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Production and biodegradability of a bio-based polylactide from corn

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Abstract

The proliferation of petrochemical plastic waste poses a threat to environmental quality and animal health. In this context, an alternative to traditional petrochemical plastics could be biodegradable bioplastics. In this study, a bioplastic based on polylactic acid (PLA) type of polyester was synthesized from local agricultural biomass. The objective is to propose a biodegradable plastic as an alternative to help reduce petrochemical plastic waste. The methodology for synthesizing the bioplastic from local corn biomass required several steps, ranging from corn germination to the polymerization of lactic acid. The natural germination method was used to obtain glucose. The glucose was then metabolized by the lactic acid bacterial strain *Pediococcus acidilactici* LabRcJ-10, leading to the formation of crude lactic acid. Liquid-liquid extraction was performed using diethyl ether. The bioplastic was synthesized with a yield of 7.87%, linked to the low growth of *Lactobacillus* in the substrate. FTIR analysis revealed an intense peak at 1748 cm^{-1}, characteristic of the C=O stretching vibration present in the esters of polylactic acid (PLA). The material also exhibited a low solubility index but a relatively high moisture content. The bioplastic degradation test, conducted by burial in three different types of matrices, revealed significant biodegradability under aerobic conditions, favored by the progressive enrichment of the matrix with microorganisms.

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1. Introduction

Côte d'Ivoire has experienced significant demographic growth since its independence, with Abidjan as the main urban center [1]. This population expansion has led to a notable increase in solid waste production, particularly from petrochemical plastics. Petrochemical plastic waste is known for being non-biodegradable and having a negative environmental impact [2]. According to the Ivorian Ministry of the Environment and Sustainable Development, the country generates more than 50,000 tons of plastic waste, which is released into the environment after use [3].

In response to this growing challenge, the Ivorian government adopted a decree in 2013 prohibiting the commercialization of petrochemical plastic packaging and encouraged the use of biodegradable plastics, also known as bioplastics [4]. Bioplastics can be obtained from bio-sourced polymers derived from conventional or non-conventional bio-resources. The synthesis of biodegradable plastic materials represents a promising solution to the problem posed by petrochemical plastics. The large-scale availability of this type of plastic could significantly reduce the proliferation of petrochemical plastic waste and thus contribute to the sustainable preservation of the environment and human health [5]. Bioplastics can be produced from conventional or non-conventional bio-resources, or as composites including modified or combined substances. In recent years, notable advances have been made in research on biodegradable plastics [5–9]. However, in developing countries like Côte d'Ivoire, their adoption remains limited, mainly due to their high cost. It therefore seems necessary in such a context to seek endogenous solutions to make this type of material more accessible, given the abundance of the required bio-resources, such as starches and sugars from tubers and cereals. Polylactic acid (PLA) is a bio-based, biodegradable thermoplastic polyester that can be obtained from lactic acid derived from agricultural sources, through bioconversion and polymerization processes [10]. It is a rigid material with greater tensile strength and low toughness compared to starch-based bioplastics. In the packaging industry, it represents a good alternative to petrochemical plastics, but its price remains relatively high compared to the latter [11].

The specific objective of this study is to synthesize polylactic acid (PLA) from its monomer derived from local biomass and to evaluate its biodegradability.

2. Materials and Methods

2.1. Study biomass

Grains of the corn variety *Zea mays SC719* were used as precursor bio-resources in the synthesis of the bioplastic for this study.

Figure 1. Grains of the corn variety *Zea mays SC719*.

The grains were collected in Barbié, a village located 19 kilometers from the city of Abengourou, in the Eastern region of Côte d'Ivoire. This biomass was chosen due to its abundance and the low interest from local populations in this variety, attributed to its relatively long maturity cycle. The dry corn cobs were harvested and transported directly to the laboratory. The corn cobs were then shelled to obtain the corn grains. The grains were sorted to retain only those of good quality.

2.2. Germination of corn grains

This step involves digesting the starch contained in the corn grains into glucose through the germination process. Indeed, to meet the energy needs of the young plant, starch is naturally hydrolyzed into glucose by the action of amylase, an enzyme present in the grains. A quantity of 500 g of corn grains was placed in a crystallizing dish, and distilled water was added in a 1:2 (V/V) ratio. The mixture was kept at ambient temperature for 10 hours to initiate the germination process. The grains, at the beginning of germination, were removed from the crystallizing dish and placed on a plant fiber sack, which had been previously sprinkled with distilled water. The corn grains were left to rest for 5 days at ambient temperature. Germination began on the 2nd day and was manually stopped on the 5th day [12]. The degermed grains were dried at ambient temperature for 48 hours and then ground using a blender (Silver Crest 5500, France) to obtain a corn powder. During the germination period, a 10 g sample of germinating corn grains was taken daily to monitor the glucose content.

