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UNITED STATES OF AMERICA
BEFORE THE FEDERAL TRADE COMMISSION
OFFICE OF THE ADMINISTRATIVE LAW JUDGES
Washington, D.C.

In the Matter of **ECM BioFilms, Inc., a corporation, also d/b/a Enviroplastics International,** **Respondent.**	**Docket No. 9358** ORIGINAL **PUBLIC**

RESPONDENT ECM BIOFILM'S MOTION TO EXTEND THE WORD COUNT LIMIT

Pursuant to Rule 3.22(f), Respondent ECM Biofilms requests that the Court extend the applicable word count limit by 2,080 words for the accompanying Combined Motion. On June 30, 2014, Complaint Counsel supplied Respondent ECM with an "expert" rebuttal report from a witness Complaint Counsel never listed on Complaint Counsel's witness lists (expert or fact). Complaint Counsel intends for that witness, Dr. Michel, to testify as a rebuttal expert in this case. Dr. Michel has been a controversial source of evidence in this case. This Court sanctioned Complaint Counsel in its March 21, 2014 Order for another act of nondisclosure, the failure to disclose documents related to Dr. Michel's scientific tests. Complaint Counsel then held out Dr. Michel as one who would not testify in this case. Complaint Counsel even secured a joint stipulation from ECM that would admit his documents without need for a fact deposition. Now Complaint Counsel has abruptly reversed course by presenting a rebuttal report from Dr. Michel the day before expert discovery closed under the Scheduling Order and without any prior notice that Complaint Counsel would call Dr. Michel as an expert witness. That act violated the Rules of Practice (e.g., 16 C.F.R. § 3.31A) and this Court's several Scheduling Orders, which all required Complaint Counsel to disclose its potential experts months ago.

The surprise revelation of a completely new expert witness at this late stage in the case raises a host of issues, and ECM seeks to file at least three motions addressing each independently, to wit: (1) a Motion to Exclude Dr. Michel; (2) a Motion for Sanctions; and (3) a contingent Motion for Leave to Add a Surrebuttal Expert. The factual background and procedure overlap substantially for these motions and, so, for the Court's and Complaint Counsel's convenience, a combined motion is likely the most appropriate course. ECM cannot fully develop each of its independent bases for relief arising from the same core of operative facts within the 2,500 words provided under Rule 3.22(c). To be sure, under that rule, were three motions filed, the total pages allowed would be 7,500. Accordingly, there is good cause to grant the relief requested which enables the filing of a single pleading of only 4,580 words. ECM respectfully requests an additional 2,080 words to submit a combined memorandum of up to 4,580 words. Complaint Counsel does not consent to this motion.

Respectfully submitted,

/s/ Jonathan W. Emord
Jonathan W. Emord (jemord@emord.com)
EMORD & ASSOCIATES, P.C.
11808 Wolf Run Lane
Clifton, VA 20124
Telephone: 202-466-6937
Facsimile: 202-466-6938

DATED: July 9, 2014

STATEMENT CONCERNING MEET AND CONFER

Pursuant to Rule 3.22(g), 21 C.F.R. § 3.22(g), the undersigned counsel certifies that, on July 8, 2014, Respondent's counsel conferred with Complaint Counsel in a good faith effort to resolve by agreement the issues raised in the foregoing Motion for Leave.

/s/ Jonathan W. Emord
Jonathan W. Emord
EMORD & ASSOCIATES, P.C.
11808 Wolf Run Lane
Clifton, VA 20124
Telephone: 202-466-6937

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In the Matter of **ECM BioFilms, Inc.,** **a corporation, also d/b/a** **Enviroplastics International,** **Respondent.**	**Docket No. 9358** **PUBLIC**

[PROPOSED]
ORDER GRANTING RESPONDENT ECM BIOFILMS, INC.'S MOTION TO EXTEND THE WORD COUNT LIMIT

This matter having come before the Administrative Law Judge on July 9, 2014, upon a Motion by Respondent ECM BioFilms, and having considered all related submissions, and for good cause appearing, it is hereby ORDERED that Respondent's Motion is GRANTED.

The Court ORDERS that the word count applicable to Respondent ECM's Combined Motion to for Sanctions Under Rule 3.38, To Exclude Complaint Counsel's Rebuttal Witness, and For Leave to Include a Surrebuttal Expert is extended by 2,080 words.

ORDERED:

D. Michael Chappell
Chief Administrative Law Judge

Date:

CERTIFICATE OF SERVICE

I hereby certify that on July 9, 2014, I caused a true and correct copy of the foregoing to be served as follows:

One electronic copy to the **Office of the Secretary** through the e-filing system:

Donald S. Clark, Secretary
Federal Trade Commission
600 Pennsylvania Ave., NW, Room H-113
Washington, DC 20580
Email: secretary@ftc.gov

One electronic courtesy copy to the **Office of the Administrative Law Judge**:

The Honorable D. Michael Chappell
Administrative Law Judge
600 Pennsylvania Ave., NW, Room H-110
Washington, DC 20580

One electronic copy to **Counsel for Complainant**:

Katherine Johnson
Division of Enforcement
Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Avenue, NW
Mail stop M-8102B
Washington, D.C. 20580
Email: kjohnson3@ftc.gov

Elisa Jillson
Division of Enfoncement
Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Avenue, NW
Mail stop M-8102B
Washington, D.C. 20580
Email: ejillson@ftc.gov

Jonathan Cohen
Division of Enforcement
Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Avenue, NW
Mail stop M-8102B
Washington, D.C. 20580
Email: jcohen2@ftc.gov

Arturo Decastro
Division of Enfoncement
Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Avenue, NW
Mail stop M-8102B
Washington, D.C. 20580
Email: adecastro@ftc.gov

I certify that I retain a paper copy of the signed original of the foregoing document that is available for review by the parties and adjudicator consistent with the Commission's Rules.

Respectfully submitted,

Jonathan W. Emord (jemord@emord.com)
EMORD & ASSOCIATES, P.C.
11808 Wolf Run Lane
Clifton, VA 20124
Telephone: 202-466-6937
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Washington, D.C.

In the Matter of **ECM BioFilms, Inc.,** **a corporation, also d/b/a** **Enviroplastics International,** **Respondent.**	**Docket No. 9358** **PUBLIC**

RESPONDENT'S COMBINED MOTION FOR SANCTIONS, TO EXCLUDE COMPLAINT COUNSEL'S CONCEALED EXPERT REBUTTAL WITNESS (NOT IDENTIFIED BY NOTICE IN ACCORDANCE WITH RULE 3.31A AND THE REVISED SCHEDULING ORDER), AND FOR LEAVE TO INCLUDE SURREBUTTAL EXPERT

Pursuant to Rules 3.38(b), 3.31A(b) & (d), 3.21(c), and this Court's Revised Scheduling Order, Respondent ECM BioFilms, Inc. (ECM) moves this Court to exclude all testimony from, and the rebuttal report of, Dr. Frederick Michel, a rebuttal expert first disclosed to ECM by Complaint Counsel on June 30, 2014 at 11:46PM, fully 68 days after the deadline in the Revised Scheduling Order for disclosure of Complaint Counsel's expert list and one day before the close of expert discovery.[1] In the alternative, if this Court permits Dr. Michel to testify despite Complaint Counsel's violation of FTC Rule 3.31A[2] and this Court's Revised Scheduling Order, ECM requests leave to present the attached surrebuttal report and surrebuttal testimony from Dr. Steven A. Grossman, Professor of Plastics Engineering at the University of Massachusetts-

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[1] Per Rule 3.31A(a), ECM files this motion within five business days from the time it received Complaint Counsel's rebuttal report and Rule 3.31A disclosures, excluding weekends and holidays. As this motion seeks relief under Rule 3.38, however, the time periods in Rule 3.31A do not apply to the bulk of ECM's requested relief.

[2] Complaint Counsel never served ECM with notice as required under Rule 3.31A and the Revised Scheduling Order at 1; instead on June 30, 2014, ECM was served Dr. Michel's rebuttal report without the required notice.

Lowell. Complaint Counsel's concealment of Dr. Michel as an expert witness, in violation of Rule 3.31A and the Scheduling Order, is an act of deception, deliberately calculated to prejudice ECM. Complaint Counsel deceived ECM into thinking Dr. Michel would not testify in this matter as a fact or expert witness, even going so far as to procure a stipulation from ECM that ECM would admit certain documents offered by Dr. Michel on the assumption that he was not providing testimony. In addition, Dr. Michel's role in this case has been controversial since at least March 2014, when this Court sanctioned Complaint Counsel for failing to disclose Dr. Michel's material in response to ECM's discovery requests. *See* March 21, 2014 Order, Dkt. No. 9358. Complaint Counsel's present further act of concealment is thus part of a pattern, one not only in violation of Rule 3.31A and the Revised Scheduling Order but also in contumacious disregard of this Court's prior sanctions decision. *See* Order Granting in Part and Denying in Part Respondent's Motion for Sanctions (March 21, 2014) (Exh. P). After the decision to sanction Complaint Counsel, one would have expected circumspection and conscientious effort to ensure timely disclosures required under the rules. The present act of concealment reveals that the prior Order had no such effect and that a more potent sanction is now warranted.

Given the remaining schedule in this case and prior limits on access to correspondence between Dr. Michel and FTC and Dr. Michel and companies in competition with ECM, ECM lacks adequate time and opportunity to vet fully Dr. Michel, a witness Complaint Counsel led ECM to believe would not be called to testify in this case. The content of the Dr. Michel rebuttal report additionally reveals it to be incompetent under Rule 3.31A (it does little more than vouch for Complaint Counsel's other scientific experts and thus does not present a true rebuttal opinion under the rule). ECM has been deprived of opportunities to pursue discovery earlier in this case which, had ECM known Dr. Michel would testify as an expert, it would have pursued with vigor.

Sanctions are appropriate under Rule 3.38(b) where, as here, a party "fails to comply with any discovery obligation imposed by [the] rules." *See* 16 C.F.R. § 3.38(b). Moreover, under the Scheduling Order, where Complaint Counsel submits improper expert rebuttal, the scope of relief may include "striking Complaint Counsel's rebuttal expert reports" or providing "leave to submit surrebuttal expert reports . . ." Finally, it is certainly beneath that minimum standard of ethics and fair play for government counsel to use an overt act of legerdemain to deceive an opponent into thinking a specific person will not be called as a witness, only to in fact call the person and do so without required notice. For the reasons discussed here, the evidence presented as attachments to the memorandum, and the legal bases defined in ECM's accompanying memorandum, the preferred relief is to strike the Michel surrebuttal report and exclude Dr. Michel's testimony. If and only if the Court decides against that alternative, we ask that Dr. Grossman be allowed to present his surrebuttal report and testify as a surrebuttal expert. In either event, for the reasons explained, sanctions should be imposed on Complaint Counsel.

Respectfully submitted,

/s/ Jonathan W. Emord
Jonathan W. Emord (jemord@emord.com)
EMORD & ASSOCIATES, P.C.
11808 Wolf Run Lane
Clifton, VA 20124
Telephone: 202-466-6937
Facsimile: 202-466-6938

DATED: July 9, 2014.

UNITED STATES OF AMERICA
BEFORE THE FEDERAL TRADE COMMISSION
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Washington, D.C.

In the Matter of **ECM BioFilms, Inc.,** **a corporation, also d/b/a** **Enviroplastics International,** **Respondent.**	**Docket No. 9358** **PUBLIC**

RESPONDENT'S MEMORANDUM IN SUPPORT OF RESPONDENT'S COMBINED MOTION FOR SANCTIONS, TO EXCLUDE COMPLAINT COUNSEL'S CONCEALED EXPERT REBUTTAL WITNESS (NOT IDENTIFIED BY NOTICE IN ACCORDANCE WITH RULE 3.31A AND THE REVISED SCHEDULING ORDER), AND FOR LEAVE TO INCLUDE SURREBUTTAL EXPERT

Pursuant to Rules 3.38(b), 3.31A(b) & (d), and 3.21(c), Respondent ECM BioFilms, Inc. (ECM) hereby moves this Court to exclude testimony from Dr. Frederick Michel and to strike his purported rebuttal report. In violation of the FTC's Rules and in violation of the Revised Scheduling Order, Complaint Counsel failed to list, identify, or designate Dr. Michel as an expert witness in this case. He was never listed on Complaint Counsel's several expert and fact witness lists. Without required notice, Complaint Counsel served Michel's rebuttal report at 11:46pm Eastern on June 30th, 117 days after Complaint Counsel was first obliged to list experts per this Court's original Scheduling Order, and only 1 day before expert discovery was set to close under the Third Revised Scheduling Order. Complaint Counsel never sought leave to add Dr. Michel as a witness. This act of concealment in violation of Rule 3.31A and the Court's Revised Scheduling Order, combined with a similar act of concealment condemned in an earlier Order imposing sanctions on Complaint Counsel, warrants imposition of additional, more stringent sanctions. If and only if this Court does not exclude Dr. Michel and his rebuttal report—despite Complaint Counsel's willful violation of the discovery rules—ECM requests leave to present the surrebuttal testimony of Dr. Steven A. Grossman, a Professor of Plastics Engineering at the University of Massachusetts-Lowell.

FACTS

A. Complaint Counsel Never Listed Dr. Michel as an Expert Witness:

Complaint Counsel provided ECM with its preliminary witness list on January 17, 2014. Dr. Michel is not listed. *See* CC's Preliminary Witness List, Exh. A. On March 5, 2014, Complaint Counsel provided ECM with its expert witness list. Dr. Michel is not listed. *See* Exh. B. On March 18, 2014, Complaint Counsel supplemented its Initial Disclosures to include Dr. Michel as someone who may have relevant information. *See* Exh. C. Despite that disclosure, and on the very next day, Complaint Counsel provided ECM with a Supplemental Expert Witness List, which did not include Dr. Michel. *See* Exh. D. On April 23, 2014, Complaint Counsel reiterated that their expert witness list, which did not include Dr. Michel, "remain[ed] unchanged." *See* Exh. E. On June 11, 2014, Complaint Counsel provided ECM with its final proposed witness list, which did not include Dr. Michel in any capacity, although it listed their other experts. *See* Exh. F. To date, despite five distinct opportunities, Complaint Counsel has failed to identify, list, or designate Dr. Michel as a witness.

B. Complaint Counsel Induced ECM to Believe that Dr. Michel Was a Non-Testifying Fact Witness and Never Represented that He Would be an Expert or Rebuttal Witness:

While never listing, designating, or identifying Dr. Michel as an expert or rebuttal expert in any disclosure, Complaint Counsel did represent that it intended to depose Dr. Michel as a fact witness in May 2014. *See* Exh. G; Exh. H. At that time, Complaint Counsel secured

Respondent's stipulation to admit Dr. Michel's documents. *See* Exh. I. That stipulation accompanied several others that were designed to avoid deposition expenses where the primary purpose was to establish authenticity and admissibility of documents.[3] The parties agreed to the joint stipulation (Exh. I) which admitted Dr. Michel's documents in lieu of a fact deposition, just as they had with other testing laboratories. *See* Exh. I. That stipulation: (1) identified Dr. Michel as a fact witness (like the other testing labs) and (2) dissuaded ECM from pursuing additional discovery regarding Dr. Michel or his article. *See* Exh. H; Exh. I. Complaint Counsel represented to ECM that, but for the stipulation, they would have taken the deposition of Dr. Michel as a fact witness. Complaint Counsel did not give notice at any time that Dr. Michel would be an expert witness or expert rebuttal witness in this case. *See* Exh. H; Exh. J.

The expert deposition schedule, including eight expert depositions, was the result of extensive negotiations between the parties. *See* Exh. K. Configuring a schedule that would minimize burden and expense was difficult. *Id.* Even then, the parties were forced to extend the discovery calendar in this case beyond the deadline set in the Revised Scheduling Order to cover all of the experts. *Id.* Depositions of those experts *properly identified* continue until July 15, 2014, just 21 days before the hearing commences. *Id.* Nevertheless, despite considerable interaction on experts and scheduling, Complaint Counsel never mentioned to ECM that Dr. Michel would be an expert or rebuttal expert in this case, or that they planned to add a new witness. *See e.g.*, Exhibit H; Exhibit J. Surely Complaint Counsel knew before June 30th that it intended to have Dr. Michel as an expert, because it necessarily had to retain him as a

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[3] For instance, ECM entered the same stipulation with respect to documents produced in discovery from O.W.S. labs, another testing group.

government witness, supply him enough information and time to become familiar with the case, request and review his report.

In fact, since at least May 21, 2014, Complaint Counsel apparently anticipated using Dr. Michel as an expert witness. Complaint Counsel deposed Elsevier, the publisher of Dr. Michel's study, on two separate occasions. Those depositions concerned only Dr. Michel's articles, even though, at the time, the parties had already stipulated to the authenticity and admissibility of his study. *See* Exh. L (limiting the deposition to "[t]he [Michel] Study, including without limitation, its selection for publication, its editing, and the peer-review process"). [redacted] The possibility that Dr. Michel could serve as a rebuttal witness was certainly known to Complaint Counsel, yet withheld to achieve strategic advantage over ECM in violation of the rules, and without seeking leave.

C. Complaint Counsel's Pattern of Discovery Abuses Concerning Dr. Michel:

On February 19, 2014, Complaint Counsel produced Dr. Michel's article to ECM during a fact deposition, after having failed to disclose the article in response to ECM's document requests. *See* Order, Exh. P. When ECM moved for sanctions, Complaint Counsel falsely stated that it did not possess Dr. Michel's work until February 14, 2014, and had not communicated with Dr. Michel in any way. *See* Exh. P. Shortly thereafter, Complaint Counsel filed a "clarification"—in contradiction to Complaint Counsel's sworn statement—explaining that FTC attorneys actually did "communicate" with Dr. Michel and did receive a copy of Dr. Michel's article well before February 14, 2014. *See* Exh. Q. This Court ultimately found that

Complaint Counsel violated the discovery rules, and sanctioned them for the guerrilla "gotcha" tactics employed. *See* Exh. P.

By submitting Dr. Michel's expert rebuttal report on June 30, 2014, at 11:46 P.M. EST, and by wholly failing to list, identify, or designate Dr. Michel as an expert witness or expert rebuttal witness at any point during this litigation, Complaint Counsel essentially recommitted the very same kind of willful non-disclosure that led this Court to impose sanctions on March 21, 2014. *See* Exhs. A–F; Exh. P; Exh. R. Complaint Counsel first served Dr. Michel's "expert" production files related to his rebuttal report on July 1, 2014, a day after the deadline to submit rebuttal reports, and just one day before the deadline to conduct expert depositions lapsed.[4] *See* Exh. S (FTP Transfer Link).

ECM cannot hope to obtain full discovery of Dr. Michel at this late stage without yet another move of the hearing date (necessitating Commission intervention), as it is exceedingly difficult for ECM to prepare for Dr. Michel's testimony while simultaneously managing the remaining obligations under the Court's third revised scheduling order. Only during a meet and confer on this matter conducted on July 2, 2014 did Complaint Counsel offer Dr. Michel for deposition, providing at that late date the following unworkable dates: July 17, 18, 29, and August 1. *See* Exh. T. By July 18, the following deadlines in this case will have passed: the deadline for filing motions for *in camera* treatment; the deadline for filing objections to witness lists; the deadline for expert exhibits; and the deadline for motions *in limine* to exclude evidence.[5] ECM would likely not receive a copy of the deposition transcript until the week

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[4] As part of the previously negotiated expert deposition schedule, the parties agreed to extend this deadline for Drs. Barlaz and Volokh only.

[5] The Scheduling Order in this Case states that "[i]t shall be the responsibility of a party designating an expert witness to ensure that the expert witness is reasonably available for

before the hearing. July 29, 2014 is actually the date of the pretrial hearing, thus making that recommendation surreal. As Complaint Counsel knows, August 1st is unavailable to ECM's counsel because they will be traveling back from an appearance in an unrelated matter in the U.S. District Court for the District of Utah, a fact made known to Complaint Counsel months ago.[6] *See* Exh. BB. This Court had provided the Respondent with fully 70 days to prepare for and conduct expert depositions following Complaint Counsel's expert list. Now Complaint Counsel intends to give ECM just two weeks to do the same, all while ECM must satisfy the pre-hearing demands in the Revised Scheduling Order, an accommodation made more difficult given the agreement of the parties to conduct two additional expert depositions during those two weeks.

ARGUMENT

1. Complaint Counsel's Failure To List, Identify, Or Designate Dr. Michel As An Expert Witness or Expert Rebuttal Witness Violates The Discovery Rules, and Precludes Complaint Counsel From Calling Dr. Michel To Testify and From Offering His Rebuttal Report Into Evidence.

Complaint Counsel's non-disclosure of Dr. Michel as an expert or rebuttal expert witness is deliberately calculated to prejudice ECM, in blatant violation of Rule 3.31A and the Revised Scheduling Order and in contumacious disregard of this Court's prior Sanctions Order. Complaint Counsel has noticed a new expert witness in this case more than two months after the deadline this Court set (April 23, 2014) to identify Complaint Counsel's expert witnesses. The Revised Scheduling Order makes manifest the need for expert disclosures by April 23, 2014.[7]

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deposition *in keeping with this Scheduling Order*." Scheduling Order, at ¶ 19(c). Thus, Complaint Counsel's deposition dates would per se violate the Scheduling Order.

[6] That date is also just days before the hearing begins, and *after* ECM files its prehearing brief.

[7] Under the original Scheduling Order Issued November 21, 2013, Complaint Counsel was obliged to disclose its expert list by March 5, 2014.

His Honor could not have intended when preparing the Revised Scheduling Order to permit just two days between the time to identify *new* rebuttal experts and the cutoff date for expert depositions. The only reasonable interpretation is that Complaint Counsel was to select from a list of already identified expert witnesses.

The Rules prohibit Complaint Counsel from calling any expert witness to testify at the hearing that it failed to list on its expert witness list. Parties to an FTC administrative action are required to "serve each other with a list of experts they intend to call as witnesses at the hearing not later than 1 day after the close of fact discovery, meaning the close of discovery except for depositions and other discovery permitted under §3.24(a)(4), and discovery for purposes of authenticity and admissibility of exhibits." 16 C.F.R. § 3.31A(a).[8] Therefore, any expert not designated in the expert witness list is not permitted to testify at the evidentiary hearing, and cannot later be designated a rebuttal expert. *See In the Matter of POM Wonderful*, 2011 WL 1429882, at n. 1 (F.T.C. Apr. 5, 2011) (Chappell, A.L.J.) ("*an expert must first be designated* and provide an expert report in order to be allowed to testify") (emphasis added, citing Rule 3.31A(b)); 16 C.F.R. § 3.31A(b) ("No party may call an expert witness . . . unless he or she has been *listed* ...") (emphasis added). Consistent with Rule 3.31A(b), the only element of the Scheduling Order that involves "listing" expert witnesses is the April 23, 2014 deadline for expert witness lists.

Rule 3.31A(d) allows a party to "depose any person who has been identified as an expert and whose opinions may be presented at trial." However, the deadline to depose an expert witness in this case, "including rebuttal experts," was July 2, 2014. *See* Third Revised

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[8] The ALJ is permitted to alter those strict deadlines, which the ALJ did here by resetting the deadline for expert lists.

Scheduling Order (May 22, 2014). It is nearly impossible—and completely impractical—for Respondent to depose an expert who is named for the first time on June 30, 2014 at 11:46PM, just a day before the deadline for expert depositions expires. *Id.* The Rules and Scheduling Order therefore envision a procedural sequence wherein the parties first list "experts they intend to call as witnesses at the hearing not later than 1 day after the close of fact discovery," the parties subsequently take depositions of the listed experts, and Complaint Counsel may subsequently designate specific expert witnesses from those listed as potential experts. The Scheduling Order thus affords Complaint Counsel an opportunity to present rebuttal expert testimony, but not an opportunity to evade the disclosure requirements in Rule 3.31A.[9]

Other applicable rules support excluding Dr. Michel. First, Rule 3.31A(c) unmistakably presupposes that any expert witness who files a rebuttal or surrebuttal report must have previously filed an initial expert report: "[a] rebuttal or surrebuttal report need not include any information *already included in the initial report* of the witness." 16 C.F.R. § 3.31A(c) (emphasis added). Second, neither the rules nor the Scheduling Order place a limit on the number of experts Complaint Counsel could have placed on their initial expert list. *See* Third Revised Scheduling Order (May 22, 2014); 16 C.F.R. § 3.31A(a). In the initial list, Complaint Counsel had the option of designating at least five experts to testify at the hearing, regardless of whether those experts would testify as case-in-chief witnesses, in rebuttal, in both capacities, or not testify at all. *See* 16 C.F.R. § 3.31A(b). By failing to include Dr. Michel on their expert

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[9] The Federal Rules of Evidence also show that any argument that rebuttal experts are exempt from expert disclosure deadlines "is not supported by law." *Mente Chevrolet Oldsmobile Inc. v. GMAC*, 728 F. Supp. 2d 662, 678 (E.D. Pa. 2010).

witness list, Complaint Counsel waived its opportunity to call Dr. Michel as a rebuttal witness or submit Dr. Michel's rebuttal report without seeking leave of Court.[10]

Finally, Rule 3.31A(a) unambiguously states that "Complaint counsel *shall* serve respondents with a *list* of any rebuttal expert witnesses …". 16 C.F.R. § 3.31A(a). Complaint Counsel failed to supply Respondent with any such list. Complaint Counsel never "designated" Dr. Michel as an expert and instead ignored the notice requirement, supplying Michel's rebuttal expert report the day before the end of expert discovery; actions of that ilk are forbidden. *See In the Matter of POM Wonderful*, 2011 WL 1429882, at n. 1 (F.T.C. Apr. 5, 2011) (Chappell, A.L.J.).

Given the Scheduling Order and its deadlines for expert discovery, the text of Rule 3.31A(a), and the impracticalities that would arise otherwise, Complaint Counsel cannot be allowed to identify, for the first time, an expert a day before the deadline for expert depositions closes. Complaint Counsel undertook no effort to seek leave for that late disclosure. Instead, they simply proceeded in a cavalier manner, without respecting Rule 3.31A or the Revised Scheduling Order.

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[10] Scheduling orders in FTC actions prior to the 2009 amendments are consistent. *See e.g., In re Polypore International, Inc.*, Dkt. 9327 (Oct. 22, 2008); *In re Gemtronics, Inc.*, Dkt. No. 9330 (Oct. 28, 2008). In those illustrative cases, a similar scheduling order was issued. The orders differentiate between the expert "list" and the process of "identifying" rebuttal experts. The word "identify" does not have the same meaning as "disclose" or "list." That distinction is recognized by the Courts. The paragraph concerning rebuttal expert reports provides that Respondent has the right to seek appropriate relief for unfair rebuttals, which includes "striking Complaint Counsel's rebuttal expert *reports* or seeking leave to submit sur-rebuttal expert *reports*," but not excluding or introducing a new witness. All of these scheduling orders leave precious little time between the rebuttal report and the "deadline for depositions of experts (including rebuttal experts)," which, again, strongly suggests that the Courts have not intended for the vague language to permit surprise witnesses at the buzzer. Even the Scheduling Order in this case has a distinction between when an expert is "first listed as a witness" (¶ 19(a)) and when their "expert report is produced" (¶ 19(b)). Here Complaint Counsel has never actually "listed" Dr. Michel in any of the required disclosures, they just provided his report.

2. Complaint Counsel Should Be Sanctioned Under Rule 3.38, and that Sanction Should be the Exclusion of Dr. Michel's Report and Testimony

Sanctions under Rule 3.38(b) are appropriate where, as here, Complaint Counsel unjustifiably chose to violate Rule 3.31A and this Court's Scheduling Order to gain a strategic advantage. "Rule 3.38 is designed both to prohibit a party from resting on its own concealment and to maintain the integrity of the administrative process." *In re Grand Union Co.*, 102 F.T.C. 812 (July 18, 1983). Sanctions are appropriate under Rule 3.38 where the failure to comply was "unjustified and the sanction imposed is reasonable in light of the material withheld and the purposes of Rule 3.38(b)." *See Matter of Int'l Telephone & Telegraph Corp*, 1984 WL 565367, at *127 (F.T.C. July 25, 1984). In other words, FTC precedent makes clear that "[t]here is no good reason to allow what is essentially surprise expert testimony," and that the FTC "should be vigilant to preclude manipulative conduct designed to thwart the expert disclosure and discovery process." *In the Matter of LabMD, Inc.*, 2014 WL 2331056, at *3 (F.T.C. May 8, 2014) (citation omitted).

Complaint Counsel's concealment was calculated to maximize ECM's prejudice, leaving ECM one day to process Dr. Michel's files, prepare for a deposition, notice a deposition, and complete same, an impossibility. ECM was also deprived of an opportunity to develop discovery surrounding Dr. Michel's work. For instance, Complaint Counsel took the deposition of Elsevier on May 30, months after Complaint Counsel's expert lists were due. Elsevier was Dr. Michel's publisher. ECM would have had a different interest while participating in that deposition had it known then that Dr. Michel would be called as an expert, particularly because the Elsevier witness, who is located in Tokyo, will likely not appear at the hearing. Instead, ECM's interest in that deposition was altered by the stipulation which admitted Dr. Michel's study document into evidence without regard to authenticity and evidentiary concerns. Moreover, ECM had subpoenaed Dr. Michel for information relating to his study of various plastic products, but ECM would have performed additional discovery, including subpoenas directly to the manufacturers who provided plastics to Dr. Michel for testing and related to Dr. Michel's financial interests in dealing with those manufacturers had it known Michel would be an expert. ECM forwent those opportunities because Complaint Counsel held Dr. Michel out as a non-testifying fact witness, like other similarly situated testing laboratories. ECM even agreed to a stipulation that would admit Dr. Michel's study documents, something ECM never would have done had it known Dr. Michel would be testifying as an expert.

Adding depositions for experts at this stage presents herculean scheduling burdens and a prejudicial timetable. Dr. Michel's deposition would come after the remaining depositions, on or beyond July 17, 2014. *See* Exh. T. That would be after the following deadlines: motions for *in camera* treatment of expert exhibits; motions *in limine*; responses to motions *in limine*; responses for motions for *in camera* treatment of proposed trial exhibits; the exchange of objections to final proposed witness lists and exhibit lists; and responses to motions *in limine*.

In accordance with the rules Complaint Counsel has violated and in light of the prejudice suffered by ECM because of those rule violations, Dr. Michel and his rebuttal report should be excluded altogether.[11]

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[11] Prejudice inures not because Michel was entirely unknown but because Michel was not identified as an expert. Evaluation of a Michel study is a far cry from an evaluation of whether that specific study author is a competent expert possessed of requisite scientific acumen born of education, training, experience, publication, a lack of bias, and evidence sufficient to meet the standard of review. Many laboratories have performed tests relevant to this matter, and the parties have taken *fact* discovery from all of them. The fact that ECM possesses some of Dr. Michel's discovery files before Complaint Counsel's surprise identification of Dr. Michel himself as its expert, does not excuse Complaint Counsel's legerdemain, which would have ECM prepared to address Michel's study alone, not to vet Michel as an expert witness.

For the foregoing reasons, ECM requests that his Honor order Complaint Counsel to be reprimanded for the offense, and in addition that Dr. Michel and his report be excluded, that the Michel/Gomez article (Exh. AA) be excluded from evidence, and that the Joint Stipulation concerning that article (Exh. I) be rescinded or voided.

3. Dr. Michel Should Be Excluded and His Report Should Be Stricken Because His Testimony and Report Are Incompetent Under Rule 3.31.

"Rebuttal evidence is appropriate only if it is offered in response to evidence first presented to the court during the defendant's case." *Heatherly v. Zimmerman*, 15 F.3d 1159, at *2 (D.C. Cir. 1993). Therefore, testimony and evidence used merely to buttress prior testimony "is not proper rebuttal testimony." *Id.* Rebuttal testimony must be limited to "that which is precisely directed to rebutting *new matter or new theories* presented by the defendant's case-in-chief." *Bowman v. Gen. Motors Co.*, 427 F. Supp. 234, 240 (E.D. Pa. 1977) (emphasis added).

Here, Complaint Counsel uses Dr. Michel's report and testimony to buttress the reports and anticipated testimony of its primary scientific experts, Dr. Stephen McCarthy and Dr. Thabet Tolyamat. For example, Dr. Michel's purported rebuttal report attempts to validate Dr. McCarthy's discredited "priming effect" theory. *Compare* Dr. McCarthy's Expert Report, Exh. U, at 8, *with* Dr. Michel's Rebuttal Report, Exh. V, at 8-10. Similarly, Complaint Counsel uses Dr. Michel's rebuttal report to buttress Dr. McCarthy's theory void of published peer reviewed support that radiolabeling is the "best-known, best accepted confirmatory test" to confirm biodegradation. *Compare* Dr. McCarthy's Expert Report, at 23, *with* Dr. Michel's Report, at 12 ("To obtain accurate evidence of biodegradation, experiments are best performed using ^{14}C-labeled substrates and measuring evolved $^{14}CO_2$ over time."). Dr. Michel's purported rebuttal report also attempts to buttress Dr. Tolaymat's theory that landfills are "dry tombs" and not biologically active. *Compare, e.g.*, Dr. Michel's Rebuttal Report at 8–9, *with* Dr. Tolyamat's

Expert Report, Exhibit W, at 10–20. Dr. Michel's report is thus not rebuttal; it is principally redundant assertion of positions already taken by McCarthy and Tolyamet. The report is thus cumulative and subject to exclusion under Rule 3.31(c)(2).

4. In the Alternative, ECM Requests Leave to Add Dr. Steven A. Grossman as a Surrebuttal Witness

If and only if this Court rules that Dr. Michel's report and testimony will be allowed, ECM argues that it be allowed a surrebuttal witness, Dr. Steven A. Grossman. If that is the Court's ruling, ECM requests leave to include its own surrebuttal expert to rebut and counter the statements in Dr. Michel's expert report (including those that extend beyond areas of fair rebuttal).[12]

A copy of Dr. Grossman's anticipated surrebuttal report is attached as Exhibit X, a copy of his CV is attached as Exhibit Y, and documentary support underlying his surrebuttal report is attached as Exhibit Z. Dr. Grossman has a Ph.D. in Polymer Science and Engineering and is in the very same department at the University of Massachusetts (Lowell) as Complaint Counsel's primary expert, Dr. Stephen McCarthy. *See* Exhibit Y. Dr. Grossman has industrial and academic experience in polymer materials, polymer chemistry, and polymer structure-property relationships. *Id.* He is also a patent attorney with expertise in patent law. *Id.*

Importantly, Dr. Grossman is a colleague of Dr. McCarthy (Complaint Counsel's lead scientific expert). *See* Exh. Y. Dr. Michel's rebuttal report is inextricably intertwined in its

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[12] Rule 3.31A(b) allows a party to call more than five expert witnesses in "extraordinary circumstances." Extraordinary circumstances now exist due to the prejudice ECM will suffer if Complaint Counsel is permitted to call Dr. Michel, an unidentified expert, as a rebuttal expert. This prejudice cannot be cured but can be somewhat ameliorated by allowing ECM to designate its own surrebuttal expert. *See Sisneros v. U.S.*, 2008 WL 4170361, at *7 (D. Colo. Sept. 5, 2008) (holding in that case that "any prejudice to Defendant could be cured by allowing Defendant to designate its own experts and rebuttal experts out of time").

content with McCarthy's opinion, and Dr. Grossman has direct testimony, contained in the surrebuttal report, that reveals falsities in the representations made by Dr. McCarthy as well as scientific evidence contradicting the rebuttal reports of both Drs. Michel and McCarthy. *See* Exh. U; Exh. V; Exh. X. Dr. Grossman would address new issues first raised in Dr. McCarthy's rebuttal report, which were not raised in his original report, or in his deposition testimony. *See* Exh. CC. Allowing Dr. Grossman to participate places the parties on an equal footing with respect to discovery burdens, and reduces ECM's prejudice resulting from Complaint Counsel's concealment of Dr. Michel and failure to serve required notice.

Complaint Counsel would consent to the addition of Dr. Grossman as a surrebuttal expert, provided that they have an opportunity to depose him. *See* Exh. DD. Their offer, while fair on its face, does not address the underlying discovery abuses described above.

5. Complaint Counsel Should Be Sanctioned

Complaint Counsel has no reasonable justification for concealing the expert status of Dr. Michel, did not seek leave from this Court to excuse its conduct, and without required notice delivered its Michel Rebuttal report twenty-four hours before the expert discovery deadline, causing Respondent to suffer prejudice. This Court should not condone those rule violations and should sanction Complaint Counsel under the general authority conferred in rule 3.38(c). *See* 16 C.F.R. § 3.38(c). Here, the most appropriate relief should include, at minimum, exclusion of the Gomez & Michel study (Exh. AA), vitiation of the earlier stipulation which was based on Complaint Counsel's false pretenses (Exh. I), exclusion of Dr. Michel's expert report, and exclusion of his expert testimony. For the foregoing reasons, ECM also requests that his Honor order Complaint Counsel to be reprimanded for the offense.

RELIEF

For the foregoing reasons, ECM requests that this Court exclude Dr. Michel as a witness in this case and strike his rebuttal report. If and only if the Court chooses not to exclude Dr. Michel and his report, ECM moves for leave to permit ECM to call Dr. Steven A. Grossman as a surrebuttal witness based on the attached surrebuttal report. ECM also requests that this Court impose sanctions on Complaint Counsel for this second offense in flagrant disregard of the Court's first grant of sanctions. In particular, ECM requests that counsel be reprimanded for the offense and that in addition to the exclusion of Dr. Michel and his report that the Michel/Gomez article be excluded from evidence.

Respectfully submitted,

Jonathan W. Emord (jemord@emord.com)
EMORD & ASSOCIATES, P.C.
11808 Wolf Run Lane
Clifton, VA 20124
Telephone: 202-466-6937
Facsimile: 202-466-6938

DATED: July 9, 2014

STATEMENT CONCERNING MEET AND CONFER

Pursuant to Rule 3.22(g), 21 C.F.R. § 3.22(g), the undersigned counsel certifies that, on July 2, 214, Respondent's counsel conferred in person with Complaint Counsel in a good faith effort to resolve by agreement the issues raised in the foregoing Motion. The parties again conferred by email on July 8th. The parties have been unable to reach an agreement on the issues raised in the attached motion.

Respectfully submitted,

Jonathan W. Emord (jemord@emord.com)
EMORD & ASSOCIATES, P.C.
11808 Wolf Run Lane
Clifton, VA 20124
Telephone: 202-466-6937
Facsimile: 202-466-6938

UNITED STATES OF AMERICA
BEFORE THE FEDERAL TRADE COMMISSION
OFFICE OF THE ADMINISTRATIVE LAW JUDGES
Washington, D.C.

In the Matter of

ECM BioFilms, Inc.,
a corporation, also d/b/a
Enviroplastics International,

Respondent.

Docket No. 9358

PUBLIC

[PROPOSED] ORDER GRANTING RESPONDENT ECM BIOFILMS, INC.'S MOTION TO EXCLUDE COMPLAINT COUNSEL'S UNIDENTIFIED EXPERT REBUTALL WITNESS AND FOR SANCTIONS

This matter having come before the Administrative Law Judge on July 9, 2014, upon a Motion to Exclude Complaint Counsel's Unidentified Expert Witness, filed by Respondent ECM BioFilms, Inc. ("ECM") pursuant to Commission Rules 3.38(b), 3.31A(b) & (d), and 3.21(c)

Having considered ECM's Motion and all supporting and opposing submissions, and for good cause appearing, it is hereby ORDERED that ECM's Motion is GRANTED; Complaint Counsel's Expert Witness, Dr. Frederick Michel, is hereby excluded as a witness in this matter, and his report is stricken from the record.

ORDERED:

D. Michael Chappell
Chief Administrative Law Judge

Date:

CERTIFICATE OF SERVICE

I hereby certify that on July 9, 2014, I caused a true and correct copy of the foregoing to be served as follows:

One electronic copy to the **Office of the Secretary** through the e-filing system:

Donald S. Clark, Secretary
Federal Trade Commission
600 Pennsylvania Ave., NW, Room H-113
Washington, DC 20580
Email: secretary@ftc.gov

One electronic courtesy copy to the **Office of the Administrative Law Judge**:

The Honorable D. Michael Chappell
Administrative Law Judge
600 Pennsylvania Ave., NW, Room H-110
Washington, DC 20580

One electronic copy to **Counsel for Complainant**:

Katherine Johnson
Division of Enforcement
Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Avenue, NW
Mail stop M-8102B
Washington, D.C. 20580
Email: kjohnson3@ftc.gov

Elisa Jillson
Division of Enfoncement
Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Avenue, NW
Mail stop M-8102B
Washington, D.C. 20580
Email: ejillson@ftc.gov

Jonathan Cohen
Division of Enforcement
Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Avenue, NW
Mail stop M-8102B
Washington, D.C. 20580
Email: jcohen2@ftc.gov

Arturo Decastro
Division of Enfoncement
Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Avenue, NW
Mail stop M-8102B
Washington, D.C. 20580
Email: adecastro@ftc.gov

I certify that I retain a paper copy of the signed original of the foregoing document that is available for review by the parties and adjudicator consistent with the Commission's Rules.

Respectfully submitted,

Jonathan W. Emord (jemord@emord.com)
EMORD & ASSOCIATES, P.C.
11808 Wolf Run Lane
Clifton, VA 20124
Telephone: 202-466-6937
Facsimile: 202-466-6938

DATED: July 9, 2014

UNITED STATES OF AMERICA
BEFORE THE FEDERAL TRADE COMMISSION
OFFICE OF THE ADMINISTRATIVE LAW JUDGES
Washington, D.C.

In the Matter of **ECM BioFilms, Inc.,** **a corporation, also d/b/a** **Enviroplastics International,** **Respondent.**	**Docket No. 9358** **PUBLIC**

DECLARATION OF PETER A. ARHANGELSKY IN SUPPORT OF RESPONDENT'S COMBINED MOTION FOR SANCTIONS, TO EXCLUDE COMPLAINT COUNSEL'S CONCEALED EXPERT REBUTTAL WITNESS (NOT IDENTIFIED BY NOTICE IN ACCORDANCE WITH RULE 3.31A AND THE REVISED SCHEDULING ORDER), AND FOR LEAVE TO INCLUDE SURREBUTTAL EXPERT

In accordance with 28 U.S.C. § 1746, I declare under the penalty of perjury that the following is true and correct:

1. I am over 18 years of age, and I am a citizen of the United States. I am employed as an attorney with Emord & Associates, P.C., which represents ECM BioFilms in this matter. I am an attorney of record in the above-captioned matter, and I have personal knowledge of the facts set forth herein.

2. The following exhibits, which are attached as Exhibits to the above-captioned motion, are true and correct copies of files received by ECM:

Exh. A.	Complaint Counsel's Preliminary Witness List
Exh. B.	Complaint Counsel's Expert Witness List
Exh. C.	Complaint Counsel's Supplemental Initial Disclosures
Exh. D.	Complaint Counsel's Supplemental Expert Witness List
Exh. E.	Email Correspondence Between Complaint Counsel and ECM Counsel Re Deposition Scheduling (April 2014)
Exh. F.	Complaint Counsel's Final Proposed Witness List (June 11, 2014)

Exh. G.	Complaint Counsel's May 1, 2014 Subpoena Ad Testificandum to Dr. Frederick Michel
Exh. H.	Email Correspondence Between Complaint Counsel and ECM Counsel Re Proposed Stipulation Concerning Dr. Michel's Documents (May 2014)
Exh. I.	Joint Motion for Entry of Proposed Order Approving Stipulation As To Admissibility of Dr. Michel's Study Documents (May 5, 2014)
Exh. J.	Email Correspondence Between Complaint Counsel and ECM Counsel Re Stipulation (May 1, 2014)
Exh. K.	Email Correspondence Between Complaint Counsel and ECM Counsel Re Deposition Scheduling (May 2014)
Exh. L.	Complaint Counsel's Subpoena Ad Testificandum to Elsevier, Inc. (May 21, 2014)
Exh. M.	***Confidential*** Email Between FTC Counsel and Dr. Michel (Nov. 2012)
Exh. N.	***Confidential*** Emails Between FTC Counsel and Dr. Michel (Nov. 2012)
Exh. O.	***Confidential*** Emails Between FTC Counsel and Dr. Michel (Sept. 2013)
Exh. P.	March 21, 2014 Order Granting in Part and Denying in Part Respondent's Motion for Sanctions
Exh. Q.	Complaint Counsel's Clarification Regarding Respondent's Sanctions Motion (March 13, 2014)
Exh. R.	Email Correspondence from Complaint Counsel Disclosing Dr. Michel Rebuttal Report and Curriculum Vitae (June 30, 2014)
Exh. S.	Complaint Counsel's Production of Dr. Michel Files (July 1, 2014)
Exh. T.	Email Correspondence from Complaint Counsel Offering Dates for Dr. Michel Deposition (July 7, 2014)
Exh. U.	Expert Report of Dr. Stephen P. McCarthy (June 4, 2014)
Exh. V.	Rebuttal Report by Dr. Frederick C. Michel (June 30, 2014)
Exh. W.	Proposed Surrebuttal Report of Dr. Steven J. Grossman (July 7, 2014)
Exh. X.	Expert Report of Dr. Thabet Tolaymat (June 4, 2014)

Exh. Y.	Curriculum Vitae, Steven J. Grossman, J.D., Ph.D.
Exh. Z.	Dr. Grossman Supporting Materials
Exh. AA.	Eddie F. Gomez, Frederick C. Michel Jr., Biodegradability of conventional and bio-based plastics and natural fiber composites during composting, anaerobic digestion, and long-term soil incubation, *Polymer Degradation and Stability* 98 (2013) 2583-2591
Exh. BB.	Email between Complaint Counsel and ECM Counsel Concerning Pretrial Hearing Date (April 11, 2014)
Exh. CC.	Dr. McCarthy Rebuttal Report (June 30, 2014)
Exh. DD.	July 8, 2014 Correspondence between Complaint Counsel and ECM Counsel

Executed this 9th day of July, 2014 in Chandler, Arizona.

Peter A. Arhangelsky
Counsel to ECM Biofilms

Respondent's Exhibit A

UNITED STATES OF AMERICA
BEFORE THE FEDERAL TRADE COMMISSION

In the Matter of	)	
	)	
ECM BioFilms, Inc., **a corporation, also d/b/a** **Enviroplastics International**	)	**Docket No. 9358**

COMPLAINT COUNSEL'S PRELIMINARY WITNESS LIST

Pursuant to the Court's Scheduling Order, Complaint Counsel hereby submits its Preliminary Witness List identifying individuals who may testify in Complaint Counsel's case-in-chief (excluding expert witnesses, rebuttal witnesses and rebuttal expert witnesses) and a brief description of their proposed testimony. This list is based upon the information reasonably available to Complaint Counsel at this time.

Significantly, Respondent's failure to comply with either its Initial Disclosure obligations or Complaint Counsel's discovery requests prevents Complaint Counsel from providing a complete list or fully detailing the subjects upon which potential witnesses may testify. Among other things, Respondent did not provide its customer list to Complaint Counsel until late yesterday and still has not complied with a majority of Complaint Counsel's document requests (including the most important requests). Respondent's failures have substantially limited the information reasonably available to Complaint Counsel, and we reserve the right to amend this list as more information becomes available.[1]

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[1] Where Complaint Counsel understands that the potential witness is represented, we will provide contact information.

Complaint Counsel's Preliminary Witness List
In the Matter of ECM Biofilms, Inc., Docket No. 9358

1. Robert Sinclair, ECM Biofilms, Inc.

Counsel for ECM Biofilms, Inc.
Jonathan W. Emord
Emord & Associates, P.C.
11808 Wolf Run Lane
Clifton, VA 20124
Email: jemord@emord.com

Proposed Testimony: We anticipate that this witness will testify about ECM Biofilms, Inc. ("ECM")'s products, distribution and sales channels, financial information, customer base, customer communications, interactions with ASTM and scientific testing facilities, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

2. Thomas Nealis, ECM Biofilms, Inc.

Counsel for ECM Biofilms, Inc.
Jonathan W. Emord
Emord & Associates, P.C.
11808 Wolf Run Lane
Clifton, VA 20124
Email: jemord@emord.com

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, financial information, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

3. Ken Sullivan, ECM Biofilms, Inc.

Counsel for ECM Biofilms, Inc.
Jonathan W. Emord
Emord & Associates, P.C.
11808 Wolf Run Lane
Clifton, VA 20124
Email: jemord@emord.com

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, financial information, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

4. Scott A. Fletcher, ECM Biofilms, Inc.

Counsel for ECM Biofilms, Inc.
Jonathan W. Emord
Emord & Associates, P.C.
11808 Wolf Run Lane
Clifton, VA 20124
Email: jemord@emord.com

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

5. Michelle R. Leicher, ECM Biofilms, Inc.

Counsel for ECM Biofilms, Inc.
Jonathan W. Emord
Emord & Associates, P.C.
11808 Wolf Run Lane
Clifton, VA 20124
Email: jemord@emord.com

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

6. Kristen A. Marineau, ECM Biofilms, Inc.

Counsel for ECM Biofilms, Inc.
Jonathan W. Emord
Emord & Associates, P.C.
11808 Wolf Run Lane
Clifton, VA 20124
Email: jemord@emord.com

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

7. Vesna Barnjak, Former Employee of ECM Biofilms, Inc.

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

8. Elizabeth M. Eberly, Former Employee of ECM Biofilms, Inc.

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

9. Chessa C. Gottron, Former Employee of ECM Biofilms, Inc.

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

10. Anne C. Hassoldt-Fenoff, Former Employee of ECM Biofilms, Inc.

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

11. Kristen M. Isabel, Former Employee of ECM Biofilms, Inc.

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

12. Michelle L. Johnson, Former Employee of ECM Biofilms, Inc.

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

13. Michael J. Montesanto, Former Employee of ECM Biofilms, Inc.

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

14. Janet Morcillo, Former Employee of ECM Biofilms, Inc.

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

15. Jamie S. Morton, Former Employee of ECM Biofilms, Inc.

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

16. Alan C. Poje, Former Employee of ECM Biofilms, Inc.

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

17. Edwin J. Senter, Former Employee of ECM Biofilms, Inc.

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

18. Kathryn M. Stuber, Former Employee of ECM Biofilms, Inc.

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

19. Jean A. Sweigert, Former Employee of ECM Biofilms, Inc.

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

20. Stacy Adams, Former Employee of ECM Biofilms, Inc.

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

21. Debra K. Browning, Former Employee of ECM Biofilms, Inc.

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

22. Kelly A. Dean, Former Employee of ECM Biofilms, Inc.

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

23. Anjie Kacperski, Former Employee of ECM Biofilms, Inc.

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

24. Brenda L. Peters, Former Employee of ECM Biofilms, Inc.

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

25. Annet A. Retofsky, Former Employee of ECM Biofilms, Inc.

Proposed Testimony: We anticipate that this witness will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

26. All current and former employees of ECM Biofilms, Inc.

Proposed Testimony: We anticipate that these witnesses will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims.

27. All persons identified in ECM's redacted document production on December 27, 2013 (Bates no. ECM-FTC-648-1859).

Proposed Testimony: We anticipate that these witnesses will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims, or regarding their purchase of ECM Additive, their use of the Certificate of Biodegradability, their use of the ECM logo, and their understanding of ECM's claims of biodegradability.

28. Representatives of entities identified in ECM's redacted document production on December 27, 2013 (Bates no. ECM-FTC-648-1859).

Proposed Testimony: We anticipate that these witnesses will testify about ECM's products, distribution and sales channels, customer base, customer communications, claims of biodegradability of plastics treated with ECM products, and substantiation for those claims, or regarding their purchase of ECM Additive, their use of the Certificate of Biodegradability, their use of the ECM logo, and their understanding of ECM's claims of biodegradability.

29. Representatives of ECM's prospective customers.

Proposed Testimony: We anticipate that these witnesses will testify regarding their purchase of ECM Additive, their use of the Certificate of Biodegradability, their use of the ECM logo, their understanding of ECM's claims of biodegradability, and their understanding of ECM's claims that it had substantiation for its biodegradable claims.

30. Representatives of ECM's current customers.

Proposed Testimony: We anticipate that these witnesses will testify regarding their purchase of ECM Additive, their use of the Certificate of Biodegradability, their use of the ECM logo, and their understanding of ECM's claims of biodegradability, and their understanding of ECM's claims that it had substantiation for its biodegradable claims.

31. Jewel Vuong and/or representative of American Plastics Manufacturing, Inc.

Mark S. Leen
Inslee, Best, Doezie & Ryder, PS
Symetra Financial Center, Suite 1900
777 108th Ave. Northeast
Bellevue, WA 98004
(425) 450-4219

Proposed Testimony: We anticipate that this witness will testify regarding their purchase of ECM Additive, their use of the Certificate of Biodegradability, their use of the ECM logo, and their understanding of ECM's claims of biodegradability, and their understanding of ECM's claims that it had substantiation for its biodegradable claims.

32. Rickard J. Locke and/or representative of MacNeill Engineering Company, Inc.

Kerry Timber
Sunstein Kann Murphy & Timber LLP
125 Summer St.
Boston, MA 02110-1618
Tel: (617) 443-9292, ext. 223

Proposed Testimony: We anticipate that this witness will testify regarding their purchase of ECM Additive, their use of the Certificate of Biodegradability, their use of the ECM logo, and their understanding of ECM's claims of biodegradability, and their understanding of ECM's claims that it had substantiation for its biodegradable claims.

33. Timothy R. Barber and/or Representative of Environ International Corp.

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

34. Timothy R. Barber and/or Representative of ChemRisk

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

35. Richard Tillinger, O.W.S. Inc.

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

36. Bruno De Wilde, O.W.S. Inc.

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

37. Patrick F. Riley, O.W.S. Inc.

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

38. Representative of O.W.S. Inc.

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

39. Mike Spilde, University of New Mexico

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

40. Morton Litt, Case Western Reserve University

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

41. Paul J. Kappus, BIOPVC, Inc.

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

42. Ramani Narayan, Michigan State University

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

43. Representative of Microtech Research, Inc.

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

44. John Lake and/or Representative of Bio-Tec Environmental LLC

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

45. William W. Ulman, Northeast Laboratories, Inc.

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

46. Garrett W. Johnson, Northeast Laboratories, Inc.

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

47. Representative of Northeast Laboratories, Inc.

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

48. Thomas Poth and/or Representative of Eden Research Laboratory

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

49. Morton A. Barlaz, North Carolina State University

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

50. Representative of ASTM International

Proposed Testimony: We anticipate that this witness will testify regarding ASTM standards, ECM's claims of biodegradability, and the substantiation for such claims.

51. Any individual associated with scientific testing that ECM contends substantiates its claims.

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

52. The representative of any entity associated with scientific testing that ECM contends substantiates its claims.

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

53. Ranajit Sahu

Kelley Drye
Washington Harbour, Suite 4000
3050 K Street, NW
Washington, DC 20007
(202) 342-8588

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

54. Charles Lancelot, Plastics Environmental Council

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

55. Clifford Moriyama, Plastics Environmental Council

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

56. Representative of Plastics Environmental Council

Proposed Testimony: We anticipate that this witness will testify regarding ECM's claims of biodegradability and the substantiation for such claims.

57. Mark Benson or Representative of APCO Insight

Judith S. Sapier
Senior Vice President, General Counsel, Secretary
APCO Insight
700 12th Street NW, Suite 800
Washington, DC
(202) 779-1000

Proposed Testimony: We anticipate that this witness will testify regarding consumer perception of biodegradability claims.

58. Keith Christman or Representative of American Chemistry Council

Karen Schmidt
Assistant General Counsel
American Chemistry Council
1300 Wilson Boulevard
Arlington, VA 22209

Proposed Testimony: We anticipate that this witness will testify regarding consumer perception of biodegradability claims.

59. Representative of Synovate (acquired by Ipsos)

Proposed Testimony: We anticipate that this witness will testify regarding consumer perception of biodegradability claims.

60. John Aiken, Investigator, Federal Trade Commission

Complaint Counsel

Proposed Testimony: Authentication of exhibits.

61. William Burton, Investigator, Federal Trade Commission (retired)

Complaint Counsel

Proposed Testimony: Authentication of exhibits.

62. Representative of The National Advertising Division of the Council of Better Business Bureaus, Inc.

Proposed Testimony: We anticipate that this witness will testify regarding complaints and challenges filed against ECM or customers of ECM; any investigation and outcomes of those complaints and challenges, including any analysis of ECM's substantiation materials; resulting case reports; and authentication of exhibits.

Dated: January 17, 2014

Respectfully submitted,

Katherine Johnson (kjohnson3@ftc.gov)
Jonathan Cohen (jcohen2@ftc.gov)
Elisa Jillson (ejillson@ftc.gov)
Federal Trade Commission
600 Pennsylvania Ave., N.W. M-8102B
Washington, DC 20580
Phone: 202-326-2185; -2551; -3001
Fax: 202-326-2551

CERTIFICATE OF SERVICE

I hereby certify that on January 17, 2014, I caused a true and correct copy of the paper original of the foregoing Complaint Counsel's Preliminary Witness List to ECM BioFilms, Inc. to be served as follows:

One electronic copy to **Counsel for the Respondent:**

Jonathan W. Emord
Emord & Associates, P.C.
11808 Wolf Run Lane
Clifton, VA 20124
Email: jemord@emord.com

Peter Arhangelsky
Emord & Associates, P.C.
3210 S. Gilbert Road, Suite 4
Chandler, AZ 85286
Email: parhangelsky@emord.com

Lou Caputo
Emord & Associates, P.C.
3210 S. Gilbert Road, Suite 4
Chandler, AZ 85286
Email: lcaputo@emord.com

I further certify that I possess a paper copy of the signed original of the foregoing document that is available for review by the parties and the adjudicator.

Dated: January 17, 2014

Respectfully submitted,

Katherine Johnson (kjohnson3@ftc.gov)
Jonathan Cohen (jcohen2@ftc.gov)
Elisa Jillson (ejillson@ftc.gov)
Federal Trade Commission
600 Pennsylvania Ave., N.W. M-8102B
Washington, DC 20580
Phone: 202-326-2185; -2551; -3001
Fax: 202-326-2551

Respondent's Exhibit B

UNITED STATES OF AMERICA
BEFORE THE FEDERAL TRADE COMMISSION

In the Matter of	)	
	)	
ECM BioFilms, Inc.,	)	**Docket No. 9358**
a corporation, also d/b/a	)	
Enviroplastics International	)	

COMPLAINT COUNSEL'S EXPERT WITNESS LIST

Pursuant to the Court's Scheduling Order issued November 21, 2013 ("Scheduling Order"), Complaint Counsel hereby identifies those experts whom Complaint Counsel may call to testify in Complaint Counsel's case-in-chief or in rebuttal at the hearing on this matter. This list is based upon the information reasonably available to Complaint Counsel at this time.

1. **Stephen McCarthy, PhD**
Distinguished University Professor
Director and Professor
M2D2 and Plastics Engineering
University of Massachusetts Lowell
One University Avenue, Office: Ball 207
Lowell, MA 01854

Dr. McCarthy's anticipated testimony will be described in his expert report, which will be produced to Respondent by the date set forth in the Scheduling Order. The general subject matter of this testimony includes, but is not limited to, biodegradation generally, biodegradation of plastic polymers, ASTM tests and standards, and evaluation and analysis of Respondent's substantiation for its biodegradability claims.

2. **Thabet Tolaymet, PhD**
Environmental Protection Agency
Acting Associate, National Program Director for Emerging Materials and Sustainability
National Risk Management Research Laboratory
26 Martin Luther King Drive
Cincinnati, OH 45268

Dr. Tolaymet's anticipated testimony will be described in his expert report, which will be produced to Respondent by the date set forth in the Scheduling Order. The general subject matter of this testimony includes, but is not limited to, biodegradation generally, biodegradation of municipal solid waste generally, conditions in municipal solid waste landfills and bioreactors, and evaluation and analysis of Respondent's substantiation for its biodegradability claims.

3. **Shane Frederick, PhD**
Yale University, Yale School of Management
52 Hillhouse Avenue Room 116
New Haven, CT 06511

Dr. Frederick's anticipated testimony will be described in his expert report, which will be produced to Respondent by the date set forth in the Scheduling Order. The general subject matter of this testimony includes, but is not limited to, consumer perception of biodegradable marketing claims and Respondent's biodegradability claims.

Dated: March 5, 2014

Respectfully submitted,

/s/ Katherine Johnson
Katherine Johnson (kjohnson3@ftc.gov)
Jonathan Cohen (jcohen2@ftc.gov)
Elisa Jillson (ejillson@ftc.gov)
Federal Trade Commission
600 Pennsylvania Ave., N.W. M-8102B
Washington, DC 20580
Phone: 202-326-2185; -2551; -3001
Fax: 202-326-2551

CERTIFICATE OF SERVICE

I hereby certify that on March 5, 2014, I caused a true and correct copy of the paper original of the foregoing Complaint Counsel's Expert Witness List to ECM BioFilms, Inc. to be served as follows:

One electronic copy to **Counsel for the Respondent**:

Jonathan W. Emord
Emord & Associates, P.C.
11808 Wolf Run Lane
Clifton, VA 20124
Email: jemord@emord.com

Peter Arhangelsky
Emord & Associates, P.C.
3210 S. Gilbert Road, Suite 4
Chandler, AZ 85286
Email: parhangelsky@emord.com

Lou Caputo
Emord & Associates, P.C.
3210 S. Gilbert Road, Suite 4
Chandler, AZ 85286
Email: lcaputo@emord.com

I further certify that I possess a paper copy of the signed original of the foregoing document that is available for review by the parties and the adjudicator.

Dated: March 5, 2014

Respectfully submitted,

/s/Katherine Johnson
Katherine Johnson (kjohnson3@ftc.gov)
Jonathan Cohen (jcohen2@ftc.gov)
Elisa Jillson (ejillson@ftc.gov)
Federal Trade Commission
600 Pennsylvania Ave., N.W. M-8102B
Washington, DC 20580
Phone: 202-326-2185; -2551; -3001
Fax: 202-326-2551

Respondent's Exhibit C

UNITED STATES OF AMERICA
BEFORE THE FEDERAL TRADE COMMISSION

In the Matter of	)	
ECM BioFilms, Inc., **a corporation, also d/b/a** **Enviroplastics International**	)	**Docket No. 9358**

COMPLAINT COUNSEL'S SUPPLEMENTAL INITIAL DISCLOSURES

Under Commission Rule of Practice § 3.31(b) and (e), Complaint Counsel supplements its Initial Disclosures. The information disclosed herein is based upon information reasonably available to Complaint Counsel. Without waiving any privileges or prejudicing the ability to supplement these Supplemental Initial Disclosures if additional information becomes available, Complaint Counsel makes the following supplemental disclosures:

I. Individuals and Entities Likely To Have Discoverable Information

In addition to the individuals and entities identified in Complaint Counsel's original Initial Disclosures, we have listed in Supplemental Appendices C and D, individuals and entities that are likely to have discoverable information relevant to the allegations asserted in the Complaint, the proposed relief, or Respondent's defenses.[1] When available, Complaint Counsel has set forth each individual's or entity's name and, if known, address and telephone number.[2]

---

[1] Documents received from ECM Biofilms, Inc. ("ECM") in response to Complaint Counsel's requests may include other individuals likely to have discoverable information relevant to the allegations asserted in the Complaint, the proposed relief, or Respondent's defenses.

[2] Any contact with Federal Trade Commission ("Commission") employees must be made through Complaint Counsel.

II. Documents and Electronically Stored Information

Attached are copies of additional documents and electronically stored information in the Bureau of Consumer Protection's possession, custody, or control that are relevant to the allegations asserted in the Complaint, the proposed relief, or Respondent's defenses. Complaint Counsel is in the process of searching for additional relevant documents, except that Complaint Counsel will not provide any documents, materials, or electronically stored information subject to the limitations in § 3.31(c)(2), privileged as defined in § 3.31(c)(4), pertaining to hearing preparation as defined in § 3.31(c)(5), or pertaining to experts as defined in § 3.31A. To the extent that such documents are produced, it is without waiver of any protections or privileges.

Dated: March 17, 2014

Respectfully submitted,

/s/ Katherine Johnson
Katherine Johnson (202) 326-2185
Elisa K. Jillson (202) 326-3001
Division of Enforcement
Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Avenue, NW
Mailstop M-8102B
Washington, DC 20580

CERTIFICATE OF SERVICE

I hereby certify that on March 18, 2014, I caused a true and correct copy of the foregoing Complaint Counsel's Supplemental Initial Disclosures to be served as follows:

One electronic copy to **Counsel for the Respondent**:

Jonathan W. Emord
Emord & Associates, P.C.
11808 Wolf Run Lane
Clifton, VA 20124
Email: jemord@emord.com

Peter Arhangelsky
Emord & Associates, P.C.
3210 S. Gilbert Road, Suite 4
Chandler, AZ 85286
Email: parhangelsky@emord.com

Lou Caputo
Emord & Associates, P.C.
3210 S. Gilbert Road, Suite 4
Chandler, AZ 85286
Email: lcaputo@emord.com

/s/ Katherine Johnson
Katherine Johnson
Division of Enforcement
Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Ave., NW, M-8102B
Washington, DC 20580
Telephone: (202) 326-2185
Facsimile: (202) 326-2558
Email: kjohnson3@ftc.gov

Supplemental Appendix C

Individuals or Entities Associated with Scientific Testing

Name and Title	Contact Information	Counsel (if known)
Ohio State University	Through counsel	Jan Alan Neiger Assistant Vice President and Associate General Counsel The Ohio State University Office of Legal Affairs 1590 North High Street, Suite 500 Columbus, Ohio 43201 Phone: 614-292-0611/FAX: 614-292-8699 E-Mail: Neiger.4@osu.edu
Frederick Michel	Contact through counsel	Jan Alan Neiger Assistant Vice President and Associate General Counsel The Ohio State University Office of Legal Affairs 1590 North High Street, Suite 500 Columbus, Ohio 43201 Phone: 614-292-0611/FAX: 614-292-8699 E-Mail: Neiger.4@osu.edu
Eddie F. Gomez	Contact through counsel	Jan Alan Neiger Assistant Vice President and Associate General Counsel The Ohio State University Office of Legal Affairs 1590 North High Street, Suite 500 Columbus, Ohio 43201 Phone: 614-292-0611/FAX: 614-292-8699 E-Mail: Neiger.4@osu.edu

Supplemental Appendix D

Current and Former FTC Employees

Name and Title	Contact Information	Counsel (if known)
Kathleen Pessolano	Unknown	Complaint Counsel
Zachary Hunter	Unknown	Complaint Counsel
Matthew Wilshire, Attorney	Federal Trade Commission 600 Pennsylvania Ave., NW Mailstop M-8102B Washington DC, 20580 Tel: contact through Complaint Counsel	Complaint Counsel
David Hendrickson, Investigator	Federal Trade Commission 600 Pennsylvania Ave., NW Mailstop M-8102B Washington DC, 20580 Tel: contact through Complaint Counsel	Complaint Counsel

Respondent's Exhibit D

UNITED STATES OF AMERICA
BEFORE THE FEDERAL TRADE COMMISSION

In the Matter of	)	
	)	
ECM BioFilms, Inc.,	)	**Docket No. 9358**
a corporation, also d/b/a	)	
Enviroplastics International	)	

COMPLAINT COUNSEL'S SUPPLEMENTAL EXPERT WITNESS LIST

Pursuant to the Court's Scheduling Order issued November 13, 2013 ("Scheduling Order"), Complaint Counsel hereby identifies those experts whom Complaint Counsel may call to testify in Complaint Counsel's case-in-chief or in rebuttal at the hearing on this matter. This list is based upon the information reasonably available to Complaint Counsel at this time.

1. **Stephen McCarthy, PhD**
Distinguished University Professor
Director and Professor
M2D2 and Plastics Engineering
University of Massachusetts Lowell
One University Avenue, Office: Ball 207
Lowell, MA 01854

Dr. McCarthy's anticipated testimony will be described in his expert report, which will be produced to Respondent by the date set forth in the Scheduling Order. The general subject matter of this testimony includes, but is not limited to, biodegradation generally, biodegradation of plastic polymers, ASTM tests and standards, and evaluation and analysis of Respondent's substantiation for its biodegradability claims.

A copy of Dr. McCarthy's *curriculum vitea* follows as Attachment A, fully describing and identifying his background and qualifications, and all publications authored by him within the preceding ten years.

2. **Thabet Tolaymet, PhD**
Environmental Protection Agency
Acting Associate, National Program Director for Emerging Materials and Sustainability
National Risk Management Research Laboratory
26 Martin Luther King Drive
Cincinnati, OH 45268

Dr. Tolaymet's anticipated testimony will be described in his expert report, which will be produced to Respondent by the date set forth in the Scheduling Order. The general subject matter of this testimony includes, but is not limited to, biodegradation generally, biodegradation of municipal solid waste generally, conditions in municipal solid waste landfills and bioreactors, and evaluation and analysis of Respondent's substantiation for its biodegradability claims.

Dr. Tolaymet is an environmental engineer and researcher in the field of solid waste management. His research is focused on solid waste management, bioreactor landfills, waste containment performance, construction and demolition waste landfills, fate and transport of environmental pollutants.

He received a Bachelor of Science degree in Environmental Engineering from The University of Florida in 1997. In 2003, he received PhD in environmental engineering from the University of Florida in Gainesville, Florida. Since 2003, he has worked for the U.S. Environmental Protection Agency (EPA) Office of Research and Development as an Environmental Engineer. His research included evaluation of the performance of Solid Waste Containment Units (municipal solid waste, hazardous waste and ash mono-fill landfills), bioreactor landfills, which are new landfill design that promotes the degradation and subsequent removal of degradable fraction of solid waste as well as organic pollutants, co-disposal of solid waste and hazardous waste, and construction and demolition waste. In 2010, he led an interdisciplinary EPA evaluating sustainable materials management and Construction and Demolition Debris. Since October of 2012, he has been the Acting Associate National Program Director for Emerging Materials and Sustainability under the Chemical Safety for Sustainability research Program. In this role, he leads EPA's national research program in the area of emerging materials and sustainability. His role is to assist the national program director in these areas and set EPA's research priority and providing resources to address the agency's regulatory needs.

He is also a member of the Interstate Technology & Regulatory Council (ITRC) Alternative Landfill Technology team, a group comprised of state and federal agency representatives (*e.g.*, EPA, Department of Energy), academics, industry stakeholders, etc., that develops guidance regarding alternative landfill covers.

As part of his duties at the EPA, he provides expert advice and future direction in the area of solid waste to the Hashemite Kingdom of Jordan, Taiwan, Russia, and Hong Kong. He also provided solid waste expertise to the World Bank and USAID.

He has not testified or been deposed in the past four years.

3. **Shane Frederick, PhD**
Yale University, Yale School of Management
52 Hillhouse Avenue Room 116
New Haven, CT 06511

Dr. Frederick's anticipated testimony will be described in his expert report, which will be produced to Respondent by the date set forth in the Scheduling Order. The general subject matter of this testimony includes, but is not limited to, consumer perception of biodegradable marketing claims and Respondent's biodegradability claims.

A copy of Dr. Frederick's *curriculum vitea* follows as Attachment B, fully describing and identifying his background and qualifications, and all publications authored by him within the preceding ten years. Dr. Frederick has not testified and has not been deposed as an expert within the preceding four years.

Dated: March 18, 2014

Respectfully submitted,

/s/ Katherine Johnson
Katherine Johnson (kjohnson3@ftc.gov)
Jonathan Cohen (jcohen2@ftc.gov)
Elisa Jillson (ejillson@ftc.gov)
Federal Trade Commission
600 Pennsylvania Ave., N.W. M-8102B
Washington, DC 20580
Phone: 202-326-2185; -2551; -3001
Fax: 202-326-2551

CERTIFICATE OF SERVICE

I hereby certify that on March 19, 2014, I caused a true and correct copy of the paper original of the foregoing Complaint Counsel's Expert Witness List to ECM BioFilms, Inc. to be served as follows:

One electronic copy to **Counsel for the Respondent**:

Jonathan W. Emord
Emord & Associates, P.C.
11808 Wolf Run Lane
Clifton, VA 20124
Email: jemord@emord.com

Peter Arhangelsky
Emord & Associates, P.C.
3210 S. Gilbert Road, Suite 4
Chandler, AZ 85286
Email: parhangelsky@emord.com

Lou Caputo
Emord & Associates, P.C.
3210 S. Gilbert Road, Suite 4
Chandler, AZ 85286
Email: lcaputo@emord.com

I further certify that I possess a paper copy of the signed original of the foregoing document that is available for review by the parties and the adjudicator.

Dated: March 19, 2014

Respectfully submitted,

/s/Katherine Johnson
Katherine Johnson (kjohnson3@ftc.gov)
Jonathan Cohen (jcohen2@ftc.gov)
Elisa Jillson (ejillson@ftc.gov)
Federal Trade Commission
600 Pennsylvania Ave., N.W. M-8102B
Washington, DC 20580
Phone: 202-326-2185; -2551; -3001
Fax: 202-326-2551

Respondent’s Exhibit E

From: Johnson, Katherine
To: Peter Arhangelsky
Cc: Cohen, Jonathan; Jillson, Elisa; Lou Caputo; Jonathan Emord
Subject: Complaint Counsel"s Expert Witness List
Date: Wednesday, April 23, 2014 5:58:37 PM

Counsel: our preliminary expert witness list remains unchanged from our prior submission.

Katherine

From: Peter Arhangelsky [mailto:PArhangelsky@emord.com]
Sent: Wednesday, April 23, 2014 06:06 PM
To: Johnson, Katherine
Cc: Cohen, Jonathan; Jillson, Elisa; Lou Caputo <LCaputo@emord.com>; Jonathan Emord <JEmord@emord.com>; Millard, Joshua S.
Subject: RE: Scheduling Depositions

Katherine,

ECM will make someone available to cover a deposition in South Carolina on May 5th.

Best,

Peter

Peter A. Arhangelsky, Esq. | **Emord & Associates, P.C.** | 3210 S. Gilbert Rd., Ste 4 | Chandler, AZ 85286
Firm: (602) 388-8899 | Direct: (602) 334-4416 | Facsimile: (602) 393-4361 | www.emord.com

NOTICE: This is a confidential communication intended for the recipient listed above. The content of this communication is protected from disclosure by the attorney-client privilege and the work product doctrine. If you are not the intended recipient, you should treat this communication as strictly confidential and provide it to the person intended. Duplication or distribution of this communication is prohibited by the sender. If this communication has been sent to you in error, please notify the sender and then immediately destroy the document.

From: Johnson, Katherine [mailto:kjohnson3@ftc.gov]
Sent: Wednesday, April 23, 2014 2:25 PM
To: Peter Arhangelsky
Cc: Cohen, Jonathan; Jillson, Elisa; Lou Caputo; Jonathan Emord; Millard, Joshua S.
Subject: RE: Scheduling Depositions

Peter:

We have several more to notice and will need to coordinate with ECM and counsel for the individual parties. There are only so many days left before the close of fact discovery and we will invariably have to have more than one on a given day. In the case of D&W, it's my understanding that it is only available May 5th or 6th. So, as between those two dates, is there a preference? ECM has at least three attorneys, and possibly Mr. Sinclair himself. Surely, you can make someone available to be there on one of those two dates?

Katherine

Katherine E. Johnson, Attorney
Division of Enforcement
Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Avenue, NW
Mail stop M-8102B
Washington, DC 20580
Direct Dial: (202) 326-2185
Fax: (202) 326-2558
Email: kjohnson3@ftc.gov

From: Peter Arhangelsky [mailto:PArhangelsky@emord.com]
Sent: Wednesday, April 23, 2014 5:10 PM
To: Johnson, Katherine
Cc: Cohen, Jonathan; Jillson, Elisa; Lou Caputo; Jonathan Emord
Subject: RE: Scheduling Depositions

Hi Katherine,

The May 6th date is unavailable. You already noticed the deposition for Kappus Plastics on May 6th in New York. We therefore object to a deposition scheduled on that same day in South Carolina.

Best,

Peter A. Arhangelsky, Esq. | **Emord & Associates, P.C.** | 3210 S. Gilbert Rd., Ste 4 | Chandler, AZ 85286
Firm: (602) 388-8899 | Direct: (602) 334-4416 | Facsimile: (602) 393-4361 | www.emord.com

NOTICE: This is a confidential communication intended for the recipient listed above. The content of this communication is protected from disclosure by the attorney-client privilege and the work product doctrine. If you are not the intended recipient, you should treat this communication as strictly confidential and provide it to the person intended. Duplication or distribution of this communication is prohibited by the sender. If this communication has been sent to you in error, please notify the sender and then immediately destroy the document.

From: Johnson, Katherine [mailto:kjohnson3@ftc.gov]
Sent: Wednesday, April 23, 2014 2:02 PM
To: Peter Arhangelsky; Lou Caputo; Jonathan Emord
Cc: Cohen, Jonathan; Jillson, Elisa; Decastro, Arturo; Millard, Joshua S.
Subject: Scheduling Depositions

Peter:

Pursuant to Scheduling Order paragraph 13, we are consulting regarding scheduling of additional depositions. We will be noticing the deposition of D&W in Greenville, SC for May 6th. Please advise immediately if this date is unavailable.

Katherine

Katherine E. Johnson, Attorney
Division of Enforcement
Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Avenue, NW
Mail stop M-8102B
Washington, DC 20580
Direct Dial: (202) 326-2185
Fax: (202) 326-2558
Email: kjohnson3@ftc.gov

Respondent’s Exhibit F

UNITED STATES OF AMERICA
BEFORE THE FEDERAL TRADE COMMISSION

In the Matter of **ECM BioFilms, Inc.,** **a corporation, also d/b/a** **Enviroplastics International**	**Docket No. 9358**

COMPLAINT COUNSEL'S FINAL PROPOSED WITNESS LIST

Pursuant to the Court's Third Revised Scheduling Order, dated May 22, 2014, Complaint Counsel hereby provides its Final Proposed Witness List to Respondent ECM BioFilms, Inc. ("ECM" or "Respondent"). This list identifies the witnesses who may testify for Complaint Counsel at the hearing in this action by deposition and/or investigational hearing transcript, affidavit, declaration, or orally by live witness.

Subject to the limitations in the Scheduling Order and Third Revised Scheduling Order entered in this action, Complaint Counsel reserves the right:

A. To present testimony by deposition and/or investigational hearing transcript, affidavit, declaration, or orally by live witness, from the custodian of records of any party or non-party from whom documents or records have been obtained—specifically including, but not limited to, those parties and non-parties listed below—to the extent necessary to demonstrate the authenticity or admissibility of documents in the event a stipulation cannot be reached concerning the authentication or admissibility of such documents;

B. To present testimony by deposition and/or investigational hearing transcript, affidavit, declaration, or orally by live witness, from persons listed below and any other person that Respondent identifies as a potential witness in this action;

C. To amend this Final Proposed Witness List to be consistent with the Court's ruling on any pending motions, including any motions *in limine* filed in this matter;

D. To question the persons listed below about any topics that are the subjects of testimony by witnesses to be called by Respondent;

E. Not to present testimony by deposition and/or investigational hearing transcript, affidavit, declaration, or orally by live witness, from any of the persons listed below;

F. To question any person listed below about any other topics that the person testified about at his or her deposition or investigational hearing, or about any matter that is discussed in any documents to which the person had access and which are designated as exhibits by either party or which have been produced since the person's deposition was taken;

G. To present testimony by deposition and/or investigational hearing transcript, affidavit, declaration, or orally by live witness, from any persons, regardless whether they are listed below, to rebut the testimony of witnesses proffered by Respondent;

H. For any individual listed below as being associated with a corporation, government agency, or other non-party entity, to substitute a witness designated by the associated non-party entity; and

I. To supplement this Final Proposed Witness List in light of Respondent's Final Proposed Witness List and Exhibit List, or as circumstances may warrant.

Subject to these reservations of rights, Complaint Counsel's Final Proposed Witness List is as follows:

Current and Former ECM Employees

1. Robert Sinclair, ECM President, in his individual capacity and as ECM designee

Mr. Sinclair will testify about ECM's advertising and products, including but not limited to ECM's distribution and sales channels; financial information; customer base; communications with prospective customers, customers, former customers, ASTM, testing facilities, scientists studying the purported biodegradability of plastics, Plastics Environmental Council, and other parties regarding the ECM additive and purported biodegradability of plastic; ECM's claims of biodegradability of plastics treated with the ECM additive; ECM's substantiation for those claims; other testing regarding the ECM additive and the alleged biodegradability of plastic; facts alleged in the Complaint; any other issues addressed in his deposition (in his personal capacity and as a corporate representative of ECM); any documents introduced into evidence by Respondent or Complaint Counsel as to which he has knowledge; or any other matters as to which he has knowledge that are relevant to the allegations of the Complaint, Respondent's affirmative defenses, or the proposed relief.

2. Kenneth C. Sullivan, Jr., ECM Chief Financial Officer

Mr. Sullivan will testify about ECM's advertising and products, including but not limited to ECM's distribution and sales channels; financial information; customer base; any other issues addressed in his deposition; any documents introduced into evidence by Respondent or Complaint Counsel as to which he has knowledge, or any other matters as to which he has knowledge that are relevant to the allegations of the Complaint, Respondent's affirmative defenses, or the proposed relief.

3. Thomas Nealis, ECM Director of Sales

Mr. Nealis will testify about ECM's advertising and products, including but not limited to ECM's distribution and sales channels; customer base; communications with prospective customers, customers, former customers, ASTM, testing facilities, scientists studying the purported biodegradability of plastics, Plastics Environmental Council, and other parties regarding the ECM additive and purported biodegradability of plastic; ECM's claims of biodegradability of plastics treated with the ECM additive; ECM's substantiation for those claims; other testing regarding the ECM additive and the alleged biodegradability of plastic; facts alleged in the Complaint; any other issues addressed in his deposition; any documents introduced into evidence by Respondent or Complaint Counsel as to which he has knowledge; or any other matters as to which he has knowledge that are relevant to the allegations of the Complaint, Respondent's affirmative defenses, or the proposed relief.

4. Alan Poje, former ECM Regulatory Specialist

Mr. Poje will testify about ECM's advertising and products, including but not limited to ECM's distribution and sales channels; customer base; communications with prospective customers, customers, former customers, ASTM, testing facilities, scientists studying the purported biodegradability of plastics, Plastics Environmental Council, and other parties regarding the ECM additive and purported biodegradability of plastic; ECM's claims of biodegradability of plastics treated with the ECM additive; ECM's substantiation for those claims; other testing regarding the ECM additive and the alleged biodegradability of plastic; facts alleged in the Complaint; any other issues addressed in his deposition; any documents introduced into evidence by Respondent or Complaint Counsel as to which he has knowledge; or any other matters as to which he has knowledge that are relevant to the allegations of the Complaint, Respondent's affirmative defenses, or the proposed relief.

ECM Customers

5. Stephen Joseph, 3M Company ("3M") Staff Scientist, as 3M's designee

Mr. Joseph will testify regarding 3M's relationship with ECM and understanding of ECM's product, claims, and testing, including, but not limited to, 3M's purchase of ECM additive; reasons for this purchase; 3M's testing of the ECM additive; 3M's evaluation of ECM's claims; 3M's evaluation of ECM's substantiation for those claims; 3M's understanding of ECM's claims and substantiation for those claims; 3M's resources and ability to test and evaluate ECM's claims and substantiation; 3M's use and understanding of the ECM Certificate of Biodegradability, the ECM logo, and other ECM promotional materials; any other issues addressed in his deposition; any documents introduced into evidence by Respondent or Complaint Counsel as to which he has knowledge; or any other matters as to which he has knowledge that are relevant to the allegations of the Complaint, Respondent's affirmative defenses, or the proposed relief.

6. Ramy Samuels, A.N.S. Plastics Corp. ("ANS") Vice President, ANS's designee

Mr. Samuels will testify regarding ANS's relationship with ECM and understanding of ECM's product, claims, and testing, including, but not limited to, ANS's purchase of ECM additive; reasons for this purchase; ANS's understanding of ECM's claims and substantiation for those claims; ANS's resources and ability to test and evaluate ECM's claims and substantiation; ANS's use and understanding of the ECM Certificate of Biodegradability, the ECM logo, and other ECM promotional material; ANS's understanding of how its customers used ECM promotional materials and products made with the ECM additive; any other issues addressed in his deposition; any documents introduced into evidence by Respondent or Complaint Counsel as to which he has knowledge; or any other matters as to which he has knowledge that are relevant to the allegations of the Complaint, Respondent's affirmative defenses, or the proposed relief.

7. Robert Ringley, BER Plastics, Inc. ("BER") Vice President, as BER's designee

Mr. Ringley will testify regarding BER's relationship with ECM and understanding of ECM's product, claims, and testing, including, but not limited to, BER's purchase of ECM additive; reasons for this purchase; BER's understanding of ECM's claims and substantiation for those claims; BER's resources and ability to test and evaluate ECM's claims and substantiation; BER's use and understanding of the ECM Certificate of Biodegradability, the ECM logo, and other ECM promotional material; BER's understanding of how its customers used ECM promotional materials and products made with the ECM additive; any other issues addressed in his deposition; any documents introduced into evidence by Respondent or Complaint Counsel as to which he has knowledge; or any other matters as to which he has knowledge that are relevant to the allegations of the Complaint, Respondent's affirmative defenses, or the proposed relief.

8. Donald Kizer, D&W Fine Pack, LLC ("D&W") Purchasing Manager, as D&W's designee

Mr. Kizer will testify regarding D&W's relationship with ECM and understanding of ECM's product, claims, and testing, including, but not limited to, D&W's purchase of ECM additive; reasons for this purchase; D&W's resources and ability to test and evaluate ECM's claims and substantiation; any other issues addressed in his deposition; any documents introduced into evidence by Respondent or Complaint Counsel as to which he has knowledge; or any other matters as to which he has knowledge that are relevant to the allegations of the Complaint, Respondent's affirmative defenses, or the proposed relief.

9. Ashley Leiti, D&W Fine Pack, LLC Southeastern National Accounts Manager, as D&W's designee

Ms. Leiti will testify regarding D&W's relationship with ECM and understanding of ECM's product, claims, and testing, including, but not limited to, D&W's purchase of ECM additive; reasons for this purchase; D&W's understanding of ECM's claims and substantiation for those claims; D&W's use and understanding of the ECM Certificate of Biodegradability, the ECM logo, and other ECM promotional material; D&W's understanding of how its customers used ECM promotional materials and products made with the ECM additive; any other issues addressed in her deposition; any documents introduced into evidence by Respondent or Complaint Counsel as to which she has knowledge; or any other matters as to which she has knowledge that are relevant to the allegations of the Complaint, Respondent's affirmative defenses, or the proposed relief.

10. Frank Santana, Down To Earth All Vegetarian Organic & Natural ("Down To Earth") Marketing Director, as Down To Earth's designee

Mr. Santana will testify regarding Down To Earth's relationship with ECM and understanding of ECM's product, claims, and testing, including, but not limited to, Down To Earth's purchase of plastic bags containing the ECM additive; reasons for this purchase; Down To Earth's understanding of ECM's claims and substantiation for those claims; Down To Earth's resources and ability to test and evaluate ECM's claims and substantiation; Down To Earth's use and understanding of the ECM Certificate of Biodegradability, the ECM logo, and other ECM promotional material; any other issues addressed in his deposition; any documents introduced into evidence by Respondent or Complaint Counsel as to which he has knowledge; or any other matters as to which he has knowledge that are relevant to the allegations of the Complaint, Respondent's affirmative defenses, or the proposed relief.

11. George Collins, Eagle Film Extruders Inc. ("Eagle") President, as Eagle's designee

Mr. Collins will testify regarding Eagle's relationship with ECM and understanding of ECM's product, claims, and testing, including, but not limited to, Eagle's purchase of ECM additive; reasons for this purchase; Eagle's understanding of ECM's claims and substantiation for those claims; Eagle's resources and ability to test and evaluate ECM's claims and substantiation; Eagle's use and understanding of the ECM Certificate of Biodegradability, the ECM logo, and other ECM promotional material; Eagle's understanding of how its customers used ECM promotional materials and products made with the ECM additive; any other issues addressed in his deposition; any documents introduced into evidence by Respondent or Complaint Counsel as to which he has knowledge; or any other matters as to which he has knowledge that are relevant to the allegations of the Complaint, Respondent's affirmative defenses, or the proposed relief.

12. David Sandry, Flexible Plastics, Inc. ("Flexible") Vice President, as Flexible's designee

Mr. Sandry will testify regarding Flexible's relationship with ECM and understanding of ECM's product, claims, and testing, including, but not limited to, Flexible's purchase of ECM additive; reasons for this purchase; Flexible's understanding of ECM's claims and substantiation for those claims; Flexible's resources and ability to test and evaluate ECM's claims and substantiation; Flexible's use and understanding of the ECM Certificate of Biodegradability, the ECM logo, and other ECM promotional material; Flexible's understanding of how its customers used ECM promotional materials and products made with the ECM additive; any other issues addressed in his deposition; any documents introduced into evidence by Respondent or Complaint Counsel as to which he has knowledge; or any other matters as to which he has knowledge that are relevant to the allegations of the Complaint, Respondent's affirmative defenses, or the proposed relief.

13. James Blood, Free-Flow Packaging International, Inc. ("FP") General Counsel, as FP's designee

Mr. Blood will testify regarding FP's relationship with ECM and understanding of ECM's product, claims, and testing, including, but not limited to, FP's purchase of ECM additive; reasons for this purchase; FP's understanding of ECM's claims and substantiation for those claims; FP's resources and ability to test and evaluate ECM's claims and substantiation; FP's use and understanding of the ECM Certificate of Biodegradability, the ECM logo, and other ECM promotional material; FP's understanding of how its customers used ECM promotional materials and products made with the ECM additive; any other issues addressed in his deposition; any documents introduced into evidence by Respondent or Complaint Counsel as to which he has knowledge; or any other matters as to which he has knowledge that are relevant to the allegations of the Complaint, Respondent's affirmative defenses, or the proposed relief.

14. Adrian Hong, Island Plastic Bags, Inc. ("Island Plastic Bags") General Manager, as Island Plastic Bags' designee

Mr. Hong will testify regarding Island Plastic Bags' relationship with ECM and understanding of ECM's product, claims, and testing, including, but not limited to, Island Plastic Bags' purchase of ECM additive; reasons for this purchase; Island Plastic Bags' understanding of ECM's claims and substantiation for those claims; Island Plastic Bags' resources and ability to test and evaluate ECM's claims and substantiation; Island Plastic Bags' use and understanding of the ECM Certificate of Biodegradability, the ECM logo, and other ECM promotional material; Island Plastic Bags' understanding of how its customers (and their customers) used ECM promotional materials and products made with the ECM additive; any other issues addressed in his deposition; any documents introduced into evidence by Respondent or Complaint Counsel as to which he has knowledge; or any other matters as to which he has knowledge that are relevant to the allegations of the Complaint, Respondent's affirmative defenses, or the proposed relief.

15. Annette Gormly, Kappus Plastic Company, Inc. ("Kappus") Vice President, as Kappus' designee

Ms. Gormly will testify regarding Kappus' relationship with ECM and understanding of ECM's product, claims, and testing, including, but not limited to, Kappus' purchase of ECM additive; reasons for this purchase; Kappus' understanding of ECM's claims and substantiation for those claims; Kappus' resources and ability to test and evaluate ECM's claims and substantiation; Kappus' use and understanding of the ECM Certificate of Biodegradability, the ECM logo, and other ECM promotional material; Kappus' understanding of how its customers used ECM promotional materials and products made with the ECM additive; any other issues addressed in her deposition; any documents introduced into evidence by Respondent or Complaint Counsel as to which she has knowledge; or any other matters as to which she has knowledge that are relevant to the allegations of the Complaint, Respondent's affirmative defenses, or the proposed relief.

16. James Bean, Quest Plastics, Inc. ("Quest") President and Chief Executive Officer, as Quest's designee

Mr. Bean will testify regarding Quest's relationship with ECM and understanding of ECM's product, claims, and testing, including, but not limited to, Quest's purchase of ECM additive; reasons for this purchase; Quest's understanding of ECM's claims and substantiation for those claims; Quest's resources and ability to test and evaluate ECM's claims and substantiation; Quest's use and understanding of the ECM Certificate of Biodegradability, the ECM logo, and other ECM promotional material; Quest's understanding of how its customers used ECM promotional materials and products made with the ECM additive; any other issues addressed in his deposition; any documents introduced into evidence by Respondent or Complaint Counsel as to which he has knowledge, or any other matters as to which he has knowledge that are relevant to the allegations of the Complaint, Respondent's affirmative defenses, or the proposed relief.

Individuals and Laboratories Associated with Scientific Review or Literature

17. Thomas Poth, Eden Research Laboratory ("Eden") Lab Director, as Eden's designee

Mr. Poth will testify regarding Eden's relationship and communications with ECM, ECM's employees, ECM's customers, and other parties interested in the alleged biodegradability of plastic; Eden's testing and evaluation of plastic that purportedly contains the ECM additive; Eden's testing facilities, resources, procedures, protocols, certifications; Eden's training and supervision of its laboratory employees; the qualifications of Eden's employees; Eden's knowledge of, use of, and departure from ASTM testing standards; Eden's financial interest in testing the purported biodegradability of plastic; any other issues addressed in his deposition; any documents introduced into evidence by Respondent or Complaint Counsel as to which he has knowledge; or any other matters as to which he has knowledge that are relevant to the allegations of the Complaint, Respondent's affirmative defenses, or the proposed relief.

18. Alyssa Ullman, Northeast Laboratories, Inc. ("Northeast") Biodegradation Studies employee, as Northeast's designee

Ms. Ullman will testify regarding Northeast's relationship and communications with ECM, ECM's employees, ECM's customers, and other parties interested in the alleged biodegradability of plastic; Northeast's testing and evaluation of plastic that purportedly contains the ECM additive; Northeast's testing facilities, resources, procedures, protocols, certifications;

Northeast's training and supervision of its laboratory employees; the qualifications of Northeast's employees; Northeast's knowledge of, use of, and departure from ASTM testing standards; Northeast's financial interest in testing the purported biodegradability of plastic; any other issues addressed in her deposition; any documents introduced into evidence by Respondent or Complaint Counsel as to which she has knowledge; or any other matters as to which she has knowledge that are relevant to the allegations of the Complaint, Respondent's affirmative defenses, or the proposed relief.

19. Timothy Barber, Principal at Environ International Corp. ("Environ")

Dr. Barber will testify regarding his relationship and communications with ECM, ECM's employees, ECM's customers, and other parties interested in the alleged biodegradability of plastic; his testing and evaluation of plastic that purportedly contains the ECM additive; his testing facilities, resources, procedures, protocols, and certifications; his training and supervision of its laboratory employees; the qualifications of personnel involved in his testing of plastics; his knowledge of, use of, and departure from ASTM testing standards; his financial interest in testing the purported biodegradability of plastic; any other issues addressed in his deposition; any documents introduced into evidence by Respondent or Complaint Counsel as to which he has knowledge; or any other matters as to which he has knowledge that are relevant to the allegations of the Complaint, Respondent's affirmative defenses, or the proposed relief.

20. Tadahisa Iwata, Professor of Polymer Chemistry at the University of Tokyo and Editor for Journal of Polymer Degradation and Stability published by Elsevier Inc. ("Elsevier"), as Elsevier's designee

Professor Iwata will testify regarding the review and evaluation of the article entitled "Biodegradability of conventional and bio-based plastics and natural fiber composites during composting, anaerobic digestion and long-term soil incubation," written by Eddie F. Gomez and

Frederick C. Michel, Jr., and published by the Journal of Polymer Degradation and Stability, an Elsevier publication, including, but not limited to, conflict-of-interest checking performed on the article's authors. Professor Iwata will also testify as to any other issues addressed in his deposition; any documents introduced into evidence by Respondent or Complaint Counsel as to which he has knowledge; or any other matters as to which he has knowledge that are relevant to the allegations of the Complaint, Respondent's affirmative defenses, or the proposed relief.

Current and Former FTC Employees

21. John Aiken, Investigator, Federal Trade Commission, Bureau of Consumer Protection, Division of Marketing Practices (formerly of the Division of Enforcement)

Mr. Aiken will testify regarding the admissibility of certain website and webpage captures produced by Complaint Counsel in this action.

22. William Burton, former Investigator, Federal Trade Commission, Bureau of Consumer Protection, Division of Enforcement

Mr. Burton will testify regarding the admissibility of certain website and webpage captures produced by Complaint Counsel in this action.

23. David Hendrickson, Investigator, Federal Trade Commission, Bureau of Consumer Protection, Division of Enforcement

Mr. Hendrickson will testify regarding the admissibility of certain website and webpage captures produced by Complaint Counsel in this action.

24. Mary Jo Vantusko, Investigator, Federal Trade Commission, East Central Region

Ms. Vantusko will testify about photographs she took of various products at ECM's office in Painesville, Ohio.

Expert Witnesses

25. Steven McCarthy, Ph.D.

Dr. McCarthy is a Professor of Plastics Engineering at the University of Massachusetts Lowell (the "University"). He teaches graduate level courses in plastics engineering, including the "Mechanical Behavior of Polymers" and "Polymers and the Environment." In addition, he is the Director of the University's Biodegradable Polymer Research Center, where he orchestrates research on biodegradable polymers. He is also the Principal Investigator for studies on plastics engineering and polymer research. Dr. McCarthy has more than three decades of experience studying both the chemical and mechanical behavior of polymers, including the biodegradability of polymers used to form conventional, commercial-grade plastics.

Dr. McCarthy will testify, from his perspective as an expert in the fields of plastics engineering, polymer science, and biodegradable polymers, about whether plastic products manufactured with the ECM additive: (1) are biodegradable; (2) will completely breakdown and decompose into elements found in nature in most landfills within 5 years, and (3) will completely breakdown and decompose into elements found in any disposal environment at an appreciably faster rate and extent than conventional plastics without the ECM additives.

26. Shane Frederick, Ph.D.

Dr. Frederick is a Professor of Marketing at Yale University. His research focuses on the area of judgment and decision-making: the study of factors that affect attitudes, preferences and behavior of consumers. In addition, he has taught courses in consumer behavior, behavioral economics, and marketing, and he has both studied and published extensively concerning judgment and decision-making, with a focus on the role of cognitive abilities on preferences, preference measurement, and cognitive biases.

Dr. Frederick will testify, from his perspective as an expert in the fields of marketing and consumer decision-making, about his analysis and/or performance of studies concerning consumers' perception of biodegradable marketing claims and ECM's biodegradability claims.

