



## Bioactive films based on babassu mesocarp flour and starch



Bianca C. Maniglia<sup>a</sup>, Larissa Tessaro<sup>a</sup>, Alessandra A. Lucas<sup>b</sup>, Delia R. Tapia-Blácido<sup>a,\*</sup>

<sup>a</sup> Departamento de Química, Faculdade de Filosofia, Ciências e Letras de Ribeirão Preto, Universidade de São Paulo, Ribeirão Preto, SP, CEP: 14040-90, Brazil

<sup>b</sup> Departamento de Engenharia de Materiais, Universidade Federal de São Carlos, São Carlos, São Paulo, CEP: 13565-905, Brazil

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### ABSTRACT

This work aimed to evaluate the properties of films prepared with babassu mesocarp flour or with starch isolated from babassu mesocarp by steeping in water (WS), alkaline steeping (KS), or acid steeping (AS). The films were obtained by casting; glycerol was used as plasticizer. Although the babassu flour presented high content of amylose, its major content of protein and fiber afforded a less mechanically resistant, more flexible, more opaque, more hydrophilic, more water vapor-permeable film with greater capacity to absorb water as compared to the babassu starch films. The WS, AS, and KS methods yielded purer starches with different contents of amylose, lipids, fibers, and total phenolic compounds, which influenced the mechanical and functional properties of the starch films. Due to its major content of amylose, the WS starch film was stronger, more rigid, less soluble, less water vapor-permeable, and more crystalline than the AS and the KS starch films. X-ray diffraction and SEM revealed that the WS starch film had denser and more ordered and homogeneous structure. However, the AS starch film had the highest antioxidant activity (70%) due to the largest amount of total phenolic compounds. The babassu flour and starch films exhibited good antioxidant activity, so they can be used as materials for packaging of oxidation-sensitive foods.

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### 1. Introduction

Biodegradable films have emerged as an environmentally friendly strategy to decrease the pollution originating from the accumulation of synthetic plastic waste. However, the application of these materials will only be feasible if they are competitive in terms of cost and functionality as compared to synthetic plastics. This might be possible if cheaper raw materials such as agroindustrial byproducts are used. This type of raw material may contain biopolymers such as protein, fiber, and starch, so it can be employed to produce films. These byproducts may also contain bioactive compounds like antioxidants and antimicrobials, which allows the production of bioactive films that offer extra benefits in relation to conventional materials (Andrade-Mahecha, Tapia-Blácido, & Menegalli, 2012; Chandra & Rustgi, 1998; Daudt et al., 2016; Dias, Müller, Larotonda, & Laurindo, 2010; Maniglia, Domingos, de Paula, & Tapia-Blácido, 2014; Pelissari, Andrade-Mahecha, Sobral, & Menegalli, 2013; Salas-Valero, Tapia-Blácido, & Menegalli, 2015).

Babassu mesocarp, a byproduct of the babassu oil extraction industry, is produced during the separation of the babassu coconut almonds. In 2014, 89.79 tons of babassu coconuts were harvested in the Northeastern, Northern, and Central Western regions of Brazil (IBGE, 2015; Machado, Chaves, & Antoniassi, 2006; Soler, Vitali, & Muto, 2007). The babassu oil corresponds to only 7% of the total weight of the fruit; the other parts like the epicarp, the endocarp, and the mesocarp (93%) are underused as biomass (Teixeira, 2008). The babassu endocarp and epicarp have high content of fixed carbon, which makes them more suitable for carbonization and burning than the babassu nuts and mesocarp, which contain less than 5% fixed carbon (Teixeira, 2008). The babassu mesocarp is usually transformed into flour and used as animal feed. In a previous work, Maniglia and Tapia-Blácido (2016) demonstrated that the babassu mesocarp flour displays antioxidant activity due to the presence of phenolic compounds. These authors isolated starch from the babassu mesocarp flour by two methodologies, steeping in water and steeping in alkaline pH. These methodologies provided starch with distinct chemical composition and functional characteristics. However, the isolation of starch did not remove the phenolic compounds, so the final starch still had antioxidant activity, which made it an interesting material to obtain bioactive films. Antioxidant activity is a desirable feature in materials that are

\* Corresponding author.

E-mail address: [delia@ffclrp.usp.br](mailto:delia@ffclrp.usp.br) (D.R. Tapia-Blácido).

used to cover fruits, meats, and cheeses, which are all sensitive to oxidation. Bioactive starch films have been produced through the addition of antimicrobial and antioxidant agents such as tannic acid (Pyła, Kim, Silva, & Jung, 2010), cinnamon and propolis essences (Kechichian, Ditchfield, Veiga-Santos, & Tadini, 2010), potassium sorbate and chitosan (Shen, Wu, Chen, & Zhao, 2010), ferulic acid (Mathew & Abraham, 2008), mango pulp and mate (Reis et al., 2015), and butter milk (Moreno et al., 2014) to the film. Research into films produced from agroindustrial residues bearing antimicrobial and antioxidant natural agents is scarce in the literature. In a previous work, Maniglia et al. (2014, 2015) prepared films from the extraction residue of turmeric pigment, to find that the film exhibited antioxidant activity due to the presence of curcuminoids.

This work aims: (1) to compare the mechanical and functional properties of babassu mesocarp flour and starch films (designated flour film and starch film, respectively), and (2) to evaluate how the methodology used to isolate babassu starch (steeping in acid, in water, or in alkaline medium) affected the properties of the starch films.

