REPORT

Preparation and composting of PHA-based film and tray prototypes

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31st of October, 2025

Verschuren Centre Inc.

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1.0 EXECUTIVE SUMMARY

Lab scale and pre-commercial film and tray prototypes were produced from commercially available, certified compostable PHA-based biopolymer resins. Prototypes were generated using hot-melt extrusion processing with the Verschuren Centre benchtop extruder located in Sydney, NS and a pilot-sized film extrusion line at Copol International Ltd. located in North Sydney, NS using established extrusion temperature profiles and protocols. The extruded film and sheet prototypes were tested as extruded, as well as secondary processing (thermoforming and/or heat-sealing) to generate articles comparable to pre-commercialization prototypes. The bench scale pre-commercialization prototypes represented 1:5 scaled versions of the full-sized pilot prototypes.

Lab scale composting trials were conducted using composted horse manure acquired from a local farmer and utilized film samples, sheet stock samples, scaled down pre-commercial prototypes as well as one of the smaller pilot-line prototypes. Samples were placed in the compost and keep moist at elevated temperatures and high relative humidity using an environmental chamber over a period of 8 weeks to simulate an active compost pile. These trials included samples produced from virgin resins and resins that had been modified at the Verschuren Centre with additives to alter the properties or function (barrier, composting speed, or antimicrobial properties) of the film or sheet. These lab trials successfully degraded many of the samples employed, and highlighted differences in the degradation rates and patterns for the individual films, sheets, and configuration of tray prototypes. The lab trials also demonstrated that certain additive modifications had a positive impact on the speed and degree of degradation.

Municipal composting trials were conducted at the Cape Breton Regional Municipality (CBRM) Solid Waste Management Facility and exclusively used films or sheets that were generated using the pilot film extrusion line. All of the film and thermoforming sheet stock underwent secondary processing (thermoforming and/or heat-sealing) to generate pre-commercial prototypes. Two trials were conducted at the CBRM Solid Waste Management Facility, each for a period of 7 weeks. Compared to the lab trials, the degree of degradation was much higher in the municipal setting for both trials, and degradation patterns mimicked those observed in the lab trials. The success of the municipal trials serves as a proof of concept and an avenue for further study to facilitate the acceptance of compostable PHA-based biopolymer resins in municipal composting waste management streams.

2.0 INSTRUMENTATION

Extrusions at bench scale were carried out using a Thermofisher Process 11 twin-screw extruder with a standard screw configuration comprised of an initial melting zone followed by 2 kneading/mixing zones. All resins and additives were dried prior to extrusion, either in a convection or vacuum oven at 55°C for a minimum of 24 h. Masterbatch preparation was carried out with a 3 mm filament nozzle and were immediately cooled in a water batch prior to pelletization with a Bay Plastics BT25 pelletizer. For film and sheet extrusions, the filament die was replaced with a 100 mm film/sheet die with a die gap of 250 µm for film extrusions and 800 µm for sheets. Extruded films and sheets were drawn down and collected by a DPM Solutions Inc. take-off machine.

Pilot scale film and sheet extrusions were carried out at Copol International Ltd. on a LabTech Engineering Company 3-layer co-ex cast film line equipped with a 700 mm film die and a die gap of approximately 250 µm. For modified film formulations, the required masterbatch was prepared using the benchtop extruder, and the masterbatch was subsequently extruded on the pilot film line.

Tray sheet stock prepared on the benchtop extruder and pilot line were thermoformed using a Mayku Multiplier pressure thermoformer. For benchtop prototypes, a reduction plate was used to accommodate the smaller dimensions of sheets produced by the benchtop extruder.

Lab composting trials utilized a Ensec Environmental Chamber. The chamber was maintained at 40°C and 90% RH for the duration of each testing period.

Thermal properties were measured using a Perkin Elmer STA-8000 simultaneous thermal analyzer. Differential scanning calorimetry (DSC) measurements were conducted under N_2 atmosphere following a heat-cool-heat cycle between 35-220°C (3 min hold at 220°C) with a ramp rate of 10°C/min. Thermogravimetric analysis (TGA) was measured under N_2 atmosphere from 35-900°C with a ramp rate of 10°C/min.

3.0 PROTOTYPES

3.1. Materials

PHA-based resins utilized in this study are composted primarily through microbial (aerobic and anerobic) pathways and were certified by the supplier for industrial (ASTM D6400), home (ASTM D6400), soil (ASTM D5988), and marine (ASTM D6991 & D5271) compostability with third-party certification through TUV Austria and possess the OK Biodegradable® certificate. Due to the propriety nature of the resins and additives, specific details cannot be published.

