



Study of the biodisintegration of a bioplastic material waste

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ABSTRACT

The aim of this work was to study the biodisintegration degree of different pieces made of a biodegradable thermoplastic material, the polylactic acid (PLA) with and without corn in its composition, is studied. The pieces of different shapes and thicknesses were obtained by both injection and extrusion processes, where also a specific foaming additive of polystyrene was added. The PLA and PLA-corn manufactured pieces were subjected to aerobic degradation at a constant temperature of 58 ± 2 °C for 90 days, following EN 14806 and ISO 20200:2004 Norms. It was found that the pieces made of PLA and PLA with foaming agent had an average biodisintegration degree of 63.6%. With regard to the pieces made of PLA-corn, an average biodisintegration degree of 79.7% was obtained. In this case, the percentage of non degraded material was independent of the size, shape and thickness of the original pieces.

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1. Introduction

Due to plastics are used in a wide range of applications, great amounts of plastic wastes are generated each year all over the world. Reusing of plastic wastes is preferable to recycling as it uses less energy and fewer resources. Mechanical recycling is problematic because of the wide variation in properties and chemical composition among the different types of plastics. Because of this variability, plastics must be sorted either by hand or by automated devices prior to recycling. Other management options as incineration, pyrolysis, hydrogenation, gasification and thermal cracking are more tolerant to impurities than mechanical recycling, although they are capital intensive and require very large quantities of used plastic to be economically viable. On the other hand, the disposal of plastics in landfills also has a significant environmental impact because most plastics are non-degradable (<http://www.wasteonline.org.uk/resources/InformationSheets/Plastics.htm>, July 9th, 2007).

Nowadays, manufacturers are developing degradable plastic materials. There are two types of degradable plastics: photodegradable plastics, which are degraded when exposed to sunlight and biodegradable plastics or “bioplastics”, which are polymers made from renewable resources as corn, sugars, potatoes, etc., and they can be degraded under controlled conditions of biodegradation, as for example, composting of the organic fraction (Körner et al., 2005; Mohee and Unmar, 2007). A plastic is called “biocompostable” when it is biologically decomposed during a composting

process at a similar rate to other compostable materials and without leaving visible toxic remainders. In order to designate a plastic as “biocompostable”, its total biodegradability, its disintegration degree, and the possible ecotoxicity of the degraded material must be determined by means of standard tests (AENOR, 2001; Tuominen et al., 2002).

This research work focused on studying the application of biodegradable plastics to the manufacturing of pieces of different sizes, shapes and thicknesses. In particular, the material object of this study was the polylactic acid or PLA, which is a biodegradable polymer (Kunioka et al., 2006; Tuominen et al., 2002). Two conformation processes, injection and extrusion, were used in order to obtain sheets, sprues and cylindrical pieces. The main objective was then to study the degree of disintegration of the manufactured pieces under an intensive aerobic composting process. Although the degradation process of PLA is initiated by a simple hydrolysis, any factor which affects the reactivity and the accessibility such as particle size, shape, water diffusion, etc., can affect the polymer degradation rate (Kale et al., 2006). In our case, the influence of the thickness and shape of the pieces on the biodisintegration degree will be also evaluated.

2. Methods

2.1. Bioplastic materials

The investigated materials were polylactic acid (PLA) and polylactic acid containing 10 wt.% of corn (PLA-corn), both provided by the Laboratoire de Physico-Chimie des Polymères of the Université de Pau et des Pays de L'Adour (Pau, France).

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2.2. Transformation processes

Transformation of PLA and PLA-corn was done by means of two conformation processes: injection and extrusion. For the injection experiments, only PLA-corn was used. Injection was carried out in a conventional injection machine Mateu Solé 55 tons at an optimum temperature of 220 °C. Cylindrical pieces (12 mm thickness and 40 mm diameter) and conical sprues (60 mm length and 6–8 mm diameter) were obtained. Also the addition of a specific foaming additive of polystyrene (PS) was tested. This chemical additive was selected because of its accessibility, low cost and processing temperature range. Its optimum amount was previously determined carrying out several experiments with PS in the injection machine. It was found that the optimum dosage of foaming agent was 2 wt.% for pieces of 12 mm thickness. However, when the PLA-corn and the foaming agent were added together to the injection machine, the PLA-corn material was degraded inside the screw, causing operational problems in the machine. As a consequence of this, no piece of foamed PLA-corn could be obtained.

