

Article

Evaluation of Biodegradability of Polylactic Acid and Compostable Bags from Food Waste under Industrial Composting

Angeliki Maragkaki ^{1,*}, Nikitas G. Malliaros ^{2,3} , Ioannis Sampathianakis ¹, Theofanis Lolos ³, Christos Tsompanidis ³ and Thrassyvoulos Manios ¹

¹ Department of Agriculture, School of Agricultural Science, Hellenic Mediterranean University, 71401 Crete, Greece; sabathianakisj@hmu.gr (I.S.); tmanios@hmu.gr (T.M.)

² Department of Chemistry, Voutes Campus, University of Crete, Crete, 70013 Heraklion, Greece; nmalliaros@uoc.gr

³ ENVIROPLAN SA, 23 Perikleous & Iras St., Gerakas, 15344 Athens, Greece; fl@enviroplan.gr (T.L.); ct@enviroplan.gr (C.T.)

* Correspondence: amaragkaki@hmu.gr

Abstract: To reduce plastic pollution, biodegradable plastics have been introduced to the market to replace petroleum-based plastics. This work investigates the biodegradation/disintegration of pure poly-L-lactic acid (PLLA_{B2B}), composed of food waste (FW), and PLLA_{B2B} bags, under industrial composting conditions, in order to determine whether they are compostable and to examine compost quality. In order to study the biodegradation, pure PLLA_{B2B} was degraded in laboratory conditions and bag samples were put into simulation systems using windrow technology. Phytotoxicity tests were carried out for every compost sample and high germination values were found (97–103.8%). The pure PLLA reached an average biodegradation value of $100.2 \pm 3.7\%$ and a disintegration value of $100.0 \pm 0.0\%$, resulting in biodegradable mature compost. After a seven week composting trial, the bag samples had fully decomposed. The samples had no influence on compost characteristics and there were no visually perceptible changes to the compost. Therefore, we suggest that food waste bioplastic can be used as a potential eco-material for compostable bags, one which could be used in industrial composting units and which offers degradable active materials with low environmental impact.

Keywords: biodegradation; food waste; polylactic acid; compostable bags; industrial composting



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1. Introduction

Plastic waste accounts for global emissions in the range of 19 to 23 million metric tons annually in fresh and sea water [1], with emissions on land also being calculated in the same order. These quantities are expected to double by 2030 [1]. At the same time most of the bioplastics used to remediate plastic pollution are xenobiotic materials and not entirely “green” [2].

Development of bio-based products has been strongly encouraged due to the depletion of fossil fuels, whereas biodegradable and compostable plastic items are developed with the hope of reducing environmental pollution or streamlining the collection of organic waste [3]. The raw materials for bioplastics used in the existing production method are most commonly based on starch (mainly from corn), using resources (water, energy, nutrients, land) and food quality crops for bioplastic production. This environmentally questionable activity will create massive social issues, especially in poorer countries, once the need for bioplastic increases. The main use of bioplastics is in packaging, which accounted for more than 48% (1.15 million tons) of the market for bioplastics in 2021 [4]. The demand for sustainable products from consumers and brands alike is on the rise due to an increased awareness of the effects on the environment and the need to lessen reliance on fossil fuels. This demand is also being fueled by the bioplastics industry’s ongoing development of new

materials with improved properties and new functionalities. The number of producers, converters, and end users is continually growing along with the quantity of materials, uses, and products. In this context, large relative growth rates are demonstrated by novel and inventive biodegradable plastics, such as polylactic acid (PLA).

On the other hand, municipal organic waste and agro-industrial byproducts have become a primary environmental, social, and economic concern in today's world [5,6]. Through the Waste Framework Directive (Directive 2008/98/EC, 2008), regulatory frameworks have been established throughout the European Union in order to create a sustainable waste management model that balances economic growth, technological advancement, and environmental and human health protection [7,8]. In this context, the circular economy could contribute to recategorizing these waste streams, as either feedstock raw materials or energy, according to the "cascading use principle".

The exploitation of bio-waste and agro-industrial organic waste through integrated processes is a rising area of investigation in the open literature, although studies are limited so far [9,10]. R&D in the area of waste valorization has focused on the production of energy and fuels but not on the production of bio-based products [11,12]. Bio-waste and agro-industrial organic wastes, that are rich in carbohydrates, protein and lipids/fats, are preferred as they can be combined into a waste stream of homogeneous composition, with large volumes, security of supply, possible funds as gate fee and simple logistics (mass transportation is possible) [13].

Due to the coexistence of biotic and abiotic elements in nature, the entire degradation process of a particular substance can be referred to as environmental degradation [14]. The material characteristics of PLA, such as its molecular first-order structure, as well as environmental aspects like humidity, temperature, and catalytic species, have an impact on how PLA degrades in the environment [15]. PLA is brittle, unclear, and opaque at low molecular weights, but it becomes stronger, more transparent, and more resistant to degradation at higher molecular weights [16].

Polymer mineralization happens under aerobic microbial biodegradation conditions [17]. Microorganisms in the compost can break down PLA in a composting setting after 45 to 60 days at 50 to 60 °C [18]. Under composting settings, commercially available PLA bottles and delicatessen containers decomposed noticeably after 30 days, with PLA bottles degrading more slowly due to their higher degree of crystallinity [19]. The term "aerobic biodegradation" describes the bio-degradation process whereby aerobic microbial respiration occurs. Due to the oxygen that is present, aerobic plastic biodegradation typically takes place in natural conditions like those associated with compost and soil [20]. Additionally, the aerobic process uses less energy and releases less methane, a more dangerous greenhouse gas than carbon dioxide, making it more energy-efficient [21].

According to several studies [17,22,23], blending affects how quickly bio-plastic films break down in soil, simulated landfills, marine water, aquatic ecosystems, and industrial composting facilities. The chemical structure of the resin, the activity of the soil and compost, and the depth at which the samples are buried all affect these rates. It has been attempted, in published research on the degradation behavior of blends, to relate the cumulative behavior of biodegradable films and articles, such as total CO₂ evolution and total weight loss, to their degradation and composting rate [24]. As already noted, one method for calculating the rate of total product breakdown under aerobic active microbial conditions is the measurement of evolved CO₂ [25,26].

In this study, the biodegradation/disintegration of PLLA_{B2B} produced from FW was examined under laboratory and real conditions (windrows), in order to assess how these proposed materials will behave after use. Additionally, the impact of the compost produced following the decomposition process was examined in terms of its phytotoxic impact on particular plants. To assess how the two different techniques affected the qualities of the materials, visual observations were carried out for each test at various periods.

