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Characterization of Babassu Mesocarp Flour as Potential Bio-Reinforcement for Poly (Lactic Acid)

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Abstract

Growing environmental concern has resulted in a strong trend towards using green materials, and the efficient use of by-products reduces resource waste and environmental pollution. Using Babassu Mesocarp Flour (BMF), a by-product of the babassu oil extraction industry, as bio-reinforcement for polymers, such as Poly (Lactic Acid) (PLA), is a promising alternative. Before any attempt to develop composite films, the industrially extracted BMF was characterized to know its chemical composition, particle size distribution, specific surface area, morphology, and structure, which is the main objective of this manuscript. The elemental analysis showed that the BMF is predominantly organic and also showed contents of inorganic elements by XRF. Its particles had an average diameter of 38.82 μm and a specific surface area of 3.02 m^2/g. Through microscopic techniques, mainly SEM, starch granules in different shapes and sizes were observed. XRD and FTIR showed structure and functional groups typical of starchy materials, evidencing the ecological character of BMF. Progress is necessary to achieve the continuous and comprehensive use of babassu coconut by-products, mainly BMF, allowing greater appreciation. The characteristics of BMF are of great interest in PLA-based films, as they are green materials and non-toxic to the environment.

Keywords: industrial by-product, biomass, babassu mesocarp flour, PLA, composite films

1. Introduction

Babassu (*Attalea speciosa*) is a kind of Brazilian palm that can reach about 20 meters, having a cylindrical trunk and cup-shaped crown (Figure 1). This palm is also known as *Orbignya speciosa*, *phalerata*, or *oleifera*, with *Orbignya phalerata* being the most common. The babassu palm is native to countries in South America, mainly in the Northeast and North of Brazil, and to a lesser extent in Bolivia, Colombia, and Suriname (Vinhal et al., 2014; Yapuchura et al., 2019; Cruz et al., 2020). In Brazil, the central area of occurrence of babassu is located in a transition zone between the tropical forests of the Amazon basin (North) and the semi-arid (Northeast) region (González-Pérez et al., 2012; Protásio et al., 2014). The Brazilian states with the most significant palm tree extensions are Maranhão, Piauí, and Tocantins, thus forming dense/dark homogenous groups due to the proximity between the palm trees (Albiero et al., 2007). Generally, there are about 15 to 25 bunches of babassu coconut in a palm tree, and the coconut has an ellipsoidal shape. It is constituted by the epicarp (11–13%), mesocarp (20–23%), and endocarp (57–63%) layers, in addition to the almonds (7–9%) (Vinhal et al., 2014; Santos et al., 2019). In Brazilian territory, babassu production is essential in extractivist (Porro, 2019; Lemos & Andrade, 2021), associated with various products and by-products from different parts of the palm tree and the fruit (Melo et al., 2019).

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Figure 1. Babassu palm trees (babassu coconut bunch highlighted) located at the Natural Sciences Center of the Federal University of Piauí

Regarding babassu layers, each one has its attributes. For example, the epicarp is a fibrous layer (outer), the mesocarp is a fibrous/starchy layer (middle), and the endocarp is a woody layer (inner). Almonds are found in the endocarp; these three layers correspond to ~93% of the coconut (Pavlak et al., 2007; Lemos & Souza, 2018). The industrial process of babassu oil extraction produces several types of by-products, including the mesocarp, which is dried and ground into a brownish powder called "Babassu Mesocarp Flour" (BMF) (Baruque Filho et al., 2000). BMF is mainly composed of starch, but in addition to starch, it also contains small contents of proteins, lipids, and ash (França et al., 2014; Ferreira et al., 2020). This flour may show traces of epicarp and endocarp, resulting in residual fiber content in its composition (Santos et al., 2019). Typically, it is common to observe fiber contents in the composition of flours. Due to the accumulation of non-biodegradable plastic waste in nature, researchers use sustainable materials to produce new composites or biocomposites (Vargas et al., 2017;

Moura et al., 2018; Oliveira et al., 2019; Vasconcelos et al., 2023). Composite materials represent an ecologically and technically promising option to replace conventional plastics in food packaging. In this perspective, BMF can be a promising alternative to be incorporated into polymer matrices (bio-reinforcement), such as the Poly(Lactic Acid) (PLA) matrix, aiming at the future development of composite films. Research aiming to produce films based on PLA and BMF has yet to be reported.

PLA is a thermoplastic aliphatic polyester derived from lactic acid, which can be produced from 100% renewable plant resources by the natural fermentation of glucose from corn, potatoes, sugar beet, sugar cane, and biomass (Shahrampour et al., 2023; Ferdináand et al., 2023). It is the most attractive biodegradable polymer for large-scale applications as a packaging material (Kharrat et al., 2020; Srimalanon et al., 2020). Through the "Food and Drug Administration" (FDA) agency located in the United States of America (USA), PLA received the title of "*Generally Recognized as Safe*" (GRAS). It can come into contact with food and beverages without risk to human health (Yusoff et al., 2021). It was explained that the biodegradation of PLA in nature does not cause ecotoxicological impacts (Araújo et al., 2014; Chyr & DeSimone, 2023). PLA is marketed as a compostable polymer. Producing films based only on PLA is not economically viable due to its relatively high cost compared to polyolefins (Bhagia et al., 2021; Mistry et al., 2023). Currently, one of the alternatives used by industries to minimize the cost of the polymer is to use a fraction of another material incorporated into the polymer matrix. Notably, such incorporation can reduce the properties of the composite in different magnitudes according to the content of the incorporated bio-reinforcement. This reduction is generally seen as an acceptable consequence of obtaining a more economically viable product. The biodegradable nature of PLA must not be drastically affected, and therefore the material to be incorporated should also be biodegradable (Sánchez-Safont et al., 2018; Volpe et al., 2018; Ghazvini et al., 2022).

Several by-products were incorporated into the PLA matrix to produce composite materials, such as fungal biomass (Asadollahzadeh et al., 2022), agave bagasse (Omar et al., 2022), hazelnut husk flour (Özdemir et al., 2022), yerba mate (Agüero et al., 2023), and cellulose (Araujo et al., 2023). BMF or starch isolated from the mesocarp have already been used for the production of films, as can be seen in the manuscript of Maniglia et al. (2017), Silva et al. (2019), Lopes et al. (2020) and Rodrigues et al. (2021). In the manuscripts in which BMF was used, the authors discussed its influence on the properties of the films produced and highlighted its great potential. However, it was observed that the intrinsic characteristics of the flour were little discussed, with little information, mainly concerning the chemical composition. Therefore, it is essential to characterize the bio-reinforcement before producing to produce films using binary formulations based on these two materials via flat die extrusion. In order to contribute to data already published in the literature, we propose – this is the main objective of this research – new characterizations in this manuscript, mainly through elemental analysis, X-ray fluorescence, granulometric distribution, and specific surface area. Morphological and structural analysis was also carried out to elucidate the organic nature of the BMF, which makes this material attractive for formulations with sustainable polymers and provides the reader with more details and direct comparisons with similar raw materials.

The idea of using an industrially extracted raw material is due to its commercial availability since, for producing products on a large scale, large amounts of flour would be required at an industrial level compared to small-scale production at a laboratory. All the considerations reported here aim to add value to this Brazilian industrial by-product, and also, due to the scarcity of technical information, the scenario was conducive to preparing a manuscript for this purpose.

2. Method

2.1 Materials

The raw material was obtained industrially through grinding and classifying the mesocarp as part of the full use of babassu coconut (Figure 2). Florestas Brasileiras S.A. (Itapecuru-Mirim, Maranhão, Brazil) carried out the procedure for obtaining the raw material. BMF is a powder with macroscopically uniform particles without significant amounts of particle agglomerates.

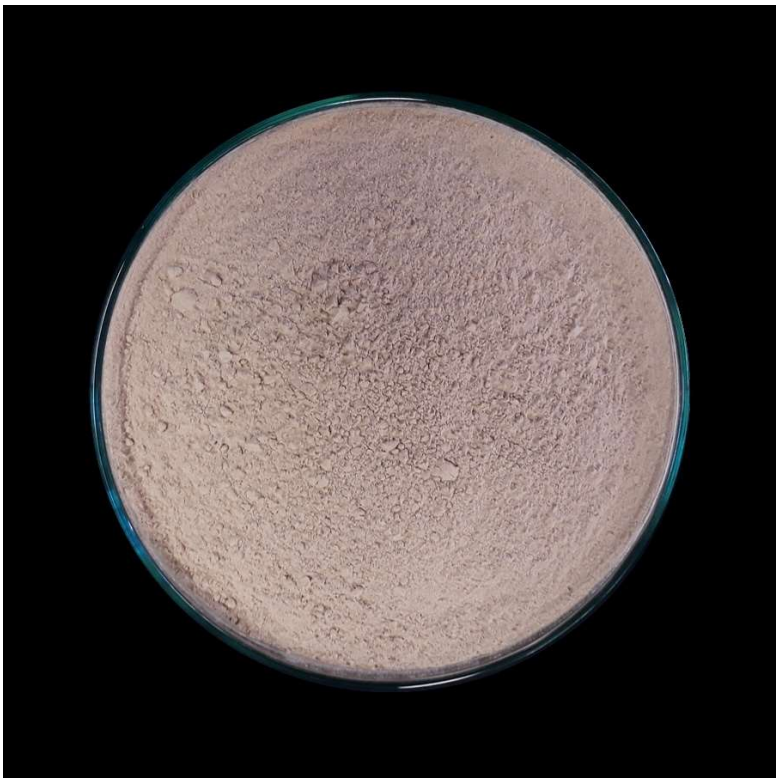

Figure 2. Visual aspect of babassu flour obtained from the mesocarp

2.2 Characterization Techniques of BMF

2.2.1 Elemental Analysis (CHN)

The percentage of Carbon (C), Hydrogen (H), and Nitrogen (N) in the BMF samples was determined using a CHNS/O elemental analyzer (Model 2400 Series II, PerkinElmer). The combustion and reduction columns' temperatures were 950 and 640 °C, respectively. Oxygen (O2) and Helium (He) gas pressure were 140 and 105 Kpa, respectively. The filling time of the combustion column was 30 s, and the total analysis time was ~5 min. The parameters used were adjusted for an efficient analysis of the samples.

2.2.2 X-Ray Fluorescence Analysis (XRF)

BMF was evaluated using a portable Niton XL3t GOLDD+ analyzer (Thermo Scientific, USA) and a "Mobile Test Stand" device, operating in "TestAll Geo" mode for ~85 s. The equipment used can measure from 10 ppm (0.001% mass/mass) to 100% mass/mass of the analyte (Krummenauer, 2017). The uncompacted BMF sample (in the form of loose powder) was evenly deposited over the entire surface of the sample holder (Camargo, 2021). The sample holder was covered with a thin Polypropylene (PP) film 4.0 µ thick and 63.5 mm in diameter (TF-240-255, Premier Lab Supply, USA).

