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Sustainable starch-based films with added babassu oil: production and technological characterization

Jésica Matos LACERDA[1], Renata Ferreira SANTANA[1], Clara Mariana Gonçalves LIMA[2], Thais Santos Moraes LIMA[1], Waseem KHALID[3], Bruno Fonsêca FEITOSA[4,5], Mônica Tejo CAVALCANTI[6], Leandro Soares SANTOS[1], Rafael Da Costa Ilhéu FONTAN[1], Evaldo Cardozo de SOUZA JÚNIOR[1], Henrique Douglas Melo COUTINHO[7], Jolanta WAWRZYNIAK[8], Silvani VERRUCK[9], Renata Cristina Ferreira BONOMO[1*]

Abstract

This study aimed to produce and characterize the technological properties of biodegradable films made from cassava starch incorporated with babassu oil and sorbitol as a plasticizer. The films were prepared using the casting technique and a 4×5 factorial experimental design, with the plasticizer and oil concentrations as independent variables. The films were characterized for thickness, solubility in water, saline solution, and acidic solution, water vapor permeability, thermal analysis, mechanical properties, Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and biodegradability. The biodegradable films exhibited good mechanical properties (maximum tensile strength: 4.78 ± 2.75 MPa and percentage elongation: 183.13 ± 75.25%) and satisfactory barrier properties, indicating their potential for use as environmentally sustainable packaging. Thermal analyses revealed the presence of three stages of weight loss common to all films, while diffractograms revealed crystalline zones at 19.3 and 30º. The polysaccharide nature of the films was confirmed by FTIR spectra. The films demonstrated significant solubility, with values ranging from 85.53 ± 22.79% to 99.72 ± 8.69%, and rapid biodegradation, suggesting a viable substitute for non-biodegradable polymers, thereby mitigating improper disposal of such materials into the environment. The films incorporated with babassu oil and sorbitol showed good water vapor barrier properties and adequate mechanical resistance, especially when sorbitol was used as a plasticizer.

Keywords: biodegradable packaging biomaterial; *Orbignya phalerata*; starch; sorbitol plasticizer; biopolymeric matrix; bio-based composite.

Practical Application: This study is relevant for the development of biodegradable materials that can replace conventional plastics, contributing to the mitigation of the environmental impact caused by the improper disposal of plastics. The incorporation of babassu oil into cassava starch-based films not only uses a renewable natural resource abundant in Brazil but can also give the material improved functional properties, such as greater hydrophobicity and potential antimicrobial activity, expanding its applications in packaging. Furthermore, by valuing regional products such as babassu, the study promotes economic and social sustainability, encouraging the use of local raw materials and innovating in the biodegradable packaging sector.

1 INTRODUCTION

The plastics industry is one of the largest revenue-generating sectors globally, with a market size of more than USD 348 billion annually. It is estimated that in this branch of industry, there will be a continuous annual increase of 4.2% from 2021 to 2026 (Agarwal, 2021). Despite its economic efficiency, the widespread use of synthetic packaging in modern society has created significant environmental challenges. Scientists and researchers are concerned about the detrimental impact of this human activity on the environment, which has driven the search for solutions based on biodegradable materials from renewable sources (Pelissari et al., 2019; Vianna et al., 2021). Unlike traditional plastics, which can remain intact for many years, bio-composites represent a promising alternative (Tabassum et al., 2024), as these biomaterials break

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[1]*Universidade Estadual do Sudoeste da Bahia, Department of Animal and Rural Technology, Itapetinga, BA, Brazil.*
[2]*Universidade Federal de Lavras, Department of Food Science, Lavras, MG, Brazil.*
[3]*University of Castilla La Mancha, Faculty of Chemical Sciences and Technologies, Department of Organic Chemistry, Ciudad Real, Spain.*
[4]*Universidade do Estado do Amapá, Amapá, AP, Brazil.*
[5]*Universidade Estadual de Campinas, Campinas, SP, Brazil.*
[6]*National Institute of the Semiarid Region, Campina Grande, PB, Brazil.*
[7]*Universidade Regional do Cariri, Crato, CE, Brazil.*
[8]*Poznań University of Life Sciences, Faculty of Food Science and Nutrition, Poznań, Poland.*
[9]*Universidade Federal de Santa Catarina, Department of Food Science and Technology, Florianópolis, SC, Brazil.*
*Corresponding author: rbonomo@uesb.edu.br
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down into smaller components that can be more easily processed by living organisms and provide nutrients to the soil, thereby increasing its fertility and ability to support vegetation. The use of biodegradable materials presents a compelling opportunity to reduce waste accumulation and mitigate environmental pollution, signaling a pivotal shift toward sustainable practices in packaging manufacturing. The use of natural elements in the production of bio-composites not only favors the sustainability of resources but also reduces greenhouse gas emissions, diverging from the petroleum-based origins of traditional plastics.

The pursuit of sustainable and environmentally friendly packaging solutions has spurred a shift from synthetic polymers to natural substitutes. Among these, starch has emerged as a particularly promising biomaterial, thanks to its favorable blend of affordability, widespread availability, thermoplastic characteristics, and high biodegradability. Abundantly found in roots, tubers, fruits, and seeds (Nevoralova et al., 2019; Pelissari et al., 2019; Rincon et al., 2021), starch consists of D-glucose units linked by glycosidic bonds, forming both amylose and amylopectin molecules. Amylose, a linear polymer connected by α1-4 glycosidic bonds, contrasts with the highly branched amylopectin, which exhibits a highly branched configuration, featuring α1-4 bonds in its linear structure and α1-6 bonds in its branched segments (Hong et al., 2016; Rahaman et al., 2021). Among various starch sources, cassava root stands out, with high potential due to its low cost and excellent film-forming properties (Jiang et al., 2020).

Biodegradable films utilizing cassava starch as a polymeric matrix exhibit remarkable attributes including homogeneity, flexibility, transparency, and rapid decomposition (Nascimento et al., 2016). However, it is noteworthy that starch-based films often suffer from poor mechanical properties, elevated solubility, and heightened permeability, factors that might not align with certain film requirements (Rangaraj et al., 2021; Wang et al., 2022). In this context, incorporating plasticizers and other compounds, such as vegetable oils, into biopolymer formulations can help overcome these limitations while increasing their functionality in food packaging applications. This enhancement also results in boosted oxidation resistance and antimicrobial activity, which are highly desirable features for maintaining food freshness and quality and extending its shelf life (Huang et al., 2021; Rangaraj et al., 2021; Wang et al., 2018). One of the widely used plasticizers in starch film preparation is sorbitol, the addition of which improves the flexibility of starch films by reducing hydrogen bonding between molecules and increasing the intermolecular spacing between polymers upon their intercalation into the starch polymer network (Lim et al., 2020).