2. Materials and Methods

2.1. Raw Materials

The food waste (FW) used in the present study was collected from the students' restaurant at the Hellenic Mediterranean University (HMU), Heraklion. The FW composition was 80% raw-fresh food (vegetables), 10% fruits and 10% salads (on a wet-weight basis). Vegetable waste (VW) was collected from a major supermarket in Crete. The HMU Department of Parks and Gardens provided material from pruning (PR) waste, branches and leaves. The PR was shredded with a Woodchipper Green Mech Abnorist 130. The commercial compostable bag (CB_c) is made of MaterBi designed for delivering organics to household waste collection systems and is compliant with the EN 13432 norm [27]. The compostable bag (CB_{B2B}) was made of PBAT with MFI = 4–5 g/10 min, 12% polylactic acid (PLLA_{B2B}), produced using FW gathered from the municipality of Heraklion in the context of the B2B Project and various other active ingredients.

The pure PLLA_{B2B} was synthesized through direct azeotropic polycondensation of L-lactic acid. For the production of lactic acid, the approach suggested by Sakai and colleagues [28] was followed and optimized for the creation of a scalable synthetic process. The PLLA_{B2B} was obtained from polymerization as a white powder with a particle size <3 mm and used without pretreatment. For the biodegradability tests the PLLA_{B2B} was cut and milled, obtaining thin, homogeneous pieces < 1 mm. Two fractions of PLLA_{B2B} were used (Figure 1): the polymeric granules 3 mm “PLLA_{B2B3}” for the disintegration test and the polymeric powder “PLLA_{B2BP}” for the composting process needed to determine the ecotoxic effect.

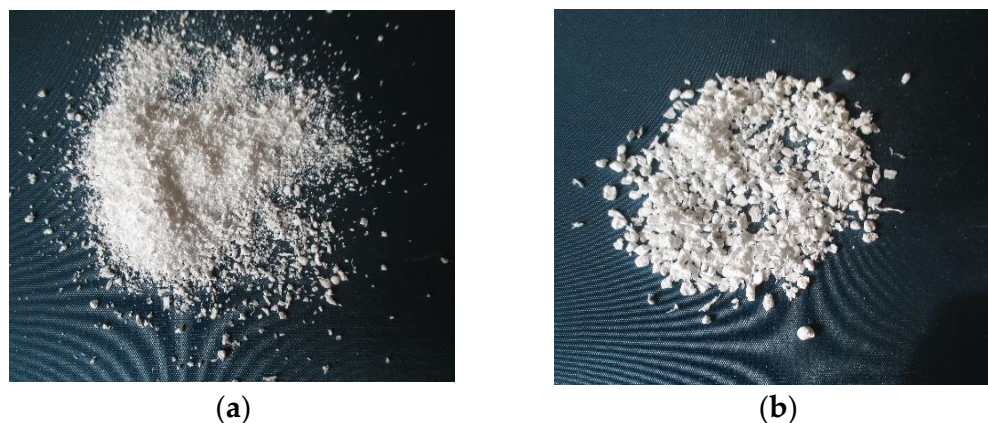


Figure 1. Sample pictures: (a) sample PLLA_{B2BP} for ecotoxicity test, (b) sample PLLA_{B2B3} for disintegration test.

For the biodegradability tests all the organic material components used to prepare the biowaste (bulking agent (BA) 24%, rabbit feed 46%, sawdust 13%, compost 13%, fruit, vegetable and cereals 3.8% and urea 0.2% dry wt) were reduced to a particle size < 50 mm. The pilot scale test was conducted under defined composting conditions according to ISO 16929:2021 [29].

The inoculum for the biodegradability test was a mature compost obtained from a composting plant treating 70% residues from public green waste management and 30% organic fraction from municipal waste collection. Before using it, the compost was sieved using a 2 mm sieve and, the fraction < 2 mm was used as inoculum. The inoculum characteristics were volatile solids (VS) 37.4%, moisture 41.1%, pH 6.3 and total nitrogen (TN) 1.8%. The mean composition of raw FW, VW, PR, CB_c, CB_{B2B}, biowaste and BA is summarized in Table 1. Table 2 provides a summary of the characteristics of the synthetic PLLA_{B2B} used in the current study.

Table 1. Characteristics of raw materials and compostable bags.

Parameter	VW	FW	PR	CB _c	CB _{B2B}	Biowaste	BA
pH	4.2 ± 0.4	4.2 ± 0.1	7.7 ± 0.1	-	-	5.9 ± 0.1	5.3 ± 0.1
Moisture (%)	88.8 ± 0.1	65.9 ± 1.5	59.6 ± 1.0	-	-	64.9 ± 1.2	31.7 ± 1.1
VS (g/kg) (DM)	904 ± 2.1	878.6 ± 11.7	890.3 ± 0.9	968.8 ± 5.5		877.0 ± 2.7	901.0 ± 1.2
N (%)	1.5 ± 0.0	2.5 ± 0.0	1.4 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	1.9 ± 0.0	0.5 ± 0.0
TOC (g/kg)	433.1 ± 4.9	634.1 ± 24.1	566.4 ± 66.1	692 ± 92.0		440.0 ± 2.3	426.2 ± 5.2
P (%)	0.1 ± 0.0	0.2 ± 0.0	0.3 ± 0.2	-	-	0.2 ± 0.0	0.1 ± 0.0

Table 2. Characteristics of produced PLLA_{B2B}.

Parameter	PLLA _{B2B}
Weight average molecular weight (M _w)	149,452
Number average molecular weight (M _n)	81,146
Polydispersity Đ	1.84

2.2. Determination of the Biodegradability Test

The biodegradability test was performed according to the UNI EN ISO 14855-1:2013 standard [30] by mixing the test sample PLLA_{B2BP} with mature compost to verify the capability of a typical microbial compost environment to biologically convert the organic fraction of the sample into CO₂, water and biomass.

The inoculum consisted of mature compost obtained from an industrial composting plant. The mature compost was sieved to eliminate the coarse fraction. The compost fine fraction obtained represented the test inoculum, while its volatile solids content had to be higher than 30% of the total compost dry solids.

The test sample PLLA_{B2BP} was mixed with the inoculum at a ratio of about 1:6 (dry solids) and introduced into the reactor. The reactors were placed in an incubator and kept at 58 ± 2 °C for the entire duration of the test.

The aerobic condition was maintained by blowing environmental air into the reactors. The aerobic condition permitted the conversion of the sample organic fraction into CO₂ during the test. The gas flow from each reactor was sent to a gas analyzer that determined the CO₂ concentration and the outlet flow rate at regular time intervals. The percentage of biodegradation was determined as the percentage of the initial theoretical carbon in the test compound that was converted into CO₂.

The sample was cut and milled to obtain thin, homogeneous pieces <1 mm. The blank consisted of three glass reactors (3 L capacity) containing testing mixture, while the reference sample consisted of three glass reactors (3 L capacity), each containing about 50 g of microcrystalline cellulose plus testing mixture, and the sample consisted of three glass reactors (3 L capacity), each containing about 50 g of milled sample plus testing mixture. Finally, the testing mixture consisted of mature compost from a composting plant mixed with an inert support (vermiculite, Sigma-Aldrich code 101532822), 300 g compost and 100 g vermiculite (dry weight) for each reactor with humidity at 50 ± 5% throughout the test.

