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BRIEF COMMUNICATION

Impact of PLA Poly(Lactic Acid) and PBAT Poly(butylene adipate-co-terephthalate) Coating on the Properties of Composites with High Content of Rice Husk

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Abstract
Composites with high content of rice husk (80% based on the solids weight) without and with PLA (5%) and PBAT (2.5%) coating were produced and characterized. Thickness, morphology, thermal and mechanical properties, water absorption capacity and hydrophilicity were evaluated. The optical microscopy indicated a more uniform surface after double coating with PLA that was confirmed by the iodine test. The modulus of elasticity and deflection were not influenced by the thin layer of PLA. On the other hand, water absorption capacity decreased with both polymers used as coating, but lower values were reached with PLA. All materials presented hydrophobic character (contact angle higher than 90°) when measured instantly, with a small decreased after 5 min. The samples coated only once with PLA or PBAT showed analogous thermograms to the uncoated composite.

Keywords Composites · Cassava starch · Rice husk · PLA · PBAT · Coating

Introduction

Polymers from non-renewable sources are used in many applications because of their resistance, durability, availability, processing facility and low cost. However, this kind of materials usually brings serious problems to the environment due to its non-biodegradability and failures in recycling collection. So, the development of environmentally friendly materials to substitute the plastics based on petroleum has intensified. Among the vegetable raw materials, starch has received special attention for being an abundant resource of low cost with capacity to produce films, foams and composites [1–6]. However, in general, the materials based on starch show poor mechanical properties and low resistance to water when compared to that with conventional plastics obtained from petroleum. To improve the properties of some polymers, the use of filler as reinforcement in composites was evidenced in the last decade because of their high performance in terms of good mechanical properties, chemical resistance and low cost [7–12].

Currently, the use of natural fibers from agroindustrial residues that besides being underused, are generally not properly disposed in the environment has attracted much attention in recent years [13–18]. Several authors have reported the addition of natural fibers or agroindustrial residuals in composites based on starch in different solid basis percentage, as follow: 10% of eucalyptus cellulosic fibers [9]; 10% of wheat straw, hemp, cotton linter or cellulose fibers [19]; 10–20% of sugarcane bagasse [13], 5–20% of malt bagasse [16], 5–40% of sugarcane or asparagus peel fiber [20]; 9% of corn husk fiber [1], 10–40% of sesame cake [18] and 2–40% of peanut skin [21]. In a previous work, Spada et al. [6] highlighted the improvements provided in starch based composites by adding different percentages of rice husk as filler. The percentage of 60% of rice husk increased the water resistance, but the surface remains hydrophilic. To the best of our knowledge, there are no studies about the preparation and characterization of cassava starch-based foams using fiber or residual content higher than 60%. Thus, the first objective of the present study was to develop composites with low quantities of starch and high percentages of rice husk reaching 80% on solid basis. Although the high fiber content results in a very rigid material, it can be used in

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different applications such as support trays in food services, flowerpots, containers for appetizers, and also to make small furniture (stands for plants and office objects, for example).

The rice husk represents a residue produced in large-scale. It is estimated a worldwide production about 100 million tons per year. After maize and wheat, rice is one of the three main staple food crops grown in the world. The worldwide production of rice in 2018/19 reached 485 million metric tons [22]. The rice is grown extensively in Brazil, which occupies the ninth position in world production. The harvest reached a value about 12 million tons in 2017, with Rio Grande do Sul being the state of largest rice production in the country [23].

The second objective was to increase the water resistance of the composites. For minimizing the hydrophilicity and the water uptake by starch, studies has reported the application of coatings or hydrophobic materials impregnation. Among materials used as biodegradable coatings, poly (lactic acid) (PLA) [24, 25] and poly (butylene adipate terephthalate) (PBAT) [26, 27] has been studied. The use of PLA and PBAT as the polymer matrix for packaging some foodstuffs as vegetables and fruits it is not economically viable, since their cost ranges between 5 and 8 U$/kg, being much than the starch (0.5–2.5 U$/kg). So, the cassava starch/rice husk composites prepared in this work were coated with PLA and PBAT to improve their water resistance.