Another ingredient that can be used for its beneficial structure-forming effect is babassu oil, which is obtained from the fruits of a tropical palm tree native to South and Central America, mainly Mexico, Peru, Bolivia, and Brazil (Hiura & Rocha, 2018). In Brazil, the plant is present mainly in the Northeast and Central-West regions, where it has been exploited sustainably for decades. The palm tree (*Orbignya phalerata* Mart.) can reach between 10 and 30 m in height and has up to five clusters that produce 250–500 fruits (coconuts) (dos Santos et al., 2017). Babassu oil is extracted from the babassu nut, which represents approximately 65% of the total mass of the almond (M. J. F. Silva et al., 2020), giving rise to high-quality oil and by-products known as babassu coconut components, including epicarp, mesocarp, endocarp, and almond cake (Ferreira et al., 2023). Babassu oil is rich in saturated fatty acids (80–91%), especially lauric, myristic, palmitic, capric, caprylic, and stearic acids. The remainder are unsaturated fatty acids (9–20%), where oleic acid and linoleic acid are present (de Oliveira et al., 2016). Due to its resistance to decomposition by hydrolysis or thermal oxidation and its low melting point, which are associated with the high content of saturated fatty acids (around 90%), babassu oil finds application in food production, the cosmetic industry, and pharmaceuticals (Bauer et al., 2020; Pereira et al., 2022).

Biodegradable materials have a broad range of potential applications, particularly in agriculture and food industries, aiming to diminish plastic usage in packaging manufacturing. They also serve to prolong the shelf life of food items by offering an extra protective layer that readily decomposes after product use. With this in mind, the aim of this study was to develop biodegradable films utilizing cassava starch, plasticized with sorbitol and enhanced with babassu oil. The research delved into a comprehensive characterization of produced films encompassing parameters such as film thickness, solubility in diverse solutions, water vapor permeability (WVP), mechanical attributes, thermogravimetric analysis (TGA), Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and biodegradability assessment.

1.1 Relevance of the work

This study is relevant for the development of biodegradable materials that can replace conventional plastics, contributing to the mitigation of the environmental impact caused by the improper disposal of plastics. The incorporation of babassu oil into cassava starch-based films not only uses a renewable natural resource abundant in Brazil but can also give the material improved functional properties, such as greater hydrophobicity and potential antimicrobial activity, expanding its applications in packaging. Furthermore, by valuing regional products such as babassu, this study promotes economic and social sustainability, encouraging the use of local raw materials and innovating in the biodegradable packaging sector.

2 MATERIALS AND METHODS

2.1 Materials

The films were developed using cassava starch as a polymeric matrix, acquired from a local company in Itapetinga, BA, Brazil. Babassu oil (*Orbignya phalerata* Mart.) was kindly provided by the Instituto Chico Mendes de Conservação da Biodiversidade—ICMBio (Florianópolis, SC, Brazil). Sorbitol 70% (Synth, Brazil) was used as a plasticizing agent, and Tween 80 (Synth, Brazil) was used as an emulsifying agent. All reagents used in the analyses were of analytical grade.

2.2 Proximate composition of cassava starch

The total starch content was determined according to the anthrone method, as proposed by Moraes and Chaves (1988).

To determine amylose content, the simplified iodine colorimetric method was used, and absorbance readings were recorded at 620 nm (Martinez & Cuevas, 1989). Amylose content was determined from the analytical curve constructed with potato amylose (Synth, Brazil), while amylopectin was determined by the difference between the amount of starch and amylose present in the sample. The total lipid content of cassava starch was assayed by the method of Bligh and Dyer (1959), using chloroform, methanol, and water as solvents in the proportions of 2:1:0.8. The moisture content was determined by the gravimetric method at 105°C until constant mass; ash content was determined by incineration in a muffle furnace at 550°C; and protein content was determined by the micro-Kjeldahl method, using the factor 5.75 as the nitrogen conversion factor (AOAC, 2006).

2.3 Production of films

Films were produced using fixed concentrations of starch (4%) and an emulsifier (0.072 g). Four babassu oil concentrations were used, along with the control (0, 0.4, 0.8, 1.2, and 1.6 g). For the plasticizer (sorbitol), four concentrations were used according to the treatments, namely, J1 (2 g), J2 (2.4 g), J3 (2.8 g), and J4 (3.2 g), as shown in Table 1.

The films were prepared as described by Farahnaky et al. (2013), with modifications. Figure 1 shows the film processing flow.

Cassava starch, the plasticizer, and the emulsifier were dispersed in 200 mL of distilled water and heated on a magnetic hotplate (LABNET—PC Model 420D, Mexico) under constant stirring. After reaching 95°C, the film-forming solution was maintained at this temperature for 5 min. For films containing babassu oil, the film-forming solution was cooled to approximately 40°C before incorporation of babassu oil. The film solution was poured onto glass plates (22.5 × 32.5 cm) and dried at room temperature of 25°C for 24 h. After drying, the films were stored in desiccators protected from light, containing saturated sodium bromide (NaBr) solution for 24 h.

Table 1. Quantitative combinations of components used for biofilm formulation.

Treatment	Oil	Water	Starch	Emulsifying agent	Sorbitol
	(g)	(mL)	(g)	(g)	(g)
J1	0.0	200	8	0.072	2.0
J1	0.4	200	8	0.072	2.0
J1	0.8	200	8	0.072	2.0
J1	1.2	200	8	0.072	2.0
J1	1.6	200	8	0.072	2.0
J2	0.0	200	8	0.072	2.4
J2	0.4	200	8	0.072	2.4
J2	0.8	200	8	0.072	2.4
J2	1.2	200	8	0.072	2.4
J2	1.6	200	8	0.072	2.4
J3	0.0	200	8	0.072	2.8
J3	0.4	200	8	0.072	2.8
J3	0.8	200	8	0.072	2.8
J3	1.2	200	8	0.072	2.8
J3	1.6	200	8	0,072	2.8
J4	0.0	200	8	0.072	3.2
J4	0.4	200	8	0.072	3.2
J4	0.8	200	8	0.072	3.2
J4	1.2	200	8	0.072	3.2
J4	1.6	200	8	0.072	3.2

2.4 Characterization of films

2.4.1 Film thickness

The thickness of the films was measured using a precision micrometer (Kit B, PANTEC, China) with 10 measurements per sample, including the edges, median, and central points. The final thickness of each sample was calculated as the average of the repetitions.

