Physical-chemical, thermal and mechanical performance of a polymer blend based on babassu coconut mesocarp starch

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ABSTRACT
The objective was to characterize the physical-chemical, thermal and mechanical properties of a polymer blend based on babassu coconut mesocarp starch, with the addition of glycerin, melamine/formaldehyde. The babassu coconut mesocarp was used as raw material and the plasticizers glycerin, melamine/formaldehyde were added. Thermogravimetric analysis (TGA), Differential Scanning Calorimetry (DSC), Fourier Transform Infrared (FTIR), Visible Ultraviolet (UV-Vis), X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and traction. The polymer blend showed non-homogeneous distribution with irregular and rough undulations, small differences in color and melamine concentrates, presence of starch residues, without pores and fissures. TGA revealed that the degradation ranges of starch and most organic compounds ranged from 210 ºC to 400 ºC. The DSC showed a predominance of endothermic events. The FTIR identified the starch present and the peaks of melamine and glycerin compounds. UV-Vis showed maximum lengths between 220 and 247 nm, and an accentuated shoulder at 281 nm referring to the electronic transitions of the double bonds present in melamine and starch. The blend is amorphous, with points of crystallinity due to starch and/or overlapping with melamine. The tensile mechanical behavior was of the ductile type. It is concluded that the proposed methodology for the formation of a polymer blend based on babassu coconut mesocarp starch showed good results for physical-chemical, mechanical and thermal properties, being an innovative product with possible potential for different applications.

Keywords: Biotechnology, Biofilm, Polymer blend, *Orbignya phalerata*.

1 INTRODUCTION

The palm tree *Orbignya phalerata* Mart. (babassu coconut) is a member of the palm family, present in several countries in Latin America. In Brazil, it is found most frequently in the North,

Northeast, and Midwest regions, mainly concentrated in the states of Maranhão, Tocantins, and Piauí (Da Silva *et al*., 2021; Da Silva Magalhães *et al*., 2022). This species plays an important social, economic, cultural, and environmental role, due to the diversity of products and by-products that can be used for edible and industrial purposes, especially the manufacture of soap, glycerin, paper, handicrafts, cosmetics, fuel, house covering, among others (Vale, *et al*. 2018; De Carvalho *et al*., 2020).

The babassu coconut is made up of three main parts: a fibrous outer layer that is called the epicarp, the middle layer of the mesocarp, and the inner layer of the endocarp, in which the almonds are inserted (Dos Santos *et al*., 2021). The mesocarp is light in color and can be easily reduced to dust and as it ages, it acquires woody stiffness and a reddish-brown color (Zuniga, 2013).

After the mesocarp is extracted, pulverized and resuspended in water, it can be used as a food supplement (Silva *et al*., 2019). Several studies also report its use in the treatment of infectious diseases, due to its proven anti-inflammatory and analgesic properties, being indicated as a healing agent (Batista *et al., 2006), in the treatment of menstrual pain (Sudre* et al., 2015), antitumor (Rennó et al., 2008), antithrombotic (De Lima et al., 2020*), antimicrobial (Barroqueiro* et al., 2016) and in the treatment of rheumatic diseases (Azevedo *et al*., 2007).

As it is an important source of starch, the babassu mesocarp has also been widely used, due to its physicochemical properties, as a thickener, stabilizer, and regulator of functional properties in the body (Da Silva *et al*., 2021). Studies also show its use for the development of biodegradable healing films (Araruna et al., *2021), in the treatment of chronic injuries (Martins* et al., 2006) and as an antioxidant, due to the presence of phenolic compounds (Nascimento et al., 2006).

Starch consists of two anhydroglucose polymers that are organized in a semicrystalline granular structure, with particle sizes ranging from 1 to 100 μm in diameter, being amylose and amylopectin. Amylose is characterized by being a linear homopolymer of α-D-glycopyranose units, joined by glycosidic α-1,4 bonds. Amylopectin, on the other hand, has a branched structure consisting of linear α-D-glucose chains joined by glycosidic bonds α-1.4 and α-1.6 (Farias *et al*., 2019).