Materials and Methods

Materials

Native cassava starch with 26% amylose content and 12% moisture (Fritz & Frida, Brazil), and rice husk from a Company of Rio Grande do Sul/Brazil (11.64% of moisture; 1.81% of protein; 0.39% of total lipids; 4% of carbohydrates and 69% of total dietary fibers) were used. PLA (Ingeo™ Biopolymer 2003D, Natureworks) and PBAT (Ecoflex® F Blend C1200, BASF) were used to coat the samples. Chloroform to dissolve the polymers.

Preparation

Rice husk and starch were added based on the solids weight at 80% (w/w) and 20%, respectively, and 1 g of water was added for each 1 g of starch. The composites were obtained by heating the mixture of starch, rice husk and water in a water bath at 80 °C under stirring until the starch gelatinization as the methodology of Spada et al. [6]. Then, 6–7 g of the mixture were placed on a Teflon mold (100 mm × 25 mm × 3 mm) and compressed at 70 bar in a hydraulic press (SL11/20E, Solab, Brazil) at 175 °C for 8 min. After cooling, the PLA (5% w/v) and PBAT (2,5% w/v) were applied by immersion method based on Schimidt and Laurindo [28]. Tests samples were immersed once and twice in the polymer solution with chloroform. After the first coating, the sample was dried at 40 °C for 20 min and then immersed again.

The composites were kept in a glass box at room temperature at 50 ± 5% relative humidity ($MgNO_3$ saturated solution). The formulations and process conditions were defined based on preliminary tests (data not shown). The coating solution was prepared by dissolving PLA and PBAT in chloroform using magnetic stirring for 2 h.

Morphology Analysis and Iodine Test

Samples with no pre-treatment were placed directly on glass slides for visualization at × 5 magnification by transmittance metallographic microscopy (Bioptika, Model B100 Series Metallographic Microscope, Brazil). A simple qualitative test was performed with lugol's iodine solution to verify the coating quality, since it promotes a blue color in contact with starch.

Water Contact Angle

The sessile drop method was used to measure the water contact angle (WCA) using the Krüss equipment (DSA, Hamburg, Germany). A microsyringe was used to drip deionized water (3 µL) on the composite surface, and the drop was measured immediately after to drip the water. The photographic images acquisitions were taken with Drop Shape Analysis (DSA4) software. The measurements were done at least in triplicate.

Water Absorption Capacity (WAC)

Cobb method described in NBR NM ISO 535:1999 [29] with adaptations was used to determine the water absorption capacity. It is measured the mass gain of the sample after direct contact of one side with water. First, weighed samples were placed in contact with 100 mL of distilled water for 5, 10, and 30 min in quadruplicate. Then, the water excess was removed using tissue paper, and the samples were weighed again.

Flexural Mechanical Properties

The three-point bending method was used to conduct the flexural tests according to ASTM D790-02 [30] with adaptations. Velocity of 1 mm min^{-1} and span setting of 50 mm were used during the tests. Strips measuring 100 mm × 25 mm were allowed to deform until break in the texture analyzer (Instron 3369, Estados Unidos) with a 2 k-N load cell. The values of maximum flexural stress and

elongation were obtained in quintuplicate for each formulation and were reported as average ± standard deviation.

Thermogravimetric Analysis

A thermobalance (TA Instruments model SDT Q600) under a nitrogen atmosphere with a rate of 100 ml min^{-1} was used to perform the thermogravimetric analysis. The composites and raw materials were heated from 25 to 800 °C at a heating rate of 10 °C min^{-1}.

Statistical Analysis

Analysis of variance (ANOVA) and Tukey's mean comparison test ($p \leq 0.05$) were performed using Statistica software 13.0 (Statsoft, USA).

Results

Appearance and Morphology

The photographic image (Fig. 1) shows the visual aspect of the composites that exhibited a homogeneous surface. The PBAT or PLA layers were adhered to the starch/rice husk base without delamination or forming cracks on the surface. Moreover, both coatings become the surface sample more opaque, and the rice husk was visually less perceptible with the second layer. Besides that, the composites PLA1X, PLA2X and PBAT 1X were slightly thicker than the uncoated sample as can be seen in Table 3.

The optical microscopies of the composites without coating and coated with PLA and PBAT (Fig. 2) showed different pattern throughout the surface. The PBAT1X (Fig. 2b) was very similar to the uncoated sample, probably due to the lower PBAT solution concentration (2,5%). Darker regions richer in solids and lighter regions with voids can be seen in PBAT1X, PBAT2X and PLA1X. The presence of light spots was not detected in PLA2X indicating that the porous fibrous structure was covered with PLA to form a smooth surface. The iodine test confirmed the better surface coating with PLA2X, since the indicator did not react with the starch, remaining yellow.