2.4.2 Solubility in water, saline solution, and acidic solution

Solubility in water, saline solution, and acidic solution was determined according to the methodology proposed by Gontard et al. (1992), with modifications. The initial mass (m_i) of the film (2 cm in diameter) was determined by drying in an oven (TECNAL, Model TE-393/I, Brazil) at 105°C for 24 h and then weighing on an analytical balance (METTLER TOLEDO, Model ME 204, China) to obtain the initial mass. Then, the samples were immersed in 50 mL of distilled water, saline solution (3% NaCl), or acidic solution (3% acetic acid) in an Erlenmeyer flask under stirring at 25°C for 24 h using a shaking table (MA 140/CF) (QUIMIS, Model Q226k, Brazil). After this period, the samples were dried in an oven at 105°C for 24 h and then weighed on an analytical balance to obtain the final mass (m_f). The determinations were performed in triplicate, and solubility (S) was expressed as a percentage of solubilized material, calculated according to Equation 1:

$$S(\%) = \left(\frac{m_i - m_f}{m_i}\right) \cdot 100 \quad (1)$$

2.4.3 Water vapor permeability

WVP was determined gravimetrically according to Gontard et al. (1992), with modifications. Disk-shaped films, 4.5 cm in diameter, were placed in a cell containing silica gel (RH = 0%; 0 Pa vapor pressure), forming a membrane to ensure moisture diffusion exclusively through the films. The cell was placed inside a desiccator containing distilled water (RH = 100%; $0.4297 \cdot 10^4$ Pa vapor pressure), in an air-conditioned room at 22°C, under constant relative humidity and vapor pressure. The cell was weighed on the aforementioned analytical balance every 24 h for 12 consecutive days. Permeability was calculated using Equation 2:

$$WVP = \frac{G \cdot V}{A \cdot T \cdot (P_1 - P_2)} \quad (2)$$

Where:

WVP: the water vapor permeability (g m^{-1} s^{-1} Pa^{-1});

G: the weight gain (g) during 24 h;

V: the average film thickness (m);

A: the permeation area (m^2) of the film;

T: the time (s);

(P_1-P_2): the vapor pressure gradient (Pa) between film surfaces ($0.4297 \cdot 10^4$ Pa).

WVP determination was performed in two repetitions.

2.4.4 Mechanical properties

Mechanical properties, i.e., maximum tensile strength (MPa), percentage elongation (%), and Young's modulus (MPa), were determined in specimens of 100 mm in length and 25 mm in width in a CT3 Texture Analyzer (Brookfield, USA) with a 25-kg load cell, coupled with a TA-DGA Dual Grip Assembly. The strain rate was 0.4 mm s^{-1} and 100% deformation at break. The mechanical properties were determined in sextuplicate according to ASTM (ASTM International, 2012), with modifications.

2.4.5 Thermogravimetric analysis

Thermogravimetric analyses of the films were performed on the STA PT-1000 apparatus (Linseis, Germany). Approximately 20 mg of each sample was placed in an open aluminum oxide crucible under a static air atmosphere. The temperature range was 25–1,000°C, with a heating rate of 10°C min^{-1} (G. M. S. Silva et al., 2020).

2.4.6 Fourier transform infrared spectroscopy

FTIR spectra were measured using a Cary630 FTIR spectrometer (Agilent Technologies Inc., Santa Clara, CA, USA). The readings were taken in the mid-infrared region from 4,000 to 400 cm^{-1} with a resolution of 4 cm^{-1} using an attenuated total reflectance and deuterated triglycine sulfate detector, at 25°C. Data were analyzed using the Microlab and Resolution Pro software by Agilent (Santa Clara, USA) (G. M. S. Silva et al., 2020).

2.4.7 X-ray diffraction

Diffraction patterns of the films were determined using a Bruker D2 Phaser X-ray diffractometer (Bruker AXS, Karlsruhe, Germany) applying the continuous-scan method (G. M. S. Silva et al., 2020), with an operating voltage of 30 kV and 10 mA. The test was performed at 25°C with a 2φ angle between 5° and 50°, and 1,600 V.

2.4.8 Biodegradability

The biodegradability of the films was evaluated by weight loss, in duplicate, following the method proposed by Chandra and Rustgi (1997). Samples of 4 cm^2 were cut, weighed, and buried in pots containing soil at a depth of 15 cm. Samples were retrieved from the soil after 7, 15, and 30 days to assess film degradation.

2.4.9 Experimental design and statistical analysis

Response surface methodology (RSM) is a statistical technique adept at modeling and optimizing complex systems. RSM has proven invaluable in refining critical variables affecting film properties. Through a meticulously designed sequence of experiments and subsequent data analysis using RSM, research was conducted to determine ideal manufacturing parameters for the formulation of composite films while measuring the impact of each variable on their properties. This systematic approach enabled an efficient exploration of formulation and characterization processes in composite films.

The experiment was performed in a 5x4 full factorial experimental design with five levels of babassu oil concentration (0.0, 0.4, 0.8, 1.2, and 1.6 g) and four levels of plasticizer (sorbitol) concentration (2.0, 2.4, 2.8, and 3.2 g). The mass of oil and plasticizer was calculated over the mass of starch used. The results