In this context, the development of more efficient processes for the study of blends based on babassu coconut mesocarp starch has great biotechnological potential and has been widely used in the pharmaceutical and biomedical industries, due to its high availability, easy access, low cost, important physicochemical properties, such as biocompatibility, biodegradability and non-toxicity, in addition to being favorable for sustainability and biodiversity conservation (Santos, 2021; De Oliveira *et al*., 2022). In the production of these biopolymers, plasticizing agents, such as glycerin and melamine/formaldehyde, can be added to increase elongation and obtain a filmogenic dispersion (Grossman et al., 2007; Espinoza Ñaupari & Oscco Villegas, 2022). The material generated after processing can be analyzed by means of: i) physical tests, such as Scanning Electron Microscopy (SEM); ii) thermal, such as the Thermogravimetric Analysis (TGA) and Differential Scanning

Calorimetry (DSC) method; iii) chemicals, such as Fourier Transform Infrared (FTIR), Visible Ultraviolet (UV-Vis) and X-ray Diffraction (XRD); and iv) mechanical strength, such as traction.

TGA is a technique used to evaluate chemical, physical, and structural changes in a material due to a change in temperature by monitoring the reactions that occur during the heating process (Nurazzi *et al*., 2021). In this way, it detects the modification of the mass as a function of temperature and time, in addition to the carbonized residue, providing information on the thermal stability, oxidation and combustion of compounds.

DSC is a method that measures the thermodynamic properties of biomolecules and nanometer-sized materials by establishing a connection between temperature and the specific physical properties of substances. The instrument used for this technique determines the temperature and heat flux associated with material transitions as a function of time. Therefore, it measures an amount of heat that is irradiated or absorbed excessively, based on the temperature difference between the sample and the reference material (Naziris *et al*., 2021).

FTIR spectroscopic imaging is a promising technique to provide information on the total chemical composition of the material, in which for biological materials the macromolecular content of the sample, such as proteins, lipids, nucleic acids, and carbohydrates, is evidenced. For non-biological materials, such as polymers, FTIR spectra can be used to determine organic functions or inform about chemical changes through functional group analysis (Wang & Wang, 2021).

UV-Vis spectrophotometry is a suitable method to excite and ionize the medium when interacting with polymeric materials. One of the effects of this interaction is the breakdown of chemical bonds and/or the formation of free radicals, which can lead to cross-linking and conjugation of macromolecular chains. One can then observe the changes in the electronic structure, optical properties, and microscopic characteristics of the polymer surfaces (Sousa *et al*., 2019).

SEM is used to observe and analyze the visual characteristics of materials. This procedure provides three-dimensional and high-resolution images, demonstrating information about the structure, morphology, and topography of the sample's surface, and has been used in recent years as an important tool for the study of microstructures (Courson *et al*., 2021).

XRD consists of the emission of electrons at a target, generating the diffraction of energy photons in the order of X-rays capable of producing constructive interference at specific angles. This interaction makes it possible to measure crystallographic planes and the degree of crystallinity in materials (Silva *et al*., 2022). On the other hand, the mechanical strength of a material reflects the relationship between its response or deformation to a force that is being applied, being verified by methods that reproduce these conditions as closely as possible. Thus, among the factors to be considered are the nature of the force used and the duration of its application (Azevedo *et al*., 2016).

Thus, the present study aimed to characterize the physicochemical, thermal and mechanical

properties of a polymeric blend based on the starch of the babassu coconut mesocarp, composed of glycerin, melamine/formaldehyde.

2 METHODOLOGY

2.1 MATERIAL FOR ANALYSIS

The mesocarp of the babassu coconut was used as raw material, a product in powder form from the state of Maranhão, acquired directly from the company União Cultural de Arari, under the commercial name of Mesovital. The descriptions of the nutritional characteristics per 100 g, previously identified on the label of the industrialized product, were as follows: 245.41 Kcal, 70.11 g of carbohydrates, 0.74 g of fiber, 0.20 g of lipids and 1.65 g of proteins.

2.2 OBTAINING THE STARCH AND POLYMER

To obtain the commercial starch, 100 g of babassu coconut mesocarp was washed with 400 mL of distilled water, decanted it in a room atmosphere and then placed in an oven to dry for 24 h at a temperature of approximately 37 ºC. The average yield was around 35 g of mesocarp starch.