The environmental air was blown through silicone rubber tubes into inlet gas flowmeters regulating the inlet flowrate of the reactors at approximately 10–15 L/h. Aquarium type pumps were used for this purpose. The aerobic condition permits the conversion of the sample organic fraction into CO₂ during the test. The air was previously humidified by bubbling in water at the same temperature as the reactors.

The air at the exit of each reactor was blown through gas-impermeable tubes into a humidity recovery system to eliminate water. Then the dry air was sent to a gas infrared analyzer that determined the CO₂ concentration and the flow rate at regular time intervals. The percentage of biodegradability was calculated as the percentage of CO₂ produced with respect to the total theoretical organic carbon content of the sample. The temperature

during the test was 58 ± 2 °C. The CO₂ measurement was carried out twice per day for the first five days of the test, then at least once per day for up to 45 days of the test. Afterwards, the measurement was performed at least five days per week up to the 90th day. The measurements were reduced to 3 times per week when the trial continued for an additional 90 days. Once a week for the whole duration of the test the reactor content was thoroughly mixed, while water was added when necessary. The mixing and water addition operations were recorded.

2.3. Determination of Degree of Disintegration

The purpose of this test was to evaluate the disintegration of the sample in a composting bin in the presence of freshly prepared biowaste. The composting process was monitored regularly and conducted until the compost was fully stabilized (3 months). At the end of the process the compost was sieved, and the disintegration of the sample was carefully measured. The ecotoxic effect of the compost obtained from this process was then evaluated to ensure the absence of any toxic effect on higher plants.

To perform the test, plastic bins of about 60 L capacity were used. The following bins were filled: (a) control bins: 2 bins each containing at least 20 kg of biowaste and (b) sample bins: 2 bins each containing at least 20 kg of biowaste with 10% sample concentration with respect to wet biowaste obtained by adding: 9% (PLLA₁: 1.87 kg, PLLA₂: 1.87 kg) polymeric granules shredded < 2 mm + 1% (PLLA₁: 208.3 g, PLLA₂: 206.5 g) polymeric granules with dimensions of 3 mm, to each bin. The two fractions of the sample were well mixed with the biowaste inside the bin. The temperature was measured at regular intervals with probes (every day for the first eight weeks and at least three times a week for the remaining weeks). Periodically the composting material was turned and visually inspected (once a week for the first four weeks and at least every two weeks for the remaining period). Water was added to the bins to restore humidity losses due to evaporation and the water content was maintained around 50–60% during the test, taking care that no free-standing water was present at the bottom of the reactor after turning of the composting material. During turning operations, the smell and appearance of the composting mixture were monitored, as was the appearance of the sample specimens. Sampling of composting material was performed to check pH at 4, 8 and 12 weeks. The reactors were aerated daily for 4 h by fluxing air to ensure that the oxygen content was >10%.

At the end of the 12 weeks of the test all of the bins were cooled. The compost was sieved with 10 mm, 5 mm and 2 mm sieves to isolate fractions of the residual sample. All the compost fractions, except the >10 mm fraction, were then used to determine the quality of the compost.

2.4. Ecotoxic Effect Evaluation

The phytotoxic effect (germination of seeds and growth of the plants) was assessed (a) on compost obtained after exposure to the composting process for 12 weeks, without the presence of the sample, and (b) on compost obtained after exposure to the composting process for 12 weeks, in the presence of the sample “PLLA_{B2BP}”. Reference compost and sample compost were mixed, before the analysis, with a reference substrate prepared by mixing vermiculite (83%) and peat (17%).

The phytotoxic effect tests were conducted with two different types of seeds: mung bean (*Vigna radiata*) (100 seeds per pot) and barley (*Hordeum vulgare*) (50 seeds per pot).

The sowing was conducted in pots filled with an 83% vermiculite/17% peat mixture containing the reference compost or the sample compost, both analyzed at two different concentrations: 25% and 50% (*w/w*). At the end of the test, germination (number of plants germinated) and biomass (dry weight of the plants) were evaluated.

Plastic containers of 500 mL volume were filled with 15 g of vermiculite, to which 50 mL of deionized water was added. Then, about 200 g of testing mixture was added and the seeds were distributed on the surface and covered by a thin layer of 83% vermiculite/17% peat mixture. The pots were covered (to preserve humidity) and incubated

in the dark till the seeds germinate, when the covering was removed, and the pots were exposed to light/dark cycles with a photoperiod of 16 h for 14 days. The plants were watered as necessary. At the end of the growth period, the plants were cut at the base where they emerge from the soil and the number of plants germinate and plant dry weight were determined.

For each seed type the following pots were set up:

- 3 pots with vermiculite/peat mixture (83% vermiculite and 17% peat), as growth control;
- 3 pots with reference compost 25% *w/w*;
- 3 pots with reference compost 50% *w/w*;
- 3 pots with sample compost 25% *w/w*;
- 3 pots with sample compost 50% *w/w*;
- for a total of 15 pots for each seed type.

2.5. Composting in Windrows

The biodegradability study was carried out in windrow technology simulation systems at the Hellenic Mediterranean University in Crete. The composting experiment was conducted in a commercial composter (BioActor, Athens, Greece, Helesi). The composter comprises ten stackable frames. Each frame consists of three integrated layers: (a) a perforated inner ventilation wall, (b) an insulating middle wall, and (c) a perforated outer ventilation wall. The FW, VW and PR were added to the composter in April 2019. PR was added to the material (FW:VW) at a volume ratio of 1:1 (FW & VW:PR). The compostable material was emptied from the composter and turned manually at one week intervals, with 7 turnings in total (7 weeks). In order to maintain moisture levels between 60% and 70% (*w/w*), water was occasionally added to the material manually. The temperature in the composter was measured on a daily basis in the core, at a depth of 40–60 cm. During turning, composite samples were taken, one from the top, 20 cm from the top of the system; one from the middle; and one from the bottom, at 10 cm from the bottom of the system. At each turning, the samples were lifted from the compost and all samples were subsequently photographed and assessed. Table 3 provides a summary of the material composition of samples used in the current study.

Table 3. Material composition of samples.

Sample	Type	Description
1	Cellulose (blank)	-
2	Starch	MaterBi EN 13432 norm-industrial compostable PBAT with MFI = 4–5 g/10 min, 12%
3	CB _{B2B}	polylactic acid (PLLA _{B2B}) and various other active ingredients

2.6. Analytical Methods

The raw composting waste and outflow were tested for moisture content, pH, electrical conductivity (EC), total organic matter (TOM), volatile solids (VS), total organic carbon (TOC), total nitrogen (TN) and total phosphorus (TP).