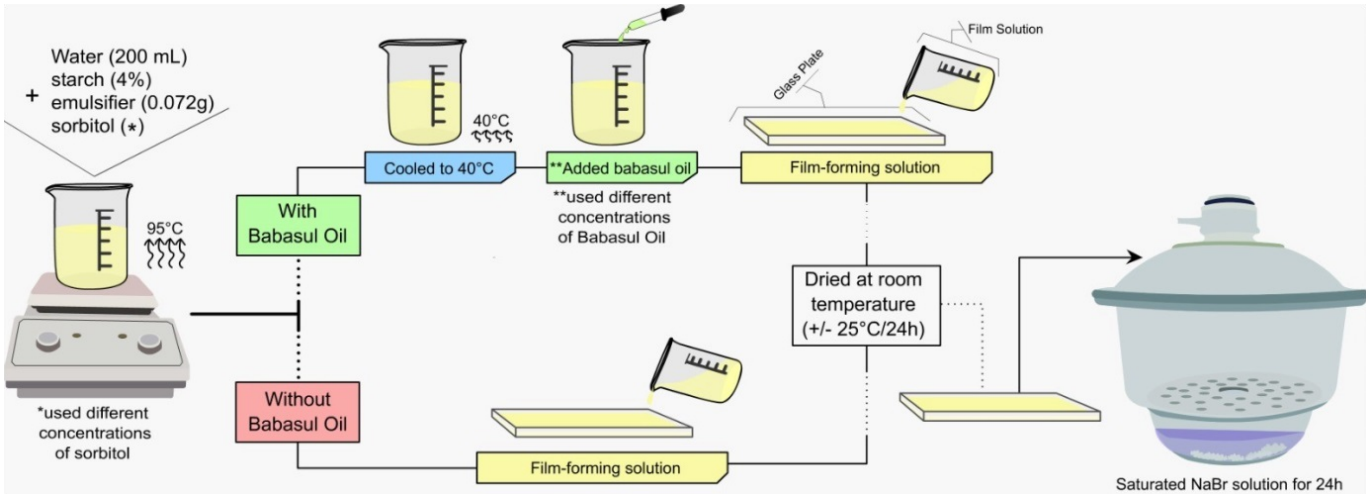

Figure 1. Processing steps for films based on cassava starch incorporated with babassu oil.

were subjected to multiple regression analysis according to the polynomial equation shown in Equation 3:

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i \cdot x_i + \sum_{i=1}^{k} \beta_{ii} \cdot x_i^2 + \sum_{i<j} \sum \beta_{ij} \cdot x_i \cdot x_j + \varepsilon \quad (3)$$

Where:

Y: the response variable;

β_0, β_i, β_{ii}, and β_{ij}: the overall constant process effect, the linear, quadratic, and interaction effects between X_i and X_j, respectively;

ε: the error.

Model fit was determined by model and parameter significance ($p < 0.05$), lack of fit ($p > 0.05$), and determination of the regression coefficient (R^2). All statistical analyses were performed in the SAS Student Statistical Package (SAS Institute Inc., Cary, NC, USA).

3 RESULTS AND DISCUSSION

3.1 Proximate composition of cassava starch

Table 2 shows the results of the proximate composition of cassava starch. For the production of biodegradable films, three constituents are considered relevant, including amylose, ash, and lipid contents, once they have a direct impact on the final product.

According to Mendez et al. (2022), the amylose content of cassava starch is closely dependent on the cultivar and soil management. In this study, amylose content was 20.57%, which was lower than that found by Mendez et al. (2022). In turn, Rolland-Sabaté et al. (2012) reported 16.8–30.3% amylose in some cassava varieties. Amylose corresponds to the linear portion of the starch, formed by glucose units that are linked by α-(1-4) bonds. In turn, amylopectin is the branched fraction, with branching points attached to α-(1-6) and linear regions of glucose units attached to α-(1-4). The proportion of these segments can determine the properties of the resulting starch-based film (Liu et al., 2009). Thus, amylose mainly contributes to the amorphous phase and favors film formation. In contrast, amylopectin molecules arranged in clusters form crystalline regions and produce weak films (Saberi et al., 2017; Thakur et al., 2019). The cassava starch used in this study showed an average amylose content (20.57%), which produced films with a continuous matrix, thus allowing diverse applications. The ash content or fixed mineral residue and lipid contents of the samples can be considered low, which makes cassava starch a good base for the formation of a polymeric matrix for biodegradable films. For film production, a high ash content can hinder the formation of bioplastics due to the potential interaction of these compounds with the constituents of the biodegradable film (Santana et al., 2018). A high lipid content in starch may cause the fixation of gel color and consequently interfere with the color of the resulting films, in addition to changes in aroma and formation of complexes (Santana et al., 2018).

Table 2. Proximate composition of cassava starch on a dry basis.

Constituents	Concentration (%) ± SD
Starch	85.47 ± 1.06
Amylose	20.57 ± 0.28
Amylopectin	64.49 ± 1.06
Moisture	8.50 ± 0.02
Ash	0.32 ± 0.02
Protein	1.12 ± 0.01
Lipid	0.05 ± 0.00

SD: standard deviation.

3.2 Characterization of biodegradable films

3.2.1 Thickness and solubility

The average thickness of the films was 0.121 ± 0.015 mm. According to Jiménez et al. (2012), film thickness should be as homogeneous as possible. Although the effect of sorbitol concentration on the films was not evidenced in this study, in general, high plasticizer concentrations lead to the formation of thicker films, since plasticizers can rapidly penetrate the starch network, increasing the interstitial space of the polymer chain (Edhirej et al., 2017). Zhang et al. (2016) evaluated the effect of glycerol and sorbitol on the production of films, using gum ghatti for the formation of the polymeric matrix. The authors reported that the films plasticized with sorbitol were thicker (0.038–0.068 mm) when compared to those plasticized with glycerol (0.033–0.047 mm), probably due to the size of sorbitol molecules, which penetrate the polymer chain faster thanks to the six free hydroxyls, thus providing stronger and thicker materials. Solubility in water, acidic solution, and saline solution of the films were 78.91 ± 24.17%, 70.44 ± 22.21%, and 98.05 ± 8.47%, respectively, which represent high solubility. This property is related to the hydrophilicity and hygroscopicity characteristics of the compounds in the film formulation (Hadi et al., 2021), and knowledge of these characteristics is relevant for better application of the film as food packaging. Highly soluble films, such as those obtained in this study, can be used in food or medicine as edible packaging, i.e., consumed together with the food, dissolving quickly in the mouth, thus facilitating digestion (Ballesteros-Mártinez et al., 2020).

3.2.2 Water vapor permeability

The WVP of the biodegradable films was affected by the plasticizer and babassu oil contents. Equation 4 describes the behavior of WVP as a function of oil (B) and plasticizer (P) concentration:

$$WVP = -1.6716 \cdot 10^{-6} \cdot B + 3.0414 \cdot 10^{-5} \cdot \cdot P + 9.6880 \cdot 10^{-6} \cdot B^2 - 4.2128 \cdot 10^{-6} \cdot P^2 \quad (4)$$
$$R^2 = 0.755$$

An increase in both oil and plasticizer concentrations led to an increase in WVP, with a maximum value at the maximum concentrations of 1.6 g of oil and 3.2 g of plasticizer within the studied range, as shown in Figure 2.

In general, the addition of oils to the film-forming matrix reduces the water absorption of films, since these compounds

are complex and highly hydrophobic mixtures. Thus, WVP decreases with the addition of a higher hydrophobic fraction to the film, with water vapor transfer due to the hydrophilic portion of the film (Caetano et al., 2018). However, this behavior was not evidenced in this study, probably due to the shrinkage of sorbitol and babassu oil used in the formulations.