For the polymeric blend, masses of 0.3 g of melamine and 0.3 g of formaldehyde were measured, followed by 25 mL of distilled water, followed by controlled heating at 60 ºC in a water bath and under constant agitation until complete dissolution. Once this was done, a solution containing 3.0 g of babassu mesocarp starch, 0.9 g of glycerin, 25 mL of distilled water was added, and heating (approximately 80 ºC) was taken to a water bath under constant agitation until the polymer was formed, which lasted an average of 10 minutes. Subsequently, the solution was distributed on a plate and had a mass of 31.191 g. After drying using an oven under controlled temperature, the film was obtained from the starch of the babassu mesocarp, in triplicate. These procedures were performed at the Multidisciplinary Laboratory of UniFacid Wyden.

2.3 THERMAL TESTS

The thermal tests, TGA/DTG and DSC, were carried out at the Laboratory of Materials Treatment – Corrosion and Plasma (LabTrat), of the Federal Institute of Piauí (IFPI). The TGA and DTG procedure in the polymeric blend of starch from the babassu coconut mesocarp was conducted in a Shimadzu thermobalance, model TGA-51, using platinum crucibles. The mass of the samples was 3.56 mg in a nitrogen atmosphere at 50 mL.min-1 and an increasing heating rate of 10 °C.min-1, from 24 to 1000 ºC (Mothé & Azevedo, 2009).

The DSC in the polymeric blend of this research was performed in Shimadzu equipment, model DSC 60 PLUS, using aluminum crucibles. Sample masses of approximately 3.5 g were submitted in a

nitrogen atmosphere with a flow rate of 50 mL.min-1 and a heating rate of 10 °C.min-1, from 25 to 601 ºC (Watson et al., 1964).

2.4 CHEMICAL TESTS

The FTIR spectra of the samples were recorded in a Bruker spectrophotometer, *model* Vertex *70 (*Bruker *Optics) with a Total Attenuated Reflectance (ATR) device and using* the OPUS software. The analyses were performed in the wavelength range of 4000-500cm-1, with a resolution of 4cm-1 and 64 scans.

The method for the determination of XRD was performed in *an Empyrean PANalytical* equipment, using copper K radiationα (Cu) of 1.54 Å, voltage of 40 kV and current of 45 mA. The procedure was conducted in a continuous scan range of 4º to 90º, with a scan size of 0 to 0.02626, and *time per step* of 97.665 s.

As for UV-Vis, the absorption spectra of the blend were identified in Thermo Scientific's *Genesys* UV-Vis model 10S dual-beam *spectrophotometer*. The scan was performed in a spectral window from 200 to 1100 nm. FTIR and UV-Vis were conducted at the Bioelectrochemistry Laboratory of the Federal University of Piauí (UFPI) and XRD at IFPI's LabTrat.

2.5 PHYSICAL TESTING

The micrographs were obtained by a high-vacuum scanning electron microscope, model 440, Leo brand, equipped with Energy Dispersive Spectroscopy (EDS), used to observe the morphology of the surfaces of the samples of polymeric blend based on the starch of the babassu coconut mesocarp. As preparation, all samples of the polymeric blend were placed on a double-sided carbon adhesive tape and grounded with carbon ink fixed on an aluminum substrate (a kind of support to place samples in the instrument for analysis). Then, the surface of the samples was coated with gold nanoparticles through the *sputtering process*. Micrographs were obtained on the frontal surface of the polymers. The magnitude ranged from 100 to 20,000 x and the power was 10 kV. The SEM was carried out at the University of São Paulo (USP).

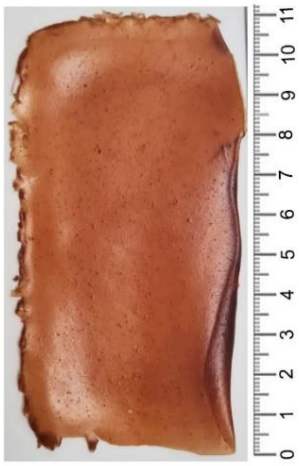
2.6 MECHANICAL TESTING

For the mechanical tensile test, a *Shimadzu Autograph AG-X universal testing machine at* 250 kN was used at the Materials Treatment Laboratory – Corrosion and Plasma (LabTrat) of IFPI, according to ASTM D3039 standards. The test on the specimen took place at a speed of 10 mm/min. Measurements of force, stress, displacement, strain displacement, strain and time were performed and a stress versus strain diagram was generated . Tests were conducted at room temperature.