Thermal Properties

Figure 3 shows the thermogravimetric analysis curves of rice husk, PLA, PBAT, cassava starch and the uncoated and coated composites. All samples showed a first mass loss step at approximately 60 °C referring to water evaporation. Two other stages of mass loss can be seen in the cassava starch (290 °C and 315 °C) and rice husk (315 °C and 350 °C). Lima et al. [31] also reported polysaccharide degradation at temperatures above 300 °C for different commercial flour. The second signal in the residue DTG curves can be related to the decomposition of cellulose and hemicellulose, which degrades between 180 and 340 °C; the third peak can indicate the thermal decomposition of lignin, which occurs between 350 and 450 °C [10].

The DTG curves of rice husk was similar to that found by Alias et al. [32] who divided the pyrolysis into three phases: drying and evaporation of light compounds (phase 1), devolatilization of most biomass (phase 2) and lignin decomposition (phase 3). Phase 2 corresponds to the degradation of hemicellulose (200–350 °C) and cellulose between 320 and 380 °C. Lignin gradually degrades in a temperature range between 380 and 700 °C [33].

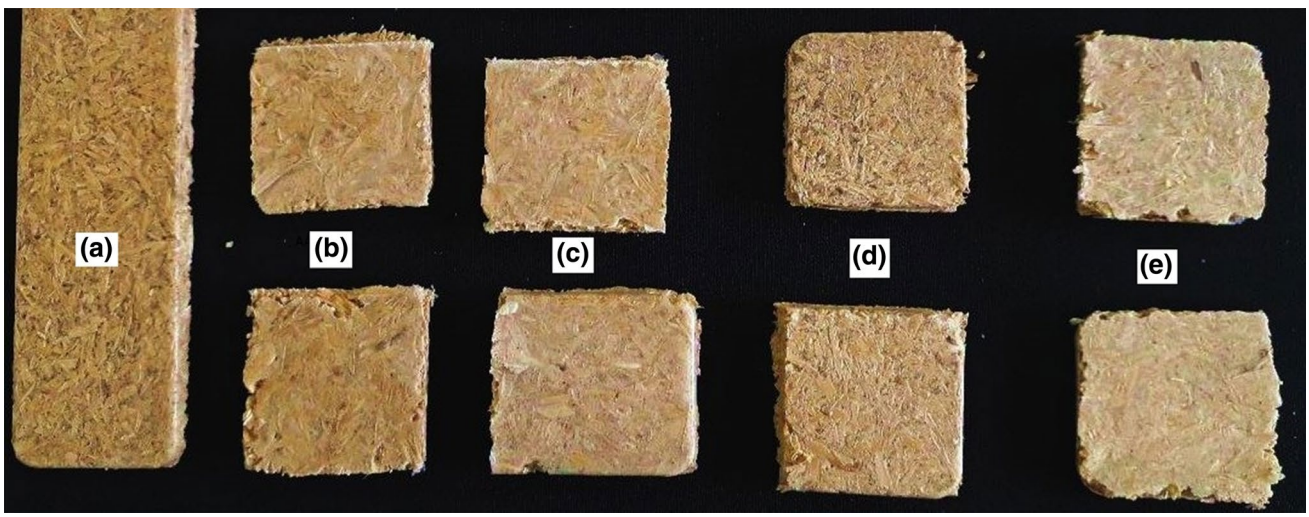

Fig. 1 Photographic images of the composites without coating (**a**) and coated: PBAT1X (**b**); PBAT2X (**c**); PLA1X (**d**) and PLA2X (**e**)