27. Thabet Tolyamet, Ph.D.

Dr. Tolyamet is an Environmental Engineer and researcher in the field of solid waste management at the U.S. Environmental Protection Agency's Office of Research and Development. His research focuses on solid waste management, bioreactor landfills, waste containment performance, construction and demolition of waste landfills, and the fate and transport of environmental pollutants. A significant part of his education, training, and experience has involved conducting and evaluating tests that purport to show biodegradation and/or replicate landfill conditions.

Dr. Tolyamet will testify, from his perspective as an expert in the field of landfill design and management, about whether plastic products manufactured with ECM's additive will completely biodegrade in five years or less under ordinary U.S. landfill disposal conditions, and whether ECM testing data are not competent and reliable scientific evidence that ECM Plastics will biodegrade in five years or less in most landfills.

Dated: June 11, 2014

Respectfully submitted,

Katherine Johnson (kjohnson3@ftc.gov)
Jonathan Cohen (jcohen2@ftc.gov)
Elisa Jillson (ejillson@ftc.gov)
Federal Trade Commission
600 Pennsylvania Ave., N.W. M-8102B
Washington, DC 20580
Phone: 202-326-2185; -2551; -3001
Fax: 202-326-2551

CERTIFICATE OF SERVICE

I hereby certify that on June 11, 2014, I caused a true and correct copy of the paper original of the foregoing Complaint Counsel's Final Proposed Witness List to ECM BioFilms, Inc. to be served as follows:

One electronic copy to **Counsel for the Respondent**:

Jonathan W. Emord
Emord & Associates, P.C.
11808 Wolf Run Lane
Clifton, VA 20124
Email: jemord@emord.com

Peter Arhangelsky
Emord & Associates, P.C.
3210 S. Gilbert Road, Suite 4
Chandler, AZ 85286
Email: parhangelsky@emord.com

Lou Caputo
Emord & Associates, P.C.
3210 S. Gilbert Road, Suite 4
Chandler, AZ 85286
Email: lcaputo@emord.com

I further certify that I possess a paper copy of the signed original of the foregoing document that is available for review by the parties and the adjudicator.

Dated: June 11, 2014

Respectfully submitted,

Katherine Johnson (kjohnson3@ftc.gov)
Jonathan Cohen (jcohen2@ftc.gov)
Elisa Jillson (ejillson@ftc.gov)
Federal Trade Commission
600 Pennsylvania Ave., N.W. M-8102B
Washington, DC 20580
Phone: 202-326-2185; -2551; -3001
Fax: 202-326-2551

Respondent's Exhibit G

United States of America
FEDERAL TRADE COMMISSION

Elisa Jillson
600 Pennsylvania Ave. NW, M-8102B
Washington, DC 20580
(202) 326-3001; ejillson@ftc.gov

Katherine Johnson
600 Pennsylvania Ave. NW, M-8102B
Washington, DC 20580
(202) 326-2185; kjohnson3@ftc.gov

Jonathan Cohen
600 Pennsylvania Ave. NW, M-8102B
Washington, DC 20580
(202) 326-2551; jcohen2@ftc.gov

May 1, 2014

VIA EMAIL AND FEDERAL EXPRESS

Dr. Frederick Michel
c/o Jan Alan Neiger, Assistant Vice President and
Associate General Counsel
The Ohio State University, Office of Legal Affairs
1590 North High Street, Suite 500
Columbus, Ohio 43201

Re: ***In the Matter of ECM BioFilms, Inc, Dkt. No. 9358*** **– Deposition Subpoena**

Dear Dr. Michel:

As you know, the FTC has initiated the above-referenced administrative proceeding against ECM BioFilms. This letter notifies you that we have subpoenaed the deposition testimony of a Dr. Frederick Michel for **Tuesday, May 13, 2014** at the **United States Attorney's Columbus Ohio Office**, located at 303 Marconi Boulevard, Suite 200, Room 2074, Columbus, OH 43215. The deposition will begin at **9:00 A.M.** before an officer authorized to take depositions. *See* FTC Rules of Practice, 16 C.F.R. §§ 3.33(c)(1) & 3.34(a).

Enclosed please find the subpoena, which contains the instructions for where and when you must appear. I am also enclosing a copy of the protective order in this matter. Please call me at (202) 326-2185 if you have any questions.

Sincerely,

Katherine Johnson
EKJ

Katherine Johnson
Complaint Counsel

Enclosures

SUBPOENA AD TESTIFICANDUM
DEPOSITION

Provided by the Secretary of the Federal Trade Commission, and
Issued Pursuant to Rule 3.34(a), 16 C.F.R. § 3.34(a) (2010)

1. TO Dr. Frederick Michel c/o Jan Alan Neiger, Assistant Vice President and Associate General Counsel The Ohio State University, Office of Legal Affairs, 1590 North High Street, Suite 500, Columbus, Ohio 43201	2. FROM UNITED STATES OF AMERICA FEDERAL TRADE COMMISSION

This subpoena requires you to appear and give testimony at the taking of a deposition, at the date and time specified in Item 5, and at the request of Counsel listed in Item 8, in the proceeding described in Item 6.

3. PLACE OF DEPOSITION United States Attorney - Columbus Office 303 Marconi Blvd., Suite 200 Room 2074 Columbus, OH 43215	4. YOUR APPEARANCE WILL BE BEFORE Complaint Counsel and other designated counsel 5. DATE AND TIME OF DEPOSITION May 13, 2014 at 9:00 AM

6. SUBJECT OF PROCEEDING

In re: ECM Biofilms, Inc., Docket No. 9358

7. ADMINISTRATIVE LAW JUDGE The Honorable D. Michael Chappell Federal Trade Commission Washington, D.C. 20580	8. COUNSEL AND PARTY ISSUING SUBPOENA Complaint Counsel Katherine Johnson (202) 326-2185 Jonathan Cohen (202) 326-2551 Elisa Jillson (202) 326-3001
DATE SIGNED 5/1/2014	SIGNATURE OF COUNSEL ISSUING SUBPOENA

GENERAL INSTRUCTIONS

APPEARANCE

The delivery of this subpoena to you by any method prescribed by the Commission's Rules of Practice is legal service and may subject you to a penalty imposed by law for failure to comply.

MOTION TO LIMIT OR QUASH

The Commission's Rules of Practice require that any motion to limit or quash this subpoena must comply with Commission Rule 3.34(c), 16 C.F.R. § 3.34(c), and in particular must be filed within the earlier of 10 days after service or the time for compliance. The original and ten copies of the petition must be filed before the Administrative Law Judge and with the Secretary of the Commission, accompanied by an affidavit of service of the document upon counsel listed in Item 8, and upon all other parties prescribed by the Rules of Practice.

TRAVEL EXPENSES

The Commission's Rules of Practice require that fees and mileage be paid by the party that requested your appearance. You should present your claim to Counsel listed in Item 8 for payment. If you are permanently or temporarily living somewhere other than the address on this subpoena and it would require excessive travel for you to appear, you must get prior approval from Counsel listed in Item 8.

A copy of the Commission's Rules of Practice is available online at http://bit.ly/FTCRulesofPractice. Paper copies are available upon request.

This subpoena does not require approval by OMB under the Paperwork Reduction Act of 1980.

FTC Form 70-C (rev. 1/97)

RETURN OF SERVICE

I hereby certify that a duplicate original of the within subpoena was duly served: (check the method used)

○ *in person.*

● *by registered mail.*

○ *by leaving copy at principal office or place of business, to wit:*

Fed Ex, per FTC Rule 4.4(a)(2)

on the person named herein on:

5/1/2014
(Month, day, and year)

Elisa Jillson
(Name of person making service)

Attorney
(Official title)

ORIGINAL
FEDERAL TRADE COMMISSION
RECEIVED DOCUMENTS
567264
OCT 22 2013
SECRETARY

UNITED STATES OF AMERICA
FEDERAL TRADE COMMISSION
OFFICE OF ADMINISTRATIVE LAW JUDGES

In the Matter of)
)
ECM BioFilms, Inc.,) DOCKET NO. 9358
a corporation, also d/b/a)
Enviroplastics International,)
Respondent.)

PROTECTIVE ORDER GOVERNING DISCOVERY MATERIAL

Commission Rule 3.31(d) states: "In order to protect the parties and third parties against improper use and disclosure of confidential information, the Administrative Law Judge shall issue a protective order as set forth in the appendix to this section." 16 C.F.R. § 3.31(d). Pursuant to Commission Rule 3.31(d), the protective order set forth in the appendix to that section is attached verbatim as Attachment A and is hereby issued.

ORDERED:

DM Chappell
D. Michael Chappell
Chief Administrative Law Judge

Date: October 22, 2013

ATTACHMENT A

For the purpose of protecting the interests of the parties and third parties in the above-captioned matter against improper use and disclosure of confidential information submitted or produced in connection with this matter:

IT IS HEREBY ORDERED THAT this Protective Order Governing Confidential Material ("Protective Order") shall govern the handling of all Discovery Material, as hereafter defined.

1. As used in this Order, "confidential material" shall refer to any document or portion thereof that contains privileged, competitively sensitive information, or sensitive personal information. "Sensitive personal information" shall refer to, but shall not be limited to, an individual's Social Security number, taxpayer identification number, financial account number, credit card or debit card number, driver's license number, state-issued identification number, passport number, date of birth (other than year), and any sensitive health information identifiable by individual, such as an individual's medical records. "Document" shall refer to any discoverable writing, recording, transcript of oral testimony, or electronically stored information in the possession of a party or a third party. "Commission" shall refer to the Federal Trade Commission ("FTC"), or any of its employees, agents, attorneys, and all other persons acting on its behalf, excluding persons retained as consultants or experts for purposes of this proceeding.

2. Any document or portion thereof submitted by a respondent or a third party during a Federal Trade Commission investigation or during the course of this proceeding that is entitled to confidentiality under the Federal Trade Commission Act, or any regulation, interpretation, or precedent concerning documents in the possession of the Commission, as well as any information taken from any portion of such document, shall be treated as confidential material for purposes of this Order. The identity of a third party submitting such confidential material shall also be treated as confidential material for the purposes of this Order where the submitter has requested such confidential treatment.

3. The parties and any third parties, in complying with informal discovery requests, disclosure requirements, or discovery demands in this proceeding may designate any responsive document or portion thereof as confidential material, including documents obtained by them from third parties pursuant to discovery or as otherwise obtained.

4. The parties, in conducting discovery from third parties, shall provide to each third party a copy of this Order so as to inform each such third party of his, her, or its rights herein.

5. A designation of confidentiality shall constitute a representation in good faith and after careful determination that the material is not reasonably believed to be already in the public domain and that counsel believes the material so designated constitutes confidential material as defined in Paragraph 1 of this Order.

6. Material may be designated as confidential by placing on or affixing to the document containing such material (in such manner as will not interfere with the legibility thereof), or if an entire folder or box of documents is confidential by placing or affixing to that folder or box, the designation "CONFIDENTIAL – FTC Docket No. 9358" or any other appropriate notice that identifies this proceeding, together with an indication of the portion or portions of the document considered to be confidential material. Confidential information contained in electronic documents may also be designated as confidential by placing the designation "CONFIDENTIAL – FTC Docket No. 9358" or any other appropriate notice that identifies this proceeding, on the face of the CD or DVD or other medium on which the document is produced. Masked or otherwise redacted copies of documents may be produced where the portions deleted contain privileged matter, provided that the copy produced shall indicate at the appropriate point that portions have been deleted and the reasons therefor.

7. Confidential material shall be disclosed only to: (a) the Administrative Law Judge presiding over this proceeding, personnel assisting the Administrative Law Judge, the Commission and its employees, and personnel retained by the Commission as experts or consultants for this proceeding; (b) judges and other court personnel of any court having jurisdiction over any appellate proceedings involving this matter; (c) outside counsel of record for any respondent, their associated attorneys and other employees of their law firm(s), provided they are not employees of a respondent; (d) anyone retained to assist outside counsel in the preparation or hearing of this proceeding including consultants, provided they are not affiliated in any way with a respondent and have signed an agreement to abide by the terms of the protective order; and (e) any witness or deponent who may have authored or received the information in question.

8. Disclosure of confidential material to any person described in Paragraph 7 of this Order shall be only for the purposes of the preparation and hearing of this proceeding, or any appeal therefrom, and for no other purpose whatsoever, provided, however, that the Commission may, subject to taking appropriate steps to preserve the confidentiality of such material, use or disclose confidential material as provided by its Rules of Practice; sections 6(f) and 21 of the Federal Trade Commission Act; or any other legal obligation imposed upon the Commission.

9. In the event that any confidential material is contained in any pleading, motion, exhibit or other paper filed or to be filed with the Secretary of the Commission, the Secretary shall be so informed by the Party filing such papers, and such papers shall be filed *in camera*. To the extent that such material was originally submitted by a third party, the party including the materials in its papers shall immediately notify the submitter of such inclusion. Confidential material contained in the papers shall continue to have *in camera* treatment until further order of the Administrative Law Judge, provided, however, that such papers may be furnished to persons or entities who may receive confidential material pursuant to Paragraphs 7 or 8. Upon or after filing any paper containing confidential material, the filing party shall file on the public record a duplicate copy of the paper that does not reveal confidential material. Further, if the protection for any such material expires, a party may file on the public record a duplicate copy which also contains the formerly protected material.

10. If counsel plans to introduce into evidence at the hearing any document or transcript containing confidential material produced by another party or by a third party, they shall provide advance notice to the other party or third party for purposes of allowing that party to seek an order that the document or transcript be granted *in camera* treatment. If that party wishes *in camera* treatment for the document or transcript, the party shall file an appropriate motion with the Administrative Law Judge within 5 days after it receives such notice. Except where such an order is granted, all documents and transcripts shall be part of the public record. Where *in camera* treatment is granted, a duplicate copy of such document or transcript with the confidential material deleted therefrom may be placed on the public record.

11. If any party receives a discovery request in any investigation or in any other proceeding or matter that may require the disclosure of confidential material submitted by another party or third party, the recipient of the discovery request shall promptly notify the submitter of receipt of such request. Unless a shorter time is mandated by an order of a court, such notification shall be in writing and be received by the submitter at least 10 business days before production, and shall include a copy of this Protective Order and a cover letter that will apprise the submitter of its rights hereunder. Nothing herein shall be construed as requiring the recipient of the discovery request or anyone else covered by this Order to challenge or appeal any order requiring production of confidential material, to subject itself to any penalties for non-compliance with any such order, or to seek any relief from the Administrative Law Judge or the Commission. The recipient shall not oppose the submitter's efforts to challenge the disclosure of confidential material. In addition, nothing herein shall limit the applicability of Rule 4.11(e) of the Commission's Rules of Practice, 16 CFR 4.11(e), to discovery requests in another proceeding that are directed to the Commission.

12. At the time that any consultant or other person retained to assist counsel in the preparation of this action concludes participation in the action, such person shall return to counsel all copies of documents or portions thereof designated confidential that are in the possession of such person, together with all notes, memoranda or other papers containing confidential information. At the conclusion of this proceeding, including the exhaustion of judicial review, the parties shall return documents obtained in this action to their submitters, provided, however, that the Commission's obligation to return documents shall be governed by the provisions of Rule 4.12 of the Rules of Practice, 16 CFR 4.12.

13. The provisions of this Protective Order, insofar as they restrict the communication and use of confidential discovery material, shall, without written permission of the submitter or further order of the Commission, continue to be binding after the conclusion of this proceeding.

CERTIFICATE OF SERVICE

I hereby certify that on May 1, 2014, I caused a true and correct copy of the foregoing document to be served by email to **Counsel for the Respondent:**

Jonathan W. Emord
Emord & Associates, P.C.
11808 Wolf Run Lane
Clifton, VA 20124
Email: jemord@emord.com

Peter Arhangelsky
Emord & Associates, P.C.
3210 S. Gilbert Road, Suite 4
Chandler, AZ 85286
Email: parhangelsky@emord.com

Lou Caputo
Emord & Associates, P.C.
3210 S. Gilbert Road, Suite 4
Chandler, AZ 85286
Email: lcaputo@emord.com

Dated: May 1, 2014

Respectfully submitted,

Elisa

Katherine Johnson (kjohnson3@ftc.gov)
Jonathan Cohen (jcohen2@ftc.gov)
Elisa Jillson (ejillson@ftc.gov)
Federal Trade Commission
600 Pennsylvania Ave., N.W. M-8102B
Washington, DC 20580
Phone: 202-326-2185; -2551; -3001
Fax: 202-326-2551

Respondent's Exhibit H

From: Johnson, Katherine
To: Peter Arhangelsky
Cc: Cohen, Jonathan; Jillson, Elisa; Lou Caputo; Jonathan Emord
Subject: RE: Scheduling Depositions
Date: Monday, May 05, 2014 1:49:18 PM
Attachments: Proposed Stipulation re Ohio State Study.pdf

Peter:

As discussed, attached is our proposed stipulation concerning the Ohio State Study. Please review and let me know whether we have your permission to sign on your behalf and file it tomorrow.

Katherine

Katherine E. Johnson, Attorney
Division of Enforcement
Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Avenue, NW
Mail stop M-8102B
Washington, DC 20580
Direct Dial: (202) 326-2185
Fax: (202) 326-2558
Email: kjohnson3@ftc.gov

From: Peter Arhangelsky [mailto:PArhangelsky@emord.com]
Sent: Friday, May 02, 2014 4:52 PM
To: Johnson, Katherine
Cc: Cohen, Jonathan; Jillson, Elisa; Lou Caputo; Jonathan Emord
Subject: RE: Scheduling Depositions

Hi Katherine,

I can inform you now that we will formally agree to stipulations with regard to Dr. Michel's documents in lieu of the deposition, as we did with OWS. Please let me know if you have any questions, or if you need to discuss.

Thanks,

Peter

Peter A. Arhangelsky, Esq. | **Emord & Associates, P.C.** | 3210 S. Gilbert Rd., Ste 4 | Chandler, AZ 85286
Firm: (602) 388-8899 | Direct: (602) 334-4416 | Facsimile: (602) 393-4361 | www.emord.com

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document.

From: Johnson, Katherine [mailto:kjohnson3@ftc.gov]
Sent: Thursday, May 01, 2014 5:22 AM
To: Peter Arhangelsky
Cc: Cohen, Jonathan; Jillson, Elisa; Lou Caputo; Jonathan Emord
Subject: RE: Scheduling Depositions

That's fine, Peter. But we are going to issue the subpoena to Dr. Michel so that we have everything in place if agreement cannot be reached.

Katherine E. Johnson, Attorney
Division of Enforcement
Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Avenue, NW
Mail stop M-8102B
Washington, DC 20580
Direct Dial: (202) 326-2185
Fax: (202) 326-2558
Email: kjohnson3@ftc.gov

From: Peter Arhangelsky [mailto:PArhangelsky@emord.com]
Sent: Wednesday, April 30, 2014 3:36 PM
To: Johnson, Katherine
Cc: Cohen, Jonathan; Jillson, Elisa; Lou Caputo; Jonathan Emord
Subject: RE: Scheduling Depositions

Katherine,

We think a stipulation agreement for Dr. Michel likely, but we cannot confirm presently without our client's authorization. Bob Sinclair is in transit today, and we have not been able to reach him. Given that we are sanguine about an agreement, may we advise you of the client's position on or before Monday next?

Best,

Peter A. Arhangelsky, Esq. | **Emord & Associates, P.C.** | 3210 S. Gilbert Rd., Ste 4 | Chandler, AZ 85286
Firm: (602) 388-8899 | Direct: (602) 334-4416 | Facsimile: (602) 393-4361 | www.emord.com

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From: Johnson, Katherine [mailto:kjohnson3@ftc.gov]
Sent: Wednesday, April 30, 2014 9:15 AM
To: Peter Arhangelsky
Cc: Cohen, Jonathan; Jilison, Elisa; Lou Caputo; Jonathan Emord
Subject: RE: Scheduling Depositions

Peter:

I'm glad we're in agreement over OWS. I will notify Christine Haaker, OWS's counsel that we will not conduct the deposition and I will file the Joint Motion today.

As for Northeast Labs, upon further consideration, we have decided that it's best to conduct the deposition. Northeast is scheduled for May 9th in New Haven, CT, which is the day that I cleared with Northeast.

Lastly, you did not state whether ECM would be willing to enter into a similar stipulation regarding Dr. Michel. if I hear nothing further, the deposition will be scheduled for Tuesday, May 13th, in Columbus, OH. Or whether you agree to the other proposed depositions: Flexible Plastics and 3M, both to take place in the St. Paul/Minneapolis, MN area, on May 15th and 16th respectively.

Katherine

Katherine E. Johnson, Attorney
Division of Enforcement
Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Avenue, NW
Mail stop M-8102B
Washington, DC 20580
Direct Dial: (202) 326-2185
Fax: (202) 326-2558
Email: kjohnson3@ftc.gov

From: Peter Arhangelsky [mailto:PArhangelsky@emord.com]
Sent: Tuesday, April 29, 2014 6:01 PM
To: Johnson, Katherine
Cc: Cohen, Jonathan; Jillson, Elisa; Lou Caputo; Jonathan Emord
Subject: RE: Scheduling Depositions

Katherine:

We agree with the stipulation concerning OWS, provided that you agree unequivocally to withdraw or withhold any subpoena ad testificandum for OWS. We request that you consider the same stipulation for Northeast Labs' documents. We are mindful, however, that certain NE Labs documents have been provided from various third parties; not all responsive documents have come from Northeast Labs itself. Nonetheless, if this type of stipulation is something you would consider, we can send a packet of NE Labs documents that would be subject to the proposed stipulation.

Best,

Peter A. Arhangelsky, Esq. | **Emord & Associates, P.C.** | 3210 S. Gilbert Rd., Ste 4 | Chandler, AZ 85286
Firm: (602) 388-8899 | Direct: (602) 334-4416 | Facsimile: (602) 393-4361 | www.emord.com

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From: Johnson, Katherine [mailto:kjohnson3@ftc.gov]
Sent: Tuesday, April 29, 2014 1:06 PM
To: Peter Arhangelsky
Cc: Cohen, Jonathan; Jillson, Elisa; Lou Caputo; Jonathan Emord
Subject: RE: Scheduling Depositions

Peter:

Attached is the proposed stipulation on the OWS production. Please let me know by the midday tomorrow if I have your permission to execute and file with the Court.

We will send out the deposition subpoenas for OWS and the other depositions I mentioned previously by 5 p.m. tomorrow.

Katherine

Katherine E. Johnson, Attorney
Division of Enforcement
Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Avenue, NW
Mail stop M-8102B
Washington, DC 20580
Direct Dial: (202) 326-2185
Fax: (202) 326-2558
Email: kjohnson3@ftc.gov

From: Johnson, Katherine
Sent: Monday, April 28, 2014 1:51 PM
To: 'Peter Arhangelsky'
Cc: Cohen, Jonathan; Jillson, Elisa; Lou Caputo; Jonathan Emord
Subject: RE: Scheduling Depositions

Peter:

The stipulation for OWS would have to include a waiver of all hearsay objections – OWS's statements (e.g., emails) and documents (e.g., tests) would come in for their truth. If that is acceptable, I can send you a proposed draft by tomorrow.

If ECM is considering deposing NE Labs, then perhaps we can just schedule the deposition. I spoke with them last week and cleared May 9th.

We are also proposing to depose Eagle Film, Thursday, May 15th with a backup of Wednesday, May 14th. The depo would be in Grand Rapids, Michigan. Let me know which date works better.

If there are other potential deponents, please put them on the table now, so we can work out possible stipulations or deposition dates. Another potential deponent for us would be Dr. Michel, unless ECM stipulates to the Ohio State Study coming in for the truth.

Lastly, I see that Mr. Sinclair filed his notice of appearance. Please let me know if you would like him copied on our correspondence and served with filings in the case.

Katherine

Katherine E. Johnson, Attorney
Division of Enforcement
Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Avenue, NW
Mail stop M-8102B
Washington, DC 20580
Direct Dial: (202) 326-2185
Fax: (202) 326-2558
Email: kjohnson3@ftc.gov

From: Peter Arhangelsky [mailto:PArhangelsky@emord.com]
Sent: Friday, April 25, 2014 9:15 PM
To: Johnson, Katherine
Cc: Cohen, Jonathan; Jillson, Elisa; Lou Caputo; Jonathan Emord
Subject: RE: Scheduling Depositions

Katherine,

I apologize for the delay responding to your below email. ECM is willing to enter stipulations in lieu of an OWS deposition. We are contemplating taking the deposition of Northeast Labs, although we might be willing to consider a similar stipulation for documents supplied by them.

Please let us know if you want to discuss this.

Best,

Peter A. Arhangelsky, Esq. | **Emord & Associates, P.C.** | 3210 S. Gilbert Rd., Ste 4 | Chandler, AZ 85286
Firm: (602) 388-8899 | Direct: (602) 334-4416 | Facsimile: (602) 393-4361 | www.emord.com

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From: Johnson, Katherine [mailto:kjohnson3@ftc.gov]
Sent: Thursday, April 24, 2014 8:04 AM
To: Peter Arhangelsky
Cc: Cohen, Jonathan; Jillson, Elisa; Lou Caputo; Jonathan Emord
Subject: RE: Scheduling Depositions

Thanks Peter.

We will be sending out a few more deposition subpoenas in the next week. In advance of that, I wanted to see whether ECM would be willing to enter into a stipulation to avoid the cost of conducting multiple additional depositions. I believe that Jonathan and you discussed the possibility of deposing OWS previously. Will ECM be noticing that deposition? If not, then perhaps we can stipulate to the authenticity and admissibility of the documents produced by OWS to avoid having to conduct that deposition? Otherwise, then we're looking to depose OWS the week of May 12th, depending on its availability.

In addition, we would like to know whether ECM is planning to issue any of its own deposition subpoenas. If so, it would be nice to know which ones and proposed dates so that we could coordinate for the remainder and avoid double-booking the rest of the days in May. At this point only the week of the 12th remains before the close of fact discovery.

If it would be easier, we could arrange another call about this.

Thanks,
Katherine

Katherine E. Johnson, Attorney
Division of Enforcement
Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Avenue, NW
Mail stop M-8102B
Washington, DC 20580
Direct Dial: (202) 326-2185
Fax: (202) 326-2558

Email: kjohnson3@ftc.gov

From: Peter Arhangelsky [mailto:PArhangelsky@emord.com]
Sent: Wednesday, April 23, 2014 6:07 PM
To: Johnson, Katherine
Cc: Cohen, Jonathan; Jillson, Elisa; Lou Caputo; Jonathan Emord; Millard, Joshua S.
Subject: RE: Scheduling Depositions

Katherine,

ECM will make someone available to cover a deposition in South Carolina on May 5th.

Best,

Peter

Peter A. Arhangelsky, Esq. | **Emord & Associates, P.C.** | 3210 S. Gilbert Rd., Ste 4 | Chandler, AZ 85286
Firm: (602) 388-8899 | Direct: (602) 334-4416 | Facsimile: (602) 393-4361 | www.emord.com

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From: Johnson, Katherine [mailto:kjohnson3@ftc.gov]
Sent: Wednesday, April 23, 2014 2:25 PM
To: Peter Arhangelsky
Cc: Cohen, Jonathan; Jillson, Elisa; Lou Caputo; Jonathan Emord; Millard, Joshua S.
Subject: RE: Scheduling Depositions

Peter:

We have several more to notice and will need to coordinate with ECM and counsel for the individual parties. There are only so many days left before the close of fact discovery and we will invariably have to have more than one on a given day. In the case of D&W, it's my understanding that it is only available May 5th or 6th. So, as between those two dates, is there a preference? ECM has at least three attorneys, and possibly Mr. Sinclair himself. Surely, you can make someone available to be there on one of those two dates?

Katherine

Katherine E. Johnson, Attorney
Division of Enforcement
Bureau of Consumer Protection
Federal Trade Commission

600 Pennsylvania Avenue, NW
Mail stop M-8102B
Washington, DC 20580
Direct Dial: (202) 326-2185
Fax: (202) 326-2558
Email: kjohnson3@ftc.gov

From: Peter Arhangelsky [mailto:PArhangelsky@emord.com]
Sent: Wednesday, April 23, 2014 5:10 PM
To: Johnson, Katherine
Cc: Cohen, Jonathan; Jillson, Elisa; Lou Caputo; Jonathan Emord
Subject: RE: Scheduling Depositions

Hi Katherine,

The May 6th date is unavailable. You already noticed the deposition for Kappus Plastics on May 6th in New York. We therefore object to a deposition scheduled on that same day in South Carolina.

Best,

Peter A. Arhangelsky, Esq. | **EMORD & ASSOCIATES, P.C.** | 3210 S. Gilbert Rd., Ste 4 | Chandler, AZ 85286
Firm: (602) 388-8899 | Direct: (602) 334-4416 | Facsimile: (602) 393-4361 | www.emord.com

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From: Johnson, Katherine [mailto:kjohnson3@ftc.gov]
Sent: Wednesday, April 23, 2014 2:02 PM
To: Peter Arhangelsky; Lou Caputo; Jonathan Emord
Cc: Cohen, Jonathan; Jillson, Elisa; Decastro, Arturo; Millard, Joshua S.
Subject: Scheduling Depositions

Peter:

Pursuant to Scheduling Order paragraph 13, we are consulting regarding scheduling of additional depositions. We will be noticing the deposition of D&W in Greenville, SC for May 6th. Please advise immediately if this date is unavailable.

Katherine

Katherine E. Johnson, Attorney
Division of Enforcement
Bureau of Consumer Protection
Federal Trade Commission

600 Pennsylvania Avenue, NW
Mail stop M-8102B
Washington, DC 20580
Direct Dial: (202) 326-2185
Fax: (202) 326-2558
Email: kjohnson3@ftc.gov

Respondent’s Exhibit I

UNITED STATES OF AMERICA
BEFORE THE FEDERAL TRADE COMMISSION

In the Matter of	)	
ECM BioFilms, Inc., **a corporation, also d/b/a** **Enviroplastics International**	)	**Docket No. 9358** **PUBLIC DOCUMENT**

JOINT MOTION FOR ENTRY OF PROPOSED ORDER APPROVING STIPULATION AS TO ADMISSIBILITY OF CERTAIN EVIDENCE

Pursuant to Commission Rule of Practice 3.31 (f) and 3.43(b)-(c), Complaint Counsel and Respondent ECM Biofilms ("ECM") respectfully request that the Court enter the attached proposed order based on the parties' stipulations regarding certain documents. Specifically, both parties stipulate and agree as follows:

1. The document produced by The Ohio State University in this matter entitled Gómez, Eddie & Michel Jr., Frederick, *Biodegradability of Conventional and Bio-Based Plastics and Natural Fiber Composites During Composting, Anaerobic Digestion, and Long-Term Soil Incubation,* POLYMER DEGRADATION AND STABILITY, Vol. 98 (December 2013): 2583-2591 (the "Ohio State Study"), attached hereto as **Exhibit CX-1**, shall be admitted into evidence at the evidentiary hearing to take place in this matter.
2. The Ohio State Study is admissible for the truth of the matters asserted therein and for all other purposes and not excludable on any grounds.
3. The parties reserve their rights to present evidence or argument that goes to the probative value or evidentiary weight to be accorded to the Ohio State Study.

Dated: May 5, 2014

Respectfully submitted,

/s/ Katherine Johnson
Katherine Johnson (kjohnson3@ftc.gov)
Jonathan Cohen (jcohen2@ftc.gov)
Elisa Jillson (ejillson@ftc.gov)
Federal Trade Commission
600 Pennsylvania Ave., N.W. M-8102B
Washington, DC 20580
Phone: 202-326-2185; -2551; -3001
Fax: 202-326-2551

And

/s/ Peter Arhangelsky
Jonathan W. Emord (jemord@emord.com)
Peter A. Arhangelsky
(parhangelsky@emord.com)
Lou F. Caputo (lcaputo@emord.com)
Emord & Associates, P.C.
11808 Wolf Run Lane, Clifton, VA 20124
Phone: (202) 388-8899
Fax: (202) 466-6938

UNITED STATES OF AMERICA
BEFORE THE FEDERAL TRADE COMMISSION

In the Matter of **ECM BioFilms, Inc.,** **a corporation, also d/b/a** **Enviroplastics International**	**Docket No. 9358** **PUBLIC DOCUMENT**

[PROPOSED] ORDER GRANTING JOINT MOTION APPROVING STIPULATION AS TO ADMISSIBILITY OF CERTAIN EVIDENCE

This matter having come before the Chief Administrative Law Judge on May 5, 2014, upon a Joint Motion for Entry of Proposed Order Based on Stipulations Regarding Admissibility of Certain Evidence, it is hereby **ORDERED** that:

1. The Joint Motion is **GRANTED**;
2. The document produced by The Ohio State University in this matter entitled Gómez, Eddie & Michel Jr., Frederick, *Biodegradability of Conventional and Bio-Based Plastics and Natural Fiber Composites During Composting, Anaerobic Digestion, and Long-Term Soil Incubation,* POLYMER DEGRADATION AND STABILITY, Vol. 98 (December 2013): 2583-2591 (the "Ohio State Study"), attached hereto as **Exhibit CX-1**, shall be admitted into evidence at the evidentiary hearing to take place in this matter.
3. The Ohio State Study is admissible for the truth of the matters asserted therein and for all other purposes and not excludable on any grounds.
4. The parties reserve their rights to present evidence or argument that goes to the probative value or evidentiary weight to be accorded to the Ohio State Study.

ORDERED:

____
D. Michael Chappell

Chief Administrative Law Judge

Date:

CERTIFICATE OF SERVICE

I hereby certify that on May 5, 2014, I caused a true and correct copy of the foregoing document to be served as follows:

One hard copy original and one courtesy copy to the **Office of the Secretary:**

Donald S. Clark, Secretary
Federal Trade Commission
600 Pennsylvania Ave., NW, Room H-159
Washington, DC 20580
Email: secretary@ftc.gov

One electronic and one paper courtesy copy to the **Office of the Administrative Law Judge:**

The Honorable D. Michael Chappell
Administrative Law Judge
600 Pennsylvania Ave., NW, Room H-110
Washington, DC 20580

One electronic copy to **Counsel for the Respondent**:

Jonathan W. Emord
Emord & Associates, P.C.
11808 Wolf Run Lane
Clifton, VA 20124
Email: jemord@emord.com

Peter Arhangelsky
Emord & Associates, P.C.
3210 S. Gilbert Road, Suite 4
Chandler, AZ 85286
Email: parhangelsky@emord.com

Lou Caputo
Emord & Associates, P.C.
3210 S. Gilbert Road, Suite 4
Chandler, AZ 85286
Email: lcaputo@emord.com

I further certify that I possess a paper copy of the signed original of the foregoing document that is available for review by the parties and the adjudicator.

Date: May 5, 2014

/s/ Katherine Johnson
Katherine Johnson (kjohnson3@ftc.gov)
Jonathan Cohen (jcohen2@ftc.gov)
Elisa Jillson (ejillson@ftc.gov)
Federal Trade Commission
600 Pennsylvania Ave., N.W. M-8102B
Washington, DC 20580
Phone: 202-326-2185; -2551; -3001
Fax: 202-326-2551

Respondent’s Exhibit J

Eric Awerbuch

From: Jillson, Elisa <ejillson@ftc.gov>
Sent: Thursday, May 01, 2014 6:58 AM
To: Lou Caputo; Peter Arhangelsky; Jonathan Emord
Cc: Johnson, Katherine; Cohen, Jonathan
Subject: ECM, No. 9358 - subpoena
Attachments: MX-M503N_20140501_091022.pdf

Counsel,

Please find attached a copy of the subpoena for Dr. Michel's deposition. If the parties reach an agreement about a stipulation that obviates the need for Dr. Michel's deposition, we will cancel the deposition.

Elisa K. Jillson
Attorney
Division of Enforcement, Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Avenue, NW, M-8102B
Washington, DC 20580
Phone: 202.326.3001
Fax: 202.326.2558

ECM Comb. Mot. to Exclude
Exh. J

Respondent's Exhibit K

Eric Awerbuch

From: Cohen, Jonathan <jcohen2@ftc.gov>
Sent: Wednesday, May 21, 2014 11:48 AM
To: Peter Arhangelsky
Cc: Johnson, Katherine; Jillson, Elisa; Lou Caputo; Jonathan Emord
Subject: RE: ECM Biofilms, No. 9358

Peter,

The EPA can host in Cincinnati.

Are we all set?

Jonathan Cohen
Enforcement Division | Bureau of Consumer Protection | Federal Trade Commission
600 Pennsylvania Avenue, N.W., M-8102B Washington, D.C. 20580
(202) 326-2551 | jcohen2@ftc.gov

From: Peter Arhangelsky [mailto:PArhangelsky@emord.com]
Sent: Wednesday, May 21, 2014 12:52 PM
To: Cohen, Jonathan
Cc: Johnson, Katherine; Jillson, Elisa; Lou Caputo; Jonathan Emord
Subject: RE: ECM Biofilms, No. 9358

Jonathan,

Thank you for your flexibility. We need to identify locations for our depositions of your asserted experts. We propose the FTC building at 400 7th Street for Dr. McCarthy on June 27th. Given the government's resources, we ask that you identify a suitable location for your experts in Cincinnati and Connecticut. Because we need to serve our Notices of Deposition soon, we may need to designate your experts' local offices until further notice. Please advise.

Thanks,

Peter A. Arhangelsky, Esq. | **EMORD & ASSOCIATES, P.C.** | 3210 S. Gilbert Rd., Ste 4 | Chandler, AZ 85286
Firm: (602) 388-8899 | Direct: (602) 334-4416 | Facsimile: (602) 393-4361 | www.emord.com

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From: Cohen, Jonathan [mailto:jcohen2@ftc.gov]
Sent: Monday, May 19, 2014 7:38 PM
To: Peter Arhangelsky
Cc: Johnson, Katherine; Jillson, Elisa; Lou Caputo; Jonathan Emord
Subject: RE: ECM Biofilms, No. 9358

Per the below.

Jonathan Cohen
Enforcement Division | Bureau of Consumer Protection | Federal Trade Commission
600 Pennsylvania Avenue, N.W., M-8102B Washington, D.C. 20580
(202) 326-2551 | jcohen2@ftc.gov

From: Cohen, Jonathan
Sent: Monday, May 19, 2014 3:52 PM
To: 'Peter Arhangelsky'
Cc: Johnson, Katherine; Jillson, Elisa; 'Lou Caputo'; 'Jonathan Emord'
Subject: RE: ECM Biofilms, No. 9358

Peter,

We accept this compromise schedule, which is what you most recently proposed:

June 23: Frederick (New Haven, CT)

June 24: Tolymat (Cincinnati, OH)

June 27: McCarthy (Washington, D.C.)

June 30: Sahu (Los Angeles, CA)

July 1: Stewart (Los Angeles, CA)

July 2: Burnette (Washington, D.C.)

July 14: Barlaz (Raleigh, NC)

July 15: Volokh (Atlanta, GA) (10:00 AM start/ECM guarantees a full day)

Jonathan Cohen
Enforcement Division | Bureau of Consumer Protection | Federal Trade Commission
600 Pennsylvania Avenue, N.W., M-8102B Washington, D.C. 20580
(202) 326-2551 | jcohen2@ftc.gov

From: Peter Arhangelsky [mailto:PArhangelsky@emord.com]
Sent: Friday, May 16, 2014 12:34 PM
To: Cohen, Jonathan
Cc: Johnson, Katherine; Jillson, Elisa; Lou Caputo; Jonathan Emord
Subject: RE: ECM Biofilms, No. 9358

Jonathan,

I think the schedule unnecessarily burdens ECM because it would require excessive travel. Your proposal separates the California depositions, which would require two separate trips to California instead of one. You have also scheduled Dr. Sahu on a date we did not say he was available. He is not available the week of July 14th, so his date will need to change. We have no objection to scheduling Burnette for July 2nd, thus separating the Washington, D.C. pair, and preventing witness depositions out of order. We are counter-proposing the following dates (swapping Barlaz with Sahu):

June 23: Frederick (New Haven, CT)

June 24: Tolymat (Cincinnati, OH)

June 27: McCarthy (Washington, D.C.)

June 30: Sahu (Los Angeles, CA)

July 1: Stewart (Los Angeles, CA)

July 2: Burnette (Washington, D.C.)

July 14: Barlaz (Raleigh, NC)

July 15 or 16: Volokh (Atlanta, GA) (10:00 AM start as long as ECM guarantees a full day)

We think this is a reasonable compromise. You would have a full day with Volokh, although a July 16th date might make more sense given the flights available. I am available to discuss today if necessary.

Thanks,

Peter

Peter A. Arhangelsky, Esq. | **EMORD & ASSOCIATES, P.C.** | 3210 S. Gilbert Rd., Ste 4 | Chandler, AZ 85286
Firm: (602) 388-8899 | Direct: (602) 334-4416 | Facsimile: (602) 393-4361 | www.emord.com

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From: Cohen, Jonathan [mailto:jcohen2@ftc.gov]
Sent: Thursday, May 15, 2014 3:47 PM
To: Peter Arhangelsky
Cc: Johnson, Katherine; Jillson, Elisa; Lou Caputo; Jonathan Emord
Subject: RE: ECM Biofilms, No. 9358

Peter,

This isn't perfect, but it accommodates your scheduling issues, as well as scheduling issues on our end. With respect to our experts, we should be able to make rooms available locally for all three depositions, but I'll need until next week to confirm that (it's very likely). Please let me know if we have an agreement.

June 23: Frederick (New Haven, CT)

June 24: Tolymat (Cincinnati, OH)

June 27: McCarthy (Washington, D.C.)

June 30: Barlaz (Raleigh, NC)

July 1: Stewart (Los Angeles, CA)

July 2: Burnette (Washington, D.C.)

July 14: Sahu (Los Angeles, CA)

July 15: Volokh (Atlanta, GA) (10:00 AM start as long as ECM guarantees a full day)

Jonathan Cohen
Enforcement Division | Bureau of Consumer Protection | Federal Trade Commission
600 Pennsylvania Avenue, N.W., M-8102B Washington, D.C. 20580

(202) 326-2551 | jcohen2@ftc.gov

From: Peter Arhangelsky [mailto:PArhangelsky@emord.com]
Sent: Thursday, May 15, 2014 5:46 PM
To: Cohen, Jonathan
Cc: Johnson, Katherine; Jillson, Elisa; Lou Caputo; Jonathan Emord
Subject: RE: ECM Biofilms, No. 9358

Jonathan,

Per our conversation this afternoon, ECM proposes the following deposition schedule:

June 23: Frederick (New Haven, CT)
June 24: Tolymat (Cincinnati, OH)
June 26: Burnette (Washington, D.C.)
June 27: McCarthy (Washington, D.C.)
June 30: Sahu (Los Angeles, CA)
July 1: Stewart (Los Angeles, CA)
July 14: Barlaz (Raleigh, NC)
July 16 (or July 15): Volokh (Atlanta, GA)

With respect to your witnesses, please let us know if we can designate a local office in New Haven, Cincinnati, and Washington, D.C. Although Dr. Volokh is available on July 15th, we would prefer July 16th to eliminate potential travel issues.

Let me know if we need to discuss this further.

Thanks,

Peter A. Arhangelsky, Esq. | **EMORD & ASSOCIATES, P.C.** | 3210 S. Gilbert Rd., Ste 4 | Chandler, AZ 85286
Firm: (602) 388-8899 | Direct: (602) 334-4416 | Facsimile: (602) 393-4361 | www.emord.com

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From: Cohen, Jonathan [mailto:jcohen2@ftc.gov]
Sent: Thursday, May 15, 2014 1:07 PM
To: Peter Arhangelsky
Cc: Jonathan Emord; Lou Caputo; Jillson, Elisa; Johnson, Katherine; Decastro, Arturo
Subject: ECM Biofilms, No. 9358

We've provided ECM with proposed dates for our experts, and when ECM responded with a request that we provide different dates for our experts within a 12-day period ECM selected, we accommodated that request in less than a day. However, with respect to the dates for ECM's experts that we proposed several days ago, we haven't heard anything back. Accordingly, we're noticing them per the attached document. Of course, we're still willing to negotiate the schedule.

I'll give you a call regarding the *Colplast* materials in about an hour.

Jonathan Cohen
Enforcement Division | Bureau of Consumer Protection | Federal Trade Commission
600 Pennsylvania Avenue, N.W., M-8102B Washington, D.C. 20580
(202) 326-2551 | jcohen2@ftc.gov

From: Cohen, Jonathan
Sent: Wednesday, May 14, 2014 11:21 AM
To: 'Peter Arhangelsky'
Cc: 'Jonathan Emord'; 'Lou Caputo'; Jillson, Elisa; Johnson, Katherine; Cohen, Jonathan; Decastro, Arturo
Subject: RE: Expert Deposition Schedule

Peter,

Per your request, we revise the proposal as follows:

June 23: Frederick (New Haven, CT)
June 24: Tolymat (Cincinnati, OH)
June 27: McCarthy (Washington, D.C.)
July 9 – Burnette (Washington, DC)
July 10 – Stewart (Los Angeles, CA)
July 11 – Sahu (Los Angeles, CA)
July 15 – Volokh (Atlanta, GA)
July 16 – Barlaz (Raleigh, NC)

Accordingly, this accomodates ECM's request that we provide dates for our experts between June 16 and June 27. Please confirm the rest of the schedule, or let us know when you'll be able to do so, as we'd like to begin making arrangements.

Best,

Jonathan Cohen
Enforcement Division | Bureau of Consumer Protection | Federal Trade Commission
600 Pennsylvania Avenue, N.W., M-8102B Washington, D.C. 20580
(202) 326-2551 | jcohen2@ftc.gov

From: Peter Arhangelsky [mailto:PArhangelsky@emord.com]
Sent: Tuesday, May 13, 2014 2:21 PM
To: Cohen, Jonathan
Cc: Jonathan Emord; Lou Caputo; Jillson, Elisa; Johnson, Katherine
Subject: RE: Expert Deposition Schedule

Jonathan,

Thanks for your proposal. We will discuss the schedule with our experts and report back shortly. Having discussed this with our team, we have conflicts the first week of July which will require changes to the proposed schedule. Please inform us when your experts are available between June 16-27. We will need to schedule your experts during that window in June.

Best,

Peter A. Arhangelsky, Esq. | **EMORD & ASSOCIATES, P.C.** | 3210 S. Gilbert Rd., Ste 4 | Chandler, AZ 85286
Firm: (602) 388-8899 | Direct: (602) 334-4416 | Facsimile: (602) 393-4361 | www.emord.com

NOTICE: This is a confidential communication intended for the recipient listed above. The content of this communication is protected from disclosure by the attorney-client privilege and the work product doctrine. If you are not the intended recipient, you should treat this communication as strictly confidential and provide it to the person intended. Duplication or distribution of this communication is prohibited by the sender. If this communication has been sent to you in error, please notify the sender and then immediately destroy the document.

From: Cohen, Jonathan [mailto:jcohen2@ftc.gov]
Sent: Tuesday, May 13, 2014 11:43 AM
To: Peter Arhangelsky; Lou Caputo; Jonathan Emord
Cc: Jillson, Elisa; Johnson, Katherine; Decastro, Arturo
Subject: RE: Expert Deposition Schedule

Peter,

One change with respect to the dates we propose for your experts:

July 2 – McCarthy (Washington, DC)
July 3 – Thabet (Cincinnati, OH)
July 8 – Frederick (New Haven, CT)
July 9 – Burnette (Washington, DC)
July 10 – Stewart (Los Angeles, CA)
July 11 – Sahu (Los Angeles, CA)
July 15 – Volokh (Atlanta, GA)
July 16 – Barlaz (Raleigh, NC)

Jonathan Cohen
Enforcement Division | Bureau of Consumer Protection | Federal Trade Commission
600 Pennsylvania Avenue, N.W., M-8102B Washington, D.C. 20580
(202) 326-2551 | jcohen2@ftc.gov

From: Cohen, Jonathan
Sent: Monday, May 12, 2014 5:43 PM
To: 'Peter Arhangelsky'; 'Lou Caputo'; 'Jonathan Emord'
Cc: Jillson, Elisa; Johnson, Katherine; Decastro, Arturo
Subject: ECM: Expert Deposition Schedule

Peter,

Per our call, we have cleared dates with our experts. Based on those dates, we propose the following overall schedule:

July 2 – McCarthy (Washington, DC)
July 3 – Thabet (Cincinnati, OH)
July 8 – Frederick (New Haven, CT)
July 9 – Burnette (Washington, DC)

July 10 – Stewart (Los Angeles, CA)
July 11 – Sahu (Los Angeles, CA)
July 14 – Barlaz (Raleigh, NC)
July 15 – Volokh (Atlanta, GA)

Please let us know whether this works. Hopefully, getting this set soon will make the process easier on everyone.

Thanks,

Jonathan Cohen
Enforcement Division | Bureau of Consumer Protection | Federal Trade Commission
600 Pennsylvania Avenue, N.W., M-8102B Washington, D.C. 20580
(202) 326-2551 | jcohen2@ftc.gov

Respondent’s Exhibit L

PLAINTIFF'S EXHIBIT
Elsevier 1

SUBPOENA AD TESTIFICANDUM
DEPOSITION

Provided by the Secretary of the Federal Trade Commission, and Issued Pursuant to Rule 3.34(a), 16 C.F.R. § 3.34(a) (2010)

1. TO	2. FROM
Elsevier Inc. c/o Nicholas Tardif, Corporate Counsel 360 Park Avenue South New York, NY 10010	UNITED STATES OF AMERICA FEDERAL TRADE COMMISSION

This subpoena requires you to appear and give testimony at the taking of a deposition, at the date and time specified in Item 5, and at the request of Counsel listed in Item 8, in the proceeding described in Item 6.

3. PLACE OF DEPOSITION	4. YOUR APPEARANCE WILL BE BEFORE
600 Pennsylvania Avenue, N.W. M8102B Washington, D.C. 20580 (TELEPHONIC/WITH TRANSLATOR)	Complaint Counsel and other designated counsel
	5. DATE AND TIME OF DEPOSITION
	8:00 PM EST May ~~[illegible]~~ 30, 2014 JC

6. SUBJECT OF PROCEEDING

In re: ECM Biofilms, Inc., Docket No. 9358

Please see attached schedule for deposition topics pursuant to 16 C.F.R. 3.33(c)(1)

7. ADMINISTRATIVE LAW JUDGE	8. COUNSEL AND PARTY ISSUING SUBPOENA
The Honorable D. Michael Chappell Federal Trade Commission Washington, D.C. 20580	Complaint Counsel Katherine Johnson (202) 326-2185 Jonathan Cohen (202) 326-2551 Elisa Jillson (202) 326-3001

DATE SIGNED	SIGNATURE OF COUNSEL ISSUING SUBPOENA
5/21/14	

GENERAL INSTRUCTIONS

APPEARANCE

The delivery of this subpoena to you by any method prescribed by the Commission's Rules of Practice is legal service and may subject you to a penalty imposed by law for failure to comply.

MOTION TO LIMIT OR QUASH

The Commission's Rules of Practice require that any motion to limit or quash this subpoena must comply with Commission Rule 3.34(c), 16 C.F.R. § 3.34(c), and in particular must be filed within the earlier of 10 days after service or the time for compliance. The original and ten copies of the petition must be filed before the Administrative Law Judge and with the Secretary of the Commission, accompanied by an affidavit of service of the document upon counsel listed in Item 8, and upon all other parties prescribed by the Rules of Practice.

TRAVEL EXPENSES

The Commission's Rules of Practice require that fees and mileage be paid by the party that requested your appearance. You should present your claim to Counsel listed in Item 8 for payment. If you are permanently or temporarily living somewhere other than the address on this subpoena and it would require excessive travel for you to appear, you must get prior approval from Counsel listed in Item 8.

A copy of the Commission's Rules of Practice is available online at http://bit.ly/FTCRulesofPractice. Paper copies are available upon request.

This subpoena does not require approval by OMB under the Paperwork Reduction Act of 1980.

FTC Form 70-C (rev. 1/97)

RETURN OF SERVICE

I hereby certify that a duplicate original of the within subpoena was duly served: *(check the method used)*

○ *in person.*

○ *by registered mail.*

● *by leaving copy at principal office or place of business, to wit:*

Fed Ex, per FTC Rule 4.4(a)(2)

on the person named herein on:

(Month, day, and year)

(Name of person making service)

(Official title)

UNITED STATES OF AMERICA
BEFORE THE FEDERAL TRADE COMMISSION

In the Matter of **ECM BioFilms, Inc.,** **a corporation, also d/b/a** **Enviroplastics International**	)))))))	**Docket No. 9358**

COMPLAINT COUNSEL'S NOTICE OF RULE 3.33(c)(1) DEPOSITION

To: Elsevier Inc.

DEFINITIONS

A. "**Include**" and "**including**" mean "without limitation," or "including but not limited to," so as to avoid excluding any documents or information that might otherwise be construed to be within the scope of any specification.

B. "**Study**" refers to Gómez, Eddie & Michel Jr., Frederick, *Biodegradability of Conventional and Bio-Based Plastics and Natural Fiber Composites During Composting, Anaerobic Digestion, and Long-Term Soil Incubation,* POLYMER DEGRADATION AND STABILITY, Vol. 98 (December 2013): 2583-2591.

C. "**You**" and "**Your**" means Elsevier Inc. ("Elsevier") or ("the Company") along with any affiliates, successors, predecessors, entities You acquired, entities You control, and entities whose information You control.

INSTRUCTIONS

A. **Protective Order:** On October 22, 2013, the Court entered an order governing discovery material in this matter. A copy of the Protective Order is attached hereto as Exhibit A with instructions on the handling of confidential information.

B. **Petitions to Limit or Quash:** Pursuant to Commission Rule of Practice 3.34(c), any motion to limit or quash this subpoena must be filed within ten days of service hereof.

DEPOSITION TOPIC

PLEASE TAKE NOTICE that Complaint Counsel will depose Elsevier, upon oral examination, pursuant to Rules §§ 3.33(c)(1) and 3.34(a), as to the matters set forth below:

The Study, including, without limitation, its selection for publication, its editing, and the peer-review process.

Katherine Johnson (kjohnson3@ftc.gov)
Jonathan Cohen (jcohen2@ftc.gov)
Elisa Jillson (ejillson@ftc.gov)
Federal Trade Commission
600 Pennsylvania Ave., N.W. M-8102B
Washington, DC 20580
Phone: 202-326-2185; -2551; -3001
Fax: 202-326-2551

CERTIFICATE OF SERVICE

I hereby certify that on May 21, 2014, I caused a true and correct copy of the foregoing document to be served by email to **Counsel for the Respondent**:

Jonathan W. Emord
Emord & Associates, P.C.
11808 Wolf Run Lane
Clifton, VA 20124
Email: jemord@emord.com

Peter Arhangelsky
Emord & Associates, P.C.
3210 S. Gilbert Road, Suite 4
Chandler, AZ 85286
Email: parhangelsky@emord.com

Lou Caputo
Emord & Associates, P.C.
3210 S. Gilbert Road, Suite 4
Chandler, AZ 85286
Email: lcaputo@emord.com

Dated: May 21, 2014

Respectfully submitted,

Katherine Johnson (kjohnson3@ftc.gov)
Jonathan Cohen (jcohen2@ftc.gov)
Elisa Jillson (ejillson@ftc.gov)
Federal Trade Commission
600 Pennsylvania Ave., N.W. M-8102B
Washington, DC 20580
Phone: 202-326-2185; -2551; -3001
Fax: 202-326-2551

Respondent's Exhibit M

CONFIDENTIAL

Respondent's Exhibit N

CONFIDENTIAL

Respondent’s Exhibit O

CONFIDENTIAL

Respondent’s Exhibit P

PUBLIC

UNITED STATES OF AMERICA
FEDERAL TRADE COMMISSION
OFFICE OF ADMINISTRATIVE LAW JUDGES

In the Matter of ECM BioFilms, Inc., a corporation, also d/b/a Enviroplastics International, Respondent.	))))))))	DOCKET NO. 9358

ORDER GRANTING IN PART AND DENYING IN PART RESPONDENT'S MOTION FOR SANCTIONS

On February 28, 2014, Respondent ECM BioFilms, Inc. ("Respondent" or "ECM") filed a Motion for Sanctions for the asserted failure of Federal Trade Commission ("FTC") Complaint Counsel to supplement its document production "in a timely manner," pursuant to FTC Rule 3.31(e)(2) ("Motion"). 16 C.F.R. § 3.31(e)(2). Complaint Counsel filed an opposition to the Motion on March 10, 2014 ("Opposition"), and a Clarification Regarding Respondent's Motion for Sanctions on March 13, 2014.

On March 12, 2014, Respondent filed a Motion for Leave to Supplement its Motion for Sanctions by submitting a complete transcript of the ECM deposition, in lieu of deposition excerpts as originally filed as RX-A to the Motion. As of the date of this Order, no opposition to that motion has been filed. Respondent's Motion for Leave is GRANTED, and citations to the deposition transcript shall refer to RX-A as supplemented by Respondent.

Having fully reviewed and considered the Motion, Opposition, and Clarification, and the exhibits thereto, the Motion is GRANTED IN PART AND DENIED IN PART, as more fully explained below.

I. Background

The Motion is based upon Complaint Counsel's use of a document at the deposition of ECM, via its designee, ECM's Chief Executive Officer Robert Sinclair, which took place on February 18 and 19, 2014. Motion RX-A (Transcript of Deposition (hereafter "Tr.")). The document, marked as Deposition Exhibit 23, is a published article titled, "Biodegradability of Conventional and Bio-Based Plastics and Natural Fiber Composites During Composting, Anaerobic Digestion and Long-Term Soil Incubation," by Eddie F. Gomez and Frederick C. Michel, Jr., who are associated with Ohio State Agricultural Research and Development Center (the "Ohio State Article" or "Article"). Tr. 366. The Ohio State Article was published on October 1, 2013, in a journal entitled *Polymer Degradation and Stability*, 98 (2013) 2583-2591.

Id.; Opposition CCX-A ¶ 5, Attachment 1.

Based on the parties' representations and the exhibits offered in support thereof, on Friday, February 14, 2014, at approximately 8:00 p.m., Complaint Counsel received a copy of the Ohio State Article. Declaration of Jonathan Cohen, Opposition CCX-A (hereafter, "Cohen Decl.") ¶ 6. Although Mr. Cohen states that the article was received "unsolicited," *Id.* ¶ 6, he does not disclose how, or from whom, the Ohio State Article was received. The Declaration further states that Complaint Counsel had not "communicated in any way" with any of the authors of the Ohio State Article, or anyone at Ohio State. Cohen Decl. ¶ 7.

Complaint Counsel's March 13, 2014 "Clarification" regarding the Motion states that on the previous day, Complaint Counsel learned that "two FTC attorneys (not representing Complaint Counsel)," one of whom worked on the investigative phase of the case but who has since left the FTC, had, in fact, received a draft of the Ohio State Article. The Clarification does not state when the draft was received.[1] According to the Clarification, the two attorneys had been working with Mr. Michel "as a consulting expert on unrelated matters involving biodegradability claims other companies asserted" and received the Article in connection with that work. Clarification at 1 and n.1.

On the afternoon of February 15, 2014, the day after Complaint Counsel received the Ohio State Article, Complaint Counsel Johnson packed for her travel to ECM's offices, where the deposition was scheduled to take place, taking the Ohio State Article with her. Tr. 374-375. February 17, 2014 was President's Day, a federal holiday. The deposition commenced on Tuesday morning, February 18, 2014. Tr. 1. After recessing for the night, the deposition reconvened the following morning, on Wednesday, February 19, 2014. Tr. 223. At approximately 1:24 p.m., after the lunch break, Complaint Counsel produced the Ohio State Article, marked it as an exhibit to the deposition, and proceeded to question Mr. Sinclair concerning the Article, including regarding the findings and conclusions of the study. Tr. 366-378. It is undisputed that this was the first time Complaint Counsel had disclosed the document to Respondent.

In response to the objections of Respondent's counsel, Complaint Counsel stated that it received the Ohio State Article the previous Friday; that Complaint Counsel "might have" had the opportunity to send a copy of it to Respondent's counsel at that time; and that someone on Complaint Counsel's staff also "possibly" could have provided a copy to Respondent's counsel. Tr. 371-372. The dialogue continued:

> MR. EMORD: So we could have been given a copy of the document so we could confirm –

---

[1] The Clarification also does not state, among other details, by whom the draft was received, where it was located, or whether or not the draft was relied upon in the investigation or prosecution of this case. *See* 16 C.F.R. § 3.31(c)(2) (Complaint Counsel must search for responsive documents "that were collected or reviewed in the course of the investigation of the matter or prosecution of the case and that are in the possession, custody or control of the Bureaus or Offices of the Commission that investigated the matter, including the Bureau of Economics. . . .). Respondent has submitted a Motion to Compel and for Sanctions with respect to the draft article and related correspondence.

> MS. JOHNSON: Well, you have a copy now. So if you want to take some time and take a look at it, that's totally fine.

Id.

> . . .
>
> MR. EMORD: . . . You gave us no advanced notice of this document. None. You had it on Friday, you knew you were going to use it in a deposition –
>
> MS. JOHNSON: I did not know I was going to use it.
>
> MR. EMORD: When did you decide you were going to use it?
>
> MS. JOHNSON: We packed up our stuff on Saturday afternoon.
>
> MR. EMORD: This is highlighted. Who did the highlighting on the document?
>
> MS. JOHNSON: I'm not going to reveal that.
>
> MR. EMORD: . . . [I]n any event, you did not turn this document over to us when you had the opportunity to do it and –
>
> MS. JOHNSON: This is your opportunity . . .

Tr. 374-375.

> . . .
>
> MS. JOHNSON: I apologize for that . . . Would you like to take a few moments to read it, we can take a break?

Tr. 375-376.

II. Analysis

A. Overview

Respondent asserts, and Complaint Counsel does not deny, that the Ohio State Article is within the scope of Respondent's First Request for Production of Documents, specifically, Requests 1 and 3, which state:

> Document Request 1: Provide all documents that concern whether plastics in general and ECM Plastics in particular will break down and decompose into elements found in nature after customary disposal or in a landfill.
>
> Document Request 3: Provide all documents that support or call into question your conclusion that ECM's biodegradable claims for degradation are false.

Motion RX-B at 7.

Respondent argues that Complaint Counsel intentionally withheld the Ohio State Article from Respondent until the afternoon of the second day of ECM's deposition in order to gain an advantage of surprise, and that such conduct violated Complaint Counsel's duty to supplement in a timely manner under Rule 3.31(e)(2). FTC Rule 3.31(e) provides in pertinent part:

> A party who has . . . responded to a request for discovery with a disclosure or response is under a duty to supplement or correct the disclosure or response to include information thereafter acquired if ordered by the Administrative Law Judge or in the following circumstances:
>
> . . .
>
> (2) A party is under a duty to amend in a timely manner a prior response to an interrogatory, request for production, or request for admission if the party learns that the response is in some material respect incomplete or incorrect.

16 C.F.R. § 3.31(e)(2).

Respondent further argues that Complaint Counsel's conduct in withholding the document was willful and unjustified, and that Complaint Counsel's questioning of ECM on the previously undisclosed document was prejudicial to Respondent. Thus, Respondent argues, Complaint Counsel should be barred from introducing into evidence at trial, or "otherwise relying on" the Ohio State Article, pursuant to Rule 3.38(b)(4). That Rule provides that if a party "fails to comply with any discovery obligation imposed by these rules, upon motion by the aggrieved party," the Administrative Law Judge "may take such action in regard thereto as is just," including a ruling "that the party may not introduce into evidence or otherwise rely" upon "improperly withheld or undisclosed materials, information, witnesses, or other discovery." 16 C.F.R. § 3.38(b)(4).

B. Timely Supplementation

Complaint Counsel does not dispute that it was under an obligation to supplement its previous document production by providing the Ohio State Article to Respondent. The disputed issue is whether, under the circumstances presented, Complaint Counsel's conduct in delaying production of the Ohio State Article until five days after its receipt, during the middle of the second day of a previously scheduled deposition, violated Complaint Counsel's obligation to supplement "in a timely manner." As explained below, and based on the record presented, by delaying production of the Ohio State Article and presenting the Article to Respondent for the first time in the midst of the second day of the deposition, when Complaint Counsel had clearly determined the relevance and possible use of the Article before the start of the deposition, Complaint Counsel did not supplement in a timely manner.

Complaint Counsel clearly recognized the relevance of the Ohio State Article, if not immediately upon receipt, then certainly no later than Saturday afternoon, February 15, 2014, when Complaint Counsel packed the document in order to use it at ECM's deposition. *See* Tr. 374-375. Yet Complaint Counsel declined numerous opportunities to provide the Article to Respondent and thereby fulfill its duty to supplement Complaint Counsel's prior document production. Putting aside whether, in the current age of instant communications, including via government issued Blackberry, Complaint Counsel had an obligation to transmit the Article to Respondent over the long holiday weekend, Complaint Counsel fails to explain or justify why it did not supplement its document production by providing the Ohio State Article to Respondent before the start of the ECM deposition, during the first day of the deposition, or at any other time prior to marking the Article as a deposition exhibit on the afternoon of the second day of the ECM deposition.

Rule 3.31(e) does not define what constitutes a "timely manner" for purposes of the duty to supplement. Furthermore, the parties do not point to a case defining the phrase in the context of the type of delayed supplementation present in this case. However the phrase "timely manner" might be defined, it manifestly does not include a conscious withholding of a responsive document that was planned to be used as a deposition exhibit, as occurred in this case.

Having determined that Complaint Counsel violated its obligation to supplement discovery in a timely manner, the analysis now turns to whether a sanction is appropriate, as requested by Respondent.

C. Sanction

Respondent argues that the appropriate sanction for Complaint Counsel's violation is to bar any use of the Ohio State Article for purposes of trial. Respondent contends that Complaint Counsel's conduct was willful and unjustified. In addition, Respondent asserts that Complaint Counsel's delay in producing the Article until the second day of the deposition prejudiced Respondent, because the delay put Mr. Sinclair in the position of answering questions about the Article without adequate preparation. According to Respondent, where a discoverable document is improperly withheld, the customary remedy is to exclude the document from any use at trial.

Complaint Counsel responds that there has been no prejudice to Respondent because Mr. Sinclair "admitted essentially nothing" about the Ohio State Article; Mr. Sinclair is free to testify at trial that he believes the study was faulty;[2] ECM's experts have until April 30, 2014 to analyze the study; and providing the document earlier "would not have affected [ECM's experts'] ability to respond . . . ." Opposition at 6.

"Rule 3.38 is designed both to prohibit a party from resting on its own concealment and to maintain the integrity of the administrative process." *In re Grand Union Co.*, 102 F.T.C. 812,

---

[2] Indeed, Complaint Counsel contends that ECM has, is in fact, gained an advantage vis a vis Mr. Sinclair's potential trial testimony concerning the study. Complaint Counsel states: "[I]f Complaint Counsel had forwarded ECM the Ohio State Study early (in the middle of the night on Friday the 14th, or at the end of the day on Tuesday February 18 (after one business day had elapsed)), Sinclair could not have testified that he had never seen the study before, which might have made it difficult for him to supplement any hastily-prepared deposition testimony at trial." Opposition at 6. Instead, according to Complaint Counsel, the fact that Sinclair had not seen the study before "gives ECM several months to figure out some sort of response." *Id.*

1983 FTC LEXIS 61 at *594 (July 18, 1983). Sanctions may be imposed for failing to comply with a discovery obligation where the failure to comply was "unjustified and the sanction imposed 'is reasonable in light of the material withheld and the purposes of Rule 3.38(b).'" *See Matter of International Telephone & Telegraph Corp.*, 1984 WL 565367 at **127 (July 25, 1984 (quoting *Grand Union*, 1983 FTC LEXIS 61 at *595)).

Regardless of whether exclusion of the Ohio State Article would be an appropriate remedy had Complaint Counsel delayed production of the document until after the discovery deadline or the start of trial – conduct present in the cases cited by Respondent – in the instant case the extent of Complaint Counsel's delay does not warrant this sanction. The fact discovery deadline in this case is presently April 3, 2014, and the close of expert discovery is presently May 16, 2014. Trial is currently scheduled to begin on June 18, 2014. Under these circumstances, exclusion of the Ohio State Article is not necessary or appropriate to remedy the asserted prejudice. Accordingly, Respondent's request to exclude the Article, as a sanction for Complaint Counsel's delayed production of the Article, is DENIED.

However, Respondent has demonstrated that Complaint Counsel violated its discovery obligation to supplement in a timely manner with respect to the Ohio State Article, and based on the facts presented, a sanction is appropriate. Accordingly, the Motion is GRANTED IN PART. Complaint Counsel has failed to show that its conduct was justified, and a sanction under these circumstances will affirm the integrity of the administrative process. Moreover, Complaint Counsel should not be permitted to benefit from its conduct. In the instant case, a reasonable and just sanction under Rule 3.38(b) is an order prohibiting Complaint Counsel from using or in any way relying upon any of Mr. Sinclair's deposition testimony regarding the Article. Rule 3.38(b) (stating that ALJ may enter any sanction order that is "just"); *see also* Rule 3.38(c) (stating that ALJ shall order relief "as may be sufficient to compensate for withheld testimony, documents, or other evidence").

III. Conclusion and Order

For all the foregoing reasons, Respondent's Motion for Sanctions is GRANTED IN PART, and it is hereby ORDERED, that Complaint Counsel may not introduce into evidence, or otherwise rely, for any purpose, including without limitation impeachment, upon testimony given at the deposition of ECM on February 18 and 19, 2014, regarding the Ohio State Article. The Motion is in all other respects DENIED.

ORDERED:

DM Chappell
D. Michael Chappell
Chief Administrative Law Judge

Date: March 21, 2014

Respondent's Exhibit Q

UNITED STATES OF AMERICA
BEFORE THE FEDERAL TRADE COMMISSION

In the Matter of	)	
	)	
ECM BioFilms, Inc., **a corporation, also d/b/a** **Enviroplastics International**	)	**Docket No. 9358** **PUBLIC DOCUMENT**

COMPLAINT COUNSEL'S CLARIFICATION REGARDING RESPONDENT'S SANCTIONS MOTION

In Complaint Counsel's Opposition to Respondent's sanctions motion, we submitted a declaration stating, in material part, that "Complaint Counsel received the article [published in POLYMER DEGRADATION & STABILITY] well after business hours on Friday, February 14, 2014, at approximately 8:00 p.m." J. Cohen Dec. ¶ 6 (Mar. 10, 2013). This is accurate.

Additionally, the declaration also provides that "Complaint Counsel has not communicated in any way with Frederick Michel, Eddie Gómez, OARDC [Ohio State Agricultural Research & Development Center], or anyone at Ohio State." *Id.* at ¶ 7. This is also accurate.

Out of an abundance of caution, however, Complaint Counsel brings to the Court's attention that yesterday, we learned that two FTC attorneys (not representing Complaint Counsel) worked with Michel as a consulting expert on unrelated matters involving biodegradability claims other companies asserted.[1] In the course of evaluating Michel's credentials, these attorneys received a draft of the article ultimately published. Complaint Counsel never had any knowledge regarding Michel's engagement (or the draft article) until yesterday. Additionally, as our Opposition explains, Complaint Counsel had no knowledge regarding the article itself before Friday evening, February 14.

---

[1] One of the two attorneys was involved in the pre-Complaint investigation of Respondent, but departed the agency approximately eight months before the Complaint was filed.

We are reviewing our prior discovery responses expeditiously to ascertain whether Complaint Counsel should amend or supplement them.

Dated: March 13, 2014

Respectfully submitted,

Katherine Johnson (kjohnson3@ftc.gov)
Jonathan Cohen (jcohen2@ftc.gov)
Elisa Jillson (ejillson@ftc.gov)
Federal Trade Commission
600 Pennsylvania Ave., N.W. M-8102B
Washington, DC 20580
Phone: 202-326-2185; -2551; -3001
Fax: 202-326-2551

CERTIFICATE OF SERVICE

I hereby certify that on March 13, 2014, I caused a true and correct copy of the foregoing to be served as follows:

One copy through the FTC's efiling system and one electronic courtesy copy to the **Office of the Secretary:**

Donald S. Clark, Secretary
Federal Trade Commission
600 Pennsylvania Ave., NW, Room H-159
Washington, DC 20580
Email: secretary@ftc.gov

One electronic and one and one paper courtesy copy to the **Office of the Administrative Law Judge:**

The Honorable D. Michael Chappell
Administrative Law Judge
600 Pennsylvania Ave., NW, Room H-110
Washington, DC 20580

One electronic copy to **Counsel for the Respondent:**

Jonathan W. Emord
Emord & Associates, P.C.
11808 Wolf Run Lane
Clifton, VA 20124
Email: jemord@emord.com

Peter Arhangelsky
Emord & Associates, P.C.
3210 S. Gilbert Road, Suite 4
Chandler, AZ 85286
Email: parhangelsky@emord.com

Lou Caputo
Emord & Associates, P.C.
3210 S. Gilbert Road, Suite 4
Chandler, AZ 85286
Email: lcaputo@emord.com

I further certify that I possess a paper copy of the signed original of the foregoing document that is available for review by the parties and the adjudicator.

Date: March 13, 2014

Katherine Johnson (kjohnson3@ftc.gov)
Jonathan Cohen (jcohen2@ftc.gov)
Elisa Jillson (ejillson@ftc.gov)
Federal Trade Commission
600 Pennsylvania Ave., N.W. M-8102B
Washington, DC 20580
Phone: 202-326-2185; -2551; -3001
Fax: 202-326-2551

Respondent’s Exhibit R

From: Johnson, Katherine
To: Jonathan Emord; Peter Arhangelsky; Eric Awerbuch
Cc: Jillson, Elisa; Cohen, Jonathan; Decastro, Arturo
Subject: ECM Biofilms, Dkt 9358
Date: Monday, June 30, 2014 8:46:05 PM
Attachments: Michel CV.pdf
Expert Rebuttal Report of Dr. Frederick Michel Jr.pdf

Counsel,

Attached please find the rebuttal expert report of Dr. Frederick Michel. The materials will follow by FTP link in the morning.

Katherine E. Johnson, Attorney
Division of Enforcement
Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Avenue, NW
Mail stop M-8102B
Washington, DC 20580
Direct Dial: (202) 326-2185
Fax: (202) 326-2558
Email: kjohnson3@ftc.gov

ECM Comb. Mot. to Exclude
Exh. R

Respondent’s Exhibit S

Peter Arhangelsky

From: pclarke@ftc.gov
Sent: Tuesday, July 01, 2014 9:14 AM
To: Peter Arhangelsky
Cc: kjohnson3@ftc.gov
Subject: ECM Biofilms (R1502)

You have received 1 secure file from pclarke@ftc.gov.
Use the secure link below to download.

Peter,

Here are the materials for Dr. Michel.

Thanks,

Katherine

Secure File Downloads:
Available until: **05 July 2014**

Click link to download:

20140701_R1502.zip
19,181.95 KB

You have received attachment link(s) within this email sent via the FTC Secure Mail system. To retrieve the attachment(s), please click on the link(s).

Secured by Accellion

ECM Comb. Mot. to Exclude
Exh. S

Respondent's Exhibit T

From: Johnson, Katherine
To: Peter Arhangelsky; Jonathan Emord; Eric Awerbuch
Cc: Cohen, Jonathan; Jillson, Elisa; Decastro, Arturo
Subject: ECM Dkt 9358: Deposition Dates for Dr. Frederick Michel
Date: Monday, July 07, 2014 10:49:35 AM

Dr. Michel is available for deposition the following dates:

July 17th and 18th; and July 29th to 1st of August.

The location would likely be in Wooster, OH.

Katherine E. Johnson, Attorney
Division of Enforcement
Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Avenue, NW
Mail stop M-8102B
Washington, DC 20580
Direct Dial: (202) 326-2185
Fax: (202) 326-2558
Email: kjohnson3@ftc.gov

Respondent’s Exhibit U

EXPERT REPORT OF DR. STEPHEN P. MCCARTHY
SUMMARY & CONCLUSIONS

I am a Professor of Plastics Engineering at the University of Massachusetts Lowell. I more than thirty years' experience studying both the chemical and mechanical behavior of polymers, including their biodegradability, and I currently serve as the Director of the University's Biodegradable Polymer Research Center. Complaint Counsel asked me to evaluate biodegradable claims made by ECM Biofilms, Inc. In my expert opinion, I conclude with at least a reasonable degree of professional certainty that:

- Plastic products manufactured with the ECM additive ("ECM Plastics") are not biodegradable;
- ECM Plastics will not completely break down and decompose into elements found in nature in most landfills within five years;
- ECM Plastics will not completely break down and decompose into elements found in nature in any disposal environment at an appreciably faster rate and extent than conventional plastics without the ECM additive;

Furthermore, no competent and reliable scientific evidence contradicts any of these facts.

6/4/14
DATED:

Dr. Stephen P. McCarthy

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I. INTRODUCTION

1. Complaint Counsel retained me to provide my expert opinion regarding certain of ECM Biofilms, Inc.'s biodegradable advertising claims. Specifically, Complaint Counsel has asked me to opine regarding whether claims that ECM's additive makes conventional plastics biodegradable are true and supported by competent and reliable scientific evidence. It is my expert opinion that ECM's biodegradability claims are false and unsubstantiated. This Expert Report sets forth the rationale for my opinions.

2. It is universally accepted that conventional plastics are resistant to biodegradation. If an additive existed that truly could render these conventional plastics biodegradable, it would have garnered enormous attention in the scientific community. It would also have untold commercial applications worldwide and be an enormous commercial success. Unfortunately, no such revolutionary product exists.

II. EDUCATION, BACKGROUND & EXPERIENCE

3. I have more than three decades of experience studying both the chemical and mechanical behavior of polymers, including the biodegradability of polymers used to form conventional, commercial-grade plastics.

4. I received a Ph.D. in Polymer Engineering from Case Western Reserve University in 1984. Prior to that, I received a Master's degree in Chemical Engineering from Princeton University (1980) and a Bachelor's of Science degree in Textile Chemistry from Southeastern Massachusetts University (1978).

5. I am a Professor of Plastics Engineering at the University of Massachusetts Lowell where I teach graduate level courses in Plastics Engineering, including the "Mechanical Behavior of Polymers" and "Polymers and the Environment." In addition, I am the Director of the University's Biodegradable Polymer Research Center, where I coordinate and supervise research on biodegradable polymers. I am the Principal Investigator for studies on plastics

engineering and polymer research. My research has led to seven patents related to polymers or plastics engineering.

6. Also during my tenure at the University, I have as the Director of the University's Bioplastics Institute and Medical Plastics Research Center, Director of the University's Institute for Plastics Innovation, and Graduate Coordinator for the Plastics Engineering Department. In addition, I served as the Director of the National Science Foundation's Center for Biodegradable Polymer Research.

7. In addition to my university activities, I am also the Editor of the JOURNAL OF POLYMERS & THE ENVIRONMENT, the official journal for the *BioEnvironmental Polymer Society*, which promotes research to develop degradable polymers. This editorship builds on the work I did as a member of the Editorial Board of the JOURNAL OF APPLIED POLYMER SCIENCE from 1995 to 2003.

8. I have received a number of awards for my work in the field of plastics engineering and polymer research, including a *BioEnvironmental Polymer Society* (BEPS) James Hammar Memorial Lifetime Achievement Award (2008), two *Society of Plastics Engineering* Best Paper Awards (Medical Plastics in 2005 and Color and Appearance in 2000 and the Society of Plastics Engineering Education Award in 2014). I also received the *Society of Plastics* Engineering Education Award earlier this year.

9. Since 1981, I have authored or co-authored more than a hundred publications related to polymer or plastics engineering. Additionally, I have lectured both around the country and around the world (in Israel, Ireland, Turkey, Canada, India, Italy, Sweden, Japan, China, Indonesia, Taiwan, Korea) on plastics engineering, "green" plastics, and biodegradable polymers. I have also given seminars in plastics engineering to research and development teams at Hewlett-Packard, Exxon, 3M, and Clariant.

10. Currently, I am a member of the American Society for Testing and Materials (now known as ASTM International, Inc.). In the past, I belonged to various professional

associations related to biodegradable polymers and plastics engineering, including the *Bio/Environmentally Degradable Polymer Society*, *Society of Plastics Engineers*, *Biomaterials Society*, *American Chemical Society*, and the *Materials Research Society*.

11. My complete *Curriculum Vitae* is attached hereto in Appendix A. Based upon my education, training, and experience, I consider myself an expert in the fields of plastics engineering, polymer science, and biodegradable polymers.

12. In the past four years, I testified as an expert in a binding alternative dispute resolution proceeding in a patent infringement case on March 19, 2012. *See Display Technologies, LLC v. Display Industries, LLC*, No. 11-civ-6390 (S.D.N.Y.). I do not have a copy of the transcript.

13. I am compensated at a rate of $100 per hour for all work in this matter, except deposition and trial testimony, which will be compensated at a rate of $325 per hour.

III. SCOPE OF ANALYSIS

14. Complaint Counsel asked me to evaluate whether the following advertising claims for ECM Biofilms, Inc.'s additive ("ECM additive") are true and supported by competent and reliable scientific evidence:

a. Plastic products manufactured with the ECM additive ("ECM Plastics") are biodegradable.[1]

b. ECM Plastics will completely break down and return to nature (*i.e.*, decompose into elements found in nature) in nearly all landfills within nine months to five years.

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[1] Complaint Counsel asked me to assume that the unqualified marketing claim "biodegradable" means that the entire treated plastic will completely break down and return to nature (*i.e.*, decompose into elements found in nature) within one year after customary disposal (*i.e.*, incinerator, landfill, or recycling). I use this definition and the scientific definition of biodegradable interchangeably in this Expert Report, because there is no substantive difference between the two that affects my analysis or my opinions.

c. ECM Plastics will completely break down and return to nature (*i.e.*, decompose into elements found in nature) in most landfills within one to five years.

d. ECM Plastics will completely break down and return to nature (*i.e.*, decompose into elements found in nature) in any disposal environment at an appreciably faster rate and extent than conventional plastics without the ECM Additive.

e. Various scientific tests, including but not limited to ASTM D5511, have shown that ECM Plastics perform as indicated in (a)-(d) above.

15. Complaint Counsel provided me with materials, including published and unpublished study reports, protocols, data, and data analysis. These materials fall into one of two broad categories:

a. Tests and other materials ECM submitted to the FTC during the pre-complaint investigation and litigation in this matter ("ECM Materials");[2] and

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[2] The ECM Materials include:
(1) Anaerobic Biodegradation of bioPVC; Analytical Report No. 1150851 regarding "ASTM D 5511 Extension Testing;
(2) Analytical Report No. 1253020 regarding ASTM D 5511: Determining anaerobic biodegradation of plastic materials under high solids anaerobic digestion conditions;
(3) Analytical Report No. 946510-01 regarding ASTM D 5511;
(4) Analytical Report No. N0946510-01 regarding ASTM D 5511;
(5) Update regarding ASTM D5511-11 Update on Clear Films 476 & 477 @ 45 days;
(6) Biopolymers and additived [sic] plastics: biodegradability, degradability and compostability. Basic concepts, comparisons and legislation: The case of ECM MasterBatch Pellets additive;
(7) Ecological Assessment of ECM Plastic;
(8) Final Report: Biodegradation Testing, Aerobic Biodegradation Under Controlled Composting Conditions for 40-gal trash bags";
(9) Report about biodegradability of a plastic artefact (full coffee capsule) produced with a compound containing Polypropylene and a biodegradability adjuvant addictive (ECM MasteBatch Pellets) for 1% of its total weight;
(10) SEM Examination of ECM Plastic;
(11) SEM imaging of bubble wrap;
(12) SEM imaging of EPS samples; SEM imaging of green PET bottles;
(13) SEM imaging of PVC samples; and
(14) Time Elapse Photographs of the ECM MasterBatch Pellets Formula in 5-Mil Cast LLDPE Film on Tryptic Soy Agar Plates.

b. Tests and other materials obtained from other parties during the course of this litigation ("Litigation Materials").

16. I also conducted my own literature research. I refer to my own research, the ECM Materials, and the Litigation Materials collectively as "Testing Materials." Appendix B contains all of the Testing Materials that I considered. In reviewing the Testing Materials and offering the opinions this Expert Report contains, I have extensively relied on my knowledge of the field of polymer science.

IV. SUMMARY OF ANALYSIS

17. ECM's claims that ECM Plastics will completely biodegrade faster than conventional plastic are false and unsubstantiated. Conventional plastics are resistant to biodegradation due to their chemical structure and can take thousands of years to biodegrade. Specifically—whether in a biofilm or otherwise—microorganisms cannot create the enzymes necessary to break down conventional plastics more rapidly. Physically blending the ECM additive with a conventional plastic does not change the conventional plastic's chemical structure. As a result, post-blending, the conventional plastic is no more susceptible to microbial attack than it was pre-blending, and ECM's claims are false.

18. ECM's claims are also unsubstantiated. For at least three reasons, nothing in the Testing Materials supports the conclusion that the ECM additive confers biodegradability to conventional plastics. First, none of the Testing Materials supports the conclusion that ECM Plastic is biodegradable or will biodegrade to completion; in fact, the only published, peer-reviewed study to address this question concludes otherwise.[3] Second, there is no scientific

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[3] *See* E. Gomez & F. Michel, *Biodegradability of conventional plastics and natural fiber composites during composting, anaerobic digestion and long term soil incubation*, 98 JOURNAL OF POLYMER DEGRADATION & STABILITY 2583-91 (2013) (testing the ECM additive and concluding that "**plastics containing additives that supposedly confer biodegradability to polymers such as polyethylene and polypropylene did not improve the biodegradability of these recalcitrant polymers**") (emphasis added).

support for ECM's hypothesis that microorganisms will combine synergistically to produce enzymes that would allow them to metabolize conventional plastic. Third, all tests that purport to show biodegradation of ECM Plastics are flawed.

19. These fatal flaws include, among many defects, test designs that do not measure biodegradation at all, gross imprecision in testing methods, misuse (or nonuse) of statistics to calculate results, and experimenters who lack the minimum qualifications necessary. To the extent that, notwithstanding these significant issues, some tests report minimal biodegradation, that purported biodegradation is the result of the "**priming effect**," *i.e.*, biodegradation of the additive (which contains organic compounds highly susceptible to biodegradation) and the organic materials of the test medium (the bacteria used for testing) rather than the plastic. Moreover, the minimal biodegradation reported is nearly always within the test's error limits, which underscores that the reported biodegradation is the result of flawed testing rather than the molecular transformation of conventional plastic into a biodegradable material.

20. In short, there is no competent and reliable scientific evidence that the ECM additive—or any other similar additive—causes plastic to biodegrade completely.

V. BIODEGRADATION & PLASTICS

21. To understand what constitutes competent and reliable scientific evidence of biodegradation in plastics, it is important to understand (i) the mechanisms of biodegradation; and (ii) the nature of plastics in general. I will describe each briefly below.

22. ***What Is Biodegradation?*** Biodegradation is a chemical process by which microorganisms such as bacteria and fungi use the carbon found in organic materials as an energy source (*i.e.*, as a food source). Microorganisms secrete enzymes that adhere to the surface of the organic materials and cause fissures in the molecular chain known as *hydrolic cleavages*. These cleavages make long-chain molecules shorter, resulting in the release of carbon and energy (heat).

23. The rate of biodegradation varies depending on numerous factors, including oxygen levels, moisture levels, pH content, and availability of nutrients. The two most important variables, however, are moisture and oxygen.

24. When biodegradation takes place in the presence of oxygen (*i.e., aerobic* biodegradation), carbon dioxide is released. Aerobic biodegradation occurs in composting facilities or when items are disposed as litter. When biodegradation takes place without oxygen (*i.e., anaerobic* biodegradation), methane gas and carbon dioxide are released. Landfills and anaerobic digesters are types of anaerobic disposal conditions.

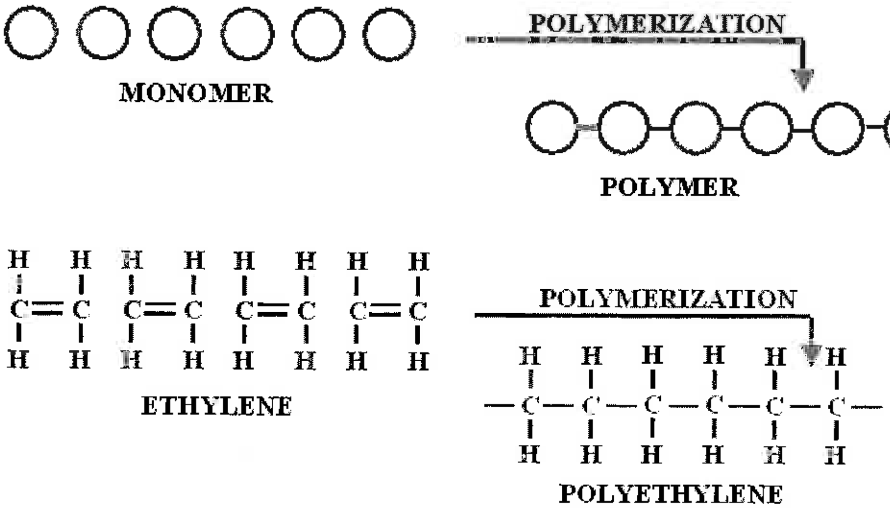
25. Importantly, *degradation* and ***bio**degradation* are <u>not</u> identical processes. As explained above, biodegradation involves microorganisms breaking down material into its constituent elements, *i.e.*, elements found in nature. Degradation (or *disintegration*) involves fragmenting of the polymer chain due to mechanical stress or chemicals.

26. Chemicals can include additives designed to fragment plastic when exposed to heat or ultraviolet rays. For example, the polyethylene blend used to make six-pack rings for soda cans is designed to disintegrate upon exposure to sunlight to limit injury to wildlife if the rings are littered. This is a type of chemically-induced fragmentation known as *photodegradation*. Similar chemical disintegration occurs in *oxodegradables*, which are polymers treated with a heavy metal that fosters oxidation in the polymer chain, causing the chain to disintegrate. Additionally, mechanical stress may disintegrate plastic, such as grinding or pulverizing (which happens during the recycling process).

27. Thus, although degradation (or disintegration) may change the plastic's physical form, it does not involve microorganisms, nor does degradation necessarily mean that the plastic will degrade into natural elements. Notably, the critical difference between degradation and **<u>bio</u>**degradation is significant to my opinions herein because ECM's Testing Materials sometimes conflate these two distinct concepts, and erroneously infer that biodegradation has occurred

merely because ECM Plastic has disintegrated. Of course, ECM markets its ECM Plastic as biodegradable, not merely degradable.

28. ***What are Plastics?*** Generically, *plastic* refers to high-molecular weight polymers. *Polymers* are large molecules composed of repeating low molecular weight molecules called *monomers.* Through a chemical process known as *polymerization,* monomers are combined to become polymers.



29. There are various plastics, but synthetic (laboratory-made), petroleum-based plastics are by far the most common. Plastics derived from petrochemicals are strong, durable, and inexpensive to manufacture, making them ideal for commercial applications. Such petroleum-based plastics, which I refer to as "conventional plastics," represent over 90% of the commercial plastic market. When laypersons talk about "plastic," they are usually referring to these conventional plastics.

30. The most common types of conventional plastic are high-molecular weight *polyethylene* (PE), which is commonly used to manufacture plastic bags, packaging material, bottles, and *polyurethane*, which is used commonly in medical and industrial applications such as adhesives and paint. Also common is *polypropylene* (PP), which is used for disposable cups,

clothing, storage containers and DVD covers, and *polystyrene* (PS), which is used most commonly for foam disposable cutlery and cups, foam packing peanuts, insulation, and fast food containers. These conventional plastics are often in the hundreds of thousands to millions in molecular weight (Mn).

31. There are a variety of plastics, but synthetic (laboratory-made), petroleum-based plastics are by far the most common. Plastics derived from petrochemicals are strong, durable, and inexpensive to manufacture, which makes them ideally suited for commercial applications. Such petroleum-based plastics, which I refer to in this report as "conventional plastics," represent over 90% of the commercial plastic market. When laypersons talk about "plastic," they are usually referring to these conventional plastics.

32. ***Bioplastics***. Most plastics are *organic*, which means that they are mostly carbon. As explained above, bacteria and fungi use carbon in organic materials as a food source. However, not all carbon-containing organics are biodegradable. Put differently, microorganisms cannot metabolize all organics. Conventional plastics are a prime example. They are organic (*i.e.*, carbon-based), but not biodegradable,[4] because the chemical structure of the conventional plastic prevents naturally-occurring microorganisms from accessing the carbon.

33. Notably, microorganisms have evolved to degrade those polymers that they have been exposed to. Since cellulose, starch, chitin, etc. are polymers that exist in nature, microorganisms have developed specific enzymes to degrade these polymers. The petroleum-

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[4] Over time, all things will likely biodegrade. Therefore, the statement that conventional plastic is not biodegradable should be read to mean "not biodegradable in any timeframe less than hundreds, thousands, or millions of years."

based conventional plastics have only existed for a hundred years or so. This is not long enough for microorganisms to evolve to degrade them. Indeed, the characteristics that make conventional plastics commercially useful—strength, durability, synthetically derived from petrochemicals—make them highly resistant to biodegradation.

34. Unlike petroleum-based, commercial-grade plastics that are **not** biodegradable, a subset of plastics called *bioplastics* do biodegrade.[5] For example, naturally-occurring polymers such as cellulose and starch are biodegradable because microorganisms have developed enzymes to degrade them. Likewise, certain types of synthetic polymers—those made from biomass, renewable resources, or some fossil materials such as polylactic acid, poly butylene succinate, and polycaprolactone—are biodegradable. These synthetic polymers are biodegradable because they are derived from or have physical and chemical properties similar to naturally-occurring polymers that microorganisms can degrade.

35. These biodegradable plastics currently have limited commercial applications because they are much more expensive to produce than conventional plastics, or they tend to be weaker and less durable than conventional plastics. For instance, polyhydroxybutyrate (PHB) is completely biodegradable under aerobic conditions, but costs several times more than the conventional plastic PE.

36. There has been significant interest in exploring ways to render conventional plastics biodegradable, especially in landfills (where most plastic is disposed). Any technology that could render conventional plastics biodegradable would **transform** the plastic industry (and the many subsidiary product and packaging industries that purchase conventional plastic),

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[5] Bioplastics only biodegrade under very specific conditions, such as aerobic composting conditions, and none would biodegrade in a landfill in any reasonable amount of time.

because such a discovery would eliminate the significant environmental problem the accumulation of these non-biodegradable materials poses. Indeed, such technology would be worth **billions** of dollars. Although bioplastics have been developed (most notably, starch-based plastics and oxobiodegradable plastics that biodegrade under certain aerobic conditions), as yet, no technology exists that enables conventional plastics to biodegrade quickly under anaerobic conditions, such as those found in a landfill.

VI. WHAT CONSTITUTES "COMPETENT AND RELIABLE SCIENTIFIC EVIDENCE" FOR CLAIMS OF BIODEGRADABLE PLASTIC

37. In light of the overwhelming scientific consensus that conventional plastics are not biodegradable after customary disposal, any test evaluating whether a conventional plastic has been transformed into a "biodegradable plastic" through the means of an additive must be rigorous enough to satisfy the scientific community that the plastic—and not merely the additive—will completely biodegrade after customary disposal. In this section, I address what constitutes competent and reliable scientific evidence to address this fundamental question.

38. To satisfy polymer scientists that 1% additive will make conventional plastics biodegradable in a stated timeframe and disposal condition, the claimant should provide the results of well-conducted scientific testing. Specifically, the evidence should take the form of appropriately-analyzed results of independent, well-designed, well-conducted, well-controlled testing. The testing should use the appropriate plastic application, load rate, inoculum, test conditions, and sample weight, over an appropriate duration of time. I discuss the significance of each of these factors below.

- **a.** ***Plastic Application.*** Testing the appropriate "plastic application" means that the test should be conducted on the type of plastic (*e.g.*, polyethylene, polystyrene, etc.) that the additive will in fact be incorporated into. Testing the intended type of plastic application is necessary, because different types of plastics have unique chemical and physical characteristics that affect their susceptibility to biodegradation. For instance, flexible polyvinylchloride (PVC) contains "plasticizers" that themselves could be biodegradable. Therefore, a test involving treated flexible PVC will likely yield different results than a plastic that does not contain biodegradable plasticizers.

b. ***Load Rates.*** The study must accurately reflect the recommended "load rate." In other words, if the additive is advertised to work when it comprises 1% of the plastic, then the test must be conducted on plastic that is 1% additive. Using the recommended load rate is important because the additive itself has biodegradable properties. Simply because a certain amount of biodegradation was observed at 5%, one cannot assume that one-fifth of that observed biodegradation will occur at 1% load rate. Rather, 5% may be the threshold amount needed to have *any* effect. Thus, studies of larger load rates of the additive do not constitute reliable evidence that a smaller amount of that ingredient will cause a proportionally reduced effect, or any effect at all. Only a study that uses the recommended load rate can yield reliable evidence.

c. ***Inoculum.*** Inoculum is the source of bacteria or other microorganisms that will use the test sample as a food source and thus cause biodegradation. It typically consists of partially digested organic material and microorganisms. The study must accurately reflect the type of bacteria that the treated plastic would be exposed to after disposal. The types of microorganisms operating in particular environmental conditions vary, and different microorganisms have different efficiencies in breaking down organic matter because they secrete different enzymes that have greater or lesser effect at breaking bonds through hydrolysis or oxidation. Thus, if the intended disposal environment is a landfill, the inoculum should be one exposed to household waste as a sole substrate (*i.e.*, sole source of food) that can survive at the temperatures, moisture and acidity levels found in landfill conditions.

d. ***Test Conditions.*** Test conditions should simulate actual disposal conditions. Thus, the test should simulate the same moisture content, temperature ranges, acidity, and oxygen level found in the conditions where the "biodegradable plastic" will decompose.

e. ***Sample Weight.*** The size of the plastic sample will affect biodegradation rate. Thus, a sample that is thinner may biodegrade faster than a sample that is thicker. Moreover, it is very important to use the appropriate sample weight relative to the quantity of inoculum. The smaller the sample, and the larger the quantity of inoculum, the less reliable the observed biodegradation is. This is because inoculum itself contains organic material that is a food source for biodegradation. As a result, when the amount of inoculum is high relative to the amount of plastic, it is likely that observed biodegradation is attributable to the biodegradation of the inoculum itself.

f. ***Test Duration.*** The study must last long enough for the sample to reach at least 60% biodegradation. The most typical type of biodegradation test is a gas evolution test, which monitors the end-products of biodegradation. Sixty percent biodegradation is the minimum benchmark for showing that the treated sample will biodegrade to completion. (Alternatively, radiolabeling the most recalcitrant part of the conventional plastic can conclusively answer the question whether the conventional plastic component of the treated plastic is biodegrading. Radiolabeling will also show that the "biodegradable plastic" can biodegrade to completion.)

g. ***Proper Controls.*** Tests should involve proper controls, so that the results of the tested sample can be compared to the results of the control samples to ensure that the results are meaningful. In other words, using proper controls decreases the likelihood that apparently meaningful results are in fact the product of testing error. Proper testing should have a negative control (*i.e.*, untreated conventional plastic of the same type as the test sample) and a positive control (typically cellulose). The negative control is a substance known *not* to be biodegradable, and, therefore, it should produce little to no methane. The positive control is a substance known to be readily biodegradable; this validates the viability of the inoculum. If the amount of methane produced by the test sample is statistically different than the negative control, and the standard deviation does not span zero, biodegradation can be reported.

h. ***Statistical Significance.*** Once a study has been completed and all data have been collected, the data for the sample and control groups must be compared through appropriate statistical analysis that will depend upon various design factors of the study. If the standard deviation spans zero, then there is a significant probability that some of the samples did not biodegrade.

