Study of the biodegradability behavior in films of starch with added rice-dust vs. films of Polylactic acid with corn starch in a controlled composting system

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Abstract*– Nowadays, plastics and oil derivatives are easier access and low-cost, which is why plastic is present everywhere. However, this trend has caused real damage to the environment due to the prolonged life span of plastics which cause accumulation. Therefore, alternatives to the use of plastic have been studied, being one of the solutions is the use of natural biopolymers. As a result, exists in the market different alternatives, i.e., biodegradables, biodegradables-compostable, oxo-biodegradables, etc. Nevertheless, it is still necessary to study their biodegradability; thus, this study aims to analyze different biopolymers' biodegradation at 25 °C (room temperature) under controlled composting conditions. For this effect, three polysaccharide base matrices (films) were made by casting varying the content of their constituents, i.e., plantain starch (AP) + cassava starch (AY) + gelatin (G) using glycerol as a plasticizer and adding up to 10% of fiber (rice-dust)-P, and compared with a commercial matrix made of Polylactic acid (PLA) and corn starch (AM). All the films were subjected to weight control and speed of biodegradation for 28 days. Additionally, the water solubility of all the films was evaluated and characterized using infrared spectrometry-FTIR. All the films made by casting showed similar behavior, being noticeable their affinity with the topsoil used for the composting. Importantly, the film with high fiber (10%), N9, presents lower water solubility, less biodegradability, and weight loss. On the other hand, the commercial films (PLA + AM) showed changes until day 7 of the composting assay, then stabilizing until the last day of the assay (day 28).

***Keywords*—Green Plantain, cassava starch, Fourier Transform Infrared Spectrometry, Solubility, Polylactic acid.**

I. INTRODUCTION

Plastics are present everywhere. They dominate the world's materials preference. Due to their lightweight, low cost, and diversity are widely used in food packaging, where their share is about 75%. An important part, about 60%, is single-use flexible containers or bags [1]. Overall, plastics are designed to resist corrosion, water vapor, microbial attack, and degradation; therefore, their lifetime is usually very long. Regrettably, they are not receiving any kind of treatment previous to being buried in landfills [2]. Furthermore, the addition of additives to improve plastics' mechanical properties results in extending their stability, lowering, even more, their degradation. Nevertheless, these advantageous characteristics of plastics make them a high pollutant material. Their stability, given by their long polymeric chains, results in prolonged degradation times that may take decades or even centuries, generating accumulation and converting plastics into an environmental burden. As a result, there is an increasing interest in research involving the study of materials (biodegradable or natural polymers) that are easier to degrade, produced by nature, or by the action of microorganisms (bioplastics) that may be used to replace plastics. Of all the plastic manufactured in 2018, 1% were bioplastics (2.11 million Tons). Being 43% biodegradable bioplastics, such as Polylactic acid (PLA), polybutylene adipate terephthalate (PBAT), and Polybutylene succinate (PBS). The rest of them are non-biodegradable bioplastics [3]. Biodegradable plastics are produced almost in equal quantities; however, their biodegradability is not the same reason why studying their degradation under different times and atmospheric conditions [4]. The PLA obtained from various sources of glucose is water-resistant but unstable to halogen hydrocarbons. It was the first biodegradable bioplastic produced at a commercial scale using blowing or injection processes, mainly used for packaging, and it takes to degrade about three weeks under controlled conditions and from 6 to 24 months when left in the environment [5] [6] [7]. Another material extensively investigated is starch, a natural polymer mainly found in cereals, tubers, and certain legumes of wide applications. For example, to give texture and consistency to foodstuff, produce paper, adhesives, or biodegradable packaging [1]. Starch is a biodegradable material, non-toxic, and able to degrade by composting under non-controlled conditions [8] [9]. Commercially there are three types of starch, i.e., native, modified, and fermented. Before its use as a thermoplastic, it needs to be destructured to eliminate its crystalline structure, usually with heat, pressure, mechanical impact treatments, or plasticizers [10]. Lately, the interest in

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starch films has increased due to their appealing characteristics, such as ease to generate, high capacity to make films, high yield, and less energy-intensive than plastics [9]. Although starch shows desirable processing and attributes, its mechanical and barrier properties are usually lower than plastics. It has been found that combining starch with other polysaccharides, proteins, fibers, among others, increases mechanical and barrier properties and allows extrusion process, therefore, replacing some types of plastics. The proteins stabilize for their tridimensional structure and non-covalent interactions, giving the matrices thermal stability and oxygen barrier [11] [12] [13]. This study aims to analyze the degradation of different matrices based on starch and fiber, comparing them with commercial biodegradable materials in the ambient and under controlled conditions, comparing its comportment, and determining its soil biodegradability.