## 2. Material and methods

### 2.1. Material

The babassu mesocarp flour was provided by industry “Coopaesp” (Esperantinópolis – Maranhão, Brazil). The flour was sieved (80 mesh–0.180 mm) and stored at 10 °C.

Sodium Hydroxide and Ascorbic Acid P.A. were acquired from Dynamic – Contemporary Chemistry (São Paulo, Brazil). The radical DPPH (2,2-diphenyl-1-picrylhydrazyl) was purchased from Aldrich (São Paulo, Brazil). Glycerol P.A. grade was supplied by Sigma-Aldrich (São Paulo, Brazil).

### 2.2. Isolation of babassu starch

Babassu starches were isolated by three different methods: steeping in water (WS), alkaline steeping (KS), and acid steeping (AS). All the methods involved steeping and wet milling steps. To this end, the babassu mesocarp flour was soaked in deionized water (neutral pH – 7.0), in a solution of sodium hydroxide 0.25% (alkaline pH – 10.0), or in a solution of ascorbic 1% acid (acid pH – 4.0), respectively, at a 1:2 ratio, and left to stand at 10 °C for 18 h. Then, the moist babassu flour was wet milled for 2 min in a food processor (Walita) operating at maximum power. The milled material was screened through 80-, 200-, and 270-mesh stainless steel sieves. The material retained in the sieves was reprocessed and sieved again four additional times. The resulting liquid was centrifuged at 1500 × g at 10 ± 2 °C for 10 min (Quimis, Q222RM, Brasil). The supernatant was discarded, and the starch was re-suspended in water. This process was repeated until the starch reached pH 7.0, for all the methods (WS, KS, and AS). The starch was dried in air-circulating oven at 40 °C for 6 h (Quimis, Q314M292, Brasil) and later milled and sieved through 80-mesh sieves. The final starches were stored in sealed dark flasks at 10 °C.

### 2.3. Chemical composition

The chemical composition of the babassu mesocarp flour and of the starches isolated by WS, KS, or AS was determined in triplicate. The contents of moisture and ash were analyzed by the AOAC 920.151 and 92.303 methodologies, respectively (AOAC, 1997). The content of proteins was measured by the AOAC 926.86 methodology (AOAC, 2005); a nitrogen conversion factor of 6.25 was applied. The content of lipids was obtained according to the methodology of Bligh and Dyer (1959). The content of dietary fiber was determined

by an enzymatic method (amylase, protease, amylo-glucosydase enzymes) by means of the AOAC 985.29 methodology (AOAC, 1990). The content of starch was calculated by difference. The content of amylose was analyzed by the simplified iodine colorimetric method, according to the methodology proposed by Juliano (1971) and adapted by Martínez and Cuevas (1989).

### 2.4. Preparation of babassu films

The films were prepared by casting from a suspension of 4% starch or babassu mesocarp flour (w/w) in deionized water. This suspension was homogenized with a magnetic stirrer (IKA® C –MAG HS7, Marconi, Piracicaba – Brazil) for 30 min. Then, the suspension was heated at 81 °C for 30 min, the plasticizer glycerol was added (19 g of plasticizer/100 g of starch), and the suspension was finally heated for 15 min. Subsequently, the solution was poured onto acrylic plates, a weight of 0.15 g/m<sup>2</sup> was maintained, and the plates were dried at 35 °C (45% RH) for 10 h in an oven with forced circulation (SL 200–364, SOLAB®, Piracicaba–Brazil). Before characterization, all the films were preconditioned for at least 48 h in desiccators containing saturated solution of sodium bromide (58% RH).

### 2.5. Optical properties of the films

The color and opacity of the films were determined on a portable colorimeter MiniScan XE (Hunterlab – Riston, Virgínia, EUA) according to the Hunterlab method (1997). A CIE-Lab color scale was used to measure the degree of lightness (L\*), redness (+a\*) or greenness (–a\*), and yellowness (+b\*) or blueness (–b\*) of the films. The results were expressed as the average of five measurements at different parts of the surface of the film. The total color difference was calculated on the basis of Equation (2):

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}, \quad (2)$$

where  $\Delta L^*$ ,  $\Delta a^*$ , and  $\Delta b^*$  are the differences between the  $L^*$ ,  $a^*$ , and  $b^*$  parameters of the samples and the standard sample ( $L^* = 93.49$ ,  $a^* = -0.77$ , and  $b^* = 1.40$ ).

### 2.6. Antioxidant activity and total phenolic compounds

The antioxidant activity of the babassu mesocarp flour, the babassu mesocarp starches, the flour film, and the starch films was measured by the DPPH methodology (Martins, Cerqueira & Vicente, 2012) as described by Maniglia and Tapia-Blácido (2016).

The content of total phenolic compounds in the babassu mesocarp flour, the babassu mesocarp starches, the flour film, and the starch films was determined by the Folin-Ciocalteu colorimetric method (Hillis & Swain, 1959). Briefly, 0.5 mL of the diluted sample extract was transferred to tubes containing 0.5 mL of a 1:7 dilution of Folin-Ciocalteu's reagent in water. After 3 min, 1.0 mL of a solution of sodium carbonate (0.5 M) was added to the sample. The tubes were allowed to stand at room temperature for 10 min; then, the absorbance at 725 nm was measured on an HP spectrophotometer (Hewlett Packard 8453, EUA). The content of total phenolic compounds was expressed as mg of gallic acid equivalents (GAE)/100 g of sample. All the analyses were performed in triplicate.

### 2.7. Mechanical properties of the films

The mechanical tests were conducted on a texture analyzer TA TX Plus (TA Instrument, England). The tensile strength (TS) and the

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