2.2.3 Particle Size Analysis

To determine the grain size distribution (average diameter in volume), a laser grain size analysis was performed using an analytical range of sizes ranging from 0.04 to 500 µm (Model 1064, CILAS). The width of the size distribution (*Span*) was calculated using Equation 1:

$$Span = \frac{D_{90} - D_{10}}{D_{50}} \quad (1)$$

Where: D_{10}, D_{50}, and D_{90} represent the diameters, in which 10, 50, and 90% of the accumulated volumes of the distribution have particle sizes smaller than this value, respectively (Jia et al., 2022; Ribeiro et al., 2022).

2.2.4 Specific Surface Area (SSA)

The SSA was determined in triplicate using Quantachrome Instruments equipment (Model NOVA 1000*e*). Initially, the samples were dried in an oven at 100 °C for 24 h to remove moisture and then in a vacuum at 100 °C for 1 h. Subsequently, for each analysis, the sample was placed in the equipment, and by molecular adsorption of gaseous Nitrogen (N_2) at -196 °C (77 K), the value of the area was obtained.

2.2.5 Optical Microscopy (OM)

BMF was analyzed in a binocular optical microscope (Model ICC50 E, Leica Microsystems), operating in transmission mode with a magnification of 100x and a scale of 200 µm. The magnification and scale used was the one that made it possible to obtain good images.

2.2.6 Scanning Electron Microscopy (SEM)

Microstructural analysis of the BMF was performed using a JEOL scanning electron microscope (Model JSM-6510LV). The sample was coated with gold using a metallizer and analyzed at an accelerating voltage of 10 kV (Model Desk V, Denton Vacuum). The micrographs were captured at different magnifications: 500, 1000, 1500, and 2000x. The analysis parameters were defined based on the best configuration for obtaining micrographs.

2.2.7 X-Ray Diffraction (XRD)

The X-ray diffractogram of the BMF was acquired using an X-ray diffractometer (Model D-5000, Siemens/Bruker) under the following conditions: voltage of 40 kV, current of 30 mA, and using as a source of monochromatic radiation Cu-Kα (λ) = 1.5406 Å. Data were detected over an angular (2θ) range of 5 to 50° at a scan rate of 0.05°/1 s. The angular range was defined according to the already known peaks for starchy materials.

2.2.8 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectrum was obtained using a spectrophotometer (Model Spectrum 1000, PerkinElmer) operating at the 4000–500 cm^{-1} range of wavenumbers. The sample was mixed with potassium bromide (KBr) and pressed into a tablet before measurement. The spectrum was obtained based on 32 scans and a resolution of 4 cm^{-1}. The wave number range used in this manuscript is usual to observe the bands of chemical groups referring mainly to starch.

3. Results and Discussion

3.1 Elemental Analysis (CHN)

Elemental analysis indicated the percentage (in dry mass) of C, H, and N in the BMF samples. The results obtained are shown in Table 1.

Table 1. Percentage of C, H, and N in the BMF

Sample	Chemical element (%)		
	C	H	N
BMF	43.47 ± 0.046	6.39 ± 0.054	0.58 ± 0.134

Data were expressed as: mean ± standard deviation (n = 3)

The results showed the presence of C and H in BMF, which refers to the organic compounds in its chemical composition, mainly starch, which is the major component. The fact that BMF mainly comprises starch explains why C is present in a higher percentage than N, the element with the lowest percentage. The high C content is also related to the energy potential of the

plant biomass, associated with the calorific value, which is why the industrial by-products of babassu are used as charcoal (Protásio et al., 2017). N refers to proteins, which can be found in plant organisms consisting mainly of amino acids, which have nitrogen in their molecule (Joye, 2019). The C, H, and N percentages for BMF are similar to those for other vegetable raw materials, such as sugarcane bagasse (Ponte et al., 2019) and rice husks (Rodrigues, 2019).

In the manuscript by Rambo et al. (2015), several lignocellulosic residues found in the North and Northeast regions of Brazil were characterized by elemental analysis, including babassu mesocarp, which obtained the following results: 47.13 (C), 5.17 (H) and 0.97% (N). The authors also observed that the biomass from the babassu industry had the highest percentage of N – 1.14% (endocarp) and 0.97% (mesocarp). Nitrogen-rich biomass can be used as soil fertilizer as it is an essential plant nutrient. Sun et al. (2020) reported that N is vital as an essential macroelement for plant growth and development, representing ~1.5 to 2% of plant dry matter and ~16% of total plant proteins. Starch from *Solanum lycocarpum* had a low percentage of N, which the authors related to the high quality and purity of the extracted starch (Pascoal et al., 2013). These results highlighted the different interpretations for the percentage of N obtained since such conclusions are based on the raw material under study in its natural or chemically modified form. This discussion highlighted the importance of the elemental analysis technique for the knowledge of the elemental chemical composition of the material.

Other manuscripts published in the literature on babassu mesocarp were mentioned in this discussion. The mesocarp of the work by Melo (2012) presented around 39.23 (C), 6.70 (H), and 0.33% (N). The epicarp was also analyzed in the same work, and it is interesting to note that the percentage obtained, respectively, was 46.72, 6.12, and 0.51%, similar to the mesocarp results. Santos (2018) obtained the following data in his work: 39.37 (C), 6.18 (H), and 0.46% (N). The C, H, and N values for babassu mesocarp are similar to those obtained for cellulose, as seen in the manuscript by Muley et al. (2016). It is normal to have differences in percentages when comparing results, as babassu is a vegetable raw material whose properties vary according to environmental, processing, and storage conditions.

3.2 X-Ray Fluorescence Analysis (XRF)

The concentration of the main chemical elements identified in the composition of the BMF is shown in Table 2. The standard deviation is related to the error associated with instrumental measurements.

Table 2. Main chemical elements identified in the composition of the BMF by XRF

Chemical element	Symbol	Concentration (ppm ± error)
Zinc	Zn	15.30 ± 4.74
Copper	Cu	33.19 ± 8.55
Chromium	Cr	43.83 ± 19.12
Iron	Fe	253.10 ± 27.19
Phosphorus	P	709.61 ± 107.62
Calcium	Ca	815.89 ± 198.35
Silicon	Si	1357.62 ± 205.54
Sulfur	S	1911.19 ± 97.55
Chlorine	Cl	5436.90 ± 82.59
Potassium	K	13175.82 ± 295.56
-	Unidentified elements*	976106.250 ± 135.14

*Represents chemical elements not identified by the equipment, that is, those with atomic number (Z) < 12, such as C, H, N, and Oxygen (O).

According to the results obtained, it can be concluded that the main chemical element present in the composition of the BMF is K. Other flours, such as green bananas (Borges et al., 2009) and hemp (Rusu et al., 2021), have K as a primary element. The identification of most of these elements can be associated with the soil characteristics where the babassu palm trees were planted or with the nutrients customarily provided by fertilizers for the growth of palm trees. To better substantiate the previous statement, similar conclusions were found in the manuscript by Vassilev et al. (2010) and Naozuka et al. (2011), where the authors explained that the concentration of elements also depends on the plant physiology, composition of the water source and pesticides used in the plantations. Plants can absorb, transport, and accumulate different elements, and each species has its requirements and different tolerance levels when absorbing and accumulating a chemical element. Protásio et al. (2014) mentioned that K gives plants resistance to adverse conditions, such as low water availability and high

temperatures.

The concentration of elements in BMF varied in descending order: K > Cl > S > Si > Ca > P > Fe > Cr > Cu > Zn. Elements such as Magnesium (Mg), Manganese (Mn), and Selenium (Se) showed concentrations below the detection limit. The elements C, H, and O are the predominant elements that constitute the organic compounds present in the composition of the BMF. Therefore, a high concentration of unidentified elements was observed, reflecting almost 98%. Due to this result, a concentration of less than 3% of minerals was obtained; however, it is crucial to emphasize that instrumental errors must be considered. The babassu mesocarp bran used in the manuscript by Miotto et al. (2012) presented 4.6% of mineral matter. In the manuscript by Nascimento et al. (2019), a content of 1.20% was observed for BMF. Based on the results, BMF is a heterogeneous mixture of organic and, to a lesser extent, inorganic matter. Despite being in lower concentration, the inorganic matter present in the composition of the BMF influences its properties and, consequently, its various uses.

The mineral composition is one of the most important indicators of the nutritional value of foods (Kale et al., 2015). Some foods with high nutrient content have been used as dietary supplements to improve nutritional values, and BMF is an example due to its composition rich in mineral salts (Azevedo et al., 2007). The mineral composition of the BMF still needs to be evidenced in the literature. However, it is possible to find some manuscripts that analyzed this raw material to know the existing minerals. Carneiro (2011) characterized the babassu mesocarp, and the results identified the following elements: Ca, P, K, Mg, Sodium (Na), Cl, Cu, Fe, and Mn. Babassu mesocarp was also evaluated in the manuscript by Fioroto et al. (2015), and the authors observed the presence of Cu, Fe, Zn, Ca, and Mg. In the work of Oliveira (2018), BMF was analyzed and presented the following composition: Cu, Fe, P, Mn, K, Se, Na, and Zn. The variation between the composition of the BMF used in this manuscript and the raw material of the referred manuscripts in terms of the identified elements and their concentration was already foreseeable. The babassu is the result of extractive; therefore, its composition is influenced by the environment of the place where it was cultivated.

3.3 Particle Size Analysis

Figure 3 presents the result of the granulometric distribution of the BMF, where it was evident that not all particles have the same size. Therefore, various sizes are present, described mathematically by a wide distribution range.