The weight loss of the sample after drying at 105 °C allowed us to determine its moisture content [31]. The pH and EC of a 1/1.5 solid/liquid aqueous extract were determined (extraction duration equal to 24 h), and total nitrogen (TN) was quantified using the semi-micro Kjeldahl method. Total phosphorus (TP) was determined by extracting the elements from solid samples and measuring them in the same way as liquid samples. Specifically, the TP was brought to a solution by acidic digestion using HNO₃. For TOC analysis, a solid sample module was utilized (SSM-5000A, Kyoto, Japan, Shimadzu). Each number was calculated as the average of three similar measurements.

The biodegradability test was performed according to the UNI EN ISO 14855-1:2013 standard [30]. The degree of disintegration test was conducted according to ISO 16929:2021 [29].

The ammonium nitrogen and nitric nitrogen were determined according to the method reported in UNI 10780:1998 [32]. An amount of 5 g of the sample is suspended in KCl for 1 h at a 1:10 ratio. After centrifugation, the solution was diluted (ten times) and Devarda's alloy was added. The solution was then distilled in the presence of NaOH boric acid. The captured nitrogen by boric acid was titrated with standard HCl. The evaluation of CO₂ concentration in the outlet gas from the reactors was carried out with a nondispersive infrared (NDIR) detector (Ecocontrol model EC100).

The ecotoxic effect was evaluated according to EN 13432:2000 [27]. The tests were conducted in triplicate. The results obtained from the reference compost and the compost sample were compared at different concentrations. The percentage of germination and the percentage of growth obtained using the sample compost were calculated as a percentage of the values obtained with the corresponding reference compost.

Regarding the germination capacity of the seeds, a layer of cotton and a filter paper disk were piled in a Petri dish and 5 mL of deionized water were added. Twenty seeds were distributed on top of the paper disk, and another previously humidified paper disk was placed on top of the seeds. The Petri dish was covered with the lid and closed by a parafilm strip. The Petri dishes were incubated for 4 days in the dark at room temperature. The germinated seeds were counted, and the percentage of germination (germination capacity) was calculated with respect to the initial seed number. The germination index (GI) was calculated using the formula found in equation [33] (GI): $GI (\%) = 100 \times (\text{average number of seed germination} \times \text{average length of treatment's roots}) / (\text{average number of seed germination} \times \text{average of root length of control})$.

Arsenic (As), cadmium (Cd), chrome (Cr), mercury (Hg), nickel (Ni), lead (Pb), copper (Cu), selenium (Se), zinc (Zn) and molybdenum (Mo) were determined according to UNI EN 15411:2011 [34]. The levels of heavy metals (Cr, Cu, Ni, Cd, Pb, Zn, Hg) were determined using atomic absorption spectroscopy (Perkin Elmer AAnalyst400, Waltham, MA, USA). An atomic fluorescence spectrometer was used to analyze As (AFS-820; Jitian, Beijing, China).

Fluorine (F) was determined according to EPA 9056A (2007) [35]. The EPA 5050 (1994): Bomb preparation method for solid waste was used for the sample preparation [36]. The sample was oxidized by combustion in a bomb containing oxygen under pressure. The liberated halogen compounds were absorbed in a sodium carbonate/sodium bicarbonate solution. The bomb combustate solution was then analyzed by ion chromatography (IC). IC analyses were conducted using a Dionex ISC-2000 reagent-free ion chromatograph eluent generator cartridge (RFIC EGCII KOH), CD 20 conductivity detector and anion self-regenerating suppressor (ASRS 300, 4 mm, all Dionex, Sunnyvale, CA, USA).

Origin 9 (OriginLab, Northampton, MA, USA) was used to undertake the statistical analysis of the data and the findings of this study (analysis of average values, variance, and standards deviation). Statistical analysis of variance (ANOVA) was used to examine significant differences.

3. Results and Discussion

3.1. Biodegradability Test

Tests on biodegradability are required to assess the environmental effects of plastic materials and to identify strategies with which to prevent the unsettling buildup of polymers after their commercial shelf life. The primary indicator typically used to calculate the rate of composting of biodegradable materials is the measurement of CO₂ evolution during composting of pure and blend samples.

Regarding the validation of the test, the degree of biodegradability of the reference material (microcrystalline cellulose) was 90% (>70% after 45 days). The difference among the percentages of biodegradability of the reference material (microcrystalline cellulose) in the different reactors was 9.2% at the end of the test (<than 20%). The CO₂ production of the compost after 10 days of test was 72.3 (between 50 and 150 mg CO₂/g of volatile solids). This means that the test was validated.

During the test setup, the milled sample was well mixed with compost and vermiculite. After four weeks of testing the sample reactor mixture was very similar to that of the blank and reference reactors.

The evolution of the total cumulative CO₂ production is reported in Figure 2 for the blank, reference and sample reactors. The evolution trend of CO₂ of the sample PLLA_{B2BP} was lower than the microcrystalline cellulose reference sample. After about 20 days it started to increase constantly until, after 50 days of testing, it exceeded the production of CO₂ of the microcrystalline cellulose reference. Table 4 provides the weight of the reactors at 0 and 91 days of test.

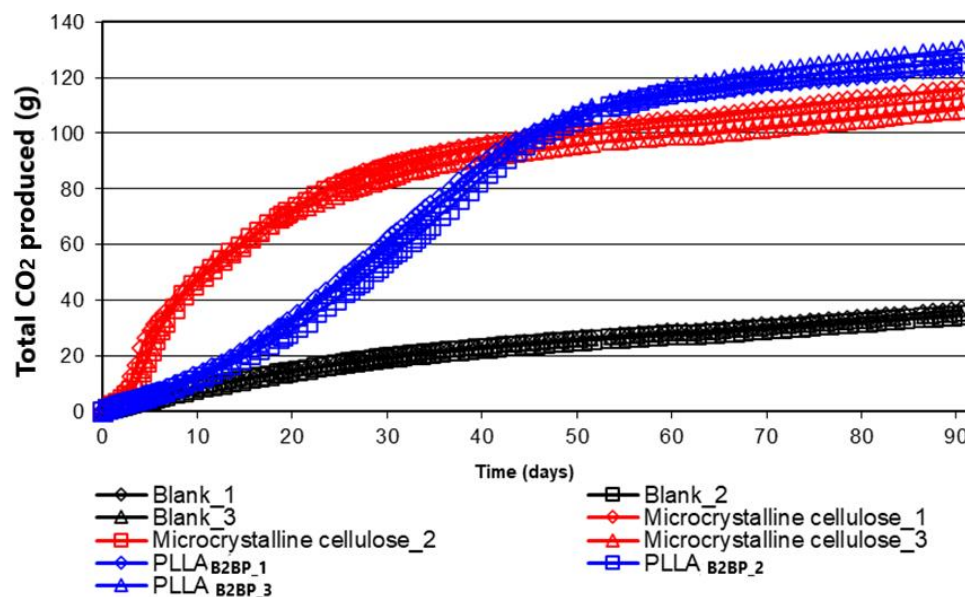


Figure 2. Total CO₂ production of the reference microcrystalline cellulose sample, PLLA_{B2BP} sample and blank.