II. MATERIAL AND METHODS

A. *Raw material*

For the film manufacture was used: green plantain (*Musa Paradisiaca*) from the farmers market of Manabí, Ecuador (AP) (37.9% amylose and 62.1% amylopectin), local cassava starch from "La Pradera" (AY) (31.2% amylose and 68.8% amylopectin), Local rice-dust (RP) with 7% of crude fiber with an average particle size between 400 – 500 µm, and 8% moisture (RH) (3.8% amylose, and 96.2% amylopectin). Also, Merck 300° Bloom bovine gelatin (G) was used to aid in the resistance and barrier properties, and Merck glycerol (GL) as a plasticizer. For comparing biodegradable and compostable film's characteristics, commercial Italian carry bags made of polylactic acid and corn starch (PLA + AM) from a supermarket in Lima, Peru, with an advised lifetime of 6 months, were used. Finally, 4 kg of topsoil was used in the composting chamber (pH 5.5 – 6.5).

B. *Manufacturing and labeling of starch-based films*

In Table 1 describes the labeling and composition of the manufactured films. The film composition used in this study results from previous feasibility assays; the selected formulas correspond to those showing the best mechanical properties. The films based on starch and fiber were compared to a film made of commercial carry bags made of PLA + AM.

The polysaccharides were mixed as dispersions. Therefore, water-based solutions were heated in a water bath at 100 °C for 30 minutes. The gelatin was dissolved in water at 80 °C using a heating plate with a magnetic stirrer (VWR USA/Hotplate stirrer BS-4HC) for 30 minutes.

The AP, AY, and G dispersions were made at 4 and 6% of total solids (TS), then 33.33% of each distribution were mixed, and added GL solution at 30% TS.

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Table 1
Manufacturing and labeling of starch-based films

INGREDIENTS	N1	N9	N13
Green plantain starch, cassava starch, and gelatin AP-AY-G	4 %	4 %	6 %
Glycerol GL	30 %	30 %	30 %
Rice-dust RP	5 %	10 %	10 %

After, the RP was added in the proportions described in Table 1. Each formula was homogenized for six minutes at 10,000 rpm (Ultra – Turrax IKA – T25 Germany). The films were manufactured by casting method; a Teflon mold of 150mm was used, with a weight sufficient to reach 4 and 6 grams of TS, depending on the formula. The molds were let to dry at room temperature (25 °C) and 53% RH; once dry, the films were stored in a desiccator at the conditions used for drying them.

C. *Solubility*

The method described by [14] was followed to analyze the solubility of the films N1, N9, N13, and PLA + AM. The film was cut in circles of 2.5 cm diameter and dried at 100 °C using a stove (Thermo Scientific – 5101451, USA) for 24 hours until constant weight. Then, the films were submerged for 24 hours in 125 ml beakers containing 100 ml of water for 24 hours and dried as before (Fig. 1). The solubility of the films in water was reported as % weight loss, using equation 1.

$$\%\, weigh\ loss = \frac{(initial\ weight - final\ weight)}{initial\ weight}\ x\ 100 \quad (1)$$

Fig.1 Film submerged in 100 mL of water.

C. *Composting Chamber*

Consisted of a 30 x 60 cm translucent glass chamber, 10 cm of topsoil was evenly added (Fig. 2). The topsoil bed was divided into four 30 x 15 cm sections for burying the films, following the method described by [15] and [16].

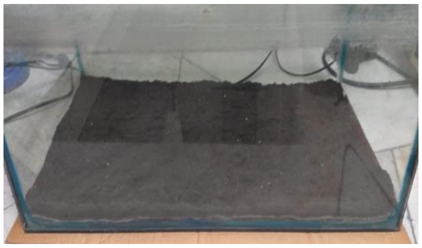
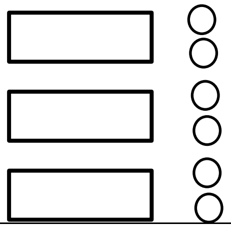


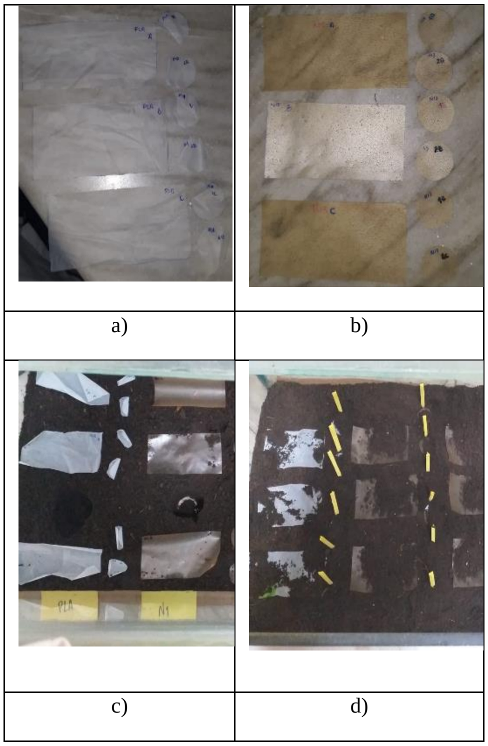
Fig. 2 Composting chamber with added topsoil