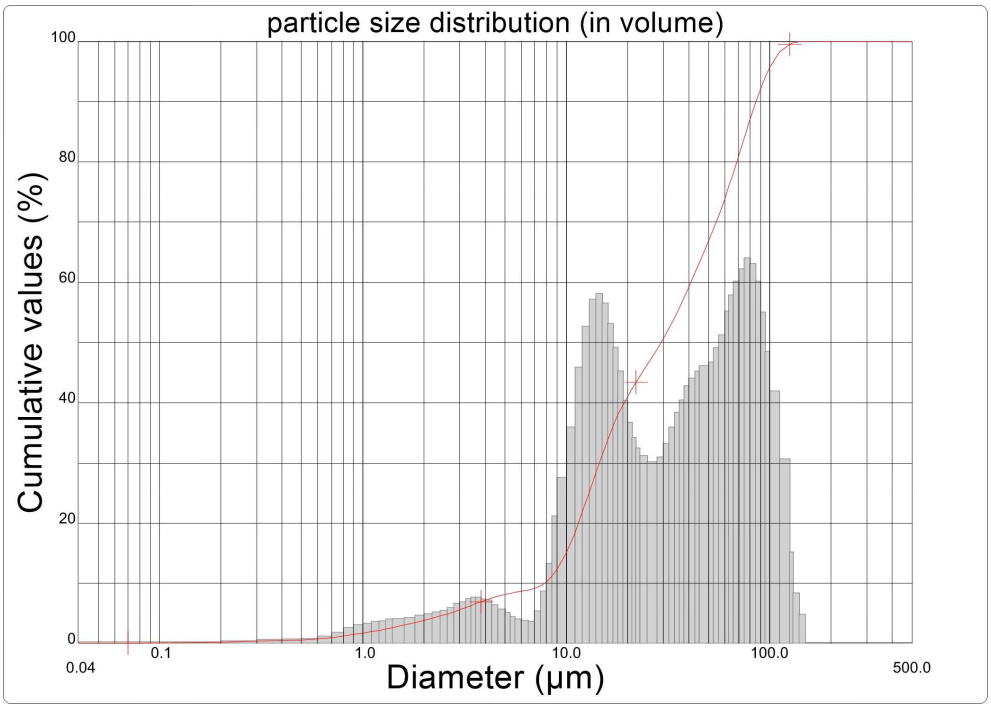

Figure 3. Histogram showing the granulometric distribution curve of the BMF

BMF showed a trimodal particle distribution curve; however, a more significant bimodal distribution was observed with the two peaks between 10 and 100 µm. This result can be corroborated by the fact that BMF is a natural raw material, leading to particle heterogeneity. Thus, a specific granulometry will not be chosen to be used in future manuscripts aimed at the development of composite films. Suppose there is research interest in using BMF with a specific granulometry. In that case, the separation and classification of the particles can be performed through sieving to minimize the heterogeneity in terms of granulometric distribution. So, as it is an industrial by-product, defining a specific granulometry and not considering the other granulometric can harm its application potential. BMF had an average diameter of 38.82 µm, and the calculated *Span* was 2.65. The BMF used in the manuscript by França et al. (2019) presented particles of 10 to 20 µm. The values for D_{10}, D_{50}, and D_{90} are shown in Table 3.

Table 3. Diameters at 10, 50, and 90%, average diameter, and *Span* of the BMF sample

Sample	Diameters (μm)				*Span*
	D_{10}	D_{50}	D_{90}	Average diameter	
BMF	7.80	29.27	85.42	38.82	2.65

It is important to note that *Span* measures the width of the particle size distribution (Hosseini et al., 2014), so if the calculated value is close to zero, the particle size is more uniform. Banakar et al. (2022) stated that particle size distribution is proportional to the *Span* value. For small *Span* values, a narrow particle size distribution is observed. The characteristics of the raw material also influence the *Span* value. This statement can be based on the result of the manuscript by Luchese et al. (2017), and Engel et al. (2020), where corn starch had a *Span* of 1.3 (average diameter = 12.2 μm) and cassava starch had the same value but with an average diameter of 11.9 μm, respectively.

Although starch is the main component of the BMF, the shape and size aspects of the granules depend on the botanical origin of the starch. Therefore, the mean diameter and *Span* of the BMF differed from the values obtained for the referred starches. Suppose the particles are not well dispersed in the liquid throughout the analysis. In that case, agglomeration may occur through interactions between particles of different sizes and cause an increase in the average diameter, although each particle may have a smaller size. It is also important to note that there is no ideal particle size or size distribution, as the ideal values depend on the application and properties of interest (Dearmitt, 2011).

3.4 Specific Surface Area (SSA)

The SSA of the BMF resulted in a value of 3.02 ± 0.21 m^2/g. Other authors employed the SSA technique in the analysis of different types of native starches (corn, potato, rice, and wheat) (Sujka, 2017), and the results showed that the surface area of all starches was smaller than the area of the BMF. Chen et al. (2019) concluded that the smaller the starch granule, the greater its SSA. This technique was also used to analyze different biomasses, such as castor seed and sunflower cake, in which the authors observed a SSA of approximately 0.37 and 1.13 m^2/g, respectively (Castro et al., 2016). Area values of 1.34 and 5.64 m^2/g (Silva, 2016) were found for the babassu mesocarp. The surface area obtained for BMF in this manuscript was more significant than for other parts of babassu used in other manuscripts, such as in the manuscript by Vieira et al. (2011a), where the value observed for the surface area of the epicarp was 1.50 m^2/g. Knowing the SSA of the bio-reinforcement material is vital since a greater surface area has more excellent contact with the polymer matrix, thus increasing adhesion (Dearmitt, 2011).

3.5 Optical Microscopy (OM)

The optical microscope equipment uses visible light and magnifying lenses (objective and eyepiece) to obtain an enlarged image of the examined sample, which can be captured digitally. It is the most used microscopic technique for analyzing the morphology of starch granules, being simple and economical concerning other advanced techniques, such as SEM (Chakraborty et al., 2020). Thus, an optical microscope was used to investigate the morphology (two-dimensional) of the BMF, and the captured micrographs are shown in Figure 4.

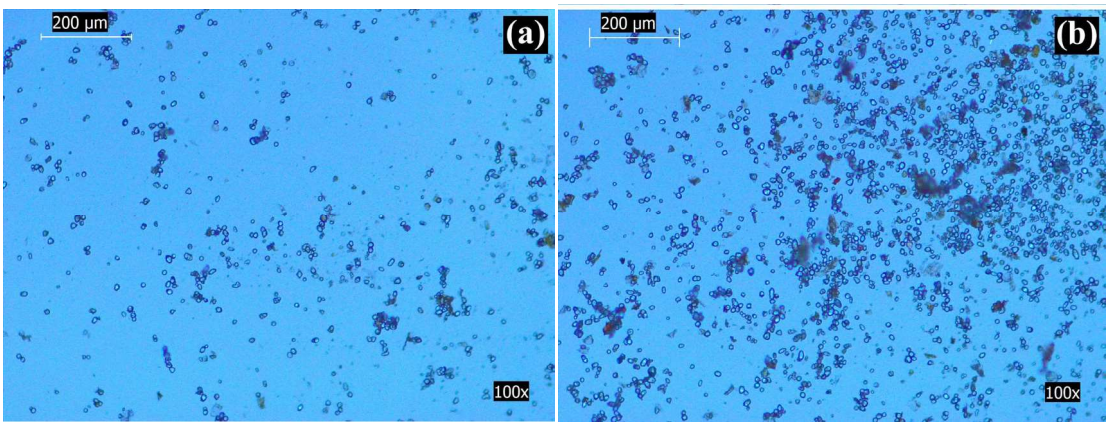

Figure 4. Micrographs: (a) and (b) obtained by optical microscopy of the BMF

Through the OM technique, it was possible to observe the starch granules of the BMF, which in turn were presented in different shapes and sizes. OM allows visualizing the differences between starch granules from different botanical origins. Govindaraju et al. (2020) used OM to study the morphology of rice and corn starch. This technique is also advantageous for verifying the starch granules swelling and loss of structural integrity due to some treatment, either by increasing the temperature (Di Paola et al., 2003) or using high pressure (Kweon et al., 2008). However, the limitations of OM arise at higher magnifications, where finer structures are difficult to visualize due to restrictions on image resolution and contrast (Jagadeesan et al., 2020). As a result, the surface details of the starch granules present in the BMF composition were not visible. Therefore, SEM analysis was performed to overcome this limitation and obtain high-resolution images. SEM remains the most advantageous technique for investigating starch granules morphology (Błaszczak & Lewandowicz, 2020).

3.6 Scanning Electron Microscopy (SEM)

The SEM technique is widely used to investigate the surface morphology of starchy materials. Because of this, the morphology of the BMF was analyzed using this technique, as shown in Figure 5.

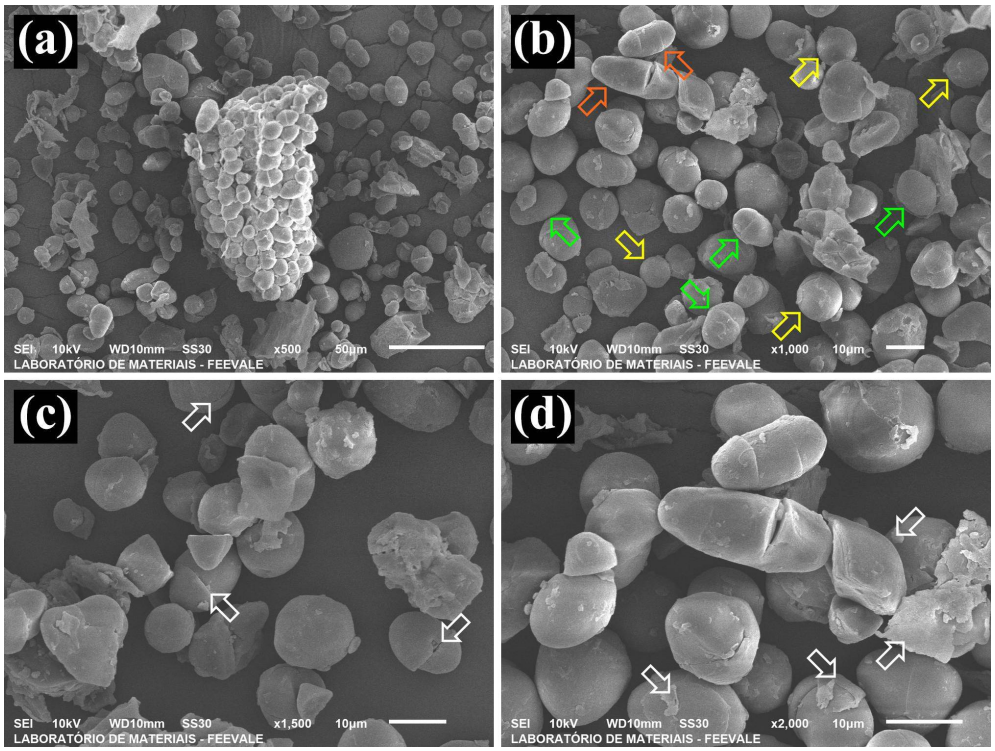

Figure 5. Micrographs obtained by SEM of the BMF: (a) 500, (b) 1000, (c) 1500, and (d) 2000x magnification

According to Figure 5, it was possible to visualize the native starch granules that make up the BMF, and the granules have different shapes and sizes. The shape and size of native starch granules are related to their botanical origin and can differ widely within the same species. Sun et al. (2021) also pointed out that the difference in morphology between granules of different types of starch can be attributed to genetic factors that regulate starch biosynthesis, amylose/amylopectin ratio, and molecular composition. As for shape, some starch granules from BMF are spherical (yellow arrow), others have an ellipsoid shape (green arrow), and others have a more elongated ellipsoid shape (orange arrow). Although many granules exhibited an ovoid shape, an irregular shape for many of them was also observed. The starch granules in BMF are similar to potatoes (Sujka & Jamroz, 2013) but very different from rice (Ma et al., 2021) and wheat starch granules (Zhang et al., 2021). As for the size, the granules showed heterogeneity. The surface morphology of starch granules is essential in studying the structure-property relationship of starch and therefore plays an important role in the processing of starchy materials (Wang et al., 2021).