3.2.3 Mechanical properties

The stress–strain curve of the cassava starch film incorporated with babassu oil is shown in Figure 3, representing the J4 1.2 g film attraction test, whose behavior was similar in all treatments. As shown in the figure, the increase in stress is linear, with low deformation, up to approximately 1.5 MPa. Then, the tension remains constant until the film ruptures under a higher tension, around 3 MPa.

No significant model was found for the mechanical properties of the produced films. These results suggest that the range of used additives (babassu oil and sorbitol) had no notable impact on this parameter. The maximum tensile strength observed in the study was 4.78 ± 2.75 MPa. These results exceeded those reported by Caetano et al. (2018), who evaluated films made with cassava starch, glycerol, and/or OEO, and minimally processed zucchini residue extract, and recorded tensile strength in the range of 0.32–1.07 MPa. In turn, Santana et al. (2018) studied films of jackfruit starch and glycerol, and Bonomo et al. (2018) evaluated jackfruit starch films incorporated with lysozyme, and they also found lower maximum tensile strength values when compared to the present study. Galus and Kadzinska (2015) pointed out that the chemical characteristics of oil, such as fatty acid composition, size, and chain saturation, can be determining factors for the mechanical behavior of films. In addition, the mechanical properties of biodegradable films are affected by various factors, including molecular weight, size, content, and the nature of applied plasticizers (Orsuwan & Sothornvit, 2018). Plasticizers are used in the production of films to inhibit intermolecular forces between film constituents, increasing mobility and thus providing flexibility and extensibility (Al-Hassan & Norziah, 2012). The mean percentage elongation of cassava starch-based films in the present study was 183.13 ± 75.25%, which was higher than that reported by Bonomo et al. (2018). Regarding Young's modulus, an average value of 5.97 ± 2.79 MPa was recorded. Caetano et al. (2018) reported higher values for films made with no addition of oil. Jaramillo et al. (2015) observed that cassava starch-based films made with yerba mate extract and glycerol showed low Young's modulus values (0.42–1.9 MPa). The most desired mechanical properties of films include resistance to rupture and flexibility to adapt to deformations. The results of the present study can expand potential applications for these films.

Sikora et al. (2021) point out that the mechanical properties of the films differ significantly from those of traditional petrochemical materials such as polyethylene, polypropylene, and polyethylene terephthalate. They are either too brittle or rigid, or their tensile strength is too low to successfully replace traditional film materials. While there are notable differences in mechanical properties, biodegradable films are continually being developed and improved to reduce these disparities so they can compete more directly with traditional plastics while maintaining environmental advantages (Collazo-Bigliardi et al., 2019; do Val Siqueira et al., 2021; Sabetzadeh et al., 2015; Sikora et al., 2021; Zhong et al., 2020).

3.2.4 Thermal analysis

From TGA and derivative of TGA (DTG) of cassava starch and babassu oil-based films the thermal properties, especially temperature stability were studied. The TGA of cassava starch films incorporated with babassu oil and sorbitol and the control film is shown in Figure 4. The TGA curves showed similar behavior of the films regardless of the incorporation of oil and

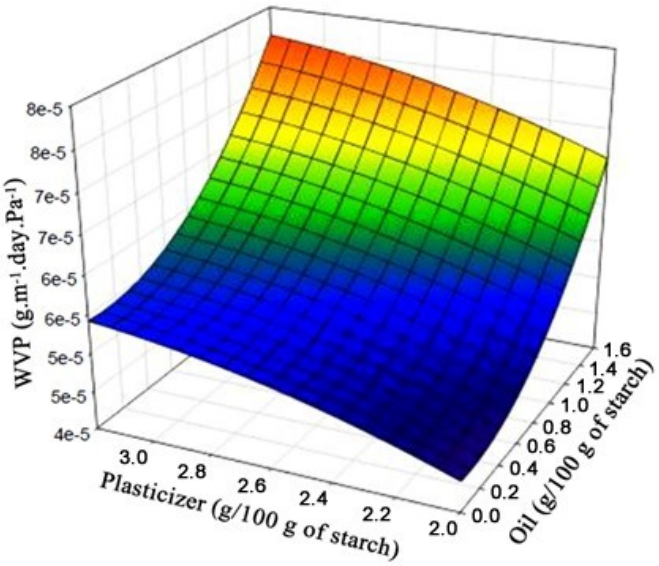

Figure 2. Changes in water vapor permeability of cassava starch-based films depending on quantities of babassu oil and plasticizer (sorbitol). For some types of foods, e.g., dehydrated foods, the use of packaging with high WVP is not preferable; however, high permeability is ideal for packaging fresh foods such as vegetables (Bonomo et al., 2018). Caetano et al. (2018) developed active cassava starch-based films with an addition of minimally processed pumpkin residue extract (0–6%) and oregano essential oil (OEO) (0–2%) and reported that the lower the plasticizer (glycerol) content, the lower the permeability value.

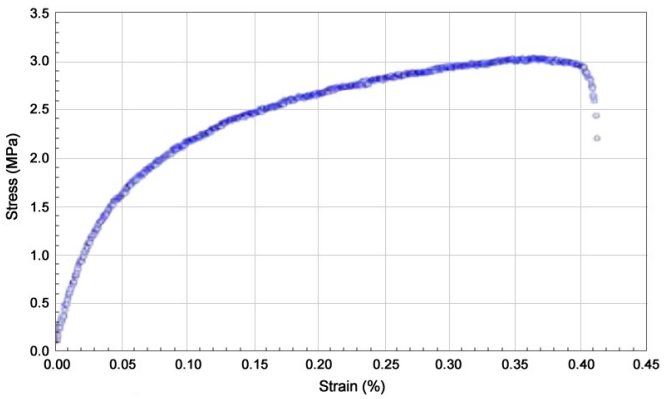

Figure 3. Stress–strain curve of the film J4 1.2 g.