D. *Film conditioning in the composting chamber*

Strips (5 x 10 cm) and circles (2 cm diameter) were obtained from each film and buried in each topsoil bed section (Fig. 3a and 3b). It was buried three rectangles and six circles per section as described in Table 2; the experiments use triplicates for the rectangles and sextuples for the circles. The temperature was kept at 25°C.

Table 2
Film Distribution in the composting chamber

Section	Film code	Distribution in each section
1	PLA+AM	
2	N1	
3	N9	
4	N13	



a) b)

c) d)

Fig. 3 Strip and circular films a) PLA + AM. b) Films N1, N9, N13. c) Film distribution in composting chamber. d) Signaling of buried films

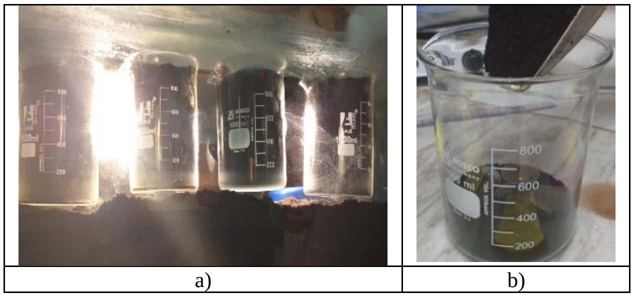
All the films, strips, and circles were labeled with codes for identification in weight control loss. Fig. 3c shows the ubication of the film in each sector of the composting chamber, and Fig. 3d shows the signaling used to locate the film after being buried. Table 3 describes the used coding.

Table 3
Film coding used in the composting chamber

Section	Film coding	Strips coding	Circle coding
1	PLA+AM	A	1A,2A
2	N1	B	1B,2B
3	N9	C	1C,2C
4	N13		

E. *Biodegradability*

The method described by [17] [18] [19] was followed, applying two temperatures, i.e., 60 and 25°C, to the strips and circular films. When working at 60 °C, the films were placed inside of chamber equipped with incandescent bulb light as a source of heating (Fig. 4a). In both cases, the temperature and moisture were registered using a Termo Pro, China. In Fig. 4b shows how the film was placed in each beaker.



a) b)

Fig. 4 a) Composting chamber 60 °C. b) Film composting preparation in beakers

All the circular films corresponding to each formula were weighed at 0, 1, 3, 7, 9, 14, 21, and 28 days using a balance with a sensibility of +/0.0001 (Boeco, model BWL61, USA). The rectangular films were weighed on days 0 and 28. When performing the control weight, the film was clean using a brush of natural hair to remove all the dirt adhered to the film to prevent error in the readings. After weight control, the films were returned to the composting chamber, taking the precaution of burying them 3 cm depth. Equation 1 was used to calculate the weight loss.

F. *Infrared Spectroscopy*

A Fourier Transform Infrared Spectrometer – FTIR (Bruker Tensor 27 instrument, Netherlands) in the spectral range 4400 – 600 cm^{-1}, with 8 cm^{-1} resolution, was used to analyze, by duplicate, the films' changes biodegradation at

25°C. For films N1, N9, and N13, the analysis was performed at days 0, 1, and 2 in the circular films. On the other hand, for the PLA + AM films, the analysis was performed on days 0, 7, 14, and 21.

G. *Statistical analysis*

Minitab® 17.1.0 was used to analyze the results using the Analysis of variance (ANOVA) test with 95% confidence level and Tukey test for multiple comparisons.

III. RESULTS AND DISCUSSION

A. *Solubility in water*

The solubility in water of the films is one of the crucial factors in the analysis of the ability of the film to biodegrade; it will define the correct use of the film. The methodology of [13] and equation one were used to determine the % solubility, between 71 and 75% for starch and fiber films; considerably higher than the PLA + AM films (Table 4).