In Figure 5, a fraction of the granules are grouped similarly to a bunch of grapes, showing a dense matrix that allows the agglomeration of the granules. It was also possible to visualize, with emphasis on Figure 5.c, that some granules are “broken” and, consequently, with a semi-spherical shape. This broken is due to the mechanical stresses inherent in the industrial processing of flour extraction, leading to surface cracks. This statement can be corroborated by Rahaman et al. (2021), in which it was mentioned that friction or shear forces are

sufficient to cause cracks on the surface of starch granules. Although these cracks were observed in several granules, the surface was predominantly smooth and did not show pores or cavities. Carrying out the flour extraction step without causing damage to the starch granules is extremely difficult in industrial practice. In several granules, it was observed that a type of material in different proportions adhered to the surface. However, this material was observed in isolation beyond the surface, always close to the starch granules (Figure 5.d). This adhered material is considered proteins, lipids, or fibers (Maniglia, 2017; Oliveira, 2018), corresponding to the matrix described above that allowed the agglomeration of many granules. As N is an element indicative of the presence of proteins, according to the topic of elemental analysis, the presence of a fraction of protein material adhered to the surface of the granules is a possibility that should not be discarded.

Anyasi et al. (2017) elucidated that the presence of united granules of green banana flour can be attributed to the occurrence of lipid and protein molecules. In the manuscript by Ozturk et al. (2021), it was observed through the SEM that several corn starch granules were retained in a protein matrix. The authors also observed that some granules had traces of protein bodies on their surfaces, and others appeared to be attached to fiber-like structures. Möller et al. (2021) reported similar observations in detail in the manuscript for the surface of starch granules extracted from pea flour. Padhi & Dwivedi (2022) highlighted that the leaf-shaped structures close to the granules confirmed the presence of fibers. The surface nature of starch granules and the presence of proteins and lipids can have relevant effects on starch properties. Granule-associated proteins and lipids are the most abundant secondary components of starch, which is believed to be incorporated into the granule during its synthesis (Pérez et al., 2009). The results in the literature showed a certain similarity with the micrographs obtained in this manuscript, highlighting the importance of the SEM technique for studying the morphology of starch granules.

3.7 X-Ray Diffraction (XRD)

The XRD pattern of the BMF is shown in Figure 6. Diffraction peaks at 2θ = 5.75, 15.08, 17.10, 20.25, and 22.75° were observed, with the sharpest peak at 17.10°.

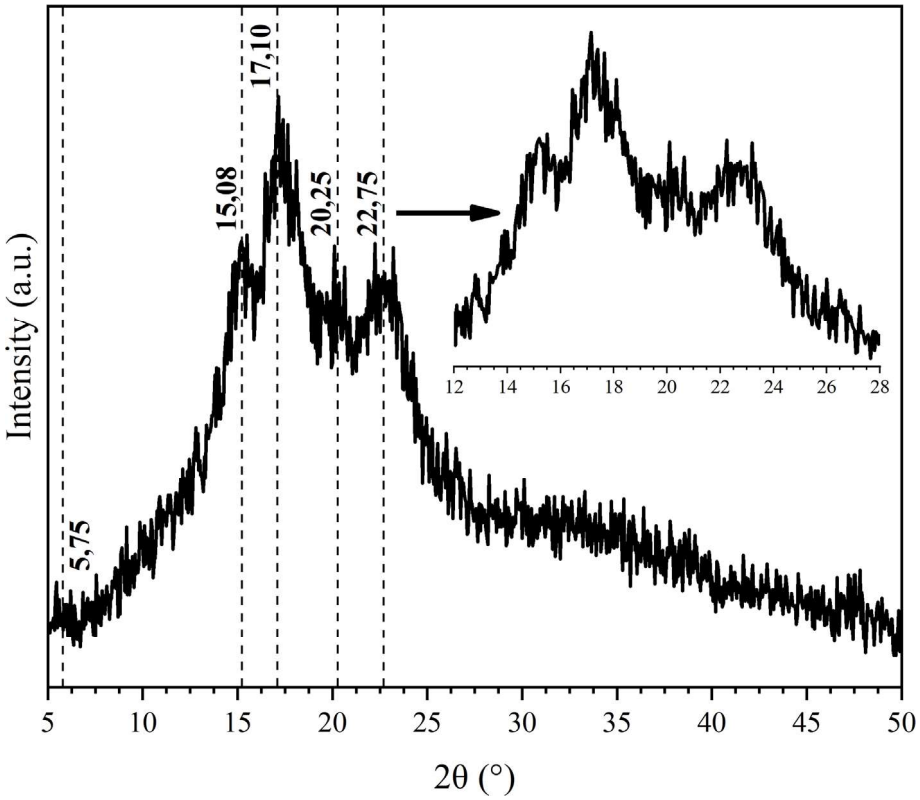

Figure 6. XRD pattern of the BMF showing characteristic peaks

The result indicated that BMF had a structure characteristic of semicrystalline materials but with a dominant amorphous character. Crystalline materials generally have intense, sharp, and narrow peaks, while amorphous materials have low-intensity and broad peaks. A hypothesis that may explain the amorphous character of the BMF is the following: starch is a semicrystalline polysaccharide. That is, it has a certain degree of molecular organization due to the amylopectin chains present in its composition, which corresponds to the crystalline region; however, this statement is more significant for starch in its isolated condition, in the case of the BMF, in addition to the starch in its composition, there are other components (e.g., proteins, lipids, and fibers) that can form agglomerates and retain the starch granules (Maniglia & Tapia-Blácido, 2016) and therefore, the structure of the trapped starch may not be easily detected, making it difficult to see a less amorphous character in the XRD pattern and making the BMF more amorphous than the isolated starch.

BMF used in the manuscript by Nascimento et al. (2019), in addition to the peaks between 5.6–26.2°, also presented peaks at 30.4 and 34°. BMF exhibited a structure with diffraction peaks at positions similar to starch from different botanical origins, such as quinoa flour (Ahmed et al., 2018) and lentil flour (Lu et al., 2018). The above references showed that the XRD technique had been widely used in detecting and characterizing starch granules crystalline patterns or verifying some amorphous condition of the starchy material. Crystallographic planes referring to the peaks or at angles close to them have been reported for some of the previously mentioned starches (Popescu et al., 2018; Cervantes-Ramírez et al.,

2020).

The planes mentioned can reference the peaks found in the XRD pattern of the BMF, obviously, with slight differences since it is flour and not material with 100% starch in its composition. A relevant contribution to the identification of crystallographic planes by XRD in native starches was reported by Singh et al. (2006). Although the botanical origin of starch plays an essential role in obtaining the XRD pattern, other aspects, such as amylopectin chain length and moisture content, also influence this pattern. Regarding moisture content, the manuscript by Wang et al. (2022) studied the effect of different moisture contents (30, 35, 40, 45, and 50%, based on dry weight) on corn starch during the extrusion process. The results showed that the higher the moisture content in the starch, the easier the absorption of water by the granules and the faster the expansion of the granules, facilitating gelatinization and reducing crystallinity. Therefore, the XRD technique was paramount for a better understanding of the structure of the BMF, evidencing its starchy structure.

3.8 Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR technique allows the determination of existing functional groups in the chemical structure of the raw material under study since the specific vibrational mode of each group promotes the appearance of its absorption band in a characteristic wavenumber. Thus, this technique was used to identify the functional groups in BMF, as shown in Figure 7.

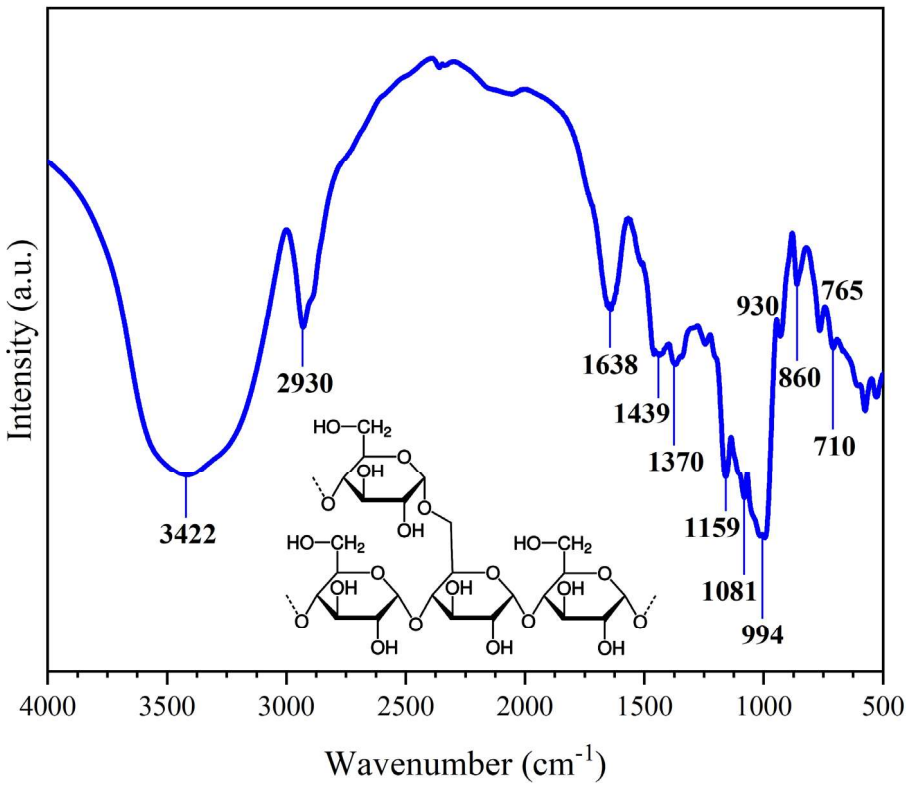

Figure 7. FTIR spectrum of the BMF investigated in the range of 4000 to 500 cm^{-1}

Figure 7 shows that BMF has a chemical structure similar to several types of starch, composed of many functional groups. According to the spectrum, their absorption bands referring to chemical bond vibrations were identified in the wavenumbers discussed below. The broad and intense absorption band at 3422 cm^{-1} refers to the hydrogen bond stretching vibrations associated with the starch structures free inter-and intramolecular hydroxyl groups (O–H). This band may also be related to free water molecules in the material. At 2930 cm^{-1}, the stretching vibrations of the C–H bonds of the methyl group (CH_3) were detected (Amin et al., 2019), and this absorption band was also associated with the stretching of CH_2 in lipids (Thivya et al., 2021). The band at 1638 cm^{-1} refers to the bending vibrations of the O–H bond in the adsorbed water molecules in the amorphous region (Pourfarzad et al., 2021). The C–H bending vibrations of the polysaccharides (Abdullah et al., 2018) were evidenced in the bands at 1439 and 1370 cm^{-1}. The other absorption bands are inherent to starch: 1159 and 1081 cm^{-1} (C–O, C–C and C–O–H stretching vibrations) (Nascimento et al., 2019; Moura et al., 2021); 994 cm^{-1} (C–O–H bending vibrations) (Xu et al., 2021); 930, 860, and 765 cm^{-1} (C–C–H and C–O–H stretching vibrations) (Singh et al., 2021).