3.2. Lab trial compost prototypes

Figure 1. Summary of sample types utilized in lab composting trials - (top row, left to right) 1:5 scaled large thermoformed tray, 1:5 scaled medium thermoformed tray, 18-20 mil thermoforming sheet stock, 2 mil barrier-

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modified film, 2 mil unmodified film. (Bottom row, left to right) 1:5 scaled large thermoformed tray with heat-sealed film lid, 1:5 scaled medium thermoformed tray with heat-sealed film lid, full-size small thermoformed tray with heat-sealed film lid.

Film and sheet prototypes at lab scale were extruded using the benchtop extruder to produce samples approximately 100 mm wide. Sample dimensions for film and sheet samples were kept consistent at approximately 20 mm x 100 mm for most samples, with some sheets thermoformed into trays that were 1:5 down scaled from their intended size of pilot prototypes.

3.3. Municipal compost trial prototypes

Figure 2. Compostable tray and bag prototypes produced using the pilot extrusion line. Displayed are small tray (top left), medium tray (top right), large tray (bottom left) and heat-sealed bag (bottom right).

Lab-scale extrusion conditions (100 mm film or sheet @ 1-2 kg/h) were translated to pilot film line which was capable of producing approximately 600 mm (24”) films or sheets at approximately 20-50 kg/h. The pilot-extruded prototypes underwent secondary processing to produce bags (heat-sealing of films), trays (thermoforming of sheets), or thermoformed trays with heat-sealed film as a lid at their full intended size for pre-commercial prototyping. Bags measured approximately 25 cm x 25 cm and an approximate gauge of 2 mil (50 µm). For thermoforming samples, small trays measured approximately 10 cm x 10 cm x 2.7 cm, medium trays 20 cm x 10 cm x 5 cm, and large trays 20 cm x 20 cm x 5 cm. The gauge of all trays was approximately 18-20 mil (400-500 µm) with the lidding stock at 2 mil (50 µm).

4.0 COMPOSTING METHODS

4.1. Initial evaluation of PHA resins by lab trials

Samples were tagged with small pieces of polypropylene line and an identifier tag. The samples were buried in steel pans measuring 13 cm by 23 cm by 8 cm, on top of a 25 mm layer of compost and were then covered to the top of the pan with compost matrix. Several trials were executed over the course of the project evaluating different compost media to determine their effectiveness with the resins.

Lab compost trials were conducted over a 6 or 9-week period. The samples were distributed across several trays to limit the local compost matrix effect in the trays. Approximately 1 kg of the mixture was required to fill the trays, and each tray was kept moist with 150 mL of deionized water every 3-4 days. Furthermore, the positions of trays were rotated at each watering period to compensate for airflow differences within the environmental chamber, as it was noted that trays on uppermost shelves dried out slightly faster than those located near the bottom. Sample degradation was evaluated based on the physical characteristics of the prototypes used for the compost trials, making note of empirical changes in the modulus (brittleness), pitting or shedding, and overall degradation of the article.

4.2. Evaluation of prototypes generated from pilot-line extruder at municipal waste management facility

The municipal compost trials were conducted at the Cape Breton Regional Municipality Solid Waste Management Facility located at 145 Sydney Port Access Road, Sydney, Nova Scotia. Two trials were conducted spanning 7 weeks (49 days) from early June to late July, and from early September to late October for 7 weeks (49 days). For ease of access and retrieval of the samples, the compost trial was conducted with material that had already undergone the primary breakdown in the main composting process and was stored in a separate, covered building to undergo secondary curing and aging (hereafter referred to as the compost). This material was still very microbially active, and we were advised by knowledgeable staff that this pile can continue to be active and reach sufficient temperature after turning to reintroduce oxygen to the pile. The pile utilized for the first study had entered the curing building in mid-March, while the second trial pile was added in late-March. Approximately 10 yards of the compost was removed from the pile, after which the film and tray prototypes were added and spread out in a random pattern, followed by covering the samples with the compost material that had been removed.

For the first compost trial, samples were tagged with 40 lb-test fishing line that had coloured identifying tape for each class of sample. Three replicate samples were included for each prototype, with no modification to the samples. For the second compost trial, samples were placed in coloured nylon mesh bags, and the empty volume of the bag was filled approximately halfway with compost material, followed by adding the designated prototype and filling the remaining volume in the bag with compost. The filled mesh bag was then placed in the compost pile. Each bag was tagged with 16-gauge galvanized wire that ran to the pile exterior with unique stainless steel tags to identify each sample. A single replicate of each sample prototype for the second trial. For thermoformed trays with a lid, a slit was cut into the lid stock to mimic an opened tray and to allow moisture and some compost matrix to permeate into the tray as degradation took place.

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5.0 RESULTS

5.1. Lab composting trials

For lab composting trials, initial evaluations were completed that evaluated virgin (unmodified) resins as received by the resin supplier, as well as those which had been modified at the VC using the benchtop extruder. These modifications incorporated various additives to alter the characteristics of the extruded prototypes. While these modifications were primarily focused on changing the barrier properties of the prototypes, it was anticipated that these modifications may alter the compostability of the resins as well. Therefore, an initial compost trial was carried out to evaluate the unmodified and modified resins and to determine if any changes in composting speed occurred.

