BIODEGRADABILITY ASSESSMENT OF ALIPHATIC POLYESTERS USING STANDARD METHODS

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Abstract

Important information concerning polymer's final fate in the environment can be achieved in biodegradation studies performed in the aquatic environment. In this context, the focus of the present work was to evaluate the biodegradability level of blends containing aliphatic polyesters using standard methods.

Blends of high density polyethylene (HDPE) and five biodegradable polymers (poly(lactic acid) (PLA), poly(ε-caprolactone) (PCL) and Mater-Bi (thermoplastic starch with PLA or PCL)) were prepared in a co-rotating twin-screw extruder, together with polyethylene modified with maleic anhydride used as compatibiliser. Biodegradation tests were carried out using the standard ISO 14851 (1999) which specifies a method for determining the biochemical oxygen demand in a closed respirometer, the standard ASTM D 5209 (1992) which specifies a method for determining the carbon dioxide evolution and the standard ASTM G 22-76 which specifies a method for determining the microbial growth of a test microorganism.

The results show that the blend containing PCL is more biodegradable than the blend containing PLA based on both microbial growth (ASTM G 22-76) and biochemical oxygen demand (ISO 14851:1999) biodegradability tests. The biodegradability of the blend containing PLA was increased by the addition of starch, in turn the same was not observed for the case of PLC. The biodegradability tests suggest that starch is more biodegradable than PLA but less than PCL.

The biodegradability of the blends evaluated in the presented study by the biochemical oxygen demand method ranged from 22 % to 48 %. Therefore the

blends may not be considered "readily biodegradable" according to the OECD standard.

Introduction

During the last decades, the demand of synthetic polymeric materials has been fairly increasing and presently they are one of the most attractive categories of materials. This success is mainly related to their properties namely, low cost, aesthetic qualities, and resistance to physical ageing and biological attack (Vert, 2005). The well known resistance to degradation of synthetic polymers, together with the growing environmental awareness and the new environmental regulations are forcing the industries to seek for more ecologically friendly materials for their products, namely in applications where they are used for a short period of time before becoming waste.

Biodegradable polymers may be derived from biological sources (corn or wood cellulose) or from petroleum sources. The best known petroleum source-derived biodegradable polymers are aliphatic polyesters or aliphatic-aromatic copolyesters. Presently, biodegradable polymers derived from renewable resources like polylactides (PLA) compete with petroleum-based biodegradable polymers. Polyolefin-starch blends are among the common materials used in the packaging industry. Blending biodegradable with non-biodegradable polymers is a method for reducing the overall production cost of the material, which offers simultaneously a way to modify both mechanical properties and biodegradation rates.

Important information concerning polymer's final fate in the environment can be achieved in biodegradation studies performed in the aquatic environment. Environmental biodegradation concerns the complete conversion of organic chemicals to inorganic products mediated by microbial processes. The degree of polymer biodegradation can be measured according to the carbon dioxide mass and/or methane evolved, oxygen consumption, degradation products (e.g., monomers) released, and polymer carbon converted into biomass (Massardier-Nageotte et al., 2006). Standard test methods have been proposed by several international organizations to assess biodegradability of polymeric materials (ASTM, ISO, OECD).

The purpose of this study was to evaluate the aerobic biodegradability of aliphatic polyesters using standard methods.

Materials and methods

Materials

Materials used in the present study are commercially available. High density polyethylene (HDPE), 2008SN60, was supplied by Total, polyethylene modified with 3.1 % (wt) maleic anhydride (PE-g-MA), Lotader 3210, was supplied by Arkema, poly(ε-caprolactone) (PCL), CAPA FB100, was supplied by Solvay, and

poly(lactic acid) (PLA), Polymer 2002D NatureWorks [a], was supplied by Novamont. Starch-based thermoplastics (TPS), Mater-Bi ®, were supplied by Novamont. Mater-Bi ® are commercially available as blends of corn starch/poly(ε -caprolactone) 30/70 (% wt), SPCL 70, corn starch/poly(lactic acid) 30/70 (% wt), SPLA 70, and corn starch/poly(Llactic acid) 50/50 (% wt), SPLA 50.

To compound the samples used in the present study (Table 1), the materials were tumble mixed and processed in a laboratory modular co-rotating twin screw extruder (Leistritz LSM 30.34) using a barrel temperature of $190^{\circ}C$, a screw speed of 100 rpm and a throughput of $3\ kg/h$. The extruded material was cooled, dried and cut in small pellets.

Table 1 Composition of the samples expressed as weight percentages

Blend	HDPE	PE-g-MA	PLA	PCL	Mater-Bi
PLA 60	30	10	60	0	0
PCL 60	30	10	0	60	0
SPLA 50	30	10	0	0	60 (50 TPS + 50 PLA)
SPLA 70	30	10	0	0	60 (30 TPS + 70 PLA)
SPCL 70	30	10	0	0	60 (30 TPS + 70 PCL)

Biodegradation tests

The aerobic biodegradation of the blends prepared in the present work was investigated using three distinct methods: microbial growth in polymeric films, biochemical oxygen demand and CO_2 evolution in the polymers previously reduced to powder.

Microbial growth test

The growth of a pure culture of Pseudomonas fluorescens was evaluated as a function of time with HDPE and all the polymer blends (Table 1) as sole carbon and energy sources. The experimental procedure was adapted from ASTM G 22-76 (the essays were carried out in liquid phase instead of solid phase). Each sample, shaped as a disc with 25 mm diameter and thickness of 0.25 mm , was decontaminated with ethanol 70 $\%(v/v)$ and placed into sterilised conical shaped 100 mL Erlenmeyer flasks containing 40 mL of R2A carbon free medium at pH 7.0. Each flask contained one disk divided into two halves and the disk's density was lower than the water density. The flasks were closed with stoppers connected to air filters and spiked with the pure culture directly from an agar plate and incubated under static conditions at room temperature ($22^{\circ}C$) during 10 weeks.

Bacterial density on the surface of the polymer, forming a biofilm, was monitored by carrying out a series of total cell counts over a period of 10 weeks. Cells were enumerated by epifluorescence microscopy after DAPI staining ($5\ min, 2\,mg/L$ final concentration) at 1000 magnification. The detailed methodology is described in Machado et al. (2007).