The FTIR result agrees with the elemental analysis data (Table 1), where the predominance of functional groups consisting of C and H was observed, in addition to oxygen, an element present in the chemical structure of starchy flours. Regarding N, in all the literature investigated on BMF, the work by Maniglia (2017) was the only one that associated absorption bands with the proteins present in the composition of this raw material. According to their results, the absorption band located at 2921 cm^{-1} may be associated with the presence of proteins and at 1320 cm^{-1} with stretching the N–H bond of the amide group of proteins. It is essential to report that the discussion of protein-related absorption bands is more common for the spectrum of flours than for isolated starch. In the manuscript by De Dios-Avila et al. (2022), avocado seed flour was characterized by FTIR, and the band present between 1720 and 1600 cm^{-1} was attributed to the stretching of the C=O bonds of the amide group. Cangussu et al. (2021) and Wang et al. (2021) also observed the amide group between 1700–1600 cm^{-1}, corroborating the abovementioned results. According to Vieira et al. (2011b), the most significant differences between the babassu mesocarp and cellulose spectrum are in the absorption bands close to 860, 769, and 710 cm^{-1}. The authors reported that these bands are associated with vibrations of esters and monosubstituted aromatic rings due to the fraction of lignin present in the material.

Despite the conclusions that the FTIR spectrum can provide, the direct comparison of the spectrum of different materials becomes delicate since the discussion is based on identifying groups at similar wavenumbers, not precisely equals. This scenario varies from case to case; however, this observation must be considered, as it is possible to find some divergences in identifying a specific functional group when analyzing different manuscripts. The main reason is that the botanical origin of the material influences its chemical structure and properties. Despite this, FTIR has proven to be efficient in characterizing starchy raw materials, and researchers have taken full advantage of its potential to investigate the molecular interactions responsible for the structural organization of natural or chemically modified raw materials. Dankar et al. (2018) used FTIR to study the molecular structure of

raw potato, commercial potato powder, and potato puree prepared with potato powder. Pozo et al. (2018) characterized 13 types of starches by FTIR. A detailed discussion was presented in both manuscripts, highlighting the importance of FTIR for a better structural understanding of the materials.

4. Conclusions

BMF was characterized by several techniques to understand its chemical composition and inherent characteristics and, in future manuscripts, explore its potential for use as bio-reinforcement in composite films based on PLA. The objective of the characterizations was to value the flour by obtaining data and discussing its characteristics; since it is an industrial by-product, its use is still scarce in research for producing composite films. The elemental analysis technique showed that BMF is a mostly organic material due to starch being the main component of its composition. The organic character is interesting because PLA is also an organic material, so combining both would reduce environmental impacts. Despite this predominant character, XRF analysis showed inorganic chemical elements in their composition. BMF showed a heterogeneous granulometric distribution, reflecting the SSA result. The starch granules in the flour were visualized using microscopic techniques, mainly SEM, showing their sustainable nature. The structural techniques, XRD and FTIR, further reinforced the starchy character of the flour and its similarity with starches and other flours from different botanical origins, which gives the potential films ecological characteristics.

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Competing Interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this manuscript.

References

Abdullah, A. H. D., Chalimah, S., Primadona, I., & Hanantyo, M. H. G. (2018). Physical and chemical properties of corn, cassava, and potato starchs. *In IOP Conference Series: Earth and Environmental Science*, *160*, 012003. https://doi.org/10.1088/1755-1315/160/1/012003

Agüero, Á., Perianes, E. C., Abarca de las Muelas, S. S., Lascano, D., de la Fuente García-Soto, M. d. M., Peltzer, M. A., Balart, R., & Arrieta, M. P. (2023). Plasticized Mechanical Recycled PLA Films Reinforced with Microbial Cellulose Particles Obtained from Kombucha Fermented in Yerba Mate Waste. *Polymers*, *15*(2), 285.

https://doi.org/10.3390/polym15020285

Ahmed, J., Thomas, L., Arfat, Y. A., & Joseph, A. (2018). Rheological, structural and functional properties of high-pressure treated quinoa starch in dispersions. *Carbohydrate Polymers*, *197*, 649-657. https://doi.org/10.1016/j.carbpol.2018.05.081

Albiero, D., Maciel, A. J. S., Lopes, A. C., Mello, C. A., & Gamero, C. A. (2007). Proposta de uma máquina para colheita mecanizada de babaçu (*Orbignya phalerata* Mart.) para a agricultura familiar. *Acta Amazonica*, *37*(3), 337-346. https://doi.org/10.1590/S0044-59672007000300004

Amin, M. R., Chowdhury, M. A., & Kowser, M. A. (2019). Characterization and performance analysis of composite bioplastics synthesized using titanium dioxide nanoparticles with corn starch. *Heliyon*, *5*(8), e02009. https://doi.org/10.1016/j.heliyon.2019.e02009

Anyasi, T. A., Jideani, A. I. O., & Mchau, G. R. (2017). Effects of organic acid pretreatment on microstructure, functional and thermal properties of unripe banana flour. *Journal of Food Measurement and Characterization*, *11*, 99-110. https://doi.org/10.1007/s11694-016-9376-2

Araújo, A., Botelho, G., Oliveira, M., & Machado, A. V. (2014). Influence of clay organic modifier on the thermal-stability of PLA based nanocomposites. *Applied Clay Science*, *88-89*, 144-150. https://doi.org/10.1016/j.clay.2013.12.005

Araujo, J. A., Azeem, M., Venkatesh, C., Mojicevic, M., Fournet, M. B., & Attallah, O. A. (2023). Color Stability Enhancement of Curcumin Bioplastic Films Using Natural Hybrid Fillers of Montmorillonite and Revalorized Cellulose. *ACS Sustainable Chemistry & Engineering*, 1-15. https://doi.org/10.1021/acssuschemeng.3c01466

Asadollahzadeh, M., Mahboubi, A., Taherzadeh, M. J., Åkesson, D., & Lennartsson, P. R. (2022). Application of Fungal Biomass for the Development of New Polylactic Acid-Based Biocomposites. *Polymers*, *14*(9), 1738. https://doi.org/10.3390/polym14091738

Azevedo, A. P. S., Farias, J. C., Costa, G. C., Ferreira, S. C. P., Aragão-Filho, W. C., Sousa, P. R. A., Pinheiro, M. T., Maciel, M. C. G., Silva, L. A., Lopes, A. S., Barroqueiro, E. S. B., Borges, M. O. R., Guerra, R. N. M., & Nascimento, F. R. F. (2007). Anti-thrombotic effect of chronic oral treatment with *Orbignya phalerata* Mart. *Journal of Ethnopharmacology*, *111*(1), 155-159. https://doi.org/10.1016/j.jep.2006.11.005

Banakar, V. V., Sabnis, S. S., Gogate, P. R., Raha, A., Saurabh, & Adak, A. K. (2022). Comparison of sonocrystallization and seeding as pretreatment approaches for scale control to improve heat transfer in thermal brine concentrator. *Desalination*, *523*, 115444. https://doi.org/10.1016/j.desal.2021.115444

Baruque Filho, E. A., Baruque, M. G. A., & Sant'Anna Jr., G. L. (2000). Babassu coconut starch liquefaction: an industrial scale approach to improve conversion yield. *Bioresource Technology*, *75*(1), 49-55. https://doi.org/10.1016/S0960-8524(00)00026-2

Bhagia, S., Bornani, K., Agrawal, R., Satlewal, A., Ďurkovič, J., Lagaňa, R., Bhagia, M., Yoo, C. G., Zhao, X., Kunc, V., Pu, Y., Ozcan, S., & Ragauskas, A. J. (2021). Critical review of

FDM 3D printing of PLA biocomposites filled with biomass resources, characterization, biodegradability, upcycling and opportunities for biorefineries. *Applied Materials Today*, *24*, 101078. https://doi.org/10.1016/j.apmt.2021.101078

Błaszczak, W., & Lewandowicz, G. (2020). Light Microscopy as a Tool to Evaluate the Functionality of Starch in Food. *Foods*, *9*(5), 670. https://doi.org/10.3390/foods9050670

Borges, A. M., Pereira, J., & Lucena, E. M. P. (2009). Caracterização da farinha de banana verde. *Food Science and Technology*, *29*(2), 333-339. https://doi.org/10.1590/S0101-20612009000200015

Camargo, P. S. S. (2021). Reciclagem de módulos fotovoltaicos de silício cristalino: separação e concentração de materiais. [Dissertation]. Porto Alegre: Postgraduate Program in Mining, Metallurgical and Materials Engineering of Federal University of Rio Grande do Sul.

Cangussu, L. B., Fronza, P., Franca, A. S., & Oliveira, L. S. (2021). Chemical Characterization and Bioaccessibility Assessment of Bioactive Compounds from Umbu (*Spondias tuberosa* A.) Fruit Peel and Pulp Flours. *Foods*, *10*(11), 2597. https://doi.org/10.3390/foods10112597

Carneiro, M. I. F. (2011). Farelo e mesocarpo do côco do babaçu na alimentação de aves. [Thesis]. Jaboticabal: Postgraduate Program in Animal Science of Paulista State University.