3 RESULTS AND DISCUSSION

The starch recovered from the mesocarp of *O. phalerata*, the babassu coconut, had a good yield. The polymeric blend, whose main component is the starch of the babassu coconut mesocarp and has glycerin, melamine and formaldehyde as ligands, was successfully obtained (Figure 1).

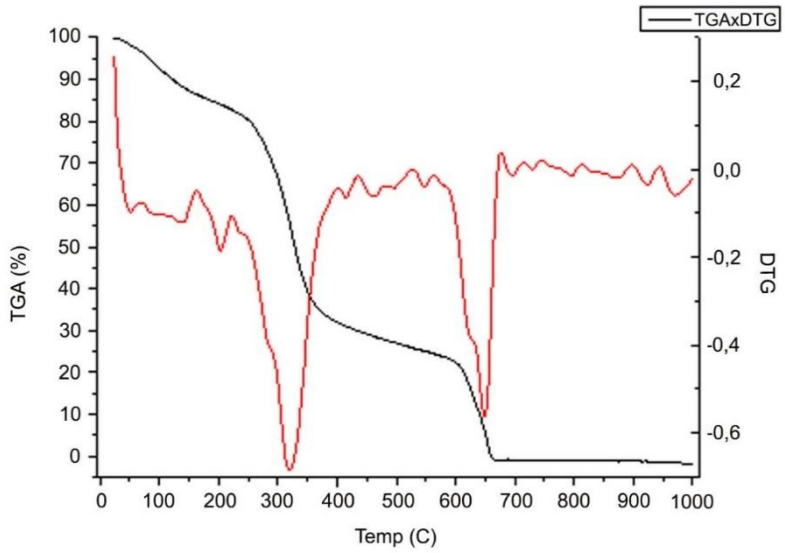
Figure 1 - Photographic record of the starch-based polymer blend obtained from the mesocarp of *O. phalerata.* Scale in cm.



Source: Authors (2023).

The TGA/DTG profile showed the mass loss due to the increase in temperature in three stages, as shown in Figure 2. In the TGA, it was possible to show that in the first stage, at a temperature ranging from 24 ºC to 200 ºC, there was the elimination of water and volatiles from the studied sample. In the second stage, above 200 ºC, there was a marked loss of mass related to sugars, characteristic of the degradation of the starch that makes up the babassu mesocarp. In the temperature range of 210 ºC to 400 ºC, the starches suffered the greatest mass loss, which can also be attributed to the degradation of the other organic compounds of the polymeric blend. And finally, there was the loss of mass related to melamine. Likewise, this fact was evidenced in the DTG curves, indicating that there are at least three peaks, in which the mass loss initially observed is attributed to the dehydration process; followed by the degradation of starch and, subsequently, of the other compounds. As shown in the DTG curves, the compounds have an unstable plateau, which precedes thermal decompositions after dehydration.

Figure 2 - Graph of the TGA/DTG profile of the polymeric blend based on starch obtained from the mesocarp of *O. phalerata.*



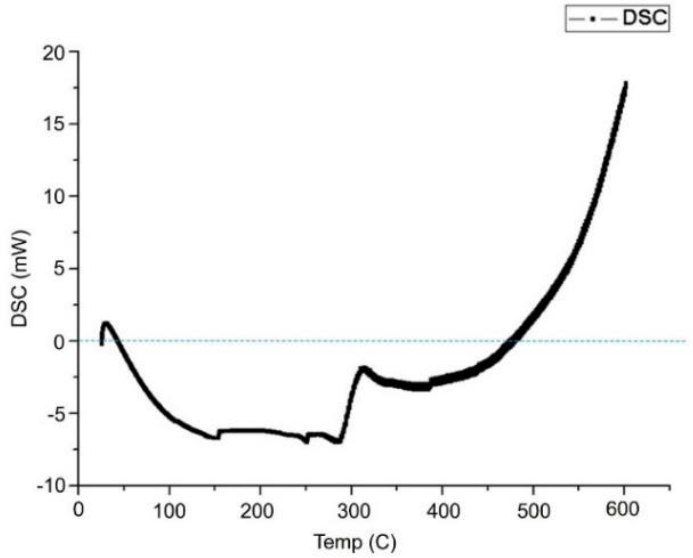
Source: Authors (2023).