Table 4. Weight of reactors at 0 and 91 days of test.

Reactor	Initial Weight (0 Day)		Final Weight (91 Days)	
	Gross	Net	Gross	Net
Blank_1	2160	793	2090	723
Blank_2	2152	793	2082	723
Blank_3	2152	796	2056	700
Microcrystalline cellulose_1	2260	896	2117	753
Microcrystalline cellulose_2	2240	877	2182	819
Microcrystalline cellulose_2	2256	886	2162	792
PLLA _{B2BP} _1	2256	888	2186	818
PLLA _{B2BP} _2	2259	888	2215	844
PLLA _{B2BP} _3	2254	885	2184	815

The biodegradability percentage was calculated with respect to the quantity of the total organic carbon initially contained in the samples. Figure 3 shows the evolution of the biodegradation percentages of the different replicates for the sample and the reference. The biodegradation of the sample PLLA_{B2BP} started lower, but after 20 days the biodegradation rate of the sample PLLA_{B2BP} increased quickly and constantly, reaching the biodegradation percentage of the reference.

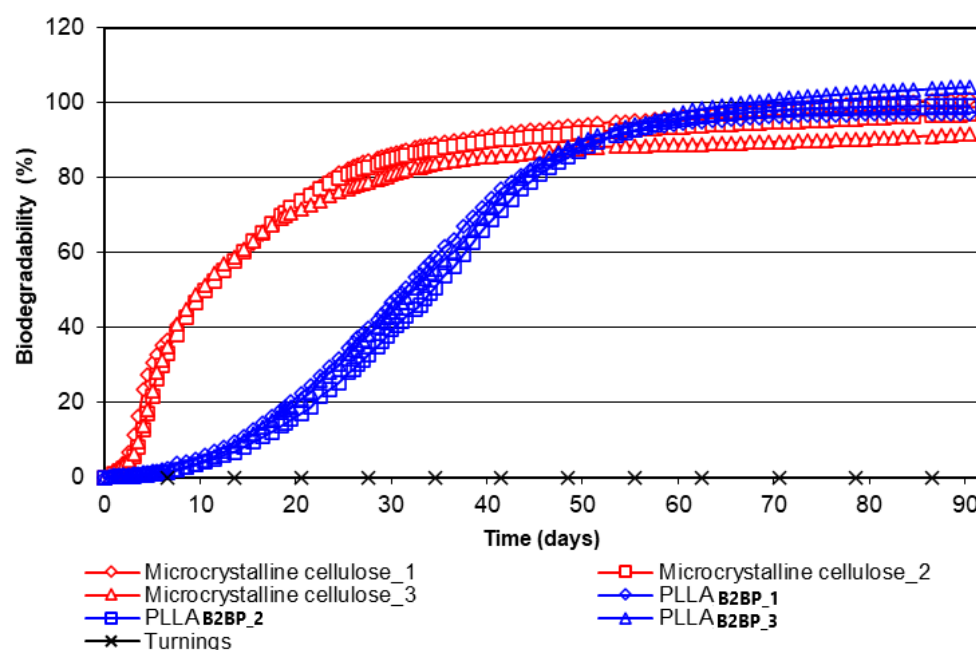


Figure 3. Evolution of aerobic biodegradability under controlled composting conditions for microcrystalline cellulose reference sample and PLLA_{B2BP}.

The biodegradability percentage was calculated with respect to the quantity of the total organic carbon initially contained in the samples. At the end of the test an average biodegradation value equal to $96.4 \pm 4.6\%$ was reached for microcrystalline cellulose and $100.2 \pm 3.7\%$ for PLLA_{B2BP}, thus fulfilling the 90% limit required by the EN 13432:2000 norm (limit value required $\geq 90\%$). Published works on the degradation behavior of blends have tried to relate the cumulative behavior of biodegradable films and articles, e.g., total CO₂ evolution and total weight loss, to their degradation and composting rate.

According to Kalita et al. [37] 80.50% to 94.20% biodegradation was observed in different PLAs at 136 to 139 days. After hydrolytic destruction, PLA oligomers and other intermediates were digested by microorganisms in the compost and turned into carbon dioxide, water, and humus [37]. For PLLA_{B2BP}, it was found that 85% of the composite fraction decomposed after 50 days as compared to 30 days for blank. Another study reported similar results, but did not include information on the lag phase, which was crucial for comparing the two systems [38,39]. This study confirms our findings on the compostability of PLA in used compost. In the case of Kalita et al. [37] the PLA biodegradation rate was comparatively slow. This could be for a number of reasons, including the material's suitability for composting, molecular structure, and mechanical strength. Additionally, several authors have observed that composite materials, such as PLA blends, disintegrated more slowly when composted [37,40].

3.2. Disintegration Test

In order to monitor the progress of the disintegration during mixing and humidity recovery, the bins were monitored for the odor and the visual appearance of the mixture and the sample under test. The odor and visual inspections evidenced a regular aerobic composting process during the 12 testing weeks overall. No deviations were registered during the test and therefore no corrective operations were adopted.

During the different process phases the following odor evolution for blank bins and sample bins was observed: (a) first 15 days: start of fermentation processes with rancid odor and increasing ammonia smell; (b) from 16 to 40 days: the rancid odor was attenuated until it disappeared completely and was replaced by an ammonia odor that became strong; (c) from 41 to 56 days: ammonia smell decreased and was replaced by fresh soil smell; and (d) from 57 to 84 days: presence of typical odor of mature compost by the end of the

test. The visual aspect of the organic waste in the bins evolved, starting from an initial darkening associated with a volume reduction that was much more visible in the sample reactor than in the Blank.

During the second week the growth of white molds was noted, which increased to cover a major part of the biowaste. At the same time, the smell of ammonia significantly increased. The biowaste in the sample reactors showed a significant volume reduction starting from the second week, while the sample mixing with the biowaste increased by disintegration and darkening.

The granules of the sample, both 3 mm and <2 mm, started to become dark and to integrate with the biowaste by the end of the first week. The disintegration process increased in the following days: after only 22 days of the test only small residues of the granules remained in the reactors. At 29 days of the disintegration test the sample completely disappeared. Figure 4 shows the sample during the composting process. Table 5 presents the final product's quality obtained at the end of the test. Table 6 presents the results of the sample PLLA_{B2B} disintegration test.