Biochemical oxygen demand and CO_2 evolution tests

Biodegradation tests were carried out in aqueous environment under aerobic conditions according to the standard ISO 14851:1999 (Determination of the ultimate aerobic biodegradability of plastic materials in an aqueous medium) which specifies a method for determining the biochemical oxygen demand in a closed respirometer, and to the standard ASTM D 5209-92 (Determining the aerobic biodegradation of plastic materials in the presence of municipal sewage sludge) which specifies a method for determining the carbon dioxide evolution. Polymers were reduced to powder to create a suspension of the polymer in the test system.

The Oxitop system contains an individual number of reactors consisting of glass bottles with a carbon dioxide trap (sodium hydroxide) in the headspace. The bottles are supplied with a magnetic stirrer and sealed with a cap containing an electronic pressure indicator. Biochemical oxygen demand (BOD) determinations were carried out in 510 mL bottles containing 62.5 g of the test sample, 2 mL of inoculum and 50 mL of mineral medium. The mineral medium contained $40\ mL/L$ of solution A ($28.25\ g/L\ KH_2PO_4, 146.08\ g/L\ K_2HPO_4$), $30\ mL/L$ of solution B ($3.36\ g/L\ CaCl_2.2H_2O, 28.64\ g/L\ NH_4Cl$), and $30\ mL/L$ of solution C ($3.06\ g/L\ MgSO4.7H_2O, 0.7\ g/L\ FeSO Fe_4.7H_2O, 0.4\ g/L\ ZnSO_4$). The source of inoculum was activated sludge freshly sampled from a municipal sewage treatment plant. The BOD of the inoculum was determined in blank tests performed only with mineral medium and inoculum. These values were subtracted from the BOD values of the samples to obtain exact values of the degradation activity. Test bottles were incubated at $30^{\circ}C$ in the dark with stirring for more than 28 d . Samples were tested in triplicate.

The amount of O_2 consumed in polymer's biodegradation (after correction with the blank test) was expressed as a percentage of theoretical oxygen demand (ThOD). The ThOD expressed as mass of O_2 per mass of polymer was determined by calculating the amount of O_2 necessary for aerobic mineralization of the polymer, i.e. complete oxidation of C to CO_2. The ThOD of the polymer $C_cH_hO_o$, with a relative molecular mass M_r, was calculated according to:

$$\text{ThOD} ¿ \left(\frac{31.9988}{M_r}\right) \cdot (c + 0.25 \cdot\ h - 0.5 \cdot o)$$

Carbon dioxide evolution was determined in 2 L Erlenmeyer flasks containing 3.12 g of the test sample, 15 mL of inoculum and 1248.5 mL of mineral medium.

The source of inoculum was activated sludge freshly sampled from a municipal sewage treatment plant. The mineral medium contained 1.5 mL/L of magnesium sulfate solution (22.5 $g/L\ MgSO_4 \cdot 7\,H_2O$), 1.5 mL/L of calcium chloride solution (27.5 $g/LCaCl$), 3 mL/L of phosphate buffer solution $C(8.5\ g/L\,KH_2PO_4, 21.75\ g/LK\,K_2HPO_4, 33.4\ g/L\ Na_2HPO_4 \cdot 7\,H_2O$, and 1.7 $g/LNH\,H_4Cl$), 6 mL/L ferric chloride solution (0.25 $g/L\ FeCl_3 \cdot 6\,H_2O$), and 1.5 mL/L ammonium sulfate solution (40 $g/L(NH_4)_2SO_4$). The flasks were placed on top of a magnetic stirrer and air was passed through the liquid at a flow rate of 0.3 mL/min. The inlet air to the flasks was passed through a series of bottles containing 700 ml of 10 M NaOH and 700 ml of 0.025 $MBa¿$ to trap CO_2 present
in the air. The exhaust gas from the flasks were passed through 3 bottles containing each 700 ml of 0.025 $MBa¿$ to trap the CO_2. The traps were replaced at frequent time intervals. The quantity of CO_2 produced was determined by titration of the excess $Ba¿$ by a standard solution of hydrochloric acid $(HCl, 1\,M)$ using phenolphthalein as indicator. The amount of CO_2 formed in polymer's biodegradation (after correction with the blank test) was expressed as a percentage of the theoretical CO_2 production $(ThCO_2)$. The $ThCO_2$ expressed as mass of CO_2 per mass of polymer was determined by calculating the amount of O_2 necessary for aerobic mineralization of the polymer, i.e. complete oxidation of C to CO_2. The $ThCO_2$ expressed as mass of CO_2 per mass of polymer was calculated from the carbon content of the polymer (Y) assuming complete oxidation of the carbon to carbon dioxide according to:

$$ThCO_2 = \left(\frac{44}{12}\right) \cdot Y$$

Characterization methods

The composition of all samples was determined by elementary analysis on a LECO CHNS-932. The samples' chemical formulas, calculated with C, H and O mass fractions of C, H and O , are the following:

PLA $60 - C_3H_5O$; PCL $60 - C_5H_{10}O$; SPLA 50 C_5H_9O; SPLA $70 - C_3H_4O$; SPCL $50 - C_3H_7O$.

The biodegradation of the polymers and blends was followed by FTIR spectroscopy (Perkin Elmer 1720 spectrometer). Measurements were recorded in the range of 4400 400 cm^{-1}, using 16 scans with a resolution of 4 cm^{-1}. Thin films of the initial materials and the residues after biodegradation were prepared by compression-moulding and analyzed directly using a solid film support.

Results and Discussion

Biodegradation tests

Blends were incubated in the presence of Pseudomonas fluorescens, under defined experimental conditions, and an increase of cell counts along time was observed in the biofilm formed on the surfaces of all blends (Table 2). HDPE was used as a negative control.

Table 2 Ratio between the biofilm bacteria number on polymeric samples quantified in the 10^{th} week and in the 2^{nd} week of experiment lifetime. Values listed in the table are the average ± 95 % confidence interval

HDPE	PLA 60	PCL 60	SPLA 50	SPLA 70	SPCL 50
1.75 ± 0.14	2.71 ± 0.31	3.49 ± 0.80	4.59 ± 1.21	2.98 ± 0.83	3.29 ± 0.27

HDPE shows the lowest cell count ratio while SPLA 50 exhibits the highest, according to data presented in Table 2. There could be two explanations for the observed results: on the one hand HDPE has a lower surface energy thus is a less favorable material to cell adhesion (Tsibouklis et al., 1999), on the other hand resistance to microbial attack is lower for SPLA 50 due to the presence of starch and PLA (Gross and Kalra, 2002).