Extrusion was carried out in a double screw extrusion machine DIGSA ZK50 with both PLA and PLA-corn. The optimum temperatures were: 190 °C for PLA and PLA-corn; 220 °C for PLA with foaming agent. Three millimeters thick plastic sheets were made of PLA, PLA and foaming agent and PLA-corn. Whereas the foaming agent could be added together with PLA, it could not be used with PLA-corn due to it produced serious operational problems in the extrusion machine. On the other hand, it was observed that PLA material with no foaming agent presented shrinkage problems during the cooling stage.

2.3. Biodisintegration tests

The biodisintegration degree of the obtained pieces was evaluated following EN 14806 Norm “Packaging. Preliminary evaluation of the disintegration of the packaging materials under simulated composting conditions in a laboratory-scale test” (AENOR, 2006a) and ISO 20200:2004 Norm “Determination of the degree of disintegration of plastic materials under simulated composting conditions in a laboratory-scale test” (AENOR, 2006b). According to these tests, the obtained plastic pieces by injection or extrusion were mixed with a solid biodegradable synthetic material and subjected to aerobic degradation. The composition of the synthetic material was the following (as percentage of dry mass): 10% compost, 40% wood sawdust, 30% rabbit food, 10% corn starch, 5% sucrose, 4% corn oil and 1% urea. The used compost corresponded to a three months old mature compost which was provided by a full scale aerobic composting plant located in Pina de Ebro (Zaragoza, Spain). The commercial compost was sieved through a 5 mm sieve before its addition to the mixture.

The manufactured pieces were cut into smaller ones and vacuum-dried at 40 ± 2 °C for 72 h before the experiments. The biodisintegration experiments were carried out by triplicate with two types of samples: “E” samples, which included pieces made of PLA and PLA with foaming agent, and “M” samples, consisted of pieces made of PLA-corn. The samples were the following:

- Samples type “E”: they contained sheets (25 mm × 25 mm × 3mm) of PLA and foamed-PLA obtained by extrusion (by triplicate, reactors E1, E2 and E3).
- Samples type “M”: they contained PLA-corn pieces (sheets of 25 mm × 25 mm × 3mm, cylindrical pieces of 15 mm × 15 mm × 12mm and original sprues) made by both extrusion and injection (by triplicate, reactors M1, M2 and M3).

Also a reactor containing wet synthetic material without plastic pieces was prepared (reactor “Blank”).

An amount of 19–20 g of plastic pieces was mixed with 1 kg of wet synthetic material (55 wt.% of water) and put in a polypropylene reactor. The polypropylene vessels of 330 mm × 180 mm × 130mm (length, width, height) were hermetically sealed to avoid excessive evaporation, but they had holes of 5 mm in the middle of each side of 180 mm for providing air exchange. The initial volume of the mixture was approximately 60% the volume of the reactor. In Table 1 are summarised the initial weights of synthetic material and the corresponding amounts of plastic added to each reactor.

Before the experiments, the solid synthetic material was checked by means of analysing volatile suspended solids (VSS) (Method 2540G of “Standard methods”, APHA, 1992), pH and C/N relationship. C/N relationship was calculated considering that the % of carbon corresponds to VSS value divided by 2 (AENOR, 2006a,b), whereas the content of nitrogen corresponds to N-Kjeldhal, which was analysed following Method 4500-N_{org} of “Standard Methods” (APHA, 1992).

The aerobic degradation was carried out in an air circulation oven at a constant temperature of 58 ± 2 °C for 90 days (Jung et al., 1999). During this time moisture, mixing and aeration of the samples were periodically controlled as Norms EN 14806, ISO 20200:2004 and Kaczmarek and Bajer (2006) establish (Table 2). Also the pH was measured in order to study its evolution.

After 90 days of composting period, the seven obtained composts were characterised by means of determining their final mass, VSS, moisture, pH and N-Kjeldhal. Each compost was dried at 58 ± 2 °C until constant mass and sieved with the objective of separating the remaining plastic pieces greater than 2mm. The recovered fragments were washed with distilled water, dried at 40 ± 2 °C and weighed for calculating the corresponding biodisintegration degree (*D*):

$$D = \frac{M_i - M_f}{M_i} 100 \quad (1)$$

where M_i corresponds to the initial dry mass of plastic material and M_f represents the dry mass of the recovered plastic material after composting and sieving.