2.3. Monitoring glucose formation

In a 100 mL beaker, 1 g of powder was dissolved in 50 mL of distilled water and homogenized. Then, 150 µL of this solution were transferred to a test tube, followed by the addition of 300 µL of 3,5-dinitrosalicylic acid (DNS). The resulting mixture developed a yellowish color. The tube was placed in a water bath at 100°C for 5 minutes. An orange color subsequently appeared, and the tube was left to stand for 15 minutes at ambient temperature before adding 2 mL of distilled water. The optical density of the resulting solution was read at 540 nm using a spectrophotometer (UV-VIS Biobase, China). The residual concentrations were determined using a calibration curve established by diluting commercial glucose in distilled water.

2.4. Production of lactic acid

The action of microorganisms is a crucial step for converting glucose into lactic acid, the essential monomer for producing polylactic acid (PLA) [13]. The lactic acid bacterial strain *Pediococcus acidilactici* LabRcJ-10 was provided by the Laboratory of Food Biotechnology and Microbiology of NANGUI ABROGOUA University (Côte d'Ivoire). This strain was isolated from tchapalo (a traditional sorghum-based beer) and identified using molecular methods by Attchelouwa et al. [14]. It can metabolize carbohydrates, such as glucose, primarily into lactic acid via the glycolytic pathway [15]. Prior to use, the lactic acid bacterial strain was cultured in MRS (De Man, Rogosa and Sharpe) broth at 30 °C for 48 hours. A quantity of 350 g of glucose-rich corn powder was placed in an Erlenmeyer flask, and 700 mL of distilled water was added. The mixture was maintained at 80 °C in a water bath (Mermmet WNB 22, Germany) for 30 minutes to promote glucose release. The mixture was then cooled and filtered through a 100 µm sieve to obtain a final glucose-rich solution. Subsequently, a 200 mL quantity of this solution was placed in a 250 mL flask, followed by the addition of a 1 mL aliquot of the *Pediococcus acidilactici* LabRcJ-10 culture. The flask was incubated at ambient temperature for 75 hours with agitation using an orbital shaker (Biobase SK-O330-Pro, China). During the incubation, 5 mL samples were taken at 15-hour intervals for pH measurement and titratable acidity determination. The

experiment was repeated twice independently. pH was measured using a pH meter (PHS-550, Ningbo, China). Titratable acidity was determined by titrating 5 mL of each sample with a 0.1N sodium hydroxide solution after first adding 2-3 drops of 1% phenolphthalein. The endpoint of the titration was marked by a persistent pale pink color. The acidity level was expressed as a percentage and calculated using the following formula:

$$P_{AL}(\%) = \frac{V_{\text{NaOH}} \times N_{\text{NaOH}} \times 0.09}{V_{PE}} \times 100 \quad (1)$$

Where:

P_{AL} : Percentage of lactic acid

V_{PE} : Volume of the test sample

N_{NaOH} : Normality of the base

V_{NaOH} : Volume of NaOH at equivalence

After 75 hours of incubation, the mixture was centrifuged at 6000 rpm for 15 minutes using a centrifuge (Bioblock Scientific 2K15, USA), and the supernatant containing the lactic acid was separated from the pellet. The collected solution was concentrated in a rotary evaporator (Heidolph Hei-VAP Silver, Germany) and acidified by adding sulfuric acid to hydrolyze calcium lactate into lactic acid. The solution was then subjected to liquid-liquid extraction with ethyl acetate in a 1:2 ratio, following the method described by Demmelmayer et al. [16] with some modifications. The mixture was stirred for 1 hour and left to settle for 10 hours for phase separation. The organic phase was then recovered, and the residual solvent was evaporated using a rotary evaporator at 80°C under a pressure of 153 mbar to obtain the lactic acid.