The cell count ratio of PCL 60 is higher than the one of PLA 60 suggesting that PCL is less resistant to bacterial attack than PLA. Massardier-Nageotte et al. (2006) reported that PCL and a SPCL blend seemed to be rather biodegradable in opposition to PLA, in a biodegradability study carried out under aerobic conditions and in liquid phase.

The effect of increasing amounts of starch-based thermoplastic (0 %, 18 % and 30 %) on the biodegradation's potential of PLA blends are presented in Table 2. The present study indicates that the ratio between bacterial counts obtained after ten and two weeks of experiment lifetime is not significantly different in the cases of the blends containing 0 % (PLA 60) and 18 % (SPLA 70) of starch but increases significantly in the case of 30 % (SPLA 50) (t-test). The results suggest that the amount of starch might have been too low or simply not available at the polymer's surface for bacterial growth in the case of the blend containing 18 % starch (SPLA 70). At 30 % (SPLA50), starch decreased the resistance of the blend to bacterial attack and promoted microbial growth. This result might be explained by the physic-chemical properties of starch, namely crystallinity and hydrophobicity (Thakore et al., 2001). Usually, the biodegradation occurs preferably in the amorphous regions of the polymer which have higher mobility of the chains and therefore are more accessible to the microorganisms. Starch, being less crystalline than PLA, is more prone to microbial attack. Additionally, its hydrophilic nature characterized by a higher number of hydroxyl groups in the structure as compared to the one present in PLA promotes swelling in the culture medium enhancing biodegradation. The effect of 18 % starch-based thermoplastic on the biodegradation's potential of the PCL blend (SPCL 70) was not significant (Table 2), as in the case of PLA, and the same explanation for the observed result might be feasible.

The biochemical oxygen demand values of the polymers (HDPE, PCL and PLA) and blends (PCL 60, PLA 60, SPLA 50, SPLA 70, SPCL 70) determined in a closed respirometer (ISO 14851:1999) are presented in Figure 1. The values were normalized by the carbon content of each sample. These results have shown that PCL presents a higher O_2 consumption than PLA. As expected, the blend containing PCL was also more biodegradable than the one containing PLA. The HDPE's O_2 consumption was negligible when compared to the other materials. The biodegradability of the PLA blend was increased by the addition of increasing amounts of starch, in turn the same was not observed for the case of the PLC blend. The biodegradability test suggests that starch is more biodegradable than PLA but less than PCL.

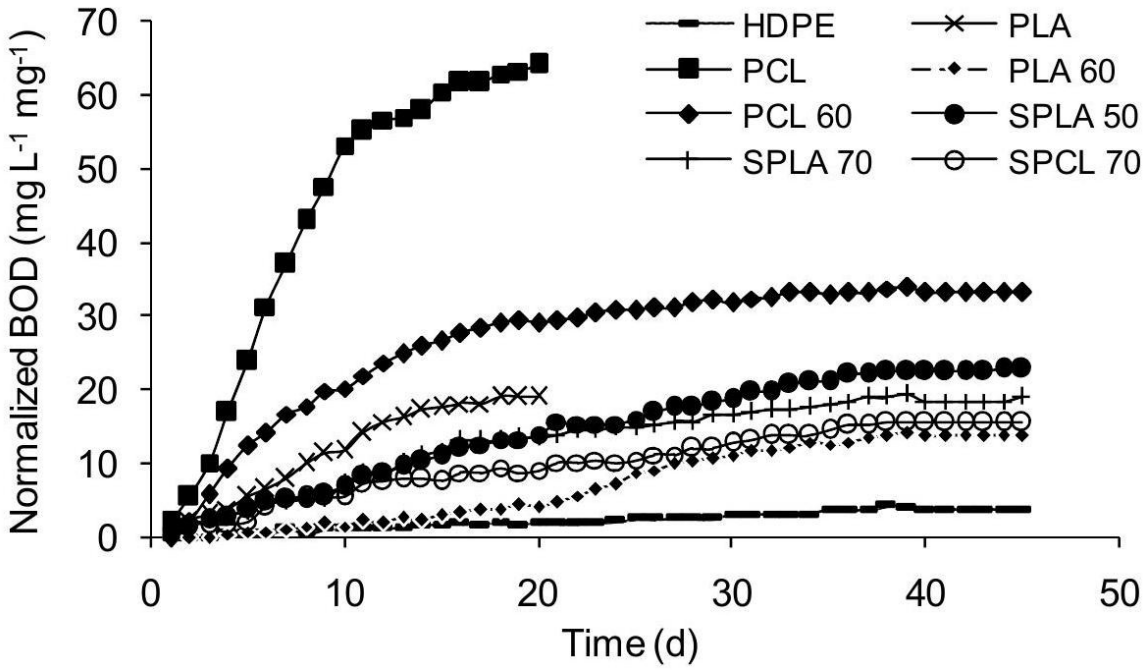

Figure 1 Normalized biochemical oxygen demand values expressed as mg/L of O_2 divided by mg of polymeric C of each sample.

The biodegradability of the polymers (tests carried out during 20 d) and blends (tests carried out during 45 d) studied in the present work was expressed as the amount of O_2 consumed during sample biodegradation divided by their theoretical oxygen demand (ThOD). Biodegradation test systems based on BOD and CO2 measurements do not differentiate between the oxidized part of the test material and the part incorporated into biomass which can be expressed by the biomass yield coefficient. Considering an average biomass, yield on polymeric carbon of 0.61 , expressed as a C molar fraction, a new ThOD was calculated.