Two sets of trays were filled with a finished horse manure compost that was collected from a local farm approximately 24 h before use in the trial. Two sets of samples were evaluated – the first was to examine if different sizes of film samples had any significant impact on degradation rate, while the second was to evaluate modified film and sheet samples. For the unmodified film samples, a reference sample of a commercially available compost bag was inserted to evaluate effectiveness of the composting process. With the modified film and sheet samples, a piece of polyethylene bag was added as a control.

Generally, the film samples showed very little difference in the speed of composting relative to the size of the prototype, but similarly little degradation was apparent for the control compostable bag over the 6-week period of the initial trial. Over the course of the trial, the film samples became opaque and a qualitatively observed decrease in tensile properties was noted. During the final observation, some degradation was observed at the terminal ends of the film samples, with small fragments lost from the primary samples. Significantly more degradation was observed for the modified film and sheet samples. Sample 8A and 8C film samples were modified with different concentrations of barrier modifier, with the higher concentration present in 8A. 8A showed increased degradation speed compared to 8C, which suggests that this formulation may have dual function by altering the barrier properties as well as the composting speed. However, even the modified film samples did not show as rapid a degradation as thermoforming and injection molding sheets (samples 3 and 4 respectively, Figure 3), which were significantly degraded after just 3 weeks under composting conditions. Only small fragments of these samples remained by 5 weeks and were indistinguishable from the compost matrix by the end of the trial at 6 weeks.

An additional compost trial was conducted to evaluate prototypes that had been generated using both the bench extruder and the pilot extrusion line using optimized formulation and conditions, as in some cases the processing parameters can have an influence on compostability. These samples included unmodified and modified film samples, as well as thermoformed trays that were 1:5 scale models that would be used in municipal compost trials. Trays were included with and without the lidding stock to evaluate if the slower degradation of film resins impacted thermoforming resin when in direct contact. The smallest prototype tray intended for the municipal trial was sufficiently small to fit in the lab compost trial trays and therefore was included in the trial as well. Additional samples were evaluated that used other additives that could influence the composting speed or impart antimicrobial properties. These additional blends were prepared at the bench scale but were not ready for pilot scale extrusion trials at the time of the study.

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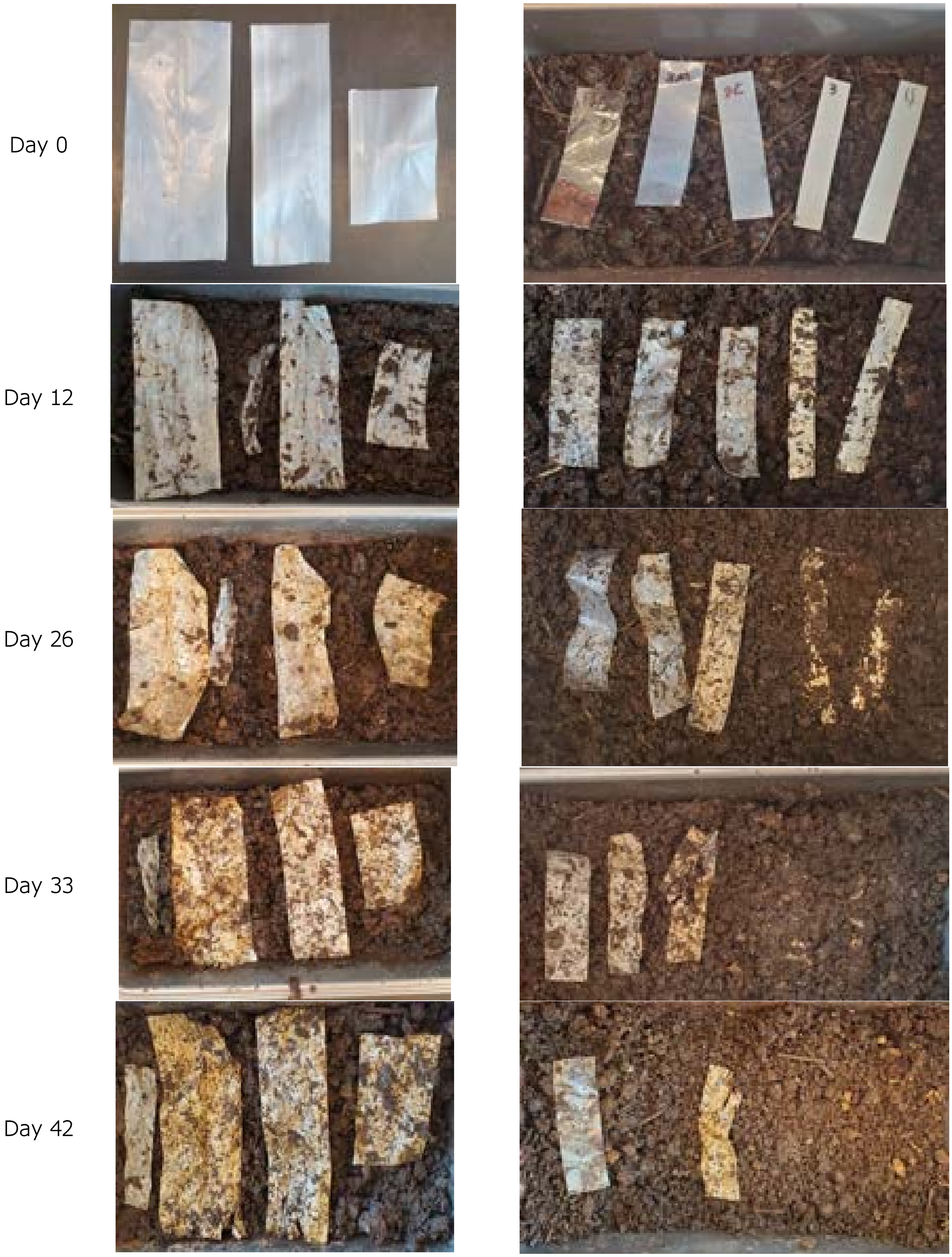