In order to validate the biodisintegration results, the volatile solids decreasing (*R*) and the variability of the biodisintegration degree were calculated:

$$R = \frac{[m_i(DM)_i(VS)_i] - [m_f(DM)_f(VS)_f]}{[m_i(DM)_i(VS)_i]} 100 \quad (2)$$

where m_i denotes the initial mass of the wet synthetic material before composting, $(DM)_i$ is the initial dry mass of synthetic waste (as % of total mass) and $(VS)_i$ represents the volatile solids of the initial synthetic material (as percentage of DM). The term m_f corresponds to the final dry mass of the obtained compost, $(DM)_f$ represents the final dried mass of compost (as % of total mass) and $(VS)_f$ is the volatile solids value of the obtained compost (as % of DM).

3. Results and discussion

3.1. Monitoring and control of composting experiments

In order to verify that the initial synthetic material was adequate for carrying out the composting reaction, some parameters as dry mass (or moisture), volatile solids and nitrogen were previously determined for each reactor (Table 3).

As it can be seen, the percentage of dry mass was adequate as the Norms recommend 45%. The average content of volatile solids was 88.67%, indicating a high content of organic matter. On the other hand, the Norms recommend an initial C/N value between 20:1 and 40:1. In this case the average C/N relationship was 32.5:1, which was considered optimum for composting.

Table 1
Amounts of synthetic waste and plastic pieces placed in each reactor.

Reactor	Weight of wet material (g)	Total amount of plastic (g)	Kind of plastic/piece (g)	Total weight (g) (including vessel)
E1	1001.0	19.5	PLA: 7.9 + foamed-PLA: 11.6	1229.2
E2	1000.1	20.3	PLA: 8.7 + foamed-PLA: 11.6	1221.0
E3	1000.1	20.3	PLA: 8.9 + foamed-PLA: 11.4	1224.7
M1	998.8	20.7	Sheets: 8.2 + sprues: 4.6 + cylindrical: 7.9	1226.5
M2	1001.5	20.6	Sheets: 8.3 + sprues: 5.0 + cylindrical: 7.3	1223.0
M3	999.9	21.0	Sheets: 7.4 + sprues: 4.3 + cylindrical: 9.3	1214.7
Blank	1000.6	0	0	1198.1

The composting reaction was periodically controlled by measuring the temperature and determining the pH and content of moisture of each reactor. The temperature was maintained at $58 \pm 2^\circ\text{C}$ as it is shown in Fig. 1. During the period of composting, temperature decreased below 55°C at days 10 and 82, probably due to changes in room temperature that affected the oven temperature. As soon as these changes were detected, the oven temperature was adjusted to increase the temperature up to 58°C .

Fig. 2 shows the pH variation of each reactor over time. The observed pH changes correspond to the theoretical evolution of a real composting plant (Haug, 1993; Tchobanoglous and Kreith, 2002).

Table 2
Task schedule during composting time.

Day of composting	Task control
0	1. Preparation of reactors 2. Weighing of reactors
1, 2, 3, 4, 7, 9, 11, 14	1. Weighing of reactors to control water mass 2. Addition of water to restore 100% of initial mass 3. Mixing and manual aeration
8, 10, 16, 18, 21, 23, 25, 28	1. Weighing of reactors to control water mass 2. Addition of water to restore 100% of initial mass
30	1. Weighing of reactors to control water mass 2. Addition of water to restore 80% of initial mass 3. Mixing and manual aeration
From day 30 to 45: twice a week	1. Weighing of reactors to control water mass 2. Addition of water to restore 80% of initial mass
45	1. Weighing of reactors to control water mass 2. Addition of water to restore 80% of initial mass 3. Mixing and manual aeration
From day 45 to 60: twice a week	1. Weighing of reactors to control water mass 2. Addition of water to restore 80% of initial mass
From day 60 to 90: twice a week	1. Weighing of reactors to control water mass 2. Addition of water to restore 70% of initial mass

Table 3
Percentages of dry mass, volatile solids, carbon, nitrogen and C/N relationship of the synthetic wastes before composting.