Table 3 Biodegradation (BOD/ThOD) of polymers (20 d essay) and blends (45 d essay) determined by the biochemical oxygen demand method

Sample	BOD (mg/L O_2)	[1] ThOD I (mg/LO_2)	[2] ThOD II (mg/L O_2)	BOD/ThOD (without growth)	BOD/ThOD (with growth)
HDPE	193	4477	2860	0.04	0.07
PCL	2523	2527	1237	1.00	2.04
PLA	597	1601	570	0.37	1.05
PLA 60	547	2527	1223	0.22	0.45
PCL 60	1486	3126	1735	0.48	0.86
SPLA 50	1024	3050	1637	0.34	0.63
SPLA 70	732	2916	1079	0.25	0.68
SPCL 70	655	2766	1520	0.24	0.43

[1] microbial growth was not considered; [2] biomass yield on polymer expressed as a C molar fraction was considered to be 0.61

The analysis of the data presented in Table 3 revealed that the BOD/ThOD ratio is higher, in the case where microbial growth was considered in the calculation of the ThOD, than in the one neglecting this process, as expected. In the case of PCL the BOD/ThOD ratio is higher than 1. One possible explanation for this result is that oxygen was consumed both for the mineralization of polymeric carbon and for the oxidation of ammonium to nitrite/nitrate (nitrification) used as a N source for microbial metabolism (Reuschenbach et al., 2003). To confirm this hypothesis, additional respirometric experiments are being conducted in the presence of allyltheorea, an inhibitor of the nitrification process. The Organization for Economic Co-operation and Development (OECD) guidelines established that a test substance is regarded as "readily biodegradable" if the degree of biodegradation based on dissolved organic carbon removal is higher than 70 % (OECD 1992). In the case of BOD determination or CO_2 production, 60 % of the theoretical values have to be reached. This removal is required to occur in a specific assay with the test material as the sole carbon source, and within 10 d after the initial lag phase. According to the results obtained in the present study, only PCL may be considered "readily biodegradable" according to the OECD standard, presenting an 83 % removal within 10 d. A removal of 100 % for PLA, 86 % for PCL 60, 63% for SPLA 50, and 68% for SPLA 70 was obtained in the case where microbial growth was considered in the calculation of ThOD. However, these results do not comply with the definition of "readily

biodegradable" established by the OECD guidelines. If a chemical does not pass the "ready"-level test, either degradation starts too late or it occurs too slowly. The results from the O_2 consumption test (Figure 1) seem to indicate that biodegradation of the polymeric blends is a slow process.

The CO_2 evolution method was used to evaluate the biodegradability of the blends SPLA 50 and SPLA 70. These results have shown that the blends SPLA 50 and SPLA 70 present approximately the same biodegradation, respectively 41 % and 43 %.

Comparison between the different methods

The results of the biodegradation tests, microbial growth test (ASTM G 22-76) and biochemical oxygen demand test (ISO 14851:1999), presented above demonstrated that the blend PCL 60 is more biodegradable that the PLA 60 blend. These results are consistent with previous studies (Massardier-Nageotte et al., 2006). As expected, it was found that the PCL 60 blend presented the highest biodegradation in the biochemical oxygen demand test. Conversely, the SPLA 50 blend performed better in the microbial growth test. This last result was not expected since previous studies (Massardier-Nageotte et al., 2006) and our own results from the biochemical oxygen demand test indicated that PCL is more biodegradable than PLA. One possible explanation is the availability of polymeric carbon to microbial degradation, which might be considerably different in both methods. In the biochemical oxygen demand test the polymer blend was reduced to fine powder and all the components were equally accessible to microbial attack. In turn, in the microbial growth test polymeric films were used where only the components present on the surface of the films were available to biodegradation. Therefore, results from biodegradation testes performed with polymeric films should be interpreted with caution since the availability of the components of the blend might play a key role in the biodegradation of the blend.

The extent of biodegradation of the blends SPLA 50 and SPLA 70 determined by the CO_2 evolution method was slightly higher than the one obtained by the biochemical oxygen demand method. One possible explanation is that an error in CO_2 determination in the scrubbing bottles occurred due to the fact the bottles were periodically disconnected promoting entry of air, which contains CO_2 into the system.

FTIR spectroscopy

Figures 2 to 4 show the FTIR spectra of the main components of the blends under study in this work, i.e., HDPE, PLA and PCL. Each figure contains two spectra, corresponding to initial and biodegraded material (biochemical oxygen demand method). While in the case of HDPE (Figure 2) no significant changes could be detected after biodegradation, major changes occurred for PLA (Figure 3) and PCL (Figure 4). FTIR spectra of PLA at 0 d and after 20 d of biodegradation are shown in Figure 3. Transmittance data on a common scale showed that all peaks

decreased in size after biodegradation. The reduction in the CH -assymetric and CH -symmetric stretches at $2920\ cm^{-1}$ and $2850\ cm^{-1}$, respectively, indicated a reduction in the molecular weight of the PLA. The decrease of peaks related to carbonyls ($1800\ cm^{-1}$ and $1700\ cm^{-1}$) and ethers ($1100\ cm^{-1}$) indicated chain scission. A reduction of the peak at $1460\ cm^{-1}$ was associated with decrease of CH_3 side groups. Changes occurred during biodegradation of PCL are depicted in Figure 4. A dramatic change in the polymer backbone took place during 20 d of biodegradation. A reduction of peaks related with CH bonds ($3000-2800\ cm^{-1}$), carbonyl ($1800\ cm^{-1}$ and $1700\ cm^{-1}$) and ethers ($1100\ cm^{-1}$) indicated chain scission and as a consequence a reduction of molecular weight.