Figure 3. Initial lab scale compost trial conducted with unmodified film samples (left column) and modified film and unmodified sheet samples (right column).

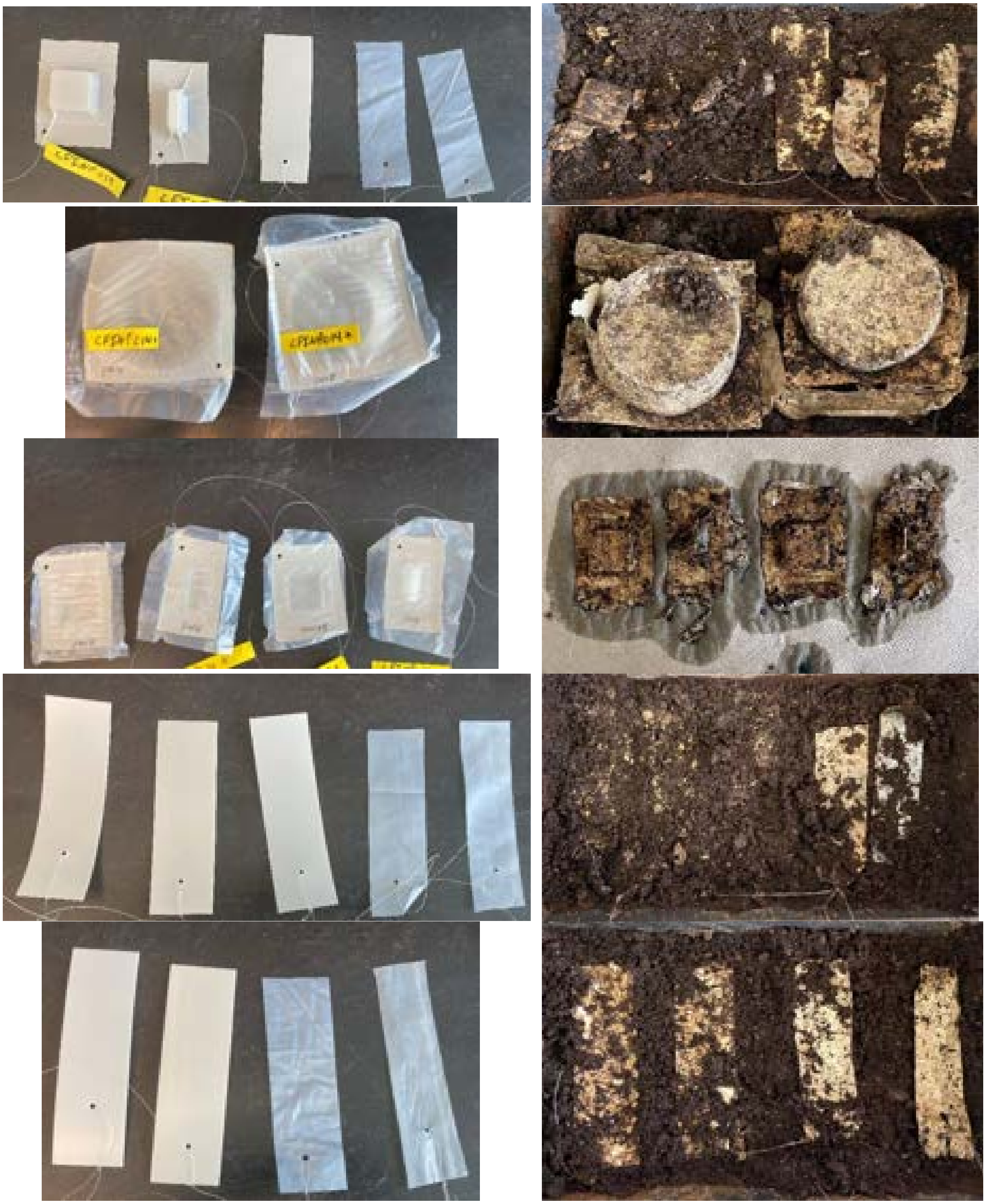

Figure 4. Samples utilized for lab compost trial #2 before insertion and at the completion of the trial after 63 days.