Castro, A. M., Castilho, L. R., & Freire, D. M. G. (2016). Characterization of babassu, canola, castor seed and sunflower residual cakes for use as raw materials for fermentation processes. *Industrial Crops and Products*, *83*, 140-148. https://doi.org/10.1016/j.indcrop.2015.12.050

Cervantes-Ramírez, J. E., Cabrera-Ramirez, A. H., Morales-Sánchez, E., Rodriguez-García, M. E., de la Luz Reyes-Vega, M., Ramírez-Jiménez, A. K., Contreras-Jiménez, B. L., & Gaytán-Martínez, M. (2020). Amylose-lipid complex formation from extruded maize starch mixed with fatty acids. *Carbohydrate Polymers*, *246*, 116555. https://doi.org/10.1016/j.carbpol.2020.116555

Chakraborty, I., Pallen, S., Shetty, Y., Roy, N., & Mazumder, N. (2020). Advanced microscopy techniques for revealing molecular structure of starch granules. *Biophysical Reviews*, *12*, 105-122. https://doi.org/10.1007/s12551-020-00614-7

Chen, L., Ma, R., Zhang, Z., Huang, M., Cai, C., Zhang, R., McClements, D. J., Tian, Y., & Jin, Z. (2019). Comprehensive investigation and comparison of surface microstructure of fractionated potato starches. *Food Hydrocolloids*, *89*, 11-19. https://doi.org/10.1016/j.foodhyd.2018.10.017

Chyr, G., & DeSimone, J. M. (2023). Review of high-performance sustainable polymers in additive manufacturing. *Green Chemistry*, *25*(2), 453-466. https://doi.org/10.1039/D2GC03474C

Cruz, G., Silva, A. V. S., Silva, J. B. S., Caldeiras, R. N., & Souza, M. E. P. (2020). Biofuels from oilseed fruits using different thermochemical processes: opportunities and challenges. *Biofuels, Bioproducts and Biorefining*, *14*(3), 696-719. https://doi.org/10.1002/bbb.2089

Dankar, I., Haddarah, A., Omar, F. E. L., Pujolà, M., & Sepulcre, F. (2018). Characterization of food additive-potato starch complexes by FTIR and X-ray diffraction. *Food Chemistry, 260*, 7-12. https://doi.org/10.1016/j.foodchem.2018.03.138

De Dios-Avila, N., Tirado-Gallegos, J. M., Rios-Velasco, C., Luna-Esquivel, G., Isiordia-Aquino, N., Zamudio-Flores, P. B., Estrada-Virgen, M. O., & Cambero-Campos, O. J. (2022). Physicochemical, Structural, Thermal and Rheological Properties of Flour and Starch Isolated from Avocado Seeds of Landrace and Hass Cultivars. *Molecules*, *27*(3), 910. https://doi.org/10.3390/molecules27030910

Dearmitt, C. (2011). Functional Fillers for Plastics. *Applied Plastics Engineering Handbook*, 455-468. https://doi.org/10.1016/B978-1-4377-3514-7.10026-1

Di Paola, R. D., Asis, R., & Aldao, M. A. J. (2003). Evaluation of the Degree of Starch Gelatinization by a New Enzymatic Method. *Starch-Stärke*, *55*(9), 403-409. https://doi.org/10.1002/star.200300167

Engel, J. B., Ginity, M. M., Luchese, C. L., Tessaro, I. C., & Spada, J. C. (2020). Reuse of Different Agroindustrial Wastes: *Pinhão* and Pecan Nutshells Incorporated into Biocomposites Using Thermocompression. *Journal of Polymers and the Environment*, *28*, 1431-1440. https://doi.org/10.1007/s10924-020-01696-w

Ferdinánd, M., Várdai, R., Móczó, J., & Pukánszky, B. (2023). A novel approach to the impact modification of PLA. *Engineering Fracture Mechanics*, *277*, 108950. https://doi.org/10.1016/j.engfracmech.2022.108950

Ferreira, D. C. M., Molina, G., & Pelissari, F. M. (2020). Effect of Edible Coating from Cassava Starch and Babassu Flour (*Orbignya phalerata*) on Brazilian Cerrado Fruits Quality. *Food and Bioprocess Technology*, *13*(1), 172-179. https://doi.org/10.1007/s11947-019-02366-z

Fioroto, A. M., Nascimento, A. N., & Oliveira, P. V. (2015). In Vitro Evaluation of Cu, Fe, and Zn Bioaccessibility in the Presence of Babassu Mesocarp. *Journal of Agricultural and Food Chemistry*, *63*(28), 6331-6337. https://doi.org/10.1021/acs.jafc.5b01947

França, D. C., Almeida, T. G., Abels, G., Canedo, E. L., Carvalho, L. H., Wellen, R. M. R., Haag, K., & Koschek, K. (2019). Tailoring PBAT/PLA/Babassu films for suitability of agriculture mulch application. *Journal of Natural Fibers*, *16*(7), 933-943. https://doi.org/10.1080/15440478.2018.1441092

França, R. C., Chagas-Junior, A. F., Carvalho, E. E. N., Guimarães, L. G. L., Erasmo, E. A. L., & Oliveira, L. A. (2014). Mesocarpo de coco babaçu (*Orbignya* sp.) como fonte de carbono para produção de meios de cultura para *Trichoderma*. *Journal of Biotechnology and Biodiversity*, *5*(1), 88-94. https://doi.org/10.20873/jbb.uft.cemaf.v5n1.franca

Ghazvini, A. K. A., Ormondroyd, G., Curling, S., Saccani, A., & Sisti, L. (2022). An investigation on the possible use of coffee silverskin in PLA/PBS composites. *Journal of Applied Polymer Science*, *139*(22), 52264. https://doi.org/10.1002/app.52264

González-Pérez, S. E., Coelho-Ferreira, M., Robert, P., & Garcés, C. L. L. (2012). Conhecimento e usos do babaçu (*Attalea speciosa* Mart. e *Attalea eichleri* (Drude) A. J. Hend.) entre os Mebêngôkre-Kayapó da Terra Indígena Las Casas, estado do Pará, Brasil. *Acta Botanica Brasilica*, *26*(2), 295-308. https://doi.org/10.1590/S0102-33062012000200007

Govindaraju, I., Pallen, S., Umashankar, S., Mal, S. S., Melanthota, S. K., Mahato, D. R., Zhuo, G.-Y., Mahato, K. K., & Mazumder, N. (2020). Microscopic and spectroscopic characterization of rice and corn starch. *Microscopy Research and Technique*, *83*(5), 490-498. https://doi.org/10.1002/jemt.23437

Hosseini, S. M., Hosseini, H., Mohammadifar, M. A., German, J. B., Mortazavian, A. M., Mohammadi, A., khosravi-Darani, K., Shojaee-Aliabadi, S., & Khaksar, R. (2014). Preparation and characterization of alginate and alginate-resistant starch microparticles containing nisin. *Carbohydrate Polymers*, *103*, 573-580. https://doi.org/10.1016/j.carbpol.2013.12.078

Jagadeesan, S., Govindaraju, I., & Mazumder, N. (2020). An Insight into the Ultrastructural and Physiochemical Characterization of Potato Starch: a Review. *American Journal of Potato Research*, *97*, 464-476. https://doi.org/10.1007/s12230-020-09798-w

Jia, N., Lin, S., Zhang, F., Zheng, D., & Liu, D. (2022). Improved effect of flaxseed gum on the weakened gelling properties of myofibrillar protein induced by catechin. *Food Chemistry*, *372*, 131136. https://doi.org/10.1016/j.foodchem.2021.131136

Joye, I. (2019). Protein Digestibility of Cereal Products. *Foods*, *8*(6), 1-14. https://doi.org/10.3390/foods8060199

Kale, S. J., Jha, S. K., Jha, G. K., Sinha, J. P., & Lal, S. B. (2015). Soaking Induced Changes in Chemical Composition, Glycemic Index and Starch Characteristics of Basmati Rice. *Rice Science*, *22*(5), 227-236. https://doi.org/10.1016/j.rsci.2015.09.002

Kharrat, F., Khlif, M., Hilliou, L., Haboussi, M., Covas, J. A., Nouri, H., & Bradai, C. (2020). Minimally processed date palm (*Phoenix dactylifera* L.) leaves as natural fillers and processing aids in poly(lactic acid) composites designed for the extrusion film blowing of thin packages. *Industrial Crops and Products*, *154*, 112637. https://doi.org/10.1016/j.indcrop.2020.112637

Krummenauer, A. (2017). Desenvolvimento e validação de metodologia analítica para análise de aços por espectrometria de fluorescência de raios X por dispersão de energia (EDXRF). [Dissertation]. Porto Alegre: Postgraduate Program in Mining, Metallurgical and Materials Engineering of Federal University of Rio Grande do Sul.

Kweon, M., Slade, L., & Levine, H. (2008). Role of glassy and crystalline transitions in the responses of corn starches to heat and high pressure treatments: Prediction of solute-induced barostabilty from solute-induced thermostability. *Carbohydrate Polymers*, *72*(2), 293-299. https://doi.org/10.1016/j.carbpol.2007.08.013

Lemos, J. J. S., & Souza, R. C. (2018). Sistemas agroextrativistas como alternativa de

preservação da palmeira de babaçu no Maranhão. *Revista de Política Agrícola*, *27*(1), 82-95.

Lemos, R. B. S., & Andrade, M. R. S. (2021). Quebradeiras de coco babaçu na Internet: a ação do MIQCB nas redes sociais digitais. *Revista Eletrônica Competências Digitais para Agricultura Familiar*, *7*(2), 82-108.

Lopes, I. A., Paixão, L. C., Silva, L. J. S., Rocha, A. A., Barros Filho, A. K. D., & Santana, A. A. (2020). Elaboration and characterization of biopolymer films with alginate and babassu coconut mesocarp. *Carbohydrate Polymers*, *234*, 115747. https://doi.org/10.1016/j.carbpol.2019.115747

Lu, Z.-H., Donner, E., Tsao, R., Ramdath, D. D., & Liu, Q. (2018). Physicochemical and digestion characteristics of flour and starch from eight Canadian red and green lentils. *International Journal of Food Science & Technology*, *53*(3), 735-746. https://doi.org/10.1111/ijfs.13649

Luchese, C. L., Spada, J. C., & Tessaro, I. C. (2017). Starch content affects physicochemical properties of corn and cassava starch-based films. *Industrial Crops and Products*, *109*, 619-626. https://doi.org/10.1016/j.indcrop.2017.09.020

Ma, M., Chen, X., Zhou, R., Li, H., Sui, Z., & Corke, H. (2021). Surface microstructure of rice starch is altered by removal of granule-associated proteins. *Food Hydrocolloids*, *121*, 107038. https://doi.org/10.1016/j.foodhyd.2021.107038

Maniglia, B. C., & Tapia-Blácido, D. R. (2016). Isolation and characterization of starch from babassu mesocarp. *Food Hydrocolloids*, *55*, 47-55. https://doi.org/10.1016/j.foodhyd.2015.11.001

Maniglia, B. C. (2017). Aproveitamento de resíduos agroindustriais para elaboração de filmes biodegradáveis. [Thesis]. Ribeirão Preto: Postgraduate Program in Chemistry of University of São Paulo.