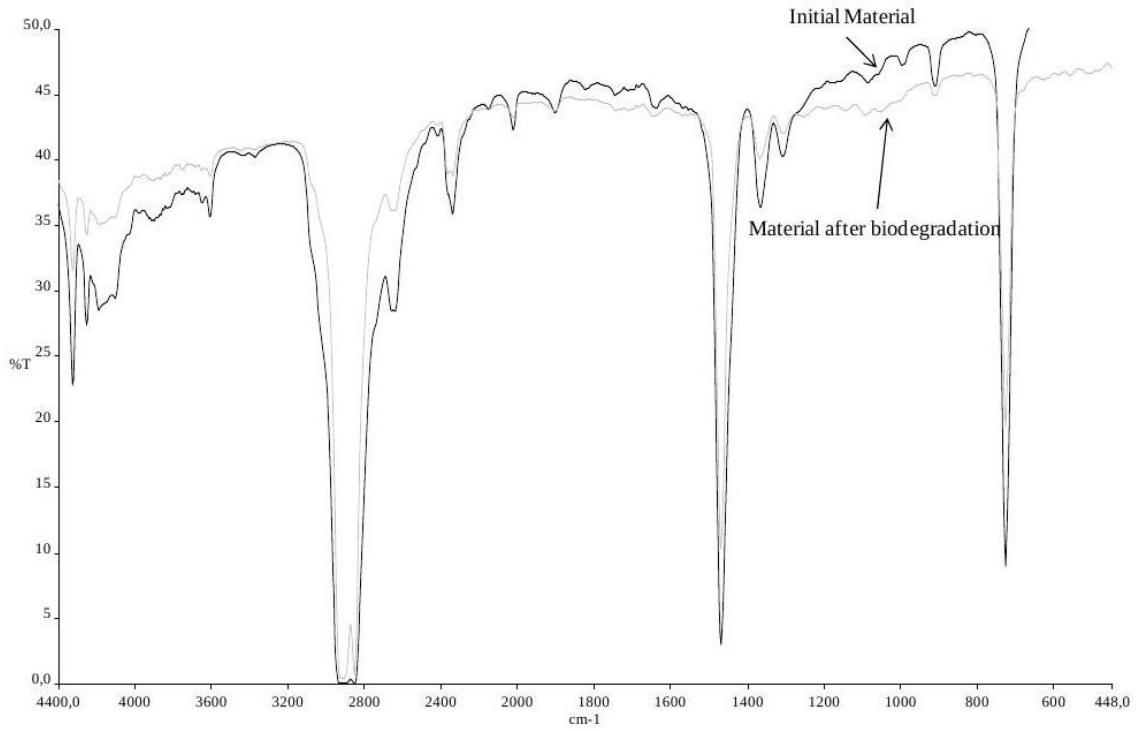

Figure 2 FTIR spectra of HDPE.

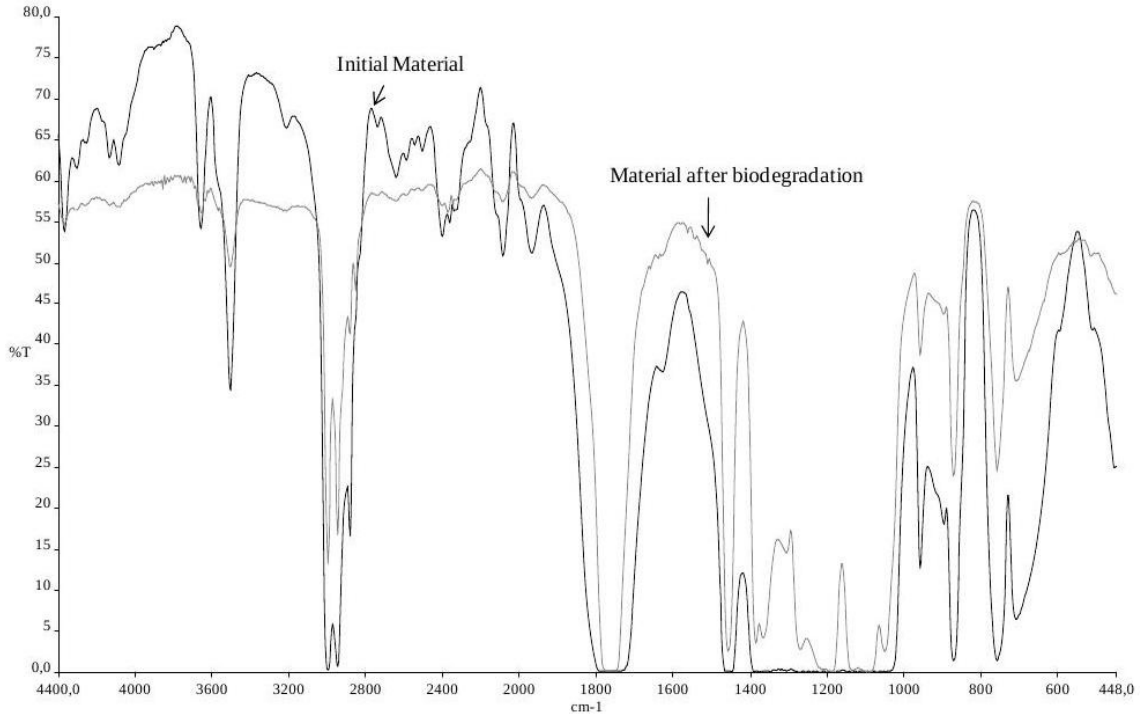

Figure 3 FTIR spectra of PLA.