The second lab compost trial saw significant degradation of the samples. Scaled down pilot trays, both lidded and un-lidded, gave some insights on how the degradation is taking place and what areas of the prototypes would degrade preferentially. 1:5 scaled down pilot trays without lid stock were uninhibited throughout the composting process with full contact to the compost matrix and were nearly fully composted over the 63-day period. In comparison, lidded 1:5 scale trays showed less degradation, particularly around the lidding area. The tray portions showed more degradation and saw that the tray portion had delaminated from the lid stock. As observed with other bench samples, the film degradation was significantly slower compared to the thermoforming resin, and modified film lid stock showing more degradation than unmodified lid stock. Full scale small trays were not significantly degraded in this trial, and could be a result of their size relative to the amount of compost in the pans. These observations support the conclusion that the more slowly degrading lid stock inhibits the degradation speed of the thermoformed tray.

Table 1. Description of samples present in each tray for compost trial #3.

Tray #	Sample #1	Sample #2	Sample #3	Sample #4	Sample #5
1	1:5 large tray (no lid)	1:5 medium tray (no lid)	Thermoforming sheet (20 mil, pilot)	2 mil film (barrier-modified, pilot)	2 mil film (unmodified, pilot)
2	Full scale small tray, barrier-modified film lid	Full scale small tray, unmodified film lid	---	---	---
3	1:5 large tray, unmodified film lid	1:5 medium tray, unmodified film lid	1:5 large tray, barrier-modified film lid	1:5 medium tray, barrier-modified film lid	---
4	Thermoforming sheet (20 mil, bench) modifier A	Thermoforming sheet (20 mil, bench) unmodified	Thermoforming sheet (20 mil, bench) modifier B	2 mil film (modified, bench) barrier + antimicrobial modifier	2 mil film (modified, bench) antimicrobial modifier
5	50:50 film:thermoforming blend sheet (20 mil, bench)	20:80 film:thermoforming blend sheet (20 mil, bench)	3 mil film (barrier-modified, pilot)	2 mil film (barrier-modified, bench)	---

Of particular note was 3 samples of thermoforming resin placed in tray 3 that utilized 2 different additives to accelerate composting speed (designated "A" and "B", sample #1 and #3 respectively in tray 3) against an unmodified thermoforming resin. We observed that small pieces of the sample would separate from the perimeters sooner than those that were unmodified. Of the 3 samples, modifier B seemed to accelerate the composting speed the most when evaluating at day 28 (Figure 6). However, all three of these samples were significantly degraded when the trial was finished. An additional set of blends was included in tray 5 that utilized a blend of film and thermoforming resin, as these particular blends had beneficial impacts in thermoforming application testing. However, there was concern based on data from the first composting trial that the degradation speed would be slowed, and this hypothesis was confirmed. Compared to tray 1 sample #3 and tray 5 samples #1, #2, and #3, which were or almost entirely degraded, the blends of film and thermoforming resin in tray 5 showed signs of degradation but were still mostly intact.

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Figure 5. Degradation pattern of lab prototype thermoformed trays.

As with previous trials, unmodified and modified film samples became opaque and more brittle over the course of the trial. Additional modified film formulations were evaluated in this trial, which included antimicrobial additives, and a mixture of antimicrobial and barrier modifiers. The antimicrobial additives seemingly had no impact on degradation speed and appeared comparable to unmodified film. The film modified with both antimicrobial and barrier modifiers performed similarly to the barrier modified film, suggesting that the barrier modifier still had a positive impact.

Figure 6. Tray #3 samples at 28 days of composting.