Table 4
Films solubility (%)

Film	Solubility (%)
PLA+AM	47.99
N1	75.44
N9	71.84
N13	72.27

The hydroxyl groups of the starch give hydrophilicity characteristics, kept in the time, raising the matrix's biodegradability. Despite the solids content, the films N9 and N13 showed similar values, maybe due to incorporating 10% fiber in its formula. Apparently, the addition of rice fiber delays the film solubility forming carbon bonds with the gelatin helical structure, improving the microstructural properties; this behavior was reported by [20] when working with cellulose fibers. According to [21], to secure the storage of films used in bags manufacturing, these should show low biodegradability. However, high values may result convenient during the cooking of coated foodstuff. The content of fiber, fats, nanoparticles, etc., added to the starch-based films improves its mechanical and barrier properties, delaying biodegradability [15]. Another factor is the glycerol content, [22] reported an indirect relationship between the content of glycerol (15, 30, and 45%) with moisture absorption, and [23] reported better thermal stability. This effect was also reported by [24] when working with arracacha starch with 66% solubility, also [25] found the values of solubility between 68 and 80% when working with potato starch, glycerol, and acetic acid. Others researchers [26] reported that high solubility, over 60% when working with cassava starch and fiber, decreased with the increment of fiber added, resulting in degradation from day 25 when working under controlled conditions.

B. *Biodegradability*

This assay started using a temperature of 60 °C for all the films in the composting chamber; however, after 48 hours, the films N1, N9, and N13 (strips and circles) presented high biodegradability to the point of being cumbersome clean the dirt from due to the brittleness of the films (Fig. 5). It was noticeable the less biodegradation presented by N13. However, it cannot be conclusive because the study did not surpass 72 hours and the possibility that the hydrolysis may be associated more accessible to the topsoil when started. On the other hand, the PLA + AM films did not show brittleness. However, it was challenging to clean them properly, maybe because of the used temperature; [27] reported a direct relationship between temperature, moisture, and PLA degradation. Therefore, the faster degradation of PLA could be reached if composting chambers with temperatures above 60°C are built, reducing the economic investment for treatment of this kind of biopolymer carried nowadays in aerobic systems, difficult to control. Also, it may be considered the burying the films at least three cm depth in topsoil, also reported by [26] [28]. As a result, it was decided to observe the results at room temperature (25°C).

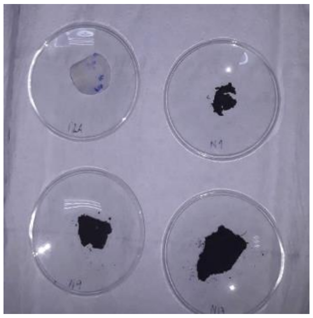


Fig. 5 PLA + AM, N1, N9, and N13 films after 48 hours of treatment

When working at 25 °C, it was possible to control the weight until 28 days; Fig. 6 shows the trend, where the average weight of the circular films, in the 1st day of control, shows a weight increment in N1, N9, and N13 probably because of the adhesion of dirt particles.

Nevertheless, a loss of weight from day three is observed due to biodegradation. The PLA + AM film showed a linear behavior in the weight loss; this may be due to acid lactic possessing stronger chemical bonds than starch, as a result, needs more time at the working conditions of the composting chamber; the reason why the PLA + AM circular films did not show a reduction on its diameter. Conversely, the starch film presented a decreasing diameter on, in approximately 0.5 cm on day 9; from this day on, a diameter of 2.0 cm was kept until the last day of this assay.

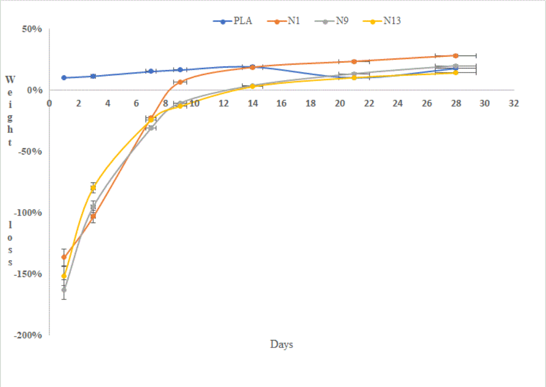


Fig. 6 Weight loss in circular films

The rectangular strip's behavior was similar to the circular films, shown in Fig. 7.

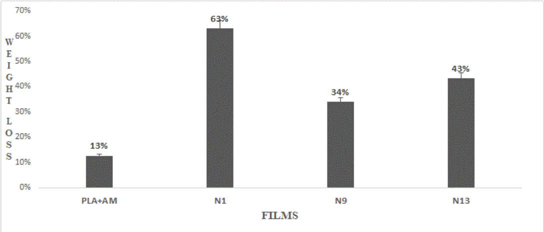
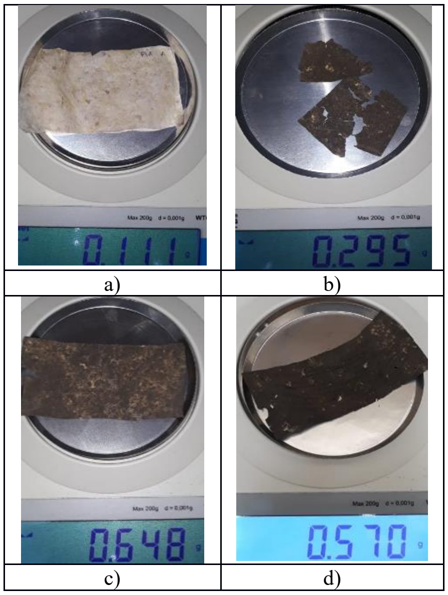