2.5. Obtaining the bioplastic via polymerization

The conversion of lactic acid to lactide was carried out according to the method proposed by Ghadamyaria et al. [17]. The lactide was then polymerized into polylactide following the method of Muabu et al. [18] with some modifications. A mixture of lactide and toluene in a 1:4 (w/v) ratio was heated under reflux at 120°C for 120 minutes. During heating, tin(II) octoate ($Sn(C_7H_{15}COO)_2$) at 6% relative to lactide and 2 mL of 0.01 M benzyl alcohol were added. After cooling, polymerization was stopped by adding 5 mL of 0.01 M methanol. The mixture was placed in a beaker, and n-hexane was added in a 1:2 ratio. After stirring, the polylactic acid produced precipitated and was recovered by heating in an oven (Memmert UN55, Germany) at 80°C for 3 hours.

2.6. Physicochemical characterization

2.6.1. Yield

The yield of bioplastic was determined relative to the glucose-rich corn powder using the following formula:

$$\text{Yield}(\%) = \frac{m_B}{350} \times 100 \quad (2)$$

Where m_B is the bioplastic mass after its production.

2.6.2. Moisture content

Into a crucible of mass m_0, a quantity of bioplastic is introduced and the total mass m_1 is measured at room temperature. The assembly is dried at 105°C in an oven for 8 hours. After cooling, the mass of the assembly is recorded as m_2. The moisture content (H) is calculated using the following formula:

$$H(\%) = \frac{m_1 - m_0}{m_2 - m_0} \times 100 \quad (3)$$

2.6.3. Water absorption

To determine the water absorption of the PLA-type bioplastic, a dry sample of mass m_s of the material was obtained after 8 hours of drying in an oven at 105°C. After cooling, the dry sample was immersed in distilled water for 24 hours to determine the water-saturated mass m_{sat}. The water absorption capacity or swelling was determined as follows:

$$\text{Abs}(\%) = \frac{m_{\text{sat}} - m_s}{m_s} \times 100 \quad (4)$$

2.6.4. Water solubility

A bioplastic sample of mass m is dried in an oven at 105°C for 8 hours, then immersed in a beaker containing 50 ml of distilled water. The setup is kept under agitation at 450 rpm using an orbital shaker ((Biobase SK-O330-Pro, China). The resulting solution is gravity-filtered through a Whatman No. 4 filter paper with a known initial mass m0. The residual bioplastic is removed, and the filter paper is dried for 8 hours. After cooling, the mass m1 of the filter paper is measured.The experiment was carried out in triplicate and the solubility determined as follows:

$$\text{Solubility}(\%) = \frac{m_1 - m_0}{m} \times 100 \quad (5)$$

2.6.5. FTIR analysis

The surface functional groups of the PLA-type bioplastic were determined by Fourier transform infrared spectrophotometry using the Agilent Cary 630 FTIR spectrometer (Santa Clara, California, USA), equipped with an ATR module. The analysis was performed in the wavenumber range of 4000 to 500 cm^{-1}.

2.7. Biodegradability

2.7.1. Burial matrices

Three types of soils were selected as burial matrices: composted soil, normal soil, and sterile soil.

Composted soil: Collected from a cattle farm, dried for 72 hours at ambient temperature, crushed, and sieved to achieve a particle size ≤ 2 mm.

Normal soil: Collected from the botanical forest of NANGUI ABROGOUA University, dried for 72 hours at ambient temperature, and sieved to achieve a particle size ≤ 2 mm.

Sterile soil (control soil): The sterile soil was obtained by calcining the normal soil in a muffle furnace at 500°C for 4 hours and sieving it to achieve a particle size ≤ 2 mm.

The organic matter content, pH, moisture content, and water absorption rate of the three matrices were determined.

Figure 2. Photograph of the different soil types

2.7.2. Biodegradability assessment

Biodegradability was assessed by introducing bioplastic into 100 g of each matrix contained in a flask. A bioplastic sample of initial mass m_0 was wrapped in a fine plastic net of known mass and then placed in each soil. The biodegradability evaluation was conducted under both aerobic and anaerobic conditions at room temperature. During the 100-day burial period, the sample, along with the net, was carefully removed every 10 days and then dried for 1 hour. The assembly was then weighed again, and the mass was recorded as m. By subtracting the mass of the net, the residual mass m_1 of the bioplastic was determined. The biodegradation rate or mass loss (%) of the bioplastic was calculated using the following equation:

$$\text{Biodegradation}(\%) = \frac{m_0 - m_1}{m_0} \times 100 \qquad (6)$$

2.8. Data processing

Statistical analysis, including the calculation of means and standard deviations from triplicate measurements, was performed using Microsoft Office Excel 2016 Professional.