Reactor	Dry mass (%)	Volatile solids (% dry mass)	Carbon (% dry mass)	Nitrogen (% dry mass)	C/N
E1	45.61	88.65	44.33	1.59	27.89
E2	45.20	88.56	44.28	1.51	29.28
E3	42.45	88.98	44.49	n.a.	–
M1	41.53	89.21	44.61	1.26	35.38
M2	43.02	87.93	43.97	1.26	34.96
M3	42.47	89.05	44.52	n.a.	–
Blank	42.75	88.32	44.16	1.27	34.79
Average	43.29	88.67	44.34	1.38	32.46

n.a.: Not analysed.

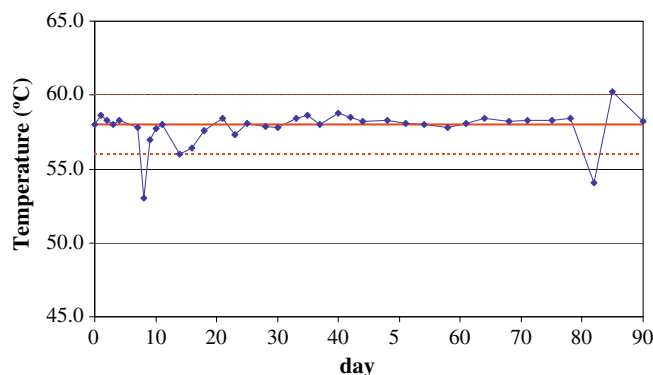


Fig. 1. Temperature variation with respect to time during the composting period.

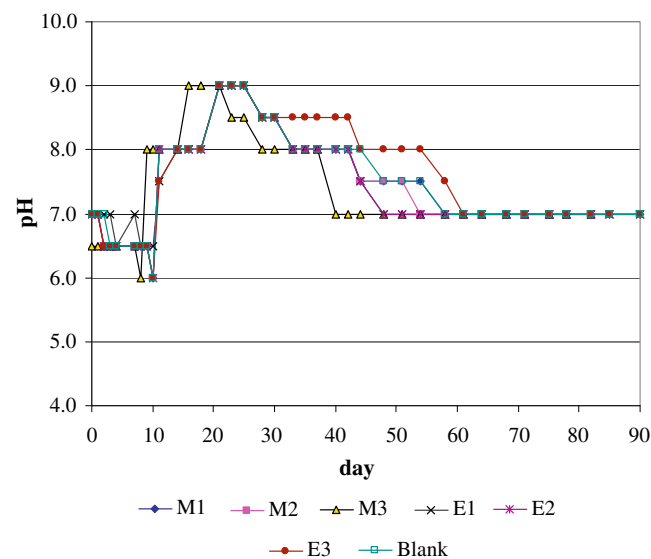


Fig. 2. pH variation with respect to time during the composting period.

The mineralization of the organic matter was responsible for the decrease of the pH at the beginning of the composting period. The organic matter of the mixture was converted to carbon dioxide and water. Furthermore, the degradation of PLA and PLA-corn occurred by depolymerisation and subsequent mineralization. In general, molecular chain break down proceeds by random hydrolytic chain scission of the ester linkages and it is accompanied by lactic acid generation (Tuominen et al., 2002). The presence of high amounts of acid lactic contributes to pH decreasing as well (Ghorpade et al., 2001). This fact could not be observed in our experiments as the pH evolution of reactor "Blank" was similar to the rest of reactors containing plastic pieces. A gradual increase of pH was observed from day 8–10, which corresponded to the release of ammonia and other basic components in the mixture

and the elimination of organic acids (Unmar and Mohee, 2008). At the end of the process, a neutral pH was observed.

Water content is an important factor affecting microbial activity in composting and ultimately the rate of degradation. Ghorpade et al. (2001) have reported that moisture content may fall 20–56%. The variation in moisture content of each reactor over time is shown in Fig. 3. As it can be seen, the initial amount of water of each reactor was 550 g and losses of water were observed in all the reactors throughout the composting period. In our case, distilled water was added to the reactors in order to maintain the optimal percentage of moisture as the Norms recommend (see Table 2).

After 90 days of composting reaction, each compost was characterised. Table 4 shows the values of final dry mass, volatile solids and nitrogen. The final content of volatile solids of each compost was slightly lower than before composting, indicating that a part of organic matter has been transformed into carbon dioxide. The reduction in volatile solids content of reactors “E” and “M” was within 5.9–7.6% and 4.7–7.8%, respectively. The reduction for reactor “Blank” had a similar behaviour (5.5%), indicating that plastic pieces did not have negative influence on the composting reaction. On the other hand, the % of organic nitrogen decreased from 1.55% to 1.19% (average values) in reactors “E” and from 1.26% to 1.20% (average values) in reactors “M”, probably due to its conversion to ammonium, nitrites and nitrates (Haug, 1993; Tchobanoglous and Kreith, 2002). The reduction of reactor “Blank” was slightly more noticeable, from 1.27% to 0.88%.