39. For a test to constitute reliable evidence that 1% additive will make conventional plastic biodegradable in a stated timeframe and disposal condition, it must involve each of these elements. This is true for three reasons. First, in a test that lacks some or all of these elements, observed biodegradation may be the result of factors other than biodegradation of the conventional plastic sample. For example, it could result from biodegradation of the additive, biodegradation of the inoculum, or testing error. Second, a test that lacks some or all of these elements cannot show that biodegradation will continue; it is equally plausible from a scientific perspective that some initial period of biodegradation could yield to subsequent recalcitrance. Third, a test that lacks some or all of these elements cannot establish whether biodegradation will occur in a specific environment.

40. Additionally, individuals with the proper educational background and training must design and oversee these scientific studies. First, only a person with a sophisticated understanding of the scientific principals involved can design a test or implement changes to a test method without affecting the validity of the underlying results. As explained above, changing parameters of the test such as inoculum, test duration, temperature, or moisture conditions, for example, can have a significant impact on the results. Changes must be planned, well thought out, and justified by commonly accepted scientific principles.

41. Second, the test should be overseen by a person with the proper education and training. Certain laboratory functions may be provided by lab technicians, such as taking readings or performing routine measurements. However, there are a number of quality control and quality assurance requirements for the operation of any laboratory that help ensure the validity and reliability of the results. Only those with the proper training and education can ensure these quality control measures are being executed properly.

42. Anecdotal evidence of biodegradation is not competent and reliable scientific evidence of biodegradation. Moreover, qualitative tests (*e.g.*, tests that evaluate changes in appearance, weight, or other physical characteristics) are, by themselves, insufficient to address the questions posed above, because qualitative changes can result from mechanical processes rather than from biodegradation.

VII. CURRENTLY AVAILABLE TESTS THAT SCIENTIFICALLY EVALUATE BIODEGRADATION OF PLASTIC

43. There are three tiers of tests used by polymer scientists to determine whether, and under what conditions, biodegradation of plastic occurs. First, an initial screening level test can show whether biodegradation is occurring at all. Second, level-1 confirmatory tests can determine whether the plastic itself is in fact biodegrading and whether it will biodegrade under specific (*e.g.*, real-world) conditions. Third, level-2, field-scale tests that are *in situ* can be used to evaluate whether biodegradation has adverse environmental effects.[6]

44. Screening level tests, by themselves, cannot definitively establish biodegradation,[7] because their optimized conditions and lack of control for the "priming effect" means that they are likely to overestimate biodegradation.[8] These factors can cause a screening-level test to essentially give a false positive—to show biodegradation because the organic

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[6] MICROBIOLOGY OF SOLID WASTE 179 – 180 (Anna C. Palmisano and Morton A. Barlaz, eds. 1996).

[7] *Id.* at 193 ("screening-level evaluations of materials do not provide definitive evidence of biodegradation.").

[8] *Id.* ("[T]he possibility of overestimation of biodegradation potential exists if the 'priming effect' occurs if material transformations are due to microbial attacks on additives rather than mineralization of a polymeric component of a material, or if a material is exposed to microbial cultures that are not representative of the environment in which the material will be disposed.")

additive (rather than the plastic) is biodegrading at ideal temperature, moisture, etc.[9] In other words, screening tests may show biodegradation because they have been "primed" to do so.

45. To substantiate its claims regarding the purported biodegradability of ECM Plastics and the role of the ECM Additive, ECM should have had both screening and confirmatory tests. Confirmatory testing such as radiolabeling is essential to establish that the plastic itself is biodegrading.

A. Screening-Level Tests Are Only The First Step.

46. The purpose of a screening-level test is to evaluate whether the test specimen is capable of biodegrading at all—under any condition. So, screening level tests are conducted under optimal conditions for biodegradation.

47. Standard test methods set by the ASTM[10] are common screening-level tests for determining biodegradability of plastics. Such ASTM test methods set forth the protocols established by the scientific community to evaluate a material. They are methods (*i.e.*, procedures and processes), not pass/fail criteria.

48. An example of a standard test method for testing biodegradability of plastics in the aerobic context is ASTM D5338: "Standard Test Method for Determining Aerobic

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[9] *Id.* (describing "material transformations [] due to microbial attacks on additives rather than mineralization of a polymeric component of a material").

[10] ASTM International, formerly known as the American Society for Testing and Materials (ASTM), develops and publishes voluntary consensus technical standards for a wide range of materials, products, systems, and services. Standards are developed within committees, and new committees are formed by request of interested members. Membership in the ASTM is open to anyone with an interest in its activities. There are approximately 20 active standards under the jurisdiction of subcommittee D20.96 on Environmentally Degradable Plastics and Biobased Products. For the past 20 years, I have been involved in helping to develop standard test methods and specifications for D20.96.

Biodegradation of Plastic Materials under Controlled Composting Conditions, Incorporating Thermophilic Temperatures." This test method sets forth a protocol for determining the extent to which a plastic can biodegrade in controlled composting conditions.

49. There are several ASTM standard test methods related to anaerobic biodegradation:

> **ASTM D5210**: "Standard Test Method for Determining the Anaerobic Biodegradation of Plastic Materials in the Presence of Municipal Sewage Sludge";
>
> **ASTM D5511**: "Standard Test Method for Determining the Anaerobic Biodegradation of Plastic Materials Under High-Solids Anaerobic-Digestion Conditions"; and
>
> **ASTM D5526**: "Standard Test Method for Determining Anaerobic Biodegradation of Plastic Materials Under Accelerated Landfill Conditions."

50. The basic methodology for each of these anaerobic test methods is the same. They are all "gas evolution tests," in which the test specimen is exposed to a source of bacteria (inoculum), and the end-products of biodegradation (biogases) are observed and measured. These tests vary in terms of test conditions (temperature, moisture, and type of inoculum) and test duration. Each test is validated only to run for a specific period.[11] Thus, each presents the optimal conditions representing a specific disposal environment (*e.g.*, high-solids anaerobic digester, or accelerated landfill conditions).

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[11] Each test protocol is conducted inter-laboratory or intra-laboratory in order to obtain the precision and bias of the test. If the inter-laboratory tests obtain similar precision and bias results under the test method, conducted for the same duration, the test method is considered validated for those factors. Tests conducted outside of these validated parameters cannot be considered reliable.

i. ASTM D5511

51. The ASTM first published the D5511 test method in 1994. It has been reconsidered periodically by the scientific community through ASTM's voluntary consensus committee D20.96 on biodegradable plastics and has undergone minor changes in 2002, 2011, and again in 2012. But the basic methodology has not changed.

52. ASTM D5511 is a gas evolution test. The test specimen is exposed to an inoculum from an anaerobic digester operating on household waste as its sole substrate (*i.e.*, sole food source). This short-term test is designed to run for 15-30 days, and is intended to replicate anaerobic digesters. In the United States, anaerobic digesters are primarily used at farms to treat animal waste, although there are also some applications for wastewater treatment. There are no anaerobic digesters treating household waste in the United States, so the ASTM D5511 inoculum is not available in this country.

53. When properly conducted, ASTM D5511 can be used as a screening-level test. It cannot, however, be used to support a scientific conclusion that a biodegradable plastic will biodegrade to completion in a landfill, for two reasons. First, this short-term test (typically 15-30 days) is not designed to run for the length of time necessary to show complete biodegradation. Second, this anaerobic-digester test is not designed to simulate conditions in the typical U.S. landfill.

ii. ASTM D5526

54. ASTM D5526 is designed to run longer than ASTM D5511: it has been validated to a period of 300 days. The method employs temperature and moisture conditions that more closely resemble a special type of landfill called a "bioreactor," a rare type of landfill in which "the gas generated is recovered or even actively promoted, or both, for example by inoculation,

moisture control in the landfill, and temperature control."[12] Because ASTM D5526 mimics bioreactors, its conditions are not representative of most landfills. As a result, although ASTM D5526 can be used as a screening-level test, it cannot be used to support a scientific conclusion that a plastic will biodegrade to completion in the typical U.S. landfill.

55. Like ASTM D5511, ASTM D5526 prohibits extrapolation of test results.[13] This means that if a test shows 10% biodegradation in 300 days, the test **cannot** be used to support a claim of 100% biodegradation in 3000 days. The reason for the prohibition on extrapolation is simple: there is no evidence that biodegradation is a linear process (and, in fact, the rate of biodegradation is likely to slow because of recalcitrance).

iii. Proposed ASTM Standard Test Method for Landfill Conditions.

56. To address rising interest in degradable plastics, ASTM is considering a proposed test method (WK 41850) for determining the rates and rate constants for plastics biodegradation in an anaerobic laboratory environment under accelerated conditions. This test method currently proposes to test for 60% conversion of the test material to carbon dioxide and methane within 18 months. In other words, if this gas evolution test shows 60% biodegradation within 18 months, the testing can be used to support a scientific conclusion that a plastic will biodegrade to completion in a landfill.

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[12] ASTM Int'l, D 5526: Standard Test Method for Determining Anaerobic Biodegradation of Plastic Materials Under Accelerated Landfill Conditions 1.2 (2012).

[13] *Id.* at 1.4; ASTM Int'l, D 5511: Standard Test Method for Determining Anaerobic Biodegradation of Plastic Materials Under High Solids Anaerobic Digestion Conditions 1.4 and 13.3 (2012).

B. Confirmatory-Level Tests Are Necessary To Establish Biodegradation.

57. As discussed above, screening-level tests establish a threshold for potential biodegradation, but confirmatory tests are necessary to provide definitive proof of biodegradation of plastic.[14]

58. Standard specifications provide a set of requirements for confirming biodegradation. In contrast to a test method, a specification is a "pass/fail" test. An example of a test specification in the aerobic context is ASTM D6400: "Standard Specification for Labeling of Plastics Designed to be Aerobically Composted in Municipal or Industrial Facilities." To pass the specification, a plastic product must biodegrade 90% (using ASTM D5538 test method) within 180 days. ASTM is considering a proposed specification for plastic materials that anaerobically biodegrade in landfills (WK34780), but there is currently no specification for customary anaerobic disposal environments such as landfills.

59. ASTM specifications are not the only way of confirming biodegradation. The best-known, best-accepted confirmatory test is radiolabeling. Radiolabeling involves tagging radioisotopes of carbon, ^{14}C of a high-molecular weight plastic, such as polyethylene (PE) before conducting a gas evolution test. During the gas evolution test, biogases are monitored for the radiolabeled ^{14}C. If the radiolabeled carbon is detected, then the conventional plastic polymer is undergoing a material transformation through biodegradation. If the radiolabeled carbon is not detected in the biogases, then the observed biogases are likely due to other factors, such as

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[14] SOLID WASTE, *supra* note 6 at 193 ("[d]efinitive proof of biodegradation, or lack thereof, may be provided through the use of isotopically labeled samples.")

biodegradation of the additive or the inoculum. Radiolabeling is particularly useful for technologies where the observed biodegradation would happen over a period of years.[15]

60. Absent an approved ASTM specification, it is my opinion that to scientifically prove a claim that the plastic—not merely the additive and inoculum—is biodegrading, the claimant must support its claim with at least one test with positive results from ^{14}C labeling of the conventional plastic.[16]

VIII. ANALYSIS OF ECM'S CLAIMS.

61. It is my understanding, based on the materials provided by Complaint Counsel, that the ECM additive is mostly a synthetic biodegradable polymer like polycaprolactone (PCL).[17] I understand further that ECM recommends that a small concentration, 1% to 5%, of its additive be melt-batch blended with a non-biodegradable conventional plastic, such as polyethylene. The resulting blended plastic, *i.e.*, ECM Plastic, is then used to manufacture a finished plastic product, such as a plastic water bottle or a golf tee.

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[15] *Id.* "a radioisotope of carbon (^{14}C) has found many applications in polymer research, especially in the design of experiments to run for long time periods (one or more years).").

[16] *See supra* note 6.

[17] The conclusion that the ECM Additive is a biodegradable polymer is based on the following. First, the materials submitted by ECM to Complaint Counsel describe the ECM additive as 60-70% biodegradable polymer. Letter from ECM's Counsel to the FTC (May 7, 2012). Complaint Counsel also asked me to assume alternatively that, based on certain materials provided to it and which I have reviewed, the ECM Additive is a blend of approximately 20% polyethylene vinyl acetate (EVA), approximately 60%, polycaprolactone (PCL), 6% linear low-density polyethylene (LLDPE), approximately 7% calcium stearate and approximately 6% starch. "Analysis Report" by Chemir Analytical Services, Chemir Analytical Job #79674, Dated December 31, 2010. Both show that most of the additive (made of biodegradable PCL) is biodegradable. Moreover, several of the tests on just the ECM Additive itself have concluded that it is biodegradable. *See* O.W.S. Inc: Final Report - Biodegradation Testing for ECM Pellets and EMC Film, concluding that the biodegradation studies performed on the additive "was statistically significant."; O.W.S. Inc: High Solids Anaerobic Digestion Test of ECM Pellets, concluding that the additive showed 24% biodegradation ± 1.5% after 15 days.

62. ECM asserts that treatment of conventional plastic with the ECM additive allows the formation of biofilms, which attract microorganisms that are able to synergistically combine to metabolize both the ECM additive and the conventional plastic until the plastic is completely biodegraded.

63. Specifically, ECM claims that: [18]

- **a.** ECM Plastics will completely biodegrade within one year in a landfill.
- **b.** ECM Plastics will completely biodegrade in nearly all landfills within nine months to five years.
- **c.** ECM Plastics will completely biodegrade in most landfills within one to five years.
- **d.** ECM Plastics will completely biodegrade in any disposal environment at an appreciably faster rate and extent than conventional plastics without the ECM Additive.
- **e.** Various scientific tests, including but not limited to ASTM D5511, have shown that ECM Plastics perform as indicated in (a)-(d) above.

Each of these claims is unsubstantiated. Moreover, although all of the claims are likely false as well, I can conclude with scientific certainty that all but one of ECM's claims are false. I discuss the false claims (a-c, e) first.

A. ECM's False Claims.

64. It is my opinion that after blending the ECM Additive with conventional plastic, the conventional plastic remains non-biodegradable. The reason for this is that a **physical** blend

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[18] *See supra* at 3-4.

of a biodegradable polymer with a conventional plastic does not alter the **chemical** structure of the conventional plastic. Because the additive does not alter the chemical characteristics that make conventional plastics resistant to biodegradation, the non-biodegradable plastic component is no more susceptible to biodegradation after blending than it was before.

65. As explained above, conventional plastics are considered non-biodegradable. Of course, all things will eventually biodegrade. The actual amount of time it would take for conventional plastics to biodegrade is unknown, but scientists have estimated anywhere from 500 to 10,000 years, depending on the molecular weight of the plastic and the environment. Even assuming that the addition of 1% ECM additive (biodegradable component) would allow the ECM Plastic to degrade faster (*e.g.*, by breaking the plastic into smaller pieces), the amount of time it would take for the conventional plastic to completely biodegrade would not be reduced to five years or even decades in any environment. ECM Plastic could take as long as the conventional plastic to biodegrade (because it still consists of 99% conventional plastic), or even longer (if the fragmented pieces become recalcitrant to biodegradation). Thus, claims that ECM Plastic will completely biodegrade in periods of time as short as five years cannot be true.

66. I cannot conclude with scientific certainty that ECM's claim that ECM Plastics biodegrade faster than conventional plastics is false. Without additional testing, it is impossible to know whether the resulting degraded plastic would biodegrade faster than untreated conventional plastic.

B. ECM's Unsubstantiated Claims.

67. All of ECM's claims identified above lack scientific support for at least three reasons. First, ECM did not conduct any confirmatory-level tests, and none of ECM's screening-

level tests adequately supports the conclusion that ECM Plastic is biodegradable or will biodegrade to completion. (Indeed, many tests indicate otherwise.) Second, there is no scientific support for the assertion that microorganisms will work synergistically to produce enzymes that would allow microorganisms to metabolize the plastic. This is contrary to widely-accepted scientific knowledge regarding how such microorganisms function with respect to conventional plastic. Third, the tests ECM relies on were conducted and/or reported in a deficient manner such that the results are insufficient to support ECM's conclusions.

C. Analysis of ECM's Claims and Testing.

i. ECM's Substantiation Does Not Support Its Claims.

68. It is my expert opinion that at least one confirmatory test must be conducted to establish that the plastic component of the ECM Plastics will biodegrade. ECM could have performed confirmatory testing by radiolabeling or by conducting a gas evolution test showing at least 60% conversion to methane and carbon dioxide within 18 months.

69. Without any confirmatory testing, ECM does not have adequate scientific support for its claims. The screening-level tests ECM relies on to support its claims extrapolate from minimal biodegradation to a conclusion of complete biodegradation. Such extrapolation is scientifically invalid because biodegradation is not linear and typically slows down due to recalcitrance.[19] (And, in fact, all of the tests performed on ECM Plastics reflect the typical plateauing associated with recalcitrance.)

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[19] For those tests conducted under the ASTM D5511 method, the invalidity of extrapolating these results to completion is reflected in the test method itself, which precludes extrapolation "Claims of performance shall be limited to the numerical result obtained in the test . . . . Furthermore, results shall not be extrapolated past the actual duration of the test." ASTM Int'l, D 5511: Standard Test Method for Determining Anaerobic Biodegradation of Plastic Materials Under High Solids Anaerobic Digestion Conditions 1.4 (2012).

70. Several of the tests submitted with the Testing Materials actually conclude that no biodegradation was observed at the conclusion of the test. For instance, two composting studies (whose composting environment presents far better conditions for biodegradation than landfill conditions) report no biodegradation. The composting study completed by O.W.S., Inc. (ECM-FTC-000124 and ECM-FTC-000244), the Final Report of Biodegradation Testing, "Aerobic Biodegradation Under Controlled Composting Conditions for 40-gal trash bags," Study PFR-5, Mar. 3, 2000, reported results of 5.2% biodegradation with a standard deviation of ±8.3%. Thus, according to the report, the observed biodegradation was within the error limits of the test and thus no conclusion can be drawn regarding whether any biodegradation occurred.

71. These findings of no biodegradation are consistent with other tests on ECM plastic. For example, another composting study performed by 3M Corporation likewise concludes that the ECM Additive "blended at 1.1% and 2.0% did not enhance the biodegradability of the polypropylene and polyethylene resins tested." Final Report: Compostability Testing of Polypropylene and Polyethylene Plastic Blended with "MasterBatch Pellets" Biodegradation Enhancement Additive," 3M Environmental Laboratory Project No. E10-0178 dated January 20, 2012, 3M-FTC-0000108.

72. These findings of no biodegradation are also consistent with the only peer-reviewed article that has addressed testing of the ECM additive. This article, by Gomez and Michel, entitled "Biodegradability of conventional plastics and natural fiber composites during composting, anaerobic digestion and long term soil incubation" tested two types of plastic blended with the ECM additive in soil incubation and anaerobic conditions. The article reported no statistically-significant biodegradation.

73. At least two additional scientific peer-reviewed publications that have evaluated blends similar to ECM Plastic conclude that these blends are not biodegradable. For example, an article reporting on tests of blends of 10% PCL (a biodegradable material) with 90% PE (conventional plastic) showed no biodegradation after 12 weeks of exposure to microorganisms. L. Tilstra and D. Johnsonbaugh, "The biodegradation of blends of polycaprolactone and polyethylene exposed to a defined consortium of fungi," Journal of Environmental Polymer Degradation, vol. 1, pp. 257-267, 1993. *See also* A. Iwamoto and Y. Tokiwa, "Enzymatic degradation of plastics containing polycaprolactone," Polymer Degradation and Stability, vol. 45, pp.205-213, 1994 (referring to blends of PCL and conventional plastics as "biodisentigrable").

ii. Microorganisms Do Not Produce Enzymes that Metabolize Plastic.

74. ECM's assertion that the presence of the additive allows biofilms to form, which in turn enables microorganisms to metabolize conventional plastic, has no scientific basis. A biofilm consists of naturally-occurring microorganisms. As discussed above, naturally-occurring microorganisms do not possess the necessary enzymes to break down high molecular weight conventional plastics, which otherwise remain resistant to microbial attack.[20] The microorganisms in the biofilm at best could only metabolize the biodegradable component. Accordingly, the presence of biofilm does not affect the conventional plastic, and ECM's claims have no scientific support.

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[20] Although there has been one documented instance of a particular strain of fungi capable of biodegrading a petroleum based plastic, *see* APPLIED AND ENVIRONMENTAL MICROBIOLOGY 6076–6084, "Biodegradation of Polyester Polyurethane by Endophytic Fungi," (Sept. 2011) there is no evidence that any of these strains exist in the U.S. or in landfills in the U.S.

iii. ECM's Test Results Are Unreliable.

75. The tests that purport to show the ECM Plastic is biodegradable are unreliable for many reasons. Each of these tests lack one or more of the necessary indicia of reliability. In addition, a number of these tests were conducted by laboratories whose practices fall far short of scientific standards.

76. In Section VI above, I identified a number of factors that must be present for a test to constitute reliable evidence for ECM's claims. The tests results that I reviewed lack several or, in some cases, all of these factors rendering each test unreliable. I discuss the factors lacking from the tests below.

77. ***Plastic Application.*** According to the Testing Materials, the only plastics that have been tested with the ECM additive are : PP, PE, PET, PS, LLDPE, EVA foam, HIPS. Thus, any claims of performance pertaining to other types of plastics are invalid. ECM's claims of biodegradability conferred to plastics generally is not adequately supported.

78. ***Load Rates.*** ECM claims that adding 1% of its additive to 99% plastic confers biodegradability on the ECM plastic as a whole. However, several of the Testing Materials included tests with higher load rates.

- "ChemRisk Assessment" (entitled "Ecologic Assessment of ECM Plastic, ECM-FTC-000106, conducted by the laboratory ChemRisk, as service of McLaren/Hart), used samples containing 50% load rate of ECM Additive, 5% ECM Plastic film, or just the ECM Additive alone.
- Advanced Materials Center, Inc. (AMC) Report conducted evaluations of ECM Plastic at 4.5%, 5% and 3% load rates.

- O.W.S. Inc: Final Report - Biodegradation Testing under Controlled Composting Conditions for 5% load films.
- O.W.S. Inc: Final Report - Biodegradation Testing for ECM Pellets and EMC Film tested a 50% film, 5% film, and the Additive.
- O.W.S. Inc: High Solids Anaerobic Digestion Test of ECM Pellets tested the ECM Additive alone.

79. Tests that do not follow the recommended load rate cannot be used to support claims regarding the recommended 1% load rate. Moreover, use of a high load rate of a known-biodegradable additive increases the likelihood that observed biodegradation is attributable to the priming effect.[21]

80. ***Inoculum.*** As noted earlier, the inoculum called for under the ASTM D5511 is unavailable in the United States. The inoculum used in quasi-ASTM D5511 tests that purport to show biodegradation of ECM Plastic were derived from a combination of compost (aerobic) and sludge from wastewater treatment plants. Neither source reflects the bacterial communities that would be found in landfills. Thus, all of the ASTM D5511 tests using this inoculum do not reflect actual conditions. Moreover, use of aerobic inoculum increases the likelihood that observed biodegradation is attributable to the priming effect.

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[21] There are also a substantial number of tests that do not identify the load rates or the specific type of plastic tested, including several included in the ECM Materials. *See, e.g.*, Analytical Report No. 1253020 regarding ASTM D 5511: Determining anaerobic biodegradation of plastic materials under high solids anaerobic digestion conditions; Update regarding ASTM D5511-11 Update on Clear Films 476 & 477 @ 45 days; SEM Examination of ECM Plastic; SEM imaging of bubble wrap; SEM imaging of EPS samples; SEM imaging of green PET bottles; SEM imaging of PVC samples.

81. ***Test Conditions.*** The temperatures for almost all of the tests are inconsistent with anaerobic conditions found in landfills. For instance, several tests were conducted under the thermophilic range more commonly associated with composting conditions. As a result, these tests cannot be used as valid scientific support for claims regarding typical landfills.

82. ***Sample Weight.*** Almost all of the gas evolution tests employ a low sample size to high inoculum weight ratio. Since the inoculum contains organic components, this skewing of the sample-to-inoculum ratio increases the likelihood that observed biodegradation is attributable to the priming effect.

83. ***Test Duration.*** No test was conducted long enough for the test samples to achieve 60% biodegradation. There are a handful of tests that were conducted for several years,[22] but the results of these tests are dubious because of the poor methodologies (*e.g.*, refreshing of inoculum, which reinvigorates the priming effect). Significantly, even these tests—conducted for as long as 900 days—did not show 60% biodegradation.

84. ***Proper Controls.*** No test controlled for the priming effect. As explained, observed biodegradation could be the result of the biodegradation of the ECM Additive itself (or the additive and the inoculum). One way to account for this possible overestimation is to use a control that includes the inoculum and the additive itself. By comparing these results to the negative control and the inoculum control, it is possible to calculate the amount of increased gas production due to the priming effect of the additive. Because no test included this type of control, none of the tests can rule out the possibility that the low-levels of biodegradation observed in the test are a result of the priming effect.

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[22] *See, e.g.*, Northeast Laboratories Report N1048340 testing ECM Plastic for 900 days; *see also* Northeast Laboratories Report 1149980 testing ECM Plastic for 365 days.

85. ***Statistical Significance.*** All but a few tests fail to report the statistical information that is necessary to understand the significance of the results. For tests conducted under ASTM methods, the standard deviation and confidence limits calculations are required to determine whether a percent biodegradation, if any, may be reported.[23] Also, as reported in the ASTM D5511 precision and bias section, the intra-laboratory sensitivity of the test is 5.1%, with a 95% confidence limit of 10.2%. This means that any single lab conducting the same test, under those same conditions, will obtain results within a 10% differential 95% of the time.[24] Given the sensitivity of the ASTM D5511 test, many of the results showing low-levels of biodegradation at the conclusion of the test should be disregarded because they are within the imprecision range of the test.

86. ***Qualitative Tests.*** A number of tests were submitted evaluating changes in the physical characteristics of the test specimen. For instance, one Northeast Laboratory test reports changes to molecular weight of the sample, stating that changes in the molecular weight demonstrate "that the polymer is breaking down." However, changes in molecular weight could also be indicative of disintegration rather than biodegradation. Importantly, the evaluation lacks a starting molecular weight for the test sample or the negative control. Assuming that the untreated sample approximates the starting molecular weight, the final molecular weight of the ECM Plastic after being exposed to anaerobic conditions for a year, demonstrate that the molecular weight has not been reduced enough to be metabolized by microorganisms. Only

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[23] *See* ASTM D5511 supra note 12, at 13.4 ("If the confidence interval on percentage biodegradation calculated in 12.5 includes zero, then the percentage biodegradation is not statistically significantly different from zero.")

[24] Although the precision and bias section does not report an inter-lab sensitivity, it is extremely likely that there would be greater error between labs.

short-chain, low-molecular weight polymers in the range of 500Mn are capable of being metabolized by naturally occurring microorganisms. This study reports the molecular weight of the test sample at the conclusion of the test to be almost 80,000Mn. Thus, this test, like many others, actually supports a conclusion that no biodegradation occurred and is consistent with possible fragmentation. Other evaluations of physical changes in appearance, mass, or chloride content of the leachate are likewise consistent with disintegration.

87. Not only do the Testing Materials reflect the lack of elements necessary for reliability, many of the tests were not conducted consistent with proper laboratory techniques or by qualified personnel.

a. Northeast Laboratories Tests

88. Based on the transcript provided to me, Northeast Laboratories, Inc. ("NE Labs") did at least four things that call into question the validity of the test results.

i. NE Labs replaced the inoculum.[25] This would likely lead to overestimation of biodegradation, expose the inoculum to oxygen, thus not simulating anaerobic conditions. This deviates from the ASTM method and calls into question the credibility of those conducting the lab.

ii. NE Labs used an inappropriate apparatus.[26] The apparatus used deteriorated over time, causing leaks and other potential problems in the system.

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[25] Transcript for the Deposition of Northeast Laboratories, Inc. ("NEL Dep. Tr.") May 9, 2014 at 87.

[26] *Id.* at 22-23, 88 (identifying metal paint cans as the containers for the test samples).

iii. NE Labs did not have someone with the proper education or training overseeing the test. [27] This is critical to ensure reliability. For instance, the gas results reported in all of its tests are inconsistent with typical biogas composition, which is roughly 55% to 70% CH_4 and 30% to 45% CO_2. Trace gases usually make up the remaining 5%. Because none of the tests report biogas compositions typical of anaerobic biodegradation, a properly trained and educated person overseeing these results would have likely evaluated the test method, apparatus, calculations, among other things, to determine the source of this anomaly.

iv. NE Labs conducted tests for periods well beyond the validation period of the test.[28]

b. Eden Laboratories

89. Likewise, Eden Laboratories, Inc. ("Eden") conducted tests using questionable modifications.

i. Eden is run by a person lacking the proper credentials and has received guidance from NE Labs on conducting biodegradation studies, indicating they likely follow common (improper) practice and techniques in this regard.[29]

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[27] *See, e.g.*, *id.* at 38 (identifying Ms. Ullman, a high school graduate completing her online degree in accounting as one of the people responsible for training lab techs).

[28] *See supra* note 22.

[29] *See, e.g.*, Transcript for the Deposition of Eden Research Laboratories, Inc. ("Eden Dep. Tr.") at 117-118.

ii. Eden acknowledged replacing the inoculum at the request of its customers.[30]

iii. Eden conducted tests for periods well-beyond the validation period of the test.

iv. Eden improperly modifies the raw data.[31]

90. The evidence indicates that the minimal biodegradation observed in the tests ECM relies on is the result of the "priming effect," *i.e.*, biodegradation of the additive (which contains organic compounds highly susceptible to biodegradation) and the organic materials of the test medium (the bacteria used for testing) rather than the plastic. Moreover, the minimal biodegradation observed is nearly always within the error limits of the test—which means that it is likely the result of testing limitations rather than true biodegradability of the plastic. In short, there is no competent and reliable scientific evidence to support the conclusion that the ECM additive—or any other similar additive—will cause plastic to biodegrade completely or more quickly than conventional plastics.

IX. CONCLUSION

84. In conclusion, in my expert opinion, ECM's claims that:

a. ECM Plastics will completely biodegrade within one year in a landfill is false and unsubstantiated.

b. ECM Plastics will completely biodegrade in nearly all landfills within nine months to five years is false and unsubstantiated.

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[30] *Id.* at 72-73.

[31] Eden Dep. Tr. at 111.

- **c.** ECM Plastics will completely biodegrade in most landfills within one to five years is false and unsubstantiated.
- **d.** ECM Plastics will completely biodegrade in any disposal environment at an appreciably faster rate and extent than conventional plastics without the ECM Additive is unsubstantiated.
- **e.** Various scientific tests, including but not limited to ASTM D5511, have shown that ECM Plastics perform as indicated in (a)-(d) above is false.

Stephen P. McCarthy
University of Massachusetts Lowell
Department of Plastics Engineering
One University Avenue
Lowell, MA 01854
Tel. 978-934-3417/Fax. 978-934-3065
Email: stephen_mccarthy@uml.edu

EDUCATION

Southeastern Massachusetts University, B.S., Textile Chemistry, 1978
Princeton University, M.S.E., Chemical Engineering, 1980
Case Western Reserve University, Ph.D., Macromolecular Science, 1984
Worcester Polytechnic Institute, Medical Device Management Certificate, 2005

PROFESSIONAL APPOINTMENTS

Owens Corning Fiberglas, Advanced R&D Engineer, 1980,
    Technical Center, Granville, Ohio
University of Massachusetts Lowell, Assistant Professor, 1984
    Department of Plastics Engineering
University of Massachusetts Lowell, Associate Professor, 1988
    Department of Plastics Engineering
University of Massachusetts Lowell, Professor, 1991
    Department of Plastics Engineering

RESEARCH INTERESTS

Mechanical, Physical, and Rheological Properties of Polymers, Blends and Composites
Medical Devices
Biodegradable Polymers
Plastic Polymer Blends and Reactive Processing
Bonding and Adhesion for Coatings, Co-extrusion, and Co-injection Molding
Design, Analysis and Rapid Tooling for Injection Molded Parts
Part Design

EDITORIAL ACTIVITIES

Editor, Journal of Polymers and the Environment, 1991-present

Editorial Board, Journal of Applied Polymer Science, 1995-2003

PROFESSIONAL ACTIVITIES

Board Member, UMCTTS Steering Committee, 2011-present
Reviewer, Biodegradable Polymer Institute (BPI), 1995 - 2012
Chair, Sections Committee, Society of Plastics Engineers, 2011-2012
Treasurer, SPE Medical Division, 2011-2012
Councilor, Society of Plastics Engineers Eastern New England Section, 2009-2012
Chair, SPE Medical Plastics Division, May 2011-present
Treasurer, BioEnvironmental Polymer Society (BEPS), July 2011-Present
Chair, SPE ANTEC Student Activities, May 2010-2011
Executive Board, ACS Division of Polymeric Materials and Engineering, 1993-2000
Councilor, Society of Plastics Engineering, 1991-1996
President, Bio/Environmental Polymer Society, 1995
Technical Program Chairman, Biodegradable Polymer Meeting, Boston, June 1994
Chairman, Fourth International Workshop on Biodegradable Polymers, Durham, N.H., October 1995
Secretary, U.S. Degradable Plastics Council, 1990-1992
Technical Program Co-Chairman, Society of Plastics Engineers, Inc. RETEC, Boston, MA, November 1990.
Technical Program Co-Chairman, Society of Plastics Engineers, Inc. RETEC, Boston, MA, October 1988

MEMBERSHIPS

American Society for Engineering Education
American Chemical Society
American Society for Standards and Materials
Bio/Environmentally Degradable Polymer Society
Biomaterials Society
Materials Research Society
Sigma Xi
Society of Plastics Engineers

AWARDS

SPE Education Award, Society of Plastics Engineers, December 2013
St. Patrick Parish Anam Cara Award, March 2013
Irish Education 100 Award, December 2012
Distinguished University Professor, University of Massachusetts Lowell, April 2012
BEPS James Hammar Memorial Lifetime Achievement Award, October 2008
SPE ANTEC Medical Plastics Division Best Paper Award, Boston, MA, May 2005
SPE ANTEC Color and Appearance Division Best Paper Award, Orlando, FL, May 2000
SPE Dedication of Service 1991-1996 Award, May 1996
SPI 46th Composites Institute Annual Conference Best Paper Award, Washington DC, 1991
The Modern Plastics Best Paper Award, Washington, DC, 1991
SPE ANTEC Advanced Composites Division Best Paper Award, Dallas, TX, May 1990
Plastics Processing Workshop Award, Taipei, Taiwan, June 1986

UNIVERSITY AND SERVICE ACTIVITIES

Faculty Athletic Rep, UMass Lowell Athletics Department
Graduate Coordinator, Plastics Engineering Department
Director, Massachusetts Medical Device Development Center
Director, UMass Lowell Bioplastics Institute (formally) Institute for Plastics Innovation
Director, NSF Center for Biodegradable Polymer Research, UMass Lowell
Co-Director, UMass Lowell Center for Irish Partnerships
Committee Member, Committee for Federated Centers and Institutes
Committee Member, Research Foundation Advisory Board
Committee Member, Plastics Engineering Department Computer Committee

Community activities

Eagle Scout Banquet Dinner for Sponsoring Scouts interested in Plastics Engineering
Spoke at Lowell High School Career Day
Assisted in helping children understand about plastics at the Plastics Museum, Leominster MA
Manufactured sled hockey bucket seats for the USA Para-Olympic Sled Hockey Team for Nagano Olympics
Lectured and gave laboratory tours of the University of Mass. Lowell Plastics Engineering Department to Local Boy Scouts for the Chemistry Merit Badge
Additional sled hockey bucket seats for the USA Para-Olympic Sled Hockey Team

INVITED LECTURES

Society of Plastics Engineers (SPE), China Plastics Conference – Medical Devices & Packaging, Shanghai, China, December 2013
Plastics in Medical Devices 2013, Waltham, MA, May 2013
UMass Lowell Delegation Trip, Kuwait City, Kuwait, January 2013
NIMAC Conference, Boston MA, October 2012
Shenkar College of Engineering & Design, Ram Gant, Israel, October 2012
MedinIreland Conference, Dublin, Ireland, October 2011
Emerging Technologies Conference, Dublin, Ireland, October 2011
Assistant Technology Turkish Workshop, Istanbul, Turkey, June 2011
Green Plastics – Current Developments & Future Perspectives Conference, Ram Gant, Israel, June 2011
BioEnvironmental Polymer Society 18th Annual Meeting, Toronto, Canada, October 2010
Boston-Ireland MedTech Conference, Galway, Ireland, October 2010
European Medical Polymers Conference 2010, Dublin, Ireland, September 2010
International Conference on Advancements in Polymeric Materials – AOM 2010 Trends & Technology, Bhubaneswar, India, February 2010
TAPPI – 2009 North American PLACE Flexible Packaging Summit, Columbus, OH, April 2009
Queens College, Belfast, February 2009
BioPharma EHS Forum, Cambridge, MA, September 2009
Medi 2008, Hartford, CT, September 2008
EASTEC 2008, West Springfield, MA, May 2008
SPE ANTEC Structures and Properties, Milwaukee, WI, May 2008
MassMEDIC's 9th Annual MedTech Investors Conference, Boston, MA, November 2007
SPE ANTEC Structures and Properties, Cincinnati, OH, May 2007
BioPlastics Processing Conference, Charlotte, NC, October 2006
The 7th Annual CAMD / CBM^2 Summer Workshop, Louisiana State University (LSU), Baton

Rouge, Louisiana, May, 2006
SPE Annual Technical Conference, Charlotte, NC., May 2006
Pacifichem Conference, Honolulu, HI, December 2005
BioEnvironmental Polymer Society 12th Annual Conference, Monterrey, Mexico, December, 2004
Green Chemistry, Gordon Conference, Bristol, RI, July, 2004
Palsto-Ispack Conference, Tel Aviv, Israel, September 2003
BioEnvironmental Polymer Society 11th Annual Conference, Denver, CO, August, 2003
Biodegradable Polymers Conference, Pisa, Italy, June 2002
BioEnvironmental Polymer Society 10th Annual Conference, Albuquerque, NM, September, 2002
ACS meeting, Polymers from Renewable Resources, Orlando, April, 2002
IOF Meeting, Washington, DC, July, 2002
Nedo Meeting, Brussels, March 2002
2001 BIOPOLYMERS - Advances in Medical and Material Science Applications, Cambridge, MA, October, 2001
Massachusetts Green Chemistry Research Symposium, University of Massachusetts, Amherst, MA October, 2001
Gordon Conference - Biodegradable Polymers, Queen's College, Oxford, UK, July, 2001
Gordon Conference, Ventura, CA, March, 1999
American Chemical Society Meeting, Anaheim, CA, March, 1999
5th International Scientific Workshop, Stockholm, Sweden, June, 1998
BioEnvironmental Degradation Polymer Societies Biodegradable Polymer Conference, Boston, MA, August, 1998
4th International Conference on Frontiers of Polymers and Advanced Materials, Cairo, Egypt, January 1997
Hamburg Makromolekulares Symposium, Hamburg, Germany, October, 1996
AICHE "Fifth World Conference of Chemical Engineering", San Diego, CA, July 1996
Corn Utilization Conference VI, St. Louis, MO, June, 1996
23rd Aharon Katzir-Katchalsky Conference, Tel Aviv, Israel, May, 1996
Thermoplastic Engineering and Design Conference, Detroit, MI, December, 1995
Pacifichem Conference, Honolulu, HI, December, 1995
"Plastics Waste Management Exhibition and Symposium", University of Connecticut, Storrs, CT, November, 1995
Annual Mold flow Conference, April 1995
TAPPI Conference, Orlando, FL, March 1995
Design Conference, Schenectady, NY, March 1995
Gordon Conference "Biodegradable Polymers", Oxnard, CA, February 1995
"Polymers from Renewable Resources and Their Degradation Symposium", Stockholm, Sweden, November 1994
"Environmentally Conscious Manufacturing for the Electronics Industry: Research on Advanced Materials and Processing, Yorktown Heights, NY, October, 1994
IUPAC Meeting, Akron, OH, July, 1994
BEDPS Conference, Boston, MA, June 1994
NSF Conference, Washington, DC, January 1994
3rd International Scientific Workshop on "Biodegradable Plastics and Polymers", Osaka, Japan, November 1993
Polymer Blends Conference, Akron, OH, October 1993
3rd Biochemical Engineering Conference, Taejon, Korea, September 1993

Gordon Conference “Biodegradable Polymers”, San Moniato, Italy, May 1993
Industrial Seminar at the University of Connecticut on Biodegradable Polymers, March 1993
Compalloy Conference, Newark, NJ, April 1993
Moldflow Conference, Valley Forge, PA, April 1993
Tandec Conference, Knoxville, TN, October 1992
Industrial Seminar on Biodegradable Polymers, Beijing, China, June 1992
Cornell University, Ithaca, NY, May 1992
Compalloy Meeting, Short Hills, NJ April 1992
International Polymer Conference, Jakarta, Indonesia, January 1992
Polymer Processing, National Taiwan University, Taipei, Taiwan, June 1986

SEMINARS

"Mechanical, Physical, and Rheological Properties of Polymers" Plastics Engineering Industrial Seminars, University of Lowell, 1985-present
Industrial Seminar at Clariant Corp. on June 2005
Industrial Seminar at Hewlett-Packard Company, Rehovot, Israel, 2003
Industrial Seminar at Exxon Company, Texas, 1999
“Medical Plastics Seminar", Plastics Engineering Industrial Seminars, University of Massachusetts Lowell, 1998
"Biodegradable Polymers and Blends", Plastics Engineering Industrial Seminars, University of Lowell, 1985
"Computer Aided Injection Molding", Plastics Engineering Industrial Seminars, University of Lowell, 1985
Industrial Seminar at 3M Company, Minneapolis, May 1992

CONTRACT AND GRANT SUPPORT

Avaya, (Kinetic Welding) (Intellectual property donation), $23,000,000
Solutia, (Nylon Pultrusion) (Intellectual property donation), $5,779,500
Commonwealth of Massachusetts, “Massachusetts Medical Device Development Center”, $4,000,000
Valyi (CSBM) (licenses of Intellectual property donation), $3,000,000
Metabolix, “Development of Novel of Biodegradable Materials, $1,500,196
NSF Center for Biodegradable Polymer Research, $1,200,000 Industrial Members (8/93-present), Principal Investigator
David Pernick UMass Lowell/Shenkar College Exchange Agreement, $ 1,000,000
Valtek, LLC, “Class A” Thermoplastic Automotive Finish without Painting, $625,000
Massachusetts Technology Collaborative John Adams Innovation Institute, “Massachusetts Medical Device Development Center”, $500,000
Polymer Degradation Research Center, $475,000, Industrial Members (8/89-8/93)
Digital, “Plastics Materials Research”, $458,706
US Army Natick, “Advanced Materials from Renewable Resources”, $365,596,
US Environmental Protection Agency, $300,004, (1998-2000), Principal Investigator
Institute for Plastics Innovation, Co-Director, $300,000, Industrial Members, (1995-present)
Lucent Technologies, Kinetic Weld, $165,000

Tycom, Undersea Photonic Connector, $161,000
Metabolix Inc., Performance of PHA Derived Chemicals and Polyols in Polyurethane, $141,465
Duracell Company, Polymeric Batteries, $140,000
3M, “Composting Research”, $155,000
Massachusetts Technology Collaborative John Adams Innovation Institute, “Massachusetts Medical Device Development Center”, $150,000
UMass Presidents office Seed Funding “Massachusetts Medical Device Development Center”, $135,000.
Monsanto Company, “Mold Analysis Research”, $123,000,
Warner Lambert, “Biodegradable Polymer Research”, $116,591
National Science Foundation, “Biodegradable Polymer Research Center”, $110,000 (8/93-8/95)
Massachusetts Centers of Excellence, “Mold Analysis Research”, $110,000
Department of the Army, “Polymer Degradation Research”, $104,000
Army Research Office “Aqueous Processing of Biopolymers”, $97,502
Digital Equipment Company, “Rapid Prototyping”, $86,500
American Composite Technology “Pultrusion Process Modelling”, $83,300
Cabot Corporation, “Material Research for Ear Plugs”, $77,740
Institute for Plastics Innovation, “Injection Molding Research”, $75,000
Massachusetts Centers of Excellence, “Institute for Plastics Innovation”, $75,000
Metabolix Inc., Performance of Polyhydroxyalkanote Derived Chemicals and Polyols in Polyurethane, $71,465
Xenith Athletics, Inc., “Development Project for Impact Protection Equipment”, $70,428
Vista Scientific, “Nanosphere-Antibiotic Corneal Contact Lens Delivery System, $ 70,000
Eastman Kodak, “Expert System Research”, $60,000
Battelle, “Biodegradable Packaging Development”, $59,865
DuPont Corian, $50,000
General Electric Company, “Mold Analysis Research”, $50,000
GTE / Sylvania Corporation, “Mold Analysis Research”, $49,000
Johnson & Johnson, “Material Research for Toothbrushes”, $45,500
Monsanto Company, “Polymer Degradation Research”, $40,000
Polysar Inc., “Polymer Degradation Research”, $40,000
Himont USA, Inc., “Mold Analysis Research”, $31,400
University of Massachusetts President Office, CVIP Technology Award, $30,000
Allied Signal, “Mold Analysis Research”, $30,000
Implant Sciences Corporation Biodegradable Implant for Radiation $30,000
Rexam Corporation, $30,000
Invista, “Evaluation of Plasticizers”, $ 28,000
Evenflo Products Company, “Baby Bottle Nipple Materials”, $27,000
Massachusetts Centers of Excellence, “Polymer Degradation Research”, $25,000
Polysar, Inc., “Permeability of Coextruded Sheet”, $24,000
United States Army ARDEC, “Materials Development for Armament Packaging”, $24,988
MoMelan Technologies, Inc., “Skin Grafting”, $23,000
Smart Surfaces Inc., “Smart Mattress”, $23,000
United States Army, “Theoretical Modelling of Transport”, $21,000
Exxon Company, “PVC Plasticizer Desorption”, $20,300
Advanced Electron Beam, “Low Energy Industrial Beams on Polymeric and Plastic Materials”, $20,000.
Eastman Kodak, “Mold Design Software Development”, $20,000
Dow Chemical Company, “Mold Analysis Research”, $20,000

Moldflow Pty Ltd, "Mold Analysis Research", $20,000
Giner, $20,000
Human Scale, $20,000
World Minerals, Inc., "Barrier Filler Research", $15,000
Osram-Sylvania Inc., Injection Molding Research, $15,000
Nypro, $10,000
Densified Solutions, "Extrusion Testing", $5,000
MMM management & Publications LLc, "Processing Polyactic Acid", $4000

PATENTS ISSUED

"Micro and Nanospheres Based on Natural Materials", Pending, with Balint Koroskenyi and Robert Nicolosi
"Novel Biodegradable Bone Plates and Bonding Systems", with Jeffrey Weinzweig, USA Patent No. US2008/0234754 A1, Effective from September 25, 2008.
"Polysaccharide-Containing Block Copolymer Particles and Uses Thereof", with Balint Koroskenyi and Robert Nicolosi, Pending, USA Patent Application 20110020227.
"Polysaccharide-Containing Block Copolymer Particles and Uses Thereof", with Balint Koroskenyi and Robert Nicolosi, USA Patent Application 7,763,663.
"Surface Finishing Compression Molding with Multi-Layer Extrusion", with Arthur Delusky, Robert Lucke, Thomas Ellison and Qing Guan, USA Patent No. 6,770,230.
"Surface Finishing Compression Molding with Multi-Layer Extrusion", with Arthur Delusky, Robert Lucke, Thomas Ellison and Qing Guan, USA Patent No. 6,964,802.
"Molded Article and Process for Preparing Same", with Thomas M. Ellison, Arthur Delusky and Qing Guan, USA Patent No. 6,670,028.
"Process and Apparatus for Preparing a Molded Article", with Thomas Ellison, and Arthur Delusky, USA Patent No. 6,506,334.
"Molded Article", with Thomas M. Ellison, Arthur K. Delusky, Robert Lucke, USA patent No. 6,440,593.
"Refined Vegetable Oils and Extracts Thereof", with Carl Lawton, Robert Nicolosi, USA Patent No. 6,197,357 B1.
"Bioresorbable Copolymers", with Richard A. Gross, Xianhai Chen, USA Patent No. 6,093,792.
"Polylactic Acid-Based Blends", with, Richard Gross, and Wenguang Ma, USA Patent No. 5,883,199.
"Composition of and Method for Forming High Molecular Weight Predominately Syndiotactic Substituted-Poly (β-Propioesters)", with Richard Gross and John Kemnitzer, USA Patent No. 5,440,007.
"Biodegradable and Hydrogradable Diblock Copolymers Composed of Poly (β-Hydroxyalkanoates and Poly (Lactones) or Poly (Lactide) Chain Segments", with Richard Gross and Michael Reeve, USA patent No. 5,439,985.
"Gamma-Poly(Glutamic Acid) Esters", with Richard Gross and Devang Shah, USA Patent No. 5,378,807.

PUBLICATIONS

Synthesis and characterization of pullulan-polycaprolactone core-shell nanospheres encapsulated with ciprofloxacin, Sally Shady, Peter Gaines, Rahul Garhwal, Charles Leahy, Edward Ellis, Kathryn Crawford, Daniel Schmidt, Stephen McCarthy, Journal of Biomedical nanotechnology, Vol. 9, Issue 9, (2013) pg. 1644-1655.

A study on recycling of polyhydroxybutyrate (PHB) copolymer and its effect on material properties, Annual Technical Conference - Society of Plastics Engineers, Bhavin Shah, Robert Whitehouse and Stephen McCarthy vol. 70, (2012) pg 260-264.

"Improved dialysis technique for core-shell pullulan-polycaprolactone (PCL) nanospheres loaded with hydrophobic ciproflaxcin", Sally Shady-Elghamrawi, Daniel Schmidt & Stephen McCarthy, (2011), 69th (vol. 3), pp1982-1988.

"Electrospun silk material systems for wound healing", Scott E. Wharram,.; Xiaohui Zhang; David L Kaplan.' Stephen P. McCarthy, Macromolecular Bioscience (2010),10(3), pp246-257.

"The Effect of Hyperbranched Polymers on Processing and Thermal Stability of Biodegradable Polyesters", Y. Shaked, Hanna Dodiuk, Samuel Kenig and Stephen McCarthy, Polymer Engineering and Science, (2009).

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"Processing and Blends of Biopolymers", 66th Annual Technical Conference – Society of Plastics Engineers, (2008)

"Improving the toughness of poly(lactic acid)(PLA) through co-continuous, immiscible, biodegradable blends with PHA". With Jinkoo Lee, Annual Technical Conference - Society of Plastics Engineers (2007), 65th 1569-1572.

"The effect of polymer surface on the wetting and adhesion of liquid systems", P.F. Rios, H. Dodiuk, S. Kenig, A. Dotan, Journal of Adhesion Science and Technology (2007), 21(3-4), 227-241

"Transparent ultra-hydrophobic surfaces", P. F. Rios, H. Dodiuk, S. Kenig, A. Dotan, Journal of Adhesion Science and Technology (2007), 21(5-6), 399-408. CODEN: JATEE8 ISSN:0169-4243.

"The Effect of Nanoclays on the Properties of PLLA-modified Polymers: Part I: Mechanical and Thermal Properties", D. Lewitus, A. Ophir, S. Kenig and S. McCarthy, J. Polymers and the Environment, Vol 14, no. 1, pp. 171-177, (2006).

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"Mechanical Testing and Characterization of Biopolymers", Society of Plastics Engineers Annual Technical Conference, (Vol. 64), (2006).

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"Biodegradable Hollow Nanospheres for Drug Delivery", with Balint Koroskenyi, Robert Nicolosi, Society of Plastics Engineers Annual Technical Conference, (2005), (Vol. 63), p. 3096-3099.

"Electrically Conductive Nano-composites in Powdered Injection Molding", with Ankur Maheshwari, Society of Plastics Engineers Annual Technical Conference, (2003), (61st) (Vol. 61), pp. 616-619.

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"Biodegradable Plasticizers for Polylactic Acid", Xu Song & Stephen McCarthy, Journal of Applied Medical Polymers, Autumn 2002, Vol 6, No.2, 64-69.

"Microwave-Assisted Solvent-Free or Aqueous-Based Synthesis of Biodegradable Polymers", with Balint Koroskenyi, Journal of Polymers and the Environment, Volume 10, Number 3, July 2002, 93-104

"Process Dynamics in Sequential Valve Gate Injection Molding-Processing Strategy and Process Control", Shin, Suk-young; Schott, Nick; McCarthy, Stephen., Society of Plastics Engineers Annual Technical Conference (2001), 59th(Vol. 1), 694-698.

"Biodegradable Plasticizers for Polylactic Acid", McCarthy, Stephen; Song, Xu. Annual Technical Conference - Soc. Plast. Eng. (2001), 59th(Vol. 3), 2546-2549.

"Large, Structural, "Class A" Thermoplastic Automotive Part Production Without Painting", McCarthy, Stephen; Guan, Qing; McCarthy, Shawn; Shetty, Malar Rohith; Ellison, Thomas, Annual Technical Conference - Soc. Plast. Eng. (2001), 59th(Vol. 3), 2448-2452.

"Biodegradable Bags Comparative Performance Study: A Multi-Tiered Approach to Evaluating the Compostability of Plastic Materials" Farrell, R. E.; Adamczyk, T. J.; Broe, D. C.; Lee, J. S.; Briggs, B. L.; Gross, R. A.; McCarthy, S. P.; Goodwin, S. ACS Symp. Ser. (2001),786 (Biopolymers from Polysaccharides and Agroproteins),

"Biodegradable Polymer Blends for Medical Applications", with P. Canale, S. Mehta, Journal of Applied Medical Polymers, Volume 5, No. 2, Pgs. 65-71, (Autumn 2001)

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"Evaluation of Oxidative Stability of Flexible Polyolefins by Oxidative Induction Testing with γ-Oryzanol and α-Tocopherol Antioxidants for Food and Medical Applications", with Shamit Hakani, Sanjay Mehta, and Philip Canale, Journal of Applied Medical Polymers, Volume 4, No. 2, (Winter 2000).

"Hydrolytic Degradation of PCL/PEO Copolymers in Alkaline Media", with S.M. Li, X.H. Chen and R.A. Gross, Journal of Materials Science: Materials in Medicine, Volume 11, pgs. 227-233, (2000).

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"Cavity Pressure Transfer Extends Prototype Tool Life", with John Dell'Arciprete and Robert Malloy, Modern Plastics, Edited by William A. Kaplan, pgs. 125-128, (January 2000).

""Kinetic" Welding of Plastic Parts", with Judith A.H. Jones, Yogish Mahadevaiah, Balint Koroskenyi, and Sanjay Mehta, 58th Society of Plastics Engineers Annual Technical Conference Proceedings, Volume 46, Number 1, pp. 1232-1236, May 2000.

"Thermoplastic Paint (A.k.a. Film Finish, Paint Film, Dry Paint) A Complementary Technology for Exterior Automotive Plastic", with Thomas Ellison, 58th Society of Plastics Engineers Annual Technical Conference Proceedings, Volume 46, Number 1, pp. 2607-2610, May 2000.

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"Biodegradable Polymer Blends for Medical Applications", with Philip Canale and Sanjay Mehta, 58th Society of Plastics Engineers Annual Technical Conference Proceedings, Volume 46, Number 3, pp. 2680-2684, May 2000.

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""Single-Site" Catalyzed Polyolefin for Fresh-Cut Produce Packaging – A Comparison Between Monoextruded Blends and CoExtruded Film", with V. Patel, S. Mehta, and S. Orroth, 58th Society of Plastics Engineers Annual Technical Conference Proceedings, Volume 45, Number 1, pp. 391-395, May 1999.

"Cavity Pressure Studies for Stereolithography Produced Tooling", with John Dell'Arciprete and Robert Malloy, 58th Society of Plastics Engineers Annual Technical Conference Proceedings, Volume 45, Number 1, pp. 467-471, May 1999.

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"A New Approach for In-Mold Finishing: The Valyi Surface Finishing/Compression Molding Process", with Qing Guan, Sandip Patel, and Thomas Ellison, 58th Society of Plastics Engineers Annual Technical Conference Proceedings, Volume 45, Number 1, pp. 2911-2914, May 1999.

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Respondent's Exhibit V

In the Matter of ECM BioFilms, FTC Docket No. 9358

Rebuttal Report by Dr. Frederick C. Michel

Rebuttal of Expert Reports by Drs. Ryan Burnette and Ranajit Sahu

Table of Contents

Qualifications

My qualifications to rebut the expert testimony include a PhD in Chemical Engineering and Bachelor's degrees in Chemical Engineering and Biochemistry. After earning my PhD, I was a postdoctoral fellow in the NSF Center for Microbial Ecology (CME), a leading research center in this area. I worked under Center Director and past chair of the American Society of Microbiology, Professor Jim Tiedje as well as with Drs. Larry Forney and Professor CA Reddy. I am currently a tenured Associate Professor in the Department of Food, Agricultural and Biological Engineering at the Ohio State University with an adjunct appointment in the Department of Chemical and Biomolecular Engineering.

For the past 25 years I have conducted research on a wide range of environmental topics including the biodegradation of plastics, bioplastics, biofoams and natural fiber materials in anaerobic digesters, composting systems and in soils. I have also conducted research on the fates of pesticides and PCBs during composting and anaerobic digestion. As a part of these projects I used C^{14} labeled compounds to determine the rate and extent of pesticide and PCB biotransformation, mineralization, volatilization and conversion to humic materials. At the CME I was the lead scientist on a project to investigating the full scale bioremediation of PCB contaminated soils. My lab is currently involved in investigations of the degradation of recalcitrant herbicides during composting and anaerobic digestion and has developed bioassays to measure the phytotoxicity that these residues confer.

For my PhD work, I investigated the production of lignin and manganese peroxidase enzymes by fungi that grow as biofilms and suspended pellets. I also investigated the ability of these organisms to decontaminate chlorinated byproducts generated by paper mills. These enzymes are known to initiate the biodegradation of some of the most recalcitrant molecules in the environment such as lignin and chlorinated organic compounds. My work at the CME and at the Ohio State University has included studies of the microbial ecology of composts and anaerobic digesters using novel nucleic acid based methods. I also have conducted investigations on the effects of composting parameters on compost quality, odor production and compost microbiology.

I have authored over 40 peer reviewed publications and many other papers and reports in these areas. In 2010, I received the USCC Rufus Chaney award for research excellence from the US Composting Council. From 2007 to the present I have served as the Editor of Compost Science and Utilization journal, an international peer reviewed scientific journal and as an Associate Editor for the Biological Engineering division of the American Society of Agricultural and Biological Engineers. I also developed, and have organized and taught the Ohio Compost Operator Education course since 2001.

In addition to my University position, I have served as consultant to a variety companies on topics related to composting, microbial ecology, and plastic and organic contaminant biodegradation. These included AllTreat Organics Composting (ON), OEW Recycling (OH), White Barn Farms (OH), United States Industry Coalition (VA), The Scotts Company (OH), DuPont (DE), Green Paper Products (OH), Keramida Environmental (IN), Ralston Instruments (OH), Indian Summer Composting (MI), Economic Development Generating Excellence (OK), US Composting Council (MD), Federal Trade Commission (DC), GT Environmental (OH), Barnes Nursery (OH) and Quasar Bioenergy.

To date, I have served as an expert witness in three (3) separate proceedings. I was retained by The City of Kirtland, OH in 2013 and by Canton OH, in 2001 to provide expert testimony on odors produced during the composting process and in 2013 by Crabbe, Brown & James, of Columbus, OH to provide expert testimony on a proposed food waste composting facility in that city (see Appendix B).

Based on my education, training and experience I have been retained by the FTC to provide this rebuttal report and testimony. I am compensated at the rate of $100 per hour and at $200 per hour for testimony and deposition.

I have previous knowledge of ECM in that my group tested the biodegradability of ECM amended plastics as part of a larger study in which we tested the biodegradability of various plastics and natural materials that could be used to make compostable or recyclable nursery pots.

Frederick C. Michel Jr., PhD

Introduction

I was asked by FTC counsel to provide rebuttal of expert reports by Drs. Sahu and Burnette. Below I present their comments (in italics) followed by my rebuttal comments.

Rebuttal of points made by Dr. Burnette

Comment by Dr. Burnette -

"26. MSW landfills are dynamic environments which attract and sustain a wide variety of microbial life. The range of conditions in MSW landfills provide ample opportunity for a variety of bacteria to thrive that possess the biochemical tools necessary for biodegradation, despite what appear at the human level to be challenging environmental conditions. The studies referenced here provide direct evidence that these biodegradation-capable microorganisms are present and thriving in MSW landfills, as they were isolated and characterized directly from MSW landfill leachate. Subsequent sections of this report will document that this diversity of microbial life possess enzymes capable of degrading both natural and synthetic plastics, and that these enzymes are biologically active in MSW landfill environments, and can behave similarly in testing conditions such as the ASTM D5511 protocol. It is reasonable to conclude that many of these microorganisms and their enzymes are present in the landfill leachate used, and will act on ECM treated plastics."

Rebuttal-

I agree with Dr. Burnette that tremendous microbial diversity exists in landfills (Zhang et al., 2011). Yet equally if not more diverse microbial communities exist in soil, compost and anaerobic digesters, including those used for biodegradability testing such as the ASTM D5511 protocol. For example Torsvik and Øvreås (2002) have shown that a single gram of soil harbors up to 10 billion microorganisms and thousands of different species. Studies have also shown extensive microbial diversity in samples of activated sludge and compost (Liu et al., 1997; Michel et al., 2002).

Since in biodegradability tests typically use more than 100 grams of soil or activated sludge, there likely is sufficient phenotypic and genotypic diversity for the biodegradation of even recalcitrant materials such as polyethylene in these test environments (assuming, of course, that such recalcitrant polymers were, in fact, biodegradable).

Comment by Dr. Burnette –

Regarding a peer review publication I co-authored (Gomez and Michel, 2013) Dr. Burnette makes the following comments about the "Plateau effect".

"33. The argument that the testing environment is the reason for the plateau effect, and not the plastic itself, can be seen even in peer-reviewed literature that contradicts biodegradation of plastics (Gomez & Michel, 2013). In this report, the positive control cellulose paper exhibited approximately 74%

biodegradation under anaerobic conditions at approximately 30 days incubation. Further biodegradation of the cellulose paper was negligible between 30 and 50 days, when the test concluded. In other words, even a known positive control experienced a plateau effect under laboratory testing conditions."

"72. These are all important confounding considerations when evaluating the results of biodegradation tests. For example, Complaint Counsel has pointed to a particular peer-reviewed journal article where ECM treated plastics were tested for biodegradation under anaerobic conditions, and they reported biodegradation of these samples was not observed (Gomez & Michel, 2013). These authors utilized a modified ASTM D5511 test protocol. You must interpret the results against the controls presented in the test. In this series of tests, the positive control cellulose paper only reached 74% biodegradation, and plateaued from 30 days until the conclusion of the test at 50 days. From this, it is reasonable to conclude that conditions that promote biodegradation expired close to the 30 day point. This is a perfect example of the importance of experimental limitations, and the impact they can have on the interpretation of results. We may surmise from this defect in test design that the environment present was not hospitable to biodegradation, because even the positive control cellulose, established in many tests to be a biodegradation catalyst, failed to sustain biodegradation beyond 30 days."

Rebuttal -

In our study (Gomez and Michel, 2013), we examined a wide range of commercially available materials used to manufacture plastic products. The biodegradability of these materials was assessed by measuring the amount of carbon mineralized from them during incubation in three environments; soil, anaerobic digestion and composting.

During soil incubation for 660 days no significant mineralization was observed for polyethylene or polypropylene plastics or the same plastics amended with ECM additive. During 50 days of anaerobic digestion, less than 2% of ECM additive containing plastics were converted to biogas (CH_4 + CO_2). After 115 days of composting, only 0.6% of an ECM additive amended polypropylene was converted to carbon dioxide. In all three tests more than 70% of the positive control (cellulose) was converted to CO_2. Two figures from this manuscript are copied below (Figures 1 and 2).

In all three environments, the biodegradation of the positive control "plateaued" at around 70-75% mineralization. Dr. Burnette states that this plateau is due to accumulation of byproducts that hinder further mineralization of the cellulose or that the environmental conditions were no longer conducive to biodegradation. However contrary to this it can be seen in the figures that some of the other materials, (e.g., Plastarch, a corn starch polyester blend and PHA) continued to biodegrade well after cellulose biodegradation had plateaued. They continued to degrade in a linear fashion to the end of the experiment. Since the plastics are a small component of the material in the reactors, this indicates that the environment was not inhibitory to the biodegradation process, but that the plateau occurred while there was still an environment conducive to biodegradation (Figure 1 and 2).

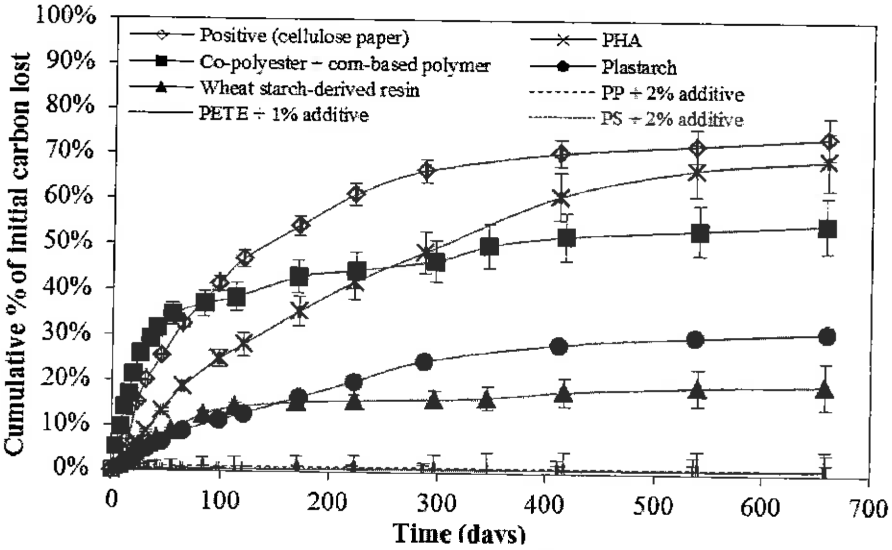

Figure 1. Cumulative carbon loss (CO_2-C) as percentage of initial carbon (± cumulative standard error) for bio-based plastics and for conventional plastics amended with additives during 660 days of soil incubation. For some data points standard error bars are smaller than markers.

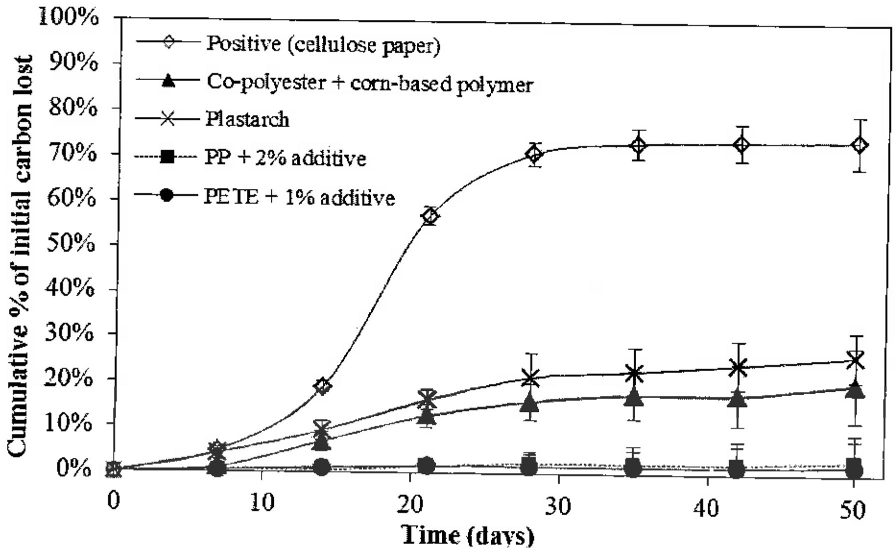

Figure 2. Cumulative carbon loss (CO_2-C) as percentage of initial carbon (± cumulative standard error) for bio-based plastics, conventional plastics amended with additives and natural fiber composites during 50 days of anaerobic digestion. For some data points standard error bars are smaller than markers.

This plateau is observed in nearly every biodegradation test of this type and is recognized in ASTM 5210, ASTM 5511 and ASTM 5526 standard tests of aerobic and anaerobic biodegradation. The reason for the plateau is that biodegradation includes not only mineralization to terminal electron acceptors like CO_2 and CH_4 but also metabolism of compounds such as proteins, cell wall components, chitin, and other biological materials by cells. Microorganisms also generate waste products including humic and fulvic acids that are recalcitrant. The carbon used for respiration is proportional to the mineralization to CO_2 and CH_4 while metabolic products do not leave this signature until they are biodegraded when cells die, which may not occur for a much longer period of time.

An analogy would be when humans consume food. One hundred percent of the food is not converted to CO_2. Some is converted to muscle, fat and other products. These products are not converted to CO_2 until a person dies. Microorganisms likewise create products other than CO_2 from the substrates they consume and these typically biodegrade much more slowly. Therefore 100% mineralization of the positive control is not expected. This is why one criterion for a valid ASTM test is that more than 70% of the positive control has been converted to CO_2, not 100%.

Burnette is mistaken in his interpretations that the plateau is evidence of a flawed test, or that the test environment has become toxic to further degradation, and that conditions are "not hospitable to biodegradation". The plateau is evidence that the biotransformation of cellulose was complete and that the remaining carbon had been converted into more recalcitrant residues.

Comment by Dr. Burnette-

30. Biofilms are easily developed on PVC surfaces (Pedersen, 1990), as evidenced by J and S traps on sinks and toilets with PVC fittings. It is likely that formation of biofilms aided in the biodegradation of an ECM treated PVC product, BioPVC®. Over a test period of 6 months, appreciable loss of mass, compared to the negative control PVC material, was witnessed with the BioPVC®. In fact, even after one month, there was statistically significant loss of mass of the BioPVC® compared to the control PVC (Environ Corp. Lab, Report dated November 29, 2007).

Rebuttal

In this study of BioPVC biodegradation (2008 Environ BioPVC Report.pdf), only a 2.5% mass loss was observed after 10 months and the loss plateaued after 7 months and did not increase from 7 to 10 months. A more likely conclusion from these data is that the ECM additive did biodegrade, but that the PVC did not.

Comment by Dr. Burnett.

"74. Further, cellulose is a better positive control to provide confidence in the overall biological activity of the test, than it is as a positive control for the biochemistry of biodegradation of plastics. Cellulose is biodegraded by an enzyme called cellulase, a widely-abundant enzyme in nature. It is unlikely that cellulase plays a direct role in biodegradation of plastics, however (see discussion on enzymes above). Therefore, these tests are not an "apples to apples" comparison of the enzymes in biodegradation. Rather, cellulose, and the cellulase enzyme that biodegrades cellulose, is really a positive control that verifies the biological viability of the assay generally. It may not verify performance in the presence of biological activity from other areas of the MSW landfill that might degrade the test article."

Rebuttal –

While it is true that the test conditions may be optimized for cellulose biodegradation and not for polyethylene degradation, these types of biodegradation tests are used and widely accepted for many different types of organic chemicals. Similar tests have been used to test polymers, herbicides, organic chemicals, natural materials, biopolymers and hydrocarbons very similar in structure to conventional plastics (Desai et al., 1990; Sanchis et al., 2013; Miles and Doucette, 2001; Gomez and Michel, 2013; Albertsson, 1977; Albertsson, 1978; Albertsson et al., 1978). Furthermore while Dr. Burnette correctly notes that Cellulase is a widely abundant enzyme produced by many different types of microorganisms, he does not explain why few if any polyethylene or polypropylene degrading enzymes (plasticases?) have been isolated from nature nor described in the scientific literature.

Comment by Dr. Burnett -

"76. One common criticism of the results (discussed below) is that the biodegradation witnessed, say, in change of dry mass, is minimal if not negligible. It is important to consider that biodegradation has only been tested in one possible environment experimentally replicated. Greater biodegradation would necessarily be observed if the test material were analyzed in a sampling of different possible MSW landfill environments, such as manipulating oxygen or pH levels. These changes in variables may provide for the rise of different microbial populations that can further the biodegradation process."

Rebuttal-

Dr. Burnette makes the point that test environments have limited ranges of temperature, pH and oxygen concentration and therefore cannot mimic the more diverse environment within a landfill where temperatures range from 10 to 50 C. Yet as noted in reports by FTC expert Dr. Thabet Tolaymat, water is actively removed and oxygen is not present within a landfill. In this type of environment, microbial diversity and activity is likely to be lower and limited as compared to environments where microorganisms are active, temperatures are warm and sufficient moisture and diverse substrates are present. These conditions are used in biodegradability tests to provide an optimal environment for microbial degradation. Furthermore, within compost, anaerobic sludge granules and soil aggregates, gradients of oxygen, pH, substrates, electron donors and minerals exist over very small spatial scales

(Kim and Crowley, 2013). So a range of these conditions is likely present even in biodegradation tests, not just the average values measured for these systems.

Given the tremendous diversity of microorganisms in these systems, biodegradable substrates should be able to be biodegraded by some member or combination of members of the community present in the tests. Therefore, while imperfect, these tests provide a very good estimate of the biodegradation of compounds under favorable conditions for microbial activity.

Comment by Dr. Burnette -

"78. An argument has been made that when test results are negative, it is because the ECM treated plastics are not biodegradable. At the same time, supporters of that argument will argue that positive results are an anomaly of the testing environment. Those arguments are fundamentally flawed; you must accept negative and positive results together and evaluate the totality of the scientific evidence. Either the test method is valid or it is not. It cannot be valid when only one outcome, negative or positive, is observed. Many of the tests with ECM treated plastics have well-designed controls, where the negative control does not biodegrade, and the positive control does biodegrade. In these cases, when the ECM treated material does also appear to biodegrade, the conclusion drawn is that the ECM treated plastics are biodegradable within design of the experiment. There is great support that the ECM treated plastics are in fact biodegradable, both in vitro and in situ."

Rebuttal –

As described by Dr. McCarthy in his report, only a few tests use proper controls and few report the actual amount of ECM additive present in the plastic. Since the additive itself may be much more biodegradable than the plastic with which it is mixed, the conclusion that "there is great support that the ECM treated plastics are in fact biodegradable", is correct to the extent that the additive itself is biodegradable. This does not mean that the ECM additive has enhanced the biodegradation of the conventional plastic.

Many of the reports where ECM amended plastics have been observed to biodegrade greater than the negative control can be attributed to the biodegradation of the ECM additive, or to the priming effect (Shen and Bartha, 1996), and not the plastic to which it has been added.

Rebuttal of points made by Dr. Sahu

General response to Dr. Sahu's "threshold question" –

Much of the report by Dr. Sahu focuses on the "threshold question" which he defines as whether conventional plastics like polyethylene and polypropylene are "biodegradable", even over very long time scales.

The "threshold question" however is really not the central issue. His threshold assessment of biodegradability would allow even rocks or metals to be defined as "biodegradable" since under certain conditions, over long eons of time, microorganisms can produce acids or other compounds that lead to their weathering, corrosion and eventual mineralization.

Biodegradation in the context of disposable consumer products must mean something different. It means that a material will biodegrade to natural products over a time frame used for municipal waste management via composting, anaerobic digestion and/or land filling. It also implies that materials will biodegrade rapidly if they end up in natural environments and will not accumulate. The massive islands of plastic pollution now collecting in the world's oceans (Figure 3), plastic particle pollution in natural bodies of water like the Great Lakes (Erikson et al., 2013) and the plastic that pollutes many of the world's beaches and natural areas demand nothing less.

It would be unfortunate if materials that claim to be "biodegradable" actually contribute to these problems. Therefore convincing scientific evidence must be presented that demonstrates biodegradation over short time frames, not decades, centuries or millennia. For these reasons, the "threshold question" in the context of conventional or ECM plastics, is moot.

Figure 3. Accumulated plastic ocean pollution.

Comment by Dr. Sahu

On Page 8, Para 2 of his report Dr. Sahu states that,

"It is my opinion that the expectation that all plastics with the ECM additive added in the usual amount (i.e., at a level of 1 or at most a few percent) should completely (another term that is used throughout the reports of Drs. McCarthy and Tolaymat) degrade in typical landfill conditions, in a time period of 1 year or even 5 years, is unrealistic."

Rebuttal

Dr. Sahu appears to agree with the central point in the case which is that it has not been demonstrated that ECM amended conventional plastics will biodegrade in a landfill in 1-5 years.

To obtain accurate evidence of biodegradation, experiments are best performed using ^{14}C-labeled substrates and measuring evolved $^{14}CO_2$ over time. One such long-term biodegradation study (Albertsson, 1970), on ^{14}C-labeled polyethylene that utilized CO_2 trapping and liquid scintillation counting, indicated that PE biodegradation is extremely slow requiring extremely long periods. After 10 years of soil incubation, <0.5% polymeric carbon (as CO_2) by weight was evolved from a UV-irradiated polyethylene sheet. The extent of conversion was even lower (0.2% w/w) in the absence of irradiation (Albertsson, 1970).

Only when PE is modified through UV irradiation for long periods of time so that carbonyl bonds are formed, or when hydrolysable linkages or unsaturated sites are introduced into the polymer does it display a very limited ability to biodegrade (Prasun et al., 2011). Pretreatment by incubating PE at high temperatures can also modify PE by reducing the molecular weight of the polymer which improves biodegradation. However without these abiotic and chemical and physical modifications, the extent of PE biodegradation is essentially nil.

Comments by Dr. Sahu

On page 52, Dr. Sahu rebuts Dr. McCarthy's Comments:

"VII.13 Specific Rebuttal to Dr. McCarthy's Testing Comments

"Next, I address some of the more specific criticisms by Dr. McCarthy. His comment/discussion is first noted in italics followed by my discussion/response within parentheses below.

[SM report para] "18.....First, none of the Testing Materials supports the conclusion that ECM Plastic is biodegradable or will biodegrade to completion; in fact, the only published, peer reviewed study to address this question concludes otherwise.3

3 See E. Gomez & F. Michel, Biodegradability of conventional plastics and natural fiber composites during composting, anaerobic digestion and long term soil incubation, 98 JOURNAL OF POLYMER DEGRADATION & STABILITY 2583-91 (2013) (testing the ECM additive and concluding that "plastics *containing additives that supposedly confer biodegradability to polymers such as polyethylene and polypropylene did not improve the biodegradability of these recalcitrant polymers")..."*

(Dr. McCarthy is wrong. Gomez and Michel is not the only published and peer reviewed study to address this question. See my discussion on the threshold question including the numerous citations (and the many more citations within the cited papers). Several of the papers that I cite specifically address PE/PP biodegradation.)"

Rebuttal-

Actually, the paper by Gomez and Michel (2013) is the only peer reviewed scientific publication to report on the biodegradation of ECM amended plastics. Even after very long incubation periods in soil, during composting and during anaerobic digestion, ECM amended plastics did not biodegrade to a greater extent than unamended polyethylene. Neither conventional nor ECM amended plastic degraded to a significant extent in any of the three environments reported in the paper, while the positive control (cellulose) and biodegradable plastics made from polyhydroxyalkanoate were mineralized to a significant extent (see Figures 1 and 2).

Sahu Comment-

"These findings of no biodegradation are also consistent with the only peer reviewed article that has addressed testing of the ECM additive. This article, by Gomez and Michel, entitled "Biodegradability of conventional plastics and natural fiber composites during composting, anaerobic digestion and long term soil incubation" tested two types of plastic blended with the ECM additive in soil incubation and anaerobic conditions. The article reported no statistically-significant biodegradation."

(Again, "no statistically-significant biodegradation" is quite different than concluding that there was no biodegradation. Dr. McCarthy is mistaken.)"

I show, unequivocally, that plastic polymers containing the ECM additive will biodegrade on a much faster timescale than such polymer plastics without the ECM additive."

Rebuttal -

It is not clear how or where Dr. Sahu has shown unequivocally that this is the case. It is true that ECM amended plastics will biodegrade to a greater extent than unamended plastics, but only because the ECM additive itself apparently biodegrades at a much faster rate than the plastics to which it has been added.

Addition of additives into conventional plastics does not increase the carbonyl content of the plastic nor does it reduce the molecular weight of the high molecular weight polymers or add hydrolysable linkages or unsaturated carbon bonds. Consistent with this fact, studies in which even large percentages of starch have been incorporated into PE (50% to >80%) do not show any improvement in the biodegradation of the PE fraction (Nakashima et al., 2002). For example blends of 50% and 83% starch added to polyethylene displayed a maximum of 49% and 78% weight loss upon 16 months incubation in soil (Nakashima et al., 2002).

Sahu comment –

On page 47, Dr. Sahu makes the following comment regarding Dr. McCarthy's assertions about C^{14} testing arguing that it is unwarranted and too difficult to do.

"VII.10 Radiological Testing is Impractical and Unwarranted

Dr. McCarthy states that the only appropriate confirmatory tests are either radiological, using ^{14}C in the plastic and tracking the resulting $^{14}CO_2$ and $^{14}CH_4$ that would evolve from biodegradation of this material. He does not, however, mention how this could be done as a practical matter. He does not mention how one can formulate (or ask a manufacturer to formulate) such materials with the ECM additive in small batch quantities, just for testing purposes; nor the many practical impediments associated with such a task – including handling the radiological materials and their proper disposal; contamination and decontamination issues in the manufacturing plant and the laboratory when such tests would be done; and, of course, the time and cost involved. Others have noted same/similar issues with this approach. "[P]roblems with handling the radioactively labeled materials and their disposal are issues on the down side to this method. In addition, in some cases, it is difficult to synthesize the target polymer with the radioactive labels in the appropriate locations, with representative molecular weights, or with representative morphological characteristics."74

74 Van der Zee, M., Analytical Methods for Monitoring Biodegradation Processes of Environmentally Degradable Polymers, Section 11.5.5.2. Chapter 11 in Handbook of Biodegradable Polymers: Synthesis, Characterization and Applications, First Edition. edited by Andreas Lendlein, Adam Sisson.

It is my opinion that while such test data, if available, would certainly be great, it is unlikely that the practical difficulties associated with arranging such materials and tests can be overcome. More importantly, even if they were to be overcome, I can only imagine the myriad objections that Drs. McCarthy and Tolaymat would raise with regards to representativeness, test conditions, qualifications, etc. of those tests, much like they have criticized the D5511 tests. Lastly, it is my opinion, that one need not wait for such tests to deliver further proof of biodegradation as Dr. McCarthy suggests. Looking at all of the evidence, collectively, as discussed in this report with regards to the threshold question (which, incidentally, also includes confirmation of biodegradation of relatively recalcitrant polymers such as PE and PP by radiological evidence of the exact type Dr. McCarthy suggests – see the papers by Albertsson et. al.) as well as the many D5511 test results and using the exercise of commonly applied scientific judgment (which is unavoidable), it is evident that the existing proof provides more than a reasonable basis for concluding that plastics containing ECM's additives biodegrade faster than plastics without those additives. "

Rebuttal –

Dr. Sahu is aware that exactly this type of testing has been conducted with pure polyethylene (Albertsson, 1977) since he cited this paper.

In fact it would be a rather straightforward matter to mix ECM additive with ^{14}C labeled polyethylene and conduct a test similar to that performed in the past by Albertsson. If ECM additive improved ^{14}C polyethylene biodegradation, then it would have a significantly faster rate of ^{14}C evolution as compared to unamended ^{14}C polyethylene.

I have conducted ^{14}C fate studies of pesticides and PCBs in composts in the past (Michel et al., 1995, Michel et al., 1997 and Michel et al., 2001) as well as tests of the biodegradation of biopolymers in compost, soil and anaerobic digestion environments (Gomez and Michel, 2013; Gomez et al., 2014). My estimate of the cost to conduct such a study under simulated landfill conditions at OSU would be approximately $100,000 (See Appendix A). This is not much more than the cost of testing ECM claims to have already performed on its additive (ECM marketing materials claim that ECM has conducted internal and external studies that have cost hundreds of thousands of dollars). Morever, it is not significantly more than some of its customers have spent over several years for ASTM 5511 testing. For example for one such test, PPC was invoiced $37,000 by Northeast Industries (see PPC Industries Invoice #1048340-01.xls and PPC Industries Invoice # 20110909-Ext.xls).

Dr. Sahu Comment –

On Page 27 of his report, Dr. Sahu makes a point that ECM will encourage biofilm, formation and therefore plastic biodegradation:

"it is without question that the biodegradation of the plastic products' polymer structure (i.e. mass loss of a plastic material itself by the biota's conversion of its hydrocarbons into biogases and biomass) will be enhanced by the presence of ECM additives, which help to set in motion the following series of steps (together, the "mechanism"):

(a) the attraction/migration of already-present, local, microbial/biological agents to the plastic part at the locations where the additive is present, since the additive acts as a food source for such agents. This is enhanced by the presence of hydrophilic defects or groups in the plastic;

(b) the consequent initial formation of biological communities or biofilms, near the additive sites;

(c) the development and growth of the biofilms – ensured by the presence of greater than a critical minimum amount of ECM Additives that must be present and properly dispersed throughout the plastic (which is at or above 1%, by weight, in the case of the ECM Additives);

(d) the spreading of the established biofilms to other areas of the plastic (i.e., to areas of the plastic beyond where additives are present) since the biofilm is no longer dependent on just the initial mass of additive – in this respect, the additive acts as an attractor/catalyst for biofilm development – i.e., the

additive and the biofilm are, respectively, attractors and delivery agents for the real actor – namely the enzymes that the biota secrete;

(e) the unavoidable microbial and enzymatic degradation of the plastic where the biofilms are present, starting with the weak links in the plastic part and then moving in deeper into the part itself – resulting in mass loss of the plastic;

(f) all of the above leading to the conversion of the initial mass of plastic to the usual and final products of organic metabolism such as methane/carbon dioxide, water vapor, and some residual humus (from the remains of the biofilm when the plastic food source is exhausted).

Rebuttal –

The mechanism described by Dr. Sahu for biofilm formation on surfaces is contrary to the scientific literature that shows that biofilms can form equally well on hydrophilic and hydrophobic surfaces, and that they can form on surfaces that are not biodegradable. The presence of a biofilm on a surface does not necessarily lead to the biodegradation of the surface upon which it is attached. For example in their study of biofilm formation Cerca et al., (2005) conclude that,

> *"The clinical isolates exhibited different cell wall physico-chemical properties, resulting in differing abilities to adhere to surfaces. Adhesion to hydrophobic substrata for all strains occurred to a greater extent than that to hydrophilic surfaces. Bacterial cell hydrophobicity seemed to have little or no influence on adhesion. Microbial adhesion to surfaces has been shown to be a complex process, involving physico-chemical, protein and polysaccharide factors [2,6,7,9,13,16,21,25,26,30]. From an overall physico-chemical point of view, microbial adhesion can be mediated by non-specific interactions, with long-range characteristics, including Lifshitz–van der Waals forces, electrostatic forces, acid–base interactions, and Brownian motion forces [3,37]. As soon as microorganisms reach a surface, they will be attracted or repelled by it, depending on the sum of the different non-specific interactions [17]. In biological systems, hydrophobic interactions are usually the strongest of all long-range non-covalent forces [5], and adhesion to surfaces is often mediated by these types of interactions [35]."*

In another study of anaerobic biofilm formation by Araujo et al., (2004) it was found that neither the ability of biofilms to attach to surfaces nor their community composition were influenced by the hydrophobicity of the surface. Biofilms formed equally on polypropylene (hydrophobic) and glass (hydrophilic) surfaces. The biofilms formed rapidly and showed nearly the same microbial composition. Furthermore, the hydrophobicity of the support material did not influence the initial development or the microbial composition of anaerobic biofilms that developed. Since both glass and polypropylene are essentially biologically inert, it can also be concluded that biofilms can form readily on surfaces which they are not capable of using as a food source or biodegrading.

In yet another study contradicting Dr. Sahu's assertions, Busscher and Weerkamp (1987) described the mechanisms for biofilm formation on surfaces. They do not indicate that additives are necessary for biofilm formation on the surfaces of plastics, but that the process involves the removal of water from the surface so that microorganisms can come into direct contact with the surface. The removal of water from a hydrophilic surface is less energetically favorable than from a hydrophobic surface. The conclusion of Dr. Sahu that ECM additive improves the hydrophilicity of the surface enhancing biofilm formation is contrary to this mechanism.

Finally, in actual testing of ECM amended plastic (Gomez and Michel, 2013) electron scanning microscopy revealed no biofilm formation or degradation of polypropylene or polyethylene terephthalate containing 2% ECM additive during tests of these materials in soil for nearly 2 years (Figure 4). Thus biofilm formation is not enhanced by ECM additive and complete biodegradation of ECM amended plastics in a landfill after 1, 5 or even 50 years, is highly unlikely.

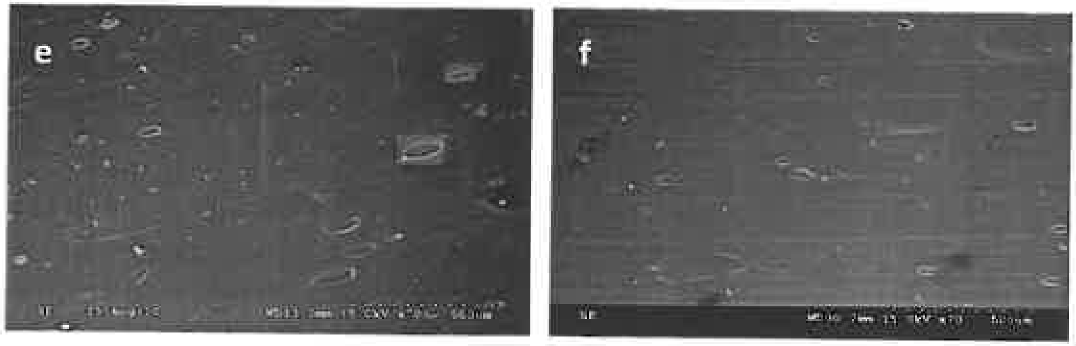

Figure 4. Scanning electron micrograph analysis of polypropylene amended with 2% ECM additive after 0 days (left) and 2 years (right) of soil incubation.

Additional Comments -

In the report submitted by Dr. Barber (2008 Environ BioPVC Report.pdf), there is a major flaw in the scientific analysis and modeling of BioPVC biodegradation data.

The first order decay model used by Barber is inappropriate for the data presented in the BioPVC test in Figure 2 and does not fit the data as well as the model he uses in the presentation of this data. This figure and its' legend are reproduced from the report by Dr. Barber below (Figure 5).

In the legend Barber states that a "logistic curve" was used to model the mass loss of BioPVC during the test. The model fits the data well with an R^2 of 0.97 at a 99% confidence level. This type of model predicts the concentration of the substrate (BioPVC in this case) and has three parameters, one of which (So) is the initial concentration of the substrate that is biodegradable (Martin and Scow, 1989). The other two, Xo and k, are the initial concentration of organisms able to degrade the substrate and the time constant for the rate at which this degradation occurs, respectively (Figure 6).

The BioPVC data is best fit by a logistic model using So, Xo and k values of 2.6%, 0.075% and 0.4 month^{-1} respectively, as shown in the graph below. These parameter values provide a fit nearly identical to that presented by Dr. Barber in Figure 2 of his report.

However an So value of 2.6% would indicate that only 2.6% of the substrate is biodegradable and the remaining 97.3% is not. Put another way, taking the mathematical limit of the logistic model used by Dr. Barber to model BioPVC degradation (by substituting t=infinity into the equation in Figure 6) would indicate that only 2.6%, and not 100% (or even 20%) mass loss of the BioPVC would ultimately occur.

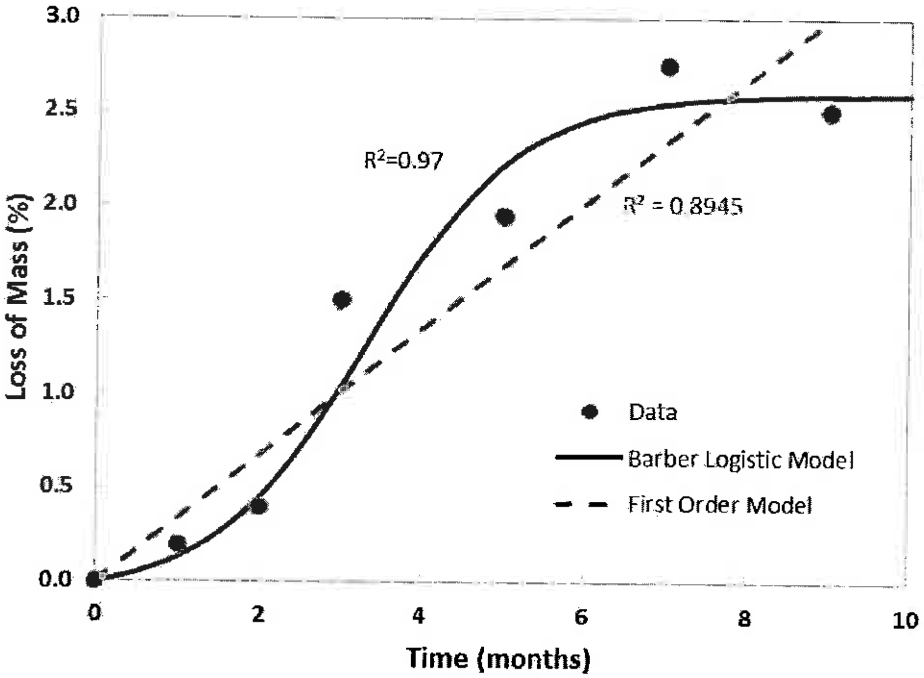

Figure 5 . Loss of mass expressed as the percent change is presented for BioPVC material over the time course of the study. A three parameter logistic curve was used by Barber to fit the data (R^2=0.97%, p=0.01). In red a first order model is also shown. The best first order curve fit has an R^2 value of 0.8945. Data reproduced from the BioPVC report.

$$-dS/dt = k_4 S(S_0 + X_0 - S)$$

$$S = \frac{S_0 + X_0}{1 + (X_0/S_0)\exp[k_4(S_0 + X_0)t]}$$

Figure 6. Differential and integrated forms of the logistic model of microbial substrate utilization.

Inexplicably, later in the same report, Dr. Barber chooses a totally different model, a "first order model", to predict "half-lives" for BioPVC. Half lives are only valid if the biodegradation kinetics are first order, but in this case a first order model does not fit the data as well as the logistic model and has a lower R^2 value of 0.89 (Figure 5). Therefore, the "half lives" he reports in the report are invalid since the kinetic data are inconsistent with a first order model.

A more appropriate kinetic model would be a two component model where one component (the ECM additive) is biodegradable over a 6-10 month period, while the other (PVC) is not biodegradable over the time period of the test. For such a two component model, the length of time for the ECM amended PVC to biodegrade could not be estimated using a first order rate constant as was done in the Environ report. But such a two component model would reduce mathematically to the very logistic model used by Dr. Barber (since So and Xo for PVC would be zero).

In summary, from the data presented, and using the logistic model used by Dr. Barber uses, the time to completely biodegrade BioPVC would be infinite.

Summary

Drs. Burnette and Sahu present very well documented, compelling and interesting reports regarding microbial ecology, plastic degradation, and ECM amended plastic degradation. However they do not provide convincing scientific evidence that ECM amended plastics will biodegrade in a landfill over 1 to 5 years as ECM has claimed. In fact most all of the studies and reports, including our own, indicate that the biodegradation of ECM amended plastics in a landfill or most any natural environment, would take many decades if not centuries. In a report cited by Dr. Burnette, the half lives used to predict the time required for the biodegradation of ECM BioPVC, are based on erroneous application of a 1st order kinetic model.

Respirometry tests that have been used to assess the biodegradability of a wide range of different chemical compounds in the scientific literature, and are accepted by the scientific community, show that ECM amended plastics degrade very slowly and to a limited extent. These tests are conducted under conditions where microbial activity and diversity are high and likely much greater than in a landfill environment.

The "threshold question" addressed by Dr. Sahu, of whether conventional plastics are biodegradable over long periods of time, such as centuries or millennia, is really a moot point. As shown by nearly all of the experts, polyethylene will degrade very slowly. Degradation can be marginally improved by irradiation to introduce carbonyl groups, by modifying PE by introducing reactive groups, by thermal treatment to reduce its molecular weight and when incubated with specific microorganisms selected for their ability to biodegrade recalcitrant materials. However, mixing ECM additive with conventional plastics causes none of these changes. This implies that ECM amended plastics will persist for very long time scales in landfills and contribute to plastic pollution if released into natural environments.

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Appendix A. Estimated cost to conduct a University research study on the biodegradability of ^{14}C polyethylene and polyethylene amended with ECM additive.

	Year 1	Year 2	Year 3	Year 4	Year 5
Personnel (3% raise per year)					
Principal Investigator (3% time)	$ 3,600	$ 3,708	$ 3,708	$ 3,708	$ 3,708
Postdoctoral Researcher (25% Y1, 10% Y2-5)	$ 11,250	$ 4,635	$ 4,635	$ 4,635	$ 4,635
Fringe benefits	$ 4,158	$ 2,336	$ 2,336	$ 2,336	$ 2,336
Supplies	$ 2,000	$ 750	$ 750	$ 750	$ 750
Synthesis of 14C polyethylene	$ 5,000				
Indirect Costs (52.5%)	$ 13,264	$ 5,829	$ 5,829	$ 5,829	$ 5,829
Yearly Total	$ 39,272	$ 17,258	$ 17,258	$ 17,258	$ 17,258
Grand Total					**$ 108,303**

Notes

OSU has a NRC license for C14, scintillation counters for measuring radioactivity and AD reactor systems

14C polyethylene synthesis would be done by American Radiolabeled Chemicals, Inc. , St Louis, MO, or Perkin Elmer.