3. Results and Discussion

3.1. Lactic acid production

During the 5-day germination period, glucose production was monitored, and the results are presented in Figure 3. Analysis of this figure shows that glucose production via the germination process was successful. The quantity of glucose produced increased until germination was halted on the 5th day. This indicates that the young plant had not yet reached the stage of exhausting its reserves. Furthermore, during the inoculation process with Lactobacillus (75 hours), the lactic acid yield resulting from the digestion of glucose was assessed by acid-base titration. The percentage yields obtained during this process, along with the change in the solution's pH, are presented in Figure 3.

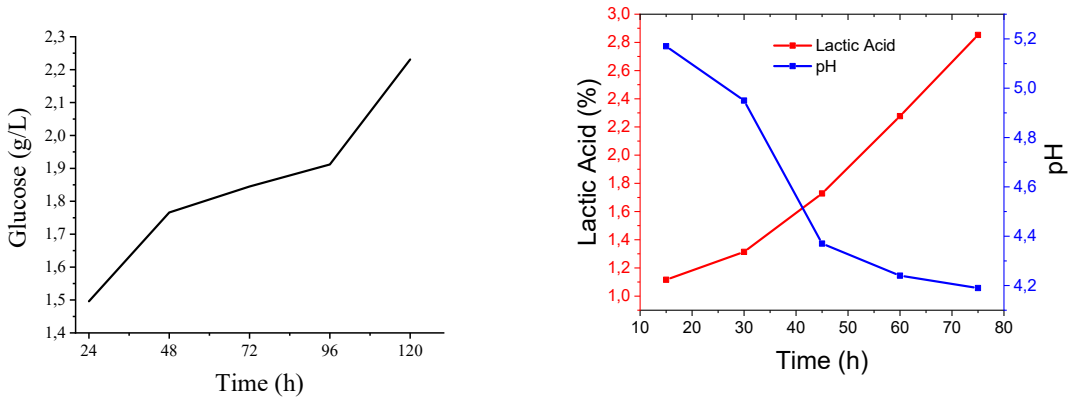

Figure 3. Curves of glucose and lactic acid production.

Lactic acid concentration increases, which is consistent with the rise in the medium's acidity (pH dropping from 5.17 to 4.19). The lactic acid bacteria being tolerant to this acidity, continues to metabolize the glucose until the pH reaches an inhibitory level. This result agrees with the findings of Boyaval et al. [19] in their study on lactic acid production by continuous fermentation.

3.2. Characteristics of the synthesized polylactic acid

The bioplastic obtained by polymerization is shown in Figure 4. It is a solid, brittle, white material with an amorphous appearance, showing some crystals on the surface.

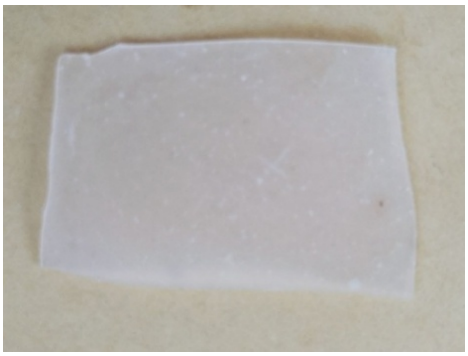

Figure 4. Bio-based polylactide from corn.

The material exhibits the characteristics recorded in Table 1

Table 1. Some characteristics of the PLA from the study

Yield (%)	Moisture Content (%)	Water Absorption (%)	Solubility (%)
7,87	4,93	5,51	0,904

An examination of the table reveals a low yield compared to that observed by Xu et al. [20], who optimized the fermentation conditions of free whole cells of three *Lactobacillus crustorum* strains to produce 3-Phenyllactic acid (3-PLA). This difference can be explained by the fact that their protocol was based on an optimized fermentation using free whole cells of selected strains of *Lactobacillus crustorum*, a condition that we did not reproduce in our study. The low yield in this study could partly be explained by the poor growth of lactobacilli in the substrate. However, high rates of moisture and water absorption are noted, which could be

attributed to the presence of pores formed during the polymerization process. The high moisture content could also be explained by the presence of residual diethyl ether solvent, a polar solvent likely to induce more or less strong interactions with the polar functional groups of the material. Furthermore, the obtained PLA exhibits non-zero solubility in water, which could be explained by the amorphous structure of the material and the presence of aggregates formed during polymerization. The results of the FTIR analysis of the surface functional groups are given in Figure 5.