5.2. Municipal composting trials

The first municipal composting trial conducted at the CBRM Solid Waste Management Facility utilized samples which had all been produced on the pilot extrusion line and were representative of pre-commercial prototypes. All films were produced under the same extrusion conditions for unmodified and modified samples, and thermoforming sheets were prepared under the same extrusion and thermoforming conditions. Three (3) replicates of each prototype were added to the compost pile without any secondary containment, which was later implemented in the second compost trial. This approach was used as academic experts consulted on the composting protocol indicated that a mesh bag, while helpful in containing the local area around a sample, may hinder the composting process and slow down the exchange of moisture, heat, and microbes and give a false result. Therefore, when added to the compost pile on in early June the prototypes were randomly cast in the compost pile before being covered. A summary of the prototypes tested included:

- small tray unmodified film lid stock
- small tray barrier-modified film lid stock
- medium tray unmodified film lid stock
- large tray unmodified film lid stock
- large tray barrier-modified film lid stock
- small bag unmodified 2 mil film
- small bag barrier-modified 2 mil film
- medium bag unmodified 2 mil film
- medium bag barrier-modified 2 mil film
- large bag unmodified 2 mil film
- large bag barrier-modified 2 mil film

Figure 7. Distribution of prototypes in compost pile for trial 1 at CBRM Solid Waste Management Facility.

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Figure 8. Covering of prototypes with compost for the first trial at CBRM Solid Waste Management Facility.

After 7 weeks (49 days), the prototypes were excavated from the compost pile. The equipment operator carefully removed compost in layers of approximately 1-2 yards and spread over the facility floor. The VC personnel then carefully combed through the material searching for the samples and prototype tag lines. As the searching began, it was evident that significant or complete degradation of the prototypes had taken place, as almost all tag lines were no longer attached to a sample remnant or fragments. Approximately 15 yards of compost was removed and scoured for intact and pieces of prototypes, and it was difficult to positively identify samples inserted for the trial due to the presence of some residual paper and plastic pieces in the municipal compost. Some samples were positively identified on site, and any samples that could potentially be related to the trial were collected. A positive consequence of this was that due to the lack of finding largely intact articles, the composting process was immediately deemed effective at degrading the prototypes. Of the samples were recovered that were still somewhat intact, it appeared that these particular prototypes were found near the top of the compost pile, where it is expected that the moisture content and temperature of the compost was lowest.

Samples collected from the trial were brought back to the VC lab for further evaluation and analysis. This evaluation provided a more qualitative assessment of the samples by conducting thermal transition analysis of the recovered samples, which allowed for more definitive conclusion on if a recovered sample or fragment was one of the compostable PHA-resins or a residual in the compost by identifying characteristics like the polymer melting point. This assessment highlighted that some of the samples returned to the lab were not part of the trial. Some of the non-trial samples included what appear to be vinyl surgical gloves, stretch wrap, and one hard polypropylene-like plastic.

Figure 9. Collage of photos collected during the first municipal compost trial prototype recovery.

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Figure 10. Samples recovered from the first CBRM compost trial.

Thermal analysis of the recovered samples was compared against the reference data collected for the resins after initial extrusion processing. From the recovered samples, the small rigid pieces were confirmed to be from the compostable prototypes, but due to the advanced degradation of the samples it was unclear which prototype(s) the fragments belonged to, except for one larger fragment that likely came from the large tray prototype based on its dimensions. For the flexible samples, the larger bags were confirmed to be compostable prototypes; however, the thermal analysis could not differentiate between the unmodified and barrier-modified resins. By comparing the degradation pattern of the samples and considering the pattern observed in the lab composting trials, most of the flexible samples appeared to be unmodified resin film. This conclusion was based on the degradation patterns of these articles in lab composting trials where unmodified films were largely intact. In contrast, the barrier-modified versions showed more holes and would break down into strands of film more frequently than unmodified film. Several smaller pieces of film were found and lacked evidence of heat sealing to another section of film resin, suggesting that these smaller pieces were either the central part of bags or lidding stock from trays.

For the second municipal compost trial, noting that significant degradation had taken place for most prototypes during the first trial, the trial protocol design was altered to obtain the best recovery of the samples and confirm the identity and degree of degradation for each unique prototype. Therefore, for the second trial only a single replicate of each prototype was utilized, and each prototype was contained within a nylon mesh bag that would not degrade under composting conditions. Furthermore, the mesh bag was tied off to a length of galvanized steel wire that was tagged with a stainless steel valve tag that contained a unique identification code. Both of these modifications would allow the tag to be traced directly to a bag with a much lower chance of

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separation. Also, each mesh bag was colour-coded for the associated prototype, that in the event the bag became separated from the line/tag combination, there was a second way to identify the unique prototype. Using mesh bags would allow for recovery of the local area around a prototype with minimal disturbance of the sample and could be evaluated more thoroughly off-site to better assess the degree of degradation. The degree of degradation in the second trial was estimated empirically as opposed to using sample weight or surface area measurements. Firstly, sample weight could be altered by residual compost materials (bulk compost material, absorbed compounds) which would artificially inflate the sample mass. Secondly, surface area measurements were also considered, but due to the advanced degradation of the samples observed in the first trial and the difficulty discerning trial samples from residual plastic fragments in the compost that surface area measurements could be skewed by materials not part of the original sample prototype.

Figure 11. Insertion of samples into the compost curing pile for the second trial at CBRM Solid Waste Management Facility.