Experiment would consist of 16 Anerobic Incubation Reactors. Each would be inoculated with activated sludge and landfill material and incubated at room temperature for 5 years. The replicates and treatments would be 4 - 14C polyethylene, 4 - 14C polyethylene plus 2% ECM additive, 4 - blanks (no plastic), 4 - cellulose (positive control

Biogas trapped in Tedlar bags, would be combusted to 100% CO2, and the radioactivity measured.

Appendix B: Occasions where Dr. Michel has provided oral testimony in depositions, at trial or in similar proceedings in the past ten years.

a. *City of Kirtland planning and zoning commission, Kirtland, OH, re: Application of Mr. Excavator, Inc. for Industrial Permit, February 2011.*
b. *Analysis of the document entitled, "Operational Procedures and Odor Management Practices for the proposed OEW Watkins Rd. Composting Facility", provided to Laura Comek, Esq. July 2013. Crabbe, Brown & James, Columbus, OH.*
c. *Testimony to Columbus, OH. Zoning Board hearing, August 2013, regarding application for a Food Waste Composting facility by OEW Inc.,* Retained by *Crabbe, Brown & James, Columbus, OH.*

Respondent’s Exhibit W

In the Matter of ECM Biofilms, FTC Docket No. 9358

Sur-Rebuttal Report of Dr. Steven J. Grossman

Sur-Rebuttal to Rebuttal Reports of Drs. Frederick C. Michel and Stephen P. McCarthy

Qualifications

My qualifications to respond to the rebuttal reports include a B.S. in Chemistry and a Ph.D in Polymer Science and Engineering and both industrial and academic experience in the field of polymer materials, polymer chemistry, and polymer structure-property relationships. More specifically, after earning my Ph.D. in Polymer Science and Engineering from the University of Massachusetts-Amherst I served as a research and development scientist in the polymer research group at the Upjohn Company, North Haven, Connecticut (1980-1984). In 1984 I joined the University of Massachusetts-Lowell as an Assistant Professor of Plastics Engineering, and was later promoted to Associate Professor and then Full Professor. I teach undergraduate and graduate courses on polymer materials which include a consideration of issues such as commercial polymer synthesis, polymer structure and property relationships, influence of additives and fillers along with a consideration of recycling and biodegradation. For example, my courses deal with issues of polymer degradation in the environment as well as a detailed consideration of polymer hydrolysis which is a key part of many biodegradation pathways.

In addition, in 1990 I obtained my J.D. degree and became a member of the New Hampshire Bar and am also licensed to practice before the United States Patent Office. I have practiced intellectual property law (patent law) since 1990 and am also the founding member of the law firm of Grossman, Tucker, Perreault & Pfleger, 55 South Commercial Street, Manchester, New Hampshire 03101.

I note that I am a colleague of Dr. McCarthy at the University of Massachusetts-Lowell, in the Department of Plastics Engineering. My observations herein are my own evaluations of the analysis and opinions Dr. McCarthy has offered in this particular case.

My complete curriculum vitae is attached which identifies prior cases where I have provided expert testimony. I note that I am being compensated at the rate of $330.00 per hour.

Introduction

I was requested by counsel for ECM Biofilms to review and provide my comments on the rebuttal reports of Drs. Michel and McCarthy.

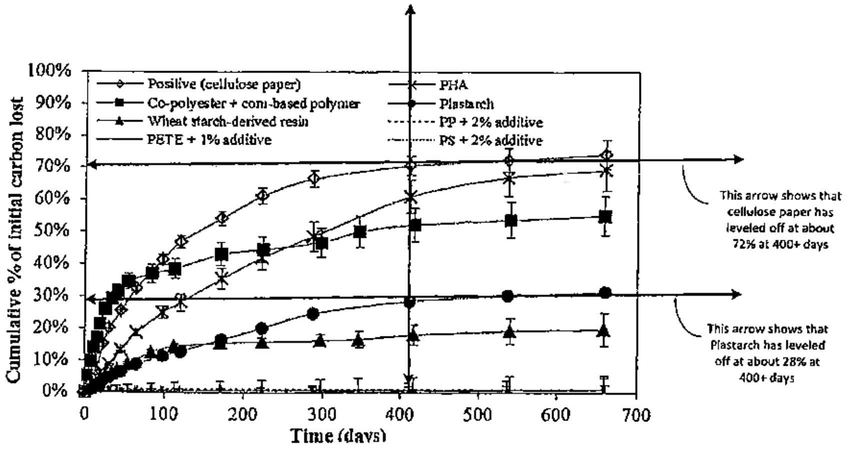
Rebuttal of Points of Dr. Frederick Michel

Comment by Dr. Michel

At page 6 of his Rebuttal report, Dr. Michel responds to the point that in his 2013 article (*Biodegradability of Conventional and Bio-based Plastics and Natural Fiber Composites During Composting, Anaerobic Digestion and Long Term Soil Incubation*) it was observed that the positive control (cellulose paper) plateaued at around 70-75%. This certainly was a relevant observation supporting the view that conditions promoting degradation had been significantly compromised. Dr. Michel's response was that this was somehow irrelevant since "some of the other materials (e.g. Plastarch, a corn starch polyester blend and PHA) continued to biodegrade well after cellulose biodegradation had plateaued." Dr. Michel asserted that such plateau is not evidence of a flawed test and presented **FIG. 1** and **FIG. 2** of his article.

Rebuttal

Looking at **FIG. 1** of the subject 2013 Michel article, when the positive control plateaued at about 72% after 400+ days, Plastarch also appeared to plateau at a level of about 28%. See below:

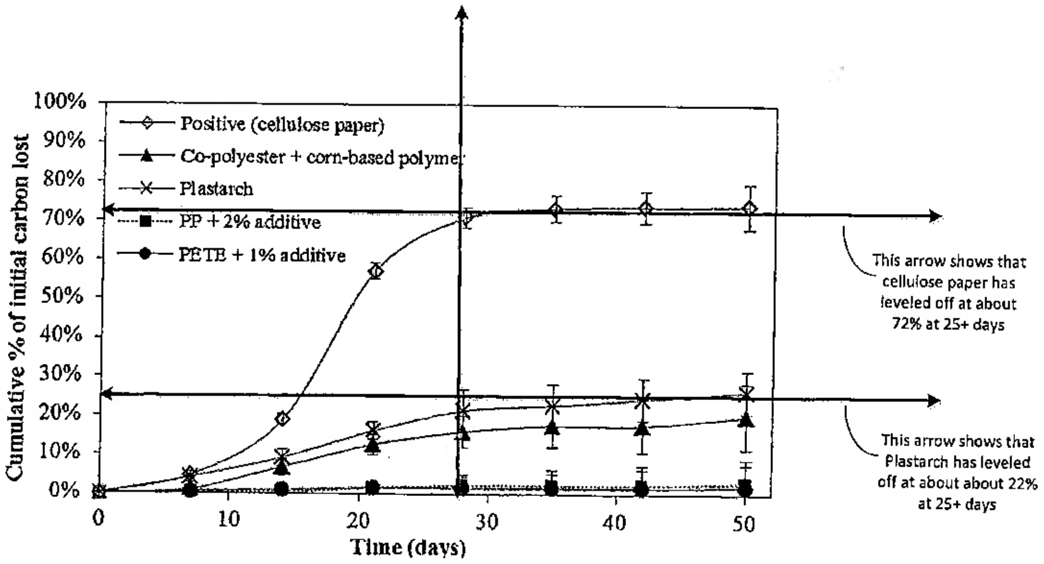


This arrow shows that cellulose paper has leveled off at about 72% at 400+ days

This arrow shows that Plastarch has leveled off at about 28% at 400+ days

In **FIG. 1** above, PHA also appeared to indicate a value that showed some small increase from day 400+ to day 500+, but then it too appeared to level-off. I did not illustrate that particular observation on **FIG. 1**.

In **FIG. 2** of the subject 2013 Michel article, once again, the positive control plateaued at about 72%. Plastarch also again appears to level off at about 22%. See below. PHA was not presented in **FIG. 2**.



This arrow shows that cellulose paper has leveled off at about 72% at 25+ days

This arrow shows that Plastarch has leveled off at about about 22% at 25+ days

Considering the above, I do not agree with Dr. Michel that Plastarch and PHA "continued to biodegrade well after cellulose biodegradation had plateaued." I am therefore of the view that the positive control of cellulose paper plateaued at around 70-75% thereby indicating that the environment present in his test became inhospitable to biodegradation supporting a conclusion that the aforementioned substances failed to biodegrade.

Comment by Dr. Michel

Dr. Michel states at page 6 of his Rebutal Report that "[d]uring soil incubation for 660 days, no significant mineralization was observed for polyethylene or polypropylene plastics or the same plastics amended with ECM additive."

Rebuttal

This is again in reference to Dr. Michel's 2013 paper noted above. I therefore reviewed this paper to see how Dr. Michel went about preparing samples for testing. I noticed as follows:

1. Dr. Michel indicates in his 2013 paper (page 2585, Table 2) that he apparently prepared a blend of polypropylene with "2% ECM MasterBatch Pellets™ additive." However, I did not see any information as to how such blend of polypropylene was prepared, such as the conditions for blending. The conditions for blending will have an impact on the polymer blend produced and can include, e.g., destruction or compromise of the additive, non-uniform distribution of the additive, inclusion in the blend of substances toxic to biodegradation, etc. Dr. Michel does indicate that he prepared the blend by "injection molding" and, as noted, no conditions are reported as to how he or others prepared the blend, which I understand to be a function ordinarily dictated by ECM with care in the creation of plastics containing its additive. Injection molding involves heating a polymer material at elevated temperature and with shear, which if not done properly, can seriously compromise the polymer as well as the distribution and integrity of the ECM additive package. It is also unclear as to whether or not the negative control of conventional polypropylene was the same polypropylene mixed with the ECM additive package and exposed to similar injection molding conditions. Such lack of information on how the polypropylene blends were prepared, and whether or not Dr. Michel followed ECM's recommended procedures for blending, and blending levels, raises serious questions regarding the accuracy of the ensuing tests regarding the ECM additive in polypropylene.
2. Similar to the above, Dr. Michel indicates in his 2013 paper (page 2585, Table 1) that he prepared a blend of polystyrene with "2% ECM MasterBatch Pellets™ additive." Again, Dr. Michel indicates that such blend was prepared by injection molding but no conditions are reported. As noted above, injection molding involves heating a polymer material at elevated temperature and with shear, which if not done properly, can seriously compromise the polymer as well as the ECM additive. Such lack of information on how the polystyrene blends were prepared, and whether or not Dr. Michel followed ECM's recommended procedures for blending, and blending levels,

raises serious questions regarding the accuracy of the ensuing tests regarding the ECM additive in polystyrene.

Comment by Dr. Michel

At page 10 of his report, Dr. Michel writes "[a]s described by Dr. McCarthy in his report, only a few tests use proper controls and few report the actual amount of ECM additive present in the plastic."

I understand this to be Dr. Michel's reference and adoption of the analysis of Dr. McCarthy that only certain tests are proper for the testing of a material to establish it is biodegradable. I report below that Dr. McCarthy's "standard" as to what makes for a material to be identified as biodegradable, and the testing protocols that he identifies as being necessary to establish a material as biodegradable, are: (1) inconsistent with U.S. Patent No. 5,883,199 where Dr. McCarthy is a named inventor; (2) not advanced in any of Dr. McCarthy's various peer-reviewed publications; (3) contrary to what Dr. McCarthy previously authored in 2003; and (4) inconsistent with a 2011 paper that appeared in the journal for which he is the reviewing editor.

Comment by Dr. Michel

At page 12 of his Rebuttal Report, Dr. Michel writes that "[t]o obtain accurate evidence of biodegradation, experiments are best performed using ^{14}C-labeled substrates and measuring $^{14}CO_2$ over time."

I disagree with Dr. Michel. I note that Dr. Michel's own study did not rely on ^{14}C labeled substrates. As noted below, Dr. McCarthy's U.S. Patent No. 5,883,199 identifies certain polymer blends as biodegradable, without the use of ^{14}C-labeled substrates and measuring $^{14}CO_2$ over time and without reliance on any ASTM recommended testing methodology (relying instead on his own testing methodology). In addition, Dr. McCarthy did not report or rely upon ^{14}C-labelled substrates in any of his published articles. As a plastics material scientist and polymer chemist, I can affirm, based on the peer reviewed literature in the field and my own years of experience, that it is not the standard among polymer scientists to rely on ^{14}C labelling to advance a claim that a particular polymer is biodegradable, and there is no evidence that accepted methods of testing reported upon in the literature, and even including the hybrid method used in Dr. McCarthy's own patent, are inappropriate for determining polymer biodegradability.

Rebuttal of Points Made by Dr. McCarthy

Comment by Dr. McCarthy:

1. The question that complaint counsel asked me to evaluate is whether ECM's claims were scientifically true or at least has an appropriate level of scientific support. The claims complaint counsel asked me to evaluate specifically are:

a. Plastic products manufactured with ECM additive ("ECM Plastics") will completely biodegrade, i.e. will completely break down and decompose into elements found in nature, within a reasonably short period of time (i.e. one year) in a landfill. McCarthy Rebuttal Report at ¶1.

Rebuttal:

I disagree with Dr. McCarthy's view that the term "biodegradable" would be understood by scientists, engineers and other professionals in the plastics industry as limited to the requirement that the plastic in question must "completely break down and decompose into elements found in nature, within a reasonably short period of time (i.e. one year) in a landfill." I have also seen no authority to support Dr. McCarthy's position.

As an initial matter, I note that Dr. McCarthy's 2003 publication entitled "*Biodegradable Polymers*" appearing in the text "*Plastics and The Environment*" states as follows:

> "The definition of biodegradable polymer varies greatly among scientists, manufacturers and consumers."

I agree with that statement of Dr. McCarthy that the definition of biodegradable varies greatly among scientists, manufacturers and consumers.

In addition, I note a 2011 article entitled "*Biodegradable Polymers-A Review On Recent Trends and Emerging Perspectives,*" by G. Luckachan and C. Pillai, which appeared in the J. Polym.

Environment (2011) 19:637-676 that Dr. McCarthy reviews and edits. The authors of that 2011 article stated:

> The various definitions of biodegradation depend on the field of application of the polymers (biomedical area or natural environment). Many different definitions have officially been adopted, depending on the background of the defining standard organizations and their particular interests. Van der Zee and Seal [11,12] review all of the definitions found in different standards. Albertsson and Karlsson [13] defined biodegradation as an event that takes place through the action of enzymes and/or chemical decomposition associated with living organisms and their secretion products. It is also necessary to consider abiotic reactions like photodegradation, oxidation and hydrolysis, which may alter the polymer before, during or instead of biodegradation because of environmental factors. So, strictly speaking, "biodegradation of a polymer" is defined as the deterioration of its physical and chemical properties and a decrease in its molecular mass down to the formation of CO_2, H_2O and CH_4 and other low molecular weight products under the influence of microorganisms in both aerobic and anaerobic conditions aided by abiotic chemical reactions like photodegradation, oxidation and hydrolysis. [14].

In view of the above, I am of the opinion that the fundamental definition of biodegradation given by Complaint Counsel and relied upon by Drs. McCarthy and Michel, stated as follows from note 1 of the McCarthy Expert report, does not appear anywhere in the peer reviewed literature, and is incorrect: "the entire treated plastic will completely break down and return to nature (i.e. decompose into elements found in nature) within one year after customary disposal (i.e. incinerator, landfill, or recycling)."[1] McCarthy Expert Report of June 4, 2014 at note 1. In my view, the definition of biodegradation "varies greatly among scientists, manufacturers, and consumers" (McCarthy 2003) and "many different definitions have officially been adopted" (Luckachan and Pillai 2011).[2]

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[1] It is unclear to me how a plastic material would be labelled as biodegradable on the basis that it was broken down by an "incinerator" which is reference to a burning of the material, not biological degradation.

[2] I reviewed Dr. McCarthy's publications produced in this matter to ECM and was unable to find any use by him outside of his expert report that the definition for biodegradation of a plastic is that the plastic "will completely

I next reviewed Dr. McCarthy's U.S. Patent No. 5,883,199 ('199) entitled "*Polylactic Acid-Based Blends*."[3] As an initial matter, I note that any inventor who files for patent protection must sign a Declaration before the United States Patent Office that he or she has reviewed and understands the contents of the application, including the claims, as well as claims that are amended. The Declaration also acknowledges a duty to disclose to the USPTO all information that is considered material to patentability in accordance with the requirements of 37 C.F.R. 1.56. Dr. McCarthy signed such Declaration on September 22, 1997, a copy of which is attached.

In the '199 patent I observed that Dr. McCarthy did *not* employ the definition he recites in this case (and incorrectly suggests is accepted in the scientific community). Instead, as discussed below, under oath before the USPTO Dr. McCarthy adopted a far more general definition as to what would qualify for a claim that a particular polymer material is "biodegradable."

The '199 patent is directed at Dr. McCarthy's claim that blends of: (1) polylactic acid (PLA) based polymers or copolymer; and (2) polymers or copolymers of any polyester, have "*excellent biodegradability and aging properties*." Col. 2, lines 10-20 of the '199 patent. See also, claim 1 at col. 12, lines 23-33.

To support the claim that the subject blends had "*excellent biodegradability and aging properties*" Dr. McCarthy identified his own testing protocol in an artificial soil environment as applied to 0.3 mm thick films. Col. 10, lines 4-10. That test he refers to as UML-7645. That test is not adopted by ASTM and is inconsistent with his definition of biodegradation in footnote 1 of his expert report. The soil degradation testing of such films was presented in **FIG. 11**, which is reproduced below:

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biodegrade, i.e. will completely break down into elements found in nature, within a reasonably short period of time (i.e. one year) in a landfill."

[3] The data and conclusions presented in U.S. Patent No. 5,883,199 also appear in Dr. McCarthy's journal publication entitled "*Advances in Properties and Biodegradability of Co-Continuous, Immiscible, Biodegradable, Polymer Blends, Macromol. Symp.* 144, 63-72 (1999).

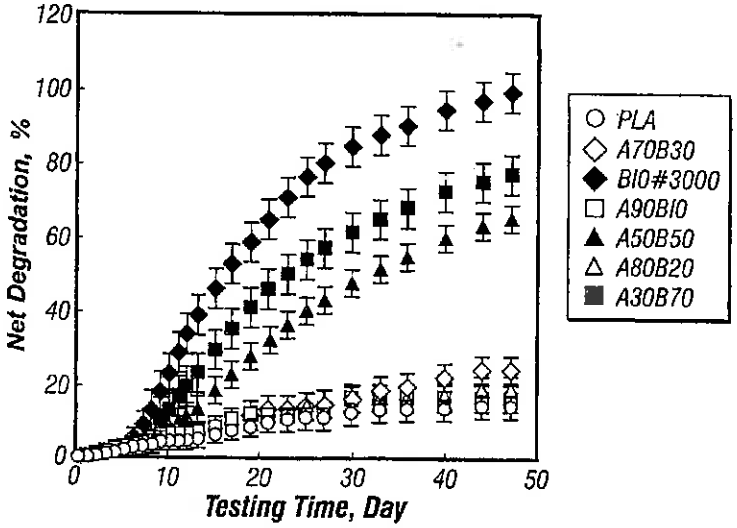

FIG. 11

In connection with the above patent **FIG. 11**, Dr. McCarthy wrote as follows:

> The soil degradation testing results of the two polymers and their blends are reported in **FIG. 11**. After degradation for 45 days, BIONOLLE#3000 degraded almost 100%, while polylactic acid degraded only about 14% by loss in weight. For blends with 70 and 50% BIONOLLE#3000, the degradation rate was relatively fast. After 45 days, the A30B70, A50B50, and A70B30 blends degraded about 77%, 65% and 25%, respectively, by loss in weight. **FIG. 11** shows that polylactic acid biodegrades in soil, but just not quickly, and the addition of the second aliphatic polymer, such as BIONOLLE#3000, increases the biodegradation rate.
>
> The importance of the soil biodegradation curves shown in **FIG. 11** is that a specific blend can now be designed such that this blend would have a certain net degradation in a given number of days within the soil.

As can be seen from the above, Dr. McCarthy asserts that biodegradation can be established by utilizing his own developed testing protocol, not an ASTM protocol, ^{14}C testing, or any other method described in his report, and that biodegradation is present in a polymer blend where it only partially degrades to levels of 20% (A80B20) or 30% (A70B30) after 45 days, with no data after this point. I do not understand the definitions and testing protocols described in his patent to be at all close to the "standard" that Dr. McCarthy asserts in his report as critically missing from ECM's testing of the ECM additive.[4]

In addition, as noted above, Dr. McCarthy's '199 patent recites that based upon his biodegradation curves, "*a specific blend can now be designed such that this blend would have a certain net degradation in a given number of days within the soil.*" Col. 11, lines 20-24. Accordingly, Dr. McCarthy has indicated that biodegradation can be assigned to a polymer blend when it demonstrates a "*certain net degradation*" in some "*given number of days*" within the soil, without articulating the existence of a minimum degradation level or how many days are relevant. Again I find the position recited by Dr. McCarthy in the '199 patent inconsistent with the definition adopted by Dr. McCarthy at footnote 1 of his report and applied against ECM.

I next note that at column 11, lines 25-62, Dr. McCarthy reports on biodegradation in compost. Here, Dr. McCarthy employed an artificial compost environment. The compositing process was carried out for 30 days at 55 °C. The results were reported in **FIG. 12** of his patent which is reproduced below:

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[4] See again, footnote 1 of Dr. McCarthy's Expert Report of June 4, 2014, where Dr. McCarthy adopts the assumption of Complaint Counsel that "biodegradable" means that "the entire plastic will completely break-down and return to nature (i.e. decompose into elements found in nature) within one year after customary disposal (i.e. incinerator, landfill, recycling)." Dr. McCarthy also stated that he used "this definition and the scientific definition of biodegradable interchangeably" in his report, "because there is no substantive difference between the two that affects my analysis or opinions."

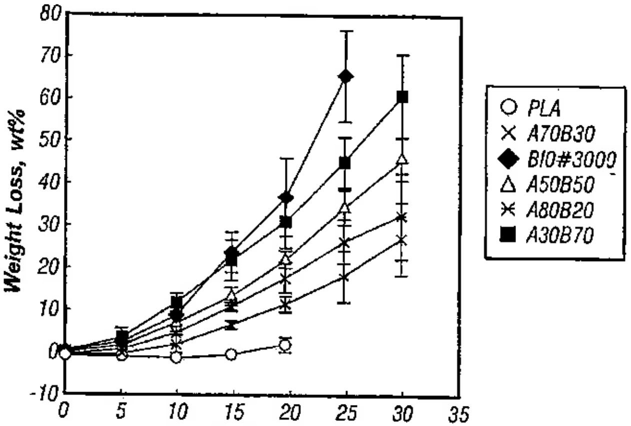

FIG. 12

In connection with **FIG. 12**, Dr. McCarthy wrote as follows:

> The compost degradation testing results of the two polymers and their blends are reported in **FIG. 12**. After degradation for 20 days, BIONOLLE#3000 degraded almost 40%, while polylactic acid degraded only about 3%, by loss in weight. For blends with 70 to 20% BIONOLLE#3000, the degradation percentage was much greater (and the rate much faster) than that of polylactic acid, e.g., after 20 days, the A30B70, A50B50, and A70B30 blends degraded about 35%, 25% and 15%, respectively, by loss in weight. **FIG. 12** shows that polylactic acid biodegrades in compost, but slowly, and the addition of even 20% by weight BIONOLLE#3000 increases this biodegradation rate dramatically.
>
> The importance of the compost biodegradation curves shown in **FIG. 12** is that a specific blend can now be designed such that this blend would have a certain net degradation in a given number of days in a composting environment.

As can be seen from the above, Dr. McCarthy asserts that a weight loss of 3% after 20 days was sufficient to identify polylactic acid as a material that "biodegrades in compost." Dr. McCarthy also wrote that 20 days of testing for blends A30B70, A50B50 and A70B30, with degradation levels well below 100% (specifically, 35%, 25% and 15%) was nonetheless sufficient to identify them as biodegradable. Once again, the position that he took under oath before the United States Patent Office is not at all close to the "standard" that Dr. McCarthy asserts in his report and applies to the ECM product.

In addition, Dr. McCarthy again wrote that based upon his biodegradation curves in **FIG. 12**, "*a specific blend can now be designed such that this blend would have a certain net degradation in a given number of days in a compositing environment.*" Col. 11, lines 58-61. Accordingly, Dr. McCarthy explains in the '199 patent that biodegradation can be assigned to a polymer blend provided it has a "*certain net degradation*" in some "*given number of days*" in an artificial compost environment.

Once again, such open standard simply requires that there be identification of some amount of "*net degradation*" over some "*given number of days*" in order to characterize a polymer material as biodegradable. This would not include any need to achieve a complete break-down into elements found in nature within one year. And here, Dr. McCarthy informs the reader that PLA "*biodegrades in compost*" based upon the observation of a weight loss of just 3% after 20 days.

Accordingly, I find Dr. McCarthy's "*Biodegradation Testing in Compost*" as recited in the '199 patent at col. 11, lines 25-62 inconsistent with the requirements Dr. McCarthy asserts in his expert report and applies to the ECM product.[5]

Comment by Dr. McCarthy

2. In my report, I defined conventional plastics as high molecular weight, commercial-grade plastics representing more than 90% of the market. See, Expert Report of Stephen P. McCarthy ("McCarthy Expert Report) ¶¶ 29-31. I conclude, based upon my professional judgment after evaluating the Testing Materials that many of ECM's claims are untrue and none have the *appropriate scientific support*." Emphasis added. McCarthy Rebuttal Report at ¶2.

Rebuttal:

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[5] At pages 163-164 of his Deposition of June 27, 2014, Dr. McCarthy indicated that he was unable to conduct accelerated control tests in the lab that could be correlated to actual landfill condition in the outside world. Specifically, Dr. McCarthy testified that "*No. We didn't. We weren't able to do it. I mean, we tried to. That was the purpose of it, but it never happened.*" As noted above, Dr. McCarthy presented results of biodegradation in an artificial compost environment at col. 11, lines 25-62 of the '199 patent to support claims of biodegradation.

I understood this to be a reference to Dr. McCarthy's Expert Report at section VI, which posed the question "*What Constitutes "Competent and Reliable Evidence" for Claims of Biodegradable Plastic*." Dr. McCarthy therein states:

> **Test Duration**. The study must last long enough for the sample to reach at least 60% biodegradation. The most typical type of biodegradation test is a gas evolution test, which monitors the end-products of biodegradation. Sixty percent biodegradation is the minimum landmark for showing that the treated sample will biodegrade to completion. (Alternatively, radiolabelling the most recalcitrant part of the conventional plastic can conclusively answer the question whether the conventional plastic component of the treated plastic is biodegrading. Radiolabelling will also show that the "biodegradable plastic" can biodegrade to completion.)

I disagree with Dr. McCarthy's assertion that "*biodegradation studies must last long enough for the sample to reach at least 60% biodegradation*." I am unaware of any such standard established in the peer-reviewed literature. I am also unaware of any such standard adopted by any industry or scientific standard setting body. As noted above, in the '199 patent Dr. McCarthy presented data for biodegradation in **FIG. 11**, which identified the "net degradation" of seven (7) polymer samples identified in **Table 1**. Dr. McCarthy identified four (4) samples as having a net degradation of *under* 60% as *nonetheless biodegrading*. See also col. 11, lines 9-19 of the '199 patent. This is clearly in contradiction to what he states in his report that "[s]ixty percent biodegradation is the minimum landmark for showing that the treated sample will biodegrade to completion." See again, the '199 patent.

I also find other inconsistencies between Dr. McCarthy's rebuttal report and the position he takes in the '199 patent. They are as follows:

- Dr. McCarthy's rebuttal reference to "*appropriate scientific report*" led me to his statement that tests must use proper controls and that "proper testing should have a negative control (i.e. untreated conventional plastic of the same type as the test sample) and a positive control (typically cellulose). The negative control is a substance known not to be biodegradable." McCarthy Expert Report at page 16. Dr. McCarthy's '199 patent makes no reference to the use of a negative control known not to be biodegradable. Moreover, I am of the view that it is an accepted practice among polymer scientists not to use a negative control as a condition precedent to a claim of biodegradation. See again, the '199 patent. See also, Dr. McCarthy's paper "*Advances In Properties and Biodegradability of Co-Continuous, Immiscible, Biodegradable, Polymer Blends*, Macomol. Symp. 144, 63-72 (1999). This published paper identifying biodegradable polymers includes no use of a negative control.

- Dr. McCarthy's rebuttal reference to "*appropriate scientific support*" led me to his statement that "[c]onfirmatory testing such as radiolabelling is essential to establish that the plastic itself is biodegrading." McCarthy Expert Report at page 19. McCarthy underscores this with the assertion that "[a]bsent an approved ASTM specification, it is my opinion that to scientifically prove a claim that the plastic-not merely the additive and inoculum-is biodegrading, the claimant must support its claim with at least one test with positive results from ^{14}C labeling of the conventional plastic." McCarthy Expert Report at page 24. Dr. McCarthy's '199 patent does not use ASTM testing and does not support its claim that the polymer blends therein are biodegradable, with at least one test with positive results from ^{14}C labeling. Moreover, I am of the view that it is an accepted practice among polymer scientists not to use ^{14}C labeling to advance a claim that a particular polymer is biodegradable. See, the '199 patent. See also, Dr. McCarthy's paper "*Advances In Properties and Biodegradability of Co-Continuous, Immiscible, Biodegradable, Polymer Blends*, Macomol. Symp. 144, 63-72 (1999). This published paper includes no use of radiolabelling to identify the subject blends as biodegradable.
- Dr. McCarthy's rebuttal reference to "*appropriate scientific support*" led me to his statement that the "screening level tests ECM relies on to support its claims extrapolate from minimal biodegradation to a conclusion of complete biodegradation. Such extrapolation is scientifically invalid because biodegradation is not linear and typically slows down due to recalcitrance." McCarthy Expert Report at page 27. Dr. McCarthy's '199 patent presents data for only five (5) particular blends of PLA with a polyester identified under the BIONELLE trademark. See **Table 1** of the '199 patent. It is not clear what the molecular weight is of the BIONELLE polymers reported in

Table 1.[6] Nevertheless, Dr. McCarthy recites in claim 1 of the '199 patent that one can prepare a "biodegradable blend" of "(a) a first polylactic acid-based polymer or copolymer, and (b) a second polymer consisting essentially of one or more polyesters." Accordingly, Dr. McCarthy extrapolated from the five (5) blends recited in Table 1 and the data presented in **FIGS. 11** and **12** to the claim that PLA copolymers, in combination with any polyester, would provide a biodegradable blend formulation. Moreover, I am of the view that contrary to Dr. McCarthy's assertion, it is accepted among polymer scientists to extrapolate biodegradation testing to support a conclusion that a polymer will biodegrade. See, the '199 patent.

Comment by Dr. McCarthy

At paragraph 12 of his rebuttal, Dr. McCarthy points out that if a polymer chain such as polyethylene is short enough it can be attacked by microorganisms. Dr. McCarthy then goes on to state that "the only way to reduce PE to this molecular weight is through a pro-oxidant (not ECM technology) or exposure to other oxidative degradation environments."

Rebuttal

I disagree with Dr. McCarthy that the only way to achieve biodegradation in PE is to utilize a pro-oxidant. In the above referenced 2011 article entitled "*Biodegradable Polymers-A Review On Recent Trends and Emerging Perspectives,*" by G. Luckachan and C. Pillai, at page 647, the authors review existing methods to facilitate polyethylene degradation. They mention: (1) insertion of weak links into the polymer; (2) compounding of polymer with preoxidants and photosensitizers; (3) blends of biodegradable and non-biodegradable polymer; and (4) grafting of natural monomers onto nonbiodegradable polymers. I note that the ECM Additive falls into the reported category of facilitating polyethylene degradation by blending of a biodegradable and non-biodegradable polymer.

Comment by Dr. McCarthy

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[6] Dr. McCarthy asserts that an evaluation for biodegradation should include "a starting molecular weight for the test sample and the negative control." McCarthy Expert Report at page 33. As noted, there is no "negative control" in the '199 patent and as noted, there is no indication of the starting MW for the particular PLA-BIONELLE blends reported in Table 1 and analyzed in **FIGS. 11** or **12**.

15. ECM and its experts assert that there is a mechanism of action initiated by the attractiveness of the ECM Additive as a food source that results in the depolymerization of otherwise non-degradable plastics. For instance, Dr. Sahu cites to papers discussing blends of biodegradable and non-biodegradable polymers. [CITE] But simple blending of two immiscible polymers (such as PE and PCL) will not increase the biodegradability of the non-degradable one. See generally, Tilsra and Tokiwa. McCarthy Rebuttal Report at ¶15.

Rebuttal

I disagree with Dr. McCarthy's statement that simply blending of two immiscible polymers (such as PE and PCL) will not increase the biodegradability of the non-degradable one. In the '199 patent, for example, Dr. McCarthy stated that polylactic acid (PLA) based polymer or copolymers and polymers of one or more polyesters (such as a polyester including PET) could be used to make "new biodegradable blends." See, col. 2, lines 10-21. See claim 9. Dr. McCarthy also asserted that compared to PLA alone, these blends "have superior tensile and mechanical properties such as stiffness, toughness, and elongation to break, *as well as excellent biodegradability*." See, Response to Office Action Dated February 24, 1998 in the prosecution history of the '199 patent. Emphasis added. Accordingly, the '199 patent claims that a biodegradable polymer (PLA) can be combined with any type of polyester (e.g. a non-biodegradable polyester containing PET) and provide a blend that has "*excellent biodegradability*."

Comment by Dr. McCarthy

22. One of the most serious flaws in the conclusions of the experts is that once biodegradation is established, it will continue to completion. In 1998, my colleagues and I discovered through research on biodegradable polymers that the amorphous regions of polymers biodegrade at a greater rate than the crystalline regions. [Footnote omitted.] M. Parikh, R. Gross, and S. McCarthy, The Influence of Injection Molding Conditions on Biodegradable Polymers, Journal of Injection Molding Technology, Vol. 2, No. 1, pp. 30-36 (March 1998). This phenomenon is critical as to why extrapolation of biodegradation is scientifically unsound. McCarthy Rebuttal Report at ¶22.

Rebuttal

As noted above, Dr. McCarthy's '199 patent presents data for only five (5) particular blends of PLA with a polyester identified under the BIONELLE trademark. See **Table 1** of the '199 patent. It is not clear what the molecular weight is of the BIONELLE polymers reported in Table 1. Nevertheless, Dr. McCarthy recites in claim 1 of the '199 patent that one can prepare a "biodegradable blend" of (a) a first polylactic acid-based polymer or copolymer, and (b) a second polymer consisting essentially of one or more polyesters." Accordingly, Dr. McCarthy extrapolated from the five (5) blends recited in Table 1 and the data presented in **FIGS. 11** and **12** to assert that PLA copolymers, in combination with any polyester (such as a polyester containing PET) would amount to a biodegradable blend formulation. I am of the view that contrary to Dr. McCarthy's assertion in his report, it is accepted among polymer scientists to extrapolate biodegradation testing to support a conclusion that a polymer will biodegrade. See, the '199 patent.

Comment by Dr. McCarthy

24. The experts rely on Dr. Timothy Barber's methodology in support of their conclusions regarding the environmental fate of ECM Plastics. The experts argue that the Barber Methodology is defensible because weight loss and evaluation of free chlorine are appropriate indicators of biodegradability. Reliance on these matrices to establish complete biodegradability is improper for two reasons. The measurement of weight loss and loss of mechanical properties as a measurement of biodegradation are not accurate. And the measurement of free chloride does not indicate biodegradation. McCarthy Rebuttal Report at ¶24.

Rebuttal

I disagree with Dr. McCarthy's assertion that measurement of weight loss and loss of mechanical properties as a measurement of biodegradation are not accurate. In the '199 patent, which is still in force, Dr. McCarthy relied upon **FIG. 12** showing the results of weight loss versus degradation time. In the specification, Dr. McCarthy wrote that "**FIG. 12** shows that polylactic acid biodegrades in compost, but slowly, and the addition of even 20% by weight BIONELLE#3000 increase this biodegradation dramatically."

Consistent with the position Dr. McCarthy took in the '199 patent and inconsistent with the position he takes in his rebuttal report, I am of the view that polymer scientists routinely use and consider reliable measurement of weight loss to support a claim of biodegradation. See, the '199 patent. See also, "*Advances in Properties and Biodegradability of Co-Continuous, Immiscible, Biodegradable*, Polymer Blends, Macromol. Symp. 144, 63-72 (1999).

On the issue of free chloride content, I reviewed Dr. Burnette's report and note that he stated as follows:

> "The observation that the addition of the ECM additive does allow for easier enzymatic breakage of the carbon-carbon bonds was seen on tests involving BioPVC®. A component of this test involved measuring the amount of chloride ion freed from the PVC structure (see structure of PVC above). ECM treated PVC showed statistically significant amounts of free chloride ion compared to the negative control of non-ECM treated PVC. The presence of this chloride ion is evidence that the stability of the ECM molecule is destabilized by the ECM additive, allowing for breakage or degradation when compared to the control." Burnette Report at ¶44.

I do not understand Dr. Burnette to claim that the "free chloride ion" is evidence of "breakage" of "the PVC" molecule meaning breakage of the C-C bonds. Read in full context, Dr. Burnette points out that a component of the testing was measurement of the chloride ion freed from the PVC structure. He points out that ECM treated PVC showed statistically significant amounts of free chloride ion compared to a negative control. He then pointed out that "the presence of the chloride ion is evidence that "*the stability of the PVC molecule is destabilized by the ECM additive, allowing for breakage or degradation when compared to the control.*" Accordingly, the presence of the chloride ion was properly interpreted as confirming that the PVC was *destabilized,* which itself would then allow for breakage or degradation. This is understandable as the loss of chlorine from the PVC chain will reportedly result in an unsaturated product which is more likely to be degraded. In addition, the metabolic needs of bacteria are reported to include carbon, water, nitrogen and chlorine. See, e.g., "*Considerations Affecting*

Biodegradability of PVC", GPEC 2006 Paper, R.F. Grossman Ph.D, RFG Consultants, J.E. Schleicher, Jr. and L. D'Alessio, Ultraflex Systems.

Conclusion

For the foregoing reasons, I disagree with the identified opinions and analysis offered by Drs. Michel and McCarthy in their respective rebuttal reports. In support of my opinion I have identified various scientific publications and Dr. McCarthy's U.S. Patent No. 5,883,199. This information supports my opinion that Dr. McCarthy has applied a standard for evaluation of whether ECM's Additive is biodegradable that is not present in the peer-reviewed scientific literature, and that is contrary to the standard that he adopted to identify a material as biodegradable in his own publications and in his U.S. Patent No. 5,883,199. With respect to Dr. Michel, I have identified what I consider to be serious deficiencies of his 2013 article *Biodegradability of Conventional and Biobased Plastics and Natural Fiber Composites During Composting, Anaerobic Digestion and Long Term Soil Incubation* and in his endorsement of the McCarthy report and analysis.

Dated: 7-7-14

Dr. Steven J. Grossman

Respondent’s Exhibit X

EXPERT REPORT OF DR. THABET TOLAYMAT
SUMMARY & CONCLUSIONS

I am environmental engineer with the United States Environmental Protection Agency ("EPA"). As an EPA engineer in solid waste management, I have substantial responsibility for, and experience with, United States ("U.S.") landfills. In my expert opinion:

- Plastic products manufactured with ECM's additive ("ECM Plastics") will not completely biodegrade in five years or less under ordinary U.S. landfill disposal conditions. The most common waste disposal method in the United States is by landfill, and most landfills do not have conditions that would allow for complete biodegradation in that timeframe (or anywhere near that timeframe).
- ECM testing data are not competent and reliable scientific evidence that ECM Plastics will biodegrade in five years or less in most landfills because none of the tests ECM offers to substantiate its claims simulate the conditions (*e.g.*, oxygen, moisture level, temperature) found in typical U.S. landfills. Additionally, the tests are based on inaccurate assumptions and faulty calculations and have severe methodological shortfalls.
- The materials I analyzed to prepare this report are listed on Exhibit A hereto, and they include the materials upon which ECM relies to substantiate its claims. I am not aware of any competent and reliable scientific evidence that ECM Plastics will biodegrade in typical landfills any more rapidly than plastics made without the ECM additive.

Dr. Thabet Tolaymat
U.S. Environmental Protection Agency

DATED: 6-4-2014

Table of Contents

I. EDUCATION, EXPERIENCE, AND TRAINING

1. I am an environmental engineer and researcher in the field of solid waste management at the U.S. Environmental Protection Agency. My research focuses on solid waste management, bioreactor landfills, waste containment performance, construction and demolition waste landfills, and the fate and transport of environmental pollutants. My *curriculum vitae* is attached hereto as Exhibit B.

2. I have both a Ph.D. and B.S. in Environmental Engineering (University of Florida, 2003 and 1997 respectively). For the past eleven years (since 2003), I have worked as an Environmental Engineer at the EPA Office of Research and Development.

3. My academic research and research for the EPA has focused primarily on waste disposal and landfills. In particular, I evaluate the performance of Solid Waste Containment Units (municipal solid waste, hazardous waste and ash mono-fill landfills), bioreactor landfills (a new landfill design that promotes degradation), organic pollutants, co-disposal of solid waste and hazardous waste, and construction and demolition waste.

4. In 2010, I led an interdisciplinary EPA team that evaluated sustainable materials management and construction and demolition debris. Between 2012 and 2013, I was the Acting Associate National Program Director for Emerging Materials and Sustainability under the Chemical Safety for Sustainability Research Program. In that role, I lead EPA's national research program for emerging materials[1] and sustainability. My role was to assist the National Program Director in these areas in setting EPA's research priorities and allocating EPA resources to regulatory needs. Currently, I am the project lead for sustainable waste and materials management at EPA's Office of Research and Development.

5. I am also part of the Interstate Technology & Regulatory Council (ITRC)'s Alternative Landfill Technology Team, a group comprised of state and federal agency

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[1] "Emerging materials" are newly-designed materials, such as nanomaterials. The term is typically used to contrast with traditional wastes. Because emerging materials are new, the implications of their disposal are not yet clear.

representatives (*e.g.*, EPA, Department of Energy), academics, industry stakeholders, etc., that develops guidance regarding alternative landfill covers.

6. As part of my responsibilities for the EPA, I provided expert advice regarding solid waste disposal for the World Bank and USAID, as well as to countries including Jordan, Taiwan, Russia, and Hong Kong.

7. A significant part of my education, training, and experience has involved conducting and evaluating tests that purport to show biodegradation and/or replicate landfill conditions. In particular, I have considerable experience conducting and evaluating tests based on large bench scale solid waste decomposition (lysimeter) studies.

8. Based on my education, training, and experience, I consider myself an expert in the field of landfill design and management.

9. I have not testified as an expert in the past four years.

10. I am not being compensated for my work with respect to this case. I volunteered to serve as an expert in this matter, and could have declined the position.

II. SCOPE OF REVIEW

11. Complaint Counsel asked me to evaluate whether, based on either (a) materials ECM Biofilms, Inc. ("ECM") submitted to the FTC, or (b) any other evidence I reviewed, the following claims are true:

a. Plastic products manufactured with ECM's additive ("ECM Plastics") will completely biodegrade in a reasonably short period of time (*i.e.*, one year) after customary disposal (*i.e.*, recycling, incinerator, landfill).

b. ECM Plastics will completely biodegrade in nine months to five years after customary disposal (*i.e.*, recycling, incinerator, landfill).

c. ECM Plastics have been shown to be biodegradable, biodegradable in most landfills, or biodegradable in nine months to five years under various scientific tests including, but not limited to, ASTM D5511.

12. Complaint Counsel provided me with the following materials relating to the ECM additive:

a. Materials, including published and unpublished study reports, protocols, data, and data analysis that, I understand, ECM submitted.

b. Materials, including published and unpublished study reports, protocols, data, and data analysis that, I understand, third parties provided in response to subpoenas from Complaint Counsel or ECM.

c. White papers, including expert reports of Dr. Ranajit Sahu submitted by ECM during the FTC investigation of ECM, and published articles cited in the white papers and/or Dr. Sahu's reports.

13. As I reviewed these materials and prepared this Expert Report, I relied extensively on my knowledge, expertise, and experience concerning landfill design and management.

I. DISPOSAL OF SOLID WASTE IN THE U.S.

14. The majority of landfills in the U.S. are "dry tomb" landfills in which biodegradation occurs very slowly. They are operated to contain waste and most waste containment protocols have the incidental effect of inhibiting biodegradation. Specifically, typical U.S. landfills have too little oxygen, are too cold, and too dry to support optimal conditions for organisms that biodegrade waste. As a result, even common organic materials such as leaves and food scraps take many years—even decades—to degrade in the average U.S. landfill.

15. ***What is Municipal Solid Waste?*** The EPA defines Municipal Solid Waste ("MSW") as waste consisting of everyday items that we use and then discard, such as product packaging, grass clippings, furniture, clothing, bottles, food scraps, newspapers, appliances, paint, and batteries. In 2012, the EPA estimated that Americans generated about 250 million

tons of waste. Biodegradable organic materials are the largest component of MSW: paper and paperboard account for approximately 28% of MSW and yard trimmings and food waste account for approximately another 28%. However, non-organic waste comprises a large portion of MSW: metals make up 9%; rubber, leather, and textiles 8%; and glass 5%. Other miscellaneous wastes make up approximately 3%. In 2012, plastic waste constituted over 12% of the total MSW generated, or about 31.7 million tons of plastic waste.

16. ***What is Customary Disposal?*** According to the EPA's latest evaluation of MSW management in the U.S. (2011), MSW is managed in one of four ways: recycling (26%), landfilling (53%), incineration (12%), and composting (8%).[2] In the United States, after the recovery of useful materials (*e.g.*, paper, glass, plastic) through recycling, the majority of MSW is customarily disposed of in incinerators or landfills.[3] Incineration involves burning MSW to produce energy, heat, or fuel. Landfilling (the topic of this report) is the placement of MSW in engineered areas in the ground. As Figure 1 shows, landfilling continues to be the dominant method for the management of MSW in the U.S.

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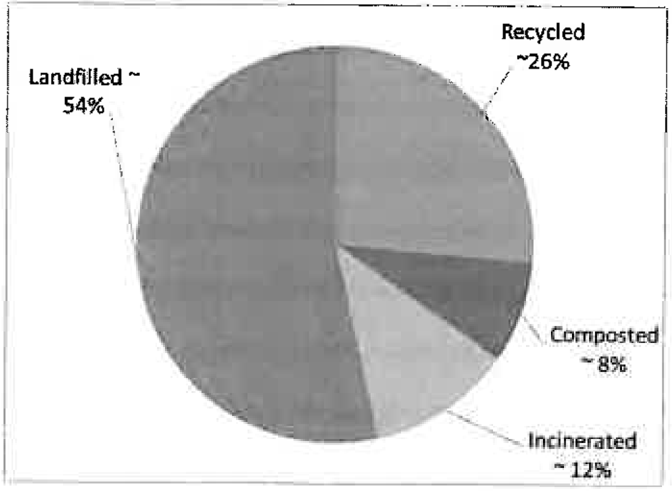
[2] No biodegradation occurs in recycling facilities or incinerators. In incinerators, MSW is combusted at a high temperature. Through recycling, materials that would otherwise be thrown away are turned into new products.

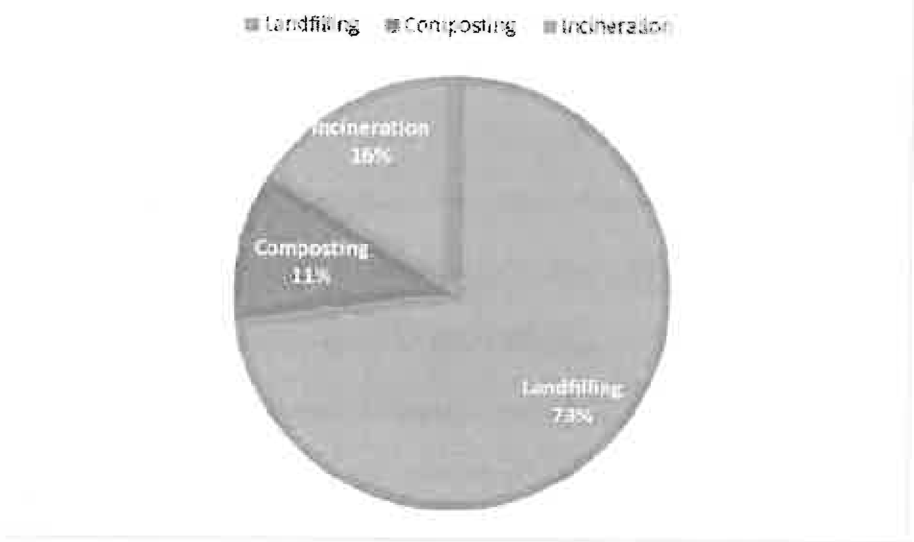
[3] A small fraction of MSW is also composted in municipal composting facilities. Composting involves collecting organic waste, such as food scraps and yard trimmings, and storing it under conditions (mainly aerobic or "with air") designed to help it biodegrade naturally. Based on data provided by the EPA, composting continues to be a relatively rare waste management method in the U.S.

Figure 1. Management Methods for Municipal Solid Waste



Indeed, when recycling is excluded, landfilling accounts for <u>73%</u> of MSW disposal and incineration 16% as presented in Figure 2.

Figure 2. Disposal of Municipal Solid Waste in U.S.



17. Landfills obviously have enormous importance to U.S. waste management, so any technology that would significantly impact landfilling would garner enormous attention.

II. BIODEGRADATION

18. Biodegradation is the decomposition of organic materials into more elementary compounds by the action of living organisms such as bacteria or fungi. These microorganisms break down organic material by using the carbon content as a food source. There are two types of biodegradation: aerobic and anaerobic.

- Aerobic biodegradation occurs in environments with oxygen, such as composting. Through aerobic biodegradation, microorganisms use oxygen to break down organic matter, yielding carbon dioxide, water, and energy, which is released as heat.[4] Biodegradation occurs much more rapidly in the presence of oxygen.
- Anaerobic biodegradation occurs in environments without oxygen, such as most landfills. During anaerobic biodegradation, microorganisms use a chemical other than oxygen (such as nitrate, sulfate, or iron) to break down organic matter, yielding methane, carbon dioxide, and energy, which is released as heat.[5] Biodegradation occurs much more slowly without oxygen.

19. There are five phases in the biodegradation process that occurs as landfill MSW stabilizes, each of which is summarized below:

- Phase I (Initial Adjustment). This is the brief period during which a new landfill actually contains oxygen—in the empty space around non-compacted waste. Because oxygen is at first present, this initial phase experiences the most biodegradation.
- Phase II (Transition). This phase describes the landfill's transformation from an aerobic (with oxygen) environment to an anaerobic one (without oxygen). As waste is added to the landfill and is compacted, the oxygen content of the landfilled solid waste decreases. As the oxygen supply is depleted, the rate of biodegradation slows and anaerobic biodegradation takes over.
- Phase III (Acid Formation). During this third phase, microorganisms called "acetogens" feed on the organic matter to produce acetic acid. As a result, the concentration of volatile fatty acids in the leachate (the liquid generated in the landfill mainly as a result of rain water passing through the waste) rapidly increases, and the pH in the landfill substantially decreases (*i.e.*, the landfill becomes much more acidic).

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[4] The formula for this process is as follows: $Biodegradable\ Organic\ Matter + O_2 \rightarrow CO_2 + H_2O + energy$.

[5] The formula for this process is as follows: $Biodegradable\ Organic\ Matter + HO_2 \rightarrow CH_4 + CO_2 + energy$. Gases released during the decomposition process are known as "biogases."

- Phase IV (Methane Fermentation). Microorganisms called "methanogens" convert organic matter into methane and carbon dioxide. As methanogens consume volatile fatty acids, the acidity of the landfill (*i.e.*, the pH) moves towards neutrality. During this phase, the majority of the wastes stabilizes.
- Phase V (Final Maturation and Stabilization). During this final (and longest-lasting) stage, biological activity decreases even more. The landfill has become mature and stable, with conditions that do not readily support biodegradation (oxygen-less, cool, dry).

20. Active landfills are dynamic and heterogeneous environments. As long as waste continues to be added to a landfill, all of the processes described in Part II above continue to occur. Indeed, in a single landfill—and even within a single landfill cell—all of the processes will occur simultaneously. Further contributing to the heterogeneity within landfills is the fact that the duration of the phases of biodegradation are highly dependent on the amount and type of organic matter, the pH (*i.e.*, acidity level), the presence or lack of oxygen, the moisture content, and the temperature.

III. LANDFILLS: ANAEROBIC, RELATIVELY COOL, "DRY TOMBS"

21. Biodegradation in landfills is remarkably slow because typical U.S. landfills are primarily anaerobic environments that are relatively cool with low-moisture. U.S. landfills are anaerobic, temperate, "dry tombs" by design: these conditions are engineered (and largely mandated by federal law) to facilitate the containment rather than stabilization of MSW.

a. Typical Landfills Are Anaerobic

22. The first significant factor that affects biodegradation in landfills is oxygen. The more oxygen, the faster the rate of biodegradation, because aerobic organisms decompose organic matter much faster than anaerobic organisms. In general, aerobic decomposition is more efficient than anaerobic and as a result faster.

23. In any given landfill, there is only one location (or "cell") that actively accepts waste. Waste is deposited into the active cell and after a day's activity, the MSW is compacted

and covered with at least 6 inches of earthen cover, which is intended to reduce odor, pests, and wind disturbances. Thus, landfill cells may start as aerobic environments rich with oxygen, thanks to the void space in non-compacted waste and exposure to air above the landfill, but the oxygen supply in the landfill does not last for long. Both the compaction and the covering reduce oxygen: covering the landfill cell reduces the amount of air that permeates the landfill, and compacting the MSW reduces air pockets. Effectively covering the landfill cells is very important to the safety and stability of the landfill; air infiltration can cause fires and explosions.

24. The oxygen level in landfills is further minimized by EPA-mandated "gas collection and control systems" (GCCS). Landfills generate significant amounts of methane and carbon dioxide, along with trace amounts of other gases that are dangerous to human health and the environment. GCCSs are designed to limit these gas effects. The efficacy of these systems depends on a constant landfill gas quality of nearly 50% carbon dioxide and nearly 50% methane. Emission of any significant amount of oxygen would disrupt the collection system and, again, create danger for fires and explosions. So, the EPA mandates monthly monitoring of every gas collection well-point at MSW landfills to ensure oxygen levels below 5% (volume).[6] Landfill operators that detect higher levels of oxygen have to recalibrate their gas collection system to decrease the amount of oxygen in the gas. This is usually done by reducing the vacuum pressure at the well-head and/or adding and packing new cover soil in the vicinity of the gas well.

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[6] There are exceptions to the EPA's rule. On a case-by-case bases, some facilities have been granted approval for a higher oxygen concentration based on a case by case evaluation and approval.

25. In short, because of these operating conditions—covering, compaction, and gas capture and collection systems—oxygen in typical MSW landfills in the U.S. is minimal. As a result, the slower anaerobic biodegradation process dominates landfills.

b. Moderate Temperatures in Typical Landfills

26. The second significant factor that affects biodegradation in landfills is temperature. Anaerobic decomposition occurs best within a thermophilic or high temperature range (50 to 60°C or 122°F-140°F). That is, biodegradation that occurs under higher temperatures happens more rapidly and has a better quality biogas production. Temperatures in typical MSW landfills in the U.S. are much lower than this: they range between 20°C and 40°C (68°F-104°F) and average around 37°C (98.6°F).[7] Biodegradation occurring at these temperatures (known as the mesophilic range) takes place more slowly and has relatively lower quality biogas production. Indeed, higher temperatures would increase the risk of surface fires and emission of greenhouse gases, so low-to-moderate temperatures are essential to good landfill management.

c. Typical Landfills Have Low Moisture Content

27. Finally, the third significant factor that affects the rate of biodegradation in landfills is moisture. The more moisture, the better suited the environment is for biodegradation, and the faster MSW will decompose. Moisture levels in typical MSW landfills in the U.S. are usually much too low for optimal biodegradation. Indeed, most landfills in the U.S. are aptly described as "dry tombs," for the reasons discussed here.

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[7] Landfill temperatures are not controlled but are often regulated by environmental conditions. For example a landfill in a hot climate, say Florida, would have a higher temperature than a similar landfill in colder climates, say Alaska.

28. The primary reason for low moisture level in typical landfills is a regulatory prohibition on addition of liquids. Regulations under the Resource Conservation and Recovery Act (RCRA), 40 CFR 239, *et seq.*, are the principal federal regulations governing disposal of solid waste. Just over 98% of the approximately 1,900 landfills in the U.S. are operated under Subtitle D of the RCRA guidelines. These are the so-called "Subtitle D landfills." A small fraction, less than 2%, operate under limited waivers.

29. The preamble to EPA's regulations under RCRA, Subtitle D recognizes that landfills are biological systems that require moisture for decomposition to occur, and moisture has real benefits:

- faster waste stabilization (because biodegradation occurs and concludes faster);
- improved leachate quality (*i.e.*, the liquid generated by landfills is less harmful); and
- better methane gas production (which can be valuable if properly captured).

But, RCRA Subtitle D also recognizes moisture causes problems as well. The preamble identifies the following negative effects from the addition of liquid:

- increased leachate production (*i.e.*, more liquids coming out of the bottom of the landfill);
- more frequent leachate collection system clogs;
- increased depth of leachate buildup on the bottom liner of the landfill, called the "hydraulic head on the liner." By law, the hydraulic head on the liner cannot exceed 30 cm.
- increased greenhouse gas generation (harmful to health and the environment if not properly collected); and
- increased odor problems.

30. At the time of the regulations, the EPA concluded that for most landfills, the costs of liquid addition outweigh the benefits. As a result, RCRA Subtitle D (40 CFR 258.27) prohibits the addition of liquids to most MSW landfills. In addition, Subtitle D mandates controls for rainwater run-off (rain water flowing away from the landfill) that serve to minimize natural addition of liquid to landfills via precipitation. In other words, all but a very small fraction of landfills in the U.S. are operated to reduce moisture content.

31. Without addition of liquid, the moisture content of a typical MSW landfill is low—in the range from 15 to 30%. This is well below the optimal range for decomposition (34-45%). The range of moisture content is broad (15-30%) because varying environmental conditions affect the moisture level. For example, a landfill in a wet environment in Florida during the rainy season will have a much higher moisture content (closer to 25% or a little higher) than a landfill in the Arizona desert (closer to 15%).

32. Leachate recirculation (also called leachate recycling)—which is permitted under Subtitle D—does increase moisture. That is, when leachate (the liquid generated in the landfill mainly as a result of rain water passing through the waste) is collected at the bottom of the landfill and added to the top of the landfill, the moisture content increases. However, even with aggressive leachate recirculation, the moisture content of the typical Subtitle D landfill still remains low. Leachate recirculation has a limited effect on moisture level because an enormous amount of liquid is needed to raise moisture content by even a small amount. To illustrate, it would take the addition of more than 1.3 million gallons of water to a small MSW landfill cell (500,000 tons MSW) to increase that cell's moisture content by a mere 1%.

33. Although the overwhelming majority of landfills in the U.S. are Subtitle D landfills with comparatively low moisture content, approximately 2% of landfills are specially

licensed by EPA to operate as "bioreactors." Bioreactors are managed as wet environments that promote biodegradation. In bioreactor landfills, the addition of bulk liquids enables the bioreactor to operate at a more optimal moisture range for biodegradation (at or above 40%). But bioreactor landfills remain a small subset of landfills because of the potential risks associated with them (described further in section IV).

34. One way to understand the differences in biodegradation rate among Subtitle D landfills and bioreactors is to compare the average predicted decay rates (rate of decomposition) and half-lives (amount of time required for half of the waste to biodegrade) of waste in each type. Decay rates and half-lives can be predicted from "landfill gas generation models," which measure landfill gas output to estimate biodegradation (because gas output is the best indicator of biodegradation in landfills). The most widely-used landfill gas generation model in the U.S. is the EPA's model (LandGEM),[8] which assumes a first order decay model. This type of model is also used by United Nation's Intergovernmental Panel on Climate Change (IPCC) to evaluate greenhouse gas emissions from landfills.

35. As Table 1 below shows, the decay rate increases with moisture, and the half-life decreases. In other words, the more moisture, the faster the rate of decomposition and the less time it takes for MSW to decompose. Table 1 demonstrates the enormous disparity in decomposition rates between wet bioreactors and arid landfills. To illustrate, if 100 pounds of fully biodegradable organics were disposed of in a bioreactor landfill, half of that mass (50

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[8] The equation that is the basis for this model is: $Q_n = k \cdot L_0 \cdot \sum_{i=0}^{n} \sum_{j=0\,0}^{0\,9} \frac{M_i}{10} \cdot e^{-k \cdot t_{i,j}}$
Where Q_n is CH_4 generation rate ($m^3\ yr^{-1}$) in year n; k is first order waste decay rate (yr^{-1}); L_o is CH_4 generation potential ($m^3\ Mg^{-1}$ wet waste); M_i is waste mass placement in year i (Mg); and t is time (yr). The US EPA presents default values for both k and L_o in its air emissions database (AP-42), however, site specific values can be determined. The waste decay constant (k) may be the most important variable in the determination of the LFG production rate.

pounds) will remain after 6 years. Comparatively, it would take almost 35 years for the same 50 pounds of mass to decompose in a Subtitle D landfill in an arid climate.

Table 1. Decay Rates and Half Life of Different Types of Landfills

Landfill Type	Precipitation (inch)	Decay Rate (yr^{-1})	Half Life (yrs)
Arid	<20	0.02	34.6
Moderate	20 to 40	0.038	18.2
Wet	> 40	0.057	12.2
Bioreactor	None	0.12	5.8

Waste, of course, is heterogeneous: different types of MSW will have different decay rates. Table 2 below shows the IPCC's decay values for various types of waste and climate.

Table 2. Ranges of Decay Rate (k) Values by Waste and Climate Type

Waste Type	Example of Waste Type	Decay Rate (k Values (yr^{-1}))		Half-life (yrs)	
		Dry Climates	Wet Climates	Dry Climates	Wet Climates
Slowly degrading wastes	Paper, textiles, wood or straw	0.01 to 0.05	0.02 to 0.07	70 to 14	35 to 10
Moderately degrading wastes	Other [non-food] organic putrescible/garden and park waste	0.04 to 0.06	0.06 to 0.1	17 to 12	12 to 7
Rapidly degrading wastes	Food waste and sewage sludge	0.05 to 0.08	0.1 to 0.2	14 to 9	7 to 3.5
Bulk wastes	Combination of above materials	0.04 to 0.06	0.08 to 0.1	17 to 12	9 to 7

These more granular decay rates show that even in wet climates MSW that is readily biodegradable (*e.g.*, food waste) will take anywhere from 7-14 years to fully biodegrade.[9] And the time for slowly degrading wastes takes up to 70 years in the same wet climate.

36. In short, whether considered by the entire landfill site, as in Table 1, or by the particular biodegradable component of the solid waste mass, as in Table 2,one would not expect complete biodegradation of even the most readily biodegradable component of MSW in the most favorable conditions in less than 7 years.

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[9] A rough estimate of the time to complete decomposition for fully degradable organics is double the half-life.

IV. DRY TOMBS VS. BIOREACTORS

a. Typical MSW Landfills: "Dry Tombs" With Minimal Biodegradation

37. All Subtitle D landfills are designed primarily as waste containment systems rather than waste treatment systems. The goal of landfilling is to minimize harms to humans and the environment (*e.g.*, groundwater pollution, greenhouse gas emission, fires, odor)—**not** to facilitate biodegradation. In fact, as discussed below, most of the tools for harm minimization have the effect of retarding biodegradation. A schematic of a typical Subtitle D landfill is presented in Figure 3.

38. The most significant feature of landfills as containment systems is the landfill containment liner system. Containment liner systems are typically constructed from two types of material: compacted earth with low permeability (*e.g.*, clay soil) and geosynthetic products such as plastic membranes. The liner system captures leachate (liquid that contacts or emerges from solid waste), and removes it from the landfill using natural and synthetic drainage layers and mechanical pumping systems.[10] As discussed in section 24 above, this feature reduces moisture, which slows biodegradation.

39. A second feature of waste containment is the waste filling plan. Waste is deposited on top of the containment system after a suitable protective layer has been placed, with the waste in designated cells. A cover of soil or alternative materials, waste compaction, grading, and rainwater run-off controls further serve to minimize odor, litter, fires, and runoff of rainfall that has contacted the waste. Again, as discussed in section 24 above, these features reduce both oxygen and moisture, which slows biodegradation.

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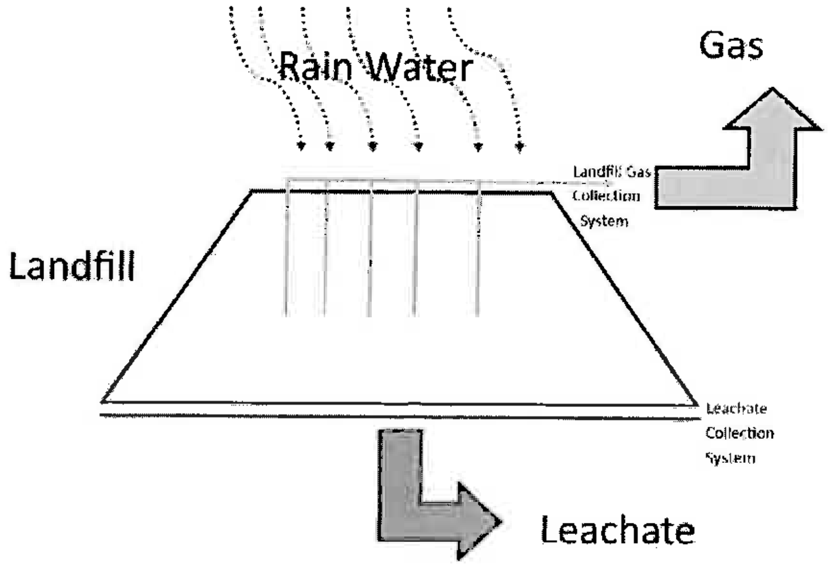
[10] U.S. Subtitle D landfill regulations require a bottom liner system to contain at least two feet of compacted soil with a permeability of less than 10^{-7} cm sec^{-1} overlain by a plastic geomembrane (typically 60-mil HDPE) and a leachate collection and removal system capable of maintaining the depth of ponded leachate on the liner to less than 30 cm (1 ft).

40. A third feature of waste containment is the monitoring protocol, by which the operator monitors the groundwater and soil vapor surrounding the containment areas, and if needed, institutes appropriate remedial measures to prevent groundwater contamination. Such remedial measures, again, often serve to reduce moisture and slow biodegradation.

Figure 3. Typical Subtitle D Landfill Schematics



41. A fourth feature of waste containment is the gas collection and control system. Under the Clean Air Act (CAA), MSW landfills must assess whether a threshold mass of non-methane organic compounds (NMOCs) are produced per year (50 Mg yr^{-1}), and if so, gas must be collected and controlled. The CAA regulations require monitoring and maintenance of the GCCS, which includes evaluating gas quality, landfill gas temperature, and extraction pressure on a routine basis from required collection points. For GCCS operated without a stringent regulatory framework, similar monitoring and maintenance measures may be employed to reduce environmental emissions and to optimize gas recovery when an energy conversion system is present. Gas collection and control systems usually include wells or trenches constructed in the waste to provide a conduit for gas removal, well-head controls at individual or clustered

extraction points, a piping system to route gas removed from the landfill to a central collection location, and a gas treatment or energy system.

42. At the end of a landfill's operating life (which widely varies), the facility must be appropriately closed, monitored and maintained for a federally-mandated period. Landfill closure involves construction of a capping system to minimize infiltration of rainfall into the waste mass and gas escape to the atmosphere. The aftercare period of an MSW landfill, referred to as post-closure care, consists of facility maintenance to ensure integrity of the containment and capping systems, removal and treatment of leachate, operation of the GCCS (if present), and continued monitoring of the environment outside the landfill (groundwater, soil vapor)—all of which have the incidental effect of suppressing oxygen, moisture, and temperature—and thereby severely inhibiting biodegradation. Current U.S. regulations require post-closure care for 30 years. Because of the operating conditions of the typical landfill during its life and this post-closure care, biodegradation will proceed at a slow rate for many decades after disposal of MSW.

b. <u>Bioreactor Landfills: Rare Landfills That Maximize Biodegradation</u>

43. The reality of the typical landfill—a "dry tomb" that incidentally minimizes biodegradation—is especially clear when the typical landfill is contrasted with a new, specialized type of landfill called a bioreactor. As discussed in section 30 above, bioreactors are an unusual type of landfill (approximately 2% of U.S. landfills as concluded in a recent ORD study) that, unlike "dry tombs," are operated to maximize biodegradation.

44. The theory behind a bioreactor is that decomposition should be maximized to limit the "active" life of the facility to those years when the site infrastructure is in its best condition, and when it is actively being monitored. Bioreactor landfill design attempts to

control, monitor, and optimize the waste stabilization process rather than simply contain wastes.[11]

45. The EPA Office of Solid Waste and Emergency Response promulgated the Research, Development, and Demonstration Permits for Municipal Solid Waste Landfills (RD&D rule) in 2004, which allows approved states to permit bioreactor landfills. Specifically, the permit waives Subtitle D's prohibition on bulk liquid introduction, which, as described in section III.c. above, limits moisture, and thereby, biodegradation. Both the RD&D rule and the permits are temporary modifications to Subtitle D regulations. RD&D permits last for 3 years, with optional 3-year renewals for a maximum of 12 years.

46. Bioreactor technology relies on maintaining moisture content near field capacity of solid waste (approximately 35 to 65%) and requires addition of liquids when it is necessary to maintain that percentage. With more moisture comes faster decomposition. With accelerated decomposition, the landfill gas generation rate increases. Gas extraction systems are used to capture and control this gas, oftentimes converting it to electricity.

47. Bioreactors remain rare and experimental: currently, only 16 states are authorized to permit bioreactors and less than 2% (32 of the approximately 1,900) operational MSW landfill sites are bioreactors. Although they present exciting possibilities for landfill control and energy generation, bioreactors do present significant risks that require further study. For example, an

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[11] The Solid Waste Association of North America defines a bioreactor landfill as: "a controlled landfill or landfill cell where liquid and gas conditions are actively managed in order to accelerate or enhance biostabilization of the waste. The bioreactor landfill significantly increases the extent of organic waste decomposition, conversion rates, and process effectiveness over what would otherwise occur with the landfill." In its final rule on National Emissions Standards for Hazardous Air Pollutants (NESHAPS) for landfills (40 CFR Part 63) Maximum Achievable Control Technology (MACT), the EPA defined bioreactor landfills as MSW landfills that utilize liquids other than leachate and gas condensate to achieve an average moisture content of 40% by weight or more.

increase in landfill moisture content tends to decrease the landfill's slope stability; from an engineering perspective and in my expert opinion, it is impossible for an above-ground landfill to have a moisture content of more than 78%. If the moisture content in a landfill is high enough it would lead to slope failure and collapse—a potentially cataclysmic result for a landfill. Because of this and other risks, EPA requires yearly monitoring reports for bioreactors and severely restricts the permitting.

48. It is, therefore, important to understand biodegradation-rich bioreactors as the exception. Biodegradation-inhibiting "dry tombs" remain the norm.

V. TYPE OF RELIABLE SCIENTIFIC EVIDENCE REQUIRED TO SHOW BIODEGRADATION IN LANDFILLS.

49. The optimal form of scientific evidence of landfill biodegradation is to conduct solid waste decomposition experiments in real landfill environments. Although feasible, such experimentation is highly time- and resource-intensive—and, therefore, rarely performed.

50. As a result, to analyze decomposition of solid waste in landfills, researchers generally simulate landfill environments in laboratory-based studies called "gas evolution tests" that analyze the Biochemical Methane Potential (BMP)[12]. The BMP is a measure of the anaerobically degradable carbon that is available in MSW for decomposition. In other words, it measures how much carbon in MSW can be metabolized by microorganisms.

51. To analyze the BMP, a researcher must first take a sample of refuse that has been dried and ground to pass 1-mm screed and add it to a batch reactor (typically a 160 mL serum bottle). Also added to the batch reactor is a growth medium (*e.g.*, vitamins and minerals) and an

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[12] Wang, Y.-S., Byrd, C. S. and M. A. Barlaz, 1994, "Anaerobic Biodegradability of Cellulose and Hemicellulose in Excavated Refuse Samples," Journal of Industrial Microbiology, 13, p. 147 - 53.

inoculum of anaerobic bacteria that typically appear in landfills. The sample is then incubated in a warm (mesophilic) environment for 60 days.

52. At the end of the 60-day period, the researcher measures the volume of methane gas that can be attributed to the refuse, because methane is a byproduct of anaerobic decomposition. By measuring the gas generated during decomposition, the rate of decomposition can be determined. This data are typically used to calculate the ultimate methane yield of a sample (*i.e.*, L_o) and thus providing information necessary for the calculation of a waste decay constant (k), which measures the half-life (*i.e.*, how long it takes for half of the material to degrade). It is noted that even the BMP gas generation value is referred to as ultimate methane yield. Meaning the maximum amount of methane generation under optimal conditions. Often, in landfill environment methane is generated at a lower rate and/or volume than the BMP predicts.

53. Other measures can provide evidence of degradation but are not themselves sufficient to establish biodegradation. For example, mass reduction can be evidence of degradation, but does not definitively establish biodegradation, because mass reduction can result from mechanical changes unrelated to biodegradation (*e.g.*, disintegration, evaporation or dissolution).

54. Changing these experimental parameters—even by a small amount—drastically impacts experimental outcomes and conclusions. For example, introducing even a small amount of oxygen will change a landfill simulation (anaerobic environment) into a compost test (aerobic environment). Increasing the moisture can change a typical landfill simulation to a bioreactor simulation. Increasing the temperature will cause activity by different bacterial communities, *i.e.*, thermophilic bacteria rather than the mesophilic bacteria that dominate dry tomb landfill

decomposition. Slight changes alone or in the aggregate will change the quality and quantity of gas generated, such that it is impossible to reliably compare the results of the test to real landfill conditions.

VI. ECM'S CLAIMS ARE FALSE AND NOT SUPPORTED BY RELIABLE SCIENTIFIC EVIDENCE.

55. My understanding is that ECM describes its additive as a propriety blend of a biodegradable polymer and conventional plastic, and that blending the ECM Additive with conventional petroleum-based plastics at 1% loading by weight will achieve a plastic that will completely biodegrade. According to the materials provided by ECM, the ECM Additive itself is at least 60%, and as much as 70%, biodegradable polymer.

56. Based on my review of the materials submitted by ECM and my knowledge and expertise in the area of landfill design and management, any claim that ECM Plastics will biodegrade in five years or less in most landfills is false. Moreover, the data ECM provided is not competent and reliable scientific evidence that supports its claims.

a. <u>ECM's Claims Are False Because They Are Incompatible With Landfill Reality</u>

57. As a threshold matter, it is important to note that even plastic comprised <u>entirely</u> of the ECM Additive—which is at least 60% known-biodegradable polymer—would not completely biodegrade in a landfill in five years or less. Based on decay constants adopted by the EPA and the United Nation's Intergovernmental Panel on Climate Change, the life of organic degradable materials in a dry tomb and bioreactor landfills are approximately 70 and 12 years respectively.

58. And, even if such plastic were located in a faster-degrading area of the landfill, it would not degrade in five years or less. As the decay rates in Table 2 (page 16) make clear, <u>even food</u> scraps will take, on average, <u>7 years</u> to biodegrade. Thus, ECM's claim that plastics treated

with its additives will biodegrade in five years or less (claimed half-life less than 2.5 years) in a landfill is tantamount to a claim that these plastics will biodegrade faster than quickly biodegrading food waste (half-life of 3.5 years from Table 2). In short, the claim that ECM Plastics will biodegrade in five years in a landfill is clearly false.

b. ECM's Claims Are Not Supported By Competent and Reliable Scientific Evidence

59. In addition to the claims of complete biodegradation in less than five years being false, these claims are unsubstantiated by the testing provided by ECM. I reviewed the following tests:

i. **ECM's Primary Substantiation:**

- The Environ Study: ECM-FTC-000069, "Anaerobic Biodegradation of bioPVC" conducted by the laboratory Environ
- The ChemRisk/McLaren Assessment: ECM-FTC-000106, "Ecological Assessment of ECM Plastic" conducted by the laboratory ChemRisk, as a service of McLaren/Hart

ii. **Composting Studies:**

- O.W.S. Composting Study: ECM-FTC-000124 and ECM-FTC-000244, the Final Report by O.W.S. Inc. of Biodegradation Testing, "Aerobic Biodegradation Under Controlled Composting Conditions for 40-gal trash bags," Study PFR-5, Mar. 3, 2000;

iii. **ASTM D5511 Reports:**

- Analytical Report No. N0946510-01 regarding "ASTM D 5511" Update regarding ASTM D5511-11 Update on Clear Films 476 & 477 @ 45 days"
- Analytical Report No. 1150851 regarding "ASTM D 5511 Extension Testing"
- Analytical Report No. 1253020 regarding "ASTM D 5511: Determining anaerobic biodegradation of plastic materials under high solids anaerobic digestion conditions"

iv. **Other Reports and Tests:**

- The Ecologica Report: ECM-FTC-000226, "Report about biodegradability of a plastic artefact [sic] . . ." by Ecologia Applicata srl., Cert No. 130/bio/180/10, Nov. 19, 2010
- Additional Biodegradation Tests of ECM Plastic (*See* Appendix A)

60. Each test suffers from faulty assumptions, poor methodology, and/or employs conditions inconsistent with typical landfills. Because of this, no test that ECM provided is competent and reliable scientific evidence to establish biodegradability, let alone biodegradability in a landfill.

i. ECM's Primary Substantiation

61. My understanding is the ECM has particularly relied on the results of two studies: the "Environ Study" and the "ChemRisk/McLaren Assessment." Neither of these highly flawed studies is competent and reliable evidence to support ECM's claims, for the reasons discussed below.

- Environ Study

62. The "Environ Study" ("Anaerobic Biodegradation of bioPVC," ECM-FTC-000069, conducted by the laboratory Environ) purports to provide data on the biodegradability of BioPVC (a type of plastic called polyvinylchloride (PVC) amended with the ECM additive). The Environ Study purports to show 2.6% biodegradation. The study design states it followed recommendations in ASTM D5526, "Standard Test Method for Determining Anaerobic Biodegradation of Plastic Materials Under Accelerated Landfill Conditions." The moisture content under the test was 62% (adjusted using landfill leachate); pH 8.0; temperature 40°C; and cellulose was used as a positive control.

63. The Environ Study has at least five defects. Each defect, by itself, prevents it from being competent and reliable scientific evidence that BioPVC would biodegrade in five years or less in a landfill. First, the moisture content of 62% greatly exceeds the moisture found in a typical dry tomb landfill in the U.S. (25-35% moisture). This flaw alone means that the test does not replicate landfill conditions.

64. Second, the test inaccurately relies on mass loss is the primary indicator of biodegradation[13] rather than following the gas generation model (*i.e.*, measuring the gas resulting from the decomposition process) set forth in ASTM D5526. Mass loss is an inadequate indicator of biodegradation because mass loss can result from other mechanical processes. To illustrate, consider the loss in the weight of a sugar cube in water; mass loss does not mean that the sugar cube is biodegrading. And, indeed, in these circumstances, it is more likely that the mass loss resulted from fragmentation of the BioPVC than from biodegradation.

65. Third, the study assumes, without scientific justification, that the initial trend of observed biodegradation will continue even when biodegradation has plateaued. The study states: "This typical microbial growth curve is an artifact of the closed system design used in this study. Under landfill disposal scenario, the microbial community would be replenished by fresh leachate, including a fresh supply of nutrients." (ECM-FTC-000075) This explanation suggests that biodegradation plateaued at 2.5% mass loss due to lack of microbes and nutrients. This explanation is implausible, because under the same test conditions, the cellulose (the control) continued to biodegrade. A more likely explanation for the plateau is that the microbes had

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[13] The study's method defines biodegradation amount as follows:

$$\%\ Biodegradation = \frac{Mass_{Initial} - Mass_{Final}}{Mass_{Initial}} X100$$

finished degrading the biodegradable carbon in the BioPVC (present because the ECM Additive is part biodegradable polymer) and could not degrade the conventional plastic.

66. Fourth, the study design assumes—without proving—the desired conclusion: that bioPVC is entirely biodegradable. The equation defining decomposition provides that the only means to achieve 100% decomposition is if the final mass ($\text{Mass}_{\text{final}}$) equals zero. This assumption is inaccurate; there will always be some assimilation of the carbon within the various microorganisms or some recalcitrant carbon that is not bioavailable for decomposition.[14]

67. Fifth, the Environ Study incorrectly reports several, varying decay rates for different stages of the degradation process, but it is well-accepted in the scientific community that a <u>single</u> decay rate describes the half-life of the <u>entire</u> degradation process under specified testing conditions. The study reports that the "first order rate constants ranged from 0.021 to 0.13 yr^{-}1" which corresponds to half-lives ranging from 4.2 to 29 years (full decomposition would range from twice that—8.4 to 58 years). Even if mass loss were an accurate indicator of biodegradation (it is not), the calculated decay constant for the process is incorrect. The correct decay constant, representing the half-life of the entire degradation process under the specified conditions, is 0.11 yr^{-1}. This was calculated by using the total mass loss (%) observed in the study over the 9 month period which is a more appropriate method for calculating the decay constant. Using the proper decay constant shows that (if the study's other flaws were ignored) the BioPVC's half-life would be 6.3 years and complete biodegradation would occur after 12.6 years—much longer than the 5 years to complete biodegradation that ECM claims.

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[14] Basically, think of the microorganisms as humans. Not everything we eat and breathe is ejected from our bodies. We "assimilate" nutrients and carbon within our bodies, which makes us grow. Recalcitrant carbon is like "fiber" to humans we can eat it but ultimately not able to digest it.

- <u>ChemRisk/McLaren Assessment</u>

68. The "ChemRisk/McLaren Assessment" (entitled "Ecologic Assessment of ECM Plastic, ECM-FTC-000106, conducted by the laboratory ChemRisk, as service of McLaren/Hart), like the Environ Study, is based on flawed methodology.

69. The first part of the Assessment consists of a short-term, 60-day aerobic composting test of film comprised of 50% ECM Additive. The second part of the Assessment discusses a long-term, 22-month aerobic test of a 5% ECM film and ECM pellets (additive). The third part of the Assessment presents the results of a 15-day anaerobic test of the ECM pellet (additive) that follows the methods under ASTM D5511-94. The Assessment purports to show that approximately 20% biodegradation was achieved for the 50% ECM film over about 60 days. It further purports to show that the ECM pellets achieved approximately 20% biodegradation over a period of 10 days.

70. The ChemRisk/McLaren Assessment has at least five defects. Each, by itself, prevents it from being competent and reliable scientific evidence of biodegradation of ECM Plastics in five years or less in a landfill. First, the aerobic test (conducted with oxygen) does not simulate the anaerobic (oxygen-less) conditions found in landfills. As discussed in section III.a. above, the phase in which MSW in a landfill would be exposed to oxygen is extremely brief, due to daily compaction of MSW. Thus, only anaerobic tests replicate landfill conditions.

71. Second, the anaerobic test purportedly conducted under ASTM D5511 contains implausible data. The figure on page ECM-FTC-000111 shows that cellulose control achieved <u>120% biodegradation</u> under aerobic composting conditions and the 50% ECM film achieved 19% biodegradation over a similar timeframe. It is not possible to achieve more than 100% biodegradation. Therefore, the reported 120% biodegradation is either an error resulting from

sloppy methodology—or it could reflect background decomposition of the organic components in the compost. If 20% of cellulose biodegradation derives from background decomposition, then a significant portion (or perhaps all) of the 19% biodegradation of the ECM film may likewise derive from background decomposition. Either way, the data is not reliable.

72. Third, even assuming the methodology and the data were correct, the 50% ECM film shows only 20% decomposition over the 60-day composting process. Since the ECM Additive is known to contain 60-70% biodegradable component and composting (unlike landfills) presents ideal conditions for biodegradation, the 20% decomposition is entirely consistent with degradation of the biodegradable component only.

73. Fourth, the ChemRisk/McLaren Assessment uses a true statement to present a misleading conclusion. On page ECM-FTC-000111, the Assessment states that: "in time, plastics produced using ECM pellets will biodegrade in aerobic conditions." That statement is true but misleading: given enough time—centuries, eons—anything will biodegrade. That does not mean that ECM plastics will biodegrade in any timeframe close to five years.

74. Fifth, the ChemRisk/McLaren Assessment erroneously conflates disintegration with degradation. The figure on page ECM-FTC-000113 claims to demonstrate that "LLDPE [plastic comprised of low-density polyethylene and ECM additive] cast film strips totally degraded" because they are fragmented in the picture. Disintegration does not necessarily entail biodegradation. To illustrate, consider how wet paper will shred; such disintegration is entirely unrelated to biodegradation. Without other reliable indicia of biodegradation, it is more likely that the ECM Additive causes fragmentation than biodegradation.

ii. Composting Studies

75. The O.W.S. Composting Study evaluated the biodegradation of 40 gal-trash bag plastic amended with ECM Additive under aerobic environment following ASTM D5338.98.[15] The temperature was 58°C, cellulose was used as a positive control. The study found that after 45 days the cellulose control decomposed 99.7% and that the trash bag decomposed 5.2% ± 8.3%.

76. It is not competent and reliable scientific evidence of ECM's claims for two reasons. First, composting studies are carried out under aerobic, oxygen-rich environments totally unlike typical MSW landfill environments. Second, the statistical analysis and uncertainties of the test undermine any conclusion that the sample biodegraded. The study reports 5.2% biodegradation with a standard deviation of ±8.3%. This standard deviation is broader than the purported biodegradation. With such wide standard deviation, biodegradation could have been zero—or the carbon in the sample could have even increased. In short, the level of uncertainty is so high that it is impossible to rely on the results.[16]

iii. Anaerobic Tests Conducted Under ASTM D5511

77. Tests conducted under ASTM D5511 (Standard Test Method for Determining Anaerobic Biodegradation of Plastic Materials Under High-Solids Anaerobic-Digestion Conditions)—no matter how well-conducted—are not competent and reliable scientific evidence of biodegradation in a landfill, because this ASTM method does not simulate typical landfill conditions. Indeed, the test protocol itself states that it is intended to replicate an anaerobic digester, whose conditions (temperature and moisture) are far more favorable to biodegradation

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[15] The report does not identify what percentage of additive the test specimen contained.

[16] And, in fact, ASTM D5511 section 1.4 specifically states that if the error of the test spans zero, then no biodegradation may be reported.

than those in a typical landfill. ASTM D5511 thus can provide data about anaerobic biodegradation, but it categorically cannot provide data about the rate of biodegradation in a typical landfill.

78. Not surprisingly, then, ASTM D5511 calls for conditions that diverge dramatically from landfills. For example, the average landfill temperature is 37°C, but ASTM D5511 calls for testing at 52°C. ASTM D5511 requires at least 20% dry solids (*i.e.*, 80% moisture); the average moisture content in landfills is much lower (15-35% moisture). These significant differences in temperature and moisture mean that ASTM D5511 tests have conditions far more favorable to biodegradation than landfills do; that the gas generated in ASTM D5511 test will, therefore, differ both in quality and quantity from gas generated by the typical landfill; and ASTM D5511 gas generation will not provide data about the rate of biodegradation in landfills.

79. ASTM D5511 also calls for use of an inoculum that is not available from a U.S. laboratory—and likely not comparable to the bacterial community found in the typical U.S. landfill. Inoculum used in ECM's ASTM D5511 tests (described on page ECM-FTC-000506) were from organic compost from New Milford Farms (founded to compost food by-product waste from Nestle USA operations) and Mattabasset Waste Treatment Facility (treats waste water and not MSW). These inoculums are very different from the bacterial community in the typical MSW landfill.

80. A limiting factor in the utility of ASTM D5511 tests is that the test method does not provide any means to distinguish biodegradation of the plastic and the additive, because the method assumes testing of homogenous material. As a result, ASTM D5511 results cannot

definitively show biodegradation of the plastic (as opposed to the biodegradable component in the ECM Additive) or the rate of biodegradation of the plastic.

81. Because the method assumes homogenous test material, it also assumes that any gas generation is synonymous with biodegradation of the test material. But this is not necessarily the case. If a non-biodegradable component were mixed with cellulose, the cellulose would generate gas, giving a false "positive" for the material as a whole. For this reason, it is impossible to tell from an ASTM D5511 test whether biodegradation resulted from the biodegradable component in the ECM Additive or from the plastic itself.

82. In addition to these overarching problems with results from ASTM D5511 testing, individual ECM ASTM D5511 tests have methodological flaws. For example, Analytical Report ASTM D5511 (ECM-FTC-000497) relies on only four gas composition readings, one of which was incomplete—even though conclusions based on only three readings are far from definitive. This same test relies on calculation methods that skewed the results to show more biodegradability than actually occurred. Indeed, the reported average methane content (39%) is higher than 6 of the 9 methane readings (reported as 26%, 26%, 26%, 30%, 31%, and 31%) for ECM Plastic meaning that three readings skewed the entire data set. Moreover, sloppiness renders the report's conclusions questionable: the average percent gas composition data used on page ECM-FTC-000500 to calculate biodegradability does not match the actual average from the readings on page 000499. The reported average methane content for PE 1% ECM with Slip Additive used on page ECM-FTC-000500 is reported to be 39%, but the actual average methane content using the data on page ECM-FTC-000499 is 34%.

83. Statistical uncertainties likewise render its conclusions questionable. The 5% assessed biodegradability is within the range of uncertainties inherent to the experimental design

of the ASTM D5511. For example, measuring gas volume using a graduated cylinder one would expect to be off by 5%. This is true because we are using the human eye and human judgment to decide if the gas volume is 8.3 mL or 9.2 mL. This is true with the ASTM D5511 methodology as a whole and not particular to this experiment. The gas volume measurement protocol proscribed in the ASTM D5511 contains an inherent high level of variability.

84. Similarly flawed is Analytical Report ASTM D5511 (ECM-FTC-000503), which purports to show 4.54% biodegradability of polypropylene clear tape in 45 days. As with the report discussed above, this minimal biodegradation is not statistically significant. Moreover, the report offers no scientific justification for the assumption that biodegradation that plateaued will resume.

iv. Other Reports and Tests

o Ecologica Report

85. The Ecologica Report, ECM-FTC-000226, "Report about biodegradability of a plastic artefact [sic] . . ." by Ecologia Applicata srl., Cert No. 130/bio/180/10, Nov. 19, 2010, purports to show aerobic decomposition of a plastic artifact containing 1% ECM additive, with 20% biodegradation over a period of 180 days. It is generous to describe this as a "test report" because the document contains no information about methodology or how biodegradation was calculated. Moreover, the report's biodegradability (on page ECM-FTC-000228) suggests that at day zero, there was 1.6% biodegradability—a scientific impossibility which undermines the accuracy of the biodegradation model and the results of this study.

o Additional Biodegradation Tests of ECM Plastic

85. I reviewed additional biodegradation tests of ECM plastic from various laboratories. Almost half of the tests showed no biodegradation or incomplete biodegradation of the plastic. The remaining tests are not competent and reliable evidence to support ECM's

claims, because the tests suffer from the same faulty assumptions, poor methodology, and/or use of conditions inconsistent with typical landfills as ECM's substantiation tests. Appendix A of this report is a table that summarizes my review of the additional biodegradation tests. The table identifies each test by name and document number and provides my assessment of the test.

VII. CONCLUSION

86. In conclusion, in my expert opinion, ECM's claims that its additive will make conventional plastic biodegrade in approximately five years or less in a landfill is false and unsubstantiated.

GLOSSARY

A.

Acetogen: Bacteria that generates mainly acetic acid as a byproduct of their respiration in an environment that lacks oxygen.

Aerobic: An environment where there is oxygen.

Anaerobic: An environment that lacks oxygen.

Anaerobic Digester: A vessel where decomposition of organic matter takes place in an environment without oxygen. Anaerobic digesters are not common in the U.S. and typically only treat farm waste or wastewater.

American Society for Testing and Materials (ASTM): A standards organization that develops and publishes voluntary technical standards for wide range of materials.

ASTM D5511 1.1: The Standard Test Method for Determining Anaerobic Biodegradation of Plastic Materials Under High-Solids Anaerobic-Digestion Conditions. The test method evaluates the degree and rate of biodegradation in an environment that lacks oxygen for plastic materials in high-solids environments.

ASTM D5526: The Standard Test Method for Determining Anaerobic Biodegradation of Plastic Materials Under Accelerated Landfill Conditions.

B.

Batch reactor: The term used to describe vessels that are used in various chemical and industrial processes.

Biochemical Methane Potential: A batch test that evaluates the degradation of materials under an environment lacking oxygen. The BMP results presents the upper range of decomposition a material can achieve in such environments.

Biogas: The gas generated a result of the decomposition of organics usually under anaerobic conditions. In these environment the gas consists of methane and carbon dioxide.

Bioreactor Landfill: A specific type of landfills that is operated with the intent to enhance the decomposition of materials mainly through but not limited to the addition of liquids.

C.

Clean Water Act: The main federal law that governs water pollution in the United States originally passed in 1972.

Compost: Organic matter that has been degraded under an oxygen rich environment.

Containment Liner System: A system of controls that is implemented to contain potential spills or contaminants. The system is usually made from plastic liners with other controls.

Control Sample: Samples that are treated as the regular experimental samples but are designed to have a well-documented outcome.

D.

Decay Rate: The rate at which organic matter is degraded to more stable forms.

E.

Environmental Protection Agency (EPA): The federal agency that is charged with protecting human health and the environment in the United States.

EPA Office of Solid Waste and Emergency Response: The EPA Office that provides policy, guidance and direction for the Agency's emergency response and waste programs.

G.

Gas Collection and Control System (GCCS): A series of piping that collects gas from the landfill and sends it either to a flaring station

Gas Evolution Test: Any test that evaluates the generation of gas under anaerobic environments.

H.

Half-Life: The amount of time it takes material to decompose to have of its original weight.

Hydraulic Head: The depth of water that builds up on the bottom liner in a landfill.

I.

Inoculum: A source material that is used to introduce a bacteria to an environment.

IPCC: Intergovernmental Panel on Climate Change.

L.

Landfill Containment Liner System: Layers of plastic and earthen material that contains the contaminants within the landfill rather than allow them to contaminate the environment.

LandGEM: Landfill Gas Emissions Model. An automated estimation tool with a Microsoft Excel interface that can be used to estimate emission rates for total landfill gas, methane, carbon dioxide, nonmethane organic compounds, and other air pollutants from municipal solid waste landfills.

Leachate: The liquid that is generated from the bottom of landfills as a result of water contacting the solid waste mass.

Leachate Collection System: A liner and piping system that is installed at the bottom of landfills. The system is designed to collect the leachate from the landfill and direct them to tanks for treatment.

Leachate Recirculation: A landfill leachate management method where the liquids generated at the bottom of the landfill are collected and then reintroduced into the top of the landfill.

Low-Density Polyethylene (LDPE): A type of plastic.

M.

Mesophilic: Bacteria that likes moderate temperature – not too hot and not too cold.

Methanogen: Bacteria that generates methane in the process of breaking down organic matter.

Municipal Solid Waste: Any solid material that is thrown out from residential homes.

N.

Non-methane organic compounds (NMOC): Carbon based trace compounds that are commonly found in the landfill gas (*e.g.*, benzene).

P.

Polyethylene (PE): A type of plastic.

Polymer: A large molecule composed of many repeated subunits.

Polypropylene (PP): A type of plastic.

Polyvinylchloride (PVC): A type of plastic.

R.

Research, Development, and Demonstration (RD&D) Permits: Permits that some states issue landfills for research and demonstration purposes.

Resource Conservation and Recovery Act (RCRA): The federal law that governs the management and disposal of solid and hazardous waste.

S.

Subtitle D: The specific provision in the federal solid waste regulations (RCRA) that governs nonhazardous solid waste.

Subtitle D Landfill: Any landfill that is designed and operated in accordance with RCRA Subtitle D.

T.

Thermophilic: An organism that thrives at relatively high temperatures, between 45 and 122°C.

U.

UN IPCC: *See* IPCC.

V.

Volatile Fatty Acids: Organic acids that are by-products of anaerobic decomposition. An example is acetic acid (vinegar).

W.

Waste Filling Plan: A plan that is developed by landfill operators that shows the sequence to be followed when filling the landfill with solid waste.

Appendix A

Summary of Review of Additional Biodegradation Tests

Test Name	Document Number	Assessment
ACM Report	ECM-112225	The test is not competent and reliable evidence because it was conducted under aerobic conditions and temperature conditions (58°C) that did not simulate a typical landfill environment. *See* ECM-112227 ("[A]ll the samples had poor aerobic compostability")
BMP-WMA	ECM-114157	The test showed incomplete biodegradation because the test reported no measurable weight loss. *See* ECM-114158 ("Quality of methane is very low. To put in perspective, if the cutlery were made with 100% cellulose, then 0.17% was converted to methane.").
Colplast Aerobic Test	ECM-112365	The test is not competent and reliable evidence because it was conducted under aerobic composting conditions that did not simulate a typical landfill environment.
Diposo Environmental Test	ECM-114679	• The test showed incomplete biodegradation because the test reported only less than or equal to 0.05% biodegradability measurements for the testing samples compared to a 100% biodegradability measurement for the positive control. • In addition, the test is not competent and reliable evidence because it was conducted under temperature conditions (58°C) that did not simulate a typical landfill environment.
DOW Test	ECM-112176	The test is not competent and reliable evidence because it was conducted under aerobic composting conditions (ASTM D-5338) that did not simulate a typical landfill environment.