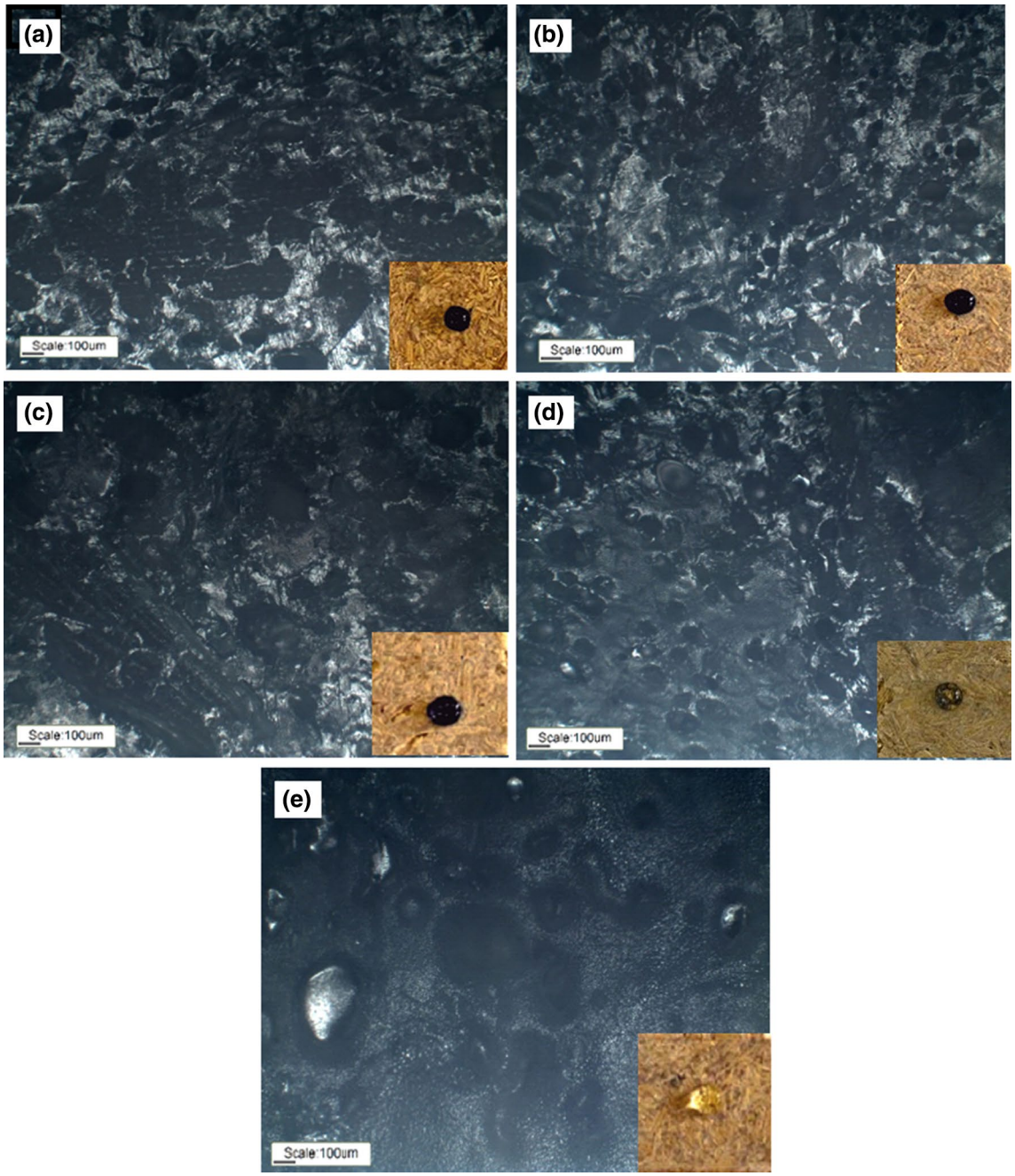

Fig. 2 Optical microscopy images at ×5 magnification of the composites without coating (**a**) and coated: PBAT1X (**b**); PBAT2X (**c**); PLA1X (**d**) and PLA2X (**e**). The photography in the right corner represents the iodine test

The composites showed similar thermograms to the rice husk (Fig. 3), maintaining the first peak pattern that represents the starch degradation. The samples coated only once with PLA or PBAT were analogous to the uncoated composite. When a second layer was made, the samples presented the peaks corresponding to PLA at 350 °C and PBAT at 420 °C. The percentage of residual inorganic compounds in the composites ranged between 23 and 30%, similar to the rice husk (29%).