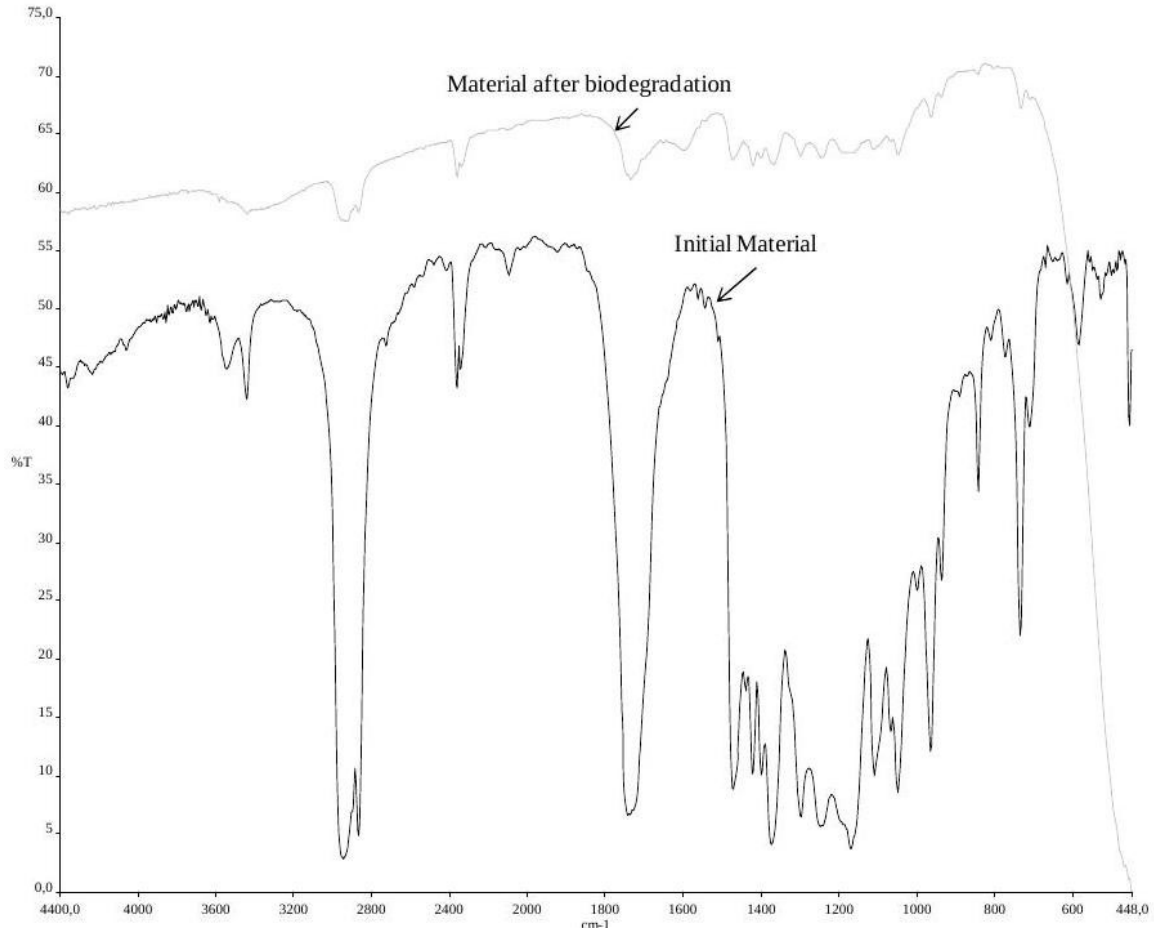

Figure 4 FTIR spectra of PCL.

Figures 5 to 9 show the FTIR spectra of all blends that were biodegraded using the biochemical oxygen demand method. Figure 5 shows the spectra of the blend PLA 60, a significant reduction in all peaks could be observed after 45 d of biodegradation. As it was seen for PLA and as expected, this blend followed the behavior of PLA. After 45 d , all peaks related to PLA decreased, which confirm a significant chain scission and as a result a decrease of molecular weight. However, the peaks related with HDPE, mainly the peak at 720 cm^{-1} are still present indicating that almost no changes occurred in this polymer.

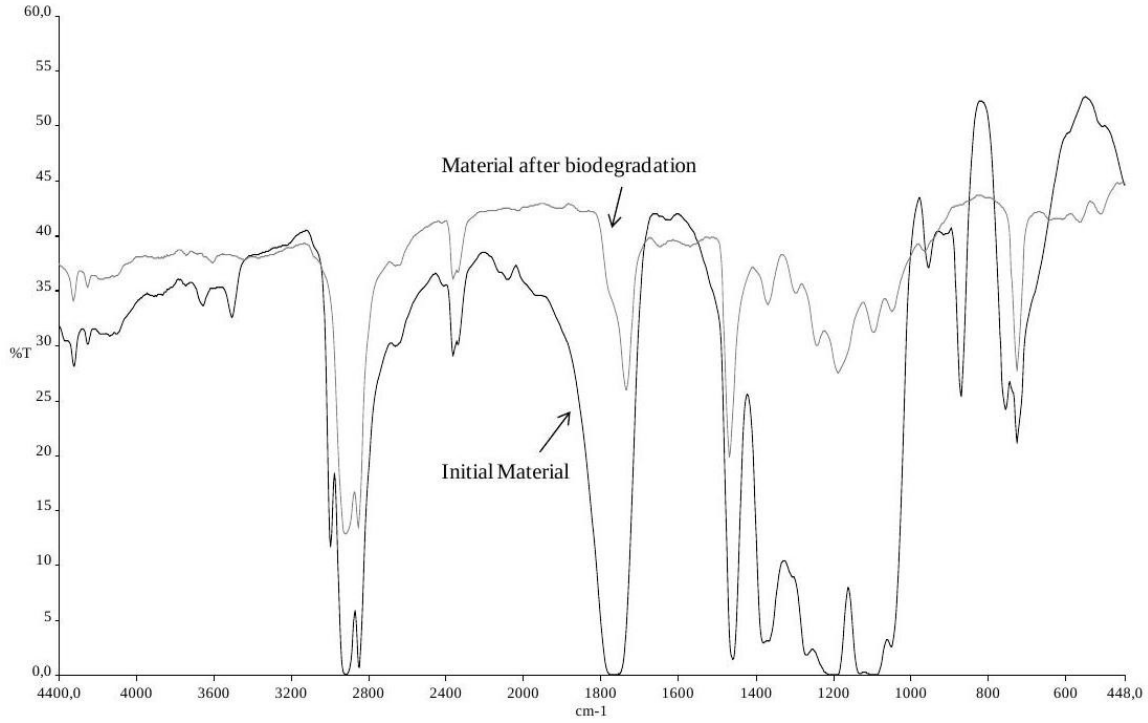

Figure 5 FTIR spectra of the blend PLA 60.

The spectra of the blend PCL 60 is depicted in Figure 6. A significant reduction of all peaks related with PCL could be detected, which indicates chain scission and a consequent decrease of the polymer molecular weight.