ETV Report	ECM-112391	The test is not competent and reliable evidence because it was conducted under aerobic conditions that did not simulate a typical landfill environment.
Measurements	ECM-113084	• The test showed no biodegradation because there was no difference in results between the samples and the inoculum. • In addition, the test is not competent and reliable evidence because it was conducted under anaerobic digester conditions (ASTM D-5511) that did not simulate a typical landfill environment.
Measurements HSAD OW Test	ECM-113608	• The test showed no biodegradation because the test showed a -3.7% degradation measurement for the testing sample compared to an 85.5% degradation measurement for the cellulose control. • In addition, the test is not competent and reliable evidence because it was conducted under anaerobic digester conditions (ASTM D-5511) that did not simulate a typical landfill environment.
Microtech 1998	ECM-111823	The test is not competent and reliable evidence because it was conducted under aerobic composting conditions that did not simulate a typical landfill environment.
Microtech Test Protocol	ECM-111884	The test is not competent and reliable evidence because it was conducted under aerobic conditions that did not simulate a landfill environment.
OWS Aerobic Test 1999-2000	ECM-111843	The test is not competent and reliable evidence because it was conducted under aerobic conditions that did not simulate a typical landfill environment.
OWS Aerobic Test On 5% Load Film	ECM-112027	The test is not competent and reliable evidence because it was conducted under aerobic conditions that did not simulate a typical landfill environment.

OWS Aerobic Test On 50% Pellet	ECM-112080	The test is not competent and reliable evidence because it was conducted under aerobic conditions that did not simulate a typical landfill environment.
OWS HSAD Test On ECM Pellet	ECM-112008	• The test is not competent and reliable evidence because it was conducted under moisture conditions (60-75%) and temperature conditions (52°C) that did not simulate a typical landfill environment. *See* ECM-112013 ("It should be noted that the results obtained in this test are only valid for the testing conditions described in this report and can therefore not automatically be extrapolated to other testing conditions.")
OWS Toxicity Tests On ECM Pellet	ECM-111891	The test is not competent and reliable evidence because it was conducted under aerobic composting conditions that did not simulate a typical landfill environment.
Shields Eden ASTM5511	ECM-114416	• The test showed incomplete biodegradation because the test reported only 8.5% and 1.1% biodegradability measurements for the testing samples compared to a 100% biodegradability measurement for the positive control. • In addition, the test is not competent and reliable evidence because it was conducted under moisture conditions and temperature conditions that did not simulate a typical landfill environment.
Shields Eden D 5511 Report	ECM-113977	• The test showed incomplete biodegradation. *See* ECM-113983 ("Prediction of time to total biodegradation is still difficult"). • In addition, the test is not competent and reliable evidence because it was conducted under moisture conditions (52%) and temperature conditions (53°C) that did not simulate a typical landfill environment. • Furthermore, the test used a once-a-week gas measurement, instead of a five-times-a-week gas measurement required by the ASTM 5511 testing protocol.

Shields HSAD OW Test	ECM-114219	• The test showed no biodegradation. *See* ECM-114223 ("the biodegradation percentage of the test items was slightly negative; this should be interpreted as zero biodegradation"). • In addition, the test is not competent and reliable evidence because it was conducted under different moisture conditions and temperature conditions that did not simulate a typical landfill environment.
Shields Testing	ECM-114059	• The test is not competent and reliable evidence because it solely relies on weight loss as an indicator of biodegradability. • In addition, the test was conducted under different temperature conditions (60°C) that did not simulate a typical landfill environment.
Sweettape ASTM D5511	ECM-114353	The test is not competent and reliable evidence because it was conducted under moisture conditions (78%) that did not simulate a typical landfill environment.
Sweettape Enterprise	N/A	• The test showed incomplete biodegradation because the test reported only a 3.8% biodegradability measurement for the testing sample compared to an 88% biodegradability measurement for the control. • In addition, the test is not competent and reliable evidence because it was conducted under different moisture conditions and temperature conditions that did not simulate a typical landfill environment.

Transilwrap	ECM112244	• The test showed incomplete biodegradation because the test reported only an 8% biodegradability measurement for the testing sample compared to a 93% biodegradability measurement for the cellulose control over a 233-day testing period. • In addition, the test is not competent and reliable evidence because it was conducted under different moisture conditions and temperature conditions that did not simulate a typical landfill environment. • Furthermore, the test used a once-a-week gas measurement, instead of a five-times-a-week gas measurement required by the ASTM 5511 testing protocol.
Tycoplas	ECM-114412	The test is not competent and reliable evidence because it was conducted under different moisture conditions and temperature conditions that did not simulate a typical landfill environment.
3M Test	3M-FTC-0000106	The test showed no biodegradation.[1] *See* 3M-FTC-0000108 ("Overall, the results show that ECM biofilm MasterBatch Pellets additive blended at 1.1% and 2% did not enhance the biodegradation of the polypropylene and polyethylene resin tested").
Ashish Mithal E-mail and Attached Excel Spreadsheet Entitled "NCSU-Test Results BMP-WMA"	N/A	The test showed minimal biodegradability because the test reported low methane quality.

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[1] The test was conducted under aerobic conditions that did not simulate a typical landfill environment. However, as discussed in Section II *supra*, such aerobic conditions serve to accelerate, not hinder, biodegradation.

Plastic Color Corp of NC	N/A	The test is not competent and reliable evidence because it was conducted under aerobic conditions that did not simulate a typical landfill environment.
MiniGrip	N/A	The test is not competent and reliable evidence because it was conducted under different moisture conditions and temperature conditions that did not simulate a typical landfill environment.
MiniGrip 365 Days[2]	N/A	• The test showed incomplete biodegradation because the test reported only a 17% biodegradability measurement for the testing sample compared to a 98% biodegradability measurement for the positive control over a one-year testing period. • In addition, the test is not competent and reliable evidence because it was conducted under different moisture conditions and temperature conditions that did not simulate a typical landfill environment.

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[2] My assessment of the MiniGrip 365 Days test also applies to the following biodegradation tests on ECM plastic I evaluated: MINIGRIP ASTM D5511 105 Day Test Report #1149980.doc; MINIGRIP ASTM D5511 120 Day Test Report #1149980 .doc; MINIGRIP ASTM D5511 120 Day Test Report #1149980 CORRECTED on 10-04 .doc; MINIGRIP ASTM D5511 135 Day Test Report #1149980 on 10-24 CORRECTED .doc; MINIGRIP ASTM D5511 15 Day Test Report #1149980.doc; MINIGRIP ASTM D5511 150 Day Test Report #1149980 11-02.doc; MINIGRIP ASTM D5511 165 Day Test Report #1149980.doc; MINIGRIP ASTM D5511 180 Day Test Report #1149980.doc; MINIGRIP ASTM D5511 195 Day Test Report #1149980.doc; MINIGRIP ASTM D5511 210 Day Test Report #1149980.doc; MINIGRIP ASTM D5511 225 Day Test Report #1149980.doc; MINIGRIP ASTM D5511 240 Day Test Report #1149980.doc; MINIGRIP ASTM D5511 255 Day Test Report #1149980.doc; MINIGRIP ASTM D5511 270 Day Test Report #1149980.doc; MINIGRIP ASTM D5511 285 Day Test Report #1149980.doc; MINIGRIP ASTM D5511 30 Day Test Report #1149980.doc; MINIGRIP ASTM D5511 300 Day Test Report #1149980.doc; MINIGRIP ASTM D5511 315 Day Test Report #1149980.doc; MINIGRIP ASTM D5511 330 Day Test Report #1149980.doc; MINIGRIP ASTM D5511 345 Day Test Report #1149980.doc; MINIGRIP ASTM D5511 365 Day Test Report #1149980.doc; MINIGRIP ASTM D5511 45 Day Test Report #1149980.doc; MINIGRIP ASTM D5511 60 Day Test Report #1149980.doc; MINIGRIP ASTM D5511 75 Day Test Report #1149980.doc; and MINIGRIP ASTM D5511 90 Day Test Report #1149980.doc.

Fellows Eden	N/A	• The test is not competent and reliable evidence because it was conducted under different moisture conditions and temperature conditions that did not simulate a typical landfill environment • In addition, graphs presented in the document were unclear, cannot distinguish between treatments, and there was no description of the samples provided.
FPI Eden	N/A	• The test is not competent and reliable evidence because it did not identify the testing method used. • The test showed incomplete biodegradation because the test reported only a 7% biodegradability measurement for the testing sample compared to a 100% biodegradability measurement for the positive control over a 290-days testing period.
Shields Eden full report	N/A	• The test showed incomplete biodegradation because the test reported only a 8% biodegradability measurement for the testing sample compared to a 100% biodegradability measurement for the positive control over a 22 weeks testing period. • In addition, the test is not competent and reliable evidence because it was conducted under different moisture conditions and temperature conditions that did not simulate a typical landfill environment.
Smith Oasis-Eden	N/A	• The test is not competent and reliable evidence because it was conducted under different moisture conditions and temperature conditions that did not simulate a typical landfill environment • In addition, graphs presented in the document were unclear, cannot distinguish between treatments, and there was no description of the samples provided.

Barlaz Biodegradability Paper	N/A	The test is not competent and reliable evidence because it did not present plastic-specific data.
FP Intl BMP Report 11-15-10[3]	N/A	• The test showed incomplete biodegradation because the test reported only a 7.8mL methane generation per gm testing sample compared to a 414.8 mL the positive control. • The test is not competent and reliable evidence because it was conducted under different moisture conditions that did not simulate a typical landfill environment.
Environ Final Report 030212.pdf[4]	N/A	• The test is not competent and reliable evidence because it has the same defects as Environ Study ECM-FTC-000069. *See supra* ¶¶ 62-67.

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[3] My assessment of the FP Intl BMP Report 11-15-10 test also applies to the following biodegradation tests on ECM plastic I evaluated: FP Intl BMP Report; R1 Loosefill Only; and NC State_Barlaz Testing.

[4] My assessment of the Environ Final Report 030212 test also applies to the following biodegradation tests on ECM plastic I evaluated: Environ-FP-Monthly Report; Environ Samples After 4 Months Exposure; Environ Final Report BioPVC; and Preliminary Report 100311.

Document	Bates Number	Prepared By
Email: from D. Rosenfield (Kelley Drye) to: K. Johnson RE: Revised Proposed Consent Agreement wth ECM		
Environmental Science and Technology: Is biodegradability a desirable attribut for discarded solid waste?	ECM-FTC-000510-000550	James w. Levis and Morton A. Barlaz
ECM BioFilms First Submission Response		
ECM BioFilms Third Submission Response		
ECM BioFilms Access Letter		
ECM BioFilms Fourth Submission Response		
ECM BioFilms Second Submission Response		
ECM BioFilms Partial Response		
ECM BioFilms TDS	ECM-FTC-000001	
ECM BioFilms MSDS	ECM-FTC-000002-000007	
ECM BioFilms MSDS	ECM-FTC-000008-000012	
ECM BioFilms Certificate	ECM-FTC-000015	
ECM BioFIlms Flyer	ECM-FTC-000017	
ECM BioFilms Mechanism for the Biodegradation of Products	ECM-FTC-000044	
ECM BioFilms Life Expectancy of Master Pellets	ECM-FTC-000053	
ECM BioFilms Technology for Biodegradation Plastics	ECM-FTC-000058	
Anaerobic Biodegradation of BioPVC Final Report prepared for BIOPVC	ECM-FTC-000069-000080	ENVIRON
Email: from: T. Barber (Environ) to P. Kappus (BioPVC) RE: Certification of results	ECM-FTC-000081	ENVIRON
Email: from: T. Barber (Environ) to P. Kappus (BioPVC) RE: Interim Repor	ECM-FTC-000082-00088	ENVIRON
Timothy R. Barber Experience Highlights	ECM-FTC-000089-00091	
Timothy R. Barber Resume	ECM-FTC-000092-000105	
Ecological Assessment of ECM Plastic for Microtech Research Inc	ECM-FTC-000106-000123	ChemRisk
O.W.S. Final Report - Biodegradation Testing	ECM-FTC-000124-000162	
SEM Imaging - EPS Samples	ECM-FTC-000163-000170	
SEM Imaging - Bubble Wrap	ECM-FTC-000186-000197	
SEM Imaging - Green PET Bottles	ECM-FTC-000171-000185	
SEM Imaging - PVC Samples	ECM-FTC-000198-000205	
SEM Examination - ECM Plastic	ECM-FTC-000206-000225	
Report about biodegradability of a plastic artefact	ECM-FTC-000226-000229	Ecologia Applicata
The case of ECM MasterBatch Pellets additive	ECM-FTC-000230-000241	Ecologia Applicata
Time Elapse Photos of the ECM MaterBatch Pellets Formula	ECM-FTC-000321	
ECM BioFilms Letter to Interested Party	ECM-FTC-000488-000489	
ECM BioFilms Letter to Interested Party	ECM-FTC-000490-000491	
Analytical Report - ASTM D 5511	ECM-FTC-000497-000500	Northeast Laboratories
ASTM D 5511 UPDATE	ECM-FTC-000501-000502	Eden Laboratories
Analytical Report - ASTM D 5511 - Extension Testing	ECM-FTC-000503-000505	Northeast Laboratories
Analytical Report - ASTM D 5511	ECM-FTC-000506-000509	Northeast Laboratories
EPA: Reducing Greenhose Gas Emissions from Municipal Solid Waste Landfills		
Open Access: Microbial Population Associated with Plastic Degradation		Himani Bhardwaj
Internal Journal of Moelcular Sciences: Biodegradability of Plastics		Yutaka Tokiwa
Critical Reviews in Biotechnology: A Review of Plastic Waste Biodegradation		Ying Zheng
First White Paper and Expert Report		Kelly Drye and Warren
Second White Paper and Expert Report		Kelly Drye and Warren
Third White Paper and Expert Report		Kelly Drye and Warren
Advanced Materials Center, Inc. (AMC) Report	ECM-112225-112239	Chris Rausch

WMA Samples removed from BMP Bottles	ECM-114156-114190	BF
Colplast Biodegradability Test	ECM-112365-112377	
Environ: Interim Test Report for Dispoz-o Products	ECM-114679-114683	Environ
Dispozo Biogedration Test Design - November 2008	ECM-114586-114589	
Dow Preliminary Report (ASTM D-5338)	ECM-112176	
Environmental Technology Verification (ETV) Report	ECM-112391-112408	
FT-IR Raw Data	ECM-114000	
Northeast Laboratories: Analytical report - ASTM D 5511	ECM-113084-113087	Northeast Laboratories
Masternet LTD: High Solids Anaerobic Digestion of Plastic Netting OWS Test	ECM-113608-113625	Lies Debeer
Microtech Research Inc.: Test results 1998	ECM-111823-111842	Microtech Research Inc.
Microtech Research Inc.: Report of Data 1999	ECM-111738-111747	Microtech Research Inc.
Microtech research Inc.: Aerobic Bucket - Scale Composting - prepared by O.W.S. Inc.	ECM-111884-111888	Microtech Research Inc.
O.W.S. Inc.: Final Report - Biodegradation Testing	ECM-111843-111883	O.W.S. Inc.
O.W.S. Inc.: Final Report - Biodegradation Testing for 5% load Film	ECM-112026-112079	O.W.S. Inc.
O.W.S. Inc.: Final Report - Biodegradation Testing for ECM Pellets and EMC Film	ECM-112080-112131	O.W.S. Inc.
O.W.S. Inc.: Final Report Infared Analysis of 40-gallon trash bags	ECM-112216-112219	O.W.S. Inc.
O.W.S. Inc: High Solids Anaerobic Digestion Test of ECM Pellets	ECM-112008-112025	O.W.S. Inc.
O.W.S. Inc: Executive Summary - Toxicity Test of ECM Pellet	ECM-111891-112007	O.W.S. Inc.
Shields Bag and Printing: ASTM D5511-11 Update	ECM-114416-114417	Eden Laboratory
Shields Bag and Printing: ASTM D5511-11 Report	ECM-113977-113983	Eden Laboratory
Shields Bag and Printing: High Solids Anaerobic Degradation Test (HSAD) of Film sampl	ECM-114219-114233	O.W.S. Inc.
Email: from L. Debeer (O.W.S.Inc.) to S. McGregor (Shields Bag and Printing) RE: SMG-1 - Anaerobic Biodegradation test – Film	ECM-114418-114427	
Sondor: Bio Foam Degradation Test	ECM-114058-114080	
Sweet Tape Enterprise: ASTM D551 Extension Test	ECM-114353-114361	Northeast Laboratories
Sweet Tape Enterprise: ASTM D551 Extension Test (30 Day)		Northeast Laboratories
Transilwrap: ASTM D5511 Report	ECM-112244-112247	Northeast Laboratories
Tycoplas: ASTM D5511 Analytical Report	ECM-114412-114414	Northeast Laboratories
Final Report: Compostability Testing	3M-FTC00001060000133	3M
Fellows: ASTM D5511 Testing Update		Eden Laboratory
FP International: ASTM DXXXX Update for FPI PE Samples (290 Days)		Eden Laboratory
Shields Bag and Printing: ASTM D5511-11 Update on Clear Films (45 days		Eden Laboratory
Smithers Oasis: ASTM D5511-12 Test 148 days		Eden Laboratory
Barlaz (NCSU): Biodegradability Powerpoint		Morton A. Barlaz
Test of Biodegradable Materials under simulated Landfill Conditions		Morton A. Barlaz
Barlaz (NCSU): FP International - summary of analysis for samples provide		Morton A. Barlaz

Barlaz (NCSU): FP International - summary of analysis for samples provided

Barlaz (NCSU): StarchTech - summary of analysis for samples provided

Biochemical Methane Potential Protocol
Barlaz (NCSU): FP International - Request for testing using BMP Protoco
Environ Tests (4 months): Samples from FP International biodegradation study
Environ Tests (4 months): Picture of diodegradation (CONC_Ae140)
Environ Tests (4 months): Copy of 4 month Interim Analysis
Environ Tests (4 months): Picture of diodegradation (COTM_Ae146)

Environ Tests (4 months): Biodegradation Testing Interim Test Report with Analysis

Environ Tests (4 months): Preliminary results from the FP International Long-term Biodegration Study
Environ Tests (4 months): Picture of diodegradation (PNC_Ae188)
Environ Tests (4 months): Picture of diodegradation (PTM_Ae075)
Environ Tests (4 months): Picture of diodegradation (PTM_An103)
Environ Tests (8 months): Biodegradation Testing Interm Test report
Environ Tests (8 months): SEM Photo Example
Environ Tests (8 months): Pictures of Test Material vs. Negative Control
Environ Tests (12 Months): Biodegradation Testing Interm Test report

Environ Tests (12 Months): Biodegradation Testing Interm Test report preliminary results

Environ Tests (24 Months): Biodegradation Testing Preliminary Final Report
Environ Tests: Final Report prepared for FP International
Environ Tests (4 months): Biodegradation Testing Interim Test Report
Environ Tests (8 months): Pictures of Test Material vs. Negative Control
Environ Tests: Samples after 4 month exposure
Anaerobic Biodegradation of BioPVC Final Report prepared for BIOPVC -

Environ Tests (12 Months): Biodegradation Testing Interm Test report preliminary results

FP Loose- Fill Biodegradation Pictures

Email: from J. Blood () to: R. Alire() RE: Summary of Testing of FP International Products

FP International: FPI Samples (120 days
FP International: FPI Samples (977 days
FP International: FPI Samples (55 days
FP International: ASTM DXXXX Update for FPI EPS Samples (117 Days) Update
FP International: ASTM DXXXX Update for FPI PE Samples (171 Days) Update
FP International: ASTM DXXXX Update for FPI EPS Samples (172 Days) Update
FP International:ASTM DXXXX Update for FPI PE Samples (290 Days) Update
FP International: ASTM DXXXX Update for FPI EPS Samples (291 Days) Update
NorthEast: ASTM D5511 Analytical report - prepared for Dansko, Inc.
NorthEast: MiniGrip 15 day test report
NorthEast: MiniGrip 30 day test report
NorthEast: MiniGrip 45 day test report
NorthEast: MiniGrip 60 day test report
NorthEast: MiniGrip 75 day test report
NorthEast: MiniGrip 90 day test report
NorthEast: MiniGrip 105 day test report

Morton A. Barlaz

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NorthEast: MiniGrip 120 day test report	Northeast Laboratories, Inc.
NorthEast: MiniGrip 135 day test report	Northeast Laboratories, Inc.
NorthEast: MiniGrip 150 day test report	Northeast Laboratories, Inc.
NorthEast: MiniGrip 165 day test report	Northeast Laboratories, Inc.
NorthEast: MiniGrip 180 day test report	Northeast Laboratories, Inc.
NorthEast: MiniGrip 195 day test report	Northeast Laboratories, Inc.
NorthEast: MiniGrip 210 day test report	Northeast Laboratories, Inc.
NorthEast: MiniGrip 225 day test report	Northeast Laboratories, Inc.
NorthEast: MiniGrip 240 day test report	Northeast Laboratories, Inc.
NorthEast: MiniGrip 255 day test report	Northeast Laboratories, Inc.
NorthEast: MiniGrip 270 day test report	Northeast Laboratories, Inc.
NorthEast: MiniGrip 285 day test report	Northeast Laboratories, Inc.
NorthEast: MiniGrip 300 day test report	Northeast Laboratories, Inc.
NorthEast: MiniGrip 300 day test report	Northeast Laboratories, Inc.
NorthEast: MiniGrip 315 day test report	Northeast Laboratories, Inc.
NorthEast: MiniGrip 330 day test report	Northeast Laboratories, Inc.
NorthEast: MiniGrip 345 day test report	Northeast Laboratories, Inc.
NorthEast: MiniGrip 365 day test report	Northeast Laboratories, Inc.
NorthEast: Minigrip Certificate of Analysis	Northeast Laboratories, Inc.
Email: from: A. Mithal (WNA) to: R. Sinclair RE: naerobic Degradation Testing of ECM Additive	
Plastics Color Corp: ASTM Preliminary Test	Soil Control Lab

Exhibit B

Thabet M. Tolaymat Ph.D.

Tolaymat.thabet@epa.gov
26 West Martin Luther King Drive
Cincinnati Ohio, 45230
513-457-2860

EDUCATION

Doctorate of Philosophy in Environmental Engineering Sciences
University of Florida Gainesville, FL 2003
Masters of Engineering in Environmental Engineering Sciences
University of Florida Gainesville, FL 1997
Bachelor of Science in Environmental Engineering
University of Florida Gainesville, FL 1995

EMPLOYMENT

Interim Associate National Program Director
USEPA/ORD Cincinnati, OH 2012- October 2013

- Assist the national program director in the areas of emerging materials (e.g., nanomaterials) and sustainability. Responsible for setting research priority and providing resources to complete the research.

Environmental Engineer
USEPA/ORD Cincinnati, OH September 2004-Present

- Project Lead for Solid Waste Management Systems Research
- Project Lead for Nanomaterials Research
- ORD Project Lead under Safe and Healthy Community Strategic Research Plan for Energy from Solid Wastes and Construction and Demolition Debris. Work in the Solid Waste Branch and conduct research in the area of solid waste and nanomaterials and assisting EPA HQ and Regional offices in the following areas:
    - Performance of Solid Waste Containment Units (municipal solid waste, hazardous waste and ash mono-fill landfills)
    - Bioreactor Landfills. New landfill design that promotes the degradation and subsequent removal of degradable fraction of solid waste as well as organic pollutants.
        - Subtitle D "dry tomb" lined landfill
        - Remediation of contaminated landfill at superfund sites
    - Co-disposal of solid waste and hazardous waste
    - Construction and demolition waste
    - Metal release, mobility from contaminated wastes
- ORD Project Lead under Chemical Safety and Sustainability Strategic Research Plan for Nanomaterials. Leading EPA's Office of Research and Development National Risk Management Research Laboratory in the area of silver nanoparticles.
    - Evaluation of the impacts of nanoparticles on human health and the environments.
    - The evaluation of the impact of environmental conditions on the fate and transport of silver nanoparticles.
    - The evaluation of the impact of silver nanoparticles use on waste management systems

Research Scientist (Federal Post-Doc)
USEPA/ORD Cincinnati, OH *December 2003-September 2004*

- Conduct Research on bioreactor technology. Research includes gathering, assimilating and assessing data gathered at the Outer Loop landfill. Coordinate EPA/ORD efforts with the Solid Waste Association of North America (SWANA) and Interstate Technology Regulatory Transfer (ITRC) to develop and distribute bioreactor landfill guidance document. Conduct research to examine the effectiveness of TCLP to simulate metal mobility in bioreactor landfills.

Graduate Research Assistant (Ph.D. Candidate)
University of Florida Gainesville, FL August 1997-December 2003

- Designed and conducted a battery of tests to evaluate risk associated with the land application of solid wastes. The research shed light on the appropriate use of dilution attenuation factors when assessing risk from ground water contamination. Carried out technical advisory group meetings with regulators (USEPA and FDEP), industry, and the general public. These meetings were designed to increase the understanding between these interested groups and direct the research to benefit the general public.

- Assisted in the design, permitting, and construction of the Polk County Bioreactor Landfill. Coordinated work between the Polk County landfill engineers, landfill operators, the Florida Department of Environmental Protection (FDEP) and fellow graduate students. Addressed design concerns that were raised by FDEP.
- Organized and assisted in evaluating risk from the use and reuse of chromated copper arsenate (CCA) treated wood. Wrote final reports and recommendations for FDEP.

Research Assistant
Applied Environmental Consulting Gainesville, FL May 1996 - August1997

- Coordinated and carried out experiments to evaluate risk associated with the exposure to naturally occurring radioactive material (NORM). Conducted interviews with phosphate workers to evaluate radiation exposure time. Assisted with drafting the final report that was submitted to the Florida Institute of Phosphate.

JOURNAL PUBLICATIONS

1. Jain, P., Powell, J., Smith, J., Townsend, T., **Tolaymat, T.,** (2014) "Life-Cycle Inventory and Impact Evaluation of Mining Municipal Solid Waste Landfills" Environmental Science & Technology 48 (5), 2920-2927
2. Huang, X., El Badawy, A., Arambewela, M., Ford, R., Barlaz, M., **Tolaymat, T.,** (2014) "Characterization of Salt Cake from Secondary Aluminum Production" Journal of Hazardous Materials (273):192-199
3. Ivask, A., El Badawy, A., Kaweeteerawat, C., Boren, D., Fischer, H., Ji, Z., Chang, C., Liu, R., **Tolaymat, T.,** Telesca, D., Zink, J., Cohen, Y., Holden, P., Godwin, H., (2014) "Toxicity Mechanisms in Escherichia coli Vary for Silver Nanoparticles and Differ from Ionic Silver" ACS Nano 8 (1), 374-386
4. Silva, T., Pokhrel, L., Dubey, B., **Tolaymat, T.,** Maier, K., Liu, X., (2014) "Particle Size, Surface Charge and Concentration Dependent Ecotoxicity of Three Organo-Coated Silver Nanoparticles: Comparison Between General Linear Model-Predicted and Observed Toxicity" Science of The Total Environment, (468) 15:968-976,
5. Gitipour, A., El Badawy, A., Arambewela, M., Miller, B., Scheckel, K., Elk, M., Ryu, R., Gomez-Alvarez, V., Santo Domingo, J., Thiel, S., Tolaymat., T. (2013) "The Impact of Silver Nanoparticles on the Composting of Municipal Solid Waste" Environmental Science & Technology 47 (24): 14385-14393
6. Nel etl. al., (2013) "A Multi-Stakeholder Perspective on the Use of Alternative Test Strategies for Nanomaterial Safety Assessment" ACSNano, (7)8:6422-6433.
7. Xu, Q., Powell, J., **Tolaymat, T.,** Townsend, T. (2013). "Seepage Control Strategies at Bioreactor Landfills." J. Hazard. Toxic Radioact. Waste, 17(4), 342–350.
8. **Tolaymat, T.,** Kim, H., Jain, P., Powell, J., and Townsend, T. (2013). "Moisture Addition Requirements for Bioreactor Landfills." J. Hazard. Toxic Radioact. Waste, 17(4), 360–364.
9. El Badawy, A.; Schekel, K.; Suidan, M.; **Tolaymat, T.** (2013) "Key Factors Controlling the Transport of Silver Nanoparticles in Porous Media" Environmental Science and Technology, 2013, 47 (9), 4039–4045.
10. Mwilu, S. K.; El Badawy, A.; Bradham, K.; Thomas, D.; Scheckel, K. G.; **Tolaymat, T**. M.; Ma, L.; Rogers, K. (2013) "Changes in Silver Nanoparticles Exposed to Human Synthetic Stomach Fluid: Effects of Particle Size and Surface Chemistry" Science of the Total Environment, (447): 90-98.
11. **Tolaymat, T.**; El Badawy, A.; Carson, D. (2013) "Estimate of the Decay Rate Constant of Hydrogen Sulfide from Drywall in a Simulated Bench-Scale Study. J. Environ. Eng. (139): 538-544.
12. Kim R. Rogers, K., Bradham, K., **Tolaymat, T.,** Thomas, D., Hartmann, T., Ma, L., Williams, A. (2012) "Alterations in Physical State of Silver Nanoparticles Exposed to Synthetic Human Stomach Fluid" Science of the Total Environment. (420):334-339.
13. Pokhrel, L., Silva, T., Dubey, B., Elbadawy, A., **Tolaymat., T.** (2012) "Rapid Screening of Aquatic Toxicity of Metal-Based Nanoparticles Using the MetPLATE Assay" Science of the Total Environment. (426):414-422.

Exhibit B

14. El Badawy, A., Scheckel, K., Suidan, M., **Tolaymat, T.** (2012) "The Impact of Stabilization Mechanism on the Aggregation Kinetics of Silver Nanoparticles" Science of the Total Environment. (429):325-331.
15. El Badawy, A., Scheckel, K., Suidan, M., **Tolaymat, T.** (2011) "Surface Charge-Dependent Toxicity of Silver Nanoparticles" Environmental Science and Technology (45)1:283-287.
16. Costanza, J., El Badawy, A., **Tolaymat, T.** (2011) "Comment on 120 Years of Nanosilver History: Implications for Policy Makers" Environmental Science and Technology. (45)17:7591-7592
17. Jain, P., Townsend, T., **Tolaymat T.,** (2010) "Steady-State Design of Vertical Wells for Liquids Addition at Bioreactor Landfills" Waste Management. (30)11:2022-2029.
18. Jain, P., Townsend, T., **Tolaymat T.,** (2010) "Steady-State Design of Horizontal Wells for Liquids Addition at Bioreactor Landfills" Waste Management. (30)12:2560-2569.
19. Bareither, C., Benson, C., Barlaz, M., Edil, T., **Tolaymat, T.** (2010) "Performance of North American Bioreactor Landfills. I: Leachate Hydrology and Waste Settlement" J. Environmental Engineering-ASCE. (136)8:824-838
20. Barlaz, M., Bareither, C., Hossain, A., Saquing, J., Mezzari, I., Benson, C., **Tolaymat, T.,** Yazdani, R. (2010) "Performance of North American Bioreactor Landfills. II: Chemical and Biological Characteristics" J. Environmental Engineering-ASCE. (136)8:839-853
21. Genaidy, A., Sequeira, R., **Tolaymat, T.,** Kohler, J., Wallace, S., Rinder, M. (2010) "Integrating Science and Business Models of Sustainability for Environmentally-Challenging Industries such as Secondary Lead Smelters: A Systematic Review and Analysis of Findings". J. Environmental Management. (91)9:1872-1882
22. Musson, S., Campo, P., **Tolaymat, T.,** Suidan, S., Townsend, T., (2010) "Assessment of the Anaerobic Degradation of Six Active Pharmaceutical Ingredients" Science of The Total Environment, (38)9: 2068-2074
23. Jang, Y., Jain, P., **Tolaymat, T.,** Dubey, B., Singh, S., Townsend, T. (2010) "Characterization of Roadway Stormwater System Residuals for Reuse and Disposal Options" Science of the Total Environment. (407)12:3686-3701
24. **Tolaymat, T.,** Green, R.,Hater, G., Barlaz, M., Black, P., Bronson, D., Powell, J. (2010) "Evaluation of Landfill Gas Decay Constant for Municipal Solid Waste Landfills Operated as Bioreactors" J. Air & Waste Management Association. (60)1:91-97
25. **Tolaymat, T**., El Badawy, A., Genaidy, A., Scheckel, K., Luxton, T., Suidan, M. (2010) "An Evidence-Based Environmental Perspective of Manufactured Silver Nanoparticle in Syntheses and Applications: A Systematic Review and Critical Appraisal of Peer-Reviewed Scientific Papers" Science of the Total Environment. (408)5:999-1006
26. Scheckel, K., Luxton, T., El Badawy, A., Impellitteri, C., **Tolaymat, T.** (2010) "Synchrotron Speciation of Silver and Zinc Oxide Nanoparticles Aged in a Kaolin Suspension" Environmental Science and Technology. (44)4:1307-1312
27. El Badawy, A. Luxton, T., Silva, R., Scheckel, K., Suidan, M., **Tolaymat, T.** (2010) "Impact of Environmental Conditions (pH, Ionic Strength, and Electrolyte Type) on the Surface Charge and Aggregation of Silver Nanoparticles Suspensions" Environmental Science and Technology. (44)4:1260-1266
28. **Tolaymat, T.,** Al-Abed, S., Jegadeesan, G. (2009) "Impact of Bioreactor Landfill Leachate Quality on As, Cd, Pb and Zn Leaching from Mine Residues" J Residuals Science & Technology (6)2: 89-96
29. Impellitteri, C., **Tolaymat, T**., Scheckel, K., (2009) "The Speciation of Silver Nanoparticles in Antimicrobial Fabric before and after Exposure to a Hypochlorite/Detergent Solution" J. Environmental Quality. (38) 4:1528-1530
30. Genaidy, A., **Tolaymat, T.,** Sequeira, R. (2009) "Health Effects of Exposure to Carbon Nanofibers: Systematic Review, Critical Appraisal, Meta Analysis and Research to Practice Perspectives" Science of the Total Environment. (407) 12:3686-3701

Exhibit B

31. Genaidy, A., Sequeira, R., **Tolaymat, T.**, Kohler, J., Rinder, M. (2009) "Evidence-Based Integrated Environmental Solutions for Secondary Lead Smelters: Pollution Prevention and Waste Minimization Technologies and Practices" Science of the Total Environment. (407)10:3239-3268
32. Jang, Y., Jain, P., **Tolaymat, T.**, Dubey, B., Townsend, T. (2009) "Characterization of Pollutants in Florida Street Sweepings for Management and Reuse" J. Environmental Management. (91)2:320-327
33. **Tolaymat, T.,** Dubey, B., Townsend, T. (2008) "Assessing risk posed by land application of ash from the combustion of wood and tires" J. Residuals Scinece & Technology. (5) 2:61-75.
34. Jjemba, P., Morris, B., **Tolaymat, T.** (2008) "Specific Energy Output from Urban Residues Degraded with Leachate and an Off-Specification Industrial Carbonated Beverage as Moisture Sources" Biomass and Bioenergy (32)1:51-59
35. Genaidy, A., Sequeira, R., **Tolaymat, T.**, Kohler, J., Rinder, M.(2008) "An Exploratory Study of Lead Recovery in Lead-Acid Battery Lifecycle in US Market: An Evidence-Based Approach" Science of the Total Environment (407)1:7-22
36. Al-Abed, S., Jegadeesan, G., Scheckel, K., **Tolaymat, T.** (2008) "Speciation, Characterization, and Mobility of As, Se, And Hg in Flue Gas Desulphurization Residues" Environmental Science & Technology. (42)5: 1693-1698
37. Townsend, T., Dubey, B., **Tolaymat, T.** (2006). "Interpretation of SPLP Results for Assessing Risk to Groundwater from Land-Applied Granular Waste" Environmental Engineering Science. (23)1:239-251
38. Williams, A., Scheckle, K., **Tolaymat, T.,** Impellitterie, C. (2006)"Mineralogy and Characterization of Arsenic, Iron, and Lead in a Mine Waste-Derived Fertilizer" Environmental Science and Technology. (40)16:4874-4879
39. Stook, K., **Tolaymat, T.,** Ward, M., Dubey, B., Townsend, T., Solo-Gabriele, H., Bitton, B. (2005). "Relative Leaching and Aquatic Toxicity of Pressure-Treated Wood Products Using Batch Leaching Tests." Environmental Science & Technology, 39(1), 155-163.
40. Jain, P., Jang, Y., **Tolaymat, T.,** Witwer, M., Townsend T. (2005)"Recycling of Water Treatment Sludge Via Land Application: Assessment of Risk" J. Residuals Science and Technology 2(1):16-20
41. Townsend, T., Dubey, B., **Tolaymat, T.,** Solo-Gabriele, H. (2005). "Preservative Leaching from Weathered CCA-Treated Wood." J. Environmental Management, 75(2), 105.
42. Townsend, T., **Tolaymat, T.,** Leo, K., Jambeck, J. (2004). "Heavy Metals in Recovered Fines from Construction and Demolition Debris Recycling Facilities in Florida." Science of The Total Environment, 332(1-3)
43. Townsend, T., **Tolaymat, T.** Solo-Gabriele, H., Dubey, B., Stook, K., wadanambi, L.(2004). "Leaching of CCA-Treated Wood: Implications for Waste Disposal." J. Hazardous Materials, 114(1-3), 75.
44. Iida, K., Pierman, J., **Tolaymat, T.,** Townsend, T., Wu, C. (2004)."Control of Heavy Metal Emissions and Leaching from Incineration of CCA-Treated Wood Using Mineral Sorbents." J. Environmental Engineering, ASCE. 1302(2), 184-192.
45. Townsend, T., solo-Gabriele, H., **Tolaymat, T.,** Stook, K., Hosein, N. (2003). "Chromium, Cooper and Arsenic Concentrations in Soil Underneath CCA-Treated Wood Structures." Soil and Sediment Contamination. 12(6), 779-798.
46. Townsend, T., Solo-Gabriele, H., **Tolaymat, T.,** Stook, K. (2003). "Impact of Chromated Copper Arsenate (CCA) in Wood Mulch." The Science of the Total Environment, 309(1-3), 173-185.
47. **Tolaymat, T.,** Townsend, T., Solo-Gabriele, H. (2000) "Chromated Copper Arsenate-Treated Wood in Recovered Wood." Environmental Engineering Science 17(1):19-28.

EPA REPORTS

1. **Tolaymat, T.,** Kremer, F., Carson, D., Davis-Hoover, W. Monitoring Approaches For Bioreactor Landfills. U.S. Environmental Protection Agency, National Risk Management Research Laboratory. Cincinnati, OH EPA/600/R-04/301, 2004.

2. Meer, S., Benson, C., **Tolaymat, T.**, Carson, D. In-Service Hydraulic Conductivity Of GCLs In Landfill Covers - Laboratory And Field Studies. U.S. Environmental Protection Agency, National Risk Management Research Laboratory. Cincinnati, OH, EPA/600/R-05/148, 2005.
3. **Tolaymat, T.** Landfill Bioreactor Performance, Second Interim Report. U.S. Environmental Protection Agency, National Risk Management Research Laboratory. Cincinnati, OH, EPA/600/R-07/060, 2007.
4. Peggs, I., **Tolaymat, T.** Non-Destructive Evaluation of Geomembrane Seam Bond Strength. U.S. Environmental Protection Agency, National Risk Management Research Laboratory. Cincinnati, OH, EPA/600/R-09/070, 2009.
5. Benson, C., Barlaz, M., **Tolaymat, T.** Bioreactor Landfills State-Of-The Practice Review. U.S. Environmental Protection Agency, National Risk Management Research Laboratory. Cincinnati, OH, EPA/600/R-09/071, 2009.

ADJUNCT APPOINTMENTS/CONSULTANCY

- Adjunct professor at East Tennessee State University
- Executive board member at the Florida Center for Solid and Hazardous Waste Management
- Solid waste expert witness for Ohio EPA, 2006
- Solid waste consultant to the World Bank
- Solid waste consultant to USAID

AWARDS

- U.S. **EPA Science Achievement Award** in 2010 for service at the Salt River Regional Landfill awarded by EPA
- U.S. EPA **Bronze Medal** for Commendable Service in 2007 for service after hurricane Katrina awarded by EPA
- U.S. EPA **Bronze Medal** for Commendable Service in 2010 for service at the Fort Deveins Superfund Site awarded by ORD
- U.S. EPA **Bronze Medal** for Commendable Service in 2010 for service at the County Wide Landfill awarded by Region 5
- Level III Scientific and Technological Achievement Award 2007
- U.S. EPA/ORD **Superior Accomplishment** award every year between 2004 and 2010

INVITED PRESENTATION AND WORKSHOPS

- Developed and taught the Interstate Technology Regulatory Council (ITRC) bioreactor landfill internet training course in 2006
- Develop web content about bioreactor landfills in 2007 with the Hinkley Center for Solid and Hazardous Waste Management and the University of Florida (see http://www.bioreactor.org)
- Develop 2-day workshops on bioreactor landfills to for EPA Regions 5, 6, and 7 between 2005 and 2006
- Develop and coordinate bioreactor landfill section of Solid Waste Association of North America (SWANA) symposium in 2007.
- Developed a bioreactor landfill workshop for the World Bank October, 2007.
- Invited by Taiwan Environmental Protection Agency to organize and host an international workshop in 2005 and 2008.
- Environmental Factors and Surface Properties of Nanoparticles Governing Their Fate, Reactivity, and Mobility. Presented at 10th International Conference on the Biogeochemistry of Trace Elements, Chihuahua, MEXICO, July 13 - 18, 2009
- Monitoring Guidance for Bioreactor Landfills. Presented at SWANA Bioreactor Meeting, San Antonio, TX, March 22 - 25, 2005.
- Mercury Speciation In FGD: Assessing Transport And Bioavailability Risk. Presented at Research and Demonstration of Agricultural Uses of Gypsum and Other FGD Materials Workshop, St. Louis, MO, September 12 - 14, 2006.
- Metal Speciation in Soil, Sediment, and Water Systems Via Synchrotron Radiation Research. Presented at EPA Science Forum, Washington, DC, May 16 - 18, 2005.

Exhibit B

- Bioreactor Landfill Design. Presented at EPA Region 5 Bioreactor Landfill Workshop, Chicago, IL, September 27, 2005.
- Monitoring Approaches for Bioreactor Landfills. Presented at EPA Region 5 Bioreactor Landfill Workshop, Chicago, IL, September 27, 2005.
- Bioreactor Landfills, Theoretical Advantages And Research Challenges. Presented at EPA Region 5 Bioreactor Landfill Workshop, Chicago, IL, September 27, 2005.
- Waste Stabilization Fundamentals For Bioreactor Landfills. Presented at EPA Region 5 Bioreactor Landfill Workshop, Chicago, IL, September 27, 2005.

EXTERNAL RESEARCH GRANTS

- Waste Management Inc. (WM) bioreactor landfill $200K in-kind per year (2001-present)
- EPA's Office of Solid Waste (OSW) "Fee for Service" $150K (2005)
- ORD's national nanomaterials research initiative $480K (2007)
- Environmental Education and Research Foundation bioreactor landfill research $40K (2007)
- Environmental Education and Research Foundation bioreactor landfill research $40K (2007)
- Regional Applied Research Effort (RARE) R5 $50k per year for two years (2008-2009)
- Regional Applied Research Effort (RARE) R10 $110k (2008)
- Environmental Research and Education Foundation secondary aluminum processing waste research $186K (2009)
- Aluminum Association secondary aluminum processing waste research $186 (2009)
- U.S. AID/Jordan the remediation of a phosphate mining site in Amman, Jordan $5,000K (2010)

Respondent's Exhibit Y

STEVEN J. GROSSMAN

EDUCATIONAL BACKGROUND

J.D. Degree (Law) Franklin Pierce Law Center, Concord, New Hampshire, January 1990.

Ph.D in Polymer Science and Engineering, University of Massachusetts, Amherst,Massachusetts, 1979. Full Research Assistantship, Thesis Topic: "Synthesis and Characterization of Novel Polymer Structures: 1. Head-to-Head Polypropylene; 2. Regular Gopolyoxamides." Thesis Advisor; Professor Otto Vogl, former Herman F. Mark Professor of Polymer Science at the Polytechnic Institute of New York. The Polymer Science and Engineering Department at the University of Massachusetts, Amherst. is recognized by industry and academic institutions as one of the world's leading centers for polymer education and research.

B.S. in Chemistry, University of Connecticut, Storrs Connecticut, 1975.

PROFESSIONAL ACCREDITATION

Member, New Hampshire Bar Association - admitted, licensed attorney at law – June 1990
Member, Federal District of New Hampshire, admitted, July, 1990.
United States Patent Office (USPTO)-Admitted To Practice-1991
Court of Appeals for the Federal Circuit -1994

EMPLOYMENT HISTORY:

A. Academia: 1984-Present

Full Professor

Plastics Engineering Department, University of Lowell, 1992 to present.

Associate Professor

Plastics Engineering Department, University of Lowell, 1987-1992

Assistant Professor

Plastics Engineering Department, University of Lowell, 1984-1987.

Lecturer

Plastic Seminars, Continuing Education, University of Lowell, 1985-1995.

Guest Lecturer

Franklin Pierce Law Center, Spring 2014
Franklin Pierce Law Center, Spring 2013
Franklin Pierce Law Center, Spring 2007
Eastern New England Society of Plastics Engineers, October 2013
Eastern New England Society of Plastics Engineer, October 2003
Eastern New England Society of Plastics Engineers, October 1999
Eastern New England Society of Plastics Engineers, October 1997
Rexene Polymers, October 1996
Amoco, Napersville Illinois, May 1996, 1997 and 1998 and 2002
Polysar Incorporated, Leominster Massachusetts, Summer, 1988.
Anglo-American Clays Corporation, Sandersville, Georgia, Summer, 1986.
Army Mechanics and Materials Research Laboratories, Watertown, 1985.
Eastern New England Section of the Society of Plastics Engineers, , 1985.

B. Work Experience

Partner, Grossman, Tucker, Perreault & Pfleger, PLLC, 2002-present

Intellectual property including patents, trademarks, copyrights, and trade secret protection. Patent prosecution and opinion work, including licensing. Litigation support is also provided by way of expert testimony in the area of polymer chemistry and plastic materials/plastics engineering. See: www.gtpp.com.

Hayes, Soloway. Hennessey. Grossman & Hage, P.C., 1989 -2002

Intellectual property including patents, trademarks, copyrights, and trade secret protection. Litigation support was also provided by way of expert testimony in the area of polymer chemistry and plastic materials/plastics engineering.

Research and Development Scientist, The Upjohn Company (1980-1984)

This industrial research position was in the Polymer Research Group of the Upjohn Company. Initial work was conducted on the preparation and characterization of aromatic amine based polyamides (engineering thermoplastics). Research continued in the area of preparation of polyamides via the novel reaction of isocyanates with dicarboxylic acids. These systems were further analyzed in the Department of Plastics Engineering at the University of Lowell as strong candidates for reactive polymer processing.

Both MS. and Ph.D. thesis have evolved from these studies. Further work at the Upjohn Company led to monomers that polymerized to condensation type polymers (e.g., polyamides) without the evolution of by-products. Additionally, research was directed at the development of new nylon-6 reaction injection molding systems.

A total of 4 U.S. Patents and 1 European Patent was filed and issued during the above period of industrial research.

ACADEMIC PROFILE

A. Teaching

1. *Area of Courses Taught (1994-2014)*

Polymer Materials I
Polymer Materials II
Polymer Materials III
Plastic Material Science
Survey of Plastic Materials 1
Survey of Plastic Materials 11
Plastics Seminar I
Plastics Seminar II
Graduate Seminar I
Graduate Seminar II
Survey of Intellectual Property (For Engineers)
New Plastic Materials
Polymer Structure/Properties/Applications

Survey of lntellectual Property was a general elective course for engineering students developed and offered for the first time in the Fall of 1990. This course allowed engineers to fully appreciate the legal issues that one may be forced to consider in order to invoke the legal system to protect technology based innovation. Once legal protection is established, students consider what. e.g., constitutes patent infringement, and what constitutes an unfair trade practices, etc. This course has now been approved (2004) as part of a business minor for students in the Department of Plastics Engineering.

The Polymer Materials Courses have provided an intensive study of the many generic classes of polymer materials that are in current production. These courses have been structured to include reviews of important raw materials; polymerization theory and practice of manufacture (which prepares the students more efficiently for higher level courses in polymer science and polymer structure/property and applications); correlation of polymer structure with engineering properties; discussions of polyblends with emphasis of related polymer chemistry (compatibility); inter and intramolecular bonding and fundamental processing considerations.

The Plastics Materials Science Courses were designed to provide the Industrial Technology student the best possible one semester review of plastics engineering. A discussion of the historical development of polymer materials was followed by a review of the major methods of materials characterization and testing. Mechanical properties of polymer materials are studied and methods to predict material performance is related to the students. Finally, a survey of the prominent resin families is included with a review of end-use applications with respect to pricing, processing and performance. This course is no longer offered.

The Plastics Engineering Seminar Courses were taught exclusively at the graduate level. These courses emphasized techniques of literature survey in plastics engineering along with effective methods of presentation. Students are challenged to develop research proposals in order to optimize commercial potential of recent technology.

Polyurethanes (University of Lowell, Continuing Education) reviewed the principles involved in the synthesis of polyurethanes and how these principles are being applied in industry. Product manufacture was reviewed (flexible and rigid foams, elastomers and coatings), the solving of problems relating to the development of improved products and new trends in formulations and polyurethane processing equipment.

Fundamentals of Plastics/Commercial Polymer Materials (University of Lowell, Continuing Education) reviewed, in a 2-3 day format, the different plastics, processes and applications of polymer materials. Basic concepts in plastics materials were reviewed which served as a foundation for a discussion of each commercial material in terms of monomer synthesis, polymerization, post-reaction treatment, large-scale production, additives, structure-property relationships, processing and applications. The course recognizes different materials as being chemically distinct arid explains actual physical properties on the basis of polymer structure.

2. **Graduate** Thesis **Supervised** (Completed):

a. Thesis Advisor (Completed)

"Plastic Materials Withdrawn From The Market", T. Richards (Fall 2004)

"Blends of LDPE with Flexible Polyolefins, M. Castillo (Spring 1999)

"Effect of Drying Conditions/Particle Size on the Recycling of PET Resins" December 1998 [M. Yurtseven]

"Effects of Reactive Processing on Polystyrene/Hyperbranched Polyol Blends", M.S. Thesis, September 1998, [Thomas I. Mulkern II]

"Recycling of Nylon-6 and Polypropylene via Reactive Polymer Processing", M.S. Thesis, August 1990, Achal Takar

"Jeffamine Plasticizers for Nylon-6,6" MS. Thesis, 1989, Arnbarish Dave

"Kinetic Studies of the Polymerization and Crystallization of Nylon Block Copolymers", Ph.D Thesis, August 1988, Tim Limtasiri, (Co-advisor with Dr. Jan-Chan Huang.)

"Synthesis of Aromatic Amine Based Polyamides from Diisocyanates and Dicarboxylic Acids: Mechanism of Polymerization and Applications for Reactive Polymer Processing", Ph.D Thesis, January 1987 (Victor Wang].

"Recent Developments in Plastics Recycling and Biodegradable Materials", MS. Thesis, January 1989, Rajen Parikh

"Polymer Blends of Recycled Poly(ethylene terepthalate) and Polypropylene with Coupling Agents", MS. Thesis, May 1988, Chong Oh Kim

"Effect of Annealing on the Moisture Absorption of Nylon-6,6 Materials", M.S. Thesis, May 1988, Chio-Chiang Pai

"Evaluation of Sorbitol as a Plasticizer for Nylon-6,6 and Reactive Processing of Nylon-6,6 with Reactive Polystyrene", MS. Thesis, April 1988, Pradleep Damle

"Preliminary Studies on Reactive Polymer Processing for the Purpose of Preparing Aromatic-Amine Based Polyarnides from Disocyanates and Dicarboxylic Acids", M.S. Thesis, February 1988, Yu-PengChen

"Thermoplastic Elastomers by Transesterification of Recycled PET and Polyethylene Glycol, M.S. Thesis, August 1987, Da-Vei Yui

"One-Step vs. Two Step Nylon Block Copolymerization", MS. Thesis, May 1987, Victor Brandalise

"Polyethylene Glycol, Polypropylene Glycol as Plasticizers for Nylon-6,6", M.S. Thesis, August 1986, Cheng-Twig Huang

"Polymer Blends of Recycled Poly(ethylene terepthalate) and Recycled Polycarbonate, M.S. Thesis, April 1986, Donald Roche

"Recycling of Styrene Polymers and Its Effect on theft Properties and Processing", M.S. Thesis, January 1986, Shreyms Morakhia

'DSC Studies for the Evaluation of Polymerization and Crystallization of Nylon-RIM Systems", M.S. Thesis, August 1985, Seng-Shiu Chung

b. Thesis Committee Member

Analysis of the Dip-Coating of Solid and Fibrous Substrates", Ph.D Thesis, June 1986, Amit Dharia

'Structure-Property Correlations in flexible Main Chain Polymeric Liquid Crystals", Ph.D Thesis, August 1986, S. Kwnar

"The Effects of Ionic Interactions on the Thennal-Mechanical Properties of Methacrylic Ampholytic lonomers and their Blends", Ph.D Thesis, May 1986, Christian M. Neculescu

"The Effect of Uterine and Vaginal fluid Constituents on the Structure, Properties and Physical Breakdown of Nylon-6", Ph.D Thesis, 1985, J. Hudson

"Viscosity of Water Soluble Polymers". M.S. Thesis, January 1989 Tsai-Jucong Liul

Rheology Study of Kraton Solutions", MS. Thesis, December 1988, Ru-Jong Jeng

"The Use of Three-Dimensional Solubility Parameter Methods to Help Predict Practical Polymer-Polymer Compatibility" M.S. Thesis, December 1986, Henry Kozlowski

"Binders for Recycled Automobile Scrap Light Fluff Fraction", MS. Thesis, September 1986 Ajay Kharodj.

"Bacterial Breakdown and/or Grow-Through Nylon-6 Film", M.S. Thesis, September 1986, Sharon Sylvester

"Theoretical and Practical Consideration of Rubber-to-Metal Bonding", MS. Thesis, August 1986, David Butler

"Reliability of Selected Marketing Projections", MS. Thesis, May 1986 (David Gorky].

"Recycling of Epoxy Resins", M.S. Thesis. May 1986. Conchita Tran-Bruni

"Polyblends of Thermoplastic Polyurethane with Acrylonitrile-Butadiene Styrene", MS. Thesis, April 1986, Ah Monochebri

"Recycling of Mixed Plastics Fraction from Junked Autos by Addition of Thermosetting Resins and Low Pressure Molding", M.S. Thesis, January 1986, Chaitanya Nadicami

"Modification of Polystyrene by Styrene/Butadiene/Styrene Thermoplastic Elastorner", M.S.

Thesis, July 1985, Jiko Chang

"Adhesives Based on Starch Graft Copolymers", M.S. Thesis, April 1985, John Martin

"Thermoset Polymers for Electronic Applications", MS. Thesis, April 1985, Philip Papoojian

"An Investigation of the Mechanical Properties and Polyblend Compatibility of Nylon-6,6 with Sodium and Zinc Based Ethylene lonomers", MS. Thesis, April 1985, Jitendra Jherwar

"Effect of Fiber Orientation on Mechanical Properties of Weld Lines", MS. Thesis, March 1985, Nitin Matini

Summarizing the above indicates the following:

-Thesis Advisor -Completed: 2 Ph.D.'s, 18 M.S.

-Thesis Comniittee Member: 4 Ph.D's, 16 M.S.

B. Research and Publications

I. Grants and Contracts (1984-1992)

a. Representative *Research Grants*	*Amount($)*
Phillips Driscopipe, Inc.	89,800*
Monsanto	50,000"
Anglo-American Clays	42,650
Gates Formed Fibre Products Inc.	26,700'
Dennison Manufacturing	23,704
Petroleum Research Fund-ACS	15,000
A.E. Staley Manufacturing	7,350

Total: $284,804.00

b. *Research and Testing Contracts*

A variety of small research/testing type contracts have been supervised in the Plastics Engineering Department. These are administered by the Testing Division of the Research Foundation.

c. *Plastic Seminar Activities*

The development of the seminar courses offered through the Department of Continuing Education (initiated in the Spring of 1985) have led to a total enrollment in excess of 200 students with a gross income of approximately $275,000.00. The income from these seminars is distributed in a manner that is similar to income from grants and contracts. Namely, a portion is utilized for salary, income or the Continuing Education Office, income for the Plastics Engineering Department,

d. Awards

Spring 2003: Plastics Engineering Department Teaching Award – award given to the outstanding faculty member as voted on by the students in the Department of Plastics Engineering

During the Spring Semester of 1988 received a $750.00 Faculty Scholarship through the office of President Hogan as support in pursuit of my J.D. degree with a concentration in patent law/intellectual property.

During the Merit Evaluation period, April 1, 1985 through March 31, 1986, I received a $2000.00 award for Professional Development from the College of Engineering.

During the Merit Evaluation period, April 1, 1986 through March 31, 1987, I received a $750.00 award for Professional Development from the College of Engineering.

2. Text Materials, Publications, Conference Papers, Patents, Presentations, Research Reports and Thesis:

a. *Text Materials*

S. J. Grossman and 0. Vogl, "Head-to-Head Polymers", Encyclopedia of Polymer Science and Technology, John Wiley Interscience (1987). [The Encyclopedia of Polymer Science and Engineering is a collection of extensive reviews by the leading authorities in the field of polymer science Many consider it as the primary reference for collecting information on advanced research and the status of current technology.]

b. *Publications*

S. Grossman & B. Filip – *The American Inventors Act: Six Key Changes In U.S. Patent Law*, 2012 SPE-ANTEC

S. Grossman & B. Filip – *Recent Procedures Introduced To Challenge Patent Grants At the United States Patent Office*, 2011 SPE-ANTEC

S. Grossman, B. Filip & J. Placker, *Ten Things Plastics Engineers Should Know About Intellectual Property*, 2005 SPE-ANTEC.

S.J. Grossman and C. Gagne. "Science and Scientific Evidence", University of Connecticut Law Review, (1993).

S.J. Grossman, "Science and Scientific Evidence", IDEA-The Journal of Law and Technology, 32(2), 179(1992).

S.J. Grossman and Achal Takar, "Reactive Processing of Nylon-6 and Polypropylene Resins, 1991 SPE-ANTEC

S.J. Grossman, "Experimental Use Or Fair Use As A Defense To Patent Infringement", IDEA The Journal of Law and Technology, 30(3), 243(1990).

C. Pai, S. S. Grossman and J. C. Huang, "Effects of Moisture on Thermal and Mechanical Properties of Nylon-6,6". Submitted to Advances in Polymer Technology (1989).

T. Limtasiri, S. J. Grossman and J. C. Huang, "Kinetic Studies of the Crystallization of Nylon-6 Block Copolymers", Poly. Eng. Sci., Accepted, in-press.

T. Limtasiri, S. J. Grossman and C. C. Huang, "Kinetic Studies of the Polymerization of Nylon-6 Block Copolymers", Poly. Eng. Science., 28,1145(1988).

S. Shah, S.F. Wang, N. Schott and S. *J.* Grossman, "Counter Rotating Twin Screw Extruder As A Devolatizer and As A Continuous Polymer Reactor, SPE-ANTEC,.45, 122 (1987).

M. Huang and S.J. Grossman, "Ethylene Glycol, Polyethylene Glycol and Polypropylene Glycol as Plasticizers for Nylon-6,6", SPE-ANIEC, 45,1284(1987)

A. Dharia and S.J. Grossman, "Variation of the Heat Distortion Temperature of Nylon-6 With Changing Water Content, SPE-ANTEC 45490(1987).

S.F. Wang and S. J. Grossman, "Synthesis of Polyamides from Aromatic Diisocyanates and Dicarboxylic Acids", Polymer Preprints, 28(1), 264(1987).

S. F. Wang and *S.J.*. Grossman, "Plastic Materials for Insulating Applications", *3.* Chem. Ed., 64(1), 39(1987).

R. Jabba, S. Chung and S. J. Grossman, "Morphological Characteristics of Nylon-RIM

Materials", SPE-ANTEC, 685(1986).

S.S. Chung and *S.J.*. Grossman, "DSC Studies of Polymerization and Crystallization of Nylon-RIM Materials-Il", Polymer Preprints, 54(1), 629(1986).

S. S. Chung and S.J.. Grossman, "DSC Studies of Polymerization and Crystallization of Nylon-RIM Materials-Polymer Preprints, 54(1), 623(1986).

S. Grossman, A. Stolarezyk and 0. Vogl, "Head-to-Head Polymers XVII. Head-to-Head Polypropylene", Monatshefte Fur Chemie, 112(11), 1279(1981).

S. Grossman. A. Yamada and 0. Vogl, "Head-to-Head Polymers XVI. Cis and Trans-l,4-Poly(2,3-dimethylbutadiene)", Journal of Macromolecular Science-Chemistry, A16 (4), 897 (1981).

A. Yamada, S. Grossman and 0. Vogl, "Head-to-Head Polymers XV. A Facile Synthesis of 2,3-Disubstituted Succinates and Butanediols-1,4, Journal of Polymer Science, Polymer Chemistry Edition, 18,1739(1980).

W. Deits, S.J. Grossman and 0. Vogl, "Novel Preparation of Specialty Polyamides by Interfacial and Solution Methods", Journal of Macromolecular Science-Chemistry, MS *(5),* 1027 (1981).

S. Grossman, D. Tirrell and 0. Vogl, "Regular Copolyoxamides VII. Water Binding in Regular Copolyoxamide Membranes", ACS Symposium Series, 127,364(1980).

D. Tirrell, S.J. Grossman and 0. Vogl, "Regular Copolyoxarnides VII. Water Absorption in Regular Copolyoxamides", Die Maicromolekulare Chemie, 180,721(1979).

0. Vogl, C.D. Dudgeon,, W. Deits, S. Grossman and D. Tirrell, "Functional Condensation Polymers and Copolyoxamides", Polymer Preprints, 19(2), 75(1978).

A, Monahan, S. Grossman et. at, "A New Synthesis of Indenones", Synthetic Communications, 7(8), 553 (1977)

c. *Patents*

S. Dai, S. Grossman and K. Onder, Polymers From Azetidine-2,4-Dione And Polyol or Polyamine, U.S. Patent 4,604,450 (1986)

S.Dai, S. Grossman and K. Onder, "Polyamnide Block Copolymer From Lactam And Azetidine-2,4-Dione", U.S. Patent 4,550,157 (1985)

S. Grossman and K. Onder, "Anionic Polymerization of Lactam With Azetidine-2,4-Diane Promoter", U.S. Patent 4,550,157 (1985)

S.Grossman and K. Onder, "Copolyamide-Inmides Based on MDI, TMA and Two Dicarboxylic Acids", U.S. Patent 4,467,083 (1984)

d. *Papers Presented at Meetings*

SPE-ANTEC 2012 (Orlando) The America Invents Act: Six Key Changes in U.S. Patent Law

SPE-ANTEC 2011 (Boston) Recent Procedures Introduced To Challenge Patent Grants At The USPTO

Eastern New England Society of Plastics Engineers, October 2005 – "Ten Things Plastics Engineers Should Know About Intellectual Property"

Eastern New England Society of Plastics Engineers, October 2003, "Recent Developments in Intellectual Propety", Massachusetts.

Eastern New England Society of Plastics Engineers, October 2001, "Recent Developments In Intellectual Property, Massachusetts.

M. Huang and S.J. Grossman, "Ethylene Glycol, Polyethylene Glycol and Polypropylene Glycol as Plasticizers for Nylon-6,6", presented at the 1987 SPE-ANTEC in Los Angeles.

S. B. Driscoll and S.J. Grossman, "Polymer Materials in the Undergraduate Program", presented at the Spring Meeting of the American Chemical Society, New York, April 13-18th (1986).

S.S. Chung and S.J. Grossman, "DSC Studies of Polymerization and Crystallization of Nylon-RIM Materials-P', presented at the Spring Meeting of the American Chemical Society, New York April 13-18th (1986).

S.S. Chung and S. J. Grossman, "DSC Studies of Polymerization and Crystallization of Nylon-RIM Materials-I ", presented at the Spring Meeting of the American Chemical Society, New York, April 13-18th (1986).

S. Grossman, P.S. Andrews and K. Onder, "Synthesis of Polyamides from Aromatic Dusocyanates and Dicarboxylic Acids", presented at the International Union of Pure and Applied Chemistry-28th Macromolecular Symposium-Amherst, Massachusetts (1982).

S. Grossman, "Synthesis of Polyamides from 4-4'~Methylene-Bis(isocyanatobenzene), Poster presentation at the 1981 Gordon Research Conference on Polymers.

S. Grossman and 0. Vogl, "Regular Copolyamides by Solution and Liquid-Liquid Interfacial Polymerization, presented at the 179th ACS National Meeting, Houston, Texas (1980).

S. Grossman, D. Tirrell and 0. Vogl, "Water Absorption in Regular Copolyoxamides", presented at the 8th Northeast Regional Meeting. American Chemical Society, Boston, Massachusetts, (1978)

e. Research Reports

"Protocol, Cardiothane Production, Chemical and Physical Test Series", Kontron Corporation, Everett, Massachusetts, October (1987). This report was made in connection with Kontron's efforts to satisfy Food and Drug Administration quality control standards for the production of polyurethane materials for medical applications.

"Reactive Processing and Compatibilization of PET/PP Mixtures", submitted to Gates Formed-Fibre Products, Summer (1987).

"New Processing Aids for PVC-Evaluation of Reactive Cellulose Esters", submitted to A.E. Staley Manufacturing, Decatur, Illinois (1986).

"Program to Improve the Moisture Resistance of Nylon-6,6 Cable Ties", submitted to Dennison Manufacturing, Framingham, Massachusetts (1986).

"Materials Testing Program for Angioflex Resin, Angioflex Solution and Angioflex Film, submitted to Abiomed, Danvers, Massachusetts *(1985).*

f. *Thesis*

"Synthesis and Characterization of Novel Polymer Structures: I. Head-to-Head Polypropylene; II. Regular Copolyoxamides", Ph.D. Thesis, Polymer Science and Engineering, University of Massachusetts, Amherst (1979).

C. **Service Contributions**

1. Professional Leadership and Achievement

a. *Recognition by Learned Professional Groups*

University of Massachusetts, Lowell, April 2006, Plastics Engineering Department Teaching Award. This award, voted by the students in the Department of Plastics Engineering, University of Massachusetts, Lowell, is given to that faculty member considered to provide outstanding teaching to the students in the Plastics Engineering Department.

University of Massachusetts, Lowell, April 2004, Plastics Engineering Department Teaching Award. This award, voted by the students in the Department of Plastics Engineering, University of Massachusetts, Lowell, is given to that faculty member considered to provide outstanding teaching to the students in the Plastics Engineering Department.

Boston Patent Law Association, Fall 1989. This award is given to an outstanding contribution in the area of intellectual property. The award-winning paper, "Experimental Use or Fair Use As A Defense To Patent Infringement", was subsequently published in Idea-The Journal Of Law and Technology.

Selected as University Scholar, Fall 1987. This award is given to outstanding faculty for achievement in teaching and research. In view of this award (a teaching load reduction) I was able to expand my academic background and initiate studies towards the Juris Doctor degree with emphasis on the law of intellectual property.

Selected as a Reviewer for the Journal of Polymer Science-Chemistry, February, 1989.

Selected as a Reviewer for the Journal of Polymer Science and Engineering December, 1988

Selected as a Reviewer for the Journal of Polymer Science-Chemistry, January, 1988

Selected as a member of the SPE-ANTEC Paper Review Board, 44th Annual Technical Conference, Boston 1986.

Selected as a Division/Special Session Technical Program Chairman, SPE-ANTEC, 44th Annual Technical Conference, Boston 1986.

Selected as a Reviewer for the Journal of Macromolecular Science-Chemistry, October 1986.

Selected as a Reviewer for the National Science Foundation-Chemical Instrumentation Program, July 1985

b. Work with Professional Societies

Former Member of the American Chemical Society.
Former Member of the Division of Polymer Chemistry, American Chemical Society
Foumer Member of the Society of Plastics Engineers
Former Member of the American Bar Association
Member, New Hampshire Bar (Admitted, May 1990)

2. Service To The University

a. *Student Advising*

Assistant Graduate Program Coordinator (with R. Deanin) in the Plastics Engineering Department (1984-1987).

Faculty Advisor for the University of Lowell, Alpine Club (1986-1988)

b. *Committee Membership*

Member, University of Lowell, Conflict in Interest Task Force (1989-90).

Chairman (1987) and Member (1985/1986) of the Joint Cumulative Exam Committee, Polymer Science and Plastics Engineering.

Member and Co-Chairman of the Research Advisory Committee (with Leon E. Beghian, 1987).

Member, Graduate School Summer Research Committee (1985,1986,1987).

c. *Attendance at Conferences*

SPE-ANTEC – Spring 2012 (Orlando)

SPE-ANTEC – Spring 2011 (Boston)

American Intellectual Property Law Association, Marco Island, January 1997

United States Trademark Association, Toronto, May, 1992

Franklin Pierce Law Center Conference on Enforcing and Litigating Patents & Trademarks, September, 1991

United States Trademark Association, San Francisco, May, 1991 Franklin Pierce Law Center, Tonference on Emerging Issues in Intellectual Property",

Boston Massachusetts, September 1990

American Chemical Society, Boston, Massachusetts, Spring 1990

ANTEC-SPE, Los Angeles, Spring 1987.

American Chemical Society, Denver, Colorado, Spring 1987.

ANTEC-SPE, Boston, Spring 1986.

SPE-SPI Plastics South, Atlanta, Spring 1986.

American Chemical Society, New York, Spring 1986.

ANTEC-SPE, Washington D.C., Spring 1985.

Gordon Research Conference on Polymers, Summer, 1983.

International Union of Pure and Applied Chemistry-28th Macromolecular Symposium, Amherst, Massachusetts, Spring 1982.

Gordon Research Conference on Polymer, Summer 1982.

Gordon Research Conference on Polymers, Summer 1981. American Chemical Society, Houston, Spring 1980.

Northeast Regional Meeting of the American Chemical Society, Boston, Summer 1978.

d. Consulting Activities

Carmody, Torrance, Sandak & Hennessey, Incardonia v. Medical Components, et al, Judicial District of Waterbury Connecticut, Docket X02-UWY-CV-07-6000812S (expert witness)

Greenburg-Traurig (G. Herbert), Orlando, Florida, expert witness, Goswami v. Universal Air-2002-CA-101 (2008)

April 2000, National Business Institute, Concord, New Hampshire, Lecturer on "Patent Infringement"

Morgan & Finnegan (M. Nicodema, 1999), expert witness, Triple S Plastics v. Capital Vial, No. CV 97-499 TUC AZ.

Jenner & Block (J. Benak, l999) expert witness, Tenneco Packaging & Consumer Products, Inc., v. S.C. Johnson & Son, Inc and KCL Corporation, Case No. 98C2679 N.D. Ill.

Pierce Atwood (Robert Stier, 1999 expert witness, Cohesive Technologies Inc. v. Waters Corporation, Civil Action 98-12308(DPWI)

Halloran & Sage (John Farley~1997/l998 expert witness/patent ownership, Fenn v. Analvtica (D.C. Connecticut))

Bernstein, Shur, Sawyer & Nelson (Robert H. Stier, Jr.) (1997 expert witness/patent infringement, Pastore/Produits v. Hydro-Innovation. (D.C. Maine))

Bronberg & Sunstein (1996 expert witness/patent infringement, E. Belt, Bailey v. M&N Plastics et.al.. Bailey v. Therma Systems Corporation. Civil Action No. 94-11 140-DPW: Bailey v. Dart. Inc. (D.C. Mass.)

Calhoun & Associates (1996 expert witness/patent infringement, TFH Publications v. Edwards. Civil Action No. 94-1945 (D.C.N.J.)

United States Government-Internal Revenue Service-District Counsel (1996, expert witness,/plastics recycling, Robert Gottsegen v. Commissioner of Internal Revenue. United States Tax Court Dkt No.23766-93.3. Dawson)

Halloran & Sage, Merocel Corp. v. Ultracell Medical Technologies. Inc., (Civil Action No. 393CV1041 (D.C. Connecticut)

Bemis Associates

Arthur D. Little Corporation

Megas Manufacturing

Ahiomed Comoration

Sandier & Travis, P.C. (1986-87 expert witness/plastic materials)

Ingersorl Rand

Laflamme, Migliori, Barron & Chabot (1987 expert witness/plastic materials)

Land & Sky Incorporated

Critikon

Kontron

Ciba-Coming

Anglo-American Clays Corporation

Polysar

McDermott, Will & Emery (1987, expert witness/patent infringement for Dennison Manufacturing before The United States International Trade Commission, Administrative Law Judge Sidney Harris (1987)) Dennison Manufacturing
Liskow & Lewis

3. Miscellaneous

Member, National Intercollegiate Soccer Officials Association and the New England Intercollegiate Soccer Officials Association. Active intercollegiate soccer official over the period 1976-2013 (37 years of active service).

Member, New Hampshire Baseball Umpires Association (inactive)

Licensed Ham Radio Operator

Licensed Commercial Pilot/Instrument Rating/Certified Flight Instructor (Helicopters)

Respondent’s Exhibit Z

J Polym Environ (2011) 19:637 676
DOI 10.1007/s10924 011 0317 1

ORIGINAL PAPER

Biodegradable Polymers- A Review on Recent Trends and Emerging Perspectives

Gisha E. Luckachan · C. K. S. Pillai

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Abstract Recent trends in biodegradable polymers indicate significant developments in terms of novel design strategies and engineering to provide advanced polymers with comparably good performance. However, there are several inadequacies in terms of either technology or cost of production especially in the case of applications in environmental pollution. So, there is a need to have a fresh perspective on the design, properties and functions of these polymers with a view to developing strategies for future developments. The paper reviews the present state-of-art on biodegradable polymers and discusses the salient features of the design and properties of biodegradable polymers. Special emphasis is given to the problems and prospects of (1) approaches adopted to make non-biodegradable synthetic polymers such as polyethylene biodegradable and (2) biodegradable polymers and copolymers made from renewable resources especially poly(lactic acid) based polymers and copolymers which are emerging as the candidate biodegradable materials for the future.

Keywords Biodegradable polymers · Environmental aspects · Recent trends · Future perspectives · Polyethylene · Poly(lactic acid)

Introduction: The Relevance of Biodegradable Polymers

Synthetic polymers are considered to be an invaluable gift of modern sciences and technology to mankind. They have become indispensable to our life with their wide range of applications in diverse fields such as packaging, agriculture, food, consumer products, medical appliances, building materials, industry, aerospace materials etc. However, the resistance of synthetic polymers to chemical, physical and biological degradation has become a serious concern when used in areas such as surgery, pharmacology, agriculture and the environment, and as a consequence time-resistant polymeric wastes are becoming less and less acceptable [1]. Naturally, the necessity for polymeric materials satisfying the conditions of biodegradability, biocompatibility and release of low-toxicity degradation products, as an alternative to these existing polymers is apparent.

The severe environmental problems, including the increasing difficulties of waste disposal and the deepening threat of global warming (due to carbon dioxide released during incineration) caused by the nonbiodegradability of a number of polymers such as polyethylene (used in packaging and agriculture field) have raised concerns all over the world. We must confront them in order to build a new society and economy free of plastic pollution in the twenty-first century. The use of biodegradable counter parts as an alternative to non-biodegradable polymers is, therefore, evoking considerable interest lately. Unfortunately, the degradable polymers available until recently have inferior physical properties in terms of strength and dimensional stability and most of them are very expensive and are technically difficult to process. As a result, attempts have been made to solve these problems by including biodegradability into polymers in everyday use through appropriate modifications of their structure.

Degradability is not only a concern from the environmental point of view. Today the medical applications of polymers are at an all time high. Biodegradable polymers

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G. E. Luckachan · C. K. S. Pillai (✉)
National Institute for Interdisciplinary Research and Technology (formerly Regional Research Laboratory), CSIR, Thiruvananthapuram 695 019, India
e mail: ckspillai@yahoo.com

from renewable resources have attracted much interest in recent years because of its desirable properties of biocompatibility, biodegradability and natural abundance [2 4]. A number of biodegradable polymers of natural and synthetic origin with good biodegradability and biocompatibility have been made recently. However, they have their inherent problems that need to be discussed and scientific and technological solutions arrived at. This review proposes to discuses the salient features of biodegradable polymers with special emphasis on the problems and prospects of (1) approaches adopted to make nonbiodegradable synthetic polymers biodegradable taking polyethylene as an example and (2) biodegradable polymers and copolymers made from renewable resources such as lactic acid. Currently, polylactide (PLA)/polyglycolide polymers are the most widely used synthetic biodegradable polymer for biomedical applications [5 8]. However, certain limitations of PLA such as low hydrophilicity and degradation rate, poor soft tissue compatibility, low thermal and physical properties, lack of processability, high cost of production along with its increased use in medicine put forward the necessity in the search of new materials that exhibit unique properties for specific applicability in the field [9, 10].

Definition of Biodegradation

The various definitions of biodegradation depend on the field of application of the polymers (biomedical area or natural environment). Many different definitions have officially been adopted, depending on the background of the defining standard organizations and their particular interests. Van der Zee and Seal [11, 12] review all of the definitions found in different standards. Albertsson and Karlsson [13] defined biodegradation as an event that takes place through the action of enzymes and/or chemical decomposition associated with living organisms and their secretion products. It is also necessary to consider abiotic reactions like photodegradation, oxidation and hydrolysis, which may alter the polymer before, during or instead of biodegradation because of environmental factors. So, strictly speaking, "biodegradation of a polymer" is defined as the deterioration of its physical and chemical properties and a decrease of its molecular mass down to the formation of CO_2, H_2O, CH_4 and other low molecular-weight products under the influence of microorganisms in both aerobic and anaerobic conditions aided by abiotic chemical reactions like photodegradation, oxidation and hydrolysis [14].

According to ASTM D 6400-99 [15 19] biodegradable plastic is defined as degradable plastic in which the degradation results from the action of naturally occurring microorganisms such as bacteria, fungi and algae. Compostable plastic is a plastic that undergoes degradation by biological processes during composting to yield CO_2, water, inorganic compounds and biomass at a rate consistent with other compostable materials and leaves no visible, distinguishable or toxic residue.

Chemistry of Biodegradation

Biodegradation is the only degradation path way that is able to completely remove a polymer or its degradation products from the environment. Biodegradation takes place in two stages (Fig. 1): The first stage is the depolymerization of the macromolecules into shorter chains. This step normally occurs outside the organism due to the size of the polymer chain and the insoluble nature of many polymers. Extra-cellular enzymes (endo or exo-enzymes) and abiotic reactions are responsible for the polymeric chain cleavage. During this phase the contact area between the polymer and the microorganism increases [20, 21].

The second step corresponds to the mineralization. Once sufficient small size oligomeric fragments are formed, they are transported into cells where they are bioassimilated by the microorganisms and then mineralized [22, 23]. Biodegradation takes place in two different conditions depending upon the presence of oxygen; aerobic biodegradation (in the presence of oxygen) and anaerobic biodegradation (in the absence of oxygen). Complete biodegradation or mineralization occurs when no residue remains, i.e., when the original product is completely converted into gaseous products and salts [24].

Biodegradable Polymers: An Overview

Several books and reviews have been published on this area covering many aspects of biodegradation and biodegradable polymers. In one of the earliest texts on the subject of degradation of polymers, Schnabel [25] discussed the mode of polymer degradation in four main categories: biodegradation, oxidation, hydrolysis and photodegradation. Ching et al. [26] and Kaplan et al. [27] presented biodegradable polymers from the point of view of packaging materials. The salient features of biodegradable polymers have been brought out by Vert et al. [28] in their books on Biodegradable polymers and plastics. Hamid et al. [20] have edited a handbook on polymer degradation.

The proceedings edited by Doi and Fakuda [29] deal with biodegradability including environmental biodegradation, synthetic techniques, morphology and blends, test methods and governmental policies. The chemistry of biodegradable polymers has been brought out in the text

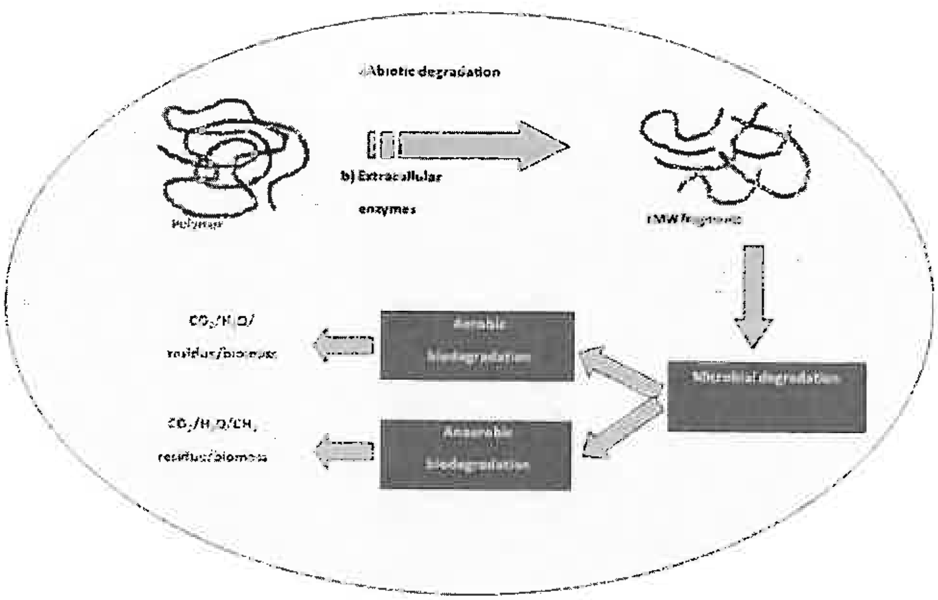

Fig. 1 Diagrammatic representation of the chemistry of biodegradation

edited by Griffin in 1994 [30]. MacGregor et al. [31] may be referred for a discussion on polymers from nature which is considered mostly biodegradable. Other books on natural/agricultural polymers are edited by Carraher et al. [32] and Glass and Swift [33]. Byrom [34] discusses biodegradable polymers from point of view of biomaterials.

Rapra Review Reports [35] provide Advances in Biodegradable Polymers edited *by* Moore and Saunders. Some interesting reviews on biodegradable polymers by Ann Christine Albertsson (pp 345 382), starch based degradable plastics by Wayne J. Maddever (pp 383 410) and degradable plastics by Hideki Omichi (pp 335 344) are available in the book. Glass and Swift discuss biodegradation and utilization of polymers from agricultural resources in the book, Agricultural and Synthetic Polymers, Biodegradation and Utilization [33]. Scott and Gilead have edited a book on Degradable Polymers [36]. Emo et al. [37] present environmentally degradable polymeric materials from renewable resources, water-soluble/swellable biodegradable polymers, biopolymers and biorelated polymers, bioresorbable materials for biomedical applications. The book also provides data on standards and regulations. The Handbook of Biodegradable Polymeric Materials and Their Applications, edited by Mallapragada and Narasimhan [38] provides a comprehensive review of the synthesis, characterization, processing, and applications of biodegradable polymers (polymeric biomaterials, biomedical engineering, food science and nutrition, green engineering, drug delivery, single-dose vaccines, protein stabilization, gene therapy, tissue engineering, orthopedics, and food packaging covering a broad spectrum of polymers such as polycaprolactones, polyesters, phosphazenes, phosphoesters, polyanhydrides, starch, chitosan and chitin).

A comprehensive first review was brought by Cassidy and Aminabhavi [39]. They considered degradation of the polymer through the reaction of the components of the environment that often promotes chemical change and correlated hydrolytic process with the enzymatic activity. Sudesh et al. [40] made an attempt from the chemical angle to present (1) the prospects for biodegradable plastics followed by the principal retrospective aspects of biodeterioration and (2) to identify possible approaches for designing new biodegradable systems and finally to review the present status of research with special reference to structure-biodegradability relationships. Bastoili [41] provided a discussion of biodegradable materials in another review. Aminabhavi et al. [42] later brought out another review focusing on the biodegradation of those polymers which have relevance in the packaging industry for food and vegetables (polyolefin plastics, with a greater emphasis on linear low-density polyethylene (LLDPE) and low-density polyethylene (LDPE)). Literature findings are covered from 1970 to 1989. The chemistry of biodegradation and the relevant methods used to study biodegradation are discussed. Several reviews [43 56] appeared between 1990 and 1993 indicating the significance of this fast growing area. Swift [47, 54, 55] wrote several reviews during this period on the complexities and significant definitions of biodegradable plastics. Kaplan et al. [57] discussed the fundamental aspects of biodegradation in 1993. Albertson [58] analyzed the biodegradation of synthetic polymers comparing it with the natural biodegradation of wood. Lenz [59] also made a good review during this period and the developments unto 1994 were summarized by David et al. [60]. The environmental biodegradation of synthetic polymers was given ample stress by Andrady [61] and thoroughly discussed by Scott [62].

Several review articles [3, 63 74] on biodegradable plastics appeared during 1996 1998. Albertsson and Karlsson [63] in a review identified four main routes for the design of biodegradable polymers. The most convenient route is to use cheap synthetic bulk polymers and add a biodegradable or photooxidizable component. A more expensive solution is to change the chemical structure by introducing hydrolyzable or oxidizable groups in the repetitive chain of a synthetic polymer. The third route to degradable polymers is to use biopolymers or derivatives of these where the bacterial polyhydroxyalkanoates are perhaps the most studied material of them all. The fourth route is to tailor-make new hydrolyzable structures e.g., polyesters, polyanhydrides, and polycarbonates. Beach et al. [64] assessed the aggregate impact of the substitution of cornstarch-based biodegradable polymer resins for petroleum-based plastic materials and resins on the US economy. The results suggest that for a DLR1.00/lb of resin subsidy, the substitution of cornstarch-based biodegradable polymer resins for petroleum-based plastic materials and resins will result in an increase in output by all producing sectors of 0.067% or about DLR542 million, a rise in the consumption of goods and services of about 0.003% or DLR110 million, a rise in total utility of 0.004% or DLR168 million, and a net increase in government expenditures of 0.047% or DLR369 million. Two comprehensive reviews [21, 67] appeared in 1998. Chandra and Rustgi [21] may be referred for a detailed survey of biodegradable polymers with an exhaustive reference collection. Amass et al. [67] have brought out the uses of biodegradable polymers in terms of their relevance within current plastic waste management of packaging materials, biomedical applications and other uses including a summary of degradation processes. De Wild and Boelens [68] enumerate the prerequisites for biodegradable plastic materials for acceptance in real-life, composting plants and technical aspects. Between 1998 and 2001, Bastioli [70 72] has several articles on biodegradable polymers giving present status and future directions and industrial applications.

Anuradha and Kumar [73] made an attempt at a concise review of environmentally degradable materials giving both mechanisms of degradability, photodegradation and biodegradation. Materials, criteria and evaluation methods are discussed. Nayak [74] discussed the challenges and opportunities of biodegradable polymers in 1999. A number of plant oils, such as castor, Lesquerella palmeri, vernonia, tung, crambe oils and cashew nutshell liquid, have been used to prepare a variety of novel bio-based polymers for industrial applications. This is a very challenging field of research with unlimited future prospects. Researchers can focus their attention on agricultural products for manufacturing monomers and polymers to keep the environment cleaner and greener in the twenty-first century. The potential of biodegradable plastics food packaging was discussed by Petersen et al. [75]. Fomin and Guzeev [76] projected the present status and future prospects of biodegradable polymers. Rouilly and Rigal [77] also propose the use of biomass to conceive new materials and discuss "plastic" properties of natural polymers, the characteristics of the different classes of polymers, the use of charge in vegetable matrices and the possible means of improving the durability of these agro-materials. Gross [78] discussed the need for biodegradable polymers for the environment and posed problems associated with the use of biodegradable polymer such as starch, cellulose and lactic acid. Scott [46, 79] proposes that polymers that degrade by peroxidation followed by bioassimilation of the oxidation products (oxo-biodegradable polymers) are in general more environmentally acceptable ('green') than the biologically produced hydro-biodegradable polymers. Chiellini [80] also made an overview on environmentally degradable polymeric materials (EDPM) in agricultural applications.

In reviewing the properties of biopolymers, Van de Vel and Kiekens [81] brought out the use of biodegradability in several applications. The available bioplastic technologies have been reviewed by Naryanan [82]. A review by Flieger et al. [83] summarizes data on the use, biodegradability, commercial reliability and production of biodegradable plastics from renewable resources. (e.g., commercially successful biodegradable plastics made by chemical synthesis such as polyglycolic acid, polylactic acid, polycaprolactone and polyvinyl alcohol, products of microbial fermentations such as polyesters and neutral polysaccharides and chemically modified natural products such as starch, cellulose, chitin or soy protein). Like Gross [78], Stevens [84] also provides a discussion on environmentally degradable plastics in his review "Environmentally Degradable Plastics". Ren [85] posed the question whether biodegradable polymers is a solution or a challenge. Pillai [86] recently reviewed the area on natural monomers and polymers and pointed out that their inherent biodegradability has been of great value in the emerging initiatives such as 'bio-inspired' polymer design, 'synthetically-inspired' materials and engineered bio-polymers inspiring prospects for polymer science at the biointerface with new materials, taking natural and synthetic polymers beyond nature's limitations. Ralston et al. [87] introduced the theme of sustainable polymers and reviewed current research on biopolymers such as casein and soy protein based plastics as well as polylactide acid.

Apart from data available in various reviews, there are a few excellent reviews [88 90] on the synthetic techniques adopted for the preparation of biodegradable plastics. Okada [88] in 2002 has brought out the various methodologies adopted in synthesizing biodegradable polymers.

Kricheldorff [89] also has reviewed the synthetic techniques for the preparation of cyclic metal alkoxides and their use as initiators for ring opening polymerization of lactones, cyclic diesters and cyclocarbonates which are biodegradable. Hori et al. [90] also have reviewed the technique used for preparing novel biodegradable polyesters. Based on polymer structure, polymer physical properties and environmental conditions, it is possible to design biodegradable polymers for a particular application. Some guidelines are as follows: a higher ratio of hydrophilic/hydrophobic ratio is better for degradation, fully carbon chain polymers are less likely to degrade than hetro atom containing polymers, chain branching is deleterious to biodegradation, condensation polymers are more likely to undergo biodegradation, lower molecular weight oligomers are more susceptible to biodegradation, crystallinity reduces biodegradation, lower surface area reduces biodegradation etc. Water solubility and purity add to possibilities for biodegradation. Environmental conditions to be considered for evaluation are temperature, pH, moisture, oxygen, nutrients, suitable microbial population, concentration and test duration. In short, parameters such as chemical structure, chemical composition, distribution of repeat units, presence of functional groups, presence of chain defects, configurational structure, molecular weights and polydispersity, presence of low molecular weight compounds, processing conditions, shape, sterilizing conditions, morphology, annealing effects, storage history etc. might be considered in selecting a biodegradable polymer.

Tang et al. [91] published a review covering research, development and production of environmentally degradable plastics, including photo degradable plastics, biodegradable plastics and disintegratable plastics; defines problems to be solved in relation to the situation in China. The biodegradation of plastic waste was considered by Zheng et al. [92] also in a recent review. In discussing the impact of Biotechnology and next generation materials, Stowers [93] has brought out the need for biodegradable plastics in many applications. In a recent review, Raquez et al. [94] discussed how reactive extrusion (REx) technique can participate in the design and development of biodegradable polymeric materials. Gunatillake et al. [95] in a recent article discuss developments in biodegradable synthetic polymers focusing on tailoring polymer structures to meet material specification for emerging applications such as tissue engineered products and therapies. This review may be referred for data on properties and biodegradability of major classes synthetic polymers such as polyesters and their copolymers including polyglycolides, polylactides and their copolymers, polyurethanes, polyphosphazenes, polyanhydrides, polycarbonates, polyesteramides and recently developed injectable polymer systems based on polypropylenefumarates, polyurethanes and acrylate/urethane systems. Demirbas [96] introduces natural biodegradable plastics based on primarily on renewable resources. The biodegradability of plastics is dependent on the chemical structure of the material. The biodegradation of plastics proceeds actively under different soil conditions according to their properties. Biodegradation of starch based polymers occurred between the sugar groups leading to a reduction in chain length and the splitting off of mono-, di- and oligo-saccharide units by a result of enzymatic attack at the glucosidic linkages. Wang et al. [97] reviewed the current development of biodegradable plastics, namely poly(lactic acid), poly(hydroxyalkanoate), poly(ε-caprolactone), poly(butylenes succinate), poly(vinyl alcohol) and its copolymer, and starch. Attention was also paid to relevant existing problems of the above biodegradable plastics.

Biodegradable Polymers in Composites

The emergence of composites and lately nanocomposites has brought in a revolution in materials science. Several articles have highlighted the importance of biodegradable polymer matrix in the development and use of composite materials [98 103]. Recent advances in polymer blends and composites from renewable resources and their potential applications have been reviewed recently by Yu et al. [98]. Among these products, natural fiber eco composites [100 106] are emerging rapidly. Bogoeva-Gaceva et al. [100] provides a brief review of the most suitable and commonly used biodegradable polymer matrices and natural fiber (NF) reinforcements in eco-composites, as well as some of the already produced and commercialized NF eco-composites. The structural aspects and properties of several biofibers and biodegradable polymers, recent developments of different biodegradable polymers and biocomposites are discussed by Mohanty et al. [101, 102] and Lodh et al. [103]. The recycling of biocomposites was reviewed by Bhakat et al. [104].

Pandey et al. [105] addresses the technical difficulties and suggest solutions in the application of biodegradable nanocomposites. Renewable resource-based biodegradable polymers including cellulosic plastic (plastic made from wood), corn-derived plastics, and polyhydroxyalkanoates (plastics made from bacterial sources) are some of the potential biopolymers which, in combination with nanoclay reinforcement, can produce nanocomposites for a variety of applications. Nanocomposites of this category are expected to possess improved strength and stiffness with little sacrifice of toughness, reduced gas/water vapor permeability, a lower coefficient of thermal expansion and an increased heat deflection temperature, opening an opportunity for the use of new, high performance, light weight green nanocomposite materials to replace conventional petroleum-based composites. The use of biodegradable natural fibers which are biodegradable has also got a new dimension as reinforcement in natural fiber composites [106 113]. Inherent shortcomings of natural polymer-based packaging materials such as low mechanical properties and low water resistance can be recovered by applying a nanocomposite technology. Natural biopolymer-layered silicate nanocomposites exhibit markedly improved packaging properties due to their nanometer size dispersion. These improvements include increased modulus and strength, decreased gas permeability, and increased water resistance. Consequently, natural biopolymer-based nanocomposite packaging materials with bio-functional properties have a huge potential for application in the active food packaging industry [114]. Yang et al. have reviewed recently the preparation, characterization, properties, and applications of nanocomposites based on biodegradable polymers such as polylactide (PLA), poly(ε-caprolactone) (PCL), poly(p-dioxanone) (PPDO), poly(butylenes succinate) (PBS), poly (hydroxyalkanoate)s such as poly(β-hydroxybutyrate) (PHB), poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV), and natural renewable polymers such as starch, cellulose, chitin, chitosan lignin, and proteins [115]. The nanoparticles that have been also utilized to fabricate the nanocomposites include inorganic, organic, and metal particles such as clays, nanotubes, magnetites, Au and Ag, hydroxyapatite, cellulose, chitin whiskers and lignin.

Biodegradable Polymers in Medicine and Surgery

The property of biodegradability of polymers when applied to medicine and surgery has immense applications. Following Kronenthal [116], several authors [117 151] have reviewed and contributed to various applications of biodegradable polymers in medicine and surgery. According to Kronenthal, the polymer degrades in vivo in four steps: (1) Hydration- disruption of van der waals forces and hydrogen bonds. (2) Strength Loss- initial cleavage of backbone covalent bonds. (3) Loss of Mass Integrity- further cleavage of covalent bonds to polymer molecular weight levels insufficient for mass coherence. (4) Mass Loss- (Solubilization) dissolution of low molecular weight species and phagocytosis of small fragments. He also gives a list of characteristics for a biodegradable implant to be successful. They are: formability, adequate initial strength and dimensional stability, controlled rate of strength loss, complete absorbability, low order of toxicity of both implant and degradation products and sterilizability. Among the different classes of biodegradable polymers, the thermoplastic aliphatic poly(esters) such as poly(lactide) (PLA), poly(glycolide) (PGA) and especially the copolymer of lactide and glycolide referred to as poly(lactide-co-glycolide) (PLGA) have generated tremendous interest because of their excellent biocompatibility, biodegradability and mechanical strength. They are easy to formulate into various devices for carrying a variety of drug classes such as vaccines, peptides, proteins and macromolecules. Most importantly, they have been approved by the United States Food and Drug Administration (FDA) for drug delivery. This review presents different preparation techniques of various drug-loaded PLGA devices, with special emphasis on preparing microparticles. An excellent treatment on the synthesis, properties and applications of aliphatic polyesters can be found in the review by Albertsson and Varma [119]. Pillai et al. induced biodegradability in the otherwise non-biodegradble liquid crystalline aromatic polyesters by introducing an aliphatic carbonyl [121, 122, 128]. In another work, Pillai et al. showed that biodegradable aliphatic poly(ester amides) having interestig properties for biomedical applications could be prepared through an amido diol route [137, 138, 140]. In the area of tissue engineering, the design and fabrication of biodegradable synthetic scaffolds is driven by four material categories: (1) common clinically established polymers, including polyglycolide, polylactides, polycaprolactone (2) novel di- and tri-block polymers (3) newly synthesized or studied polymeric biomaterials, such as polyorthoester, polyanhydrides, polyhydroxyalkanoate, polypyrroles, poly(ether ester amide)s, elastic shape-memory polymers and (4) biomimetic materials, supramolecular polymers formed by self-assembly and matrices presenting distinctive or a variety of biochemical cues [139]. Degradable polymeric biomaterials are preferred candidates for developing therapeutic devices such as temporary prostheses, three-dimensional porous structures as scaffolds for tissue engineering and as controlled/sustained release drug delivery vehicles. Each of these applications demands materials with specific physical, chemical, biological, biomechanical and degradation properties to provide efficient therapy. These aspects including the synthesis, biodegradability and biomedical applications of biodegradable synthetic and natural polymers as biomaterials have been discussed in a recent review by Nair and Laurencin [152]. The use of biodegradable natural polymers in tissue engineering has\been reviewed by Mano et al. [153]. Williams recently reviewed the synthesis and characterization of functionalized aliphatic polyesters which are attracting attention as sustainable alternatives to petrochemicals for applications in medicine [154]. Biodegradable elastomers have a number of potential applications in the biomedical area, especially in the emerging field of soft-tissue engineering where the mechanical properties of the polymer scaffold should match those of the tissue to be grown. The synthesis strategies and the properties of these elastomers are discussed by Amsden elaborating the factors that influence the characteristics of these elastomers including mechanical properties, degradation rate, and mechanical property change during degradation in terms of the design of the elastomer and their advantages and disadvantages for biomedical applications [155].