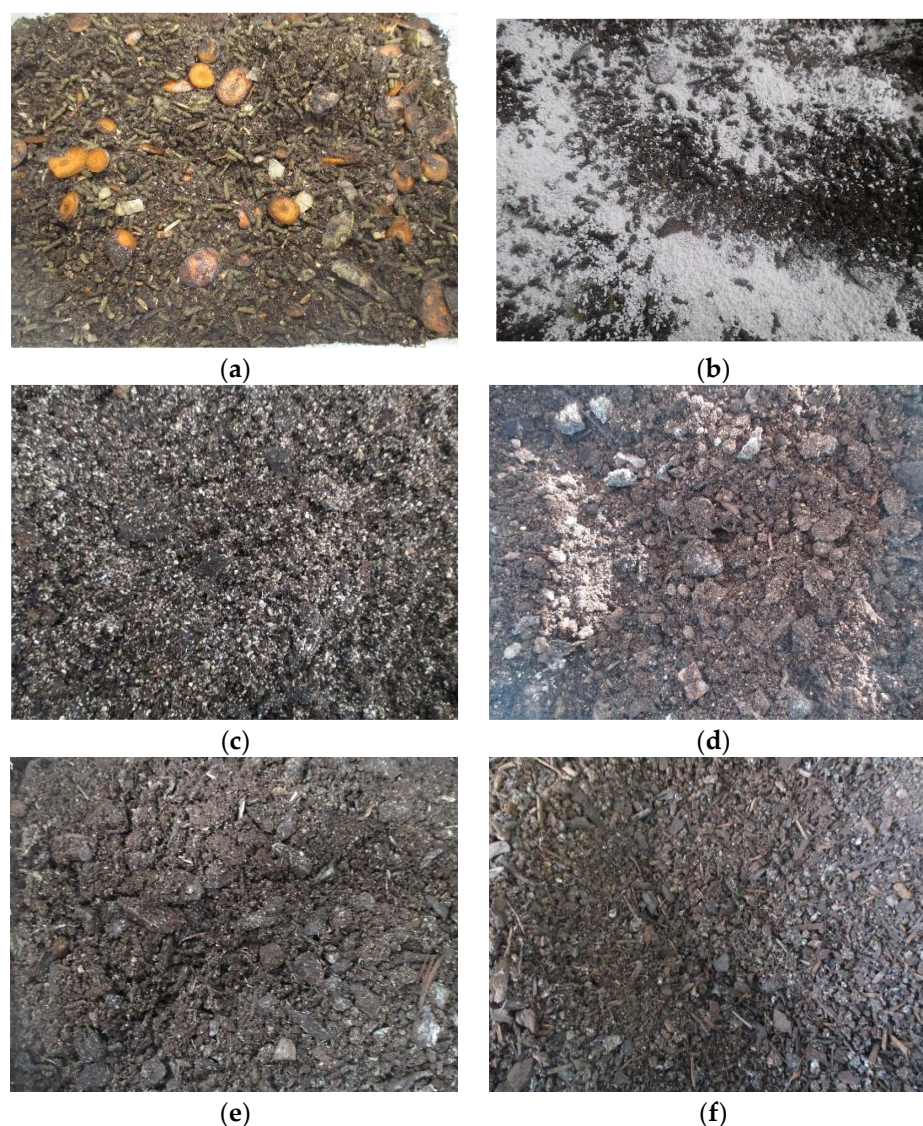


Figure 4. Photo of the sample during the test, at the end of the test and the final residues of the sample. (a) Fresh Biowaste at the beginning of the test, (b) sample granules at the beginning of the test, (c) disintegration of the granules after 8 days, (d) residues of the granules after 22 days, (e) final sample compost after 12 weeks, (f) final sample compost < 10 mm sieving fraction.

Table 5. Chemical characterization of the compost obtained at the end of the test.

Parameter	Blank ₁	Blank ₂	PLLA _{B2B1}	PLLA _{B2B2}
Moisture (%)	49.2 ± 0.5	39.0 ± 0.3	35.5 ± 0.1	50.1 ± 0.1
Volatile solids (%)	73.8 ± 2.1	72.5 ± 4.5	73.2 ± 2.6	72.5 ± 3.3
pH	6.9 ± 0.1	8.0 ± 0.1	7.9 ± 0.1	7.2 ± 0.1
NH ₄ -N (mg/Kg)	2343 ± 6.1	2145 ± 3.6	3176 ± 2.7	2770 ± 9.3
NO _x -N (mg/Kg)	3692 ± 4.9	3951 ± 8.1	3090 ± 21.1	3387 ± 10.2
TN (%)	2.9 ± 0.0	2.8 ± 0.0	2.9 ± 0.0	2.9 ± 0.0
C/N	12.7 ± 3.1	13.2 ± 2.7	12.7 ± 4.2	12.5 ± 0.1

Table 6. Results of the sample PLLA_{B2B} disintegration test.

Sample Bin	Weight of Sample Residues (g) as Dry Weight				Disintegration (%)	Average Disintegration (%)	Disintegration Limit (%)
	Initial	At the End					
		2–10 mm	>10 mm	Total			
PLLA _{B2B1}	204.5	0.0	0.0	0.0	100.0	100.0	≥90.0
PLLA _{B2B2}	202.7	0.0	0.0	0.0	100.0		

The odor and visual controls evidenced a regular aerobic composting process during the 12 testing weeks overall. At the end of the test the compost obtained from the composting process was mature, as the maximum temperature measured during the self-heating test remained below 30 °C: Rottegrad = V as requested by ISO 16929:2021. No deviations were registered during the test and therefore no corrective operations were adopted. The sample of PLLA_{B2B} polymeric granules with dimensions of 3 mm, disintegrated at 100% value, within the limits specified by the EN 13432:2000/AC:2005. In contrast with earlier studies on thicker samples, the results of PLLA_{B2B} disintegration demonstrate extended stability [41,42]. The sample of polymeric granules with a large surface area and high porosity may be responsible for the rapid breakdown [43,44]. As set out in Table 5, PLLA_{B2B} did not affect the chemical characteristics of the compost obtained at the end of the test.

3.3. Phytotoxic Effect Test

Using composts produced from the breakdown of the materials studied in a lab, phytotoxicity tests were conducted to determine the compost quality.

The compost obtained from the disintegration test in the presence of the sample PLLA_{B2B} did not present an inhibiting effect on either the germination or growth of both plants analyzed at the different compost concentrations tested. The results are shown in Table 7. Figure 5 presents photographs of ecotoxic test.



Figure 5. Photos of ecotoxic test (a) growth of ecotoxic test (4th day), (b) end of ecotoxic test (Wastewater and Solid Waste Management Laboratory of the Department of Agriculture).

Table 7. Percentage of germination and growth on the Sample Compost with PLLA_{B2B} respect the values obtained with the Reference Compost.

Compost (%)	Barley		Mung Bean	
	Germination (%)	Grown (%)	Germination (%)	Grown (%)
25	98.5	109.3	103.8	110.6
50	101.5	118.0	97.0	95.3

High germination values were found in the phytotoxicity test: 97.0–118.0%.