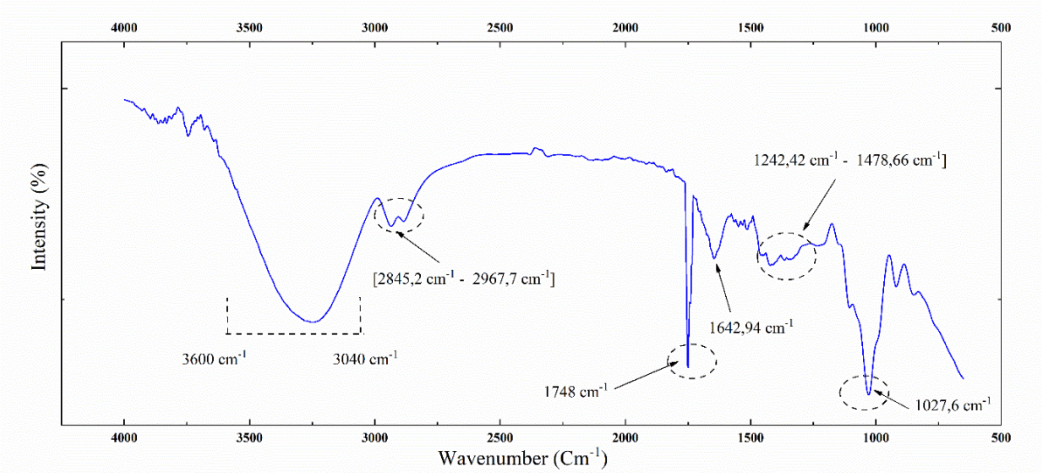

Figure 5. FTIR spectrum of corn-based polylactide.

Between 1242.42 cm^{-1} and 1478.66 cm^{-1}, complex bands indicate vibrations associated with C-O-C and C-O stretching in the ester chains of the corn-based polylactide.

Between 2845.2 cm^{-1} and 2967.7 cm^{-1}, medium bands are observed, representing the C-H stretching vibrations of methyl (CH_3) and methylene groups. This indicates that the carbon chains of the PLA are intact. According to Bajpai et al. [21], these groups are essential for the mechanical and thermal stability of the corn-based polylactide.

Between 3040 cm^{-1} and 3600 cm^{-1}, a broad, medium band is observed, corresponding to the O-H stretching vibration of carboxylic acid and alcohol groups. The presence of hydroxyl (OH) groups could also be partly related to residual moisture or to hydroxyl groups introduced during the extraction with diethyl ether solvent.
The intense peak at 1748 cm^{-1} is characteristic of the C=O carbonyl stretching vibration present in the esters of the corn-based polylactide. The observation of this peak indicates the presence of ester groups associated with polylactic acid (PLA) in the analyzed sample. Carbonyl groups are essential structural markers of PLA, and their presence indicates that the polymer chains have not undergone major degradation. The peak at 1642.94 cm^{-1} indicates the C=O carbonyl stretching vibration of the lactide. This suggests that the polymerization was not complete.

3.3. Biodegradability of corn-based polylactide

The biodegradability tests of the synthesized PLA were conducted by burial over a period of 100 days. The burial took place at ambient temperature, using three soil matrices under both aerobic and anaerobic conditions.

3.3.1. Composition of the burial matrices

The composition of the burial matrices is given in the following table:

Table 2. Soil composition

Burial Matrice	pH	Organic Matters (%)	Water Absorption (%)	Moisture Content (%)
Composted Soil	7.33	46.1	56.24	13.14
Normal Soil	7.16	2.54	53.2	11.7
Sterile Soil	6.72	0.39	45.01	3.8

The sterile soil has a more acidic pH and a low organic matter content. In contrast, the composted soil has a higher pH, likely due to the calcium and iron oxides present in the cow dung, as observed by Nekhubvi [22]. As for the normal soil, its pH is close to neutral and it has a moderate amount of organic matter. The water absorption capacity of the composted soil remains the highest, which could be explained by the ability of organic matter to adsorb water via hydrogen bonds.