Fig. 7 Weight loss in rectangular films

The film N9 showed the least biodegradability (34%) in the timespan of the assay. Researchers [29] working with cassava and potato starch, glycerol, acetic acid, obtained by cooking and molding process and stored in composting environment observed eight days the biodegradability before the track was lost because the amylose and amylopectin chains made the film brittle; results that are similar to the reported in this study. Fig. 8 shows the films at the end of the assay.



a) b) c) d)

Fig. 8 a) PLA+AM b) N1 c) N9 d) N13 films biodegradation after 28 days.

C. *Biodegradation Speed*

It was observed a slow increment in the speed of biodegradation for the starch-based circular films; contrarily, that of the PLA + AM film was almost constant during the time of this assay (1, 3, 7, 14, 21, and 28 days). Notably, the starch-based films showed negative values of speed of biodegradation until day 14 of this assay, probably due to error caused for dirt adhered to the surface of the film acting as a shield against biodegradation or because of lack of mass transfer (moisture accumulation) between the surface of the film and the inner matrix (time required to start hydrolysis). In Table 5 shows the different values for the film's biodegradation speed.

The average biodegradation speed for strip films was determined on day 28 (Table 6). It was observed a slower speed of biodegradation on film N9; its formula (10% fiber) may be the reason for this result. Other researchers had reported digestion under household (35°C) and industrial composting conditions (50°), i.e., mesophilic and thermophilic temperature regimes, respectively, registering an increase in speed of biodegradation. For pure PLA, severe conditions are required for biodegradability; however, when mixed with starch, the presence of microorganisms accelerates biodegradation, as shown by [30] [31]. Furthermore, [27] showed how temperature affects PLA-based waste kinetics. The speed of biodegradation at high temperatures results in a reorganization of the biopolymer chains, provoking the crystallinity increment destroying the macromolecular support forming sub-products, an effect reported by [32]. The incorporation of clay nanoparticles to the starch (Montmorillonite-Na) impeded the enzymatic attack and delayed the biodegradability, which we could relate to the good behavior presented for film N9 [33].

D. *Infrared spectrometry – FTIR*

The PLA+AM films do not show differences in the ranges of absorption between day 0 and 7. however, beginning on day 14 it was observed differences in peaks, i.e., in the ranges of 800 cm^{-1} to 1000 cm^{-1}, 1800 cm^{-1} to 2000 cm^{-1}, and 3000 cm^{-1} to 3200 cm^{-1} the trend continues until the end of the assay (Table 7). Studies performed by [34] determined that 3200 to 3400 cm^{-1} wavelengths correspond to additives used in PLA items manufacture, which appear in the same wavelength in this study. The peaks in the range of 1800 cm^{-1} to 2000 cm^{-1} reduced significantly until they disappeared on day 14; this may be due to the rupture of aliphatic bonds; this particularity was observed by [35], who worked with PLA at different combinations. The peaks in the range of 2000 cm^{-1} to 2800 cm^{-1} were absent.

Table 5

Speed of biodegradation of circular films (g/day)
at 1,3,7,14,21, and 28 days

	1	3	7	14	21	28
PLA+AM	0.001±0.002	0.001±0.001	$3x10^{-4}$±0.000	$2x10^{-4}$±0.000	$2x10^{-4}$±0.000	$1x10^{-4}$±0.000
N1	-0.110±0.034	-0.028±0.016	-0.003±0.001	0.001±0.000	0.001±0.000	0.001±0.000
N9	-0.147±0.017	-0.029±0.005	-0.004±0.001	-0.001±0.001	$3x10^{-4}$±0.001	0.001±0.000
N13	-0.252±0.018	-0.044±0.005	-0.006±0.002	-0.002±0.001	$4x10^{-4}$±0.001	0.001±0.001

Table 6

Rectangular films biodegradation at day 28

Film	[g/day]
PLA+AM	0.0091
N1	0.020
N9	0.010
N13	0.021

Table 7

PLA + AM FTIR spectroscopy peaks at 0, 7, 14, and 21 days

Wavelength (cm^{-1})	0	7	14	21
4400-3800				
3800-3600				
3600-3400				
3400-3200	X	X	X	X
3200-3000	X	X		
3000-2800	X	X		X
2800-2600				
2600-2400				
2400-2200				
2200-2000				
2000-1800	X	X		
1800-1600	X	X	X	X
1600-1400	X	X	X	X
1400-1200	X	X	X	X
1200-1000	X	X	X	X
1000-800	X	X		
800-600	X	X	X	X