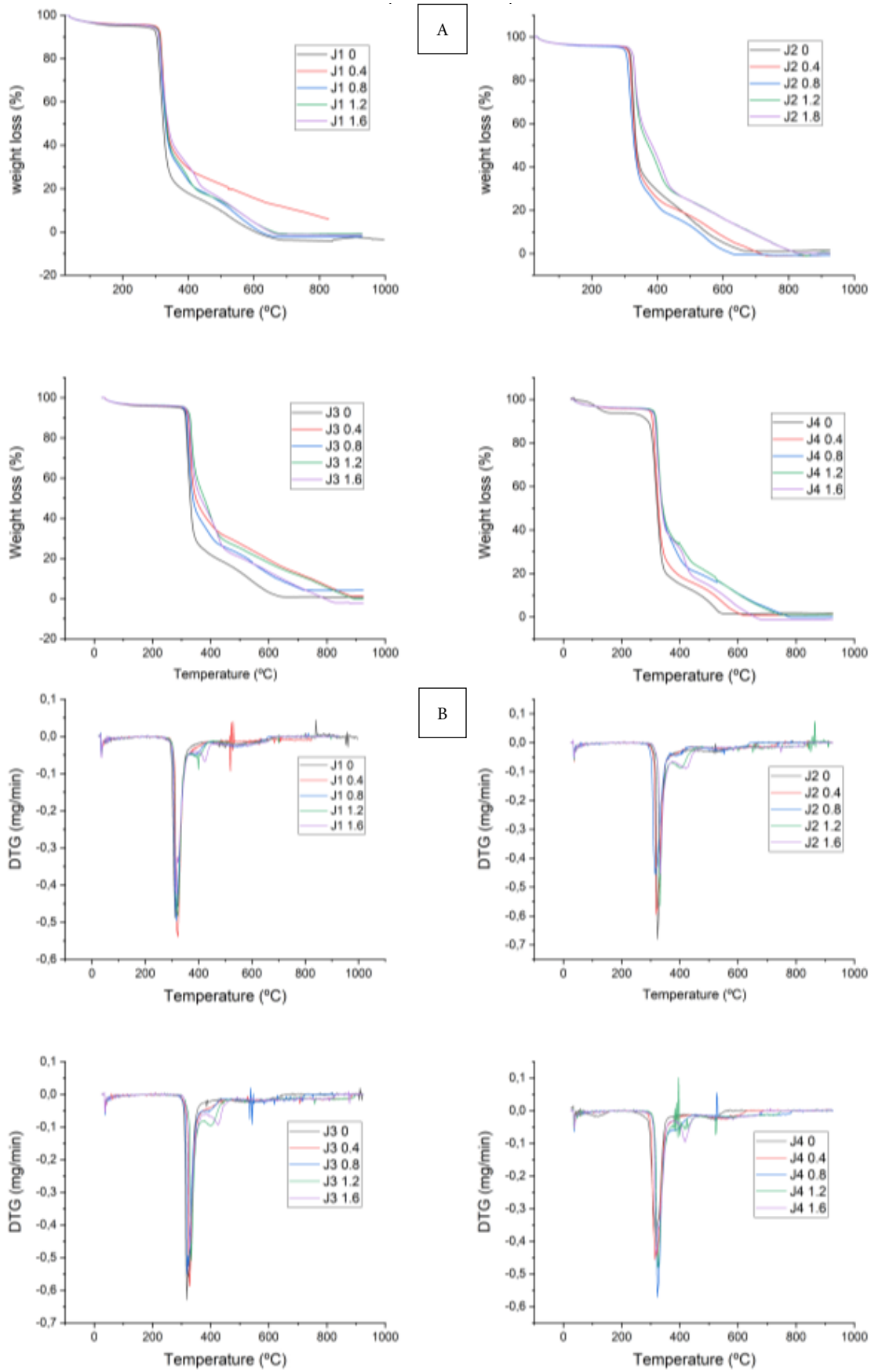

Figure 4. (A) Thermograms of cassava starch-based films enriched with babassu oil plasticized with sorbitol. (B) DTG of cassava starch-based films enriched with babassu oil plasticized with sorbitol.

plasticizer, with three stages of weight loss, as described in the literature for starch-based films, while lower thermal stability was observed for all the treatments without the addition of babassu oil, except for films made with 2.4 g of sorbitol (J2). The first stage of weight loss occurred from 30 to 95°C, probably due to the loss of free water present in the films. According to Costa et al. (2023), the percentage of mass loss at these stages depends on the moisture level of samples. The second stage of weight loss was observed in the temperature range between 253 and 379°C and was associated with volatile matter, decomposition of starch and plasticizer, and loss of water bound to the polymer matrix (Shen & Kamdem, 2015). This was the main stage of mass loss, representing around 69%. In addition, the determination of the initial degradation temperature of biodegradable films is important for the evaluation of their thermostability and consequently leads to a better application of them. According to the results obtained, it can be predicted that the developed films have good thermal stability when compared to others obtained in similar studies of biodegradable films by starch, mucilages, and other plant compounds (Costa et al., 2023; Jasem odhaib et al., 2024; Mannai et al., 2023).

The last thermal event occurred from 380 to 467°C, and it can be related to starch decomposition due to the breaking of amylose and amylopectin bonds in the polymer (Zhang et al., 2018), with residual mass ranging from 0.32% (J2) to 2.85% (J1). Caetano et al. (2018) reported higher values than those found in the present study for the residual mass of the films, which ranged from 6.5 to 9.5%, while the highest percentage corresponded to the film made with the highest concentration of OEO. It is worth noting that the residual mass in the TGA depends on several factors, including inorganic compounds and impurities of starch, the nature of additives, and the conditions of analysis in an inert atmosphere (N_2), which may prevent complete combustion of organic compounds (Perazzo et al., 2014). According to the DTG graph, the most pronounced mass loss for all films was at temperature values around 327°C, which may be an indication that these films exhibit relatively higher resistance to heat exposure, as well as a strong interaction between their constituents.

TGA and DTG are effective techniques for evaluating biodegradable films, mainly used to understand and predict the thermal and oxidative stability of samples (Jafarzadeh & Jafari, 2021). The TGA thermogram allows the understanding of thermal events that involve loss or gain of mass, in addition to the identification of the compounds that make up the materials. DTG, in turn, allows for a more precise identification of these thermal events. These techniques provide detailed information on decomposition temperatures and degradation steps, allowing the optimization of formulations for improved thermal performance (Haykiri-Acma et al., 2010; Kuprianov & Arromdee, 2013). Additionally, they help develop films that are durable during use and environmentally sustainable after disposal, balancing performance needs with sustainability considerations.

3.2.5 Fourier transform infrared spectroscopy and X-ray diffraction

The films were submitted to FTIR in the absorbance mode, and their spectra are shown in Figure 5. A similar behavior was observed for all the films, thus showing that differences in the concentrations of the components caused no chemical changes in the material. However, the films containing 2.4% sorbitol exhibited a lower peak intensity.