The successful performance of the first synthetic poly(-glycolic acid) based suture system (The first biodegradable synthetic suture called DEXONs that was approved by the United States (US) Food and Drug AdministrationFDA) in 1969 was based on Polyglycolide) during the late 1960s led to the design and development of a new array of biodegradable polymers as transient implants for orthopaedic and related medical applications [152, 156]. Pillai and Sharma [156] has recently reviewed and brought to focus the scattered data on chemistry, properties, biodegradability, and performance of absorbable polymeric sutures. Pillai et al. [157] have discussed the significane of chitosan for various biomedical applications emphasising its biodegradability and biocomppatibilty and pointed out the unique characteristics of chitosan for fiber application.

Biodegradable Polymers from Renewable Resources

Biodegradable polymers can be broadly divided into different categories based on the origin of the raw materials (renewable or non-renewable) and the processes used in their manufacture [158 161]. Shifting the resource base for chemical production from fossil feedstocks to renewable raw materials provides exciting possibilities for the use of industrial biotechnology-based process tools [161]. Among renewable resource based materials, biomass is generally constituted of polymers, oligomers, monomers, and other non-polymerizable simple organic compounds including metallic salts and complexes [159 163]. Polymers are, of course, the major constituents and have been serving human civilizations from time immemorial [14, 164, 165]. The outstanding aspect of natural polymers is their wide variety which gives innumerable opportunities for structural modifications and utilization. Depending on the nature of the hetero atom inserted in the main chain, the polymers can be classified into four major types [166 168] as hydrocarbon polymers (e.g., natural rubber), carbon-oxygen (e.g., carbohydrates- cellulose [starch, chitin, chitosan, pullulan etc.; phenolics - lignin, humus etc. and polyesters-shellac), carbon-oxygen -nitrogen/sulphur (e.g., proteins with the exception of phospho proteins) and carbon-oxygen-nitrogen-phosphorus (e.g nucleic acids) containing polymers. These polymers especially, polysaccharides, additionally offer CO_2-neutrality, partial independence from petrochemistry-based products and the exploitation of natures synthesis capabilities via photosynthesis [169]. Polysaccharides (cellulose [170 176], starch [14, 177 182], chitin and chitosan [148, 182], proteins (gelatin, casein, pectin etc. [107, 183] and others prepared from natural monomers [184 186] have found innumerable applications in biodegradable products. Nature has a plethora of natural enzymes for degrading natural polymers. The rate of degradation and the formation of the ultimate metabolites depend very much on the structural complexity of the material and the environmental conditions selected for the degradation trial. Several reviews have appeared on various aspects of biodegradability and applications of these natural polymers. Among the polysaccharides, cellulose [168 175] is an almost inexhaustible polymeric raw material with fascinating structure and properties. Cellulose is a linear condensation syndiotactic polymer of anhydrogluose units joined together by β-1,4-glycisidic bonds. Properties such as its hydrophilicity, chirality, biodegradability (readily biodegradable by many microorganisms due to activity of cellulase enzyme complex.), broad chemical modifying capacity, and its formation of versatile semicrystalline fiber morphologies etc. make it a polymer of unique possibilities. Based on general considerations on the correlation between biodegradability and molecular structure, cellulose derivatives allow both thermoplastic processing and post-consumer waste management via biological decomposition [169].

The significance of natural polymers can be explained by taking chitosan, as an example, which is well known to elicite minimal foreign body reactions with little or no fibrous encapsulation upon implantation. Figure 2 shows the tissue responses to chitosan gels when compared to biodegradable surgical suture Vicryls. Chitosan gels elicited very minimal inflammatory response compared to Vicryls (a copolymer of polyglycolide and polylactide) which showed a pronounced inflammatory reaction. The gels with lower cross-linking density (FDG) showed significant weight loss after 14 days of implantation; approximately 80% (subcutaneous) and approximately 91% (intraperitoneal).No significant decrease in weight was observed for highly cross-linked gels 9SDG) after 14 days of implantation [187] implantation on the in vivo degradation of chitosan based implants. The study clearly showed the effect of cross-linking density and the site of implantation on the in vivo degradation of chitosan based implants. In vivo, chitosan is also known to elicite minimal foreign body reactions.

Appropriate chemical modification improves the biodegradability. NM-chitosan and partially N-acylated chitosan films were assessed by exposure of the films to laboratory-scale aerobic thermophilic compost reactors and abiotic control vessels (Fig. 3) [188].

Starch [176 182] one of the most inexpensive and most readily available of all natural polymers, is related in structure to cellulose by having the same repeat unit. The 1,4-α-glycosidic linkage of glucose units in starch rather than the 1,6-β-glycosidic linkage in cellulose gives rise to a

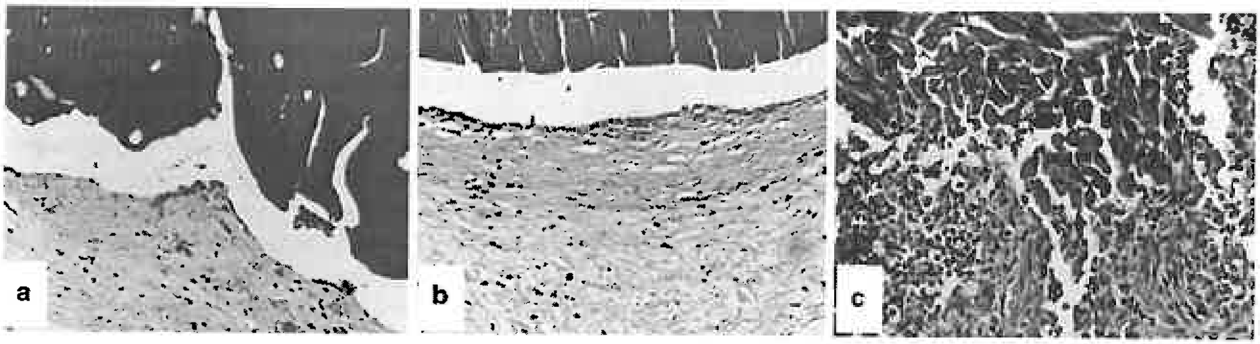

Fig. 2 Histological sections demonstrating minimal tissue response in the surrounding tissues: (**a**) highly cross linked chitosan implant, (**b**) lighly cross linked chitosan implant and (**c**) chronic foreign body reaction in the tissues surrounding surgical sutures such as vicryl. (Reprinted from Ref. [187] permission from Elsevier)

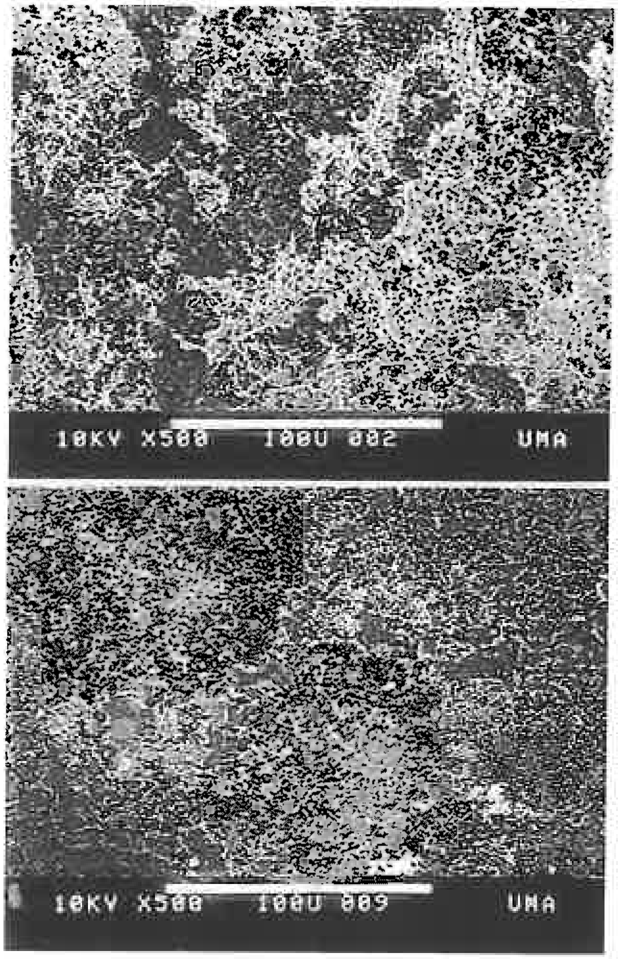

Fig. 3 Scanning electron micrographs of the degraded samples by aerobic thermophilic compost: N acylated (*top*) and NM chit (not modified chitosan) after 21 days (*bottom*). (Reproduced from Ref. [188] with permission from Elsevier)

branched structure with concomitant change in properties. The high strength and rigidity of cellulose originate thus from the linear structure and the hydrogen bonding made possible. Apart from other uses as gums and adhesives, a promising application is in the area of biodegradable polymers either after grafting with suitable polymers or after blending with other synthetic polymers and can be processed into thermoplastic materials only in the presence of plasticizers and under the action of heat and shear. Poor water resistance and low strength are limiting factors for the use of materials manufactured only from starch, and hence the modification of starch is often achieved by blending aliphatic polyesters. The biodegradable rates of blends can be controlled to a certain extent depending on the constitutions of blends, and the mechanical properties of blends are close to those of traditional plastics such as polyethylene and polystyrene. The reduction of their sensitivity to humidity makes these materials suitable for the production of biodegradable films, injection-molded items, and foams. Thermoplastic starch and its blends are known to cater to a sustainable development, which means that the responsible use of available natural resources and production processes take environmental aspects and natural circulations into consideration [180]. Starch foam is one of the major starch-based packaging materials. It is produced by extrusion or compression/explosion technology. This product has been developed as a replacement for polystyrene which is used to produce loose-fillers and other expanded items. The major starch-derived polyesters in the market now are polylactic acid and polyhydroxyalkanoate [176]. Proteins (gelatin, casein, soy products etc.) [116, 189] have also found innumerable applications in biodegradable products.

Natural biodegradable polymers being renewable can be considered under the category of green materials [165, 166, 190]. One can alter the structure of natural biodegradable polymers to impart certain additional properties or to improve the biodegradability itself [191]. There are a variety of natural existing monomers which can be polymerized to obtain with various degrees of success in biodegradability [165, 168, 192 194]. Monomers can also be obtained by extraction and/or fermentation from biomass constituents and biodegradable polymers could be synthesized [191 195]. Monomers such as 1,3-propanediol, 1,4-butane diol, γ-butyrolactone, adipic acid, N-methylpyrrolidone, succinic acid, lactic acid etc. are produced by fermentation from renewable resources [51, 83, 97]. It is, however, not indented in this review to provide a serious discussion on natural biodegradable polymers except that of lactic acid polymers.

Microbially Produced Polyesters

The family of bacterial polyesters commonly referred to as poly(hydroxyalkanoate)s have generated much interest because of their useful properties such as biodegradability for disposable plastic applications. They are the gift of microbiology community to polymer chemists as described first by Lemoigne. Biodegradable polyesters enzymatically synthesized using bacteria have emerged as industrially important biodegradable plastics [47, 70, 196 224]. Unlike petroleum based polymers that take several decades to degrade, polyhydroxyalkanoates (PHA)s can be completely bio-degraded within a year by a variety of microorganisms to give carbon dioxide and water. Attempts based on various methods have been undertaken for mass production of PHAs. Promising strategies involve genetic engineering of microorganisms and plants to introduce production pathways. This challenge requires the expression of several genes along with optimization of PHA synthesis in the host. Suriyamongkol et al. considered that although excellent progress has been made in recombinant hosts, the barriers to obtaining high quantities of PHA at low cost still remain to be solved [224]. The commercially viable production of PHA in crops, however, appears to be a realistic goal for the future.

PHAs are naturally occurring biodegradable polyesters produced as energy storage materials by many bacteria. The most common PHA, poly (3-hydrobutyrate) (PHB), is an isotactic semi-crystalline polyester. Hydrolytic degradation occurs by surface erosion, making it an attractive material for controlled release applications. The homopolymer PHB has relatively high melting point and crystallizes rapidly, making entrapment of drug technically difficult. The related copolymers with 3-hydroxyvalerate, P (HB-HV)s, have similar semi-crystalline properties though their slower rates of crystallization result in matrices with different properties. PHB and P (HB-HV) matrices lose mass very slowly when compared to bulk-degrading poly(lactide-glycolide) systems. Therefore the applications of these materials in drug delivery are likely to depend on the formulation of suitable blends with other biocompatible polymers.

Patnaik suggests that while the biochemistry of PHB synthesis by microorganisms is well known, improvement of large-scale productivity requires good fermentation modeling and optimization [225]. He proposes hybrid models, combining mechanistic, cybernetic and neural models that offer a useful compromise. All three kinds of basic models are discussed with applications and directions toward hybrid model development that expresses adequately all the important features of large-scale non-ideal fermentations.

PHAs are the polymers of hydroxyalkanoates that accumulate as carbon/energy or reducing-power storage material in various microorganisms. Until now, however, industrial production of PHAs has encountered only limited success. The main barrier to the replacement of synthetic plastics by PHAs has been the higher cost. The use of mixed cultures and renewable sources obtained from waste organic carbon can substantially decrease the cost of PHA and increase their market potential. Lu [226] reviews two main methods of PHA production by mixed cultures, anaerobic-aerobic processing and aerobic transient feeding processing, and analyzed the metabolic and effective factors.

The isolation and the purification of bacterial polyhydroxyalkanoates are the key step of the process profitability in the fermentation system [227]. Medium-chain-length polyhydroxyalkanoates (MCL-PHAs), which have constituents with a typical chain length of C_6 C_{14}, are polyesters that are synthesized and accumulated in a wide variety of Gram-negative bacteria, mainly pseudomonads [228]. These biopolyesters are promising materials for various applications because they have useful mechanical properties and are biodegradable and biocompatible. The versatile metabolic capacity of some Pseudomonas spp. enables them to synthesize MCL-PHAs that contain various functional substituents; these MCL-PHAs are of great interest because these functional groups can improve the physical properties of the polymers, allowing the creation of tailor-made products. Moreover, some functional substituents can be modified by chemical reactions to obtain more useful groups that can extend the potential applications of MCL-PHAs as environmentally friendly polymers and functional biomaterials for use in biomedical fields.

In the very near term, metabolic engineering will help solve many problems in promoting PHA as a new type of plastic material for many applications. PHA monomer structures and their contents strongly affect the PHA properties. Using metabolic engineering approaches, PHA structures and contents can be manipulated to achieve controllable monomer and PHA cellular contents [229].On the application side, the rate of degradation is important where fast or slow degradation may be required. Figure 4 shows that increasing the concentration of the C6 comonomer increases the rate of degradation [227].

Medium-chain-length polyhydroxyalkanoates (MCL-PHAs), which have constituents with a typical chain length of C_6-C_{14} that are synthesized and accumulated in a wide variety of Gram-negative bacteria, mainly pseudomonads are promising materials for various applications. These

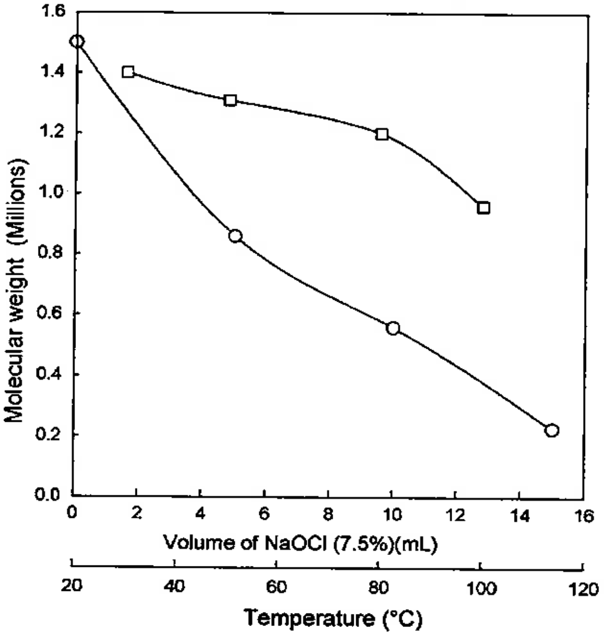

Fig. 4 Degradation of bacterial polyhydroxyalkanoates. Increasing the concentration of the C6 comonomer increases the rate of degradation. (Reproduced from Ref. [227])

MCL-PHAs are of great interest because the functional groups present in them can improve the physical properties of the polymers, allowing the creation of tailor-made products [228]. In the very near term, metabolic engineering will help solve many problems in promoting PHA as a new type of plastic material for many applications. A similar view is held by Sun et al. who reviewed the area recently [230].

The process known as "feast and famine" or as "aerobic dynamic feeding" seems to have a high potential for PHA production by mixed cultures [202]. Production of PHA by transgenic plant may be economically viable if efforts are concentrated at improving this process [231]. It may be possible in the near future to see farmers growing plastics in their field and a new agricultural product "plastics fruit" may appear in our market by transgenic plant [232, 233]. Several reviews have appeared recently covering various aspects of the production of biodegradable PHAs listing their properties and applications [234 242]. There are also attempts to evolve novel methods of production of aliphatic polyesters with controlled macromolecular parameters by still "greener" ways [242]. Copolymerization has been advocated to improve biodegradability and other properties [243 246].

Industrial firms are now commercializing PHA as natural plastics made from renewable resources such as corn sugar and vegetable oils. Within the foreseeable future, this technology will also enable the production of these plastics directly in non-food plant crops. These high performing new materials have the potential to put a large portion of the plastics and chemicals industry on a sustainable basis.

However, these bioplastics are currently far more expensive than petrochemical based plastics and are therefore used mostly in applications that conventional plastics cannot perform, such as medical applications. PHAs are immunologically inert and are only slowly degraded in human tissue, which means they can be used as devices inside the body. Recent research has focused on the use of alternative substrates, novel extraction methods, genetically enhanced species and mixed cultures with a view to make PHAs more commercially attractive.

Polylactic acid and Copolymers

The significance of polylactic acid as an emerging biodegradable polymer can see from the large number of reviews that have appeared on the system recently [88, 247 279]. (See discussion provided later).

Synthetic Biodegradable Polymers

Synthetic polymers, in general are non biodegradable. However, polymers with hydrolysable backbones such as polyesters [97, 119, 276, 280 282], polycaprolactone [51, 83], polyamides[282 290], polyurethanes [291 299], polyureas [300], polyanhydrides [301 306], poly(amide-enamine)s [96], poly(ester amide)s [96], poly(orthoesters) [307], poly(phosphoesters) [308, 309] and vinyl polymers containing easily oxidizable functional groups (e.g., poly(vinyl alcohol), poly(vinyl esters) and polyacrylates) are susceptible to hydrolysis and enzymatic biodegradation [310, 311]. These polymers have interesting properties and potential applications. The cost of production is the main barrier for the wide utilization of these polymers. Structural analysis indicates that a linear polyamide backbone is key to the total biodegradability of the acid-catalyzed polyaspartates. Polyesters represent one of the most promising families of biodegradable polymers which are used in fields such as packaging for industrial products, mulching for agriculture, or bioresorbable biomaterials for hard tissue replacement and controlled drug delivery systems. Their degradation products are fully nontoxic substances or the starting monomers that satisfy the environmental requirements. A few monomers such as 1,3-propanediol, 1,4-butane diol, γ-butyrolactone, adipic acid, n-methyl-pyrrolidone, succinic acid and lactic acid produced by fermentation from renewable resources are potential candidates for production of biodegradable polyesters.

The synthetic strategies involve (1) production of oligomeric or copolymeric α,β-dihydroxyl-terminated oligosuccinates, by thermal polycondensation of succinic acid with 1,3-propanediol/1,4-butane diol, or a mixture of 1,3-propanediol and 1,4-cyclohexanedimethanol. (2) Chain extension with via the chloroformate synthesis, affording polyester-carbonate (PEC), and (3) Production of hydrophobic/hydrophilic segmented copolymers between α,β-dihydroxyl-terminated oligo(propylene succinate)s and poly(ethylene glycols) (PEG) or alicylclic polyols of different molecular weights were synthesized, affording poly(ester ether-carbonates) terpolymers [70, 88, 97]. An alternative to the aliphatic/aromatic polyester approach is the design and use of cycloaliphatic or aliphatic/cycloaliphatic copolyester.

As a carbon-intermediate chemical, fermentation-derived succinate has the potential to supply over 2.7 × 108 kg industrial products/year including: 1,4-butanediol, tetrahydrofuran, α-butyrolactone, adipic acid, n-methylpyrrolidone and linear aliphatic esters. Succinate yields as high as 110 g/l have been achieved from glucose by the newly discovered rumen organism *Actinobacillus succinogenes* [97, 312]. Succinate fermentation is a novel process because the greenhouse gas CO2 is fixed into succinate during glucose fermentation. Succinic acid can be produced by *Anaerobiospirillum succiniciproducens*, using glucose, but even lactose, sucrose, maltose and fructose as carbon sources. New developments in end-product recovery technology, including water-splitting electrodialysis and liquid/liquid extraction have lowered the cost of succinic acid production to U.S. $ 0.55/kg at the 75,000 tonne/year level and to $ 2.20/kg at the 5,000 tonne/year level.

Biodegradable Polymer Blends

The blending of biodegradable polymers from renewable and non-renewable resources is a method of reducing the overall cost of the material and offers a method of modifying both properties and degradation rates [14, 21, 67, 98, 186, 313, 314] natural polymers, such as starch, protein and cellulose; (2) synthetic polymers from natural monomers, such as polylactic acid; and (3) polymers from microbial fermentation, such as polyhydroxybutyrate are described with an emphasis on potential applications. The hydrophilic character of natural polymers has contributed to the successful development of environmentally friendly composites, as most natural fibers and nanoclays are also hydrophilic in nature. More recently there has been an increasing interest in starch based biodegradable blends. The melt processability, humidity resistance, and mechanical properties of starch can be improved by blending starch with synthetic polymers such as PVA, PLA, PCL, and PHAs etc. to make these materials suitable for the production of biodegradable films, injection molded items and foams. The strategy adopted for preparation of biodegradable polymer blends usually involves blending a thermoplastic resin with a biodegradable one. The blending must produce fine enough dispersion that, after disintegration of the biodegradable part, the remaining thermoplastic part will not contaminate the environment.

Need of Polyethylene Biodegradation

Plastics, made from petrochemical feed stocks are portrayed by the "green" lobby as ecologically undesirable, because they are made from fossil carbon resources and it is assumed that they are very resistant to biodegradation in the environment. It is ironical that the very physical property of durability, that have made the polyolefin so commercially successful in packaging and other agricultural applications, is a disadvantage, when the material appears in the waste stream [315, 316]. The environmental impact of persistent plastic wastes is growing more global concern, and alternative disposal methods are limited. Incineration may generate toxic air pollution, and satisfactory landfill sites are limited. Recycling is, at present, only viable for high cost, low volume specialty plastics [317, 318]. The high cost or the lack of commercially acceptable performance of fully biodegradable polymers available until recently has meant that they have never replaced traditional non-degradable plastics in the mass market [319, 320]. Hence, it is increasingly felt that the best solution for rectifying the environmental pollution caused by the non-biodegradability of existing commercial plastics would be making them biodegradable.

Polyethylene Modifications to Facilitate Biodegradation

The methods adopted to facilitate polyethylene disintegration and subsequent biodegradation are discussed below [21]:

Insertion of Week Links into Vinyl Polymer

Particular emphasis on this approach of insertion of 'weak links' on non biodegradable polymers has been placed on two types of polymer modifications: namely the insertion of functional groups in the main chain, especially ester groups (Fig. 5), which can be cleaved by chemical hydrolysis, and the insertion of functional groups in or on the main chain that can undergo photochemical chain-cleavage reactions, typically carbonyl groups (Fig. 6). On irradiation with ultraviolet light, the activated ketone groups present can take part in two different types of free radical bond-breaking reactions referred to as Norrish I and Norrish II reactions (Fig. 7).

ECM Comb. Mot. to Exclude
Exh. Z

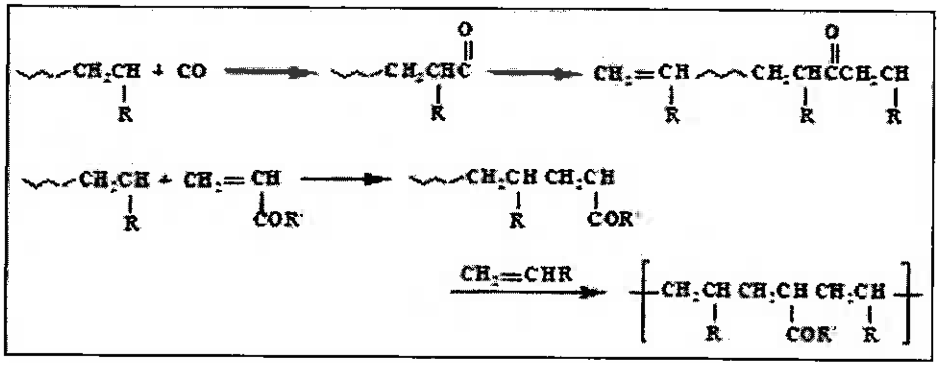

Fig. 5 Insertion of ester group into vinyl polymer

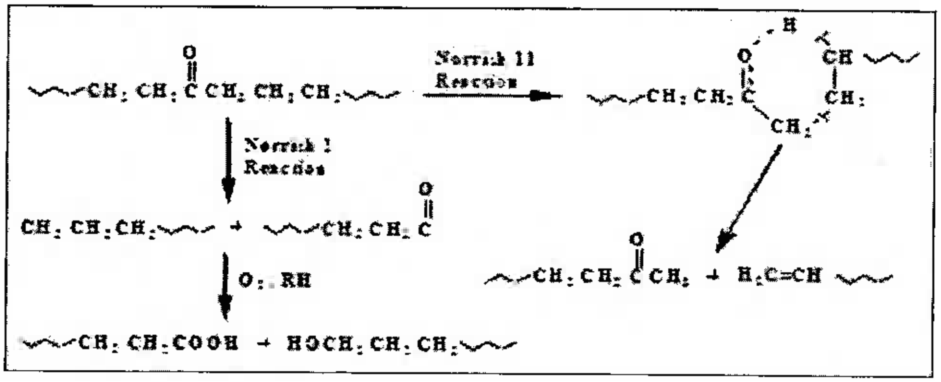

Fig. 6 Insertion of ketone group into vinyl polymer

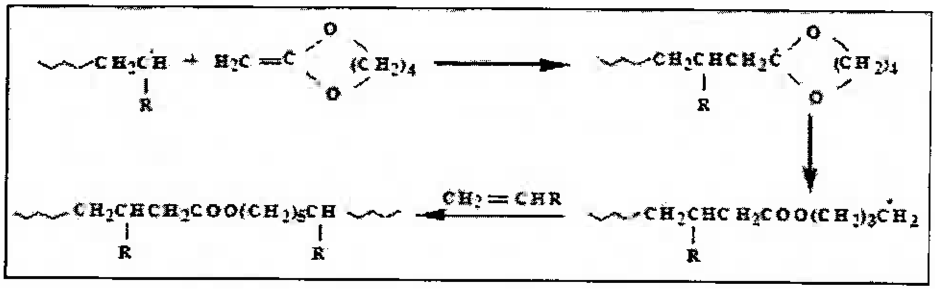

Fig. 7 Norrish I and Norrish II reaction mechanisms for the degradation of copolymers of ethylene

Compounding of Polymer with Pro-Oxidants and Photosensitizers

The thermal (Δ) and/or photolytic (hυ) pre-abiotic treatment, which constitutes the major route for promoting the eventual biodegradation of polyethylene, could be enhanced by using pro-oxidant additives [321, 322]. The pro-oxidants most generally used for this purpose are divalent transition metal salts of higher aliphatic acids, such as stearic acid and transition metal complexes such as dithiocarbonates as photosensitizers or photoinitiators [323 325]. Soaps of transition metal salts such as Mn, Co, Zn, Ag, Co, Ni, Fe, Cr, V and Cu were reported to act as effective oxidation catalysts when combined with unsaturated elastomers as autooxidizable substances [321 323]. During photo and thermo oxidative degradation the material degrades by a free radical chain reaction involving the formation of hydroperoxides, carbonyl groups and finally to low molecular mass oxidation products such as carboxylic acids, alcohols, ketones, esters and low molecular mass hydrocarbons (Fig. 4) [326]. Peroxidation and carbonyl group formation also lead to hydrophilic surface modification friendly to microorganisms that are thus able to bioassimilate the low molar mass oxidation products [327, 328].

Transition metals catalyze the hydroperoxide decomposition step of the oxidation mechanism according to the reaction given below (Fig. 8) [322, 329]. The catalytic activity of the metal was reported to correlate with the redox potential of the metal and requires several oxidation states of comparable energy for the metal. The peroxide decomposition products react further to yield volatile low

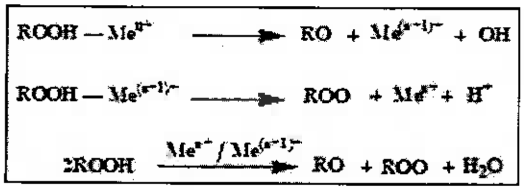

Fig. 8 Reaction of metal catalysts with hydroperoxides

molecular weight fragments. Unsaturated compounds, which are very prone to oxidation, generate free radicals much more readily than saturated groups. These free radicals catalyze the initiation step of LDPE degradation by transforming LDPE from RH into free radical $R^{\cdot}$.

Blends of Biodegradable and Non Biodegradable Polymers

The most frequently adopted approach to degradability design of LDPE has been to introduce pro-degradant additives such as starch and cellulose into synthetic polymers. According to "percolation theory and experiments" a large amount of starch of the order of 30% by volume needs to be blended for improving the accessibility of the whole system to different degrading factors [330, 331]. At high starch content, the incompatibility of LDPE and starch due to the different polar character will affect mechanical properties of the blend negatively. By using compatibilizers or chemically modified starch and polyethylene the amount of incorporated starch as well as the mechanical properties of the blend could be increased. It was found that the compatibilized blends have only a slightly lower biodegradation rate compared to the uncompatibilized blends [332]. The rapid enzymatic hydrolysis of starch under biotic exposure conditions produces a plastic film with a porous structure that will further evoke environmental problems [322]. Incorporation of pro-oxidants containing transition metal ions and unsaturated elastomers as autooxidizable substances along with starch in LDPE/starch blends was found to increase the degradation of LDPE. Where as starch behave as antioxidant by effectively blocking the free radicals and forming inactive species [333, 334].

Grafting of Natural Monomers Onto Nonbiodegradable Polymers

A search on available literature on biodegradable polymers reveals a number of attempts to achieve biodegradability, but grafting of naturally available carbohydrate monomers which can induce biodegradation onto polyolefins has not been given much emphasis. Successful methods of preparation of biodegradable polyethylene-natural monomer graft composites are limited. Grafting of natural monomers such as cardanol, twelve hydroxy stearic acid, vanilic acid etc. onto polyethylene can give rise to grafted polymers in which the biodegradable component is evenly distributed such that biodegradation can proceed smoothly. Grafting of the natural monomers onto polyethylene will be conducted by irradiation polymerization or ionic polymerization. Anchoring of simple sugars such as monosaccharide or disaccharides instead of starch onto polyethylene can reduce the negative effects on physical properties observed in the case of polysaccharide grafting [335].

Historical Perspective

The potential degradability and ultimate biodegradability of polyethylene has been started to consider in the early 1970s as specific attributes for applications in packaging and agricultural market segment. In 1972 Potts and his collaborators reported that polyethylene was not biodegradable, but the linear paraffin molecules below a molecular weight of about 500 were utilized by several microorganisms [336]. Later Hueck emphasized the role of criteria for the inertness of polyethylene as its hydrophobic nature and its large molecular dimensions [337]. Otake et al. observed the signs of degradation in pure polyethylene film during soil burial for 32 years [338]. This suggests that the PE film could not be degraded significantly. Albertsson studied the oxidative effects of microbes and abiotic forces on the degradation of PE by measuring the conversion of ^{14}C present in the HDPE film to respiratory $^{14}CO_2$ during a 2 year aerated cultivation with soil or with *Fusarium redolens*. She reported that the effective oxidation of somewhat longer polyolefin chains was done by microbes than abiotic forces [339, 340].

In 1978 Griffin had first presented the idea of using granular starch as filler in polyethylene in order to increase the biodegradability of the resulting material. Where as the opposite polarity of starch and LDPE affected the mechanical properties of the blend negatively [341]. Since that time several techniques in which starch is used as a biodegradable additive have been patented [341 351]. Peanasky et al. investigated the accessibility of starch in PE starch blends by computer simulation, percolation theory, and acid hydrolysis experiments. If the concentration of starch (*p*) is less than the percolation threshold concentration ($pc = 31.17\%$)., a small amount of starch is removed from the surfaces only, but at $p < . pc$ connected pathways existing throughout the bulk of the material facilitate large amounts of starch extraction [331]. Goheen and Wool reported a similar conclusion in the biodegradation of PE starch blends in soil over a period of 8 months [352]. They did not observed any significant chemical changes in the polyethylene matrix after 240 days. Arvanitoyannis et al. reported that the presence of high starch contents (>30%, w/w) had an adverse effect on the mechanical properties of LDPE/rice or potato starch blends [353]. Composites containing various forms of starch/LDPE blends were prepared by several teams to study the effect of starch content on mechanical properties and water absorption [354 356]. High starch contents promoted brittleness and resulted in lower tensile strength, elongation at break, and modulus and higher GP and WVTR. Tsao et al. studied the influence of soil macro invertebrates such as earthworms, cockroaches, termites, sowbugs, and crickets on primary biodegradation of starch-containing polyethylene films [357]. The degree to which the films were attacked and consumed by macroinvertebrates was directly related to the starch content of the film. Doble et al. have reviewed recently biodegradation of polyethylene and polypropylene giving emphasis on the mechanism of biodegradation, the various bacterial and fungal organisms that have been reported for the same, methods adopted for the studies and different characterization techniques followed to measure the extent of degradation [358].

The modification of either the starch or LDPE is necessary to make compatibilized starch/LDPE blends, which in turn increase the mechanical properties and the amount of starch in the blend. Cornstarch treated with a siliane coupling agent was used by Griffin to provide compatibility with hydrocarbon polymers [359]. Kang et al. used a modified starch (MS) to improve the mechanical property of starch-filled polyethylene films by introducing cholesterol moiety as a hydrophobic biomaterial [360]. However, a decrease in the enzymatic degradation rate of the MS-filled LDPE than the rate of the native starch-filled LDPE was observed. Another approach is graft copolymerization of a vinyl monomer on to the starch backbone. Fanta and Doane have made an extensive study of the synthesis and properties of starch-g-poly(methyl acrylate) [161]. Patil and Fanta prepared S-g-PMA copolymers containing 55 60% PMA from cornstarch, high amylase cornstarch, and waxy cornstarch with ceric ammonium nitrate initiation [361]. Henderson et al. grafted PMA onto wheat starch by γ-irradiation and chemical initiation, respectively [362]. Dennenberg et al. prepared S-g-PMA copolymers having grafted side chains with molecular weights of less than 500,000 [363]. Although Dennenberg et al. confirmed that the starch portion of these graft copolymers is indeed susceptible to fungal attack, PMA is resistant to biodegradation. Jane et al. reported that the tensile strength and elongation of starch/PE composites was improved by the addition of oxidized PE (OPE) [364, 365]. Tena-Salcido et al. used thermoplastic starch to impart biodegradability to PE. PE1/TPS blends having 32% starch remained ductile after 45 days of exposure to bacterial attack [366].

Another approach to bring about some compatibility in starch and synthetic polymer is blending starch with polymers containing polar functional groups such as ethylene acrylic acid (EAA) copolymer, ethylene maleic anhydride (MAH) copolymer, ethylene vinyl alcohol (EVA) copolymer etc. that can interact with starch. Of all these compatibilizers, EVA is a highly hydrophilic polymer, which restricted its application in moist environments. EAA is a very effective compatibilizer between starch and polyethylene, but only a high content of EAA could result in good compatibility. However, the higher the EAA content was, the lower was the biodegradability. So far, PE-g-MAH was considered as the most effective compatibilizer between starch and polyethylene and only very small amount of MA is needed to attain a large improvement in mechanical properties [367 372]. But, its price is high and the manufacture is difficult. Otey et al. produced blown films containing up to 40 50 wt% gelatinized starch along with EAA and ammonia [182, 373]. Bikiaris et al. reported that the tensile strength of LDPE/PLST blends containing high amounts of starch (up to 30 wt%) could be retained to a great extend the tensile strength of LDPE by using PE-G-MA copolymer as a compatibilizer [332, 374]. The biodegradation studies showed a slightly lower biodegradation rate for the compatibilized blends compared to their uncompatibilized counterparts. Later Shujun et al. observed better mechanical properties, morphology properties, thermal properties and rheology properties in the case of TPS/LLDPE blends compatibilized with MAH compared with the blends without MAH [371].

Several teams have reviewed the progresses made in the synthesis of biodegradable polyethylene [21, 56, 359, 375 377]. In a series of works, Bikiaris et al. used ethylene-co-acrylic acid (EAA) as compatibilizer in LDPE/plasticized starch (PLST) blends [321, 334]. They reported that EAA increases the thermal oxidative degradation of LDPE, where as PLST inhibits it. Sastry et al. synthesized films of polyethylene (PE) starch blends containing vegetable oil as a compatibilizer [378]. Kiatkamjornwong et al. used cassava starch-g-poly(acrylic acid) prepared by γ- ray irradiation which was further modified by esterification and etherification with poly(ethylene glycol) 4,000 and propylene oxide, respectively, for a biodegradable LDPE sheet. They reported that hydrophobically modified starch/LDPE blends have better mechanical, thermal and morphological properties as well as better biodegradability than LDPE/native starch [379].

The thermo-oxidative aging studies done by Sharma et al. revealed that the incorporation of pro-oxidant which consisted of metal salts (Manganese stearate) and unsaturated elastomer (SBR and ENR-50) enhanced the degradation rate of sago starch filled LLDPE composites [323]. Where as Khabbaz et al. reported that starch have an inhibiting effect on the thermal degradation of LDPE containing pro-oxidant and photosensitizers [380, 381].

Abd El-Rehim et al. observed an accelerating effect in biodegradation of LDPE/starch blend subjected to soil burial treatment after UV-irradiation [382]. Jakubowicz [383, 384], Bonhomme et al. [385], Chiellini et al. [315] and Manzur et al. [386] have done the biodegradation of thermally-oxidized polyethylene to evaluate their ultimate biodegradation (e.g., mineralization) in soil. They observed bioassimilation of PE films after its thermo-oxidative degradation into low molar mass products. More recently Raghavan et al. prepared starch/PE/poly(lactic acid) composites [387].

An economical and commercially viable approach to form graft or block copolymers is in situ reactive blending by using polymers containing reactive functional groups so that reaction occurs during melt blending, which improves the compatibility and interfacial adhesion of the two immiscible polymers. Chandra and Rustgi reported the synthesis and biodegradation studies of MA-g-LLDPE/starch blends. Biodegradation studies showed only the degradation of starch, while the MA-g-LLDPE matrix remained intact [320]. It has, however, been noted that the starch gets enzymatically hydrolyzed in a biologically active environment leaving the polymer in a disintegrated form. It is also now well understood that the molecular weight of LDPE remains unaffected even after the complete degradation of starch causing further environmental pollution [322]. A combined methodology of in situ oxidative modification of polymer backbone by introducing >C = O groups followed by grafting of simple sugars was tried by our team [335] to prepare a sugar grafted LDPE (Su-g-LDPE). They reported that the increase in the amount of grafted sugar moieties in the polymer enhances intermolecular interactions such as hydrogen bonding at higher grafting percentages giving rise to improved mechanical properties. During biodegradation in soil environment, the microbial oxidation of LDPE backbone was established by observing a decrease in carbonyl index of Su-g-LDPE with time of degradation.

Grafting Reactions on to Polyethylene to Improve Biodegradability

Sugar End-Capped Polyethylene: Melt Phase Grafting of Glucose on to Polyethylene

A combined methodology of in situ oxidative modification of polymer backbone by introducing >C = O groups followed by grafting of simple sugars is expected to be an another interesting technique to graft sugar/carbohydrate molecules that can reduce the negative effects observed in the case of polysaccharide grafting. Low density polyethylene (LDPE) was modified to introduce biodegradability by grafting highly hydrophilic monomers (which can act as nutrients for microorganisms) such as glucose by a novel melt phase reaction in Brabender plasti-corder in the presence of ceric ammonium nitrate (CAN) to obtain 4-O-hydroxymethyl D-arabinose (Sugar) end-capped LDPE (Su-g-LDPE) at a maximum grafting of 16%. The increase in the amount of grafted sugar moieties in the polymer enhances intermolecular interactions such as hydrogen bonding at higher grafting percentages giving rise to improved mechanical properties. The biodegradability of Su-g-LDPE was carried out by soil burial test and by optical density measurements in presence of an aerobic bacterium *Pseudomonas sp.* During biodegradation studies in compost soil environment a decrease in ketone carbonyl index (CI (ν_{1719}/ν_{1465})) of Su-g-LDPE with time of degradation was observed, indicating the initiation of LDPE chain degradation (Fig. 9). A stepwise cleavage of LDPE from the chain ends can be envisaged with microbes consuming the sugar units on the surface of Su-g-LDPE and further oxidizing the ketonic carbonyl to carboxylic acid. The microbial degradation resulted in the increase in crystallinity and the decrease in viscosity of the degraded samples. SEM showed surface erosion caused by microbial degradation on Su-g-LDPE (Fig. 10) [335].

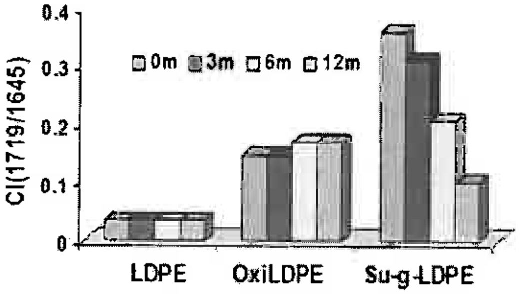

Fig. 9 Carbonyl index of LDPE samples subjected to biodegradation

Grafting of Vinyl Monomers

Kaur et al. report that gelatin grafting improved biodegradation [388]. They modified PE through the graft copolymerization of vinyl monomers such as acrylamide (AAm) and acrylic acid (AAc) by a phase-transfer-catalyst method to impart biodegradability. The grafting percentage of AAm and AAc for PE was found to be dependent on the dibenzoyl peroxide concentration, monomer concentration, time, temperature, and concentration of the phase-transfer catalyst. Some AAm- and AAc-grafted PE samples were prepared by chemical, UV, and γ-radiation methods. The biodegradation of samples of PE, PE-g-polyacrylamide, and PE-g-poly(acrylic acid) prepared by all these methods was studied. The weight loss of the samples over a period of time was observed with soil-burial tests. The grafted samples prepared by the phase-transfer-catalyst method

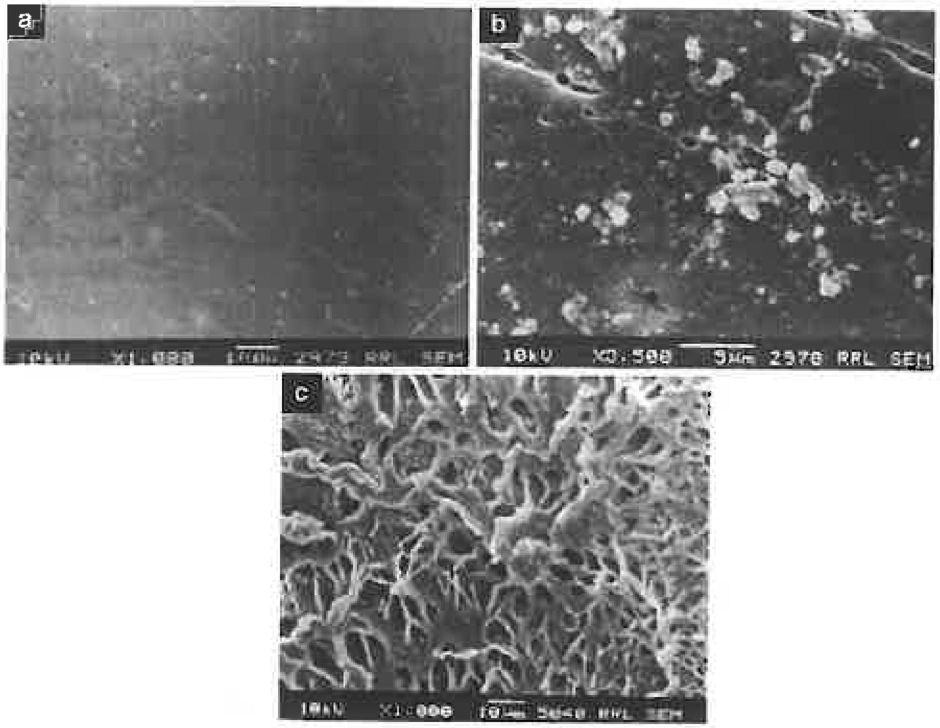

Fig. 10 SEM micrographs of Su g LDPE: (**a**) before biodegradation, (**b**) after 4 months biodegradation, (**c**) after 12 months biodegradation

showed better biodegradation results than those prepared by other methods of grafting. Simialrly, Maharana and Singh investigated graft copolymerization of low-density polyethylene (LDPE) onto starch using glucose-cerium(IV) redox initiator in an aqueous sulfuric acid medium under nitrogen atmosphere [389]. The graft yield was influenced by various parameters like reaction time, temperature, and concentrations of acid, glucose, polyethylene (PE), starch, and initiator. A maximum graft yield of 85.66% was obtained at a temperature of 50 °C and at higher concentration of starch. Biodegradability of starch-grafted PE has been tested applying soil-burial test.

Low-density poly(ethylene) (LDPE) was modified by graft copolymerization of acrylonitrile/acrylic acid by El-Arnaouty et al. who showed that that the increase of AAc ratio in the composition increases the hydrophilicity of the films and the degradation rate [390]. In comparison, they found that PET polymer is generally found to be more resistant to the biodegradation than LDPE in the two types of soils tested. Highest degradation rate was found to be achieved using agricultural soil. It is found that the isolated strains belonging to the genus Pseudomonas were mainly responsible for the degradation of both polymers. Ghosh et al. also report preparation of starch-based biodegradable low-density polyethylene (LDPE) films by graft copolymerization of vinyl acetate with ceric ammonium nitrate (CAN) in aqueous acidic medium as redox initiator with nitric acid [391].

Copolymers and Blends

Several groups have investigated copolymer formation and blends for improving the biodegradability [392 400]. High energy irradiation is reported to enhance biodegradation as shown by Senna et al. [401]. Cassava mesocarp carbohydrate and its modified form were used as fillers in low density polyethylene to give plastic films that were biodegradable [396]. It was found that the tensile strength of the films decreased with an increase in the amount of the filler incorporated. The water absorption results of the films showed that modification of the cassava mesocarp carbohydrate made it hydrophobic and therefore more compatible with the polyethylene.

Rutkowska et al. report very little microbial degradation was observed for polyethylene blends in the sea water, which was explained by low sea water temperatures, moderate biological activity of microorganisms and low amounts of solar radiation reaching the films [397]. The introduction of pro-degradant additive into the polyethylene improves biodegradability of this blend in natural aqueous biological environment. Figure 11 shows weight changes of polyethylene after incubation in the Baltic Sea water indicating biodegradation of the starch containing batches [397].

Moura et al. prepared biodegradable polymeric materials based on blends of a synthetic high density polyethylene (HDPE) and biodegradable polymers such as polylactic

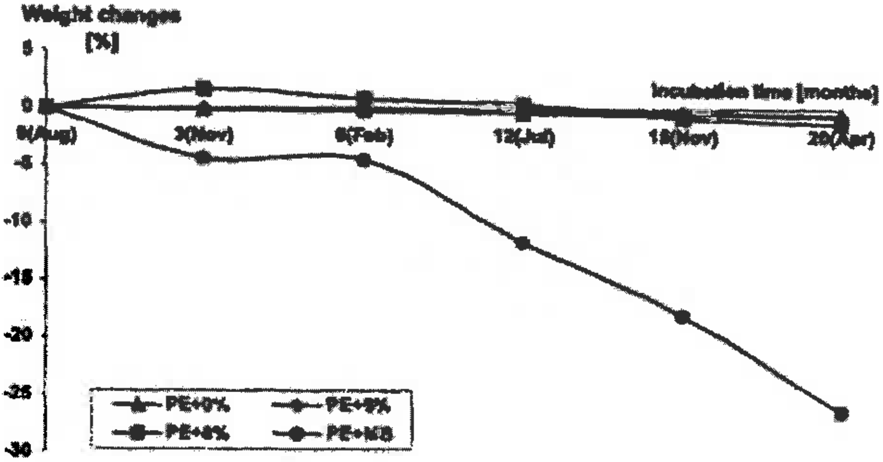

Fig. 11 Weight changes of polyethylene after incubation in the Baltic Sea water. (Reproduced from Ref. [397])

acid (PCL) and poly(caprolactone) (PLA), in a co-rotating twin-screw extruder [400]. A polyethylene modified with maleic anhydride was used as compatibilizer. The mechanical results showed that the addition of PLA improves the blends stiffness while the addition of PCL leads to materials with a greater elongation at break and a lower Young modulus. This feature is related with the mechanical properties of each material as well as the adhesion between them. Concerning the biodegradability tests, it was found that HDPE/PCL blend presents the highest degree of biodegradability.

Chemistry of Polyethylene Biodegradation

Biodegradation of polyethylene involve a complex interaction of abiotic and biotic mediated oxidative processes [402 404]. The carboxylic acids formed by the abiotic reactions undergo β-oxidation, where two carbon fragments are removed from the carboxylic molecule by reaction with coenzyme A, leaving behind a still activated acid. The activated acid repeatedly participates in the same process until the whole chain is split into two carbon fragments (Fig. 12). The two carbon fragments enter the citric acid cycle, from which carbon dioxide and water are released [405 408]. The various mechanisms, which include photo-oxidative degradation, thermal degradation, ozone-induced degradation, mechanochemical degradation, catalytic degradation and biodegradation, are discussed in detail by Shah et al. and Zheng et al. [409, 410].

It appears there is a limit in enhancing the biodegradability of PE. Incorporation of photosensitizers is considered a mechanistic way by which one may approach this problem. But, this method has to be in combination use biodegradable blend components. This was shown by Wiles et al. when they used antioxidants and stabilizers, developed to increase the durability of polyolefins, in combination with prooxidant transition metal complexes to provide industrial products with widely variable but controlled lifetimes [411]. The low molar mass oxidation products formed during photo-oxidation and thermal oxidation are biodegradable and oxo-biodegradable polyolefins are now widely used in agricultural applications and in degradable packaging as examples.

Polylactide, a Dominating Biopolymer

There is a growing interest in utilizing renewable resources as a functional biopolymer in a broad range of scientific areas such as biomedical, agricultural, food, cosmetics, wastewater management and environmental field [77, 98, 408]. Polylactides (PLA) are considered as the most versatile material among biodegradable polymers because of its inherent biodegradability, biocompatibility, high mechanical strength and Young's modulus comparable with those of polyethylene and polystyrene and the easy availability from renewable agricultural sources.

Recent developments indicate a great increase in the publication of polylactide (PLA) polymers spurt in the [412 418]. These attributes make them a leading candidate in biomedical and pharmaceutical industries as a resorbable implant material, wound closure, bone fixation devices and a vehicle for controlled drug delivery [416 418]. It is also used as an environment friendly plastic, although their market is still limited due to its higher cost and slow degradation rate as compared to the waste accumulation rate [419, 420]. However, their clinical applications are sometimes affected by the high hydrophobic and consequent poor water uptake, which results in a slow hydrolytic degradation rate [2, 421]. Another potential disadvantage is the complications resulting from the accumulation of lactic acid produced in the process of PLA degradation, and its poor processability [422, 423]. Ultra high molecular weight is required for processing PLA into strong fibers. PLA is

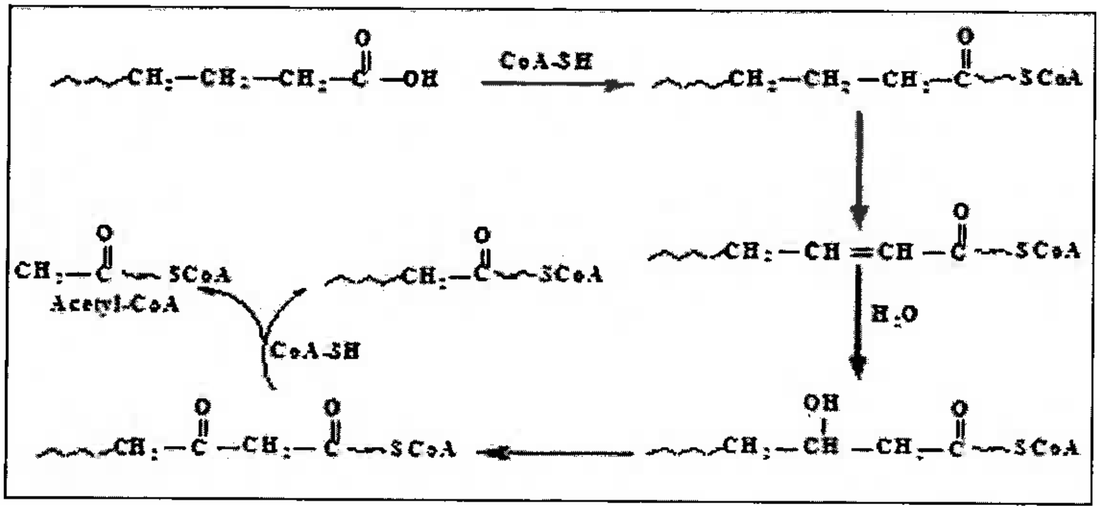

Fig. 12 Biotic conversion of carboxylic acids (β oxidation) [407, 408]

belonging to the group of thermally less stable polymers and have poor physical properties and high cost of production [424, 425]. Copolymerization of lactide with other comonomers and/or polymers is used to modify the properties of PLA and to control its degradation behavior suitable for the specific applications in the field. Its immense applications in a number of growing technologies such as orthopedics, drug delivery, sutures, and scaffolds have enhanced the interest of researchers in this novel area [425].

Lactic Acid

Lactic acid or 2-hydroxypropionic acid is an "Alpha-Hydroxy-acid" (AHA) and was discovered by the Swedish Scientist Scheel in 1780 being first isolated from sour milk. This gave rise to a whole series of claims and counter claims in the academic literature concerning Scheels' findings. The French scientist Fremy produced lactic acid by fermentation and this gave birth to industrial production in 1881.

Lactic acid exists as in racemate form, made up of stereo-isomers as shown in Fig. 13. The isolated acid is a white crystalline solid which is hygroscopic. As this is the case it is typically manufactured and consumed as a concentrated solution of around 88% weight. In this form lactic acid exists not only as individual molecules, but oligomers and polymers. In addition, lactic acid can self-esterify providing a mixture of esters in equilibrium with acid and oligomers. Hence, purifying lactic acid can be a challenge. Synthetic lactic acid made from petrochemical feedstocks is optically inactive, i.e., a racemate mixture. Lactic acid made biochemically by fermentation is optically active and suitable organisms can selectively produce laevo- or dextro- rotatory enantiomers. However, salts of lactic acid tend to be found in the laevo state only [264, 274].

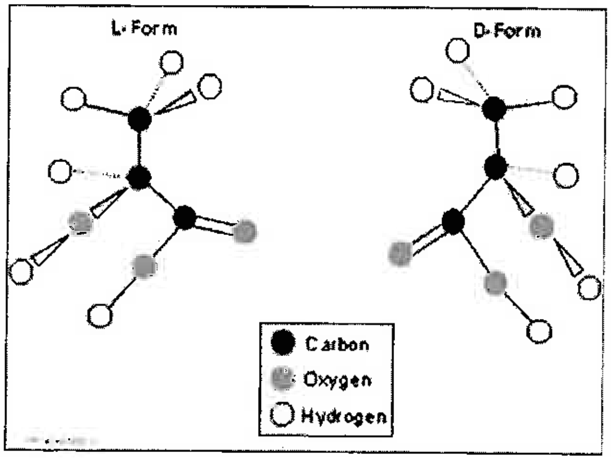

Fig. 13 Lactic acid stereo isomers

Structural Aspects

The presence of asymmetric carbon atoms generates structural particularities, which make lactic acid-derived polymers rather special when compared with other polymers. Because of the chirality of the lactyl unit, the lactic acid cyclic dimers exist in three diastereoisomeric forms, i.e., L-lactide, D-lactide and meso-lactide, the latter containing a L-lactyl unit and a D-lactyl one in the ring. The equimolar mixture of L- and D-lactide is referred to as racemic DL-lactide [2, 417]. The thermal, mechanical, and biodegradation properties of lactic acid polymers are known to depend on the choice and distribution of stereoisomers within the polymer chains. High-purity L- and

D-lactides form stereoregular isotactic poly(L-lactide) (PLLA) and poly(D-lactide)(PDLA), respectively. These are semicrystalline polymers (crystallinity around 37%) with a high melting point (Tm ~180 °C) and a glass transition temperature in the 55 80 °C range. These types of materials exhibit high tensile strength and low elongation, and consequently have a high modulus that makes them more suitable for load-bearing applications such as in orthopedic fixation and sutures [412, 426 428]. PLLA finds a wide range of uses, from biomedical implantation devices, e.g., sutures, rods, plates and pins, through artificial skin and drug delivery agents to environmentally benign film and fibers for packaging and clothing [427, 428]. The *meso-* and DL-lactide, on the other hand, form atactic poly(DL-lactide) (PDLLA) which is an amorphous transparent material with a glass transition temperature of 50 ± 60 °C. It can be used for the production of transparent films and glues. This material has lower tensile strength, higher elongation, and a much more rapid degradation time, making it more attractive as a drug delivery system [412]. The degradation time of PLLA is much slower than that of DLPLA, requiring more than 2 years to be completely absorbed. The presence of CH_3 side groups impart a hydrophobic nature to PLLA. Copolymers of L-lactide and DL-lactide have been prepared to disrupt the crystallinity of L-lactide and accelerate the degradation process. The methyl substituent's steric shielding effect of the ester group reduces the hydrolytic degradation rate of PLLA on comparing with that of other polylactones [67].

Synthetic Methods of PLA

Lactic acid polymers are prepared by two quite different ways: (1) polycondensation (condensation polymerization) of lactic acids and (2) ring-opening polymerization of lactides [429, 430]. Polycondensation method is relatively simple and inexpensive, but it has several disadvantages. Only low-molecular-weight polymers can be derived by the direct condensation of the corresponding alpha-hydroxy acids. The molecular weight, the molecular weight distribution and the end groups are difficult to control and syntheses of copolyesters with controlled sequences are impossible. Therefore, high molecular weight PLA are prepared by the ring-opening polymerization (ROP) of lactides. The ROP of lactides can be induced by four different classes of initiators and reaction mechanisms [10, 412, 429 433].

(a) cationic polymerizations,
(b) anionic polymerizations,
(c) coordination-insertion mechanisms,
(d) enzymatic polymerizations.
(e) solid state polymerization.

Extremely strong acids or carbenium ion donors are capable of initiating a cationic polymerization of lactides. The cationic mechanism consists of a protonation or alkylation of the carbonyl O-atom (exocyclic oxygen) with the consequence of an electrophilic activation of the O-CH bond. This bond is then cleaved by the nucleophilic attack of another monomer, a process which is repeated in every propagation step until a monofunctional nucleophile (e.g., water) causes a termination step [431]. (Fig. 14). This mechanism involves a nucleophilic substitution at the chiral carbon, and it was found that optically pure poly(L-lactide) can be prepared only at temperatures ≤50 °C.At higher temperatures the cationic polymerization causes more or less racemization, which dramatically changes the physical and mechanical properties of the resulting polylactide. Where as the cationic polymerization is rather slow below 50 °C and only yields low to moderate molecular weights, and thus, this polymerization method is not attractive from the preparative point of view.

The anionic polymerization of lactides is initiated by alkali metal alkoxides. At higher temperatures phenoxides and carboxylates are also active. Both the initiation and the propagation step consist of a nucleophilic attack of an anion onto the CO-group of the lactide, followed by the cleavage of the CO O bond (Fig. 15). Therefore, the chain growth proceeds via an alkoxide, which is so basic that is, can also deprotonate the monomer in a-position. Due to the planarity of the delocalized anion this deprotonation/reprotonation reaction involves racemization. Hence, racemization is an unavoidable side reaction of anionic polymerization.

Coordination-insertion mechanism is based on metal alkoxides having a covalent metal-oxygen bond and the character of weak Lewis acids. The lactide plays temporarily the role of a ligand coordinated with the metal atom via the carbonyl O-atom [432, 433] (Fig. 16). This coordination enhances the electrophilicity of the CO-group and the nucleophilicity of OR-groups, so that an "insertion" of the lactone into the metal O-bond may occur. Typical initiators of this mechanism are the alkoxides of magnesium, aluminium, tin, zirconium, titanium and zinc. Another advantage of the covalent initiators is the easy control of the molecular weights via the monomer/initiator ratio. Furthermore, the risk of side reactions is much lower compared to ionic initiators so that much higher molecular weights can be obtained. Moreover, the covalent nature of these initiators significantly reduces the risk of racemization even at high temperatures. This aspect is important for the technical production of polylactides, because a final reaction temperature around 190 200 °C is needed to extrude the molten poly(L-lactide) from the reactor. In summary, the coordination-insertion initiators are for preparative purposes and far more useful than ionic initiators. The ring-

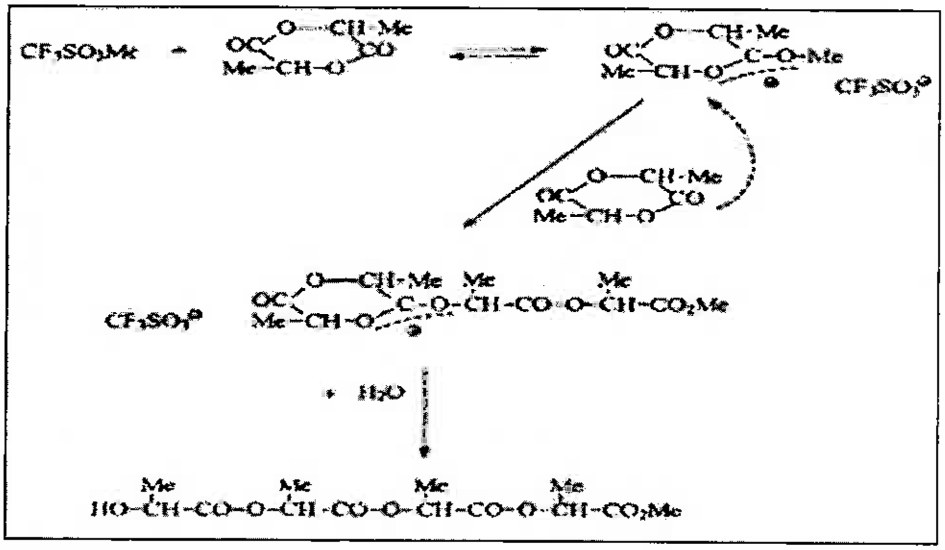

Fig. 14 Cationic polymerization of lactides

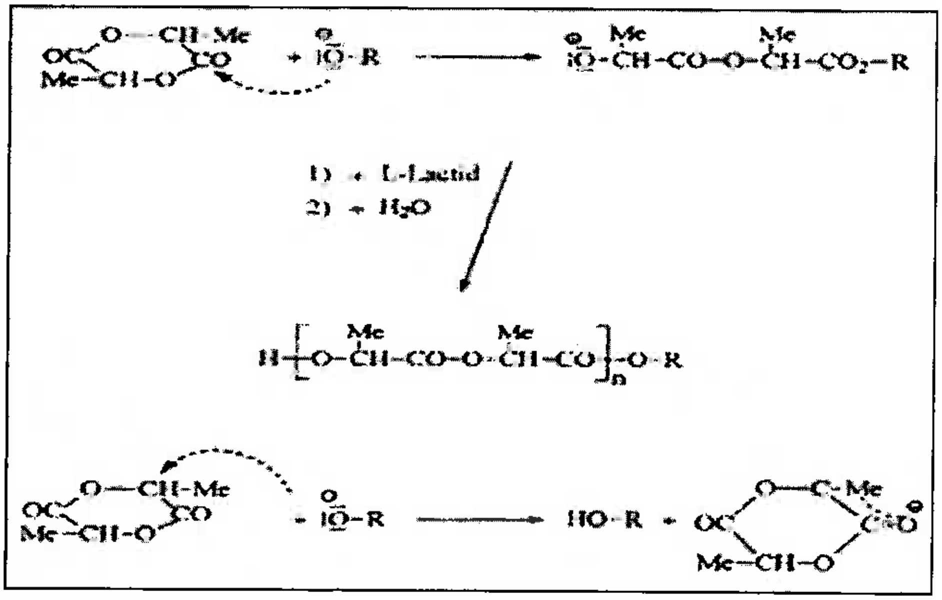

Fig. 15 Anionic polymerization of lactides

opening polymerization of lactide typically involves metallic promoters. Recent achievements report metal-free systems via organocatalytic (nucleophilic, cationic and bifunctional) as well as enzymatic approaches [434].

Enzymatic polymerization is an ecofriendly process of polymer synthesis and is based on using easily renewable resources as starting materials. Enzyme catalyzed polymerizations have several advantages over conventional chemical methods. These include (a) mild reaction conditions, i.e., temperature, pressure, pH, and absence of organic solvents, (b) high enantio and regioselectivity, and (c) recyclabiliy of catalysts [435].

Direct solution polycondensation of lactic acid has been reported recently, and the process is much simpler. However, the azeotropic solvent used in polymerization is not easy to purge. Therefore, direct polycondensation without solvents has attracted increasing interest [436]. Melt/solid state polymerization involves heating a semi-crystalline, solid prepolymer (of relatively low molecular weight) in powder, pellet, chip or fiber form up to a temperature below the melting temperature with the simultaneous removal of by-products from the surface of the material either (by volatilizing) under reduced pressure or with a carrier, for example, blowing inert gas [437, 438]. This method uses low operating temperatures and hence gives polymer of improved properties because of control over the side reactions as well as thermal, hydrolytic, oxidative degradations.

Catalysts have played a very important role in the production of PLA with controlled properties. A large number of catalysts have been studied for the ring opening

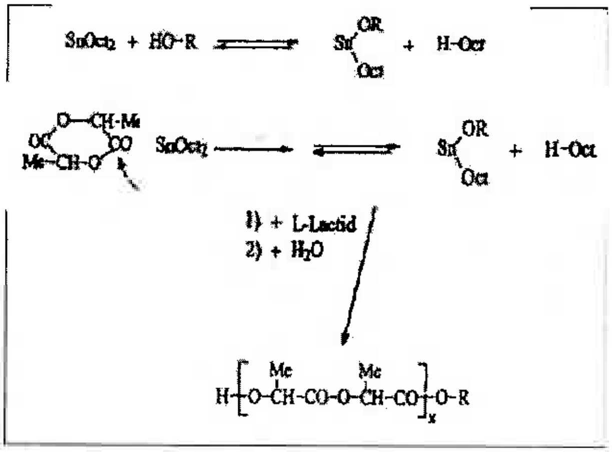

Fig. 16 Ring opening polymerization of lactides

polymerization of lactide [425]. Among the innumerable metal salts and complexes studied, organometallic compounds have been very effective in the synthesis of high molecular weight PLA. Gupta and Kumar have reviewed the catalyst performance in their recent review [425].

Properties of PLA

PLA is prepared by a variety of methods involving ring opening polymerization of lactide [429 436].

A set of unique properties such as good processability, biocompatibility and biodegradability make PLA an industrially important polymer for various applications. As indicated earlier, the chirality at α carbon of lactic acid gives rise to three isomers, L, D and DL isomers and hence three polymers, poly(L-lactic acid), poly(D-lactic acid) and poly(DL-lactic acid) which differ in their degradation rates, physical and mechanical properties. PLLA has a melting point of 170 183 °C and a glass-transition temperature of 55 65 °C while PDLLA has (Tg) 59 °C [203, 214]. Density of PLLA and PDLLA are 1.25 1.29 1.27 g/cc respectively [258]. PLLA is crystalline whereas PDLLA is completely amorphous biodegradable polymer. PLLA has more ordered and compact structure and hence it has better mechanical properties and longer service time. However, the annealed PLLA has better mechanical properties than un-annealed PLLA [425]. The mechanical properties are mainly dependent on the molecular weight, crystallinity, the method of production and processing conditions. The flexural strength of PLLA increased from 64 to 106 MPa on increasing of molecular weight of from 23 to 67 k, but the tensile strength remains the same at 59 MPa [439]. In the case of poly(DL-lactide) when molecular weight is increased from 47.5 to 114 k [252, 439], tensile and flexural strength increased 49 53 and 84 88 MPa, respectively [252]. The life cycle of the polymer from crops to the monomer, polymer and degradation to carbon dioxide is presented in Fig. 17. Table 1 gives a comparison of properties of PLA with PET and PS.

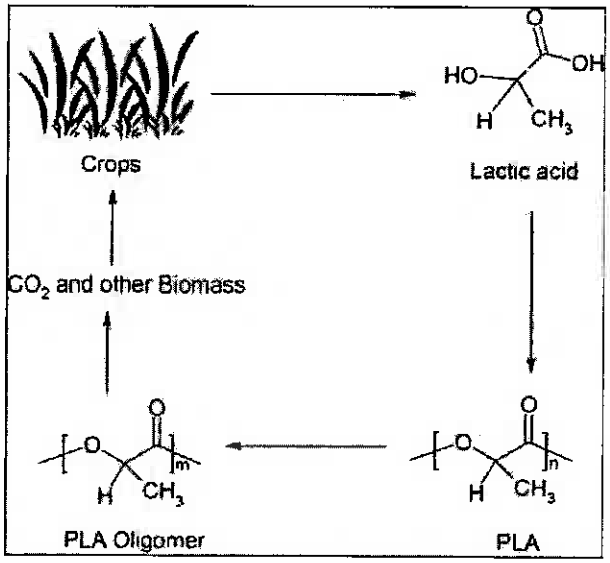

Fig. 17 Life cycle of PLA (adapted from Ref. [425] with permission from Elsevier)

Table 1 Comparison of properties of PLA with PS and PET [266, 440, 441]

	PLA	PET	PS
Modulus(GPa)	3.8	2.8 4.1	3.2
Density (kg/m3)	1.26	1.40	1.05
Modulus/density	2.8	1.71	
% Increase in stiffness	38.78		
Elongation at break (%)	4 7	2.8 4.1	3
Tensile strength (MPa)	59	57	45

Mechanism of Degradation of PLA

The physical and chemical structures of the polymers are the basic properties that affect the degradation and biodegradation mechanisms of PLA polymers [5]. PLLA can form highly crystalline structures that are known to be more difficult to hydrolyze than the amorphous parts of the polymer. The mechanism of hydrolytic degradation of PLA polymers is quite unusual. In general, a bulk erosion mechanism has been considered as the main degradation path way for PLA and its copolymers such as PLGA: random chain scission on the linkage of ester bonds in the polymer backbone proceeds homogeneously throughout the device. Recently it was reported that massive devices (>1 mm thick) of PDLA, PLA and PLGA degrade via a heterogeneous mechanism i.e., the degradation proceeds more rapidly in the center than at the surface. This was attributed to the autocatalytic action of the carboxylic acid end groups of degrading products which were trapped in the matrix [442, 443]. This heterogeneous degradation was assigned to the contribution of two phenomena: (1) easier diffusion of soluble oligomers from the surface into the external medium than from inside, and (2) neutralization of carboxylic end groups located at the surface by the external buffer solution [443]. Both phenomena contribute to reduce the acidity of the surface layer, where as in the internal part, the degradation rate is enhanced by autocatalysis due to carboxylic end groups. If the surrounding tissues cannot eliminate the acid by products of a rapidly degrading implant then an inflammatory or toxic response may result. However, the extent of heterogeneity depends on a number of factors such as the molecular characteristics of the polymer and the degradation medium [443]. Grizzi et al. showed that devices of small-size such as thin films and microspheres degraded homogeneously since no internal autocatalysis occurred [444]. The release of the soluble carboxyl-terminated oligomers depends on their solubility in the surrounding aqueous medium and thus factors like pH, ionic strength, temperature and buffering capacity become important [425].

Recently, there have been an increasing number of reports on the enzymatic hydrolysis of PLA [445 451]. These studies revealed that enzymatic degradation preferentially occurred in the amorphous regions of PLA. In addition, the disordered chain-packing regions of crystalline lamellae have been suggested to be preferentially degraded compared with the tight chain-packing regions [450, 452]. Enzymatic hydrolysis of PLA proceeds mainly via the surface erosion mechanism and the surface area of polymer materials would have a great influence on the enzymatic degradation [451]. Abiotic hydrolysis is known to participate at the initial stage of microbial biodegradation of PLA in nature. The released degradation products could be consumed by microorganisms [419, 453].

PLA Based Copolymers

In 1932, Carothers demonstrated the synthesis of poly(L-lactide) (PLLA) by ring opening polymerization of lactide [454]. Since that time PLA has been used as a bioabsorbable material in the medical and pharmaceutical fields [455 457]. However, the application scope of PLA is limited because of the certain weaknesses mentioned above. Copolymerization of L-lactide with other monomers has been recognized as an important tool to modify PLLA suitable for specific applications in the field [458]. The most employed comonomers are D-lactide [459], meso-lactide [460, 461], glycolide [462 466], caprolactone (ε-CL) [467 470] and trimethylene carbonate (TMC) [472]. Grijpma and Pennings have done a detailed study on the synthesis, thermal properties and hydrolytic degradation of these copolymers [471]. Glycolide copolymers are much more hydrophilic than PLLA, while ε-CL and TMC reduce the glass transition temperature of the L-lactide copolymer. All comonomers decrease the crystallinity of the polylactide and provide optimal mechanical properties to the materials.

Another approach for enhancing the hydrophilicity and degradation rate of polylactide was the copolymerization with other polymers such as polyethylene glycol [472 482] and chitosan [483 487]. PEG has many outstanding properties such as solubility in water and in organic solvents, lack of toxicity, and absence of antigenicity and immunogenicity, which are essential for drug formulations and other medical applications in implantation and wound treatment. To date, there have been many reports focused on the synthesis and characterization of block copolymer of L-lactide and poly(ethylene glycol) [472 482] and on their biomedical applications [473, 488]. However, such AB or ABA-type block copolymers have difficulty in getting a PLLA/PEG copolymer with a high molecular weight. Later many researchers synthesized multiblock copolymers constructed with alternated short PEG and PLA blocks having high molecular weight with remarkable mechanical strength suitable for constructing cell scaffolds for tissue engineering purposes [489, 490]. Zhou et al. in their review article focuses mainly on the properties of polylactide co-poly(ethylene glycol) (PELA) microspheres adjuvant as vaccine delivery systems [428]. The results indicate that compared with the commonly used PLA and PLGA, PELA showed several potentials in vaccine delivery systems, which may be due to the block copolymer have its capability to provide a biomaterial having a broad range of amphiphylic structure. PELA microspheres can control the rate of release of entrapped antigens and therefore, offer potential for the development of single-dose vaccines. They are extremely flexible delivery systems capable of encapsulating a wide range of antigens. Otsuka et al. gave a review article regarding the self assembly of poly(ethylene glycol)-PLA block copolymers for biomedical applications [491].

Among renewable resources chitosan, a unique amino polysaccharide has attained great interest because of its special structure, properties and its inexpensive abundant resources [157, 182, 492 496]. The specific properties of chitosan such as biocompatibility, biodegradability, bioactivity, low immunogenicity and multifunctionality increase its potential as a beneficial material for various applications in biomedical field [497 506]. However, the extended applications of chitosan are frequently limited by its insolubility in water and neutral pH due to its rigid crystalline nature and poor processability. The physicochemical properties and biodegradability of chitosan and polylactide could be enhanced substantially by graft copolymerization of L-lactide onto chitosan. The copolymer may be able to be degraded faster than polylactide. Unlike chitosan, all chitosan-lactide graft copolymers are converted to hydrogels in aqueous environment. Albertsson et al. prepared a pH- sensitive physically cross linked hydrogel by grafting D,L-lactic acid onto amino groups in chitosan without using a catalyst [452]. Later Dutta et al. reported that the molecular mechanism of gelation involves interaction between chitosan and lactic acid [507]. Yao et al. reported the in vitro fibroblast static cultivation on a cytocompatible poly (chitosan-g-L-lactic acid) film and the cell growth rate on the copolymer film was found to be much faster than that of the chitosan film [484]. In another work, Liu et al. have reported the synthesis of a brush like copolymer of polylactide grafted onto chitosan [485]. Later, Wu et al. studied the amphiphilic properties of a graft copolymer of water-soluble chitosan and polylactide prepared by using triethyl amine as catalyst [508]. In 2005 Peesan et al. [509] prepared hexanoyl chitosan/PLA blend films and in 2006 Wan et al. [510] prepared a biodegradable polylactide/chitosan blend film. The biodegradability of these chitosan/polylactide graft copolymers was, however, not studied in these works.

The introduction of functional groups in polylactide for the modification of its properties has been investigated since 1990s. Barrera et al. synthesized a copolymer of poly(lactic acid-co-lysine) containing lysine residues [511]. The free amino group in the lysine residue was used to chemically attach a biologically active peptide GRGDY [511, 512]. Gonsalves et al. [513] and Ouchi et al. [514] reported the introduction of hydroxyl and carboxylic acid groups on the surface of polylactide by copolymerization with the amino acids, serine (P(lac-Ser)), and aspartic acid (P(Lac-Asp)) respectively. Recently, Wang et al. synthesized a series of polylactide polyurethanes (PLAUs) having the property of shape recovery temperature near to the body temperature from poly(L-lactide) diols, hexamethylene diisocyanate, and 1,4-butanediol [515].

In 1992, Simone and coworkers [516] and Castaldo et al. [517] introduced the idea of random multiblock PEAs containing poly(L-lactide) blocks as hydrolytically degradable segments. They reported that the presence of amide functions in the polymer chain might lead to better physical properties than aliphatic polyesters usually have. Poly(ester amide)s are a class of biodegradable condensation polymers having regular enchainment of ester and amide groups as the integral part of polymer chain [518]. The ester component, in fact, reduces the crystallinity of polyamide segments and increases the chain flexibility of PEAs needed to fit into the active sites of the enzymes. Since amide groups are polar, their incorporation into the ester main chain leads to an increase of hydrophilicity [519]. The enhanced hydrophilicity increases the amount of water sorption in the amorphous region followed by degradation. Qian et al. reported a decrease in the degradation rate of PEAs with increase of amide content, macromolecular weight, and thickness of the test samples, and increase with incubation temperature and pH of the degradation medium [520 524]. Angelo et al. prepared poly(ether-ester-amide)s based on poly(L,L-lactide) macromer for the delivery of bioactive compounds [525]. They reported that an enhancement of biodegradability of PEAs could be achieved by the incorporation of flexible hydrophilic oligo(ethylene glycol) segments into the main chain. It was reported in the literature that the crystal structure and physical properties of PLLA could be increased by the incorporation of cycloaliphatic amide segments. The rigidity, arising from the conformational restriction in the cyclic ring, makes them has relatively high thermal properties in comparison with their linear aliphatic counterparts [526].

PLGA demonstrates good cell adhesion and proliferation making it a potential candidate for tissue engineering applications. Various studies have been performed so far using micro- and nanofabrication techniques to form three-dimensional biodegradable scaffolds based on PLGA [527, 529]. Figure 18 shows porous three-dimensional structures developed from PLGA using (a) gas foaming [527] (b) microsphere sintering [528] and (c) electrospinning techniques [529].

Chitosan/oligoL-Lactide Graft Copolymers

The physicochemical properties and biodegradability of chitosan and polylactide could be enhanced substantially by graft copolymerization of L-lactide onto chitosan. Chitosan/oligoL-lactide graft copolymer was synthesized in DMSO at 90 °C in presence of $Ti(OBu)_4$ as ring opening catalyst [530]. Grafting of hydrophobic side chains results in the decrease of chitosan crystallinity by loosening the hydrogen bonds and increasing the number of free hydrophilic hydroxyl groups and amino groups of chitosan, which in turn can hold water molecules more strongly. The graft copolymers are converted to hydrogels on exposure to deionized water. This property is of special interest in biomedical applications such as wound dressing and in controlled drug delivery systems. At higher grafting percentages due to the self-assembling of longer OLLA side chains by the hydrogen bonding and dipole interactions, the hydrophobic nature of the side chains become dominant, which results in the lower swelling of graft copolymers at higher grafting percentages (Fig. 19). A decrease in hydrolytic degradation was observed with increase of lactide content in CL graft copolymers (Fig. 19). CL graft copolymers showed a higher susceptibility towards lipase

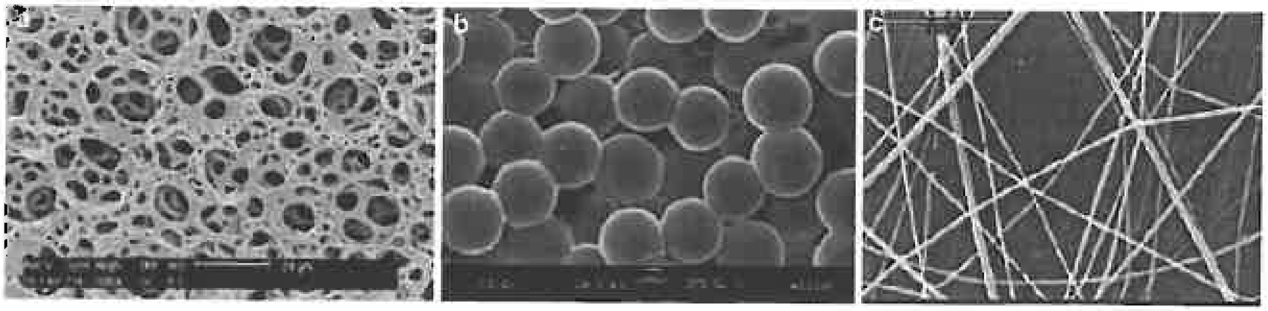

Fig. 18 Porous three dimensional structures developed from PLGA using (**a**) gas foaming (reprinted from Ref. [527] with permission from Elsevier), (**b**) microsphere sintering (reprinted from Ref. [528] with permission from Elsivier) and (**c**) electrospinning (reprinted from Ref. [529] with permission from Wiley Interscience

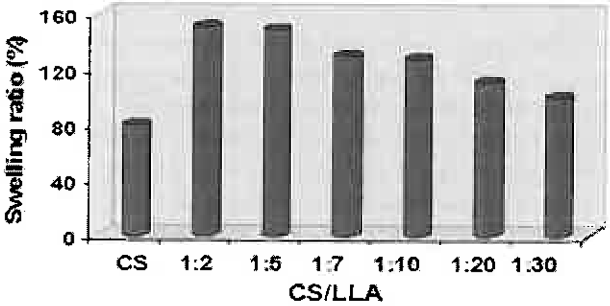

Fig. 19 Swelling studies of CS and CL graft copolymers

than the original chitosan (showed no significant degradation in lipase), which indicated that the chitosan segment consisting of glucosamine residues is not accessible to the lipase active site and the random distribution of OLLA side chains is at least required to be adsorbed to the active sites of the lipase. As lipase is one of the main enzymes present in the human body, the higher susceptibility of CL graft copolymers towards lipase than that of chitosan and OLLA would increase its potential in biomedical field. SEM micrographs of CL graft copolymers (Fig. 20) taken after 8 h immersion in papain medium showed severe surface erosion in many of the graft copolymers where the latic acid content is low. These results indicate that the physicochemical properties and the rate of degradation of graft copolymers as a biomaterial could be controlled by adjusting the amount of LLA in the CL graft copolymers.

Random Multiblock Poly(Ester Amide)s Containing Poly(L-Lactide) and Cyclo Aliphatic Amide Segments

The lack of certain optimum properties such as thermal, mechanical and processing limits their wider use of aliphatic polyesters (PE) mainly poly(L-lactic acid) (PLLA), poly(glycolic acid) (PGA) and their copolymers which are the best known biodegradable, biocompatible polymers used intensively in biomedical field. Aliphatic polyamides (PA) have better thermal and mechanical properties than aliphatic polyesters, but their hydrolytic degradation rate is too low to classify them as biodegradable polymers. Therefore, by incorporating polyamide segments into the PLLA main chain, it could be possible to synthesize a polymeric material possessing good material and processing properties and biodegradability than their homopolymer counterparts.

Pillai and Luchachan have prepared random multiblock poly(ester amide)s containing PLLA macromer and cycloaliphatic amide segments by the "two-step" interfacial polycondensation method [526]. The resulting polymers exhibit inherent viscosities in the range of 0.37 0.48 dL 4]/g. PEAs having lower content of PLLA, PEA 1 and PEA 2, show a single diffraction signal at 20.8° 2θ and a single melting transition at 166 and 164 °C, respectively, indicating an amide-adjacent crystallization of copolymers. In PEA 3, the diffraction signals (16.7 and 18.8° 2θ) and melting transition (101 °C) characteristic of PLLA macromer are also appeared. This was attributed to the development of a crystalline structure of the PLLA type at higher ester content, revealing the blocky nature of PEAs. The random nature of PEAs is confirmed by the appearance of single T_g values. Even though the prepared PEAs are in low molecular weight (0.35 0.44 dL/g), they show a thermal stability unto a temperature of 230 °C. This may be attributed to the presence of strong hydrogen bond interactions between ester and amide groups and the presence of rigid cyclic ring structures in the polymer backbone. Since no evidence of thermal decomposition was observed below a temperature of 200 °C, they can be processed from the melt state. All PEAs are susceptible to lipase promoted degradation and the rate of degradation increased with increase in PLLA content. Spectroscopic investigations and thermal analysis of degraded products show the breakage of ester linkages. The results indicate that by the incorporation of cycloaliphatic amide segments onto the PLLA macromer it could be possible to synthesize PEAs having good thermal properties and biodegradability at the lower molecular weight that would increase its potential in the biomedical applications.

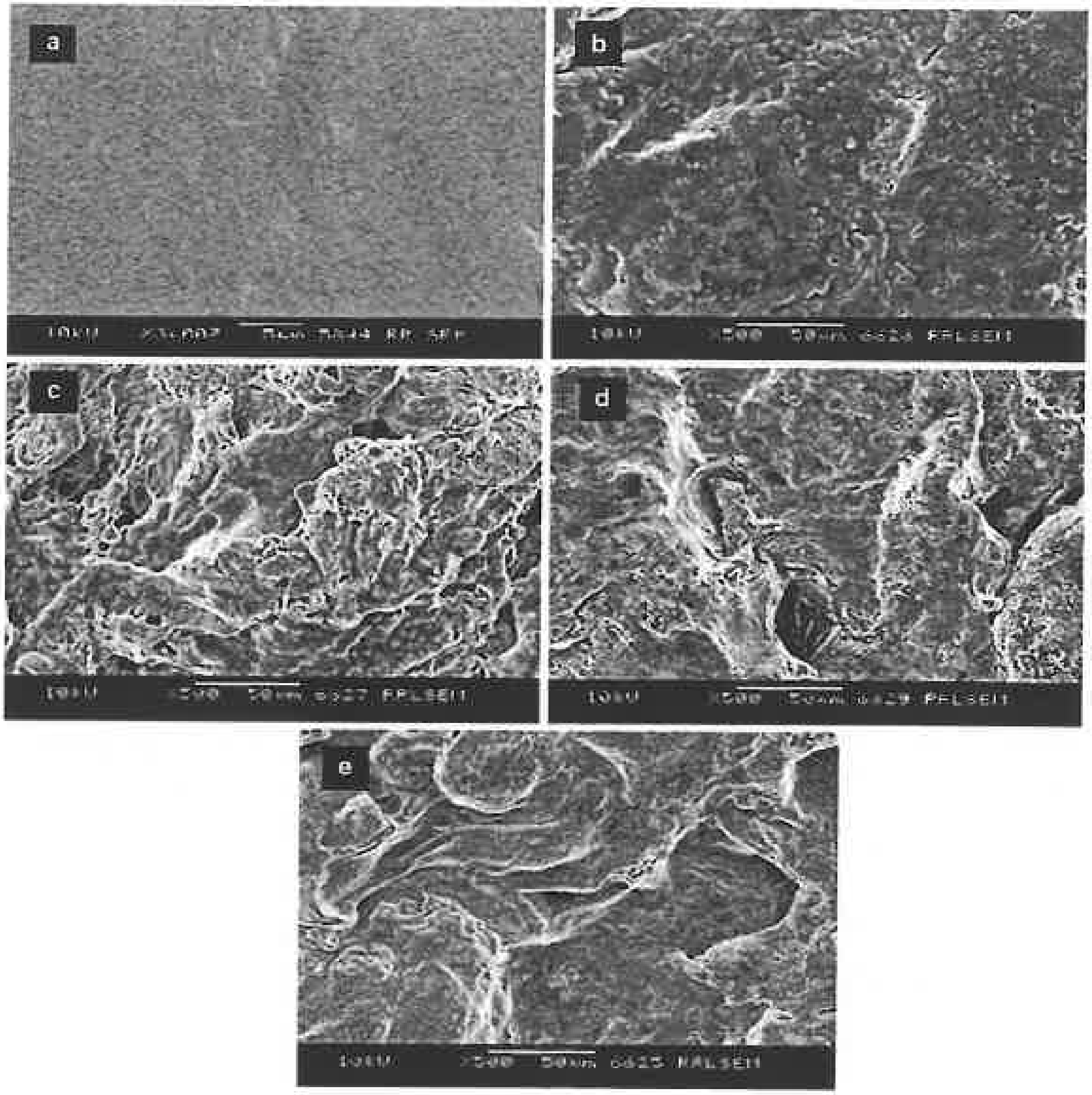

Fig. 20 SEM micrographs of hydrolytically degraded samples taken after 80 days of immersion in deionized water: (**a**) CL graft copolymer before degradation, (**b**) Chitosan, (**c**) CL 2, (**d**) CL 10 and (**d**) CL 30 graft copolymers

Current developments [531 563] include continued stress on new techniques of production and processing novel applications [531, 532]. In a comparative evaluation of two synthetic routes: direct polycondensation reactions without organic solvents, and in a supercritical medium, Jahno et al. showed that the poly condensation reaction proved to be an excellent synthetic route to produce PLA polymers with different molar mass [533]. It was observed that cell adhesion and viability was not disturbed by the presence of the polymer, although the proliferation rate was decreased when compared to control.

Microbial fermentations are preferred over chemical synthesis of lactic acid due to various factors [534]. Refined sugars, though costly, are the choice substrates for lactic acid production using Lactobacillus sps. Complex natural starchy raw materials used for production of lactic acid involve pretreatment by gelatinization and liquefaction followed by enzymatic saccharification to glucose and subsequent conversion of glucose to lactic acid by Lactobacillus fermentation. Reddy et al. showed that economical production of lactic acid can be done by direct conversion of starchy biomass to lactic acid by bacteria possessing both amylolytic and lactic acid producing character thus eliminating the the two step process. Lactobacillus amylophilus GV6 is an efficient and widely studied amylolytic lactic acid producing bacteria capable of utilizing inexpensive carbon and nitrogen substrates with high lactic acid production efficiency [534].

Recent advances in simultaneous saccharification and fermentation, molecular genetic approaches, and enzymetic and metabolic pathways involved in the production of lactic acid by fungal strains are discussed by Zhang et al. [535]. In another study involving osteointegration of poly(L-lactic acid)PLLA and poly(l-lactic acid)PLLA/poly(ethylene oxide)PEO implants in rat tibiae, Corac et al. showed that newly formed bone was found around both PLLA and PLLA/PEO implants. PLLA/PEO blends had a porous morphology after immersion in a buffer solution and in vivo implantation. The proportion 50/50 PLLA/PEO blend was adequate to promote this porous morphology, which resulted in gradual bone tissue growth into the implant [536].

Petrie et al. investigated the osteogenic potential of a novel stem cell was analyzed on biodegradable poly(lactic-co-glycolic acid) (PLGA) biomaterials as a step toward creating new cell-materials constructs for bony regeneration [537]. Dura mater stem cells (DSCs), isolated from rat dura mater, were evaluated and compared to bone marrow stem cells (BMSCs) for proliferative and differentiative properties in vitro. DSCs demonstrated greater cell coverage and enhanced matrix staining on 2D PLGA films when compared with BMSCs. These cells can be isolated and expanded in culture and can subsequently attach, proliferate, and differentiate on both TCP and PLGA films to a greater extent than BMSCs. This suggests that DSCs are promising for cell-based bone tissue engineering therapies, particularly those applications involving regeneration of cranial bones.

Cheung et al. showed that the use of silk fibers as reinforcements in PLA was a good candidate for the development of polymeric scaffolds for tissue engineering applications [538]. Lu et al. synthesized a series of biodegradable poly(L-lactide-co-ε-caprolactone) (PCLA) copolymers with different chemical compositions. These polymers exhibited shape-memory behaviors [539]. The tensile strength of copolymers decreases linearly and the elongation at break increases gradually. By means of adjusting the compositions, the copolymers exhibit excellent shape-memory effects with shape-recovery and shape-retention rate exceeding 95%. The degradation results show that the copolymers with higher ε-CL content have faster degradation rates and shape-recovery rates, meanwhile, the recovery stress can maintain a relative high value after 30 days in vitro degradation.

Chiu et al. report the fabrication carbon nanotubes (CNTs) embedded in a polylactic acid (PLA) matrix to obtain a composite nanostructure. Purified CNTs showed better dispersing and properties in the CNTs PLA Nanocomposites [540]. The nanoclay-loaded PLLA substrates were used for preparing polymer photovoltaic devices [541]. Devices were realized by application of a conducting transparent anode comprising an aluminium grid with a thin over layer of silver and spin-coated PEDOT:PSS. The active layer consisted of microfibrillar P3HT and PCBM to encompass the low processing temperatures for PLLA and finally an evaporated aluminium cathode. It was found that PLLA as a substrate holds potential, but there are several challenges beyond the photovoltaic itself which must be met before general application within this field can be envisaged.

With the increasing interest in developing fully biodegradable materials for industrial applications, use of natural fibers in PLLA matrix poses an interesting alternative economically. Composite combining biodegradable polymer and natural fiber could be a fully biodegradable material. This work focus on the investigation on China jute fiber reinforced PLA composites. Composites with three different fiber volume fractions were fabricated by film stacking hot press method. Mechanical properties have been investigated and the microstructure of the composites was observed. Fiber surface treatment by silane was applied in order to investigate the effects on the properties of the composites. The moisture absorption rates in a hydrothermal environment were also measured. In terms of the mechanical properties, the wood-fiber-reinforced poly(lactic acid) composites were comparable to conventional polypropylene-based thermoplastic composites [542]. The mechanical properties of the wood-fiber-reinforced poly(lactic acid) composites were significantly higher than those of the virgin resin. The flexural modulus (8.9 GPa) of the wood-fiber-reinforced poly(lactic acid) composite (30 wt % fiber) was comparable to that of traditional (i.e., wood-fiber-reinforced polypropylene) composites (3.4 GPa The incorporation of the wood fibers into poly(lactic acid) resulted in a considerable increase in the storage modulus (stiffness) of the resin. The addition of the maleated polypropylene coupling agent improved the mechanical properties of the composites. Microstructure studies using scanning electron microscopy indicated significant interfacial bonding between the matrix and the wood fibers. The specific performance evidenced by the wood-fiber-reinforced poly(lactic acid) composites may hint at potential applications in, for example, the automotive and packaging industries.

PLA (polylactic acid) was reinforced with Cordenka rayon fibres and flax fibres, respectively [543]. The mechanical properties of these composites which are examples for completely biodegradable composites were tested and compared. The samples were produced using injection molding. The highest impact strength (72 kJ/m^2) and tensile strength (58 MPa) were found for Cordenka reinforced PLA at a fibre-mass proportion of 30%. The highest Young's modulus (6.31 GPa) was found for the composite made of PLA and flax. A poor adhesion between the matrix and the fibers was shown for both composites using SEM. The promising impact properties of the presented PLA/Cordenka composites show their potential as an alternative to traditional composites.

In another work focus was on the investigation on China jute fiber reinforced PLA composites [544]. Composites with three different fiber volume fractions were fabricated by film stacking hot press method. Mechanical properties have been investigated and the microstructure of the composites was observed. Fiber surface treatment by silane was applied in order to investigate the effects on the properties of the composites. The moisture absorption rates in a hydrothermal environment were also measured.

Standards developed by ASTM and ISO evaluate the biodegradation of biodegradable plastic materials in simulated controlled composting conditions. However, a more detailed understanding of the biodegradation of complete packages is needed in order to have a successful composting operation. Kale et al. investigated the biodegradation performance of polylactide (PLA) bottles under simulated composting conditions according to ASTM and ISO standards using two simulated composting methods were used in this study to assess biodegradability of PLA bottles: (a) a cumulative measurement respirometric (CMR) system and (b) a gravimetric measurement respirometric (GMR) system [545]. Both CMR and GMR systems showed similar trends of biodegradation for PLA bottles and at the end of the 58th day the mineralization was 84.2 ± 0.9% and 77.8 ± 10.4%, respectively. The biodegradation observed for PLA bottles in both conditions explored in this study matched well with theoretical degradation and biodegradation mechanisms.

To make PLA economically viable, Bhtia et al., used blends of PLLA with poly (butylene succinate) (PBS) to explore suitability of these compositions for packaging applications [546]. Tensile strength, modulus and percentage (%) elongation at break of the blends decreased with PBS content. However, tensile strength and modulus values of PLA/PBS blend for up to 80/20 composition nearly follow the mixing rule. Rheological results also show miscibility between the two polymers for PBS composition less than 20% by weight. PBS reduced the brittleness of PLA, thus making it a contender to replace plastics for packaging applications. This work found a partial miscibility between PBS and PLA by investigating thermal, mechanical and morphological properties.