The ranges 1400 cm^{-1} to 1800 cm^{-1} corresponding to carbonyl functional group (-C=O), 1200 cm^{-1} to 1400 cm^{-1} corresponding to the functional group (-CH), and 1000 cm^{-1} to 1200 cm^{-1} corresponding to the functional group (-C-O) did not have changes until day 21 of this assay; in these ranges are the starch peaks, seems that the combination of PLA with corn starch is homogenous and did no present changes during the time of this study.

The biodegradability happens selectively, first in the amorphous regions and then in the crystalline zones of pure PLA; it is not affected by composting until after seven months when working with pure PLA. [36] reported that the combination of PLA with starch speeds up its biodegradability, it is identified by the presence of the carbonyl group (-C=O) (range of 1800 cm^{-1} to 2000 cm^{-1}), which contributes to the biodegradability and are the first to disappear (Table 7), this was also reported by [37] who worked with PLA and corn starch, also finding stretch in the hydroxyl groups (-OH), glycosidic groups (-C-O-C), and carbonyl groups (-C=O); diminishing its intensity provoked by the loss of the starch chains after 30 days.

The films N1, N9, and N13, in red in Table 8, show peaks in the same frequency which are kept during the assay timespan, i.e., from 1200 cm^{-1} to 1800 cm^{-1} carbonyl groups and sections of carbonyl groups (-C=O / -C-O-), 2000 cm^{-1} to 2200 cm^{-1} aliphatic fragments, and 2800 cm^{-1} to 3200 cm^{-1} (-C-H) aliphatic groups.

The wavelength range 3800 cm^{-1} to 3600 cm^{-1} marked in blue in films N1, N9, and N13, absent at the assay start, appears on days 1, and 2 of the assay; are related to additives used in the assay and stabilized after 24 hours. Notably, the film N9 with 10% of fiber (marked in green) present a peak in the wavelength range of 1200 cm^{-1} to 1000 cm^{-1} and 3200 cm^{-1} to 3400 cm^{-1} that corresponds to the functional groups (–C=O) and (–C-H), respectively, maintained since de assay start. It could be compounds that contribute to reducing biodegradability. The FTIR of films N1 and N9 are similar with the same peaks and wavelengths, similar to the reported by [34], who worked with animal gelatin and blends of sunflower oil and related the peaks to the gelatin amino groups [13].

Table 8

N1, N9, and N13 FTIR spectroscopy peaks at 0, 1, and 2 days

Wavelength	N1	N9	N13	N1	N9	N13	N1	N9	N13
(cm^{-1})	0 day			1 day			2 days		
4400-3800			X	X	X	X	X		X
3800-3600				X	X	X	X	X	X
3600-3400			X	X	X		X		X
3400-3200	X	O		X	O	X		O	
3200-3000	X	X	X	X	X	X	X	X	X
3000-2800	X	X	X	X	X	X	X	X	X
2800-2600			X	X		X	X	X	
2600-2400						X			
2400-2200						X			
2200-2000	X	X	X	X	X	X	X	X	X
2000-1800									
1800-1600	X	X	X	X	X	X	X	X	X
1600-1400	X	X	X	X	X	X	X	X	X
1400-1200	X	X	X	X	X	X	X	X	X
1200-1000		O			O			O	
1000-800				X		X	X	X	X
800-600					X	X	X	X	

At the end of the FTIR, the films N1, N9, and N13 were left at room conditions turning more fragile due to the continuity of changes in their structure favors biodegradability (Fig. 9). Fig. 10, 11, 12, and 13 correspond to films PLA+AM, N1, N9, and N13 diffractograms, whose results were summarized in tables 7 and 8.

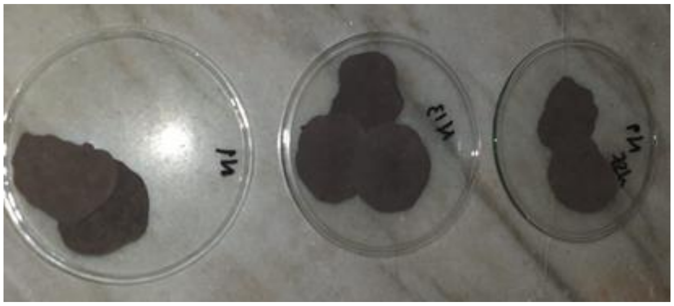


Fig. 9 N1, N9, N13 films left at room conditions.