For the second compost trial, the following samples were included:

- small tray unmodified film lid stock
- small tray barrier-modified film lid stock
- medium tray unmodified film lid stock
- medium tray barrier-modified film lid stock
- large tray unmodified film lid stock

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- large tray barrier-modified film lid stock
- large bag unmodified 2 mil film
- large bag barrier-modified 2 mil film

5 yards of compost was removed from the pile, followed by an additional 5 yards from inside the pile that was used for filling the sample bags. Each bag was half-filled with compost before inserting the sample, followed by covering the sample and filling the remaining bag volume with compost. The trays and bags were intentionally not filled with compost, as while this would maximize contact to the compost matrix and enhance degradation, this scenario would be highly unlikely in the true process. The mesh bag was securely closed and then added to the compost pile. The galvanized wire and tags were located such that the tags rested outside of the compost pile and could be followed during excavation and expedite recovery.

Figure 12. Distribution of samples in the compost pile during the second trial. From left to right, unmodified large bag (white), small tray with unmodified film lid (green), large tray with barrier-modified film lid (orange), barrier-modified large bag (white), small tray with barrier-modified film lid (pink), medium tray with barrier-modified film lid (yellow), large tray with unmodified film lid (red), medium tray with unmodified film lid (blue).

Figure 13. Sample retrieval for the second municipal compost trial. Highlighted with a red circle are sample tags during excavation (top-left), sample mesh bags (middle-left and middle right). Recovered sample bags are shown in the bottom-right picture.

After 49 days, the samples in the second trial were retrieved from the compost pile. The modifications to the protocol for sample recovery greatly improved the speed that samples were recovered and positively identified. Of the 8 bags inserted into the pile, only one lost its wire but was recovered. It was noted that the wire had corroded significantly leading to the failure. One of the mesh bags was punctured and torn during excavation by the loader (blue bag in Figure 12 containing medium tray with unmodified film lid); however, no contents of the bag were lost. Besides

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these minor issues, this protocol worked well to keep the local environment within the bag contained over the course of the trial and during recovery.

The sample bags were brought back to the VC for closer inspection and evaluation of the degree of degradation. None of the samples were found intact or in their original shape, and each showed a significant degree of degradation, which correlates with the observations of the first trial. It was understandable that sample recovery was difficult in the first trial, as all samples in the second trial had pieces that were on average no more than 2-4 mm in size, with some outliers in the 5-12 mm range. Some larger clusters of degraded material were found but may have been due to the local area of compost matrix and will be discussed further below. The mechanical integrity of the samples was significantly diminished, as any handling, especially for tray stock, would cause the samples to fall apart easily. As previously observed, film samples lost elasticity and were more brittle than prior to the compost trial.

Figure 14. Comparison of degradation for unmodified film bag (left column) and barrier-modified film bag (right column).

For film bags, the barrier-modified bag had more advanced degradation compared to the unmodified bag which were estimated at >90% and >75% respectively. Both types of bags showed more advanced degradation than the recovered bags from the first municipal trial. Some trends in the sample degradation were noted upon examination of the mesh bag contents and aligned with previous lab and municipal trials.

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Figure 15. Sample pictures of degraded tray samples. The top row features (from left to right) small, medium and large trays with unmodified film lid. The bottom row features (from left to right) small, medium, and large trays with barrier-modified film lids.

The barrier-modified bag saw much smaller pieces of film (< 5 mm) with little to no areas remaining intact, while there were areas of the unmodified bag that were degraded but still loosely intact (Figure 14). There were no apparent differences in the loss of mechanical integrity between the recovered sample fragments, as both seemed similarly diminished. For both samples, it appears that the lowest amount of degradation occurred near larger clumps of compost, as shown in the top row of pictures in Figure 14, and may have created a localized environment that lacked either the temperature, moisture, or resources required to advance the degradation. There may also have been a slight effect on the positions in the compost pile, as the barrier-modified bag sample was placed slightly more centralized in the pile, and may have had more favourable conditions during the 7 week period. Thermal analysis of the degraded film fragments showed the disappearance of the glass transition temperature, and a decrease in the onset of degradation by 50°C. In contrast, the fragments of the unmodified film bag onset of degradation dropped by approximately 40°C. While this measurement is not a true representation of average molecular weight of the resin, it

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does support the breakdown of the polymer structure and supports the more advanced degradation observed for the barrier-modified film over the unmodified film.

Figure 16. Before (left) and after (right) completion of the second municipal compost trial for the sample bag containing the medium tray with barrier-modified lid.

Figure 17. Example of degraded tray (small tray with unmodified film lid) with residual film stock on one surface face of the original tray (left), and localized area of diminished degradation for tray stock with film lid.