The degradation process at high temperatures promotes the random breaking of the C-C bonds of organic molecules, affecting the molecular and/or polymeric chains of these compounds (Maniglia, 2017; Queiroz, 2018). Thus, the peak of approximately 350 °C for the starch degradation of the babassu mesocarp observed in the present study suggests that this polysaccharide contains large amounts of these organic compounds. When biomass is subjected to high temperatures (above 500ºC), it undergoes thermal decomposition of its chemical constituents due to the carbonization process (Santos, 2012).

In TGA/DTG, the peak temperature and the percentage of mass loss in each thermal event depend on the components and the crystal structure of the starch, through which it is possible to draw conclusions regarding the thermal stability of the sample, composition and stability of the compounds, as well as the final product (Denari & Cavalheiro, 2012). In the present study, the thermogravimetric analysis and its derivatives showed that the loss of mass related to moisture, volatiles, starch, glycerin and melamine occurred up to a temperature of approximately 600ºC.

As for the DSC, curves with defined peaks characteristic of starches that do not have a high degree of purity were observed (Figure 3). The thermogram showed a slight exothermic peak at approximately 50 ºC and a variation of endodermal peaks observed at about 75 ºC to 400 ºC. After 450 ºC, the sample showed a marked release of energy marked by a large exothermic event, possibly due to the fact that the sample had a lower purity content.

Figure 3 - Graph of the DSC profile of the starch-based polymer blend of the mesocarp of *O. phalerata.*

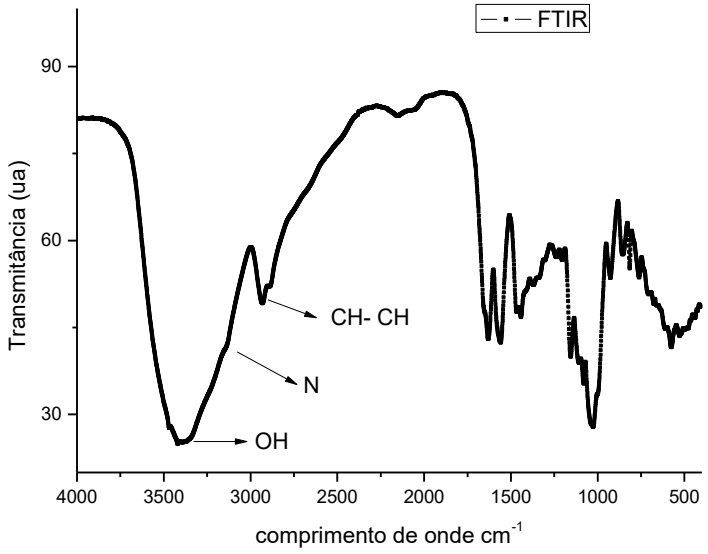


Source: Authors (2023).

According to Franco (2010), endothermic and exothermic enthalpic transitions occur due to changes in physical states (fusion, boiling, sublimation and vaporization) or chemical reactions, such as dehydration, dissociation, decomposition, oxidation and reduction. In general, the processes of melting, vaporization, and reduction produce endothermic effects, while crystallization, oxidation, and some decomposition reactions produce exothermic effects.

Regarding the FTIR spectra, Figure 4 shows the characteristic result of a polymer material with wide bands, due to the high molar mass of the polymer chains.

Figure 4 - Spectra in the infrared region of the starch-based polymer blend of the mesocarp of *O. phalerata.*

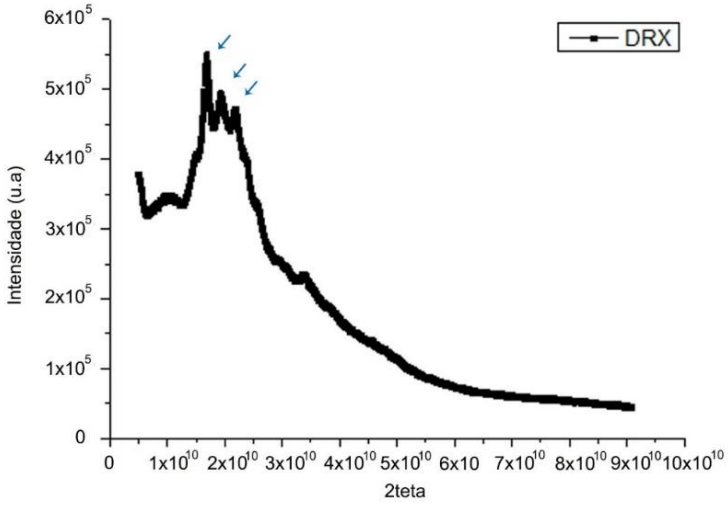


Source: Authors (2023).