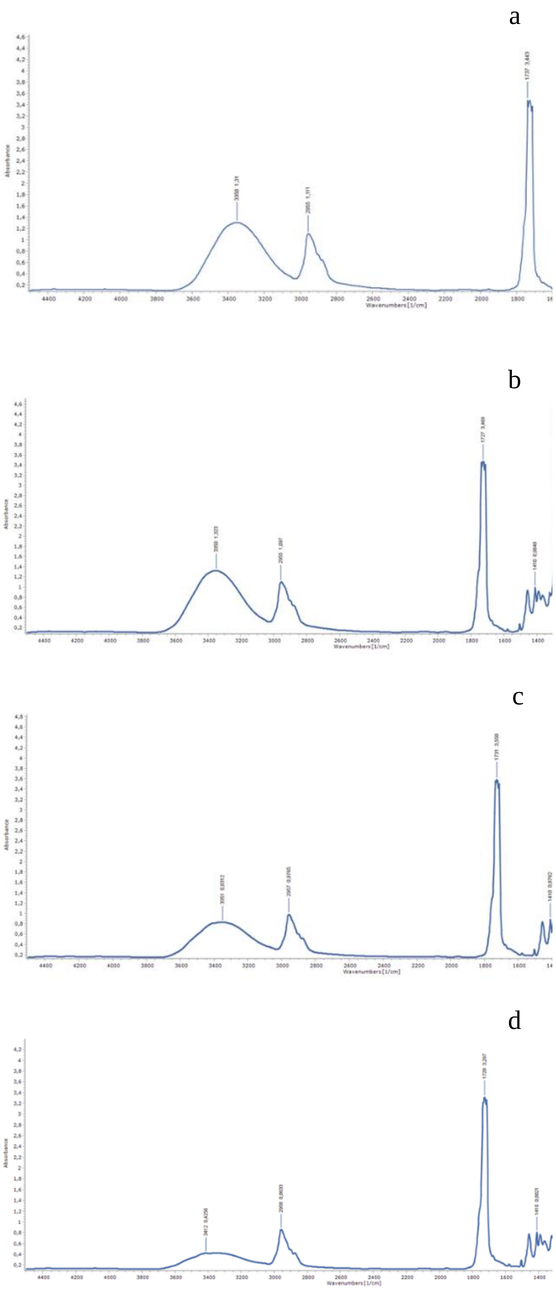


Fig. 10 PLA + AM FTIR at a) 0 days b) 7 days c) 14 days d) 21 days

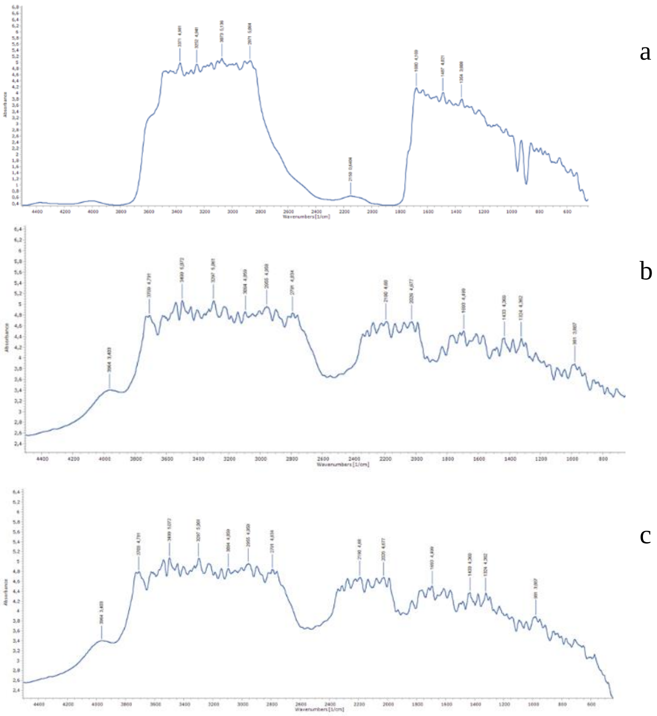


Fig. 11 N1 film FTIR at a) 0 days b) 1 day c) 2 days

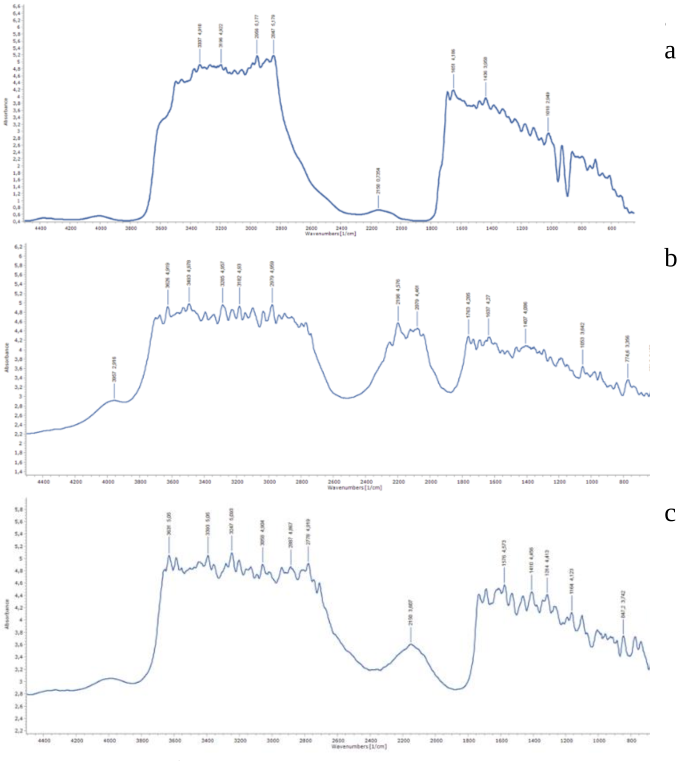


Fig. 12 N9 film FTIR at a) 0 days b) 1 day c) 2 days

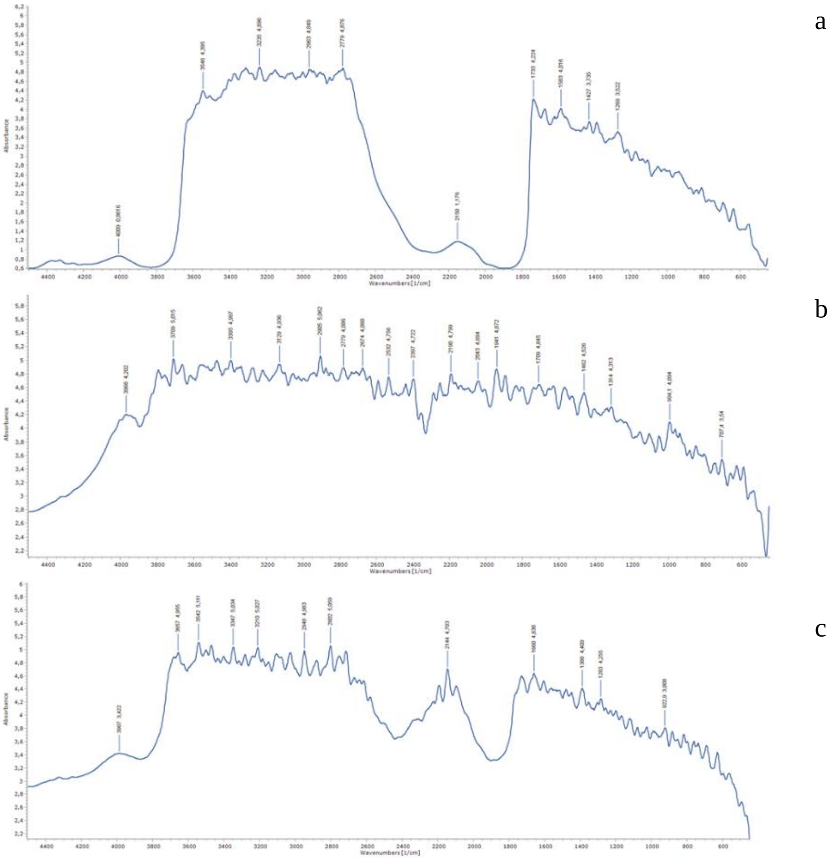


Fig. 13 N13 film FTIR at a) 0 days, b) 1 day, and c) 2 days

IV. CONCLUSION

All the films made of native starches show similar behavior in the controlled parameters, i.e., water solubility percentage and speed of biodegradation. Notably, film N9 has the lowest values meaning that adding 10% fiber improves its physical properties. In the biodegradability assay, at 25 °C, films N1, N9, and N13 showed a high affinity with the topsoil used in the composting chamber, which relates to the biodegradability at environmental conditions. The film N9 needs less time for biodegradation in soil, with 34% weight loss in 28 at the composting chamber. The PLA+AM film showed a biodegradability speed of about 0.0091 g/day, corresponding to 13% of weight loss until day 28 at the composting chamber, being stable in its composition, i.e., the FTIR at day 14 showed peaks at the same wavelength. Notably, only film N9 showed peaks in the wavelength range of 1200 cm^{-1} to 1000 cm^{-1}, and 3200 cm^{-1} to 3400 cm^{-1} which are maintained until day 28; these can be related to the presence of functional groups (–C=O) and (–C-H) that contributes to reduce the biodegradability speed.

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