Several attempts are reported to improve the mechanical properties of PLLA [441, 540, 547]. Possessing the stiffness and tensile strength of polyethylene terephthalate (PET), polylactide is being marketed for a variety of fiber and packaging products. Fibers produced from polylactide exhibit low odor retention and excellent moisture wicking properties. Additionally, polylactide bottles, films, and thermoformed containers are becoming increasingly popular for food packaging because of their resistance to fats and oils, as well as their ability to "block" flavors and aromas. The use of clay composites for maintaining the stiffness and tensile modulus of polylactide on toughening have shown promise. Andertson et al. discuss three most common strategies for polylactide toughening : orientation, plasticization, and blending. It is well known that in general, semi-crystalline polylactide homopolymer shows improved tensile and impact properties relative to the amorphous. So, orientation is expected to increase the tensile modulus and elongation at break. The use of small molecule plasticizers (e.g., lactide, citrate esters) as well as oligomeric/polymeric plasticizers (such as PEG) in polylactide are useful ways to obtain significant increases in the elongation at break, making polylactide much more elastic. Blending with other bio degradable non biodegradable fibers and composite formation are good toughening mechanisms. The use of clay composites for maintaining the stiffness and tensile modulus of polylactide on toughening have shown promise.

A significant increase in impact resistance (540 + 100 J m21) was observed in a PLA/LLDPE blend compatibilized with 5 wt% of a PLLA-b-PE block copolymer [547] (See Fig. 21).

Copolymer formation is considered a good method to improve properties [548 560]. To tailor the properties of PLA for a particular application, several parameters, apart from what have been discussed such as catalyst and catalyst concentration, molecular weight, crystallinity, end capping, copolymer ratio etc. have to be controlled [425]. The selection and use of an appropriate catalyst is of paramount importance to produce PLA at a lower cost. The mechanical properties of PLA can be varied to a large extent ranging from soft, elastic plastic to stiff and high strength plastic. With the increase of molecular weight the mechanical properties also increase. With the increase of molecular weight of PLLA from 23 to 67 k, flexural strength increased from 64 to 106 MPa but the tensile strength remains the same at 59 MPa [561]. In the case of poly(DL-lactide) when molecular weight is increased from 47.5 to 114 k [561] tensile and flexural strength increased 49 53 MPa and 84 88 MPa, respectively. Tissue engineering is the most recent domain where poly(lactic acid) is being used and is found to be one of the most favorable scaffold materials [562]. PuraSorbsPLG is a semicrystalline bioresorbable copolymer of L-lactide and glycolide with a monomer ratio of 80L: 20G [563] A co-polymer containing 90% glycolic acid (GA) and 10% L-lactic acid (LA) was initially used for the development of the multifilament suture Vicryls [156]. An irradiated version of Vicryls to increase the rate of degradation is currently in the market under the trade name, Vicryl Rapids. PANACRYL s is another commercially developed suture from the co-polymer with a higher LA/GA ratio in order to decrease the rate of degradation.

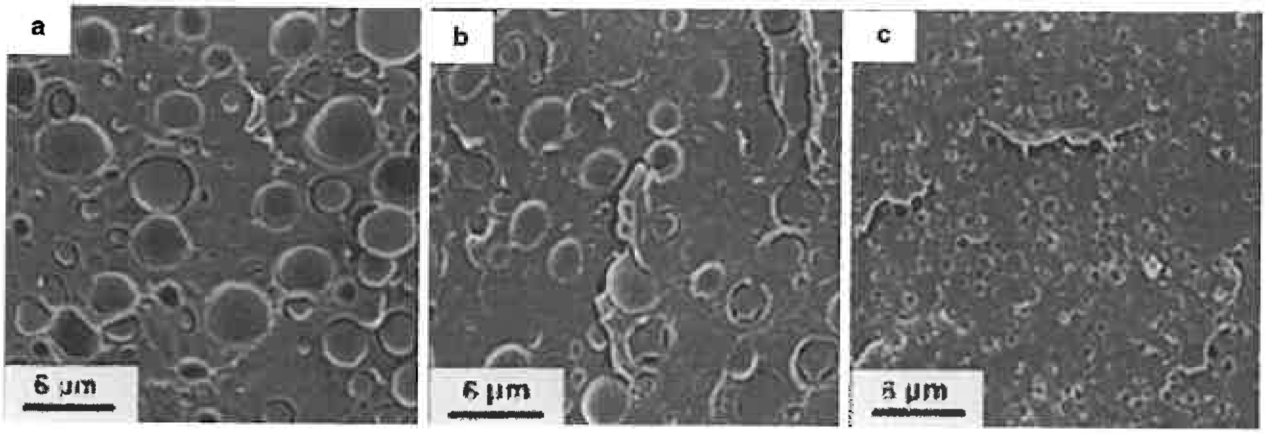

Fig. 21 SEM images demonstrating the differences in morphology for 80/20 wt% PLA/LLDPE Blends. (**a**) binary blend, (**b**) addition of 5 wt% of PLLA PE (5 kg/mole 30 kg/mole) block copolymer, (**c**) addition of 5 wt% of PLLA PE (30 kg/mole 30 kg/mole) block copolymer. (Reprinted from Reference [547] with permission from Wiley)

The Issue of Sustainability and Environmental Impact

Due in part to a growing emphasis on "green" chemical processes, there has been increasing interest in the development and utilization of more environmentally benign polymeric materials. This classification can describe a continuum of polymers, ranging from those that are obtained from annually renewable feedstocks, to those that ultimately degrade to benign by-products after their useful lifetime. One of the key polymers in this regard is polylactide, or poly(lactic acid), which is one of a subset of thermoplastic polymers that is viable in both of these categories, as it is derived from plant starches and is ultimately degradable into biologically and/or environmentally manageable by-products when composted [2].

Applications

For economic reasons, for practical reasons, even for health and safety reasons, biodegradable plastic products are establishing themselves as viable alternatives in a number of important niche markets. Some of these lie in the fields of medicine, textiles (clothing and fabrics), hygiene and agriculture, as well as some specific packaging applications. Because of their specialized nature and greater unit value, medical device applications have developed faster than the others.

Medical Applications

Biodegradable polymers have been widely used in vascular and orthopedic surgery as implantable matrices for the controlled long-term release of drugs inside the body, as absorbable surgical sutures and plastic staples, various pins and screws, ligature clips and vascular grafts, artificial skin and bone fixation devices [564, 565]. Commonly used PLA based biodegradable polymers in medical field are given in Table 2.

A number orthopedic products based on PLLA have come up: the Phantom Soft Thread Soft Tissue Fixation Screws, Phantom Suture Anchors (DePuy), Full Thread Bio Interference Screws (Arthrex), BioScrews, Bio-Anchors, Meniscal Stingers (Linvatec), and the Clearfix Meniscal Darts (Innovasive Devices) are gaining momentum [152]. PLLA fiber was FDA approved in 1971 for the development of an improved suture over DEXONs. Due to the high strength of PLLA fibers, it has been investigated as scaffolding material for developing ligament replacement or augmentation devices to replace nondegradable fibers, such as Dacron [566 568].

Agricultural Applications

Biodegradable polymers in agricultural field as agricultural mulches, agricultural planting containers, and in controlled release of agricultural chemicals are also of interest. Biodegradable films based on starch with poly(vinyl alcohol), poly(ethylene-co-acrylic acid) and poly(vinyl chloride) have been developed as agricultural mulch films [569]. Starch, cellulose, chitin, alginic acid and lignin are among the natural polymers used in controlled release systems and polycaprolactone are used as agricultural planting containers [487].

Packaging

The niche markets of biodegradable polymers in packaging field include composting bags for yard waste or food scraps, carrier bags, food-service applications, such as single-use compostable plastic cups, straws, plates and cutlery, and food wrappers and containers (Table 3) [313].

Table 2 Biodegradable PLLA based polymers used in medical field [562, 564]

Application	Trade Name	Composition	Manufacturer
Sutures	Vicryl	PLLA(8%) co PGA(92%)	Ethicon
	Polysorb	PGA LPLA	U.S. surgical
Interference screws	Sysorb	DLPLA	Synos
	Endofix	PGA TMC or LPLA	Acufex
	Arthrex	LPLA	Arthrex
	Bioscrew	LPLA	Linvatec
	Phusiline	LPLA DLPLA	Phusis
	Biologically quiet	PGA DLPLA	Instrument Makar
	Full thread bio sheathed	LPLA	Arthrex
	Bio	LPLA	Arthrex
	Phantom	LPLA	DuPuy
Suture anchor	Bio statak	LPLA	Zimmer
	Bankart tack	SR LPLA	Bionx implants
	SmartAnchor D	SR LPLA L	Bionx implants
	SmartAnchor L	R LPLA	Bionx implants
	Phantom suture anchor	LPLA	DuPuy
	BioROC EZ 2.8 mm	LPLA	Innovasive devices
	BioROC EZ 3.5 mm	LPLA	Innovasive devices
	Biologically quiet	85/15 DLPLG	Instrument makar
	Biosphere	85/15 DLPLG	Instrument makar
	Bio anchor	LPLA	Mitek products
	GLS	LPLA	Mitek products
	Panalok	82/18 LPLG	Surgical dynamics
	Panalok RC	82/18 LPLG	Surgical dynamics
	SD sorb 2 mm	82/18 LPLG	Surgical dynamics
Dental	Drilac	DLPLA	THM Biomedical AHP
Angioplastic plug	Angioseal	PGA DLPLA	Bionx
Screw	SmartScrew	LPLA	Bionx
Tack	SmartTack	LPLA	Lorenz
Plates, mesh, screws	LactoSorb	PGA LPLA	
Ligating clips, bone pins and rods	Biofix	LPLA or PGA	Bionx
	Resor Pin	LPLA DLPLA	Geistlich
	Lactasorb	LPLA	Davis and Geck
Guided tissue	Antrisorb	DLPLA	Atrix
	Resolut	PGA DLPLA	W.L. Gore
	Guidor	DLPLA	Procordia
Drug delivery products	Lupron Depot	PLA/PLGA	TAP
	Zoladex	PLA/PLGA	AstraZeneca
	Decapeptyl	PLA/PLGA	Ferring

Textiles and Hygiene Applications

Woven and non-woven fabrics, made from biodegradable polymers are used as industrial wipes, filters, and geotextiles for erosion control and landscaping. Personal hygiene products, such as disposable diapers with a biodegradable plastic liner or tampon applicators are made from biodegradable resins.

Thoughts on Future Perspectives

Biodegradable polymers are expected to play an increasingly important role in a society moving towards a sustainable and environmentally responsible materials base with possibilities for implementing the principles of industrial ecology. They offer a possible alternative to traditional non-biodegradable polymers. One of the main

Table 3 The niche markets of biodegradable polymers in packaging [313]

Use	Trade name	Composition
Packaging and agriculture	Biopol	Poly(3 HB co 3 HV)
	Bionolle	Poly(butylene succinate) Poly(ethylene succinate)
	Aqua NOVON and Vinex	Starch/PVOH
	BIOCETA	Cellulose acetate
	Tone and enviroplastic C	Poly(ε caprolactone)
	EcoPLA'	PLA
	Natureworks	PLA
	PLAQUCEL.TM	Polycaprolactone
	BAk 1095	PEA
Disposable items, bottles, personal care and hygiene	Biomax	PET modified
	Cereplast	Corn and potato starch
	Mater Bi	Starch and vegetable oil

driving force behind this technology is the solid waste problem, particularly with regard to the decreasing availability of landfills, the litter problem and the pollution of marine environment by non-biodegradable plastics. Thermoplastic starch-based polymers and aliphatic polyesters are the two classes of biodegradable materials with the greatest near-term potential. Biodegradability requires complete assimilation of the plastic by the microbial populations present in the selected disposal environment in a defined time frame as opposed to degradable or partially biodegradable. Therefore, biobased content and biodegradability are essential elements for single use, short-life disposable packaging and consumer plastics. For example, thermoplastic starch in combination with poly(vinyl alcohol) or some aliphatic polyesters like poly(hydroxybutyrate-co-hydroxyvalerate), poly(lactic acid), poly(ε-caprolactone) and poly(butanediyl succinate) appears to have gaining approval as it meets the basic requirements for a product to be declared compostable [70]. Complete biodegradability of the product in a time period compatible with composting, measured through respirometric tests (ASTM D5338-9, ISO/CD14855, etc.); disintegration of the material during the fermentation phase and no negative effects on compost quality etc. could be easily met by these combinations.

However, in long term perspectives aliphatic polyesters such as poly(hydroxybutyrate-co-hydroxyvalerate) and poly(lactic acid) are expected to gain momentum because of possible outcomes low cost production possibilities looming on the research front. For durable, long life articles bioplastics needs to be engineered for long-life and performance, so biodegradability may not be an essential criterion and in this respect also, these aliphatic polyesters may gain additional support. The biotechnological production of bulk biopolymers such as polyhydroxyalkanoates and polylactic acid, which act as substitutes for polyolefins might play a major role in building of a sustainable bioeconomy as the next generation of materials [570]. The success of polylactide highlights the commercial and environmental potential for plastics sourced from plants, with renewable life cycles and which are carbon neutral [154]. The applications for polylactide are enhanced by its biocompatibility and its ability to be absorbed and degraded in vivo; furthermore, it is an FDA approved substance for use in therapy. It has been used for some time in biomedical applications such as sutures, stents, dental implants, vascular grafts, bone screws and pins. It has also been investigated as a vector for drug delivery, for example in the long-term delivery of antimicrobial drugs, contraceptives and prostate cancer treatments. PLA has been widely used in the field of tissue engineering as a scaffold material to support cell and tissue growth. However, for many applications, PLA is not an ideal material due to its high crystallinity, brittleness, lack of total absorption and thermal instability. Further drawbacks are that it is unfunctionalized and hydrophobic; these properties impede its biodegradation and prevent targeting of the treatment their specialist applications in niche medical markets PLA is currently manufactured on a large scale in the US and by smaller enterprises in the EU and Japan.

One of the major developments that may sustain the growth of biodegradable plastics is the emergence of a number of industrial firms such as NatureWorks, NE, owned by Cargill (biobased polylactic acid material from corn sugars; Cargill Dow LLC (a 50/50 joint venture between Cargill Inc. and The Dow Chemical Co.) has recently developed a solvent-free, low-cost continuous process for the reduction of PLA from corn-derived dextrose. The resulting PLA, commercialized under the trade name Nature Works, is the first synthetic polymer to be produced from annually renewable resources [432]. with material mirroring styrenics in mechanical properties)' Germany's BASF (biodegradable aliphatic-aromatic copolyesters)' Ecovio L (biodegradable foams that are making a potential heir apparent to the polystyrene foam containers)' Rejuven8 Plus sheet is a strength modified grade with impact properties similar to polyethylene terephthalate (PET), Braskem Technology, Brazil-based petrochemical firm (biobased high density polyethylene (HDPE) to cite a few. Researchers at the Pacific Northwest National Laboratory has also reported a breakthrough in converting common sugars, fructose, and glucose into fuel and polyester building blocks. Metabolix is commercializing a family of polymers (PHA natural plastics) made from renewable resources such as corn sugar and vegetable oils. Within the foreseeable future, this technology will also enable the production of these Natural Plastics directly in non-food plant crops. These high performing new materials have the potential to put a large portion of the plastics and chemicals industry on a sustainable basis.

There is pressing need to enhance the versatility of these bioplastics, so that it can compete with conventional polymers. However, in situations that require a high level of impact strength, the toughness of polylactide in its pristine state is often insufficient. As such, there has been tremendous effort in developing ways to improve this material property deficiency. Several lines of futuristic thoughts are on the anvil. Bringing down the production cost by using better production technologies, toughening the polylactide through various means such as orientation, plasticization, copolymerization and blending, using nanostructured reinforcements, are some of the strategies being explored [425, 441]. Use of special catalysts in metal assisted ROP of lactide or use of multifunctional initiators might provide structure controlled macromolecules with specific functional properties [432].

Blending is agreeably an area of great promise. For example, when a small amount of ductile PHA is blended with poly(lactic acid) (PLA), a new type of polymer alloy with much improved properties is created. The toughness of PLA is substantially increased without a reduction in the optical clarity of the blend. The synergy between the two materials, both produced from renewable resources, is attributed to the retardation of crystallization of PHA copolymers finely dispersed in a PLA matrix as discrete domains [571]. Similarly, the use of silk fibers was found to be a good candidate, as reinforcements in PLLA for the development of polymeric scaffolds for tissue engineering applications [538]. The combination of biodegradable polymers and CNTs opens a new perspective in the self-assembly of nanomaterials and nanodevices [572]. Modeling and related studied might throw more light on the intrinsic brittle nature of poly(lactide)s. For example, the stiffness of a polymer chain, expressed as the characteristic ratio C_{∞} plays an important role in the deformation mechanism of amorphous polymers [573].

Another question is the recyclability of industrial production as the economic viability of PLLA has become industrially feasible [574]. Large enzyme quantities (500 600 wt%) are necessary to achieve high to complete depolymerization in 24 48 h [54]. The enzymatic approach is interesting not only for the ROP of lactide but also for the PLA recycling via depolymerization. Increasing interest has also been dedicated to chemically modified poly(a-hydroxy acids). In this regard, the preparation and polymerization of various substituted 2,4-dioxane-2,5-diones have demonstrated that the polymer properties are tunable across a broad range. Poly (a-hydroxy acids) featuring pendant functional groups also appear particularly promising sustainable materials and further developments in this direction should be facilitated using O-carboxy anhydrides which are readily available, structurally moldable and highly polymerizable monomers [575]. Methods such as organocatalyzed ROP of lacOCA are indeed competitive routes to make PLAs of controlled molecular weights and narrow polydispersities under particularly mild conditions [576].

Reddy et al. demonstrates that with many applications in food, pharmaceutical, leather, textile industries and as chemical feed stock, microbial fermentations are preferred over chemical synthesis of lactic acid due to various factors and direct conversion of starchy biomass to lactic acid by bacteria possessing both amylolytic and lactic acid producing character will eliminate the two step process to make it economically viable [577]. Refined sugars, though costly, are the choice substrates for lactic acid production using Lactobacillus sps. Complex natural starchy raw materials used for production of lactic acid involve pretreatment by gelatinization and liquefaction followed by enzymatic saccharification to glucose and subsequent conversion of glucose to lactic acid by Lactobacillus fermentation.

Higher activities and better control can be expected in due course by further optimizing the initiator structure and polymerization procedure [578, 579]. In particular, further improvements may be anticipated for the stereocontrolled ROP of lactide using well-defined complexes, with non-metallic systems being likely to attract increasing interest [432].

Poly (hydrohybutyrate-co-hydrohyvalerate) (PHBV) copolymers have received significant scientific attention recently due to the fact that they are biodegradable, and their properties can be easily controlled by the content of hydroxy valerate (HV). PHBV copolymers with different content of hydroxyvalerate have been used recently as matrices in eco-composites [1, 2], where different natural fibers were applied as reinforcement [100]. Recent research has focused on the use of alternative substrates, novel extraction methods, genetically enhanced species and mixed cultures with a view to make PHAs more commercially attractive [242, 580]. The classical microbiology and modern molecular biology have been brought together to decipher the intricacies of PHA metabolism both for production purposes and for the unraveling of the natural role of PHA [581]. It is easel to develop a commercially attractive recombinant process for large scale production of PHAs. PHA synthase (PhaC) enzymes, which catalyze the polymerization of 3-hydroxyacyl-CoA monomers to P(3HA)s, were subjected to various forms of protein engineering to improve the enzyme activity or substrate specificity [220]. The use of mixed cultures and renewable sources obtained from waste organic carbon can substantially decrease the cost of PHA and increase their market potential [198].

Commercially available soy products, e. g., soy oil, soy protein isolate, soy flour, and soy protein concentrate, have attracted significant attention because of their abundance, low cost, and good biodegradability. Compared to the other techniques such as heat treatment, co-blending with PCL enhanced tensile strength and lowered water absorption. Novel design strategies and engineering to provide advanced polymers from natural sources are being made [582]. The belief that sustainable production of chemicals via "white biotechnology" and "green chemistry" is indispensable for chemical industry is getting strengthened. What are emerging are new boundaries where synthetic and natural polymers converge. Initiatives such as bio-inspired polymer design and engineered biopolymers 'synthetically-inspired' materials inspiring prospects for polymer science at the bio-interface with new materials taking natural and synthetic polymers beyond nature's limitations are promises to deliver advanced biodegradable plastics. What are emerging are new boundaries where synthetic and natural polymers converge to ecologically sustainable and economically viable products and processes.

Composite panels consisting of blended soybean oil-based epoxy with synthetic epoxy resin and E-glass fiber were shown to have a comparable dynamic stiffness, flexural modulus, and flexural stiffness with those of synthetic systems [583]. Natural fiber-reinforced materials offer environmental advantages such as reduced dependence on non-renewable energy/material sources, and less pollutants and greenhouse emissions [100, 584, 585]. Their low cost, high toughness, low density, good specific strength properties, reduced tool wear (nonabrasive to processing equipment), enhanced energy recovery, CO2 neutral when burned, and biodegradability offer along with their hollow and cellular nature make excellent reinforcements for a variety of applications [586]. Nano fiber reinforcements give additional edge on properties over other fibers [587]. Although initially expensive, the increasing numbers of biodegradable matrices that have appeared as commercial products offered by various producers indicate the growing popularity of biodegradable polymers.

Conclusions

Biodegradable polymers for short time applications have attracted much interest all over the world in different sectors such as surgery, pharmacology, agriculture and the environment. The reason behind this growing interest is the incompatibility of the polymeric waste with the environment where they are disposed after the usage. The recovery of polymeric waste as a solution to this problem is not easy or feasible, like in surgery for obvious reasons, or in the environment in the case of litter. The development of novel biodegradable polymers satisfying the requirement of degradability, compatibility with the disposed environment and the release of low-toxicity degradation products are the ultimate solution to these issues. The recent technological advances offer great promise towards achieving biodegradability with less pollutants and greenhouse emissions. Linking performance with cost is a tremendous task which needs imaginative steps in the selection of materials, processes, product structures and production schedules. It appears that there is a peaceable shift to derive more carbon from the renewable resources to preserve the eco system. The petrochemicals as a sole resource for polymers may have to undergo a shift towards the fermentation industry wherein developments in genetic engineering might generate high yielding microbes to produce monomers and polymers at economically viable routes making way for cost effective and ecologically sustainable biodegradable plastics.

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GPEC 2006 Paper Abstract #9B

Title: Considerations Affecting Biodegradability of PVC

Authors: Richard F. Grossman, PhD, RFG Consultants, John E. Schleicher, Jr. and Lorna D'Alessio, Ultraflex Systems

ABSTRACT

Carbon-chlorine bonds are not a barrier to biodegradation. Bacteria and fungi exist that can, for example, metabolize PCBs and pentachlorophenol. The difficulty with polymers of all types is molecular weight. Biodegradation of PVC film and sheet under landfill conditions requires an additive package that, under such conditions, initiates degradation and directs its course to chain scission rather than crosslinking. A class of such additives has been found. Since food metabolism occurs outside the micro-organism, it is further necessary to direct the sensory capability of bacteria and fungi towards the article by including migratory components that will be recognized as nutrients. A discussion of useful fillers and plasticizers is presented as well as the degradation path envisioned.

Considerations Affecting Biodegradability of PVC

Richard F. Grossman
RFG Consultants

Background

Polyvinyl chloride (PVC) is one of the most useful materials available to the fabricator, being capable of formulation so as to produce a range of forms from soft coatings to durable structural articles. PVC combines good physical properties, chemical, flame, abrasion and weathering resistance with low cost. It has, nevertheless, been subject to strong non-economic competition based on the allegedly evil effects of chlorine. Thus articles are promoted as "halogen free," despite the fact that in the absence of chlorine, all life on earth would cease.

Much of this attack has been based on perceived difficulty of disposal. Considerable industrial PVC scrap is recycled as work-off into the original products or into lower end articles, such as garden hose tube. Incineration of post-consumer scrap is unattractive since HCl from combustion must be absorbed, requiring a level of investment not consistent with the fuel value of the scrap. This places emphasis on the ability to dispose of post-consumer PVC scrap by landfilling.

Hitherto PVC has been considered immortal in the landfill [1]. A number of halogenated compounds are, nevertheless, subject to biodegradation. Bacteria and fungi exist that can, in their search for organic carbon, metabolize PCB's and pentachlorophenol and are, in fact, in use in remediation of contaminated sites. These compounds are, however, of low molecular weight. In general, microorganisms can metabolize almost any organic carbon compound of relatively low molecular weight, as well as polymers, such as cellulose, if they have evolved enzymes to reduce it to low MW, such as cellulose to sugar. To date, microorganisms have not been reported that have developed enzymes capable of MW reduction of synthetic addition polymers such as PVC.

The goal, therefore, is to formulate so as to aid the microorganism. Consider generally the degradation of a polymer that contains carbon-hydrogen bonds (R-H):

$$\text{R-H} \rightarrow \text{R}\bullet \qquad (1)$$

A proton is removed by one means or another to generate a polymer radical R•. Normally this reaction is caused by heat, shear or UV light absorption. We would prefer that it occur at ordinary temperatures, under no stress, in the dark, in a landfill. Therefore the additive package must promote the above reaction under these conditions without unduly compromising processability or service life of the PVC compound under more usual conditions.

We would like R• to break apart into smaller pieces so as to facilitate microbial attack:

$$\text{R}\bullet \rightarrow \text{R'} + \text{R''}\bullet \qquad (2)$$

We wish to avoid recombination of the radicals:

$$\text{R}\bullet + \text{R}\bullet \rightarrow \text{R-R} \qquad (3)$$

This would lead to higher MW and greater resistance to biodegradation. Normally in PVC, both (2) and (3) occur, but (3) predominates. High energy in the radical tends to favor reaction (3). Therefore, an additive system favorable to biodegradation should complex the radical R• and allow it to dissipate energy in vibration as heat, favoring reaction (2).

In the presence of oxygen, the R• radicals are readily oxidized:

$$\text{R}\bullet + O_2 \rightarrow \text{R-O-O}\bullet \qquad (4)$$

This does not suit our needs. The resultant R-O-O• radicals are energetic. They react with the polymer to form more radicals:

$$\text{R-O-O}\bullet + \text{R-H} \rightarrow \text{R-O-O-H} + \text{R}\bullet \qquad (5)$$

This generates a self-sustaining chain reaction. Even worse, the R-O-O-H hydroperoxide formed itself decomposes:

$$\text{R-O-O-H} \rightarrow \text{RO}\bullet + \bullet\text{OH} \qquad (6)$$

Both of these species can remove protons from R-H, forming more R• radicals. This series of reactions is useful to our purpose to the extent that reaction (2) above dominates reaction (3). Here the landfill helps in that it limits oxygen availability so that the system is not flooded with initiating radicals to the point where reaction (3) cannot be overcome. Nevertheless, the additive system must be present at a level such that most R• radicals can be complexed and slowed down.

In the case of PVC, degradation is complicated by the loss of HCl. This is useful in that the resultant unsaturated product is more readily degraded, but unsuitable in that HCl is not only an undesirable landfill effluent, but also a hindrance to the extent to which it may convert the radical reaction sequence into ionic processes. Therefore fillers that are acid absorbers, such as fine particle calcium or magnesium carbonate should be present.

If the polymer molecular weight is reduced through degradation to the point where microbial digestion becomes possible, the fate of residual chlorine is open to speculation. The metabolic needs of bacteria and fungi are carbon, water, nitrogen and trace elements, including chlorine. The extent to which chlorine is required seems far less than would be provided by metabolism of, for example, pentachlorophenol. When chlorinated biocides are present at too low a level to be effective, it appears that certain organisms, such as the fungus *Trichoderma* and the bacterium *Pseudomonas fluorescens* then consume them (if more easily processed diet is unavailable). [2] In the case of the biocide Captan®, most of the chlorine content appears to end up as sodium and calcium salts.

A further factor is that the PVC article must contain additional food to sustain microorganisms as the polymer degrades to a molecular weight that can be metabolized, or additives that favor conversion of other ingredients, such as plasticizer, into food. This poses a problem with rigid PVC compositions where little in the way of foodstuffs is normally included. The systems developed for plasticized PVC should, however, apply to foamed rigid PVC composites with wood fiber or other cellulose derivatives.

Experimental

Following ASTM D 5526 – 94 (Reapproved 2002), Standard Test Method for Determining Anaerobic Biodegradation of Plastic Materials Under Accelerated Landfill Conditions, a mixture was prepared comprising 60% by weight of sterilized dehydrated manure (to simulate processed household waste), 30% distilled water, and 10% fermenting inoculum from an active composter. 50 g were used in sealed Petri dishes with 1/2 by 1 in. plastic samples. All experiments were run at 35° C in a dark incubator.

Control samples. Samples of untreated filter paper showed mold growth with in week and were consumed in 30 days. A sample of polylactic acid (PLA) 2 mil film was completely consumed in 7 days. A sample of 1 mil LDPE film was unchanged after 90 days. Samples of polyolefin film described as biodegradable (brown trash bags) showed slight embrittlement but were otherwise unchanged after 90 days.

Control PVC sample: A plastisol was mixed consisting of 100 parts PVC (Geon® 121), 80 parts di-isodecyl phthalate (DIDP), 10 phr Ultracarb® fine particle calcium carbonate and 2 parts dibutyltin dilaurate (DBTDL) heat stabilizer; coated as a 2 mil film on release paper and fused. Samples were unchanged after 90 days landfill exposure. This was repeated using di-octyl adipate (DOA) in place of DIDP. After 90 days, there was visible mold growth on the film but no other evidence of decomposition. This sample is shown as Figure 1.This was repeated with the addition of 2.5 parts of a 4% solution of isothiazolone biocide (MicroChek 11, Ferro). In this case, there was no evidence of mold growth after 90 days. DBTDL was used as the stabilizer because it is also an active hydrolysis catalyst to promote plasticizer degradation.

Sample A: The above plastisol was mixed using DOA, DBTDL plus 5 parts of Additive A, a titanium-based adduct containing a methacrylamide group [3]. Fused samples were consumed in the test landfill within 10 days, vanishing to the visible eye. Suspended strips of moist pH paper remained in the range of 6-7, indicating little or no HCl evolution. This sample is shown as Figure 2. The experiment was repeated adding 2.5 parts of Micro-Chek® 11 biocide, with identical results. Control samples containing calcined clay in place of Ultracarb® calcium carbonate also decomposed but rapidly acid discolored pH paper.

Sample B: The above plastisol was mixed with the di-isononyl ester of cyclohexane dicarboxylic acid (DINCH®, BASF) replacing DOA. Fused samples disappeared in 7 days, with or without added biocide.

Sample C: The last was repeated with Additive B, the zirconate analog of Additive A, and with basic magnesium carbonate replacing calcium carbonate. Fused samples disappeared within 10 days. Again, suspended strips of moist pH paper showed little or no HCl volatilization.

Sample D: The above plastisol was mixed using DINCH, Additive A and dibutyltin maleate ester heat stabilizer (PlastiStab 2808, Halstab) in place of DBTDL. Fused samples disappeared within 10 days.

Sample E: The same plastisol was mixed using DINCH, Additive A, and 2 parts of a liquid calcium/zinc stabilizer (PlastiStab 3002, Halstab) in place of DBTDL organotin. After 90 days, the fused sample had heavy mold growth and had fragmented but was still visibly of the same dimensions.

Discussion

The systems developed for PVC are quite different from those for polyolefins, usually based on manganese or cobalt prodegradants. The latter appear to be ineffective in PVC. In related experiments, replacement of Additive A or B with cobalt or manganese stearate yielded samples that appeared unaffected after 60 days landfill exposure at 30 C, other than the appearance of slight mold growth.

The elements of successful landfill biodegradation of PVC comprise a combination of additives to absorb HCl, promote plasticizer hydrolysis, facilitate HCl elimination during degradation, and to stabilize radicals formed long enough to favor reaction (2) above over reaction (3). The use of an organotin carboxylate stabilizer provides a combination heat stabilizer/hydrolysis catalyst.

Variations of samples A through D and the control PVC sample, all containing 5 phr titanium dioxide, were subjected to 1000 hours UV-A exposure at 30 C. All showed ΔE increases in color in the range of 1-2 units, with little or no differences among them. It is likely that such products would be useful in non-permanent outdoor displays such as billboard backings. This is an area where landfill disposal of significant quantities of flexible PVC would have utility.

It remains an open question whether unplasticized PVC could be formulated to show a similar response. This might be possible with PVC/wood fiber composites, cellulose substituting for more or less edible plasticizers.

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3. US Patent pending. Assignee Ultraflex Systems will license.

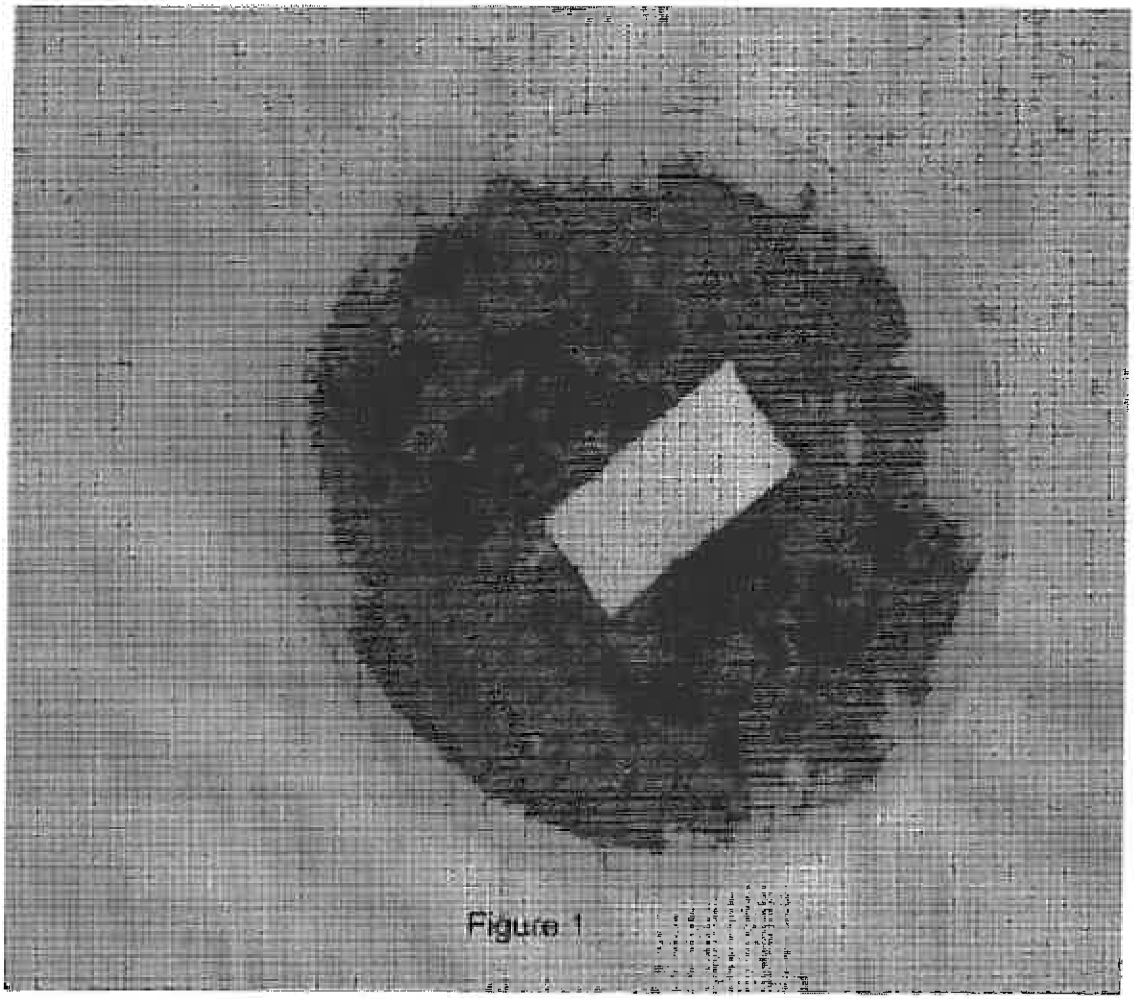

Figure 1

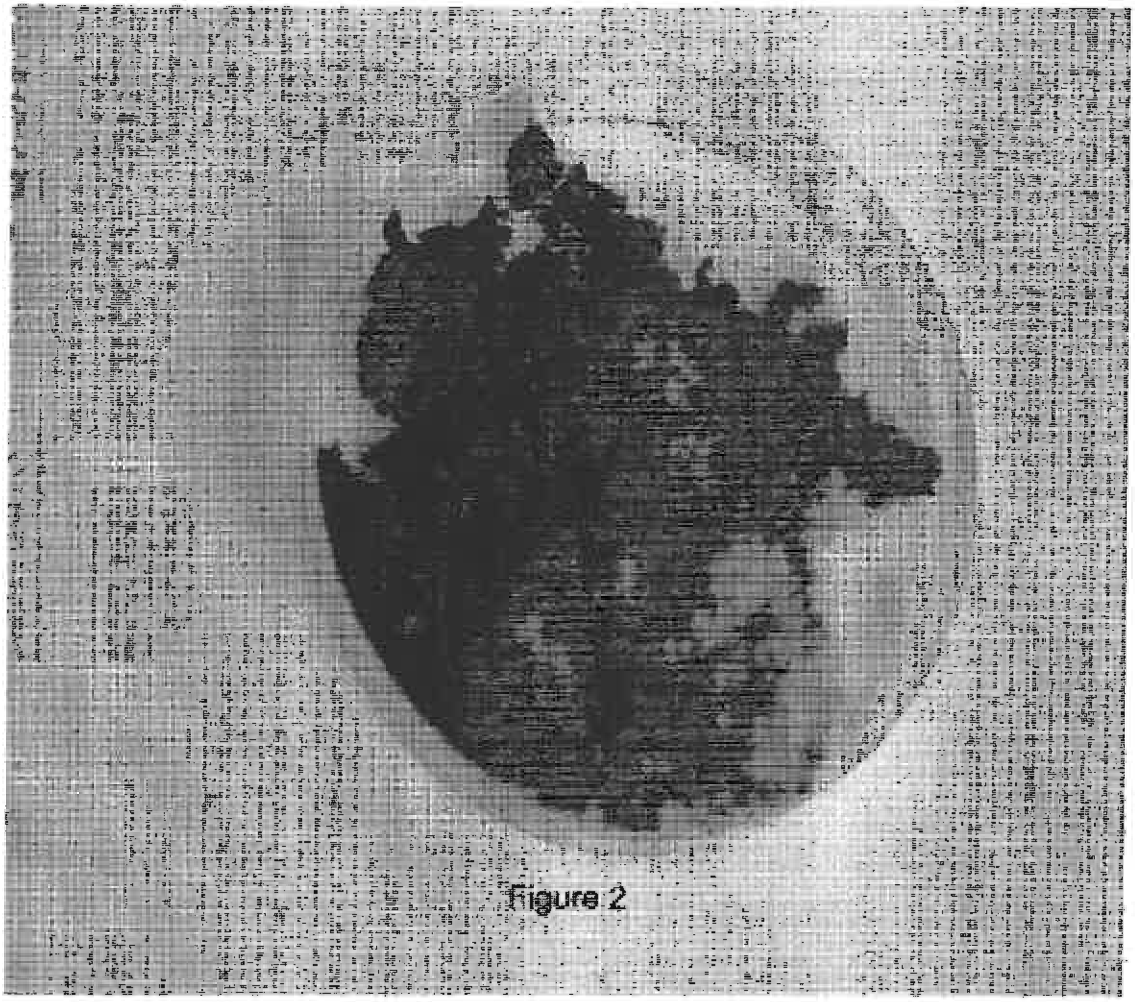

Figure 2

#3

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PATENT & TRADEMARK OFFICE

PATENT
ATTORNEY DOCKET NO: 08688/025001

COMBINED DECLARATION AND POWER OF ATTORNEY

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled POLYLACTIC ACID BASED BLENDS, the specification of which

☐ is attached hereto.
■ was filed on 04/03/97 as Application Serial No. 08/825,810 and was amended on _________.
☐ was described and claimed in PCT International Application No. ________________ filed on _______________ and as amended under PCT Article 19 on _______________.

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose all information I know to be material to patentability in accordance with Title 37, Code of Federal Regulations, §1.56.

I hereby appoint the following attorneys and/or agents to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith: J. Peter Fasse, Reg. No. 32,983, Y. Rocky Tsao, Reg. No. 34,053 2

Address all telephone calls to J. Peter Fasse at telephone number 617/542-5070.

Address all correspondence to J. Peter Fasse, Fish & Richardson P.C., 225 Franklin Street, Boston, MA 02110-2804.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patents issued thereon.

Full Name of Inventor: Stephen P. McCarthy 1-00
Inventor's Signature: ____________ Date: 9/22/97
Residence Address: Tyngsboro, Massachusetts MA
Citizen of: U.S.A.
Post Office Address: 7 Westland Road, Tyngsboro, Massachusetts 01879

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over

Revised: August 24, 1994 (391DECL.MRG)

COMBINED L ARATION AND POWER OF A RNEY CONTINUED

Full Name of Inventor: Richard A. Gross 2-00

Inventor's Signature: Date: Oct 3 1997

Residence Address: Chelmsford, Massachusetts MA

Citizen of: U.S.A.

Post Office Address: 11 Ruthellen Road, Chelmsford, Massachusetts 01824

Full Name of Inventor: Wenguang Ma 3-00

Inventor's Signature: Date: 8/4/97

Residence Address: 9 Ellis Avenue, 1st Floor, Lowell, Massachusetts 01854 MA

Citizen of: China

Post Office Address: 9 Ellis Avenue, 1st Floor, Lowell, Massachusetts 01854

08825810.040397

Revised: August 24, 1994 (391DECL.MRG)

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JUL 27 1998

PATENT
ATTORNEY DOCKET NO. 08688/025001

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : McCarthy et al.
Serial No.: 08/825,810
Filed : April 3, 1997
Title : POLYLACTIC ACID-BASED BLENDS

Art Unit: 1314
Examiner: A. Woodward

Assistant Commissioner for Patents
Washington, DC 20231

RESPONSE TO OFFICE ACTION DATED FEBRUARY 24, 1998

Please enter the following amendments.

In the Claims:

Amend claims 1, 2-9, and 11-22 as follows.

A1

1. (Amended) A biodegradable blend comprising:

(a) a first polylactic acid-based polymer or copolymer, and

(b) a second polymer [or copolymer] consisting essentially of one or more polyesters,

wherein said first and second polymers are present in a ratio of 9:1 to 1:9 by weight, and wherein the second polymer is a homopolymer or random copolymer that forms a continuous or co-continuous phase in the blend.

Claims 2-9 and 11-22, first word, replace "A" with --The--.

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GROUP 1300
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Claim 9, line 2, insert --polyethylene-- before "terephthalate".

Claim 18, line 4, delete "general".

Date of Deposit July 24, 1998
I hereby certify under 37 CFR 1.8(a) that this correspondence is being deposited with the United States Postal Service as first class mail with sufficient postage on the date indicated above and is addressed to the Assistant Commissioner for Patents, Washington, D.C. 20231.

Madeleine E. Betancourt

REMARKS

Claims 1 to 25 are pending. Claims 1 to 9 and 11 to 22 have been amended. These amendments add no new matter. In particular, support for the claim language "the second polymer forms a continuous or co-continuous phase in the blend" is found in the specification, e.g., page 10, lines 22-26. The concept that the second polymer is a homopolymer or a random copolymer is described in the application, e.g., at page 10, lines 17-21. Support for the term "polyethylene terephthalate" is found in the specification, e.g., page 11, line 12. Other amendments are grammatical in nature. Claims 10 and 23 to 25 have been restricted by the Examiner; however, applicants have not cancelled these claims, in view of their request for consideration of the restriction requirement, which is discussed in further detail below.

The Invention

The invention is based on the discovery that polylactic acid (PLA)-based polymers or copolymers and polymers of one or more polyesters can be used to make new biodegradable blends. Compared to PLA alone, these blends have superior tensile and mechanical properties such as stiffness, toughness, and elongation to break, as well as excellent biodegradability and aging properties. The PLA component can be based on lactic acid homopolymers and/or block, graft, or random copolymers of lactic acid with other organic units. The polyester component can be based on homopolymers or random copolymers of C_2 to C_{20} aliphatic or aromatic diacids and diols. The polyester component forms a continuous or co-continuous phase in the biodegradable blends of the invention.

Restriction Requirement

Applicants hereby affirm the election of Group I, claims 1 to 9 and 11 to 22. This election is made with traversal of the restriction requirement for the following reasons.

Applicants respectfully submit that with respect to at least Group II, claim 10, there would be no serious additional burden on the Examiner to include this claim in her search. M.P.E.P. at section 803 provides, "If the search and examination of an entire application can be made without serious burden, the examiner must examine it on the merits, even though it includes claims to distinct or independent inventions."

With respect to the claims of Groups I and II, applicants submit that the addition of a compatibilizer (Group II) to the constituents of the biodegradable blend (Group I) does not impose a serious additional search burden on the Examiner. Furthermore, the compositions in the already cited prior art of Sinclair '050, Sinclair '642, and Kakizawa are all three component polymer blends. The search already conducted therefore presently encompasses three component blends. Accordingly, applicants respectfully request reconsideration and withdrawal of the restriction requirement between Group I and Group II, which includes only a single claim, claim 10.

With respect to Groups III and IV, applicants submit that if the blend of claim 1 is patentable, then products made of this blend are also patentable, with no additional search required. Thus, claims 23 to 25 should also be added to Group I and the restriction withdrawn.

35 U.S.C. § 112, Second Paragraph

Claims 1-9 and 11-22 have been rejected as being allegedly indefinite. Applicants respectfully traverse the rejections, in light of the claim amendments and for the following reasons.

The term "polylactic acid-based polymer" in claims 1, 3-5, 18, 20, and 21 was said to be redundant. Applicants submit that the term "polylactic acid" is in widespread use in the field of polymer chemistry. The material is commercially available, and is used to make further polymers. Applicants appreciate the Examiner's concern with respect to potential confusion in the claim language. However, considering the well-established usage of the term "polylactic acid" to refer to the homopolymer and copolymers made from that material, applicants respectfully request that the rejection on this ground be withdrawn.

The phrase "polymer or copolymer consisting essentially of one or more polyesters" in claim 1 was said to be unclear. In light of the claim amendment deleting the words "or copolymer," applicants respectfully request that the rejection on this ground be withdrawn.

The preambular --The-- was suggested by the Examiner instead of the preambular "A" in claims 2-9 and 11-22. Although applicants do not agree that this amendment is required, or changes the scope of the claims, they have made this change requested by the Examiner in the interest of moving the application towards allowance. The rejection on this ground can thus be withdrawn.

The term "homopolymer of polylactic acid" in claims 3, 20, and 21 was said to be unclear. In light of the arguments made above regarding the well-established use of the term "polylactic acid," applicants respectfully request withdrawal of the rejection on this ground.

The term "terephthalate" in claim 9 was said to be indefinite. In view of the claim amendment changing the term to --polyethylene terephthalate-- (supported in applicant's specification, e.g., at page 11, lines 10-12), applicants respectfully request withdrawal of the rejection on this ground.

The term "general" in claim 18 was found to be indefinite. This word has been deleted from the claim; applicants respectfully request withdrawal of the rejection on this ground.

The formula in claim 19 was asserted to be indefinite because it lacked end groups. Claim 19 depends from claim 1 which is directed to a biodegradable blend comprising first and second polymers. End groups are not included in the structure shown in claim 19 because the claim does not require the polyester structure to be linked to any particular group, but rather it can be linked to a variety of different groups. For example, the structure shown in claim 19 can be linked to another polyester unit, or a polylactic acid based polymer or copolymer. This method of identifying polymer segments is well known to those of ordinary skill in the art. Thus, applicants submit that claim 19 as it stands is clear and definite, and respectfully request reconsideration and withdrawal of the rejection on this ground.

Applicants believe that the amended claims meet all requirements of 35 U.S.C. § 112, second paragraph, and respectfully request reconsideration and withdrawal of the rejections.

35 U.S.C. § 102(b) and/or 103(a) over Japanese Abstract 96-231,837

Claims 1-5, 7-9 and 11-18 have been rejected as allegedly anticipated, or in the alternative as allegedly obvious over the Japanese Abstract to Shimadzu Corp. Applicants respectfully traverse the rejection for the following reasons.

The Shimadzu abstract discloses a composition made by mixing fraction A, a lactic acid-based copolymer, with fraction B, a 2-component block copolymer having segments B_1, an aliphatic polyester other than those derived from lactic acid, and B_2, a polyalkylene ether. The ratio of segments B_1 to B_2 can be, for example, 1:1, 1:2, 2:1, etc., but the weight fraction of B_1 is desirably 70-95% of the total weight of fraction B. Thus, the composition of Shimadzu requires the presence of at least 5% by weight of a polyalkylene ether in block copolymer B.

The biodegradable blend composition of the present invention, in contrast, does not contain any polyalkylene ether component. As summarized above, applicants' composition comprises a blend of first lactic acid homo- and copolymers, and a second polymer <u>consisting essentially of</u> various polyesters. Polyalkylene ethers do not appear in any claim of the invention, and the presence of any polyalkylene ethers is not required for the advantageous properties of the inventive blend.

The biodegradable blend composition of the present invention also does not utilize a block copolymer as the second polymer. Instead, the second polymer is a homopolymer or a random copolymer. The Shimadzu reference requires that fraction B of that composition be a block copolymer. The present invention does not mention the use of block copolymers of any of its components, and refers specifically to the use of homopolymers and "random copolymers" (e.g., page 10, lines 17-21) of aliphatic polyesters for the biodegradable blends of the invention. Therefore, the Shimadzu reference does not anticipate applicants' biodegradable blend.

Furthermore, the Shimadzu reference does not make the inventive blend obvious. Shimadzu does not disclose or otherwise suggest that polyalkylene ethers could be omitted from their compositions. Likewise, Shimadzu does not disclose or otherwise suggest that block copolymers could be omitted in favor of random copolymers in their compositions. Absent even a suggestion, or motivation to modify the disclosures in the prior art, a *prima facie* case of obviousness has not been established. Applicants respectfully request reconsideration and withdrawal of the rejections based on Shimadzu.

<u>35 U.S.C. § 102(b) or 103(a) over Sinclair (U.S. Patent No. 5,216,050) or Sinclair (U.S. Patent No. 5,252,642)</u>

Claims 1, 3-5, 11-18, and 22 have been rejected as allegedly anticipated by, or in the alternative, obvious in view of the Sinclair '050 or '642 patents. Applicants respectfully traverse these rejections for the following reasons.

Sinclair '050 discloses degradable compositions comprising blends of polylactic acid and polymers based on polyethylene terephthalate and other polymers. No other examples of polyesters which could be blended with polylactic acid are given in Sinclair '050. The compositions are made up of "heterogeneous domains of 3 microns or less" as revealed by optical microscopy at 310 x (Col. 4, lines 63-65). These domains are spherical, and are believed to consist of polylactic acid which is "mostly buried" (Col. 8, lines 3-6).

In view of the amendment to claim 1, reciting the formation of a continuous or co-continuous phase of the second polymer in the blend, applicants submit that the invention is not anticipated by Sinclair '050. In applicants' claimed blends, the first and second polymers form an interlocking lattice of materials. The polylactic acid component of the claimed blend is not at all "mostly buried," but instead is evenly distributed and accessible throughout the blend, as demonstrated by applicants' Example 5 in the present application.

Example 5 shows that when a 70% PLA, 30% Bionolle #3000 blend was exposed to acetone, approximately 95% of the PLA was dissolved, leaving a continuous sheet of polyester material with interconnecting holes where the PLA used to be. This result demonstrates that this polyester component forms a continuous structure within the blend. Furthermore, since 95% of the PLA dissolved, the PLA component of the blend is also continuous and accessible to the acetone, and not "buried," as in Sinclair '050. If PLA were mostly buried in the present blends, 95% PLA dissolution could not take place.

The presence of spherical, heterogeneous domains in the blend of Sinclair '050 is quite different from applicants' claimed continuous or co-continuous phase of the second polymer in the blend, and thus Sinclair '050 does not anticipate the present claims.

In addition, Sinclair '050 fails to render the present claims obvious. Sinclair '050 contains no suggestion that blends of polylactic acid and polyethylene terephthalate have a continuous or co-continuous nature, or that other polymers could be added to create such a co-continuous blend. To the contrary, Sinclair '050 specifically notes that the polylactic acid in his blends is present in mostly buried spherical heterogeneous domains of 3 microns or less. This stands in strong contrast to applicants' teaching, which is that the polyester is present as a continuous or co-continuous phase in the claimed blends.

Thus, the claimed invention is neither anticipated by, nor made obvious by the Sinclair '050 patent, and applicants respectfully request reconsideration and withdrawal of the rejection on this ground.

Sinclair '642 discloses degradable compositions comprising blends of lactic acid with block copolymers of hard, crystalline segments of polybutylene terephthalate and soft, long chain segments of polyether glycol. The block copolymers act as impact modifiers, are "present as small spherical domains" (Col. 8, lines 17-21) and "must be elastomeric and intimately bound into the poly(lactic acid) as a discrete heterogeneous phase" (Col. 8, lines 38-40).

The present invention, in distinct contrast, does not contain block copolymers of polybutylene terephthalate blended with polylactic acid as a discrete heterogeneous phase in small spherical domains. The presently claimed blends contain a continuous or co-continuous phase of homopolymers or random copolymers of polyesters with polymers or copolymers of polylactic acid. Thus, Sinclair '642 does not anticipate the present invention.

Moreover, Sinclair '642 does not render the present invention obvious. As noted with respect to Sinclair '050, Sinclair '642 does not disclose or suggest compositions which could be made by replacing the block polybutylene terephthalate with another polyester to achieve Applicants' claimed blends, which have a continuous or co-continuous phase of polyester within the polylactic acid. Again, absent a suggestion or motivation in the art to modify the reference to exclude block polybutylene terephthalate from the compositions of Sinclair '642, a *prima facie* case of obviousness has not been established.

Applicants submit that Sinclair '642 neither anticipates nor makes obvious the present invention, and respectfully request reconsideration and withdrawal of the rejection over the reference.

Rejection under 35 U.S.C. § 102(e) or 103(a) over Kakizawa (U.S. Patent No. 5,686,540)

Claims 1-9 and 11-22 have been rejected as allegedly anticipated by, or in the alternative, made obvious by Kakizawa. Applicants respectfully traverse these rejections because Kakizawa is not prior art.

The subject matter of the claimed invention was invented before the filing date of Kakizawa (September 27, 1996), and is therefore not prior art. Applicants earlier date of invention is supported by the appended Declaration under Rule 131, submitted herewith as Exhibit A.[1] Specifically, applicants formulated various of the claimed blends of polylactic acid and polyesters and measured a number of their physical properties prior to Kakizawa's filing date (Exhibit A, paragraphs 5 and 6).

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[1] The attached Declaration has been signed by two of the inventors. Applicants will submit a Supplemental Amendment including the Declaration signed by all three inventors as soon as possible.

Based on applicants' evidence of prior invention, Kakizawa is not prior art. Accordingly, applicants respectfully request reconsideration and withdrawal of this rejection.

CONCLUSION

The present invention is not anticipated or made obvious in light of the prior art cited by the Office Action. Applicants respectfully request withdrawal of all rejections and a Notice of Allowance as the next mailing from the Office. Please apply any further charges or credits to Deposit Account No. 06-1050, making reference to Attorney Docket No. 08688/025001.

Respectfully submitted,

Date: July 24, 1998

for Gary E. Hill R-42,148
J. Peter Fasse
Reg. No. 32,983

Fish & Richardson P.C.
225 Franklin Street
Boston, MA 02110-2804

Telephone: 617/542-5070
Facsimile: 617/542-8906
36662.N11

US005883199A

United States Patent [19]

McCarthy et al.

[11] **Patent Number: 5,883,199**

[45] **Date of Patent: Mar. 16, 1999**

[54] **POLYACTIC ACID-BASED BLENDS**

[75] Inventors: **Stephen P. McCarthy,** Tyngsboro;
Richard A. Gross, Chelmsford;
Wenguang Ma, Lowell, all of Mass.

[73] Assignee: **University of Massachusetts**, Boston, Mass.

[21] Appl. No.: **825,810**

[22] Filed: **Apr. 3, 1997**

[51] **Int. Cl.^{6}** **C08F 20/00**; B29D 22/00

[52] **U.S. Cl.** **525/437**; 525/450; 604/212; 604/370; 604/403; 604/408; 428/35.2; 428/35.7; 428/36.92

[58] **Field of Search** 525/437, 450; 604/403, 212, 358, 370, 408; 428/34.1, 35.2, 35.7, 36.92

[56] **References Cited**

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Sheth et al., "Biodegradable Polymer Blends of Polylactic Acid (PLA) and Polyethylene Glycol (PEG)", *ANTEC '95*, 1829–1833 (1995).

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Primary Examiner—Nathan M. Nutter
Attorney, Agent, or Firm—Fish & Richardson P.C.

[57] **ABSTRACT**

Biodegradable blends including a first, polylactic acid-based polymer or copolymer, and a second polymer or copolymer including one or more polyesters, e.g., an aliphatic polyester or a polyester of one aliphatic C_2 to C_{20} diacid or of a combination of two more different aliphatic C_2 to C_{20} diacids, wherein the first and second polymers are present in a ratio of 9:1 to 1:9, are described.

25 Claims, 7 Drawing Sheets

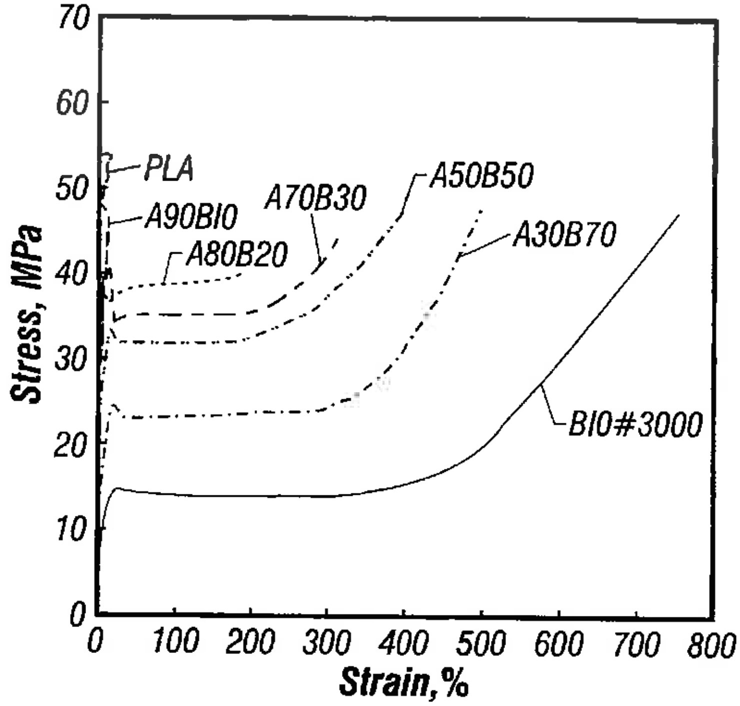
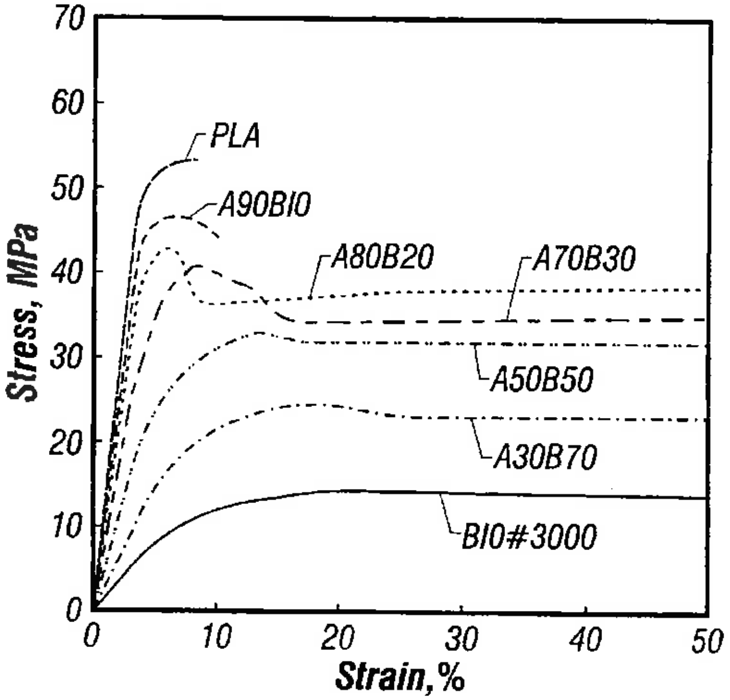

70
60
50
40
30
20
10
0
Stress, MPa
0
100
200
300
400
500
600
700
800
Strain,%
PLA
A90B10
A70B30
A80B20
A50B50
A30B70
B10#3000

FIG. 1


70
60
50
40
30
20
10
0
Stress, MPa
0
10
20
30
40
50
Strain,%
PLA
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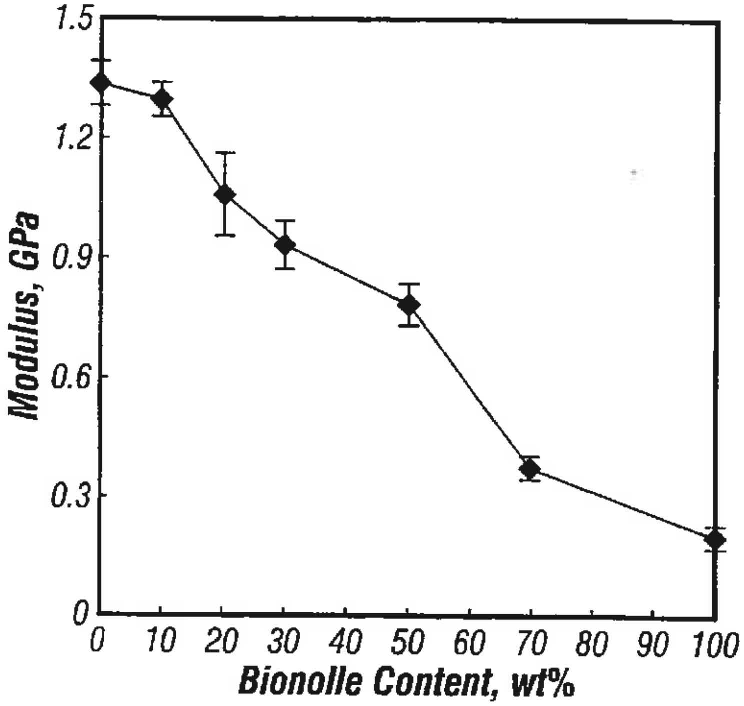
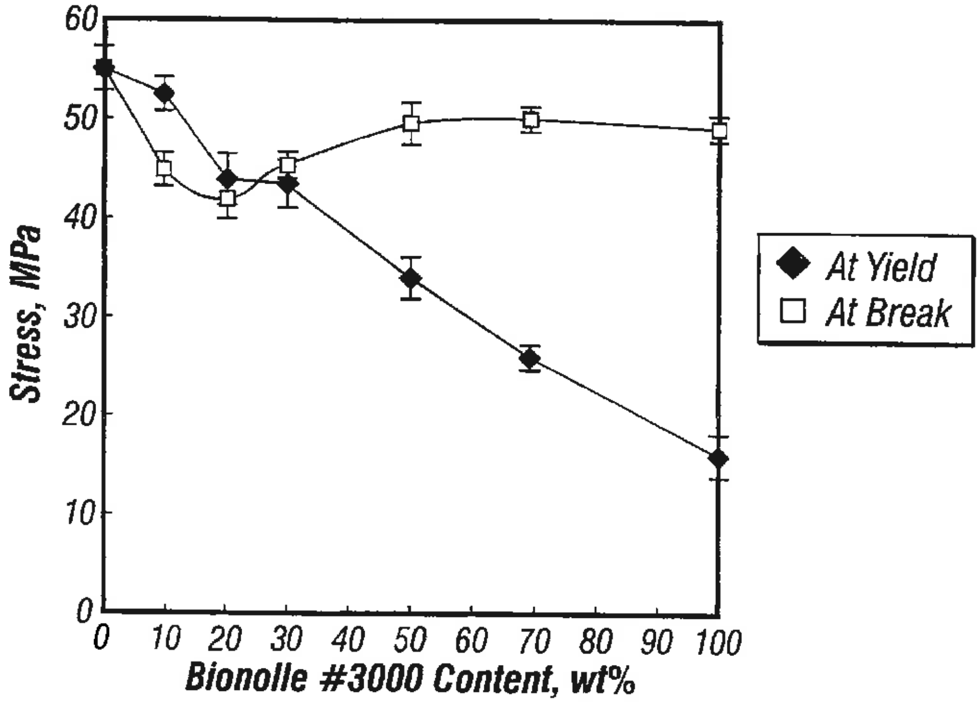
A80B20
A70B30
A50B50
A30B70
B10#3000

FIG. 2



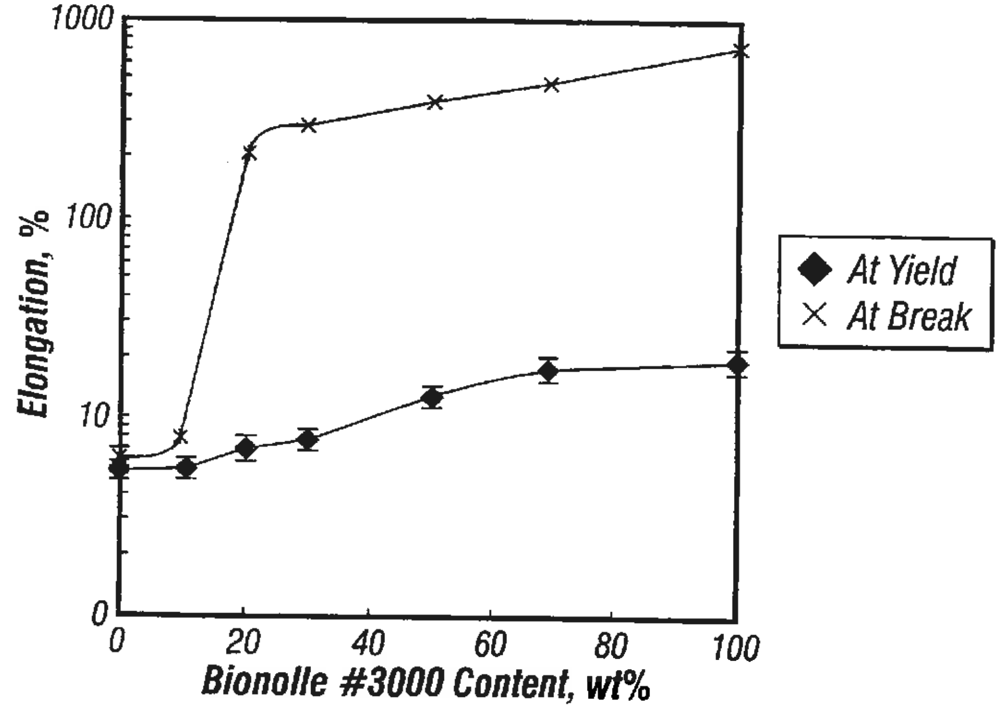
1.5
1.2
0.9
0.6
0.3
0
Modulus, GPa
0
10
20
30
40
50
60
70
80
90
100
Bionolle Content, wt%

FIG. 3



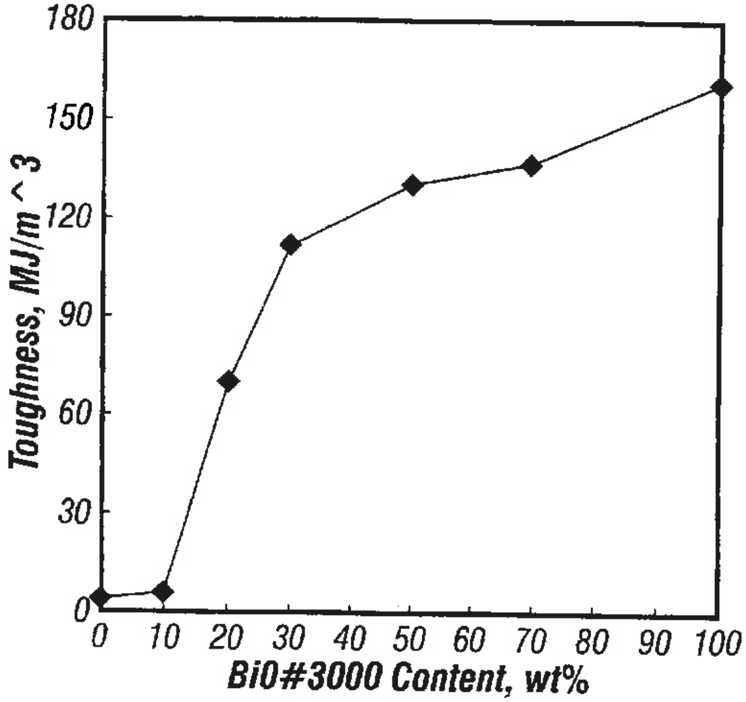
60
50
40
30
20
10
0
Stress, MPa
0
10
20
30
40
50
60
70
80
90
100
Bionolle #3000 Content, wt%
◆ At Yield
☐ At Break

FIG. 4



1000
100
10
0
Elongation, %
0
20
40
60
80
100
Bionolle #3000 Content, wt%
◆ At Yield
× At Break

FIG. 5



180
150
120
90
60
30
0
Toughness, MJ/m ^ 3
0
10
20
30
40
50
60
70
80
90
100
BiO#3000 Content, wt%

FIG. 6

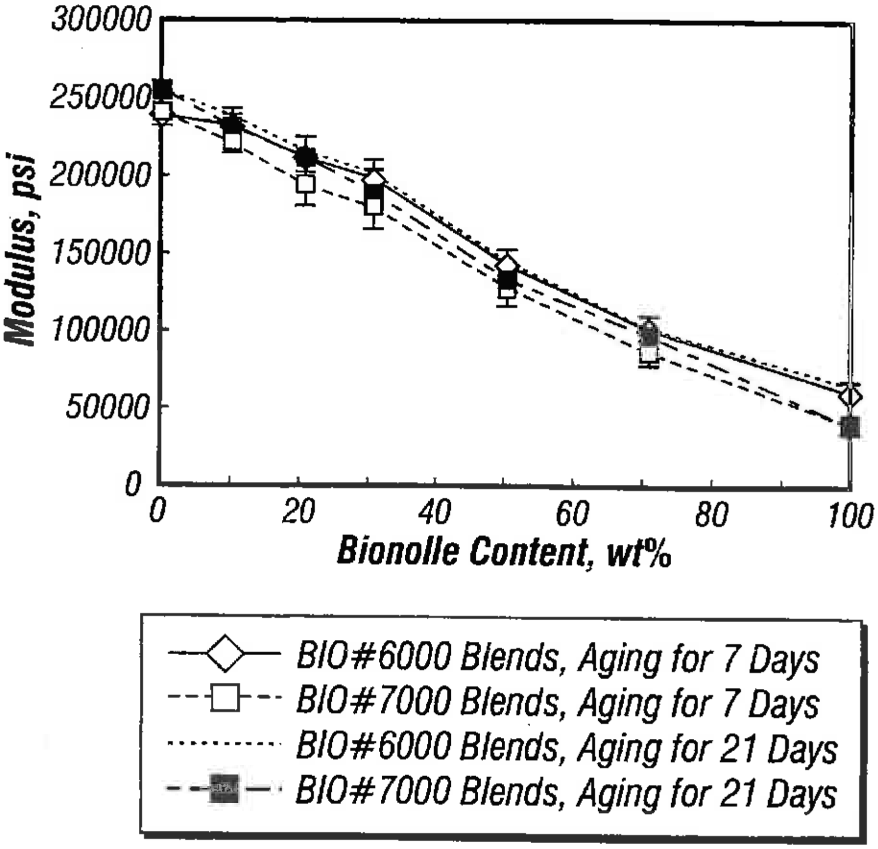

300000
250000
200000
150000
100000
50000
0
Modulus, psi
0
20
40
60
80
100
Bionolle Content, wt%
BIO#6000 Blends, Aging for 7 Days
BIO#7000 Blends, Aging for 7 Days
BIO#6000 Blends, Aging for 21 Days
BIO#7000 Blends, Aging for 21 Days

FIG. 7

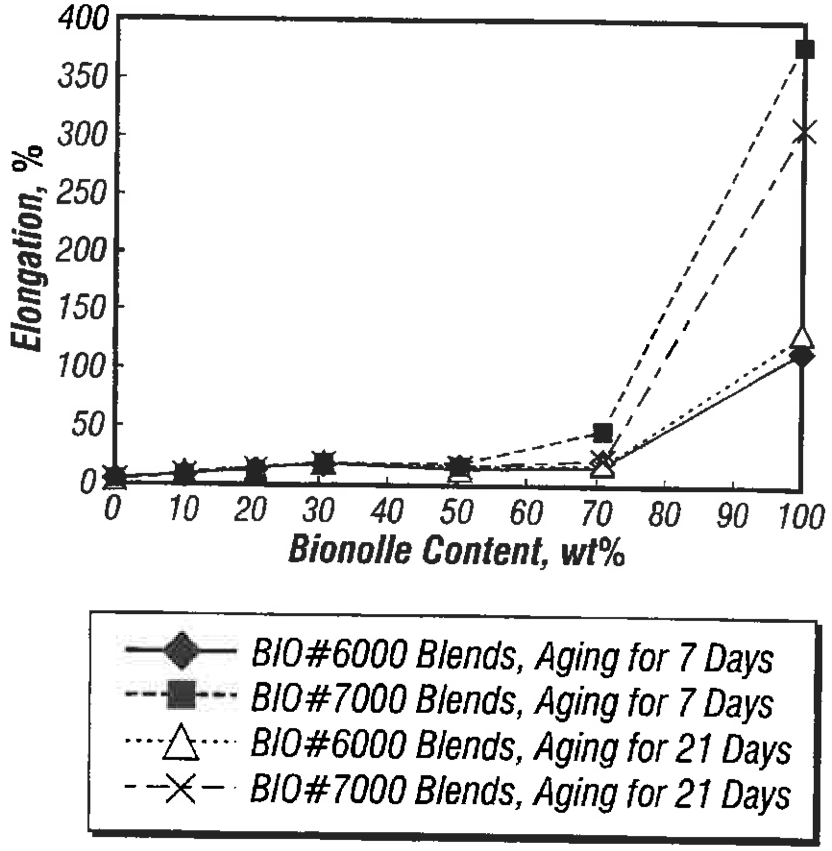
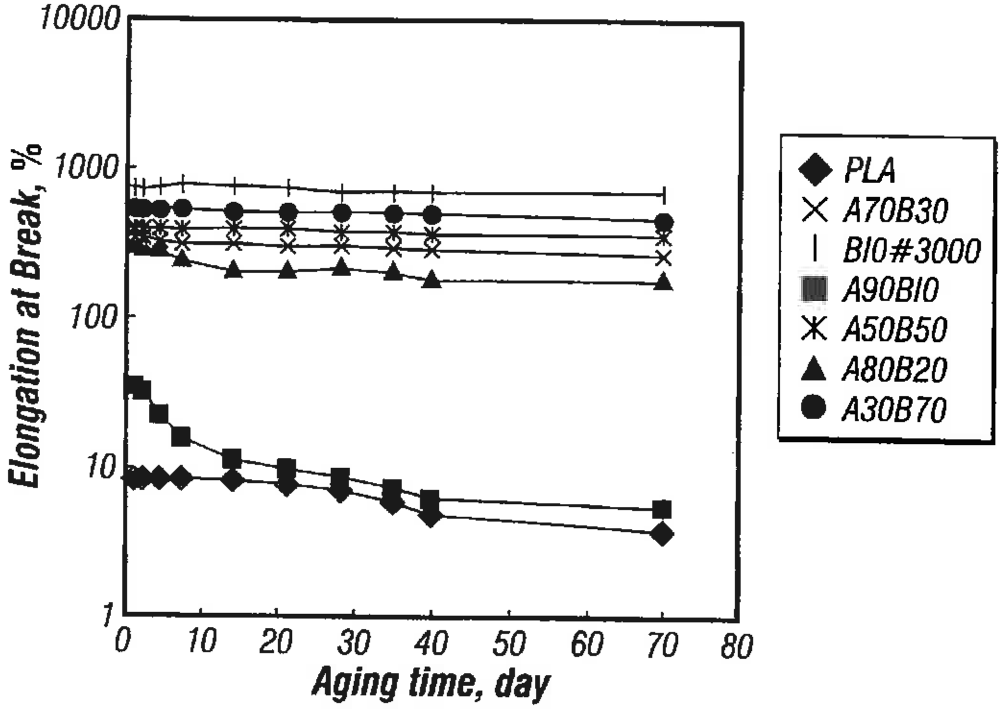
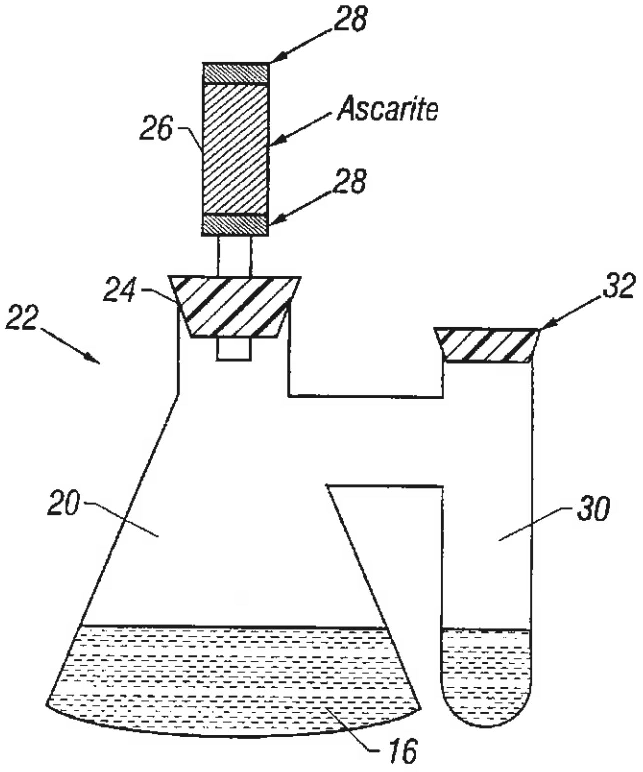

400
350
300
250
200
150
100
50
0
Elongation, %
0
10
20
30
40
50
60
70
80
90
100
Bionolle Content, wt%
BIO#6000 Blends, Aging for 7 Days
BIO#7000 Blends, Aging for 7 Days
BIO#6000 Blends, Aging for 21 Days
BIO#7000 Blends, Aging for 21 Days

FIG. 8



10000
1000
100
10
1
Elongation at Break, %
0
10
20
30
40
50
60
70
80
Aging time, day
◆ PLA
× A70B30
| BI0#3000
■ A90BI0
✳ A50B50
▲ A80B20
● A30B70

FIG. 9



28
Ascarite
26
28
24
22
32
20
30
16

FIG. 10

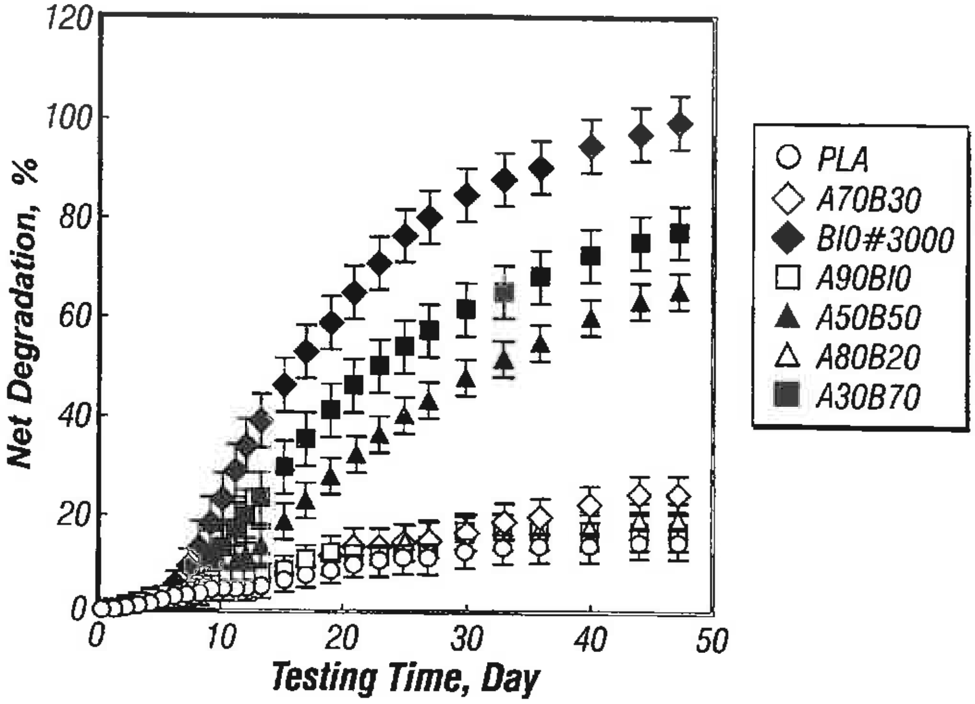

120
100
80
60
40
20
0
Net Degradation, %
0
10
20
30
40
50
Testing Time, Day
○ PLA
◇ A70B30
◆ BI0#3000
□ A90BI0
▲ A50B50
△ A80B20
■ A30B70

FIG. 11

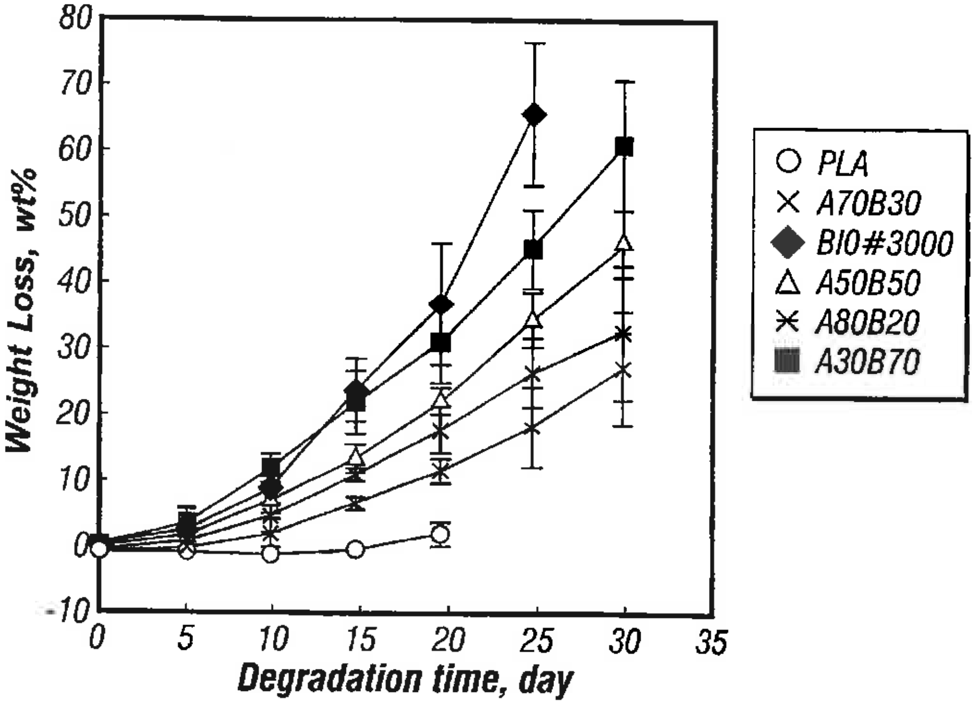

80
70
60
50
40
30
20
10
0
-10
Weight Loss, wt%
0
5
10
15
20
25
30
35
Degradation time, day
○ PLA
× A70B30
◆ BI0#3000
△ A50B50
⋇ A80B20
■ A30B70

FIG. 12

POLYACTIC ACID-BASED BLENDS

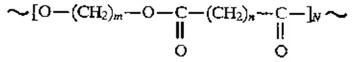
STATEMENT AS TO FEDERALLY SPONSORED RESEARCH

Funding for the work described herein was partially provided by the National Science Foundation under grant number EEC-9314562. The Government has certain rights in the invention.

BACKGROUND OF THE INVENTION

The invention relates to polylactic acid-based blends.

Succinic acid and diols can form biodegradable aliphatic polyesters and copolyesters through coupling and polycondensation reactions. The main unit structure resulting from these reactions is:



$$\sim[O-(CH_2)_m-O-\underset{\underset{O}{\|}}{C}-(CH_2)_n-\underset{\underset{O}{\|}}{C}-]_N\sim$$

Examples of biodegradable aliphatic polyesters and copolyesters having the unit structure shown above are polybutylene succinate (PBSU), where m is 4 and n is 2, polyethylene succinate (PESU), where m is 2 and n is 2, a random copolymer of polybutylene succinate adipate (PBSU-AD) where m is 4 and n is 2 or 4, and polyethylene succinate adipate (PESU-AD) where m is 2 and n is 2 or 4.

These polyesters and copolyesters have interesting properties including biodegradability, melt processability, and thermal and chemical resistance. One of these, BIONOLLE®, a commercially available aliphatic succinate-adipate polyester, has excellent physical properties. For example, the thermal resistance of BIONOLLE is equivalent to that of polyethylene, but the yield strength is higher than polyethylene. The stiffness of BIONOLLE is between high density and low density polyethylene (LDPE). Particularly for BIONOLLE #3000, its impact strength is equivalent to that of LDPE, while its elongation at break is higher than that of LDPE.

Polylactic acid can be made from lactic acid (lactate). Lactic acid is a natural molecule that is widely employed in foods as a preservative and a flavoring agent. It is the main building block in the chemical synthesis of the polylactide family of polymers. Although it can be synthesized chemically, lactic acid is procured principally by microbial fermentation of sugars such as glucose or hexose. These sugar feed stocks can be derived from potato skins, corn, and dairy wastes. The lactic acid monomers produced by fermentation are then used to prepare polylactide polymers.

Lactic acid exists essentially in two stereoisomeric forms, which give rise to several morphologically distinct polymers: D-polylactic acid, L-polylactic acid, D,L-polylactic acid, meso-polylactic acids and any combinations of thereof. D-polylactic acid and L-polylactic acid are stereoregular polymers. D,L-polylactic acid is a racemic polymer obtained from a mixture of D- and L-lactic acid, and meso-polylactic acid can be obtained from D,L-lactide. The polymers obtained from the optically active D and L monomers are semicrystalline materials, but the optically inactive D,L-polylactic acid is amorphous.

Lactic acid has a hydroxyl group as well as a carboxylic group, and hence can be easily converted into a polyester. These polyesters have some potential advantages when compared to other biodegradable polymers such as polyhyroxybutyrate and polycaprolactone, as to their strength, thermoplastic behavior, biocompatibility, and availability from renewable sources, and have been classified as "water sensitive," because they degrade slowly compared with "water soluble" or "water swollen" polymers. However, while polylactic acid is a biodegradable polymer with generally good processability, it is brittle, and the brittleness increases with time due to physical aging, i.e., densification of the polymer at a molecular level.

SUMMARY OF THE INVENTION

The invention is based on the discovery that polylactic acid (PLA)-based polymers or copolymers and polymers or copolymers of polyesters, e.g., polybutylenesuccinate, polybutylene succinate-adipate or polybutylene succinate-terephthalte (wherein the diacids of the polyester would be, for example, succinic acid, adipic acid, terephthalic acid, or any combinations thereof), can be used to make new biodegradable blends that, compared to PLA, have superior tensile and mechanical properties such as stiffness, toughness, and elongation to break, as well as excellent biodegradability and aging properties.

In general, the invention features a biodegradable blend including a first, polylactic acid-based polymer or copolymer, and a second polymer or copolymer including one or more polyesters, e.g., an aliphatic polyester or a polyester of one aliphatic C_2 to C_{20} diacid or of a combination of two more different aliphatic C_2 to C_{20} diacids, wherein the first and second polymers are present in a ratio of 9:1 to 1:9, by weight, e.g., 5:1 to 1:5, or 2:1 to 1:2, or 1:1. For example, the first polymer can be a homopolymer of polylactic acid, e.g., D-polylactic acid, L-polylactic acid, D,L-polylactic acid, meso-polylactic acid, and any combination of D-polylactic acid, L-polylactic acid, D,L-polylactic acid and meso-polylactic acid. In addition, the first polymer can be a copolymer having at least 50, 60, 70, or more, up to 100 percent, by weight, of polylactic acid.

The second polymer or copolymer can be, for example, a polybutylenesuccinate homopolymer, polybutyleneadipate homopolymer, polybutylenesuccinate-adipate copolymer, polyethylenesuccinate homopolymer, polyethyleneadipate homopolymer, or a polyethylenesuccinate-adipate copolymer, or a copolyester of an aliphatic polyester and up to 50 percent, by weight, of an aromatic polyester, such as terephthalate, as long as the overall copolyester (and second polymer) is biodegradable.

The blend can further include a compatibilizer including one or more polyesters, polyethers, or polyvinyl alcohols.

The new biodegradable blends have an elongation at break of at least 10 percent, for example, at least 50, 100, 200, 300, 400, and up to 500 percent or more. The blends also have an elongation at break of at least 10 percent, e.g., 50, 100, 200, 300, 400, and up to 500 percent or more after 70 days of aging. In addition, the blends have a toughness of at least 10 MJ/m^3, e.g., 20, 40, 60, and up to 120 MJ/m^3 or more.

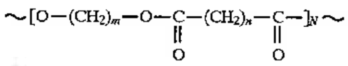
The second polymer can be present in the new biodegradable blends as a co-continuous phase with the first polymer, and at least the first or the second polymer or copolymer is present in a continuous phase in the blend.

The first, polylactic acid-based polymer or copolymer can be a homopolymer of lactic acid or a block, graft, or random copolymer of lactic acid having the general formula:

$$-(R_1)_a-(R_2)_b-$$

wherein R_1 is a lactic acid unit, R_2 is caprolactone, glycolide, trimethylene carbonate, dioxanone, butyryl lactone, or ethylene oxide, a is 10 to 10,000, e.g., 100 to 7,500, or 1000 to 5000, and b is 0 to 10,000, e.g., 100 to 7,500, or 1000 to 5000.

The polyester of the second polymer or copolymer can have the formula:



$$\sim[O-(CH_2)_m-O-\underset{\displaystyle O}{\underset{\|}{C}}-(CH_2)_n-\underset{\displaystyle O}{\underset{\|}{C}}-]_N\sim$$

wherein m is 2 to 20, e.g., 4, 8, or 12; n is 2 to 20, e.g., 2 and 4, or 6, or 8; and N is 10 to 10,000, e.g., 500, 3,500, or 5000.

The new biodegradable blends can include the first, polylactic acid-based polymer or copolymer as a polylactic acid homopolymer, and the second polymer or copolymer as a polybutylenesuccinate homopolymer, polybutyleneadipate homopolymer, polybutylenesuccinate-adipate copolymer, polyethylenesuccinate homopolymer, polyethyleneadipate homopolymer, or a polyethylenesuccinate-adipate copolymer.

In another embodiment, the invention features articles manufactured from the new biodegradable blends. For example, the invention features sheets or films, bags, containers, such as bottles and disposable cups, disposable diapers, and other items including the new blends.

A "polylactic acid-based polymer or copolymer" is a homopolymer or a copolymer having at least 50% by weight of polylactic acid. As used herein, the term "polylactic acid," without further designation, includes any one or more of four morphologically distinct polylactic acid polymers: D-polylactic acid, L-polylactic acid, D,L-polylactic acid, and meso-polylactic acid. "D-polylactic acid" and "L-polylactic acid" are dextro-polylactic acid and levo-polylactic acid, respectively, and both of them are optically active polymers that rotate a light vector when transmitted through the polymer. "D,L-polylactic acid" is a racemic polymer, i.e., a copolymer of D-polylactic acid and L-polylactic acid having a well-defined conformation of D- and L-polylactic acid units. "Meso-polylactic" is a random copolymer of D-polylactic and L-polylactic. An "aliphatic polyester of a diacid and a diol" is a polyester formed by the reaction of a diacid and a diol.

The invention provides several advantages. Polylactic acid by itself is a brittle material having poor toughness and low elongation to break, and these properties worsen with time due to its physical aging behavior. Furthermore, the biodegradability of polylactic acid is slow. The new blends overcome these deficiencies of polylactic acid. Moreover, the new blends are environmentally friendly and commercially attractive for making biodegradable plastic films, sheets, and other plastic products made by conventional processing methods such as blown film, extrusion, and injection molding. These plastic products can be used for food packaging, compost bags, and other disposable items. The new blends provide an entry for polylactic acid in the potentially large market of biodegradable polymers.

Unless otherwise defined, all technical and scientific terms used herein have the same meaning as commonly understood by one of ordinary skill in the art to which this invention belongs. Although methods and materials similar or equivalent to those described herein can be used in the practice or testing of the present invention, suitable methods and materials are described below. All publications, patent applications, patents, and other references mentioned herein are incorporated by reference in their entirety. In case of conflict, the present specification, including definitions, will control. In addition, the materials, methods, and examples described herein are illustrative only and not intended to be limiting.

Other features and advantages of the invention will be apparent from the following detailed description, and from the claims.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. **1** is a graph showing complete stress-strain curves of polylactic acid, BIONOLLE#3000, and their blends.

FIG. **2** is a graph showing stress-strain curves of polylactic acid, BIONOLLE#3000, and their blends in the strain range of 0 to 50%.

FIG. **3** is a graph showing stiffness (modulus) of polylactic acid, BIONOLLE#3000, and their blends.

FIG. **4** is a graph showing stress at yield and break of polylactic acid, BIONOLLE#3000, and their blends.

FIG. **5** is a graph showing percent elongation at yield and break of polylactic acid, BIONOLLE#3000, and their blends.

FIG. **6** is a graph showing toughness of polylactic acid, BIONOLLE#3000, and their blends.

FIG. **7** is a graph showing stiffness (modulus) of polylactic acid, BIONOLLE#6000, BIONOLLE#7000, and their blends.

FIG. **8** is a graph showing percent elongation at yield and break of polylactic acid, BIONOLLE#6000, BIONOLLE#7000, and their blends.

FIG. **9** is a graph showing percent elongation at break of polylactic acid, BIONOLLE#3000, and their blends as a function of aging.

FIG. **10** is a schematic of a biometer for soil biodegradation testing.

FIG. **11** is a graph showing net percent biodegradation of polylactic acid, BIONOLLE#3000, and their blends as a function of test time in soil.

FIG. **12** is a graph showing net percent weight loss due to biodegradation of polylactic acid, BIONOLLE#3000, and their blends as a function of test time in compost.

DETAILED DESCRIPTION

Polylactic acid-based polymers and polymers of polyesters, e.g., aliphatic polyesters of diols and diacids, can be used to make new blends that have surprisingly good mechanical and biodegradable properties compared to polylactic acid alone. The new blends provide tough, biodegradable plastics that can be used to make biodegradable plastic films, sheets, and other products made by conventional blown film, extrusion, and injection molding processing methods. These plastic products can be used for food packaging, compost bags, and other disposable items.

Compared to polylactic acid, the new blends provide a large increase in elongation (e.g., from 5% to 500%), toughness enhancement (from less than 10 MJ/m^3 to more than 120 MJ/m^3), and increased biodegradation rate. The modulus of these blends decreases with increasing amount of the aliphatic polyester, i.e., Bionolle#3000 (from 1.3 GPa of polylactic acid to 0.3 GPa of Bionolle#3000), and elongation to break increases with increasing amount of the aliphatic polyester (e.g., from 5% to 500%). The blends with more than 20% by weight of Bionolle#3000 possess toughness of more than 70 MJ/m^3, more than 200% elongation at break and other excellent tensile properties, which are retained even after the blends have aged for 70 days in the temperature range of −15° to 60° C. Compared to polylactic acid, these blends also have a relatively high degradation rates in soil and composting environment.

Materials

The main components needed to make the new blends are polylactic acid-based polymers and polyesters, e.g., aliphatic polyesters of diols and diacids. Optionally, a compatibilizer may be added to the blends.

The simplest polylactic acid-based polymer is polylactic acid, which can be obtained from, e.g., Cargill Inc. (EcoPla Division, Minnesota). The polylactic acid used for the experiments described herein had an 8% meso content (96% L) and a number average molecular weight of 70,100. Other polylactic-based polymers can also be used to make the new tough blends with aliphatic polyesters of diols and diacids.

For example, a polylactic-based polymers can be either a homopolymer of lactic acid or a block, graft, or random copolymer of lactic acid having the general formula:

$$—(R_1)_a—(R_2)_b—$$

wherein R_1 is a lactic acid unit and R_2 is caprolactone, glycolide, trimethylene carbonate, dioxanone, butyryl lactone, or ethylene oxide. When the polylactic acid-based polymer is a homopolymer, the b term is zero in the general formula.

Commercially available aliphatic polyesters of diols and diacids include the BIONOLLE family of polymers, e.g., BIONOLLE #1000, #2000, #3000, #6000, and #7000, which can be obtained from, e.g., Showa Highpolymer Co., Ltd, Japan. Bionolle #3000, #6000, and #7000, which have molecular weights (M_W) of 23,300, 250,000 and 270,000, respectively, and melting points of about 91°, 102°, and 89° C., respectively, were used to make the new blends which were tested as described below. Other aliphatic polyesters of diols and diacids can also be used.

Examples of diols in the aliphatic polyesters include any aliphatic diols including ethylene glycol and 1,4-butanediol. Examples of diacids in the aliphatic polyesters include any individual diacids or combinations of two or more aliphatic diacids, in the range of C_2 to C_{20}, in a weight percent from 0 to 100, e.g., oxalic acid, malonic acid, succinic acid, glutaric acid, adipic acid, n-butylmalonic acid, succinic acid, azelaic acid, sebacic acid, ethyl diethylmalonate and dibutyl succinate. Specific aliphatic polyesters include polybutylene succinate (PBSU), polyethylene succinate (PESU), random copolymers of polybutylene succinate adipate (PBSU-AD), and polyethylene succinate adipate (PESU-AD).

Among other features of the aliphatic polyesters used in the new blends are that these polyesters are biodegradable and that they impart ductility to polylactic acid-based polymers by forming a continuous or co-continuous phase in the morphology of the blends. The polylactic acid-based polymers and the aliphatic polyesters are immiscible, but synergistically compatible in the blends, i.e., the properties of the blends are greater than that of the mixtures of polylactic acid-based polymer and aliphatic polyester determined by the additive rule of mixture. The range of weight average molecular weights of the polylactic acid-based polymer and the aliphatic polyester that can be used is 5,000 to a million,

for example 10,000 to 500,000 or 15,000 to 250,000. The range of melting points of the polylactic acid-based polymer and aliphatic polyester that can be used is 50° to 300° C., for example 60 to 200° C., e.g., 80° to 150° C.