For tray samples, all the tray stock portions, regardless of initial size, showed advanced degradation in the range of 65-75%. There was no evidence of the samples in their original dimensions. When comparing the small, medium, and large trays' degradation pattern, the small and medium trays showed very similar patterns of pieces in the 2-4 mm range. It was noted that the large tray with unmodified film lid had a small portion of larger pieces of up to 12-24 mm in length which appeared to be areas of the tray lids where film stock had been heat-sealed to it. While pieces of this type were observed in trays with barrier-modified film lids as well, the larger-sized outliers were not as apparent or non-existent for barrier-modified film lid trays. This observation was not surprising considering that both of the film resins degraded more slowly than the thermoforming resin in bench trials and would reduce direct surface area exposure of tray stock to compost microbes. As with the film bag samples, it was noted that the large tray with unmodified film lid had a localized area of decreased degradation near a larger clump of compost material. While the overall piece of tray stock

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appeared larger (~24-48 mm, Figure 17) this area was severely fractured and comprised of many smaller pieces that were easily broken with mechanical manipulation. Similar to degraded film samples, thermal analysis of the tray stock samples showed evidence of degradation with decreases in the onset of degradation by up to 40°C indicating breakdown of the polymer at the molecular level. Furthermore, some thermograms showed emergence of new peaks, which further support breakdown of the polymer molecular structure.

6.0 CONCLUSIONS AND CLOSING REMARKS

In summary, the composting trials in the lab and municipal setting were successful, showing that these biopolymer resin prototypes degraded under both conditions. While the rate of degradation was slower under the lab conditions, it still provided insight and trends into the relative rates of degradation, and differences in the degradation pattern for each of the PHA resins and their prototype's features. This project also demonstrated that degradation of these samples proceeds rapidly at the CBRM Solid Waste Management Facility and serves as a proof of concept for the feasibility in other municipal compost facilities that employ comparable protocols. Through the trials conducted in this study, we can present the following recommendations that would give more insight into how the degradation pattern may change depending on where the prototypes are introduced into the municipal composting process, and how further studies may be improved to further accelerate the degradation.

Since the samples were introduced at the final (curing or aging) stage of the compost process, it is expected that if the compostable prototypes were introduced at an earlier stage in the municipal composting process, or immediately after completing the primary composting protocol, that degradation would accelerate and be more advanced for several reasons. Firstly, the introduction of the prototypes at the curing stage was done for ease of access and recovery, but it is expected that introduction at the beginning of the overall composting or curing process would increase the degree of degradation. This would give the prototypes further exposure to microbes and conditions during the initial two-stage composting process prior to curing but would also expose the prototypes to mechanical stress, mixing, and oxygenation during the transfer process at the completion of each stage. Mechanical agitation would facilitate the breakdown of prototypes by increasing the surface area. This would be particularly advantageous for tray stock, which had severely diminished mechanical properties during both the lab and municipal trials. Introduction at an earlier stage of the composting process, particularly during the first composting stage, would present additional logistical challenges for placement, tracking, and retrieval of samples, as well as challenges in any screening processes that take place before compost is transferred to the curing building.

A better understanding of typical and preferential composting conditions could give further insights for other municipal composting operations to replicate the results of this trial at the CBRM Solid Waste Management Facility. During the first trial, we noted that prototypes located near the top and sides of the compost pile were still somewhat intact. Furthermore, it was noted in the second compost trial that some portions of the prototypes showed decreased degradation around larger clumps of compost. Better understanding of common compost parameters such as C:N ratio, moisture content, oxygenation, pH, temperature at different locations and localized environments within the compost pile would be beneficial. This could be correlated with the corresponding degree of degradation to give a more complete picture of what conditions and locations will produce the fastest degradation of these particular resins. Additional adaptations to the prototype protocol could be made, evaluating the influence of filling the interior portions of the prototype bags and trays with compost to increase the exposure surface area, rather than leaving them unfilled as in this study.

Further expansion of the testing prototypes would add further robustness to the study. A single gauge of film and tray stock was utilized in this study, and while these are common gauges used in the industry, it does not cover the wide range of product structures employed in the plastics manufacturing. It would be expected that increasing the thickness of films or tray stock would slow degradation, and multi-layer film or tray structures (i.e. – 2-layer, 3-layer, or 5-layer structures) with differing layers could influence the degradation – either negatively or positively. Furthermore, the evaluation of additives that improve composting speed, such as those used in the lab trial, could be incorporated with prototypes of larger thickness to attempt to bring composting speed to a comparable level of thinner gauged prototypes.

With the data collected from the CBRM Solid Waste Management Facility, it would be beneficial to expand the number of participating municipal waste management facilities. Ideally, this would evaluate facilities that have both similar and differing composting protocols. The collection of data from other composting sites could be used to establish the baseline parameters required to quickly and successfully compost these resins and prototypes. These parameters could then potentially be integrated within the composting facility guidelines for municipal waste management sites across Nova Scotia to ensure complete degradation of PHA-based biopolymer products.

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