Hydrophilicity and Water Absorption Capacity (WAC)

As showed in Table 1, the contact angles were statistically equal among the samples when measured instantly, indicating a hydrophobic character (> 100°). Because of the hydrophilic nature of gelatinized starch and rice husk [34], the samples presented a significantly reduction in the contact angle with time. After 1 min and 5 min, the uncoated

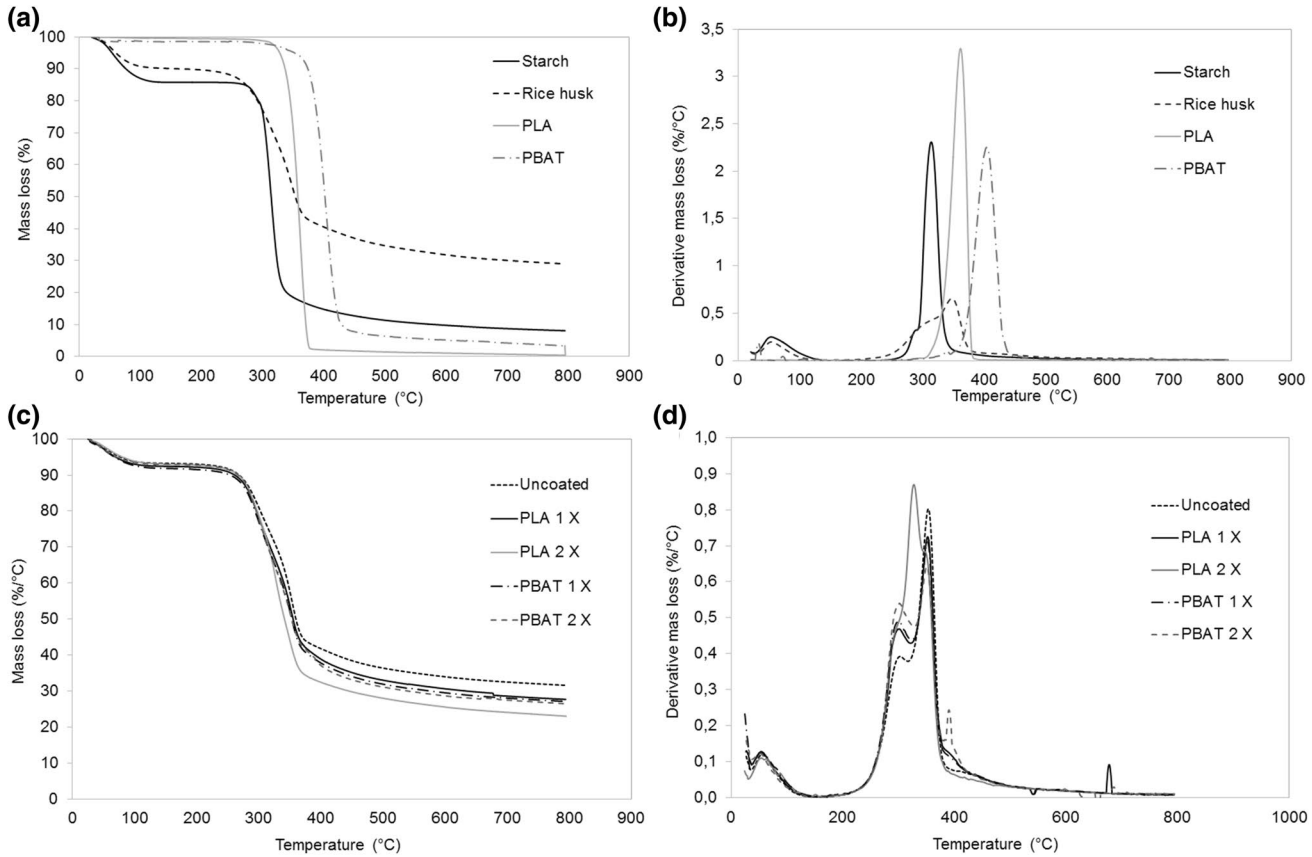

Fig. 3 **a** TGA and **b** DTG curves of cassava starch, rice husk, PLA and PBAT. **c** TGA and **d** DTG curves of the uncoated (control) and coated composites with PLA and PBAT

Table 1 Water contact angles of the uncoated and coated composites with PLA or PBAT

Sample	Water contact angle (°)		
	Instantly	1 min	5 min
Uncoated	103 ± 3^{aA}	101 ± 1^{bA}	89 ± 2^{cB}
PBAT1X	102 ± 2^{aA}	98 ± 2^{bA}	90 ± 2^{cB}
PBAT2X	101 ± 1^{aA}	99 ± 1^{bA}	95 ± 1^{bB}
PLA1X	102 ± 1^{aA}	100 ± 1^{bA}	95 ± 1^{bB}
PLA2X	107 ± 1^{aA}	106 ± 1^{aA}	104 ± 1^{aA}

Different lowercase letters in the same column indicate significant differences ($p < 0.05$) between the samples (Tukey's test) and different uppercase letters in the same line indicate significant differences ($p < 0.05$) between the contact time for the same sample (Tukey's test)

samples were equal to PBAT 1X, i.e., the wettability was equal regardless the coating presence. Differently, the surface presented higher values with PLA2X and remained with the same behavior during the analysis.