Besides a purely aliphatic polyester of diols and diacids, a copolyester of an aliphatic polyester and an aromatic polyester can be used so long as the copolyester is biodegradable and imparts ductility to polylactic acid-based polymers. An example of an aromatic polyester that can be used (in up to 50 percent by weight) in the copolyester is polyethylene terephthalate. Other aromatic polyesters can be used.

Examples of compatibilizers include AB block or AB graft copolymers that consist of a polylactic acid-based polymer or a polymer which is miscible with the polylactic acid-based polymers, and an aliphatic copolyester of polymers based on diols and diacids or polymers which are miscible with these aliphatic copolyesters. These compatibilizers can be added to the blend in an amount ranging from, e.g., 0.1 to 10 percent, e.g., 2, 3, or 5 percent.

Preparing Polylactic-Based Polymer Blends

Standard melt processing equipment and processing conditions can be used to prepare the new blends. Examples of polymer melt processing equipment that can be used to make the new blends include melt mixers (Banbury mixer), blenders, extruders for sheet, film, profile and blown-film extrusion, vulcanizers, calenders, and spinnerets for fiber spinning, molding, and foaming.

The polylactic acid-based polymers and the polymers or copolymers of polyesters were carefully dried at 40° C. under vacuum for at least 24 hours to minimize hydrolytic degradation of polylactic acid-based polymer during the subsequent melt processing. Blending was done on a single screw extruder operating between 150° and 160° C. and a screw speed of 50 rpm. Each sample was extruded twice. This protocol can be varied as long as the polymers and polyesters form a continuous or co-continuous phase blend.

The composition and sample code for each blend made up of polylactic acid and BIONOLLE are reported in Table 1. The A in each sample code refers to the percentage of polylactic acid-based polymer in the blend, and the B refers to the polyester, BIONOLLE#3000, BIONOLLE#6000, or BIONOLLE#7000, which were used to make the new blends with polylactic acid.

TABLE 1

Sample Code	PLA	A90B10	A80B20	A70B30	A50B50	A30B70	Bio#_
PLA wt %	100	90	80	70	50	30	0
BIONOLLE wt %	0	10	20	30	50	70	100

Sample Preparation

Rectangular shaped samples of each blend were prepared to enable uniform testing of characteristics. The tensile test samples were made according to a modified specification in ASTM D 882. In particular, samples of about 0.3 mm thickness, 12.7 mm width, and 38.1 mm length between the grips of the tensile test machine holding the sample, i.e., gage length, were compression molded at 155° C. and cooled in a cooling press machine at 20° C. and 700 psi. Thin film samples were made by melt blending on an extruder and then compression molding to 0.3 mm thickness. The films were cut into 20 mm×20 mm samples for testing biodegradation in soil and in composting environments.

Testing Methods

Tensile test properties of blends were obtained 1, 2, 4, 7, 14, 21, 35, 40, and 70 days after making the samples. During this interim time period between making and testing, the samples were physically aged at room temperature and atmospheric pressure. The tensile test was done according to ASTM D 882 with the following modifications. The grip separation used was 38.1 mm (1.5 inches) instead of 50 mm (2 inches), and the grip separation rate was 2 inches/minute even for samples with elongation at break greater than 100%, while ASTM D 882 specifies that the grip separation rate be 20 inches/minute for samples with elongation at break greater than 100%.

Biodegradation testing in an artificial soil environment was performed on films of the blends using the respirometric method developed at the NSF Biodegradable Polymer Research Center, University of Massachusetts Lowell and designated UML-7645. This test method covers the determination of the degree and rate of aerobic biodegradation of synthetic plastic materials (including formulation additives) in contact with moist soil under controlled laboratory condition. Carbon dioxide production, as a fraction of the measured theoretical carbon content of the test materials, is reported as a function of time. The test is designed to determine the biodegradability of plastic materials, relative to that of a comparative standard material, in an aerobic environment. The test applies to all plastic materials that do not inhibit bacteria and fungi present in soil.

Biodegradation testing in an artificial compost environment was conducted on film samples in a simulated municipal compost as described in Example 4.

In addition, morphology of the blends was observed under polarizing optical and scanning electron microscopy.

Uses of Polylactic Acid-Based Blends

Like wood and paper, these blends are stable in the atmosphere but biodegradable in compost, in moist soil, in water with activated sludges, and in the sea, where a large number of microorganisms are present. These blends can be incinerated with only slight damage to the furnace since the heat of combustion is relatively low, and no toxic gases are generated. The blends made by this invention can be used to make biodegradable plastic film, sheets, and other products by conventional processing methods such as blown film, extrusion, and injection molding methods. The resulting blends can be used to manufacture bags, food packaging, laminated papers, food trays, fishing line, net, rope, diapers, disposable medical supplies, sanitary napkins, shampoo, drug, cosmetic, and beverage bottles, cutlery, brushes, combs, molded and extruded foamed articles such as packing material and cups, and cushions for flexible packing. These blends provide not only the excellent processibility of polyethylene, but also posses excellent properties like those of polyethylene terephthalate. In addition, these blends can be processed into films that are heat-sealable, unlike polyethylene terephthalate.

EXAMPLES

The following examples further describe the invention without limitation.

Example 1

Tensile Testing

The tensile test was done according to ASTM D 882 with the modifications in the sample length between grip separation and the grip separation rate, as stated above. Specifically, tensile testing was done by using an Instron Tensile machine, model 1137, at grip separation rates of 0.5 and 2.0 inches/minute.

Tensile test properties of blends were obtained 1, 2, 4, 7, 14, 21, 35, 40, and 70 days after making the samples. During this interim time period between preparing and testing, the samples were physically aged at room temperature and atmospheric pressure.

The stiffness of the blends was determined from the slope of the initial linear portion of the stress-strain curve. Stress was measured as the nominal stress defined as force per unit original area. Strain and elongation are used as synonymous terms, and they were measured as percent change in length per unit length of a sample. The yield point of the blends, i.e., where a large inelastic deformation starts (yielding occurs), but the material continues to deform and absorb energy long beyond that point, was characterized as the intersection of the initial linear portion of the stress-strain curve and the flat horizontal portion of the stress-strain curve.

The toughness of the blends, which can be defined as the tensile energy to break according to ASTM D 822, was measured according to ASTM D 822 by integrating the area under the stress-strain curve.

Specifically, a load range such that a specimen would fail within its upper two thirds was selected. The cross sectional area of the specimen at several points along its length was measured to an accuracy of 0.0025 mm. The initial grip separation was at 38.1 mm. The rate of grip separation rate was set at 0.5 inches/minute for samples with less than 20% elongation at break, and at 2 inches/minute for samples with more than 20% elongation at break. The load cell of the Instron tester was balanced, zeroed, and calibrated for measuring and recording force. The rectangular test specimen was placed in the grips of the Instron testing machine, taking care to align the long axis of the specimen with an imaginary line joining the points of attachment of the grips to the machine. The grips were tightened evenly and firmly to the degree necessary to minimize slipping of the specimen during test. The Instron machine was started and stress versus grip separation was recorded.

Tensile stress (nominal) was calculated by dividing the load by the original minimum cross-sectional area of the specimen in the loading direction. The modulus value was determined from the initial slope of the stress-strain curve. Tensile strength (nominal) at break was calculated in the same way as tensile stress except that the load at break was used in place of the maximum load. Percentage elongation at break was calculated by dividing the extension (i.e., grip separation) at the moment of rupture of the specimen by the initial length of the specimen between the grips. Yield stress and percentage elongation at yield were determined by recording the stress and percent elongation at the yield point, which was established as noted above.

Tensile stress-strain curves of blends of BIONOLLE#3000 and polylactic acid are shown in FIGS. **1** and **2**. These blends were aged for 14 days. FIG. **1** shows the complete stress-strain curves of samples coded in Table 2 as PLA, A90B10, A80B20, A70B30, A50B50, A30B70, and BIO#3000. FIG. **2** is a expanded view of the initial portion of the stress-strain curves in FIG. **1**, i.e., up to a strain of 50%. The excellent strain hardening characteristics of these blends is exhibited in FIG. **1** by the rapid increase in stress prior to break. For example, strain hardening in A30B70 occurred in the strain range of 300–500%, and the corresponding increase in stress was from about 25 MPa to about 50 MPa.

FIG. **2** shows that both stiffness and stress at yield decrease with increasing BIONOLLE#3000 content, while elongation at yield and at break increase with increasing BIONOLLE#3000 content Based on the data in FIGS. **1** and **2**, FIGS. **3** and **4** show modulus (i.e., stiffness) and stress at yield and break, respectively. The outstanding strain hardening behavior of these blends was further exemplified by the increasing difference in stress at break and stress at yield with increasing BIONOLLE#3000 content.

FIG. **5** shows that the elongation at both yield and break of polylactic acid/BIONOLLE#3000 blends increase with BIONOLLE#3000 content, with a dramatic increase at break above 10 percent BIONOLLE. FIG. **6** shows that the toughness of polylactic acid/BIONOLLE#3000 blends increases as a function of BIONOLLE#3000 content above 10 percent. Both FIGS. **5** and **6** show a surprising and unexpected increase in the elongation at break of the blends when the BIONOLLE#3000 content was increased to over about 10 weight percent to about 30 weight percent in the polylactic acid/BIONOLLE#3000 blends, and in toughness of the blends when the BIONOLLE#3000 content was increased to over about 10 eight percent to about 40 or 50 weight percent in the polylactic acid/BIONOLLE#3000 blends.

Tensile properties (modulus and elongation at break) after aging for 7 and 21 days as a function of BIONOLLE#6000 and BIONOLLE#7000 content are shown in FIGS. **7** and **8**. The modulus decreases (FIG. **7**) and the elongation at break increases (FIG. **8**) with increasing BIONOLLE#6000 and BIONOLLE#7000 content. As the aging time increases from 7 to 21 days, the modulus shows a slight increase (FIG. **7**), and the elongation at break shows a slight decrease (FIG. **8**). Since BIONOLLE#7000 is a softer polymer than BIONOLLE#6000, polylactic acid/BIONOLLE#7000 blends have a lower modulus and a higher elongation at break compared with those of polylactic acid/BIONOLLE#6000 blends.

Unlike BIONOLLE#3000, BIONOLLE#6000 and BIONOLLE#7000 do not increase the elongation at break significantly when 10 to 40% by weight of BIONOLLE#6000 or BIONOLLE#7000 is blended with polylactic acid. This may be due to the fact that pure BIONOLLE#6000 and BIONOLLE#7000 do not possess the same tensile properties of BIONOLLE#3000, and also more importantly, the compatibility of polylactic acid with BIONOLLE#6000 and BIONOLLE#7000 is not as good as that of polylactic acid and BIONOLLE#3000. However, the compatibility of polylactic acid with BIONOLLE#6000 and BIONOLLE#7000 can be improved with the addition of a suitable compatibilizer, such as a small amount of BIONOLLE#3000.

Example 2

Aging Effect

The effect of aging on the blends was measured by physically aging the samples at room temperature and atmospheric pressure, and subsequently testing the samples by tensile testing according to ASTM D 882 with the modifications already stated above.

FIG. **9** shows elongation at break of polylactic acid, BIONOLLE#3000, and their blends, as a function of aging. The elongation at break of polylactic acid was below 8%, and decreased to about 5% with aging. Similarly, the elongation at break of A90B10 was rather low (about 50%) and decreased to less than 10% with aging. However, blends having a BIONOLLE#3000 content of 20% or more by weight showed outstanding elongation at break (200% elongation for 20% BIONOLLE#3000, and similarly, 300% for 30%, 400% for 50%, and 500% for 70%, respectively). In addition, these BIONOLLE#3000 containing blends did not exhibit any significant reduction in elongation after aging.

Example 3

Biodegradation Testing in Soil

Soil testing in an artificial soil environment was performed on 0.3 mm thick films of the blends using the respirometric method developed at the NSF Biodegradable Polymer Research Center, University of Massachusetts Lowell and designated UML-7645. A standard soil mix (1:1:0.1 potting soil:sand:dehydrated cow manure by weight) was prepared and characterized. The soil test materials were exposed to the soil under controlled aerobic conditions at 30+±2° C. Carbon dioxide production, expressed as a fraction of the measured of theoretical carbon content of the test materials, was measured as a function of time. The degree of biodegradation of the test material is assessed by comparing the amount of CO_2 produced from the test material to that produced from a standard material, i.e., one that is known to biodegrade (here PLA was used for comparison).

Specifically, the soil biodegradation test was conducted as follows. Fifty grams (oven-dry weight basis) of soil was weighed into a large (14 cm) disposable weighing boat. Enough distilled water was added to the soil and mixed thoroughly to bring the soil to a moisture content of 60 to 70%. Approximately 15 g of the moist soil was set aside. The test specimen, or standard material, was added to the soil and the amended soil was mixed thoroughly. As shown in FIG. **10**, the amended soil **16** was transferred to a large chamber **20** of a 250-mL biometer flask **22**, packed to a uniform depth (about 2.5 cm), and covered by the 15 g of the moist soil set aside. The large chamber **20** was then closed with a rubber stopper **24** connected to a 3-mL plastic syringe **26** packed with a material **26** that removes any carbon dioxide from air entering the biometer during incubation, such as sodium hydroxide-coated silicon (e.g., Ascarite™), between plugs of a filter material **28**, e.g., glass wool or cotton, that allows air, but not the Ascarite™, to pass.

The combined weight of the flask, rubber stopper, and amended soil containing the test specimen was determined and recorded. Twenty mL of 0.4M sodium hydroxide was pipetted into the side-arm chamber **30** of the biometer flask **22** and the side-arm chamber **30** was sealed with a rubber stopper **32**. The biometer flask was placed in an environmental chamber at **30**° C and this chamber was kept dark.

The carbon dioxide analysis was done by reacting the carbon dioxide produced in the biometer with the sodium hydroxide in the side-arm chamber to form an aqueous solution of sodium carbonate. The amount of carbon dioxide produced was monitored by removing the sodium hydroxide from the trap and transferring it to a glass test tube to which 5 mL of 1.5M barium chloride was added. The barium chloride reacts with the sodium carbonate to form a precipitate of barium carbonate. The amount of carbon dioxide evolved was calculated by standard stoichiometric calculation.

The net degradation was measured as the ratio of carbon dioxide evolved to the amount of theoretical maximum carbon dioxide production possible by the test specimen. The theoretical maximum carbon dioxide production was determined by the total organic carbon content of the test material (by calculation, if the chemical composition was well established, or elemental analysis). The maximum amount of carbon dioxide that can be theoretically evolved was calculated by the equation:

$$Maximum\ carbon\ dioxide=[(W\times C)/100]\times[44/12]$$

where W is the weight of the test specimen; C is the percent organic carbon in the test specimen, 44 is the molecular weight of carbon dioxide, and 12 is the equivalent weight of carbon.

The biodegradation testing in soil showed that the biodegradation rate of BIONOLLE#3000 by itself was extremely fast, while the biodegradation rate of polylactic acid by itself was relatively slow.

The soil degradation testing results of the two polymers and their blends are reported in FIG. **11**. After degradation for 45 days, BIONOLLE#3000 degraded almost 100%, while polylactic acid degraded only about 14% by loss in weight. For blends with 70 and 50% BIONOLLE#3000, the degradation rate was relatively fast. After 45 days, the A30B70, A50B50, and A70B30 blends degraded about 77%, 65% and 25%, respectively, by loss in weight. FIG. **11** shows that polylactic acid biodegrades in soil, but just not quickly, and the addition of the second aliphatic polymer, such as BIONOLLE#3000, increases the biodegradation rate.

The importance of the soil biodegradation curves shown in FIG. **11** is that a specific blend can now be designed such that this blend would have a certain net degradation in a given number of days within the soil.

Example 4

Biodegradation Testing in Compost

Biodegradation testing in an artificial compost environment was conducted on film samples in a simulated municipal compost. Biodegradation testing in an artificial compost environment was conducted on compression molded film samples of dimensions 20 mm×20 mm×0.3 mm in a simulated municipal compost mixture consisting of 60% by weight of water and the rest containing shredded leaves, shredded paper, mixed frozen vegetables, meat waste, urea, and commercial compost seeds. The carbon to nitrogen (C:N) ratio of the starting mix was 14:1. The composting process was carried out for 30 days at 55° C. Triplicate test samples were removed from the composting bioreactors at an interval of 5 days and weighed to measure the weight loss per surface area in the units of $\mu g/mm^2$.

After 20 days in the composting environment at 55° C., BIONOLLE#3000 had a high weight loss rate while polylactic acid had negligible weight loss. The weight loss rates in the blends of polylactic acid and BIONOLLE#3000 after 20 days in the composting environment were between the rates of polylactic acid and BIONOLLE#3000.

The compost degradation testing results of the two polymers and their blends are reported in FIG. **12**. After degradation for 20 days, BIONOLLE#3000 degraded almost 40%, while polylactic acid degraded only about 3%, by loss in weight. For blends with 70 to 20% BIONOLLE#3000, the degradation percentage was much greater (and the rate much faster) than that of polylactic acid, e.g., after 20 days, the A30B70, A50B50, and A70B30 blends degraded about 35%, 25% and 15%, respectively, by loss in weight. FIG. **12** shows that polylactic acid biodegrades in compost, but slowly, and the addition of even 20% by weight BIONOLLE#3000 increases this biodegradation rate dramatically.

The importance of the compost biodegradation curves shown in FIG. **12** is that a specific blend can now be designed such that this blend would have a certain net degradation in a given number of days in a composting environment.

Example 5

Morphologv

Samples were analyzed by microscopy to investigate the morphology of the phases of polylactic acid versus the phases of BIONOLLE#3000. The blends were exposed to acetone to dissolve the polylactic acid component without affecting the BIONOLLE#3000 component. For the blend containing 70% by weight of polylactic acid and 30% by weight of BIONOLLE#3000, 67% of the material, or approximately 95% of the polylactic acid, was dissolved. The remaining material was in a sheet form, and the BIONOLLE#3000 phase in the original blend formed a continuous or co-continuous phase, while the dissolved polylactic acid left behind holes in the sheet-like structure of BIONOLLE#3000. This continuous or co-continuous structure of the BIONOLLE#3000 phase in the original blend explained the outstanding toughness shown in the graph of FIG. **6**.

OTHER EMBODIMENTS

It is to be understood that while the invention has been described in conjunction with the detailed description thereof, that the foregoing description is intended to illustrate and not limit the scope of the invention, which is defined by the scope of the appended claims. Other aspects, advantages, and modifications are within the scope of the following claims.

What is claimed is:

1. A biodegradable blend comprising:

(a) a first polylactic acid-based polymer or copolymer, and

(b) a second polymer consisting essentially of one or more polyesters,

wherein said first and second polymers are present in a ratio of 9:1 to 1:9 by weight, and wherein the second polymer is a homopolymer or random copolymer that forms a continuous or co-continuous phase in the blend.

2. The biodegradable blend of claim **1**, wherein said one or more polyesters are of one aliphatic C_2 to C_{20} diacid or of a combination of two more different aliphatic C_2 to C_{20} diacids.

3. The biodegradable blend of claim **1**, wherein said first, polylactic acid-based polymer is a homopolymer of polylactic acid.

4. The biodegradable blend of claim **1**, wherein said first, polylactic acid-based polymer is selected from the group consisting of D-polylactic acid, L-polylactic acid, D,L-polylactic acid, meso-polylactic acid, and any combination of D-polylactic acid, L-polylactic acid, D,L-polylactic acid and meso-polylactic acid.

5. The biodegradable blend of claim **1**, wherein said first, polylactic acid-based polymer is a copolymer having at least 60% by weight of polylactic acid.

6. The biodegradable blend of claim **1**, wherein said second polymer or copolymer is selected from the group consisting of polybutylenesuccinate homopolymer, polybutyleneadipate homopolymer, polybutylenesuccinate-adipate copolymer, polyethylenesuccinate homopolymer, polyethyleneadipate homopolymer and polyethylenesuccinate-adipate copolymer.

7. The biodegradable blend of claim **1**, wherein said polyester is an aliphatic polyester.

8. The biodegradable blend of claim **1**, wherein said second polymer or copolymer is a copolyester of an aliphatic polyester and up to 50 percent, by weight, of an aromatic polyester.

9. The biodegradable blend of claim **8**, wherein said aromatic polyester is polyethylene terephthalate.

10. A biodegradable blend of claim **1**, further comprising (c) a compatibilizer consisting essentially of one or more polyesters or polyvinyl alcohols.

11. The biodegradable blend of claim **1**, said blend having an elongation at break of at least 10%.

12. The biodegradable blend of claim **1**, said blend having an elongation at break of at least 200%.

13. The biodegradable blend of claim **1**, said blend having an elongation at break of at least 10% after 70 days of aging.

14. The biodegradable blend of claim **1**, said blend having an elongation at break of at least 200% after 70 days of aging.

15. The biodegradable blend of claim **1**, said blend having a toughness of at least 10 MJ/m^3.

16. The biodegradable blend of claim **1**, said blend having a toughness of at least 70 MJ/m^3.

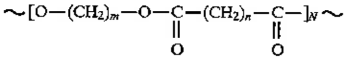
17. The biodegradable blend of claim **1**, wherein said second polymer is present in said blend as a co-continuous phase.

18. The biodegradable blend of claim **1**, wherein said first, polylactic acid-based polymer or copolymer is a homopolymer of lactic acid or a block, graft, or random copolymer of lactic acid having the formula:

$$-(R_1)_a-(R_2)_b-$$

wherein R_1 is a lactic acid unit, R_2 is caprolactone, glycolide, trimethylene carbonate, dioxanone, butyryl lactone, or ethylene oxide, a is 10 to 10,000, and b is 0 to 10,000.

19. The biodegradable blend of claim **1**, wherein said polyester has the formula:



$$\sim[O-(CH_2)_m-O-\underset{\displaystyle O}{\underset{\|}{C}}-(CH_2)_n-\underset{\displaystyle O}{\underset{\|}{C}}-]_N\sim$$

wherein m is 2 to 20, n is 2 to 20, and N is 10 to 10,000.

20. The biodegradable blend of claim **1**, wherein said first, polylactic acid-based polymer or copolymer is a polylactic acid homopolymer, and wherein said second polymer or copolymer is a polybutylenesuccinate homopolymer.

21. The biodegradable blend of claim **1**, wherein said first, polylactic acid-based polymer or copolymer is a polylactic acid homopolymer, and wherein said second polymer or copolymer is a polybutylenesuccinate-adipate copolymer.

22. A film comprising a biodegradable blend comprising:

(a) a first polylactic acid-based polymer or copolymer, and

(b) a second polymer consisting essentially of one or more polyesters,

wherein said first and second polymers are present in a ratio of 9:1 to 1:0 by weight, and wherein th second polymer is a homopolymer or random copolymer that forms a continuous or co-continuous phase in the blend.

23. A bag comprising a biodegradable blend comprising;

(a) a first polylactic acid-based polymer or co-polymer, and

(b) a Second polymer consisting essentially of one or more polyesters.

wherein said first and second polymers are present in a ratio of 9:1 to 1:0 by weight, and wherein the second polymer is a homopolymer or random copolymer that forms a continuous or co-continuous phase in the blend.

24. A container comprising a biodegradable blend comprising:

(a) a first polylactic acid-based polymer or copolymer, and

(b) a second polymer consisting essentially of one or more polyesters,

wherein said first and second polymers are present in a ratio of 9:1 to 1.0 by weight and wherein the second polymer is a homopolymer or random copolymer that forms a continuous or co-continuous phase in the blend.

25. A disposable diaper comprising a biodegradable blend comprising:

(a) a first polylactic acid-based polymer or copolymer, and

(b) a second polymer consisting essentially of one or more polyesters,

wherein said first and second polymers are present in a ratio of 9:1 to 1:0 by weight, and wherein the second polymer is a homopolymer or random copolymer that forms a continuous or co-continuous phase in the blend.

* * * * *

UNITED STATES PATENT AND TRADEMARK OFFICE

CERTIFICATE OF CORRECTION

PATENT NO. : 5,883,199
APPLICATION NO. : 08/825810
DATED : March 16, 1999
INVENTOR(S) : McCarthy et al.

Page 1 of 1

It is certified that error appears in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

On the title page item (54), and col. 1, line 1, – replace “Polyactic” with -- Polylactic –

Signed and Sealed this

Twentieth Day of February, 2007

Jon W. Dudas

JON W. DUDAS
Director of the United States Patent and Trademark Office

UNITED STATES PATENT AND TRADEMARK OFFICE

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Page 1 of 1

It is certified that error appears in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

Column 14, Claim 22, Line 4
Delete "1:0" and Insert --1:9--

Column 14, Claim 23, Line 15 (Approx)
Delete "1:0" and Insert --1:9--

Column 14, Claim 24, Line 27 (Approx)
Delete "1.0" and Insert --1:9--

Column 14, Claim 25, Line 39
Delete "1:0" and Insert --1:9--

Signed and Sealed this
Eighth Day of March, 2011

David J. Kappos

David J. Kappos
Director of the United States Patent and Trademark Office

Respondent’s Exhibit AA

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Biodegradability of conventional and bio-based plastics and natural fiber composites during composting, anaerobic digestion and long-term soil incubation

Eddie F. Gómez, Frederick C. Michel Jr. *

Department of Food, Agricultural and Biological Engineering, The Ohio State University, OARDC, 1680 Madison Avenue, Wooster, OH 44691, USA

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Bioplastics

A B S T R A C T

Plastics are a major constituent of municipal solid waste that pose a growing disposal and environmental pollution problem due to their recalcitrant nature. To reduce their environmental impacts and allow them to be transformed during organic waste recycling processes, various materials have recently been introduced to improve the biodegradability of plastics. These include conventional plastics amended with additives that are meant to enhance their biodegradability, bio based plastics and natural fiber composites. In this study, the rate and extent of mineralization of a wide range of commercially available plastic alternative materials were determined during composting, anaerobic digestion and soil incubation. The biodegradability was assessed by measuring the amount of carbon mineralized from these materials during incubation under conditions that simulate these three environments and by examination of the materials by scanning electron micrography (SEM). The results showed that during a 660 day soil incubation, substantial mineralization was observed for polyhydroxyalkanoate plastics, starch based plastics and for materials made from compost. However, only a polyhydroxyalkanoate based plastic biodegraded at a rate similar to the positive control (cellulose). No significant degradation was observed for polyethylene or polypropylene plastics or the same plastics amended with commercial additives meant to confer biodegradability. During anaerobic digestion for 50 days, 20 25% of the bio based materials but less than 2% of the additive containing plastics were converted to biogas ($CH_4 + CO_2$). After 115 days of composting, 0.6% of an additive amended polypropylene, 50% of a plastarch material and 12% of a soy wax permeated paper pulp was converted to carbon dioxide. SEM analysis showed substantial disintegration of polyhydroxyalkanoate based plastic, some surface changes for other bio based plastics and coconut coir materials but no evidence of degradation of polypropylene or polypropylene containing additives. Although certain bio based plastics and natural fibers biodegraded to an appreciable extent in the three environments, only a polyhydroxyalkanoate based resin biodegraded to significant extents during the time scale of composting and anaerobic digestion processes used for solid waste management.

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1. Introduction

Plastics are synthetic and semi synthetic polymeric compounds, derived primarily from fossil carbon sources such as crude oil and natural gas. Their mechanical properties and characteristics such as low cost, durability and processability, have led to their widespread use for diverse applications. However most commonly used plastics are very resistant to biological degradation [1]. This has led to major challenges for waste management operations especially those that are moving toward more sustainable waste management practices such as recycling, composting and anaerobic digestion.

It is estimated that of the 31 million tons of plastic waste generated annually in the U.S. only 8% is recycled [2]. Therefore, a large percentage of plastic waste is currently landfilled, or released into the environment. Throughout the world, roadsides, parks, beaches, oceans and natural areas are inundated with plastic debris pollution [3]. Waste management systems are also affected by high volumes of plastics that are often commingled with organic wastes (food scraps, wet paper, yard trimmings, soil and liquids), making it difficult and impractical to recycle both organic fractions and/or the plastics mixed with them without expensive cleaning, separation and sanitizing procedures [4].

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* Corresponding author. Tel.: +1 330 263 3859; fax: +1 330 263 3670.
E-mail address: michel.36@osu.edu (F.C. Michel).

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The fact that plastics are made from non renewable resources and their persistence in the environment and during organic recycling has resulted in global concern and intensive efforts to develop plastic materials that not only have acceptable prices and similar performance to conventional plastics, but also are made from renewable feedstocks and/or undergo biodegradation in a reasonable amount of time without leaving toxic residues [5].

Although biodegradable bio based plastics are meant to improve the sustainable use of resources, a complete life cycle analysis including disposal must be conducted [6] to insure that the solution is not worse than the problem. Many factors impact the life cycle carbon balance of plastics including the source of the feedstock used to make them, whether the material is recycled and the extent and type of biodegradation during disposal. For example, most plastics are derived largely from fossil sources such as natural gas or crude oil [7]. However the monomers used to make them can also be made from renewable resources. In Brazil, ethylene, the building block of one of the most widely used plastics, polyethylene [8] is made from ethanol derived from sugar cane. Although made from a biomass feedstock, this type of polyethylene is still essentially not biodegradable. On the other hand, petroleum can also be used to make plastics that are biodegradable. The lactic acid used to make polylactic acid (PLA) can be produced both by fermentation and synthetically from petroleum [9], and either type is biodegradable. On this basis, plastics can be classified into four types with respect to whether they are biodegradable and the source of the feedstock used to make them. These four types are conventional plastic, bio based plastic, biodegradable plastic and biodegradable bio based plastic (Table 1). Understanding the environmental benefits of these four classes of materials (Table 1) and the impact of their use on GHG emissions can be confusing and is not always straightforward.

Plastics made from petroleum, such as polyethylene, have a well defined life cycle. When landfilled, the carbon in the plastic will be sequestered and not contribute to global warming. Recycled polyethylene may contribute even less fossil CO_2 to the environment if less energy is used to recycle it than is used to make it in the first place. In these cases, conventional plastics may have less impact on GHG emissions that those designed to biodegrade.

For reasons presented above, efforts have been made to develop durable plastics made from renewable biomass feedstocks [5]. These are called "bio based plastics". On balance this type of plastic offers a great potential to reduce greenhouse gases in the atmosphere by sequestering carbon. This is because atmospheric CO_2 is fixed into the carbohydrates used as their feedstock. If the plastic is eventually landfilled, this carbon will become locked for millennia within the landfill and on balance reduce atmospheric CO_2. However these plastics also pose pollution problems [10].

Biodegradable bio based plastics, are also made from biomass but are designed to be compostable and/or biodegradable. These types include PLA and polyhydroxyalkanoates based resins (PHA) made from corn. This class of polymer is carbon neutral from the standpoint of the carbon in the plastic, but a substantial amount of fossil energy is used to produce the plastic and the biomass feedstocks.

Table 1
Classes of plastics.

Class	Source	Biodegradable	Example	Reference
I	Petroleum/natural gas	No	Polyethylene, polypropylene.	[7]
II	Petroleum/natural gas	Yes	PLA[a] from petroleum.	[9]
III	Biomass (Corn, sugar cane, etc)	No	Polyethylene derived from corn ethanol.	[8]
IV	Biomass (Corn, sugar cane, etc)	Yes	PHA[b], PLA derived from starch.	[14]

[a] Polylactic acid.
[b] Polyhydroxyalkanoates-based resin.

The class with perhaps the greatest potential to contribute to greenhouse gas emissions is biodegradable plastics made from petroleum. This is because not only is fossil energy used to produce them in the first place, but fossil carbon is released when the material ultimately biodegrades. If this biodegradation occurs in a landfill, then it usually will generate methane (CH_4), which is a greenhouse gas with 21 times the warming potential of CO_2. Most landfills do a poor job of capturing this gas, even those with methane recovery systems [11]. So landfilled biodegradable plastics, eventually contribute both methane and carbon dioxide to the atmosphere when they degrade.

Some novel polymers combine both biomass and fossil derived resins to decrease production prices, increase the bio based content and improve material performance [5] (e.g. a plastarch containing a blend of a starch based polymer and conventional plastics such as polypropylene). The biogenic renewable carbon contained in these and other biomaterials can be determined from the radioactive C_{14} signature of the product [12]. Yet these hybrid materials likely are neither recyclable nor completely biodegradable and therefore are likely worse than conventional plastics from a GHG emissions perspective.

Composting plays an important and growing role in sustainable organic waste management and recycling. However, plastics are one of the main contaminants in composts. Biodegradable plastics are meant to address this problem. Composting of these materials also reduces their environmental impact in that they will largely be converted to CO_2 and not to CH_4 as they would be in a landfill. Since this CO_2 was originally fixed from the atmosphere into renewable biomass, on balance it will not increase atmospheric CO_2.

Biodegradation is the mineralization of materials as a result of the action of naturally occurring microorganisms such as bacteria and fungi [13]. The biodegradation of plastics is limited by their molecular weight, chemical structure [14], water solubility and the fact that most plastics are xenobiotic. That is, they were not present in the environment until very recently so that the evolution of metabolic pathways necessary for their biodegradation, a process that takes millions of years, has yet to occur.

In contrast, the biodegradation of natural polymers, such as starch or cellulose by microorganisms occurs relatively rapidly. It begins with the excretion of extracellular enzymes that depolymerize these materials. Once the polymer is reduced to a size that is water soluble and able to be transported through the cell wall, microbial metabolic pathways can then mineralize it [15]. Even though microorganisms drive the biodegradation process, other non biotic chemical processes such as photo oxidation and chemical degradation may also take place before or in parallel.

Biodegradable materials are used in diverse applications. Many different biodegradable plastics are used for food packaging and for waste containment. They have also been developed for medical applications, including medical devices and for drug delivery [16]. Biodegradable plastics are used widely in agriculture, as mulching films and low tunnels [17,18] as well as guide strings and plant nursery containers [19]. The physical properties and performance of biodegradable plastics made from PLA and natural fibers were found to be similar to conventional plastics for greenhouse crop production [20]. In addition, biodegradable potting containers have gained a high degree of acceptance among consumers [21].

Recently, various materials have begun to be marketed that claim to be biodegradable or compostable. Terms such as "degradable", "oxo biodegradable", "biological", "compostable" and "green" are often used to describe and promote different plastics. These materials include conventional plastics amended with additives meant to enhance biodegradability as well as bio based plastics and natural fiber composites. There has been little research on the extent to which these materials truly degrade and/or biodegrade over the time scale of waste management processes such as composting and anaerobic digestion (AD) or in natural settings [22].

The objective of this study was to compare the relative biodegradability of a range of novel plastics and natural fiber composites during composting, AD and in soil conditions. The hypothesis was that materials that are referred to as biodegradable, compostable (or similar terms), and plastics containing additives designed to enhance biodegradability, mineralize during the time scale of waste treatment processes and in reasonable amounts of time in the environment and at rates comparable to natural materials known to be biodegradable and or compostable (e.g. cellulose paper).

2. Materials and methods

Standardized laboratory scale experiments were conducted to study the biodegradability of various materials during soil incubation, composting and AD conditions [23–25]. The extent of biodegradation was calculated by measuring the average carbon (CO_2 and or CH_4) mineralized from each treatment minus the average carbon evolved from blanks, and dividing this by the total amount of sample carbon added to each treatment. Reactors containing only the inoculum (AD), soil (soil tests) or compost (compost tests) were used as blanks.

2.1. Materials

Materials tested included plastics designed to be biodegradable, conventional plastics amended with additives that are meant to enhance biodegradability, bio based plastics and natural fiber composites (Tables 2 and 3). The positive and negative controls used for all experiments were cellulose paper (Fisher Scientific, PA, U.S.) and 100% conventional polypropylene (PP), respectively. Materials were tested both after grinding (a preliminary soil experiment only) and as 1 × 1 cm squares (thicknesses shown in Table 3).

2.2. Biodegradation in soil incubation

The extent of long term biodegradation of polymeric materials in contact with soil was determined based on ASTM D5988 03 [24]. These included PP + 2% additive, polystyrene (PS) + 2% additive, polyethylene terephthalate (PETE) + 1% additive, plastarch, a co polyester + corn based plastic, a wheat starch derived plastic and PHA (Tables 2 and 3). Six natural fiber composite materials were also tested: paper pulp, paper pulp + asphalt, coconut coir, rice hull, composted cow manure and peat fiber. All samples were incubated in triplicate for a period of 660 days.

The soil media used for the experiments was a mixture of 43% certified organic top soil, 43% no till farm soil collected at coordinates: 40.778633, 81.930873 and 14% sand. Soil was sieved to less than 2 mm particle size and large plant materials, stones, and other inert materials were removed. The chemical properties of the soil mixture are shown in Table 4. The soil media was amended with ammonium phosphate (Fisher Scientific, PA, U.S.) to maintain a C:N ratio of 20:1 based on the carbon content of the test specimen.

The soil mixture (300 g dry) was placed in the bottom of a 2 L (working volume) wide mouth jar (Ball® Corporation, item # 383178). Distilled water was added to bring the moisture content of the mixture to 60% of the moisture holding capacity. The test specimens (1 g of sample carbon) were then mixed thoroughly into the soil. A solution containing 20 ml of potassium hydroxide (KOH) 0.5 N (Fisher Scientific, PA, U.S.) was placed in a cup suspended from the lid of each vessel to trap evolved CO_2. All vessels were sealed and incubated at room temperature (20 ± 2 °C).

Carbon dioxide produced in each vessel reacted with the KOH in the cup to form potassium bicarbonate. The amount of CO_2 produced was determined by titrating the KOH solution with 0.25 N hydrochloric acid (Fisher Scientific, PA, U.S.) to a phenolphthalein end point. The experiment was designed so that the headspace volume was sufficient to prevent the oxygen concentration in the vessel from falling below 18%. The KOH traps were removed and titrated at time intervals that assured that their absorption capacity was not exceeded. The KOH traps were refilled at a rate dependent on the rate of CO_2 generation in each flask. At the time of removal of the traps, the vessel was flushed and allowed to sit open to allow fresh air to fill the headspace. In addition, distilled water was added to the soil to the original weight to maintain adequate moisture.

The effect of particle size on biodegradation rate was determined by comparing the biodegradability of 1 cm squares to ground samples. Samples were ground in liquid nitrogen using a IKA® A11 basic Analytical mill (IKA® Works Inc., NC, U.S.) for 10 s. Test specimens included PP + 2% additive, co polyester + corn based plastic, wheat starch derived plastic, paper pulp, paper pulp + asphalt, coconut coir and rice hull (Tables 2 and 3). Samples were incubated in triplicate for 660 days.

Table 2
Material information for commercially available bio-based plastics, plastics amended with additives and natural fiber composites.

Material	Material description	Formation process[a]
PP + 2% additive	Blend of polypropylene (PP) with 2% ECM MasterBatch Pellets™ additive (ECM BioFilms Inc., OH, U.S.)	1
PS + 2% additive	Blend of polystyrene (PS) with 2% ECM MasterBatch Pellets™ additive (ECM BioFilms Inc., OH, U.S.)	1
PETE + 1% additive	Blend of polyethylene terephthalate (PETE) with 1% EcoPure® additive (Bio-Tec Environmental LLC., NM, U.S.).	2
Plastarch	A blend of polypropylene with corn starch.	3
Co-polyester + corn-based plastic	Blend of an aliphatic aromatic co-polyester with a corn starch-derived polymer (Ecobras™, BASF).	1
Wheat starch-derived plastic	Made from a wheat starch-derived resin (OP-47 Bio®, Summit Plastic Company, OH, U.S.).	3
PHA	Made from polyhydroxyalkanoates-based resin (Metabolix, MA, U.S.).	1
Paper pulp + soy wax	Paper pulp pot permeated with soy wax.	4
Paper pulp	Recycled (74% minimum) paper pulp.	4
Paper pulp + asphalt	Blend of recycled (74% minimum) paper pulp + asphalt.	4
Coconut coir	Made from coconut husk.	7
Rice hull	Made from rice hull.	5
Composted cow manure	Made from composted cow manure.	6
Peat fiber	Made from Canadian sphagnum peat moss + wood pulp.	6

[a] 1 injection molding; 2 blow molding; 3 thermoforming; 4 vacuum forming; 5 compression forming; 6 pressure forming; 7 other.

Table 3
Chemical and physical properties of the test specimens.

Material	Chemical and physical properties[a]				
	Total solids (%)	Volatile solids (%dw)	Total carbon (%dw)	Total nitrogen (%dw)	Film thickness (mm)
Positive	90.3 ± 5	57.4 ± 1.1	41.8 ± 0.1	0.03 ± 0.01	0.35 ± 0.01
Negative	99.8 ± 0.1	96.3 ± 2	82.9 ± 0.1	0.06 ± 0.003	0.37 ± 0.01
PP + 2% additive	99.8 ± 0.1	97.7 ± 0.1	82.9 ± 0.3	0.04 ± 0.01	0.37 ± 0.03
PS + 2% additive	99.9 ± 0.1	97.0 ± 1.5	88.8 ± 1	0.05 ± 0.01	0.23 ± 0.01
PETE + 1% additive	99.4 ± 0.5	99.9 ± 0.1	64.6 ± 0.1	0.01 ± 0.002	0.36 ± 0.01
Plastarch	90.9 ± 2.1	57.5 ± 3	60.9 ± 0.2	0.07 ± 0.01	0.48 ± 0.03
Co-polyester + corn-based plastic	95.2 ± 0.1	99.8 ± 0.1	51.9 ± 0.3	0.10 ± 0.01	0.72 ± 0.02
Wheat starch-derived plastic	97.8 ± 0.4	98.5 ± 0.5	49.4 ± 0.1	0.74 ± 0.004	0.50 ± 0.01
PHA	99.4 ± 0.4	90.4 ± 0.5	50.7 ± 0.3	0.45 ± 0.01	0.62 ± 0.01
Paper pulp + soy wax	94.3 ± 1	91.0 ± 0.4	46.9 ± 0.3	0.06 ± 0.01	2.14 ± 0.03
Paper pulp	92.0 ± 0.1	92.0 ± 0.1	42.1 ± 0.1	0.10 ± 0.01	2.74 ± 0.01
Paper pulp + asphalt	93.4 ± 0.5	90.6 ± 0.3	46.9 ± 0.03	0.22 ± 0.02	2.61 ± 0.1
Coconut coir	96.8 ± 0.3	98.5 ± 0.5	46.7 ± 0.3	0.26 ± 0.002	1.09 ± 0.02
Rice hull	94.0 ± 0.4	89.6 ± 0.4	38.3 ± 0.1	14.1 ± 0.06	1.24 ± 0.02
Composted cow manure	92.5 ± 0.1	89.4 ± 1.0	40.5 ± 0.01	1.12 ± 0.05	2.40 ± 0.1
Peat fiber	92.1 ± 0.3	97.8 ± 0.5	45.4 ± 0.3	0.49 ± 0.07	1.74 ± 0.05

[a] Values are means ± SD of three replicates.

2.3. *Biodegradation during composting*

Three materials were tested under simulated composting conditions. These included PETE + 1% additive, plastarch and paper pulp + soy wax (Tables 2 and 3). The experiments were conducted in triplicate for a period of 115 days.

The test conditions used were based on a protocol described in ASTM D5338 98 (2003) [25]. This test is a measure of the degree and rate of carbon conversion to CO_2 under conditions that mimic a commercial scale industrial composting facility.

An 80 g sample of each test specimen was mixed with 350 g dry of mature compost inoculum (Table 4). The compost inoculum was obtained from a full scale windrow composting facility featuring a concrete surface and controlled aeration system at OARDC. The compost contained a mixture of dairy manure and hardwood sawdust as described elsewhere [26].

The compost was collected at various locations on the windrow and screened to less than 10 mm and large inert items were discarded. The screened compost was amended with ammonium phosphate (Fisher Scientific, PA, U.S.) to give a C:N ratio of 20:1 including the carbon content of the test specimen. The initial moisture content of the mixture was adjusted to 60% (wet weight basis).

The compost and test specimens were incubated in 4 L (working volume) vessels (length 30 cm and diameter 15 cm), made of PVC pipe placed in a 55 °C incubator (BioCold Environmental Inc., MO, U.S.). Each vessel contained approximately 1100 g of material on a wet weight basis. The reactors were aerated from below at 100 ± 1 ml/min to maintain aerobic conditions. To avoid drying during the experiment, air was saturated by bubbling through bottles containing water at the incubator temperature. The air exiting the vessels was passed through flasks in a separate water bath set at 9 °C to condense moisture from the off gas. The off gas was then analyzed for percent CO_2 using an infrared gas analyzer (Vaisala model GMT 220, range 0–20%). CO_2 data was automatically recorded using a Campbell Scientific model 23XL data logger for each vessel every hour. Each vessel was also equipped with a K type thermocouple to measure the temperatures of the composts mix near the center of the compost vessel, and was recorded automatically every 12 min. A more complete description of the laboratory scale composting system can be found elsewhere [27].

2.4. *Biodegradation during anaerobic digestion*

The biodegradation of four materials was compared during high solids batch anaerobic digestion. These included PP + 2% additive, PETE + 1% additive, plastarch and a co polyester + corn based plastic (Tables 2 and 3). The experiments were conducted in triplicate for a period of 50 days.

The anaerobic degradation of the polymeric materials was compared under high solids AD conditions based on a protocol described in ASTM D5511 02 [23] international standard. The test measured the conversion of samples to CO_2 and CH_4 during incubation under controlled anaerobic conditions. For this study test specimens were exposed to an active methanogenic inoculum derived from a full scale anaerobic digester treating municipal sewage sludge. These conditions resemble those found in high solids AD digestors and in biologically active landfills, but not in typical landfills where water is excluded and removed.

Table 4
Initial mean characteristics of the aerobic and anaerobic organic substrates.

Organic substrate	Chemical and physical properties[a]				
	Total solids (% ww)	Volatile solids (% dw)	Total carbon (% dw)	Total nitrogen (% dw)	pH
Compost[b] inoculum	24.3 ± 2.0	88.9 ± 1.0	48.7 ± 5.5	2.37 ± 0.2	7.95 ± 0.04
Soil mixture[c]	87.4 ± 0.1	2.96 ± 0.1	1.19 ± 0.2	0.13 ± 0.02	7.43 ± 0.4
Anaerobic seed[d] sludge	8.92 ± 0.5	59.5 ± 2.0	36.8 ± 1.0	7.21 ± 0.2	8.30 ± 0.01
Medina County[e] OFMSW	47.2 ± 7.2	60.3 ± 1.2	89.6 ± 1.3	0.92 ± 0.2	7.50 ± 0.4

[a] Values are means ± SD of three replicates.
[b] Dairy manure and hardwood sawdust mature compost.
[c] This is the value before adding water to reach 60% of the water holding capacity.
[d] Methanogenically active municipal sewage sludge.
[e] OFMSW the organic fraction of municipal solid waste.

The AD assays were conducted in 2 L (working volume) laboratory scale batch reactors. Temperatures were maintained at a mesophilic (37 ± 1 °C) range by means of incubators. Test specimens (25 g of sample carbon) were mixed with 750 g wet of methanogenically active sludge obtained in October of 2010 from a full scale (3000 m^3) anaerobic digester located at the City of Akron wastewater treatment plant and operated by KB Compost Services, Akron, Ohio [28]. This was mixed with 187.5 g wet of the organic fraction of municipal solid waste (OFMSW) of the Medina County, Ohio Solid Waste District to achieve the desired solids content for the test and to provide supplemental nutrients for the anaerobic microbial consortia. The chemical properties of the seed sludge and OFMSW substrate are shown in Table 4. Ammonium phosphate (Fisher Scientific, PA, U.S.) was added to the mixture to adjust the C:N ratio to a value of 20:1 considering the carbon content of the test specimen.

The volumetric production and CO_2 and CH_4 content of the biogas produced in the AD experiments were analyzed by volume displacement and gas chromatography as described by Gómez et al. [28], respectively. This information was used to calculate the moles of carbon emitted from each reactor.

2.5. Analytical methods

Solids content in soil, organic substrates and test specimens was determined by drying samples to a constant weight at 80 °C. The volatile solids content was determined using an ashing oven set at 500 °C for 4 h. pH was determined using a pH electrode (TMECC 04.11 A 1:5 slurry method, mass basis). Carbon (TMECC 04.01 A combustion with CO_2 detection) and nitrogen content (TMECC 04.02 D oxidation, Dumas method) were determined by the Service Testing and Research laboratory at the OARDC.

Selected test specimens were also analyzed before and after soil incubation using scanning electron microscopy (SEM) (Hitachi S 3500N, Hitachi High Technologies America, Inc., CA, U.S.). Samples were coated with platinum to a thickness of 0.2 kA° using a Hummer® 6.2 sputtering system (Anatech USA, CA, U.S.). A 15 Kv electron beam was applied.

2.6. Statistical analysis

Three independent replicates were used for each treatment. Analysis of variance (ANOVA) was calculated for the average final cumulative percent of carbon loss for each of the studies. Comparisons for all pairs of final cumulative biodegradation means were performed using Tukey–Kramer HSD analysis. All conclusions were based on a significant difference level of α 0.05. The statistical analyses were performed using JMP statistical program version 9 (SAS Institute Inc., SAS Campus Drive, NC, U.S.).

3. Results and discussion

3.1. Biodegradation during soil incubation

The importance of understanding the biodegradability of plastics in soil has increased since these are released inadvertently into the environment where they may persist. Plastics comprise a relatively large fraction of the ubiquitous pollution found world wide in both land and ocean environments [29]. In addition, intensive and semi intensive agriculture utilizes large quantities of these materials annually in the form of mulches, as plantable pots, nursery containers [30].This has resulted in the recent development of biodegradable agricultural plastics for these applications [31,32]. One example of this is biodegradable plant nursery pots. Some containers are designed to be plantable pots (e.g. rice hull and coconut coir) allowing them to degrade in the soil after planting, or to be composted at plant nurseries rather than being landfilled.

An initial experiment was conducted to assess the effect of particle size on biodegradation during soil incubation. Seven materials were tested and the amount of carbon converted to CO_2 was compared using student's *t* method for particle size effect. Student's *t* method revealed that out of the seven materials studied in this experiment, only one, a co polyester + corn based plastic, showed a significant effect of particle size on biodegradability. A significantly greater extent of biodegradation was observed for co polyester + corn based plastic in 1 × 1 cm square film form (55.1 ± 2.1%) after 660 days as compared to a ground sample of the same material (39.71 ± 2.4%). For the rest of the materials, there was not a significant effect of particle size on biodegradation. Results from this study suggested that for most of the materials studied, biodegradability in soil was not greatly affected by particle size under the experimental conditions used in the study.

A second soil experiment was conducted to evaluate the relative biodegradability of thirteen different test specimens in 1 × 1 cm square film form. These included bio based plastics, plastics amended with additives that are meant to enhance biodegradability and natural fiber composites. The experiment was conducted for a period of 660 days. The initial moisture content of the mixes was 16.6 ± 2.1% and the final mean soil moisture content on a wet weight basis across all treatments was 14.3 ± 3.3% (wet weight basis) which is 84.9 ± 2.4% of the 60% moisture holding capacity of the soil mixture. The positive control (cellulose paper) exhibited 74.2 ± 4.5% conversion during the period of incubation.

For some bio based plastics and the positive controls (cellulose paper), the initial rate of mineralization was rapid (Fig. 1). Most of the mineralization took place during the first 300 days of incubation (Fig. 1). The most rapid initial rate of conversion was observed for co polyester + corn based plastic with almost 34.6 ± 2.4% mineralized during the first 55 days of the experiment. The extent of PHA biodegradation was initially lower, but its extent surpassed that of co polyester + corn based plastic after approximately 280 days reaching a value of 48.5 ± 4.6%. For the wheat starch derived plastic and plastarch conversion rates were 14.2 ± 0.8 and 24.6 ± 1.4% after 110 and 280 days of experiment, respectively.

Final (660 days) cumulative biodegradation values during soil incubation for the positive control, PHA and co polyester + corn based plastic were 74.2 ± 4.5, 69.2 ± 6.4 and 55.1 ± 6.1%, respectively. For the wheat starch derived plastic and plastarch the final conversion reached 19.7 ± 1.1 and 31.3 ± 1.7%, respectively.

SEM images of PHA and co polyester + corn based plastic before and after mineralization showed substantial changes in the

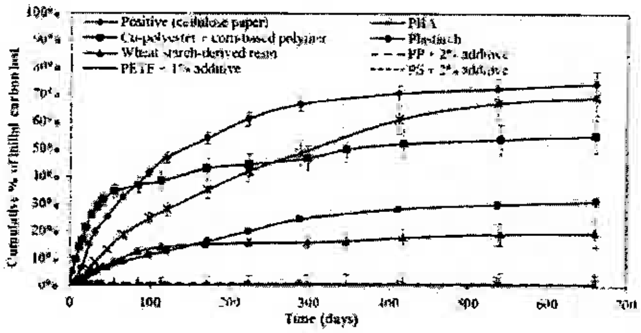

Fig. 1. Cumulative carbon loss (CO_2 C) as percentage of initial carbon (±cumulative standard error) for bio-based plastics and for conventional plastics amended with additives during 660 days of soil incubation. For some data points standard error bars are smaller than markers.

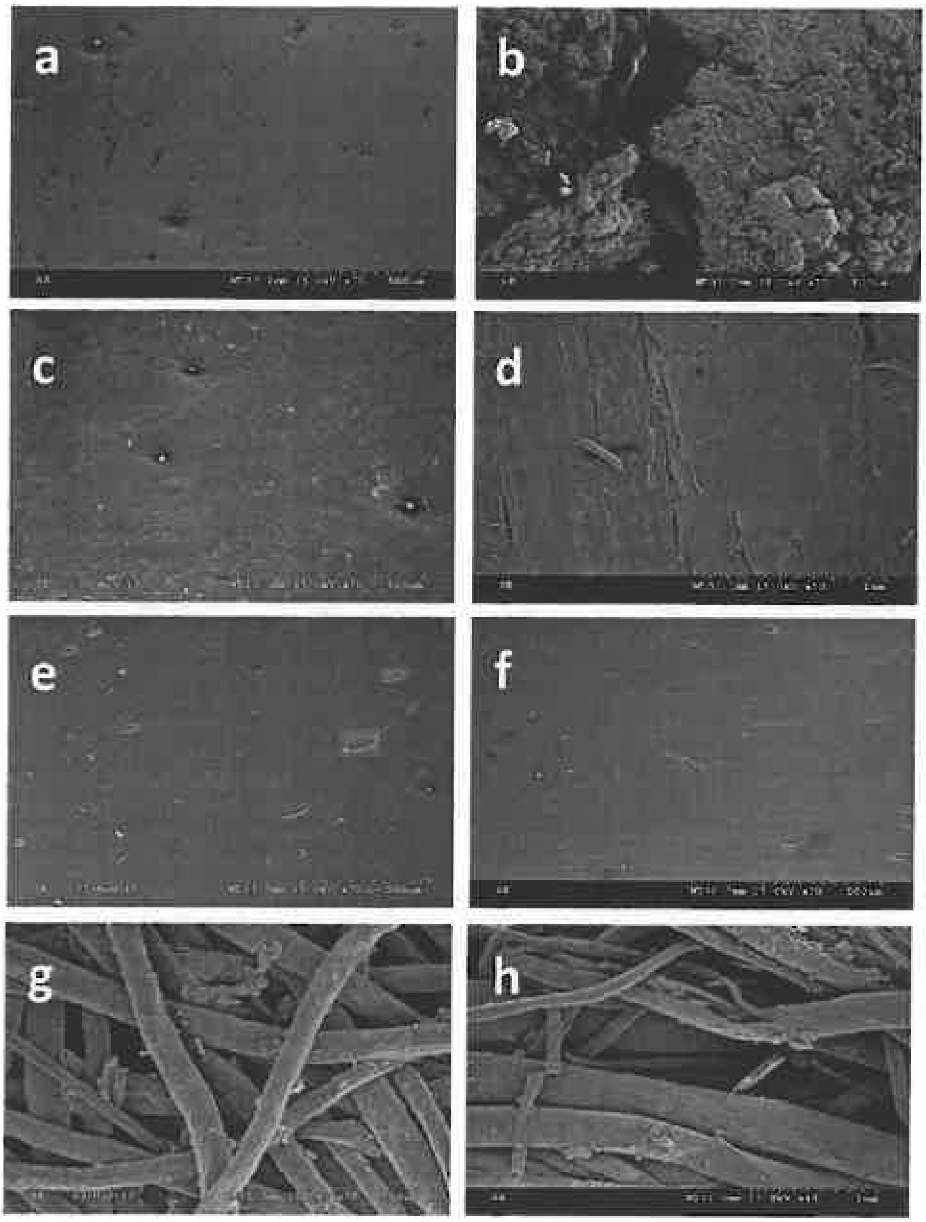

Fig. 2. Scanning electron micrographs of plastics during 2 years of soil incubation. From top to bottom: PHA (a: initial, b: final), co-polyester + corn-based plastic (c: initial, d: final), polypropylene + 2% additive (e: initial, f: final) and coconut coir (g: initial, h: final).

surface of the PHA material (Fig. 2A and B) and some degradation of the co polyester + corn based plastic (Fig. 2C and D).

For conventional plastics and the same plastics amended with additives that were supposed to enhance biodegradability, almost no biodegradation was observed after nearly two years of incubation in soil (Fig. 1). The highest observed conversion during soil incubation was 1.0 ± 0.1% (PP + 2% additive). For all other plastics amended with additives, the final cumulative biodegradation ranged between 0.9 and 1%. These values were less than that measured for the negative control (PP) which reached a final cumulative conversion of 1.3 ± 0.7%. Although they were not significantly different. SEM images did not reveal qualitative changes in the appearance of PP or PP + 2% additive after the 2 year incubation period (Fig. 2E and F).

The mineralization in soil of the natural fiber composite materials was most rapid during the first 65 days of the experiment

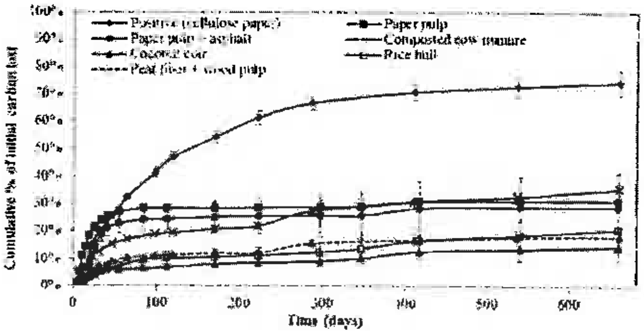

Fig. 3. Cumulative carbon loss (CO_2 C) as percentage of initial carbon (±cumulative standard error) for natural fiber composites during 660 days of soil incubation. For some data points standard error bars are smaller than markers.

(Fig. 3). This was followed by a period of slow mineralization until the termination of the experiment (Fig. 3). After 660 days, the mineralization percent of the composted cow manure, paper pulp and paper pup + asphalt were 35.5 ± 2.3, 31.3 ± 3.6, 29.4 ± 2.1%, respectively. Lower final conversion values were observed for rice hull, peat fiber and coconut coir with values of 21.1 ± 2.6, 18.3 ± 0.7 and 14.4 ± 2.5%, respectively. SEM images of coconut coir revealed some surface changes indicative of biodegradation (Fig. 2G and H).

Approximately 74.2% of cellulose added to soil was converted to CO_2 after 660 days. This is similar to the conversion of cellulose of 80% reported in a 800 day soil incubation conducted to evaluate how carbon substrates affect microbial biomass yield in soil biodegradation tests [33].

The highest biodegradability observed during soil incubation was reported for PHA (70%); a polyhydroxyalkanoate based plastic. This was similar in magnitude to the extent of mineralization of the cellulose positive control (cellulose paper). Bacterial polyhydroxyalkanoates are intracellular aliphatic polyesters of various chain lengths [34]. Several studies have been conducted to study the biodegradability of aliphatic polyesters under different conditions [35–38]. Mineralization of these polymers is mainly achieved by cleavage of the ester bonds which occurs due to both enzymatic and chemical hydrolysis [39].

Statistically analysis revealed that significant differences in the extent of biodegradation ($F_{15,32}$ 822.2, $P < 0.0001$) existed between group means. Tukey–Kramer HSD analysis revealed that among bio based plastics, the difference between PHA and the positive control (cellulose paper) was not significant. Analyses also revealed that differences were not significant between plastics amended with additives that are meant to enhance biodegradability and the negative control (PP). For natural fiber composites all test specimens differed significantly from both the positive and negative controls (Fig. 3).

The results of this study indicate that conventional plastics containing additives do not biodegrade any faster than non additive containing plastics in soil. Manufacturers of these additives claim that if at least 1–5% (by weight) of their additive is added to plastics products, these will fully biodegrade when disposed of in microbe rich environments. These claims are not supported by the findings of this study.

The greatest extent of biodegradation among the fiber composite materials tested was the composted cow manure (35%). This was unexpected since low carbon conversion rates were anticipated for the composted cow manure since it had previously been biologically degraded. After undergoing a composting cycle, much of the carbon contained in the cow manure was expected to be stable and humified [26,40]. However, much less extents of degradation were observed for uncomposted composites produced from rice hulls, from peat fiber pot and coconut coir. For these materials, the extent of degradation in soil ranged from 14 to 21% (Fig. 3). These materials have been used as natural composites due to their low price and structural strength [41,42]. Approximately 46% of coconut coir is lignin [43] as is 21–40% of rice hulls [44] which may have limited their biodegradation.

3.2. Biodegradation during composting

Three different materials were evaluated for their relative rate of degradation during composting. The materials were composted at 55 °C under aerobic conditions for a period of 115 days. The tested materials included plastarch, paper pulp + soy wax and PETE + 1% additive (Tables 2 and 3).

The initial moisture content was adjusted to 60% and the final mean compost moisture content across all treatments was 64.2 ± 3.3% (wet weight basis).

Mineralization under composting conditions occurred at a rapid initial rate for both the positive control and the plastarch material during the first 80 days (Fig. 4). Overall, the positive control (cellulose paper) exhibited 78.4 ± 3.5% conversion during composting.

For paper + soy wax, a majority of the mineralization took place during the first 15 days. For PETE + 1% additive no significant conversion was observed over the entire period of study (Fig. 4). The final cumulative biodegradation during composting for plastarch, paper + soy wax and PETE + 1% additive was 51.3 ± 4.9, 12.4 ± 2.7 and 0.6 ± 3.7%, respectively. The ANOVA indicated that statistically significant differences in the extent of biodegradation ($F_{4,7}$ 496.6, $P < 0.0001$) existed between group means. Tukey–Kramer HSD analysis revealed that all test specimens differed from the positive control. However, PETE + 1% additive did not differ significantly from the negative control.

None of the tested materials mineralized at rates comparable to the positive control material. The highest cumulative biodegradation during composting was observed for the plastarch containing material (51.3%). Starch is made of repeating glucose units linked by glucosidic bonds that are susceptible to enzymatic attack. Uses and applications of starch in its native form or blended with other materials have been discussed [45,46]. Biodegradation of the starch containing portion of the material has been reported [47,48]. However the reason that the plastarch degraded more slowly than cellulose is not known.

After 20 days, only 12% of the paper pulp composite was converted to CO_2 during composting. The low level of cumulative degradation could be related to inhibitory properties of the soy derived wax on the microbial consortia or limiting water accessibility. For plastics containing additives, no degradation was observed. Additives did not improve the biodegradability of PETE during composting.

3.3. Biodegradation during anaerobic digestion

Understanding the biodegradation of different materials in anaerobic conditions such as in industrial sewage sludge AD systems, landfills and anoxic environments is important since under these conditions, microorganisms mineralize organic substrates to both CO_2 and methane. Methane itself can be used as a fuel source but if not captured it has a global warming potential 21 times stronger than CO_2. Since in the U.S. only 30% of the landfills capture methane and among those that do capture, only a small percentage of the methane produced is recovered, then biodegradable plastics in landfills have a greater potential than composted biodegradable plastics to contribute to global warming.

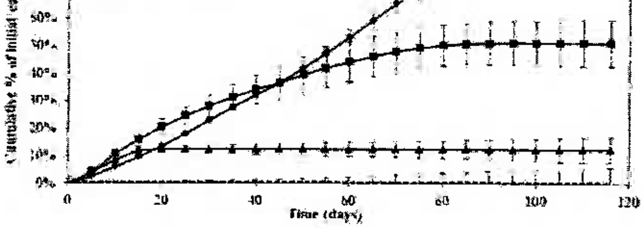

Fig. 4. Cumulative carbon loss (CO_2 C) as percentage of initial carbon (±cumulative standard error) for bio-based plastics, conventional plastics amended with additives and natural fiber composites during 115 days of thermophilic composting. For some data points standard error bars are smaller than markers.

The biodegradability of polymeric materials exposed to an active methanogenic inoculum was studied under controlled laboratory conditions that resemble those found during active AD for a period of 50 days. They likely differ somewhat from the conditions within a landfill where moisture is usually removed and a greater diversity of materials is present. Yet the extent of biodegradation is likely similar to what would ultimately occur over many years in a landfill environment.

Materials tested included plastarch, co polyester + corn based plastics, PP + 2% additive and PETE + 1% additive (Tables 2 and 3). The mean methane content in the biogas across treatments during the entire period of study was 54.1 ± 6.1%.

During the AD incubation, the positive control (cellulose paper) exhibited 74.1 ± 4.8% conversion. For plastarch, the carbon conversion rate to biogas was similar to the positive control (cellulose paper) for the first 7 days (Fig. 5). However, after this period, the rate of conversion slowed as compared to the positive control through day 28. In contrast, no significant mineralization was observed for the plastics containing additive samples over the entire period of the study.

The final cumulative carbon conversion during AD for plastarch and co polyester + corn based plastic were 26.4 ± 3.5 and 20.2 ± 4.4%, respectively. The final conversion values for PP + 2% additive and PETE + 1% additive were 3.1 ± 3.7 and 2.2 ± 1.6%, respectively. The ANOVA indicated that statistically significant differences in the extent of biodegradation ($F_{5,12}$ 50.7, $P < 0.0001$) existed between group means. The Tukey–Kramer HSD analysis revealed that the bio based plastics were significantly different than the positive control but not different from each other. There was no significant difference in the carbon conversion of the negative control (PP) and the plastic containing the additive.

The biodegradability of different bio based materials including cellulose and starch [49,50] has been investigated previously under anaerobic conditions [51,52]. Yagi et al. [53] studied the biodegradability of cellulose powder under mesophilic (35 °C) and thermophilic (55 °C) AD conditions. Cellulose powder reached a cumulative conversion of 80% under both temperature conditions. Other authors have also studied the anaerobic mineralization of aliphatic polyesters. Abou Zeid et al. [54] conducted a study to determine the biodegradability of the natural polyesters poly(b hydroxybutyrate) (PHB), poly(b hydroxybutyrate co 11.6% b hydroxyvalerate) (PHBV) and the synthetic polyester poly(o caprolactone) (PCL) using different anaerobic sludges and individual strains. Biodegradability of the powdered materials was measured as the percent of weight loss. They found that almost all the PHB was converted in 9 days, but only 60 and 30% weight loss was observed for the PHBV and PCL, respectively. Similar results were reported by Shin et al. [55] in which nearly complete conversion was observed for the natural bacterial polyester but no biodegradability for synthetic analogs was observed under simulated landfill conditions.

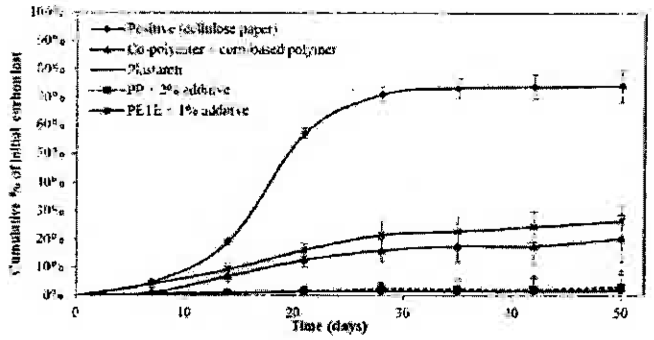

Fig. 5. Cumulative carbon loss (CO_2 C) as percentage of initial carbon (±cumulative standard error) for bio-based plastics, conventional plastics amended with additives and natural fiber composites during 50 days of anaerobic digestion. For some data points standard error bars are smaller than markers.

The results of this study indicate that materials have different rates of mineralization under different end of life scenarios. For example, the positive control reached 70% conversion in 25 days during AD while 75 and 400 days were needed to reach the same extent of conversion under composting and soil incubation conditions, respectively. The plastarch material degraded faster under composting conditions reaching 50% conversion in 85 days than under AD and soil incubation conditions where only 26 and 30% was converted after 50 and 660 days, respectively. For co polyester + corn based plastic 20% of the material was converted during 20 days of soil incubation while 50 days were needed to reach the same value during AD. Ultimately, co polyester + corn based plastic reached 55% conversion after 660 days of soil incubation. Conventional plastics and those containing additives did not degrade at all under any of the three conditions.

Biodegradable plastics are potential alternatives to petroleum based materials that can be incorporated into organic recycling schemes based on anaerobic digestion or composting. They also could potentially reduce the pollution associated with conventional plastics and therefore lead to the development of products that are more environmentally friendly. Ideally, biodegradable materials must be useful for a predetermined service life and then biodegrade in a short period of time, leaving no visible fragments and no toxic residues when composted or anaerobically digested. Disposal of these materials in landfills as opposed to anaerobic digestions is not recommended since under anaerobic conditions they biodegrade to form methane and most landfills capture only a small fraction of the methane created [56].

4. Conclusion

In this study, the relative biodegradability of a range of polymeric materials and natural fiber composites used for various commercial applications was investigated under composting, soil incubation and anaerobic digestion conditions. The validity of the tests was confirmed in that positive controls (cellulose paper) biodegraded by more than 70% in all three systems in a reproducible manner.

While some of the bio based plastics and natural fibers biodegraded to an appreciable extent, plastics containing additives that supposedly confer biodegradability to polymers such as polyethylene and polypropylene did not improve the biodegradability of these recalcitrant polymers. SEM analysis confirmed that substantial biodegradation of polyhydroxyalkanoate based plastics occurred and that some surface changes occurred in co polyester + corn based plastic and coconut coir materials. However, SEM confirmed that no degradation of polypropylene and polyethylene occurred, even after amendment with additives meant to confer biodegradability.

The relative biodegradability of the materials during long term soil incubation was PHA > co polyester + corn based plastic > composted cow manure > plastarch > paper pulps > natural fibers > conventional plastics containing additives to enhance biodegradability conventional plastics. For anaerobic digestion and composting the relative biodegradability was plastarch > co polyester + corn based plastic > conventional plastics with additives and plastarch > paper pulp + soy wax > conventional plastic with additives, respectively.

Over the time scale of organic recycling processes (composting and anaerobic digestion) most of the bioplastics biodegraded to only a limited extent. Furthermore, under anaerobic incubation, some of the bio-based plastics biodegraded to generate methane, a potent greenhouse gas that unless captured may negate the perceived environmental benefits of using these materials. Biodegradable plastics made from petroleum (Class II), may have more adverse environmental impacts than conventional plastics (Class I) if their ultimate fate is landfilling and anaerobic conversion to methane.

Acknowledgments

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Respondent’s Exhibit BB

Peter Arhangelsky

From: Johnson, Katherine <kjohnson3@ftc.gov>
Sent: Friday, April 11, 2014 10:48 AM
To: Peter Arhangelsky
Cc: Cohen, Jonathan; Jillson, Elisa; Lou Caputo; Jonathan Emord
Subject: RE: Docket 9358 - 041014 Second Revised Scheduling Order

Peter, we don't have any objection to moving the pre-trial conference to another date, but it doesn't seem pragmatic to move it to the day before the evidentiary hearing (and I'm not sure the ALJ would approve it). Is there really no other date, e.g., 1st or the 30th? We would agree that both parties could file final stipulations on the 29th, if the 30th would work for the pre-trial.

Katherine E. Johnson, Attorney
Division of Enforcement
Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Avenue, NW
Mail stop M-8102B
Washington, DC 20580
Direct Dial: (202) 326-2185
Fax: (202) 326-2558
Email: kjohnson3@ftc.gov

From: Peter Arhangelsky [mailto:PArhangelsky@emord.com]
Sent: Friday, April 11, 2014 1:32 PM
To: Johnson, Katherine
Cc: Cohen, Jonathan; Jillson, Elisa; Lou Caputo; Jonathan Emord
Subject: FW: Docket 9358 - 041014 Second Revised Scheduling Order

Katherine,

Under the Second Revised Scheduling Order issued yesterday, we have a calendaring conflict for the July 31, 2014 Pretrial Conference date. We are scheduled to appear in Salt Lake City for a hearing in the U.S. District Court for the District of Utah on that same day. Given our travel schedules, the next date we are available is Monday, August 4th. Please let us know if you agree to moving the prehearing conference from July 31 to August 4. If you are in agreement, we would prepare a joint motion to continue the pretrial conference date to August 4. I am available this afternoon to speak about this if needed.

Thanks,

Peter

Peter A. Arhangelsky, Esq. | **EMORD & ASSOCIATES, P.C.** | 3210 S. Gilbert Rd., Ste 4 | Chandler, AZ 85286
Firm: (602) 388-8899 | Direct: (602) 334-4416 | Facsimile: (602) 393-4361 | www.emord.com

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From: Gross, Dana [mailto:DGROSS@ftc.gov]
Sent: Thursday, April 10, 2014 1:13 PM
To: Peter Arhangelsky; Jonathan Emord; Jillson, Elisa; Johnson, Katherine
Cc: Arthaud, Victoria; Gebler, Hillary; Pelzer, Lynnette; Clark, Donald S.; Tabor, April; Frankle, Janice Podoll
Subject: Docket 9358 - 041014 Second Revised Scheduling Order

Dear Counsel,

Attached is a courtesy copy of the above order issued today by Judge Chappell.

Sincerely,

Dana L. Gross
Legal Support Specialist
Office of Administrative Law Judges
Direct:(202) 326-3723
Main: (202) 326-3637

Respondent’s Exhibit CC

SUMMARY OF REBUTTAL REPORT OF STEPHEN P. MCCARTHY, PhD

Complaint Counsel has asked me to review and respond to the expert reports of Dr. Sanjit Sahu and Dr. Ryan Burnette submitted on behalf of ECM Biofilms, Inc. ("ECM"). This rebuttal report responds to the critiques and analyses raised in those reports and can be summarized as follows:

- Dr. Sahu and Dr. Burnette incorrectly frame the question as whether there is any evidence that plastics biodegrade leading to an irrelevant evaluation of the scientific literature regarding known biodegradable polymers. ECM does not claim (as far as I know) to enhance the biodegradability of plastics that are already biodegradable. Rather, ECM asserts that it can turn "non-degradable" plastics into biodegradable ones.
- Dr. Sahu and Dr. Burnette both agree that the ASTM D5511 test is not a perfect test for evaluating ECM's claims, and that no single test is perfect. However, they offer very little to support whether the ECM Materials or the Litigation Materials support ECM's claims, nor do they address the issue of recalcitrance. Instead they both appear to rely on faulty and incomplete understanding of the mechanisms at issue. While I do not opine that any test must be perfect to answer the questions raised here, the tests performed must be of sufficient quality and quantity to demonstrate that a non-degradable plastic will completely biodegrade. The Testing Materials I have reviewed simply do not pass muster.

DATED: 6/30/14

Dr. Stephen P. McCarthy

I. THE CONCLUSIONS REACHED BY ECM'S EXPERTS ARE NOT SUPPORTED BY THEIR UNDERLYING ANALYSIS.

1. The question that complaint counsel asked me to evaluate is whether ECM's claims were scientifically true or at least had an appropriate level of scientific support. The claims complaint counsel asked me to evaluate specifically are:

   a. Plastic products manufactured with the ECM additive ("ECM Plastics") will completely biodegrade, *i.e.*, will completely break down and decompose into elements found in nature, within a reasonably short period of time (*i.e.*, one year) in a landfill.
   b. ECM Plastics are completely biodegradable in nearly all landfills.
   c. ECM Plastics will completely biodegrade in nearly all landfills between 9 months to 5 years.
   d. ECM Plastic will completely break down and decompose into elements found in nature in any disposal environment at some rate and extent that exceeds plastic without the ECM Additive.
   e. ECM Plastics have been shown to completely biodegrade in most landfills in one year, nine months to five years, or faster than conventional plastics under various scientific tests, including ASTM D5511.

2. In my report, I defined conventional plastics as high molecular weight, commercial-grade plastics representing more than 90% of the market. *See* Expert Report of Stephen P. McCarthy ("McCarthy Expert Report") ¶¶ 29-31. Thus, I based my evaluation of and conclusions regarding ECM's claims on these conventional plastics. I conclude, based on my professional judgment and after evaluating the Testing Materials, that many of ECM's claims are untrue and none have the appropriate scientific support.

3. Before I begin my critique of the evaluations of Drs. Sahu and Burnette, it is important to note that there are a wide variety of definitions for the term "biodegradable" and "biodegradability." As aptly stated by one source cited by Dr. Sahu, "Because of the slightly different definitions or interpretations of the term biodegradability, the different approaches [to analyses] are not equivalent in terms of information they provide or the practical significance . . . . Before choosing an assay to simulate environmental effects in an accelerated manner, it is

critical to consider the closeness of fit that the assay will provide between substrate, microorganisms, or enzymes and the application or environment in which biodegradation should take place." Drs. Sahu and Burnette limit their definitions of biodegradation to the mechanism of break down by biological organisms. While I agree that is one possible definition, this differs from what complaint counsel asked me to assume. The primary point of departure is whether the definition includes the concepts of complete biodegradation or complete biodegradation in a specific timeframe. While I agree that "biodegradable" is not always used to describe complete mineralization in a specific timeframe, I evaluated the evidence in terms of whether it satisfies that definition of biodegradation provided to me, which does include those concepts.

4. Dr. Sahu concludes that ECM Plastics will unavoidably biodegrade faster than untreated conventional plastics.[1] He reaches this conclusion starting with a faulty "threshold question": whether plastics with additives will biodegrade at all. *See* Expert Report of Dr. Ranjit (Ron) Sahu ("Sahu Report") at 24. Likewise, Dr. Burnette concludes that many microorganisms have enzymes capable of degrading plastics. Expert Report of Dr. Ryan N. Burnette ("Burnette Report") ¶55 and ¶83(g). I strongly disagree with these conclusions and how they were reached.

5. I have been involved in plastics and polymers since 1976. I currently teach in Plastics Engineering and have done so for 30 years. Although suggested by ECM's experts that my report is "sourced with very few citations," I included my Vitae as support for my conclusions. My Vitae includes hundreds of publications, doctoral dissertations and masters theses on biodegradable polymers and biodegradation of polymers. In addition, I am the Editor of Journal of Polymers and the Environment, a peer-reviewed journal dominated by peer reviewed

---

[1] Dr. Sahu does not define "conventional plastics" but seems to ignore the difference between high-molecular weight plastics and low-molecular weight plastics that have the same name.

publications of biodegradable polymers and polymer biodegradation. During my 20 years as editor of this journal, I have seen no publications and no evidence that any conventional plastic (*i.e.*, a high-molecular weight synthetic polyolefin such as PE or PVC) will completely biodegrade in the times claimed by ECM, the conclusion that ECM Plastic will biodegrade faster than untreated alternatives, or for the mechanism of action proposed by ECM.

6. Although I stated that I cannot conclude with scientific certainty that conventional plastics treated with the ECM Additive would biodegrade faster than untreated plastics, it is my professional judgment and expert opinion that these claims are false. Nothing presented in any of the expert reports presents a compelling argument otherwise.

A. The Experts Rely On Irrelevant Authorities.

7. Dr. Sahu begins his analysis with an unnecessary personal attack before launching into a largely irrelevant discussion of several inapposite papers.[2] The threshold question is not whether there is evidence that plastics (with or without additives) biodegrade. My report discusses several subsets of plastics that can undergo biological, enzymatic degradation. McCarthy Report ¶¶32-35. In order to evaluate the claims identified by complaint counsel, however, the appropriate threshold question is as follows:

- Given what we (the scientific community) know about the non-biodegradability of commercial-grade conventional plastics (as I have defined them in my report), has ECM provided sufficient quantity and quality of scientific evidence to demonstrate that these plastics treated

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[2] It appears that most of Dr. Sahu's knowledge of plastics and polymers came from merely reading and quoting *Lampman, S., Characterization and Failure Analysis of Plastics, ASM Incorporated, 2003* and other cited sources. For instance, Dr. Sahu uses the term "mer." Most professionals teaching plastics and polymers would never use the word "mer." The correct term is "polymer repeating unit."

with its additive will completely biodegrade in the stated timeframes and disposal environments.

The answer to this question is no.

8. Dr. Sahu's supporting evidence is irrelevant to ECM's technology for two reasons: (1) the authorities cited (except one)[3] do not evaluate non-degradable plastics, the subject of ECM's claims; and (2) they do not support ECM's claimed mechanism of action.

1. ECM Additive is intended to enhance the biodegradability of non-degradable plastic.

9. The ECM Additive is intended to enhance the biodegradability of non-degradable plastics. For example, in the exhibits attached to the ECM complaint, ECM distinguishes the ECM additive from other "alternative products" on the market such as bioplastics (PLA, Mater-Bi®, PHB and combinations) and oxo-biodegradable plastics. Thus, ECM has distinguished its technology as NOT belonging to this subset of plastics, making many of the authorities referenced by Dr. Sahu irrelevant.[4]

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[3] Both experts cite to the 1978 study by Albertsson establishing that untreated, virgin polyethylene could biodegrade under certain environmental conditions. They omit three significant points in their discussion. First, the test condition was a super-oxygenated environment, not applicable to any landfill. Second, the rate of degradation was extremely slow (0.36 ± 0.03% over two years for PE film) and attributed by the author to biodegradation of the *low-molecular weight* portion of the polyethylene. See Albertsson at 3432 ("It is assumed that the source of the ^{14}C metabolization in these degradational experiments must have been mainly low molecular weight polyethylene . . . ."). And third, Albertsson utilized radiotagged ^{14}C to evaluate the biodegradation, thus establishing that radiolabeling is the appropriate method for evaluating slowly degrading materials.

[4] If Dr. Sahu's analysis is relevant at all, it is with respect to the extremely limited subset of high-molecular weight, commercial-grade, synthetic plastics that are similar enough to naturally occurring polymers to be susceptible to enzymatic attack. In this group are certain aliphatic polyesters, aliphatic-aromatic polyesters, certain polyurethanes, polyvinyl alcohol, etc. These high-molecular weight plastics mimic naturally-occurring polymers and are susceptible to hydrolysis. Because they are recognized as biodegradable, it is also likely that these are not the type of plastics to which ECM is referring (because, again, ECM states it can

10. Dr. Sahu cites to several articles that discuss biodegradability of PCL and other synthetic biodegradable plastics. See, e.g., L. Tilstra and D. Johnsonbaugh, "The biodegradation of blends of polycaprolactone and polyethylene exposed to a defined consortium of fungi," Journal of Environmental Polymer Degradation, vol. 1, pp. 257-267, 1993; Shah, A.A., et. al., Biological degradation of plastics: A comprehensive review, Biotechnology Advances Vol. 26, 2008, p. 246–265. As discussed in my prior report, high-molecular weight synthetic polymers such as PCL, PLA, and poly butylene succinate, contain similar molecular structure to naturally-occurring polymers, such that microorganisms have developed enzymes over time that will biodegrade these. McCarthy Report ¶ 33-35. The biodegradability of PCL was not discussed in my report at length, other than to note that if it is the main proprietary "ingredient" of the ECM Additive, it is considered biodegradable. This, in turn, affects how one designs and conducts a biodegradation study evaluating a blend containing PCL and a non-degradable plastic.

11. Dr. Sahu also referenced articles and reports addressing the biodegradability of oxidized and low-molecular weight polyethylenes in support of his conclusion that conventional plastics (undefined by him) are subject to biodegradation. See Sahu Expert Report at ¶ VI.3. These are irrelevant for at least two reasons: (1) such technology requires oxidation through additives (such as pro-oxidants) and/or pretreatments, which does not occur in landfills (no or low-oxygen environments); and (2) any polyethylene that had undergone such oxidation prior the addition of the ECM Additive would have no commercial application.

12. As mentioned in my report, if the polymer chain is short enough, it can be attacked by microorganisms. McCarthy Report ¶86. For instance, the Tokiwa article discusses biodegradation of low-molecular weight PE. Tokiwa Y., et al, Biodegradability of Plastics, Int.

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turn "non-degradable" plastic into biodegradable plastic) and they only represent less than 5% of the commercial plastic market.

J. Mol. Sci. Vol. 10, 3722-3742, at 3735 ("PE cannot be easily degraded with microorganisms, However, it was reported that lower molecular weight PE oligomer (MW= 600-800) was partially degraded . . . while high molecular weight PE could not be degraded."). The only way to reduce PE to this molecular weight is through a pro-oxidant (not ECM technology) or exposure to other oxidative degradation environment. See for example *Tokiwa*, discussing blends of PE with starch, which enhances the auto-oxidation, but concludes "despite all these attempts to enhance the biodegradation of PE blends, the biodegradability with microorganisms on the PE part of the blends is still very low"; *Shah*, discussing attempts to make traditional polyolefins that are "non-susceptible to degradation by microorganisms" into ones that are biodegradable and concluding that polyethylene can only be degraded following "photodegradation and/or chemical degradation." While this technology does result in an increase in aerobic biodegradation, the lack of oxygen in landfills would be problematic for such a mechanism.

13. ECM's technology likely does not pertain to low-molecular weight PE for another reason: such low-molecular weight plastics would serve few commercial applications. Plastics at this low molecular weight would have the consistency of wax. It is extremely unlikely that ECM is confining its technology to these low-molecular weight plastics— these are not the plastics possessing the mechanical characteristics that ECM's customers would be manufacturing.

14. Therefore, whether this small subset of plastics are susceptible to biodegradation with or without the ECM additive is not the starting point of the analysis.

2. The Papers Cited Do Not Support the Mechanism of Action.

15. ECM and its experts assert that there is a mechanism of action initiated by the attractiveness of the ECM Additive as a food source that results in the depolymerization of otherwise non-degradable plastics. For instance, Dr. Sahu cites to papers discussing blends of biodegradable and non-biodegradable polymers. [CITE] But simple blending of two immiscible polymers (such as PE and PCL) will not increase the biodegradability of the non-degradable one. See generally, *Tilstra* and *Tokiwa*.

16. If we assume that the ECM Additive is a blend of approximately 20% polyethylene vinyl acetate (EVA), approximately 60% PCL, 6% linear low density polyethylene (LLDPE), approximately 7% calcium stearate, and approximately 6% starch as discussed in my expert report (see McCarthy Expert Report at n.17), then the enzymes produced from the microorganisms which are attracted to the PCL food source would be hydrolytic enzymes, such as lipases. The conventional non-degradable plastics discussed in my two reports (PE, PP, PVC, and PS) cannot be hydrolyzed because they do not have an ester bond that can be cleaved by hydrolysis. Because the remaining plastic would remain unaffected by the hydrolytic enzymes, there is no mechanism of action that would result. The statement that addition of the ECM Additive causes the C-C bonds to "weaken" has no support in the literature (nor did they cite to any).

B. A Series of Faulty Tests Cannot Support ECM's Claims.

17. I started this rebuttal report by counter-proposing the following question to Dr. Sahu's "threshold question": given what we know about the non-biodegradability of commercial-grade conventional plastics, has ECM provided sufficient quantity and quality of scientific evidence to

demonstrate that these plastics treated with its additive will completely biodegrade in the stated timeframes and disposal environments.

18. The conclusions that the Testing Materials in the aggregate demonstrate biodegradation and that, once it begins, it will go to conclusion, are flawed for three reasons: (1) a series of faulty tests, conducted with improper controls, over insufficient duration, and under inapplicable conditions do not answer the threshold question as I presented it; (2) the experts do not address any authority supporting the conclusion that a polymer's recalcitrance can be ignored; and (3) the tests conducted under the Barber Method are methodologically and scientifically unsound.

1. ASTM D5511 Does Not Support ECM's Specific Marketing Claims.

19. Although Dr. Burnette and Dr. Sahu seem to be at odds with whether ASTM D5511 is appropriate, both agree that no one test could simulate every environmental condition and that the tests performed are not without flaws.

20. I agree with Dr. Sahu and Dr. Burnette in two respects. No test is perfect and test representativeness involves scientific judgment. While no test is perfect, tests must be well-designed, well-executed, and well-controlled, and suited to address the relevant question.

21. As explained in my report, ASTM D5511 cannot be used to support the types of claims ECM is making. Specifically, it is not intended to and (without modification under professional judgment and guidance) cannot address whether a blend like an ECM Plastic will completely biodegrade in a stated timeframe under landfill conditions.

2. ECM's Experts Present No Authorities that Support Extrapolation.

22. One of the most serious flaws in the conclusions of the experts is that once biodegradation is established, it will continue to completion. In 1998, my colleagues and I

discovered through research on biodegradable polymers that the amorphous regions of polymers biodegrade at a greater rate than the crystalline regions.[5] M. Parikh, R. Gross, and S. McCarthy, The Influence of Injection Molding Conditions on Biodegradable Polymers, Journal Of Injection Molding Technology, Vol. 2, No. 1, pp. 30-36, March (1998). This phenomenon is critical as to why extrapolation of biodegradation rates is scientifically unsound.

23. The first material to biodegrade is the amorphous region of a polymer, which biodegrades at a fast rate. If the material were 50% crystalline, then the biodegradation rate would be very rapid until it reached 50% biodegradation. Once the amorphous region was degraded, the crystalline region would be left. The biodegradation rate would severely decrease (or even stop altogether) since the crystalline region is very difficult to biodegrade, as even Dr. Sahu concedes. Sahu Report at 30. Therefore, during a biodegradation test, if the biodegradation rate calculated from the first 50% were to be extrapolated to 100%, an unrealistic (or impossible) rate of complete biodegradation would be the result.

3. The Barber Method Is Scientifically Unsound.

24. The experts rely on Dr. Timothy Barber's methodology in support of their conclusions regarding the environmental fate of ECM Plastics. The experts argue that the Barber Methodology is defensible because weight loss and the evaluation of free chlorine are appropriate indicators of biodegradability. Reliance on these metrics to establish complete biodegradability is improper for two reasons. The measurement of weight loss and loss of

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[5] It is easy to understand the difference between amorphous and crystalline when considering water and ice. The liquid phase of water is amorphous. The solid phase of ice is crystalline. The crystallinity of any given polymer is a result of its chemical structure and processing history. High density polyethylene is highly crystalline, while low density polyethylene is less crystalline due to branching.

mechanical properties as a measurement of biodegradation rate is not accurate. And the measurement of free chloride does not indicate biodegradation.

25. In 1992, there was the belief that weight loss was a suitable measurement to demonstrate biodegradation. In fact, until 2004 ASTM Standard Test Method D5247 utilized this methodology. Accordingly, it is not unusual that some of the early literature and journals discussing biodegradability of plastics rely on this measurement. I myself have publications from this period that also utilize weight loss as a prediction of biodegradability. While weight loss and loss of mechanical properties are easy to measure, they are inaccurate—the sample may fragment, making it difficult to retrieve all of the sample for weighing; it is usually difficult to clean the sample effectively to ensure that weight loss is accurately measured. ASTM Standard Test Method D 5247 was withdrawn in 2004 for these and similar reasons.

26. This is consistent with many of the sources cited by Dr. Sahu, which indicate that weight loss is by itself not an accurate predictor of biodegradability on its own. For instance, one of the papers cited by Dr. Sahu discusses the various methods available to monitor the degradation process of environmentally degradable polymers. Notably, in the discussion over laboratory-scale simulated accelerated environments, the author discusses the use of changes in mechanical and chemical properties as a basis for determining biodegradation. The author recognizes that one of the "inherent drawbacks in the use of mechanical properties, weight loss, molecular weights, or any other property which relies on the macromolecular nature of the substrate is that in spite of their sensitivity, these can only address the early stages of the biodegradation process. Furthermore, these parameters can give no information on the extent of mineralization. Especially in materials blends or copolymers, the hydrolysis of one component can cause significant disintegration (and thus loss of weight and tensile properties), whereas other

components may persist in the environment, even in disintegrated form. Blends of starch, poly (з - hydroxybutyrate) or poly (ε- caprolactone) with polyolefins are examples of such systems." Van der Zee, M., Analytical Methods for Monitoring Biodegradation Processes of Environmentally Degradable Polymers, Section 11.5.6.3, Chapter 11 in *Handbook of Biodegradable Polymers: Synthesis, Characterization and Applications*, First Edition, edited by Andreas Lendlein, Adam Sisson.

27. Dr. Burnette claims that the existence of "free chloride ion" is evidence of "breakage" of the "PVC molecule." In fact, the chlorine atoms of the PVC are located as a side atom on the polymer chain as shown in Figure 7 of Dr. Burnette's report. The loss of the chlorine group from the PVC chain will not result in breakage of the C-C bonds that form the polymer chain. Thus, the existence of the free chlorine does not indicate anything about the most difficult, *i.e.*, strongest and most recalcitrant, bonds that need to be broken to truly result in biodegradation. Therefore, the tests conducted by Dr. Barber are invalid and ECM's expert's reliance on those reports is unfounded. [6]

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[6] Notably, none of the experts identify any peer-reviewed literature discussing whether the measurement of free chloride is a basis for determining biodegradability.

Respondent's Exhibit DD

Peter Arhangelsky

From: Peter Arhangelsky
Sent: Wednesday, July 09, 2014 11:53 AM
To: 'Johnson, Katherine'
Cc: Cohen, Jonathan; Jillson, Elisa; Decastro, Arturo; Jonathan Emord; Eric Awerbuch
Subject: RE: Meet & Confer; Motion for Leave to Exceed Rule 3.22 Word Count

Katherine:

Thank you for your response. I explained last Wednesday that the failure to disclose Dr. Michel as an expert in any of your witness lists was in violation of Rule 3.31A and the Court's Scheduling Order. Rule 3.38 provides the mechanism for relief for failing to meet discovery obligations, and sanctions are therefore part and parcel to our motion to exclude. Moreover, the failure to disclose Dr. Michel seems to have been an attempt to secure a competitive advantage in litigation. Because Rule 3.31A requires that you provide advance notice for experts in the original expert list (including rebuttal experts who you later designate), and you have no reasonable justification for this unfair surprise, sanctions are in fact appropriate. We therefore disagree that I failed to raise this point in our meeting; I think the issues were clearly drawn. You even responded to my concerns that ECM had been deceived.

We also note that you secured Dr. Michel's "stipulation" based on false pretenses, to wit, the objective representation that testimony was unnecessary if you could rely on his stipulation alone to introduce documents. We recognize that it was your intent (not ECM's) to depose Dr. Michel. But you held him out as a fact witness, and the facts surrounding his proposed deposition strongly suggested that he either would not testify in this case, or would be doing so as a fact witness. ECM lost significant discovery opportunities and suffered prejudice from complaint counsel's failure to abide by the scheduling order.

While we appreciate your offer to depose Dr. Michel, which would certainly be necessary, that remedy alone will not suffice given the circumstances. However, given your representations below, we would expect that ECM be afforded an opportunity to present its surrebuttal testimony if that is the direction the ALJ chooses to take.

Best,

Peter A. Arhangelsky, Esq. | **Emord & Associates, P.C.** | 3210 S. Gilbert Rd., Ste 4 | Chandler, AZ 85286
Firm: (602) 388-8899 | Direct: (602) 334-4416 | Facsimile: (602) 393-4361 | www.emord.com

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From: Johnson, Katherine [mailto:kjohnson3@ftc.gov]
Sent: Wednesday, July 09, 2014 12:14 AM
To: Peter Arhangelsky; Jonathan Emord; Eric Awerbuch
Cc: Cohen, Jonathan; Jillson, Elisa; Decastro, Arturo
Subject: RE: Meet & Confer; Motion for Leave to Exceed Rule 3.22 Word Count

Peter:

ECM already stipulated to the admissibility of the Ohio State Study, so it seems a bit disingenuous to attempt a second (or third or fourth, as the case may be) bite at this apple. In fact, the whole purpose of the stipulation, as I understood it, was for Complaint Counsel to avoid having to take Dr. Michel's deposition *solely* to establish the admissibility and authenticity of the study during fact discovery. The parties specifically reserved rights to introduce additional evidence regarding the weight and probative value of the study.

Obviously, we disagree that there is any basis to move to exclude Dr. Michel's rebuttal report or him as an expert witness generally. Or that identifying him as a rebuttal expert warrants sanctions. In fact, because you failed to raise this in the "meet and confer", I am at a loss to see what basis ECM could possibly have to support sanctions.

As for the word count, we will not consent given that (1) there is no basis to exclude the study given the stipulation; (2) there is no basis to exclude Dr. Michel as a rebuttal expert; (3) and there is no basis for sanctions. To the extent that ECM seeks leave of the court to name a surrebuttal expert, we would consent to that relief, provided that, we have the opportunity to depose the expert regarding the scope of the surrebuttal.

Best,
Katherine

Katherine E. Johnson, Attorney
Division of Enforcement
Bureau of Consumer Protection
Federal Trade Commission
600 Pennsylvania Avenue, NW
Mail stop M-8102B
Washington, DC 20580
Direct Dial: (202) 326-2185
Fax: (202) 326-2558
Email: kjohnson3@ftc.gov

From: Peter Arhangelsky [mailto:PArhangelsky@emord.com]
Sent: Tuesday, July 08, 2014 5:58 PM
To: Johnson, Katherine
Cc: Cohen, Jonathan; Jillson, Elisa; Jonathan Emord; Eric Awerbuch
Subject: Meet & Confer; Motion for Leave to Exceed Rule 3.22 Word Count

Counsel:

As we discussed last week, ECM intends to file a combined motion to exclude Dr. Michel, his report, and his study. That motion stems from the failure to disclose Dr. Michel as a witness. Last week we met and conferred on the relief we intend to request in those motions. However, ECM now requests a conference concerning its anticipated motion for leave to exceed the 2,500 word count in Rule 3.22(c). We intend to request an additional 2,000 words in support of a combined motion: (1) to exclude Dr. Michel and his report; (2) for sanctions; and (3) for leave to add a surrebuttal expert. Although ECM could use 2,500 words for each motion individually, the overlapping facts and procedure make a combined motion most appropriate.

ECM would of course agree to a reciprocal extension for your opposition. Please let us know if you consent to the extension or if you would oppose it by noon Eastern tomorrow. I am available to discuss if you have any questions.

Best,

Peter A. Arhangelsky, Esq. | **EMORD & ASSOCIATES, P.C.** | 3210 S. Gilbert Rd., Ste 4 | Chandler, AZ 85286
Firm: (602) 388-8899 | Direct: (602) 334-4416 | Facsimile: (602) 393-4361 | www.emord.com

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