3.4. Heavy Metals

The concentrations of heavy metals and other toxic and hazardous substances of sample PLLA_{B2B} were determined within the limits specified by the EN 13432:2000. The results are shown in Table 8.

Table 8. Chemical characterization of the compost obtained at the end of the test.

Substance (mg/kg)	PLLA _{B2B}	Limits EN 13432:2000 (mg/kg)
Chrome	0.2 ± 0.1	50
Cobalt	<0.1	38 (a)
Nickel	<0.1	25
Copper	1.9 ± 0.4	50
Zinc	4.7 ± 0.9	150
Arsenic	<0.1	5
Selenium	<0.1	0.75
Molybdenum	<0.1	1
Cadmium	<0.1	0.5
Lead	0.2 ± 0.1	50
Mercury	<0.05	0.5
Fluorine	<50	100

The extended uncertainty values refer to a 95% confidence interval. Coverage factor $k = 2$. (a) Canadian Certification Program (CAN/BNQ 9011-911-1/2007).

3.5. Composting in Windrows

At the end of the experiment at the HMU, the samples were taken to the Wastewater and Solid Waste Management Laboratory of the Department of Agriculture, where they were subjected to detailed evaluation. In all samples, a visual comparison was made of their initial and final states. In terms of this visual assessment all samples exhibited the highest degree and rate of decomposition. Photographs of the initial condition of the samples, their condition in each week, and the final condition of the samples at the end of the experiment are presented in Figure 6. At the end of the experiment, all samples were decomposed to about 100% of their initial condition. Moisture and temperature were monitored weekly over the course of the experiment (Figure 7).

Due to water absorption and the creation of low molecular weight compounds, the whitening process and formulation opacity are linked to changes in the refractive index [45] while, during degradation, the formation of certain holes on the materials and an induced increase in crystallinity are observed [46].

Figure 7 illustrates the development of composter temperatures and the material moisture during the experimental period. As shown in the graph, the highest composter temperatures were reached in the 2nd turning when the temperature amounted to 70 °C. Composter temperatures in the 3rd and 4th turnings were 67.6 and 47.2 °C, respectively. To determine the compost quality, phytotoxicity tests were carried out using composts resulting from the disintegration of the materials tested under laboratory conditions. The compost GI value was found to be 67% for the 1:10 dilution ratios.

The composition of the blank samples verified the favorable composting conditions. According to Angelica et al. (2020) [47], the ideal moisture content required for composting

is between 50 and 60%. The initial moisture content was 61.0, 0.1%, within the 50–60% range recommended in the literature to support the composting process.

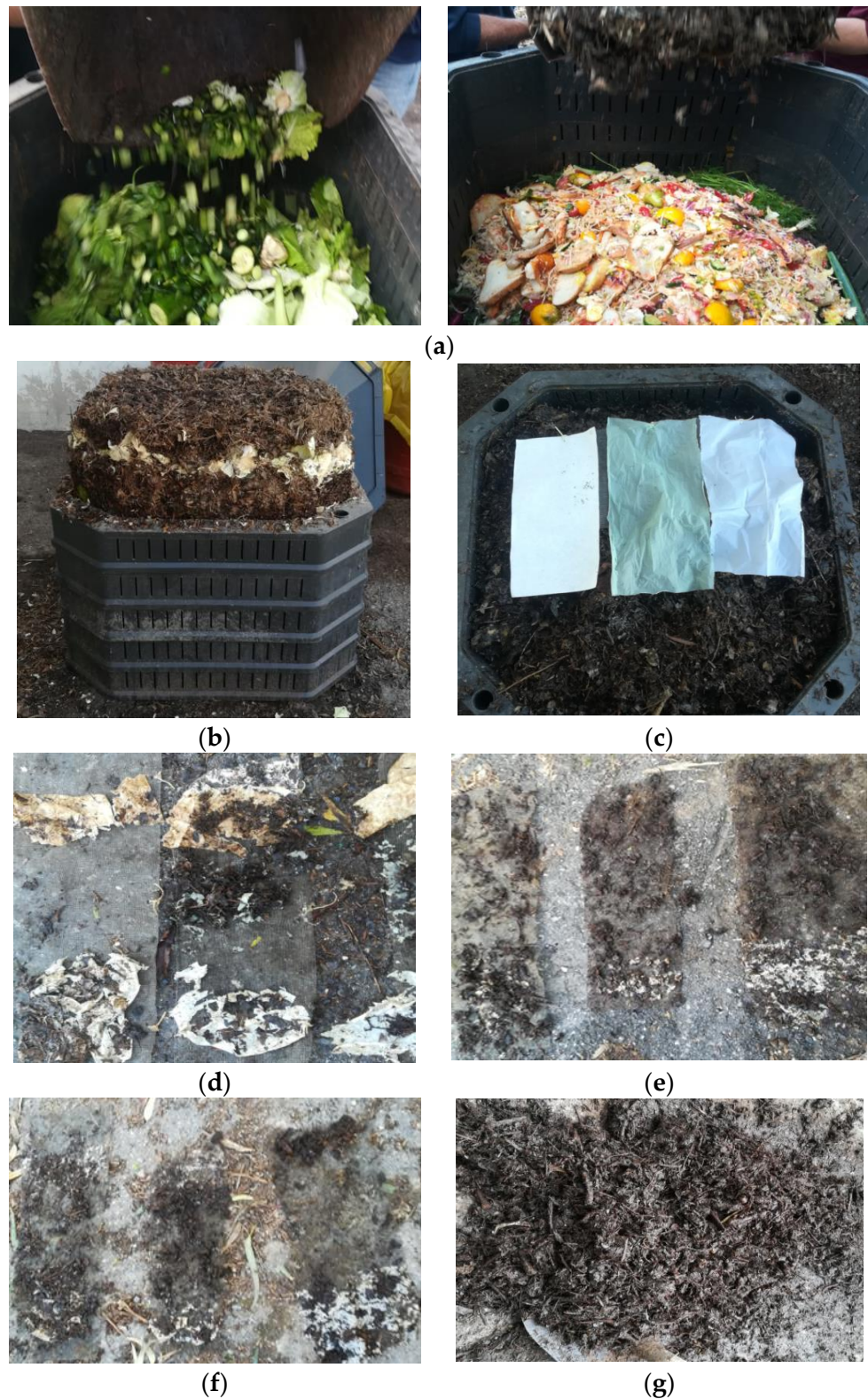


Figure 6. Photos of the sample during the test. (a) Fresh FW, VW and PR at the beginning of the test, (b) turning 1 after 5 days, (c) turning 2 after 7 days and with addition of the samples, (d) turning 3 after 7 days, (e) turning 4 after 7 days, (f) turning 5 after 13 days, and (g) turnings 6 and 7 after 7 days.