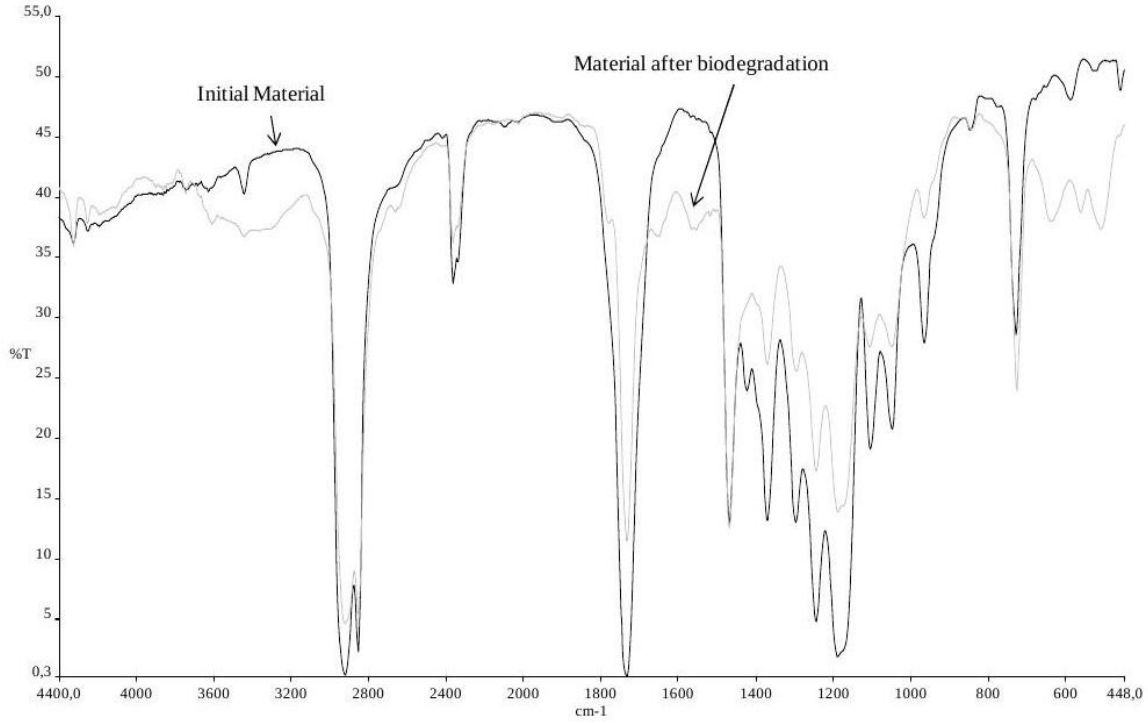

Figure 6 FTIR spectra of the blend PCL 60.

Figures 7 to 9 show the spectra of blends containing HDPE, starch and PLA or PCL. Comparing the spectra with those blends containing only PLA or PCL, it was possible to observe the presence of the OH peaks, between $3600\ cm^{-1}$ and $3200\ cm^{-1}$, related with the presence of starch. As expected, the spectra of all blends showed a significant decrease of all peaks after biodegradation with the exception of the peak at $720\ cm^{-1}$, which is related to the CH_2 of HDPE. Beside the decrease of intensity of the peaks stated above, corresponding to PLA and PCL, a decrease of the OH peak also occured indicating that bond scission took place in both polymers (PLA or PCL and starch). From the intensity of the peaks (Figures 7 and 8) it is possible to point out, in a qualitative way, that the extent of biodegradation was higher for the blend with high starch content (SPLA 50). Blends with the same amount of starch (Figure 8 and 9) showed a similar decrease of the intensity of the peaks in all spectrum range, i.e., similar biodegradation extension is achieved with PLA or PCL in presence of the same amount of starch.

The FTIR spectra of the blends SPLA 50 and SPLA 70 biodegraded using the CO_2 evolution method is similar to the ones shown in Figures 7 and 8. A significant decrease of all peaks related to PLA and starch were observed.

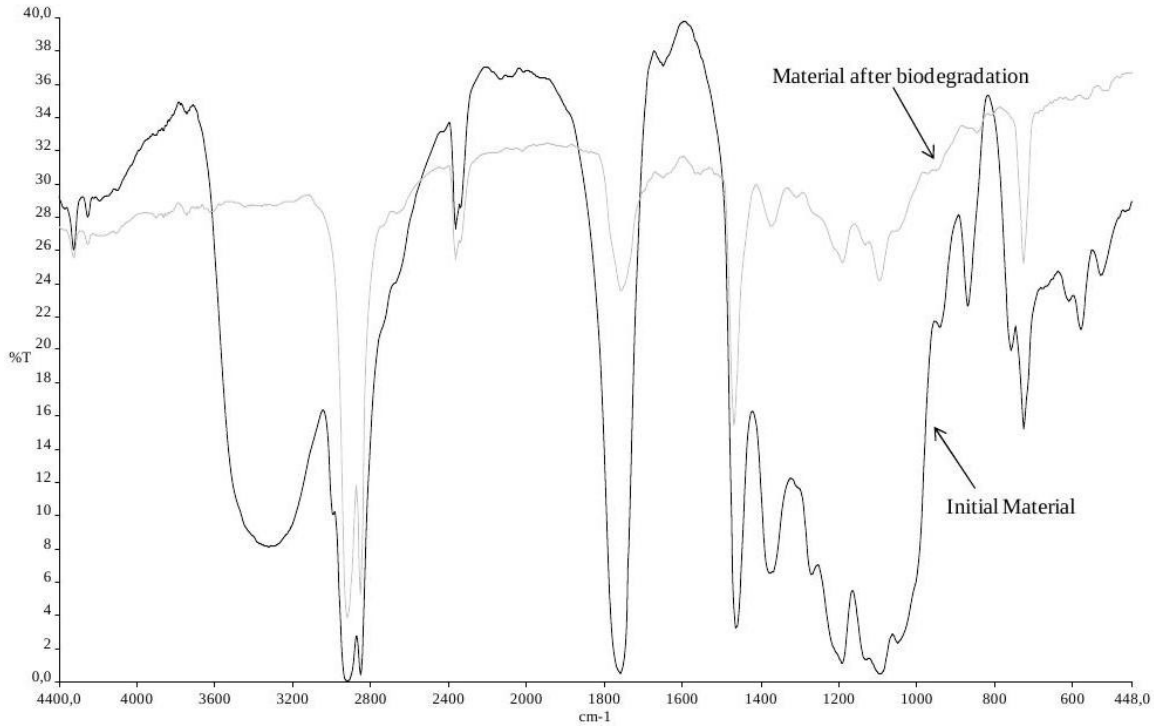

Figure 7 FTIR spectra of the blend SPLA 50, containing starch (30 %) and PLA.