This technique has been used to ascertain the starch present in the structures (Capron *et al*., 2007). Regarding the functional groups, the spectrum showed a wide band in the range of approximately 3470 to 3250 $^{cm-1}$ indicative of deformation of the OH bond of glucose monomers. Subsequently, the binding of CH-CH was observed in wavenumbers of approximately 2980 cm-1 to ^{2875} cm-1, corresponding to the starch striker, and N around 3100 $^{cm-1}$ referring to melamine. These data were approximated to those reported by Rodrigues (2021), in FTIR of the precursors of starch, melamine, urea and fertilizer composites. It is then presumed that it was possible to characterize the starch present and identify the expected peaks of the compounds inserted in the formation of the polymeric blend

The X-ray diffractogram, shown in Figure 5, was typical of an amorphous compound with small crystallinity points observed in three peaks, between approximately 17º and 25º.

Figure 5 - X-ray diphratogram of the polymeric blenda at the base of the O. *phalerata mesocarp amide*.

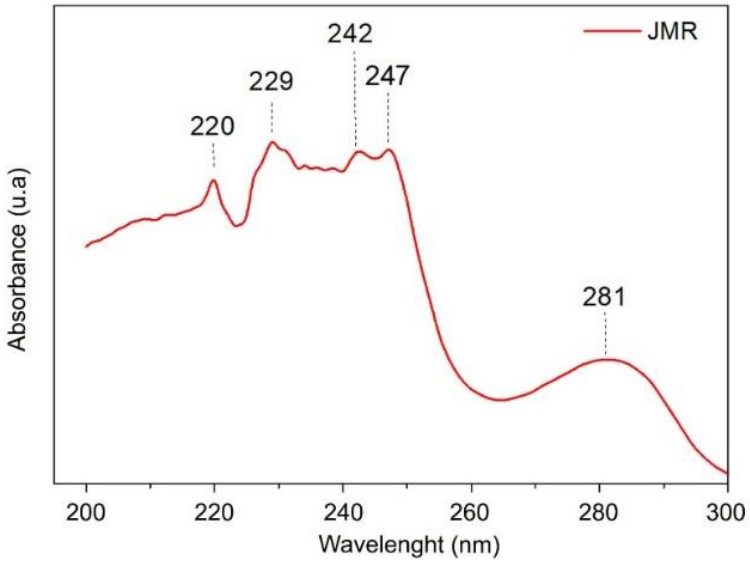


Source: Authors (2023).

In a study to obtain biodegradable composites from babassu coconut, the starch diffractogram showed peaks at 5.91º, 11.45º, 13.12º, 17.52º, 20.03º, 23.33º and 26.63º, characteristic of the type C pattern that is intermediate between types A (more compact, monoclinic arrangement) and B (less compact, hydrated center, hrxagonal arrangement) (Elfstrand *et al*., 2004; Moura *et al.*, 2021). As for melamine, in the tests for degradation of fertilizer composites containing urea, the peaks were between 15º and 30º (Rodrigues, 2021). Thus, the crystallinity points found in the polymeric blend of this study may be related to starch residues and/or overlap with melamine.

Regarding the UV-Vis analysis, the absorption spectrum showed maximum lengths between 220 and 247 nm and a steep shoulder at 281 nm (Figure 6).

Figure 6 - Absorption spectrum of the starch-based polymeric blend of *O. phalerata mesocarp*.