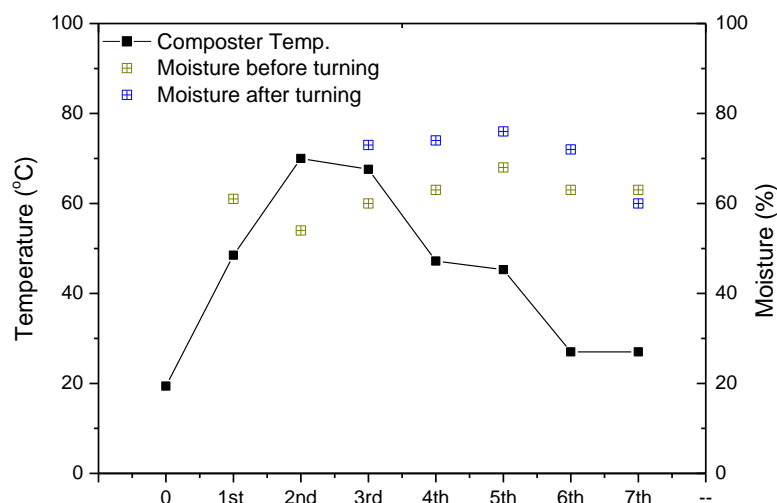


Figure 7. Composter temperature and moisture variation during composting experiment before and after turning of the materials.

Table 9 presents the analysis of materials during the composting process. The pH increased from 5.9 ± 0.0 to 8.6 ± 0.0 . The pH tends to rise during the first two to three weeks of composting as ammonia gas is produced from the breakdown of nitrogen, but it tends to fall later as organic acid breaks down into organic matter, according to He et al. (2020) [48]. EC decreased from 4.8 ± 0.0 mS/cm to 3.2 ± 0.0 mS/cm with the composting process. The high salt content in the raw materials presents a significant challenge because the salts would be released into the final compost after composting, and if it were used as fertilizer, it might result in too much salt in the soil, which might prevent the soil from absorbing other salts [49]. The EC was less than 4 mS/cm after the fifth week, which is favorable for plant growth [50]. TN concentration increased (from 1.4% to 2.5%); this was due to an organic mass reduction in the composting process. Composting produced more stable, mature compost and reduced TOC, increased TN, and decreased C/N ratios. The nitrogen content was found to be at an acceptable level when compared with other compost [51].

Table 9. Analysis of materials during the composting process.

Turnings	Days	T (°C)	Moisture B ¹ (%)	Moisture A ² (%)	pH	EC (mS/cm)	TN (%)	TOC (mg/g)	Ash (%)	TP (%)
Beginning	0	19.4	-	-	-	4.8 ± 0.0	-	561.0	-	-
1st Turning	5	48.5	61.0 ± 0.1	-	5.9 ± 0.0	3.6 ± 0.1	1.4 ± 1.1	545.0	12.3	0.3 ± 0.1
2nd Turning	12	70.0	54.0 ± 1.0	-	7.9 ± 0.0	4.5 ± 0.0	2.2 ± 1.2	508.0	15.5	0.6 ± 0.0
3rd Turning	19	67.6	60.0 ± 0.9	73.0 ± 0.9	8.9 ± 0.0	4.3 ± 0.0	2.1 ± 0.0	495.0	24.6	0.5 ± 0.1
4th Turning	26	47.2	63.0 ± 0.0	74.0 ± 0.0	9.0 ± 0.0	4.1 ± 0.2	2.4 ± 5.2	450.0	29.6	0.9 ± 0.2
5th Turning	39	45.3	68.0 ± 66.1	76.0 ± 66.1	8.6 ± 0.0	3.9 ± 0.1	2.5 ± 0.0	344.0	23.8	2.0 ± 0.0
6th Turning	46	27.0	63.0 ± 0.2	72.0 ± 0.2	8.7 ± 0.0	3.3 ± 0.1	2.4 ± 0.0	440.0	10.1	1.1 ± 0.3
7th Turning	53	27.0	63.0 ± 0.2	60.0 ± 0.2	8.6 ± 0.0	3.2 ± 0.0	2.5 ± 0.0	447.0	6.4	0.9 ± 0.0

¹ before turning; ² after turning.

The goal of our experiment was to test the decomposition of the above-described samples in real conditions of industrial composting in windrows technology simulation. The temperatures reached over a long term in the compost pile ranged from 50–70 °C. Tibu et al. [52] recorded the same range. Sample 2 (certified as compostable) and 3 were decomposed. Samples 2 and 3 exhibited the highest decomposition rate (before the expiration of the 7 weeks). Sample 1 was a control reference sample with which to confirm that the conditions of decomposition were suitable during the experiment, which was the case. This was to check the potential of biological decomposition in the tested environment. This agreed with other studies [53,54].

Composting seems to be the most promising waste management option for degradable plastics because the composting process is designed to degrade waste. There are, however, obstacles that make many communities reluctant to accept plastic bags for composting. The plant growth phytotoxic test revealed that PLLA_{B2B} had no negative effects on the composting procedure or compost quality. In conclusion, this article describes the biodegradability and compostability of PLLA_{B2B} and indicates that the EN 13432 standards [27] for industrial composting are fulfilled. Additionally, the degradation degree of compostable bags that were tested in a real composting environment was reached at 100%. The knowledge gained in this study will also contribute to the development of policies and assessments for bioplastic waste, as well as provide direction for future bioplastics research and development.

4. Conclusions

This study reports on the biodegradation/disintegration of pure PLLA composed of food waste and the bioplastics produced from this PLLA, ascertaining and their ability to form compostable materials. Only the question of whether the PLLA_{B2B} material is biologically degradable is addressed by the substantiation of degradation in laboratory conditions in accordance with current standards. It does not, however, address whether the bags made from these PLLA_{B2B} are successfully degradable in the setting of an industrial composting operation, so simulation systems using windrow technology were used to study their biodegradation/disintegration. Laboratory tests for PLLA_{B2B} reached a biodegradation level of $100.2 \pm 3.7\%$ with high germination values, and concentrations of heavy metals and hazardous substances which meet the stringent requirements of the European standard EN 13432:2000 for compostability. Furthermore, the results of the tests conducted under actual composting conditions were comparable with laboratory tests. These findings demonstrate the potential for the development of a novel, sustainable bioplastic. Therefore, we suggest food waste bioplastic as a new potential eco-material for compostable bags and their use in industrial composting units, as they can be considered promising degradable active materials with low environmental impact. In conclusion, the results of this study open up new opportunities for material manufacturers, who should prioritize testing in real settings using different raw materials, mainly waste.

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Abbreviations

Poly-L-lactic acid (PLLA), biowaste to bioplastic (B2B), total nitrogen (TN), total phosphorus (TP), total organic matter (TOM), volatile solids (VS), total organic carbon (TOC), germination index (GI), polylactic acid (PLA), food waste (FW), pruning (PR), compostable bag (CB), vegetable waste (VW), Hellenic Mediterranean University (HMU).

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