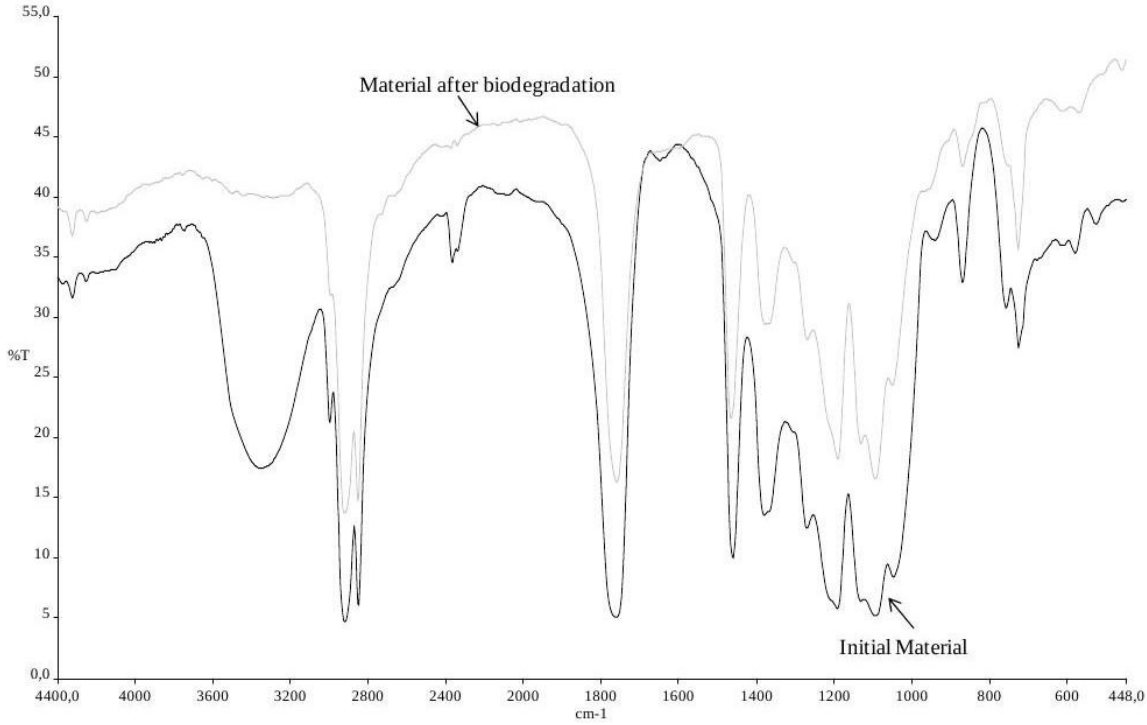

Figure 8 FTIR spectra of the blend SPLA 70, containing starch (18 %) and PLA.

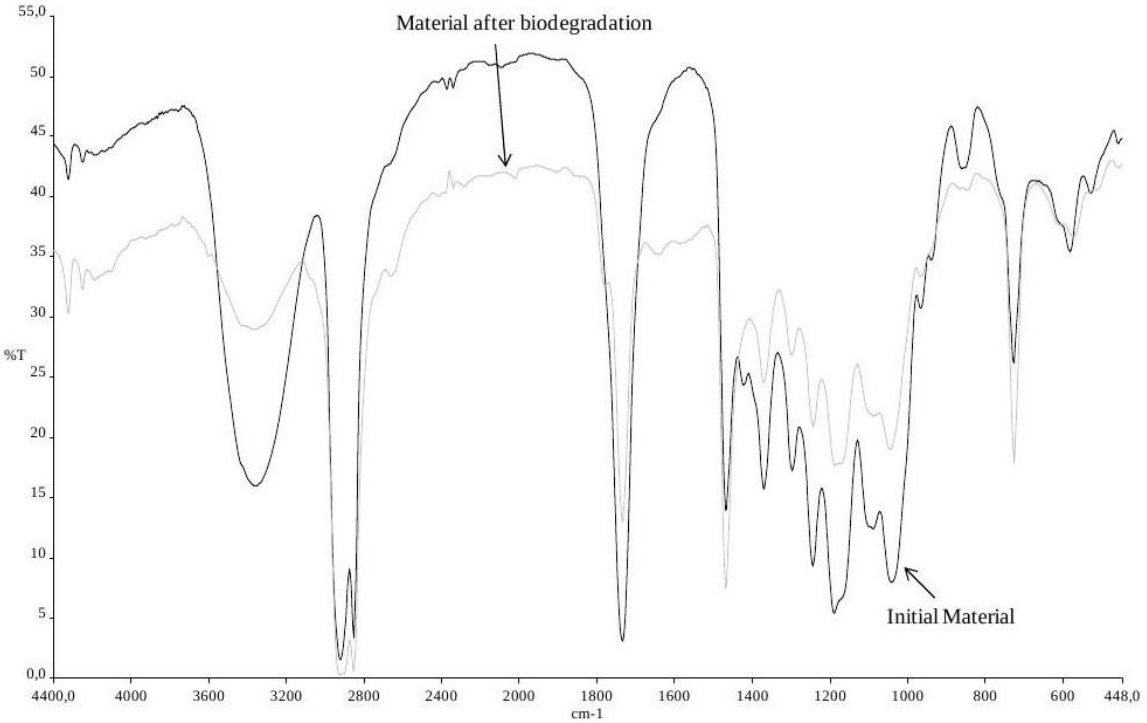

Figure 9 FTIR spectra of the blend SPCL 70, containing starch (18 %) and PLA.

Conclusions

The results obtained have shown that the blend containing PCL is more biodegradable than the blend containing PLA based on both the microbial growth (ASTM G 22-76) and biochemical oxygen demand (ISO 14851:1999) biodegradability tests. The biodegradability of the blend containing PLA was increased by the addition of starch, in turn the same was not observed for the case of PLC. The biodegradability tests suggest that starch is more biodegradable than PLA but less than PCL.

The biodegradability of the blends evaluated in the presented study by the biochemical oxygen demand method ranged from 22 % to 48 %. Therefore they may not be considered "readily biodegradable" according to the OECD standard.

The qualitative results of FTIR spectroscopy of initial and biodegraded polymeric blends are in agreement with the ones obtained in the standard biodegradability tests.

Biodegradability of fine grinded polymeric blends was tested using the biochemical oxygen demand and CO_2 evolution tests. It is important to point out that the surface area of the polymeric material available to microbial attack was increased considerably compared to the film samples. Thus, an acceleration factor was introduced in the tests compared to real environment.

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