The FTIR spectra provide information concerning chemical groups and their vibrational state, related to changes in the chemical composition of the raw materials (Garcia-Salcedo et al., 2018) and their interaction in the films. A broadband was identified at 3,274.27 cm^{-1} for all the films, with lower intensity for the J2 films. Peaks at 3,600–3,000 cm^{-1} represent the stretching vibration of the O–H group, while the region from 2,850.10 to 2,925.14 cm^{-1} may be related to asymmetric and symmetric C-H stretching, respectively (Wang et al., 2018). The peak at 1,643.49 cm^{-1} corresponds to the stretching vibration of C¼O (amide I) (Dankar et al., 2018) and water molecules in the amorphous region (Kizil et al., 2002). The region between ~1,120 and 900 cm^{-1} is generally recognized in carbohydrates and polysaccharides, with band intensity related to the degree of hydration of the sample (Costa et al., 2020), while vibrations at 1,147.75 to 1,078 cm^{-1} are assigned to the C–O–H bonds of starch. In these spectra, the most defined peak common to all the films was observed at 990.57 cm^{-1}, which confirms the polysaccharide characteristic of the filmogenic matrix, representing typical stretching of C–C–H bonds (Barizao et al., 2020).

Figure 6 shows diffractograms of cassava starch-based films incorporated with babassu oil and plasticized with sorbitol and the respective crystallinity index.

Crystalline zones were observed in the material studied, with peaks located at approximately 19.3 and 30º, except for films J1 1.2 (2 g sorbitol and 1.2 g babassu oil) with an absence of peaks, indicating their amorphous character. The peak around 19° is characteristic of type C and has intermediate crystallinity. This peak is attributed to the recrystallization of amylopectin during film storage. Over time, starch molecules can reassociate into crystalline or "retrograde" segments (Medina-Jaramillo et al., 2020; Wu et al., 2009). Thus, the crystallinity of starch films results from the crystallization of amylose, which occurs in the initial phase of film formation, and the crystallization of amylopectin, which is slower and occurs during storage (Ghasemlou et al., 2013). Films with this type of crystalline structure have bonds with intermediate strength intensity, resulting in lower resistance to hydrolysis and, consequently, greater degradability. However, this characteristic also affects the mechanical resistance of the material, resulting in lower stiffness and greater elasticity, as demonstrated by the results of this study. La Fuente et al. (2019) studied native cassava starch films subjected to the ozonation process and reported peaks of 16.8 and 19.2º for native cassava starch-based films. Lim et al. (2020) investigated the effects of plasticizers on physicochemical properties, crystallinity, and heat sealing of cassava starch films using glycerol and sorbitol at the ratios of 4:0, 3:1, 2:2, 1:3, and 0:4, respectively. X-ray diffraction revealed a decrease in crystallinity with an increasing sorbitol content, indicating an association between plasticizer content and film crystallinity. In the present study, film J3 (1.6) showed a higher degree of crystallinity, which is represented by the highest peak at 19.3°.

Maniglia et al. (2017) developed films based on starch and babassu mesocarp flour, using glycerol as a plasticizer. Those

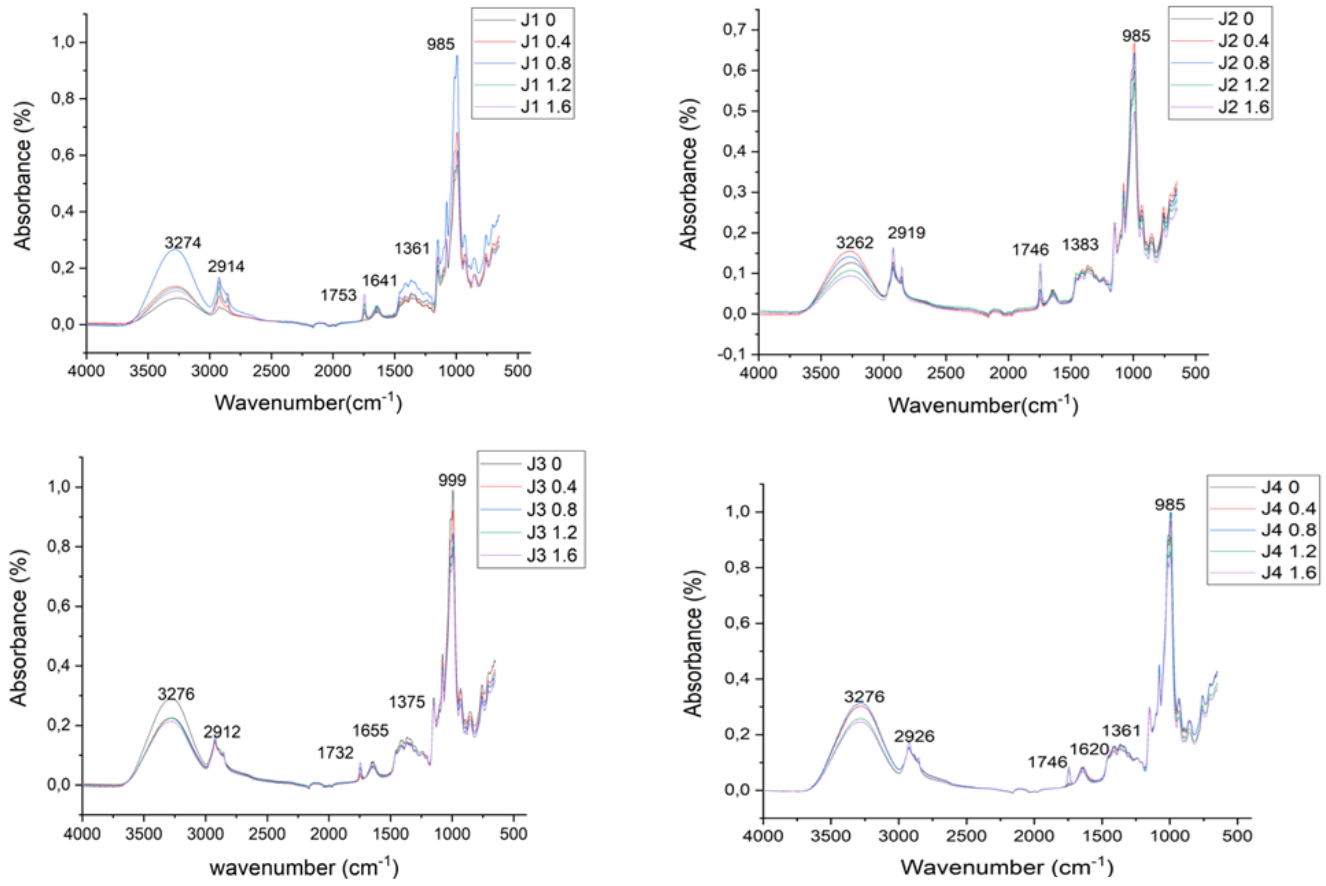

Figure 5. FTIR spectra recorded for biofilms (J1), i.e., cassava starch-based films enriched with 2% sorbitol and 0.0, 0.4, 0.8, 1.2, and 1.6% babassu oil.