Maniglia, B. C., Tessaro, L., Lucas, A. A., & Tapia-Blácido, D. R. (2017). Bioactive films based on babassu mesocarp flour and starch. *Food Hydrocolloids*, *70*, 383-391. https://doi.org/10.1016/j.foodhyd.2017.04.022

Melo, E., Michels, F., Arakaki, D., Lima, N., Gonçalves, D., Cavalheiro, L., Oliveira, L., Caires, A., Hiane, P., & Nascimento, V. (2019). First Study on the Oxidative Stability and Elemental Analysis of Babassu (*Attalea speciosa*) Edible Oil Produced in Brazil Using a Domestic Extraction Machine. *Molecules*, *24*(23), 4235. https://doi.org/10.3390/molecules24234235

Melo, J. C. P. (2012). Celulose e lignocelulósicos como suportes na remoção de contaminantes em líquidos. [Thesis]. Campinas: Postgraduate Program in Chemistry of State University of Campinas.

Miotto, F. R. C., Restle, J., Neiva, J. N. M., Resende, P. L. P., Lage, M. E., Prado, C. S., Padua, J. T., & Araújo, V. L. D. (2012). Farelo de mesocarpo de babaçu (*Orbygnia* sp.) na terminação de bovinos: composição física da carcaça e qualidade da carne. *Ciência Rural*,

42(7), 1271-1277. https://doi.org/10.1590/S0103-84782012005000041

Mistry, A. N., Kachenchart, B., Pinyakong, O., Assavalapsakul, W., Jitpraphai, S. M., Somwangthanaroj, A., & Luepromchai, E. (2023). Bioaugmentation with a defined bacterial consortium: A key to degrade high molecular weight polylactic acid during traditional composting. *Bioresource Technology*, *367*, 128237. https://doi.org/10.1016/j.biortech.2022.128237

Möller, A. C., van der Padt, A., & van der Goot, A. J. (2021). From raw material to mildly refined ingredient – Linking structure to composition to understand fractionation processes. *Journal of Food Engineering*, *291*, 110321. https://doi.org/10.1016/j.jfoodeng.2020.110321

Moura, A., Bolba, C., Demori, R., Lima, L. P. F. C., & Santana, R. M. C. (2018). Effect of Rice Husk Treatment with Hot Water on Mechanical Performance in Poly(hydroxybutyrate)/Rice Husk Biocomposite. *Journal of Polymers and the Environment*, *26*, 2632-2639. https://doi.org/10.1007/s10924-017-1156-5

Muley, P. D., Henkel, C., Abdollahi, K. K., Marculescu, C., & Boldor, D. (2016). A critical comparison of pyrolysis of cellulose, lignin, and pine sawdust using an induction heating reactor. *Energy Conversion and Management*, *117*, 273-280. https://doi.org/10.1016/j.enconman.2016.03.041

Naozuka, J., Vieira, E. C., Nascimento, A. N., & Oliveira, P. V. (2011). Elemental analysis of nuts and seeds by axially viewed ICP OES. *Food Chemistry*, *124*(4), 1667-1672. https://doi.org/10.1016/j.foodchem.2010.07.051

Nascimento, J. M., Oliveira, J. D., & Leite, S. G. F. (2019). Chemical characterization of biomass flour of the babassu coconut mesocarp (*Orbignya speciosa*) during biosorption process of copper ions. *Environmental Technology & Innovation*, *16*, 100440. https://doi.org/10.1016/j.eti.2019.100440

Oliveira, N. A. (2018). Caracterização da farinha do mesocarpo e do óleo das amêndoas de Babaçu (*Orbignya phalerata*, Mart.) obtidos via extração com fluído supercrítico e líquido pressurizado: estudos pré-clínicos e toxicológicos. [Thesis]. Pirassununga: Faculty of Animal Science and Food Engineering of University of São Paulo.

Oliveira, T. A., Mota, I. O., Carvalho, L. H., Barbosa, R., & Alves, T. S. (2019). Influence of Carnauba Wax on Films of Poly (Butylene Adipate Co-Terephthalate) and Sugarcane Residue for Application in Soil Cover (Mulching). *Materials Research*, *22*, 1-10. https://doi.org/10.1590/1980-5373-MR-2019-0040

Omar, H.-C., Isidro, D.-C., Philip, L. J., & Adriana, E.-O. (2022). Film-stacking method as an alternative Agave tequilana fibre/PLA composite fabrication. *Materials Today Communications*, *31*, 103853. https://doi.org/10.1016/j.mtcomm.2022.103853

Özdemir, F., Ayrilmis, N., & Yurttaş, E. (2022). Mechanical and thermal properties of biocomposite films produced from hazelnut husk and polylactic acid. *Wood Material Science & Engineering*, *17*(6), 783-789. https://doi.org/10.1080/17480272.2021.1955972

Ozturk, O. K., Kaasgaard, S. G., Palmén, L. G., Vidal Jr., B., & Hamaker, B. R. (2021). Protein matrix retains most starch granules within corn fiber from corn wet-milling process. *Industrial Crops and Products*, *165*, 113429. https://doi.org/10.1016/j.indcrop.2021.113429

Padhi, S., & Dwivedi, M. (2022). Physico-chemical, structural, functional and powder flow properties of unripe green banana flour after the application of Refractance window drying. *Future Foods*, *5*, 100101. https://doi.org/10.1016/j.fufo.2021.100101

Pascoal, A. M., Di-Medeiros, M. C. B., Batista, K. A., Leles, M. I. G., Lião, L. M., & Fernandes, K. F. (2013). Extraction and chemical characterization of starch from *S. lycocarpum* fruits. *Carbohydrate Polymers*, *98*(2), 1304-1310. https://doi.org/10.1016/j.carbpol.2013.08.009

Pavlak, M. C. M., Zuniga, A. D., Lima, T. L. A., Arévalo-Pinedo, A., Carreiro, S. C., Fleury, C. S., & Silva, D. L. (2007). Aproveitamento da farinha do mesocarpo do babaçu (*Orbignya martiana*) para obtenção de etanol. *Evidência*, *7*(1), 7-24.

Pérez, S., Baldwin, P. M., Gallant, D. J. (2009). Structural Features of Starch Granules I. *Chemistry and Technology Food Science and Technology*, 149-192. https://doi.org/10.1016/B978-0-12-746275-2.00005-7

Ponte, M. R., Gadelha, A. M. T., Machado, Y. L., Lopes, A. A. S., Malveira, J. Q., Mazzetto, S. E., Lomonaco, D., & Rios, M. A. S. (2019). Blendas de bagaço de cana-de-açúcar, podas de mangueira e cajueiro: caracterização das propriedades e investigação de seus potenciais energéticos. *Matéria*, *24*(2), 1-12. https://doi.org/10.1590/S1517-707620190002.0687

Popescu, M.-C., Dogaru, B.-I., Goanta, M., & Timpu, D. (2018). Structural and morphological evaluation of CNC reinforced PVA/Starch biodegradable films. *International Journal of Biological Macromolecules*, *116*, 385-393. https://doi.org/10.1016/j.ijbiomac.2018.05.036

Porro, R. (2019). A economia invisível do babaçu e sua importância para meios de vida em comunidades agroextrativistas. *Boletim do Museu Paraense Emílio Goeldi. Ciências Humanas*, *14*(1), 169-188. https://doi.org/10.1590/1981.81222019000100011

Pourfarzad, A., Yousefi, A., & Ako, K. (2021). Steady/dynamic rheological characterization and FTIR study on wheat starch-sage seed gum blends. *Food Hydrocolloids*, *111*, 106380. https://doi.org/10.1016/j.foodhyd.2020.106380

Pozo, C., Rodríguez-Llamazares, S., Bouza, R., Barral, L., Castaño, J., Müller, N., & Restrepo, I. (2018). Study of the structural order of native starch granules using combined FTIR and XRD analysis. *Journal of Polymer Research*, *25*, 1-8. https://doi.org/10.1007/s10965-018-1651-y

Protásio, T. P., Trugilho, P. F., César, A. A. S., Napoli, A., Melo, I. C. N. A., & Silva, M. G. (2014). Babassu nut residues: potential for bioenergy use in the North and Northeast of Brazil. *SpringerPlus*, *3*, 1-14. https://doi.org/10.1186/2193-1801-3-124

Protásio, T. P., Guimarães Junior, M., Mirmehdi, S., Trugilho, P. F., Napoli, A., & Knovack, K.

M. (2017). Combustão da biomassa e do carvão vegetal da casca do coco babaçu. *Cerne*, *23*(1), 1-10. https://doi.org/10.1590/01047760201723012202

Rahaman, A., Kumari, A., Zeng, X.-A., Farooq, M. A., Siddique, R., Khalifa, I., Siddeeg, A., Ali, M., & Manzoor, M. F. (2021). Ultrasound based modification and structural-functional analysis of corn and cassava starch. *Ultrasonics Sonochemistry*, *80*, 105795. https://doi.org/10.1016/j.ultsonch.2021.105795

Rambo, M. K. D., Alexandre, G. P., Rambo, M. C. D., Alves, A. R., Garcia, W. T., & Baruque, E. (2015). Characterization of biomasses from the north and northeast regions of Brazil for processes in biorefineries. *Food Science and Technology*, *35*(4), 605-611. https://doi.org/10.1590/1678-457X.6704

Ribeiro, E. F., Polachini, T. C., Alvim, I. D., Quiles, A., Hernando, I., & Nicoletti, V. R. (2022). Microencapsulation of roasted coffee oil Pickering emulsions using spray- and freeze-drying: physical, structural and *in vitro* bioaccessibility studies. *International Journal of Food Science & Technology*, *57*(1), 145-153. https://doi.org/10.1111/ijfs.15378

Rodrigues, C. R. (2019). Degradação Térmica da Casca de Arroz e Utilização do Carvão Obtido no Processo para Polimento de Águas Cinzas. [Dissertation]. Palmas: Postgraduate Program in Environmental Engineering of Federal University of Tocantins.