3.3.2. Biodegradation

The results of monitoring PLA degradation in the three types of matrices, under both aerobic and anaerobic conditions, are presented in Figures 6-a) and 6-b).

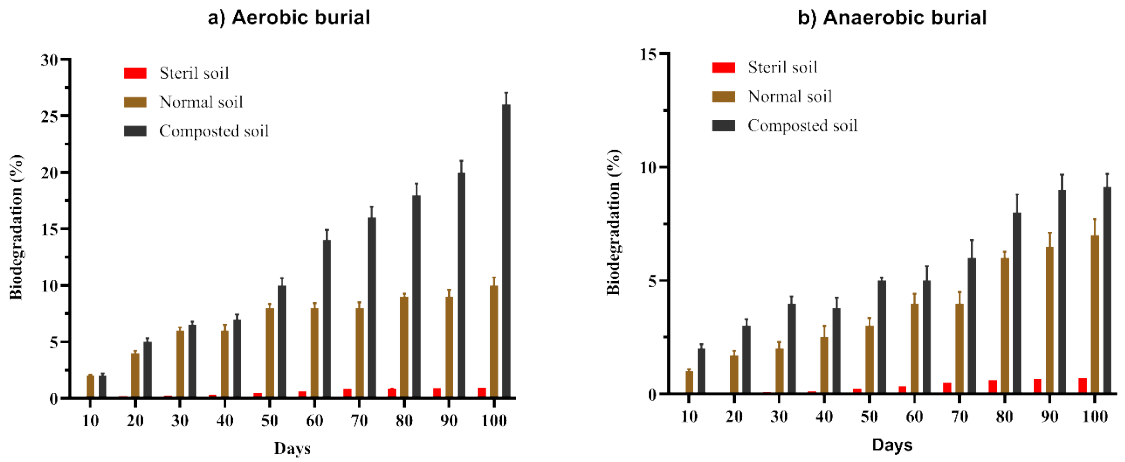

Figure 6. Biodegradation of corn-based polylactide under two different environmental conditions.

In sterile soil, whether under aerobic or anaerobic conditions, the PLA shows almost no mass loss over the entire 100-day study period. The structural properties that could make the material more or less degradable have a low impact in the sterile soil matrix. The low organic matter content, and consequently the low microorganism content, of the sterile soil matrix could partially explain this relative stability of the material in this environment. This result agrees with the study by Birinci et al [23], which showed that the absence of biotic factors limits degradation. In contrast, in the normal soil and composted soil matrices, significant biodegradation was observed under both aerobic and anaerobic conditions, thus confirming the predominant role of biotic factors in the PLA degradation mechanism. In normal soil, the degradation under both aerobic and anaerobic conditions is less significant than that observed with the composted soil matrix, which could be justified by a difference in microbial population, favoring the composted matrix. However, it is noted that for the composted soil, the degradation proportions observed under aerobic conditions are greater than those observed under anaerobic conditions. This difference could be linked to several favorable factors. Indeed, the microorganisms present in the composted matrix, such as *Thermobifida fusca* are predominantly aerobic

bacteria [24]. The aerobic nature of the environment is therefore conducive to microbial multiplication. Furthermore, the contribution of ambient moisture promotes the hydrolysis of PLA through the cleavage of ester bonds (CO-O). The amorphous structure of the PLA could also facilitate water mobility compared to a crystalline structure, as also observed by Silveira [25]. This more or less dense structure of the PLA could be an essential factor in the degradation process by microorganisms. In the composted soil, degradation is more pronounced due to the activity of microorganisms, which play a crucial role in the process of organic matter degradation [26]. These microorganisms secrete enzymes capable of breaking down PLA into lactate monomers and oligomers, which justifies the rapid increase in mass loss.

Conclusion

In this study, a bio-sourced polylactic acid (PLA) type bioplastic was synthesized from local corn biomass. The developed material was characterized through several physicochemical properties and an analysis by Fourier Transform Infrared (FTIR) spectroscopy. The biodegradability was assessed under both aerobic and anaerobic conditions. The results show that the biodegradation of PLA in the different burial matrices depends on the quantity of microorganisms and the nature of the environment. Locally, this study opens the field for the exploration of other local biomasses potentially capable of yielding a higher polylactide output using the processes described in this study.

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Conflict of Interest

There are no conflicts of interest in relation to this manuscript.

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