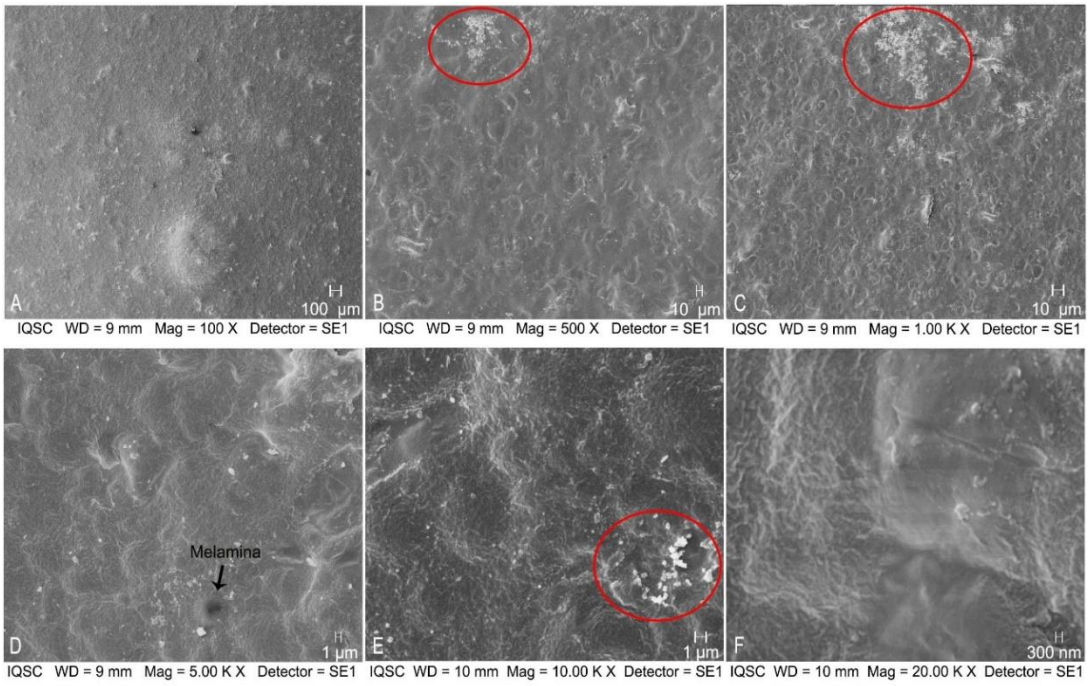


Source: Authors (2023).

The absorption spectra allow the deducation of electron transitions and thus obtain information to aid the recognition of molecular structures (Martinho, 1994). Corroborating these findings, absorption spectra with wavelengths between 200 nm and 260 nm have been identified for melamine, which may present a low-intensity shoulder at 250 nm (Zhang *et al.*, 2016; Correia Junior, 2019). In the free aqueous extract of the babassu coconut mesocarp, two absorption peaks were obtained, of 210 nm and 280 nm (Silva, 2017). This reinforces that the energy absorbed in the UV-Vis region for the polymeric blend of this research corresponds to the electronic transitions of the double bonds present in melamine and starch, as well as their interactions.

The micrographs revealed that the surface of the blend presented irregular and rough undulations, easily perceived in the images of Figure 7D, 7E and 7F, with differences in color in the film and melamine concentration (Figure 7D). The distribution of the polymeric blend was not uniform, and crossed lines (Figure 7F) and starch island residues, which are the white dots highlighted in the gray material (Figure 7B, 7C and 7E) were observed.

Figure 7 - SEM images of the starch-based polymer blend of the mesocarp of *O. phalerata* at magnification of 100 µm (A), 10 µm (B and C), 1 µm (D and E) and 300 nm (F). Red circle in B, C, and E indicate starch granules. Arrow in D points to melamine concentration.



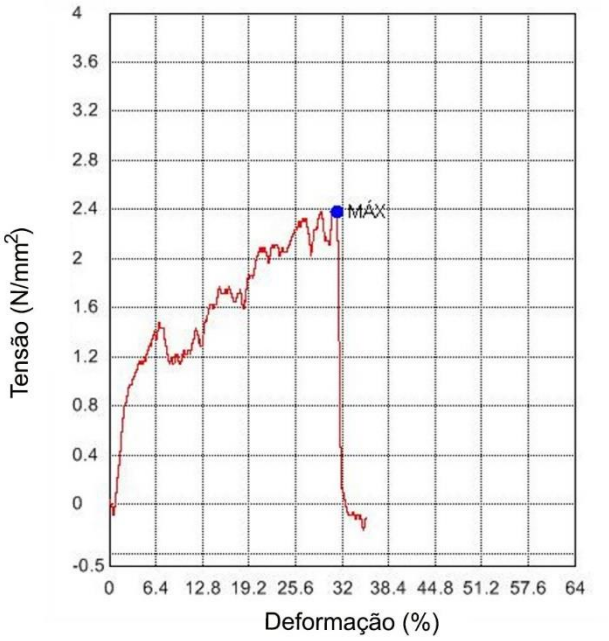
Source: Authors (2023).