Rodrigues, S. C. S., Mesquita, F. A. S., Carvalho, L. H., Alves, T. S., Folkersma, R., Araújo, R. S. R. M., Oliveira, A. D., & Barbosa, R. (2021). Preparation and characterization of polymeric films based on PLA, PBAT and corn starch and babassu mesocarp starch by flat extrusion. *Materials Research Express*, *8*(3), 1-10. https://doi.org/10.1088/2053-1591/abeaca

Rusu, I.-E., Marc, R. A., Mureşan, C. C., Mureşan, A. E., Filip, M. R., Onica, B.-M., Csaba, K. B., Alexa, E., Szanto, L., & Muste, S. (2021). Advanced Characterization of Hemp Flour (*Cannabis sativa* L.) from Dacia Secuieni and Zenit Varieties, Compared to Wheat Flour. *Plants*, *10*(6), 1237. https://doi.org/10.3390/plants10061237

Sánchez-Safont, E. L., Aldureid, A., Lagarón, J. M., Gámez-Pérez, J., & Cabedo, L. (2018). Biocomposites of different lignocellulosic wastes for sustainable food packaging applications. *Composites Part B: Engineering*, *145*, 215-225. https://doi.org/10.1016/j.compositesb.2018.03.037

Santos, A. M. S. (2016). Carbono ativado por pirólise a partir de biomassa de babaçu (*Orbignya speciosa*) e sua aplicação como adsorvente. [Dissertation]. Teresina: Postgraduate Program in Materials Science of Federal University of Piauí.

Santos, J. A. (2018). Celulose e mesocarpo de babaçu como complementos vetoriais para transformação genética bacteriana. [Dissertation]. Teresina: Postgraduate Program in Materials Science and Engineering of Federal University of Piauí.

Santos, P. A. C., Parente, M. O. M., Parente, H. N., Zanine, A. M., Moreira Filho, M. A., Alves, A. A., Ferreira, D. J., Gomes, R. M. S., & Santos, V. L. F. (2019). Babassu mesocarp flour in diet of finishing lambs. *Italian Journal of Animal Science*, *18*(1), 236-245.

https://doi.org/10.1080/1828051X.2018.1504635

Shahrampour, D., Razavi, S. M. A., & Sadeghi, A. (2023). Evaluation of green tea extract incorporated antimicrobial/antioxidant/biodegradable films based on polycaprolactone/polylactic acid and its application in cocktail sausage preservation. *Journal of Food Measurement and Characterization*, *17*(1), 1058-1067. https://doi.org/10.1007/s11694-022-01670-1

Silva, D. C., Lopes, I. A., Silva, L. J. S., Lima, M. F., Barros Filho, A. K. D., Villa-Vélez, H. A., & Santana, A. A. (2019). Physical properties of films based on pectin and babassu coconut mesocarp. *International Journal of Biological Macromolecules*, *130*, 419-428. https://doi.org/10.1016/j.ijbiomac.2019.02.151

Silva, J. S. (2016). Remoção de medicamento antineoplásico em meio aquoso por materiais adsorventes provenientes do babaçu (*Orbignya sp.*). [Dissertation]. Teresina: Postgraduate Program in Materials Science of Federal University of Piauí.

Singh, R., Kaur, S., & Aggarwal, P. (2021). Exploration of potato starches from non-commercial cultivars in ready to cook instant non cereal, non glutinous pudding mix. *LWT*, *150*, 111966. https://doi.org/10.1016/j.lwt.2021.111966

Singh, V., Ali, S. Z., Somashekar, R., & Mukherjee, P. S. (2006). Nature of Crystallinity in Native and Acid Modified Starches. *International Journal of Food Properties*, *9*(4), 845-854. https://doi.org/10.1080/10942910600698922

Srimalanon, P., Prapagdee, B., & Sombatsompop, N. (2020). Soil Inoculation with *Pseudomonas geniculata* WS3 for Accelerating the Biodegradation Process of In Situ Compatibilized PBS/PLA Blends Doped with HPQM. *Journal of Polymers and the Environment*, *28*, 1138-1149. https://doi.org/10.1007/s10924-020-01670-6

Sujka, M., & Jamroz, J. (2013). Ultrasound-treated starch: SEM and TEM imaging, and functional behaviour. *Food Hydrocolloids*, *31*(2), 413-419. https://doi.org/10.1016/j.foodhyd.2012.11.027

Sujka, M. (2017). Ultrasonic modification of starch – Impact on granules porosity. *Ultrasonics Sonochemistry*, *37*, 424-429. https://doi.org/10.1016/j.ultsonch.2017.02.001

Sun, X., Sun, Z., Saleh, A. S. M., Zhao, K., Ge, X., Shen, H., Zhang, Q., Yuan, L., Yu, X., & Li, W. (2021). Understanding the granule, growth ring, blocklets, crystalline and molecular structure of normal and waxy wheat A- and B- starch granules. *Food Hydrocolloids*, *121*, 107034. https://doi.org/10.1016/j.foodhyd.2021.107034

Sun, Y., Wang, M., Mur, L. A. J., Shen, Q., & Guo, S. (2020). Unravelling the Roles of Nitrogen Nutrition in Plant Disease Defences. *International Journal of Molecular Sciences*, *21*(2), 572. https://doi.org/10.3390/ijms21020572

Thivya, P., Bhosale, Y. K., Anandakumar, S., Hema, V., & Sinija, V. R. (2021). Exploring the Effective Utilization of Shallot Stalk Waste and Tamarind Seed for Packaging Film Preparation. *Waste and Biomass Valorization*, *12*, 5779-5794.

https://doi.org/10.1007/s12649-021-01402-4

Vargas, C. G., Costa, T. M. H., Rios, A. O., & Flôres, S. H. (2017). Comparative study on the properties of films based on red rice (*Oryza glaberrima*) flour and starch. *Food Hydrocolloids*, *65*, 96-106. https://doi.org/10.1016/j.foodhyd.2016.11.006

Vasconcelos, G. C. M. S., Carvalho, L. H., Araújo, F. H., Pinto, F. A. N. C., Barbosa, R., Santos, M. S. F., & Alves, T. S. (2023). Thermal and Acoustic Performance of Green Polyethylene/Cork Composite for Civil Construction Applications. *Materials Research*, *26*, 1-8. https://doi.org/10.1590/1980-5373-MR-2022-0232

Vassilev, S. V., Baxter, D., Andersen, L. K., & Vassileva, C. G. (2010). An overview of the chemical composition of biomass. *Fuel*, *89*(5), 913-933. https://doi.org/10.1016/j.fuel.2009.10.022

Vieira, A. P., Santana, S. A. A., Bezerra, C. W. B., Silva, H. A. S., Chaves, J. A. P., Melo, J. C. P., Silva Filho, E. C., & Airoldi, C. (2011a). Removal of textile dyes from aqueous solution by babassu coconut epicarp (*Orbignya speciosa*). *Chemical Engineering Journal*, *173*(2), 334-340. https://doi.org/10.1016/j.cej.2011.07.043

Vieira, A. P., Santana, S. A. A., Bezerra, C. W. B., Silva, H. A. S., Chaves, J. A. P., Melo, J. C. P., Silva Filho, E. C., & Airoldi, C. (2011b). Epicarp and mesocarp of babassu (*Orbignya speciosa*): characterization and application in copper phtalocyanine dye removal. *Journal of the Brazilian Chemical Society*, *22*(1), 21-29. https://doi.org/10.1590/S0103-50532011000100003

Vinhal, J. O., Lima, C. F., & Barbosa, L. C. A. (2014). Analytical pyrolysis of the kernel and oil of babassu palm (*Orbignya phalerata*). *Journal of Analytical and Applied Pyrolysis*, *107*, 73-81. https://doi.org/10.1016/j.jaap.2014.02.005

Volpe, V., De Feo, G., De Marco, I., & Pantani, R. (2018). Use of sunflower seed fried oil as an ecofriendly plasticizer for starch and application of this thermoplastic starch as a filler for PLA. *Industrial Crops and Products*, *122*, 545-552. https://doi.org/10.1016/j.indcrop.2018.06.014

Wang, B., Gao, W., Kang, X., Dong, Y., Liu, P., Yan, S., Yu, B., Guo, L., Cui, B., & Abd El-Aty, A. M. (2021). Structural changes in corn starch granules treated at different temperatures. *Food Hydrocolloids*, *118*, 106760. https://doi.org/10.1016/j.foodhyd.2021.106760

Wang, B., Dong, Y., Fang, Y., Gao, W., Kang, X., Liu, P., Yan, S., Cui, B., & Abd El-Aty, A. M. (2022). Effects of different moisture contents on the structure and properties of corn starch during extrusion. *Food Chemistry*, *368*, 130804. https://doi.org/10.1016/j.foodchem.2021.130804

Wang, J., Wang, J., Vanga, S. K., & Raghavan, V. (2021). Influence of high-intensity ultrasound on the IgE binding capacity of Act d 2 allergen, secondary structure, and *In-vitro* digestibility of kiwifruit proteins. *Ultrasonics Sonochemistry*, *71*, 105409.

https://doi.org/10.1016/j.ultsonch.2020.105409

Xu, F., Zhang, L., Liu, W., Liu, Q., Wang, F., Zhang, H., Hu, H., & Blecker, C. (2021). Physicochemical and Structural Characterization of Potato Starch with Different Degrees of Gelatinization. *Foods*, *10*(5), 1104. https://doi.org/10.3390/foods10051104

Yapuchura, E. R., Tartaglia, R. S., Cunha, A. G., Freitas, J. C. C., & Emmerich, F. G. (2019). Observation of the transformation of silica phytoliths into SiC and SiO_2 particles in biomass-derived carbons by using SEM/EDS, Raman spectroscopy, and XRD. *Journal of Materials Science*, *54*, 3761-3777. https://doi.org/10.1007/s10853-018-3130-6

Yusoff, N. H., Pal, K., Narayanan, T., & Souza, F. G. (2021). Recent trends on bioplastics synthesis and characterizations: Polylactic acid (PLA) incorporated with tapioca starch for packaging applications. *Journal of Molecular Structure*, *1232*, 129954. https://doi.org/10.1016/j.molstruc.2021.129954

Zhang, K., Zhao, D., Guo, D., Tong, X., Zhang, Y., & Wang, L. (2021). Physicochemical and digestive properties of A- and B-type granules isolated from wheat starch as affected by microwave-ultrasound and toughening treatment. *International Journal of Biological Macromolecules*, *183*, 481-489. https://doi.org/10.1016/j.ijbiomac.2021.04.180

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