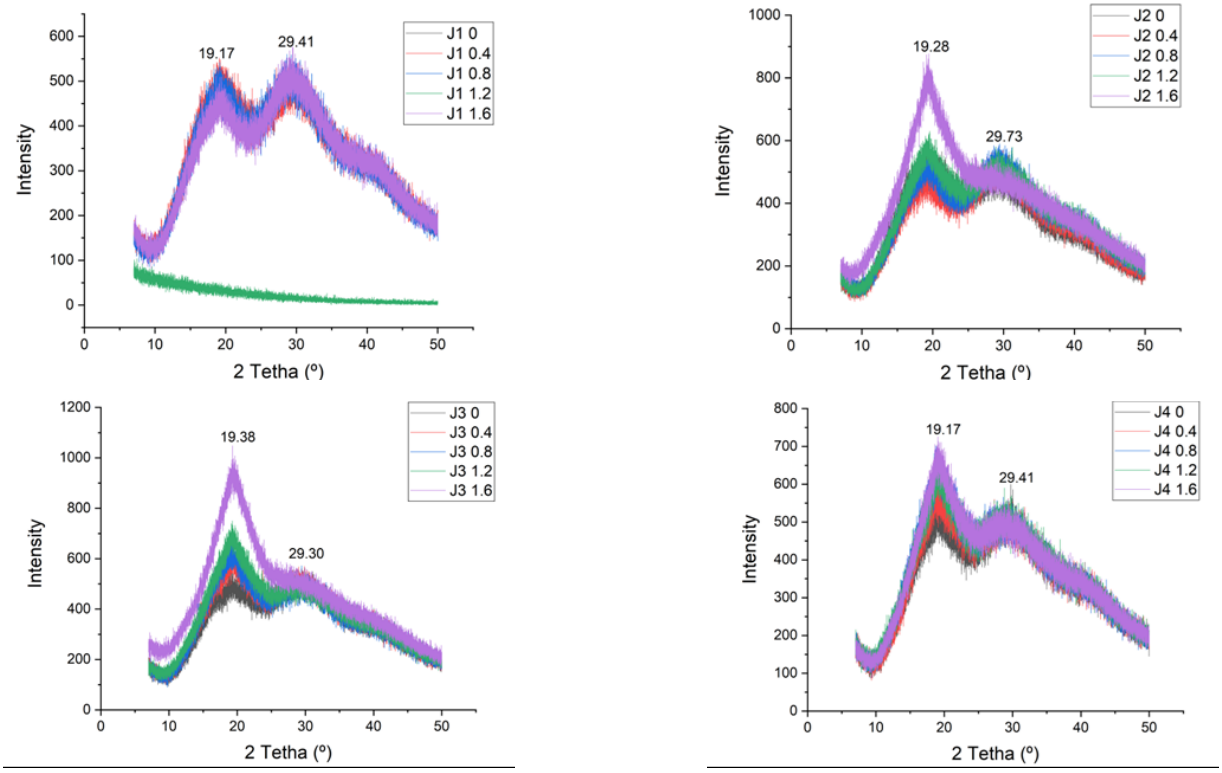

Figure 6. The diffractogram of cassava starch-based films with babassu oil plasticized with sorbitol.

authors reported that all starch-based films exhibited peaks at 15, 17, 19, and 22°, respectively (2θ), characterizing them as type B starch. Jimenez et al. (2012) pointed out that amylose and amylopectin molecules are rearranged during film formation, while hydrogen bonds are known to form during reassociation. Thus, the crystallinity of starch-based films depends on the ability of the chains to form crystals, as well as the mobility of the chains during the crystallization process (Rindlav-Westling et al., 1998), drying and storage conditions (temperature and relative humidity), and the content of plasticizer used to produce the films.

3.2.6 Biodegradability

The films were completely degraded within 7 days of analysis, proving to be an effective alternative to replace non-biodegradable polymers. A similar result was observed by Jaramillo et al. (2015) in cassava starch-based films with yerba mate extract, which exhibited rapid biodegradability in 12 days, demonstrating their usefulness in developing biodegradable packaging and replacing packaging from non-biodegradable polymers. Seligra et al. (2016) also observed rapid degradation in the first 15 days, with significant degradation at the end of 30 days. Biodegradable films made from starch and glycerol, known for their hydrophilic nature, show significant weight loss during the biodegradation process. This occurs as a result of the proliferation of natural microorganisms in the soil (bacteria and fungi), which, through the production of degradative enzymes, decompose the constituents of the films into simpler compounds, which are then metabolized by the microorganisms, producing carbon dioxide, water, and biomass that is returned to nature through the biocycle process (Onyeaka et al., 2022; Seligra et al., 2016). The combination of these actions results in a significant weight reduction.

The rapid degradation of the films in just 7 days demonstrates their high effectiveness as alternatives to non-biodegradable polymers, which can persist in the environment for decades or even centuries. This degradation efficiency indicates that biodegradable films contribute significantly to reducing the negative environmental impact caused by the accumulation of plastic waste.

However, films made exclusively from starch as a polymeric matrix present some challenges, as they produce films with poor mechanical properties, low water stability, high sensitivity to humidity, the presence of a weak moisture barrier due to strong hydrophilic behavior, and brittle behavior at room temperature and its high moisture content (Thakur et al., 2018). These properties can be improved with the modification of starch, the production of films with more than one polymeric matrix, and also the inclusion of hydrophobic compounds and others (Onyeaka et al., 2022).

4 CONCLUSIONS

Cassava starch-based films enriched with babassu oil using sorbitol as a plasticizer were produced and provided promising results. Babassu oil contributed to the formation of an amorphous and thicker film structure. In addition, the films showed good water vapor barrier properties and adequate mechanical strength when sorbitol was used as a plasticizer. The addition of oil strengthened the polymeric matrix, increasing the modulus of elasticity and tensile strength (maximum tensile strength: 4.78 ± 2.75 MPa and percentage elongation: 183.13 ± 75.25%). The films showed rapid biodegradability (100%), proving to be an effective alternative to replace non-biodegradable polymers, thus avoiding inappropriate disposal of these materials in the environment.

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