3.2. Biodisintegration calculation

Table 5 presents the exact amounts of plastic pieces before and after composting of each reactor, as well as their corresponding biodisintegration degree (D) calculated according to Eq. (1). As it can be seen, in all cases more than 50% of the initial plastic material was degraded. In particular, the biodisintegration degree of PLA-corn (“M” samples) had an average value of 79.7%, which is greater than the value obtained for PLA and foamed-PLA (average value 63.6%). This fact could be explained because of this bioplastic material contains a 10% of corn in its composition. Due to corn is a high biodegradable component, the material was more available to microorganisms and improved the degradation of the PLA fraction. These results are in accordance with those reported by Gattin et al. (2002), where PLA-starch extruded films were subjected to com-

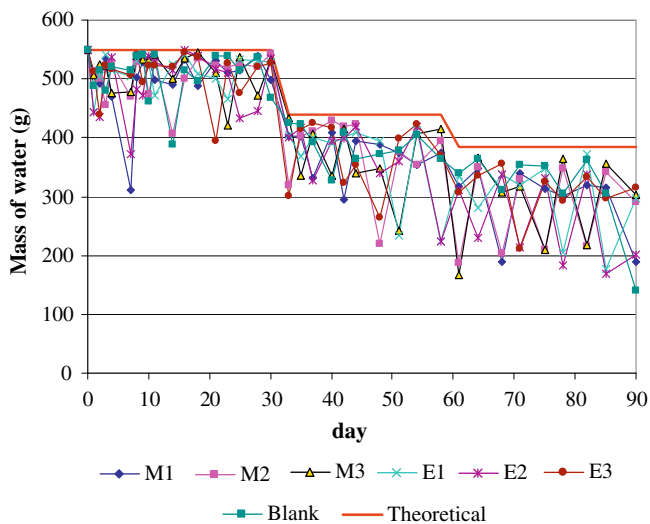


Fig. 3. Variation of the amount of water during the composting time.

Table 4

Percentages of dry mass, volatile solids, and nitrogen of the obtained composts.

Reactor	Dry mass (%)	Volatile solids (% dry mass)	Nitrogen (% dry mass)
E1	95.44	83.42	1.24
E2	91.96	81.83	1.15
E3	92.23	83.69	n.a.
M1	91.32	82.75	1.10
M2	96.36	83.83	1.30
M3	90.39	82.10	n.a.
Blank	92.29	83.42	0.88
Average	92.85	83.00	1.13

n.a.: Not analysed.

Table 5

Amounts of plastic materials before and after composting and calculated biodisintegration degree (D).

Reactor	Initial mass (g) M_i	Final mass (g) M_f	D (%)
E1	19.5044	4.8342	75.2
E2	20.2807	8.7561	56.8
E3	20.2495	8.3612	58.7
Average “E” samples			63.6
M1	20.6944	5.0345	75.7
M2	20.5288	3.4703	83.1
M3	21.0146	4.1211	80.4
Average “M” samples			79.7

posting in similar conditions for 45 days. The final mineralization percentage was 71%, which was facilitated by the presence of starch. The biodisintegration degree of PLA-corn did not depend on the shape and/or size of the manufactured pieces, as the behaviour of all the tested pieces was the same.

On the other hand, the biodisintegration degree of PLA and foamed-PLA is lower if compared with the results obtained by other authors in similar composting conditions. Tuominen et al. (2002) reported an 80% of degradation for PLA prepolymer after 90 days of composting. Kunioka et al. (2006) obtained up to 90% of degradation of PLA powder after 20 days. These differences could be attributed to the lower initial size of PLA particles (lower than 0.6 mm in both cases), which favour the degradation rate (Kunioka et al., 2006).

3.3. Validation of biodisintegration tests

In order to validate the results of the biodisintegration experiments of both “E” and “M” samples, the volatile solids decreasing (R) was calculated for each reactor according to Eq. (2). Table 6 shows the obtained values.