This is due to: i) the drying process in an oven at a constant temperature of 37 ºC, indicating that the stirring and heating time were insufficient for the complete rupture of the starch granules, in agreement with Bastos (2010) in obtaining the thermoplastic starch films; ii) the binding capacity of glycerin in combination with melamine and formaldehyde, as mentioned in the tests by Zuo *et al.* (2015), who verified variations in the surfaces of the films due to the action of different plasticizers; and iii) the amount of glycerin used in the formation of the blend, as verified by Ramos Junior (2023), in a characterization of thermoplastic starch in which there was a decrease of such particles with the increase in the concentration of this plasticizer in the composition.

However, the starch of the babassu coconut mesocarp together with glycerin and melamine/formaldehyde were able to form a monophasic polymeric structure with only starch residues, different from the common biphasic arrangement of this polysaccharide, in which the formation of a continuous matrix composed mainly of amylose and a phase with swollen gelatinous granules is expected (Morris, 1990; Thiré, 2003). In addition, the presence of pores and cracks was not noticed, which is favorable for a slight permeability and resistance of the blend. In contrast to this finding, the SEM of babassu coconut epicarp fiber composites and different polymeric residues, using Urea-Formaldehyde (UF) resin as matrix, demonstrated porosity, cracks, and voids (Rodrigues, 2019).

The polymer blend has been tested in tensile strain up to 64%. The maximum strain was 31.25% at 2.35 N/mm2 of tension, under a force of 7.6 N and a maximum displacement of 13.44 mm (Figure 8). The ductile behavior demonstrated can be associated with good adhesion between the components.

Figure 8 - Stress versus strain graph for the starch-based blend of the mesocarp of *O. phalerata*. Blue circle indicates point of maximum strain and stress.



Source: Authors (2023).

These values are close to those found in the production of biofilm based on chitosan and glycerol, which also revealed ductile behavior, tension of 2.65 N/mm2, but greater elasticity, with maximum deformation occurring in 54.91%, being reached by the presence of the plasticizer (Machado, 2021). The proportion of amylopectin present in starch also alters the mechanical tensile behavior, as reported by Corradini *et al*. (2005) in comparative tests of thermoplastic starches derived from corn.

4 CONCLUSION

The proposed methodology for the development of a product based on the mixture of starch from the babassu coconut mesocarp with glycerin, melamine and formaldehyde, led to the formation of a polymeric blend with an inhomogeneous distribution presenting irregular and rough undulations, exhibiting small differences in color and melamine concentrates, in addition to the presence of starch residues. No pores and cracks. The test and the analysis of the data of the TGA/DTG curves identified and described that the degradation ranges of starch and most organic compounds was from 210 ºC to 400 ºC. Below this temperature, water and volatiles were degraded, and above melamine. From the DSC it was possible to infer that the molecular interaction allowed the predominance of endothermic events, with two exothermic peaks in the range of 50 ºC and after 450 ºC.

The spectra in the infrared region characterized the starch present and identified the peaks of melamine and glycerin compounds, which were the plasticizers used. The electronic transitions of the double bonds present in melamine and starch, as well as their interactions, showed maximum lengths

between 220 and 247 nm, and a pronounced shoulder at 281 nm. The polymeric blend is amorphous, with crystallinity points referring to starch residues and/or overlapping with melamine. The mechanical behavior was ductile due to the good adhesion between the constituents, with a maximum strain of 31.25% at 2.35 N/mm2 of stress.

Because it is an innovative product and because of the scarcity of studies in the area, all the tests conducted were compared to similar polymers with starches also coming from other sources and the use of different plasticizers. This comparison was of paramount importance to recognize the feasibility of using this polymeric biofilm. It is worth mentioning that it was a raw material that adds value to the babassu coconut production chain, cooperating with economic, social and environmental development.

Thus, in this work, the methodology proposed for the formation of the polymeric blend based on the starch of the babassu coconut mesocarp presented good results for the physicochemical, mechanical and thermal properties, with possible potential for various applications. However, comparative tests with different proportions of the components, their performance in more mechanical tests added to other physicochemical methodologies are necessary to elucidate the best composition and viability of the blend obtained from the mesocarp of the babassu coconut, being a future research of the main researcher.

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