According to Norm ISO 20200:2004, the results of R can be considered valid as all the calculated values for the seven reactors (including “Blank” without plastic pieces) were greater than 30%. On the other hand, the variability of the biodisintegration degree (D) must be lower than 10%. As it can be seen in Table 5, the variability depends on the initial tested material. “M” samples can be considered valid as the obtained values were not divergent (75.7–83.1%). However, values for “E” samples could not be considered valid due to the differences among results were greater than 10% (values between 56.8% and 75.2%). It can be observed that reactor E1 showed a disintegration degree of 75.2, whereas reactors E2 and E3 presented similar degrees near to 57–58%. This fact can be explained taking into account the initial amounts of PLA and foamed-PLA added to each reactor (Table 7).

It can be noticed that the amount of PLA added to reactor E1 (7.9 g) was lower than the ones added to reactors E2 and E3 (8.7 and 8.9 g, respectively), being the difference of almost 1 g. This difference can be important as reactor E1 has shown a better

Table 6Values for calculating the volatile solids decreasing (*R*) of each reactor.

Reactor	m_i (g)	$(DM)_i$ (%)	$(VS)_i$ (%)	m_f (g)	$(DM)_f$ (%)	$(VS)_f$ (%)	<i>R</i> (%)
E1	1001.0	45.61	88.65	266.5	95.44	83.42	47.6
E2	1000.1	45.20	88.56	270.3	91.96	81.83	49.1
E3	1000.1	42.45	88.98	276.5	92.23	83.69	43.5
Average "E" samples							46.7
M1	998.8	41.53	89.21	268.4	91.32	82.75	45.2
M2	1001.5	43.02	87.93	267.3	96.36	83.83	43.0
M3	999.9	42.47	89.05	278.9	90.39	82.10	45.3
Average "M" samples							44.5
Blank	1000.6	42.75	88.32	293.2	92.86	83.01	41.1

Table 7

Amounts of PLA and foamed-PLA added to reactors E1, E2 and E3.

	E1		E2		E3	
	Mass (g)	%	Mass (g)	%	Mass (g)	%
PLA	7.9	40.5	8.7	42.8	8.9	43.8
Foamed-PLA	11.6	59.5	11.6	57.2	11.4	56.2
Total	19.5	100.0	20.3	100.0	20.3	100.0
Foamed-PLA/PLA	1.46		1.33		1.28	

biodisintegration degree, possibly due to its greater content of foamed-PLA in relation with the PLA amount (foamed-PLA/PLA = 1.46). In general, the addition of a chemical foaming agent to PLA during plastic pieces manufacturing generates gases (ammonia, carbon dioxide, etc.) within solidifying matrices producing highly porous structures (Macosko, 1989; Yoon and Park, 2001). The characteristics of PLA without foaming agent (texture and lower porosity) may difficult the action of microorganisms, whereas foamed-PLA, which presents a highly porous structure, can favour the microorganisms attack. These results are in accordance with those obtained by Yoon and Park (2001), who reported that highly porous PLGA (lactic/glycolic) structures may have different degradation behaviours in contrast to non porous matrices. The exchange of aqueous fluid between within and outside the sheets can take place in a more facilitated manner due to the presence of macropores, thus enhancing the biodegradation degree.

4. Conclusions

Pieces of PLA and PLA with 10% of corn (PLA-corn) of different thicknesses and shapes have been manufactured by injection and/or extrusion in a similar way to other thermoplastic materials. Sprues and cylindrical pieces of PLA-corn were obtained by injection. Sheets of PLA and PLA-corn were manufactured by extrusion. A specific foaming agent for polystyrene was compatible with PLA in extrusion tests, where foamed-PLA sheets were obtained. Nevertheless, this foaming agent was incompatible with PLA-corn in both injection and extrusion processes.

With regard to biodisintegration tests, it was found that the pieces made of PLA and PLA with foaming agent (thickness smaller than 5 mm) present an average biodisintegration degree of 63.6%. After analysing the not biodisintegrated materials, it was observed that the PLA without foaming agent is disintegrated in a lower degree than foamed-PLA. The pieces made of PLA-corn present an average biodisintegration degree of 79.7%. In this case the biodisintegration degree is neither influenced by the thickness nor the shape of the pieces.

According to all these results, it can be said that the application of PLA and PLA-corn to the manufacturing of plastic pieces has a promising future due to their wastes present a high degree of disintegration under composting conditions.

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