Likewise, Stoffel et al. [35] also verified a lower wettability of trays based on starch after coating with 10, 12.5 and 15% (w/v) of PLA. While the uncoated trays presented a measurement of 62° after 10 min, the coated samples presented values higher than 70°. After 20 min of contact with the water drop, a reduction in the contact angle value was also observed; the trays having a cover of 10, 12.5 and 15% of PLA had 67.7°, 65.4° ± 2.4 and 84.4°, respectively. The uncoated tray also showed a reduction, changing to 55.2°.

The results are in agreement with Rhim et al. [36] who obtained contact angle measurements of 57.7 ± 12.6° and close to 80° for uncoated and coated cardboard with PLA. Differently, Shankar and Rhim [27] stated that the wettability of the Kraft paper was not influenced by the coating with PBAT.

By increasing the amount of husk, the sample structure becomes more closed and with less pores, making water diffusion difficult [6]. In samples made only with starch, this value is up to four times higher. Moreover, after gelatinization starch becomes water soluble, losing its integrity when immersed in water for long times. Rice husk is insoluble and presents high proportion of lignin and silica (15–20%) [37], components with low water affinity [38].

The water absorption capacity for 5, 10 and 30 min are shown in Table 2. All coated composites presented lower WAC when compared to that without coating. The WAC of

Table 2 Water absorption capacity (WAC) at different immersion times of the composites without coating and coated with PLA or PBAT

Composite	WAC (%) 1 min	WAC (%) 5 min	WAC (%) 10 min	WAC (%) 20 min
Uncoated	10 ± 2^{bA}	14 ± 3^{bA}	26 ± 4^{aA}	24 ± 4^{aA}
PLA1X	5 ± 2^{aB}	7 ± 3^{aBC}	6.4 ± 0.9^{aC}	6 ± 1^{aC}
PLA2X	1.5 ± 0.2^{bC}	3 ± 1^{aC}	4 ± 0.5^{aC}	5 ± 1^{aC}
PBAT1X	5 ± 1^{cB}	8.1 ± 0.3^{bB}	12 ± 2^{aB}	13 ± 2^{aB}
PBAT2X	4.2 ± 0.8^{cB}	7 ± 1^{bBC}	10 ± 2^{aB}	12.8 ± 0.6^{aB}

Different lowercase letters indicate significant differences between the samples in the same line by Tukey's test ($p < 0.05$)

Different uppercase letters indicate significant differences between the samples in the same column by Tukey's test ($p < 0.05$)

Table 3 Flexural mechanical properties of the composites without coating and coated with PLA or PBAT

Sample	Thickness (mm)	Stress at break (MPa)	Strain at break (%)	Modulus of elasticity (MPa)
Uncoated	3.59 ± 0.12^{b}	6 ± 1^{b}	1.1 ± 0.1^{a}	759 ± 91^{a}
PBAT1X	3.56 ± 0.10^{b}	5 ± 1^{b}	1.2 ± 0.3^{a}	535 ± 92^{b}
PBAT2X	3.76 ± 0.15^{a}	5 ± 1^{b}	1.4 ± 0.2^{a}	461 ± 30^{b}
PLA1X	3.77 ± 0.08^{a}	9 ± 2^{a}	1.2 ± 0.1^{a}	723 ± 118^{a}
PLA2X	3.84 ± 0.07^{a}	10 ± 1^{a}	1.1 ± 0.3^{a}	808 ± 87^{a}

Different lowercase letters in the same column indicate significant differences ($p < 0.05$) between the samples (Tukey's test)

the PLA1X remains the same after 20 min. Differently, the PLA2X differ between 1 and 5 min, but it did not differ for longer times. The WAC for PLA was very low even after 20 min in contact with water, confirming its water resistance. The WAC varied significantly from 10% (1 min) to 26% (10 min) for the composite without coating, but it was statically equal between 10 and 20 min. A similar behavior was observed for the PBAT samples (1X or 2X) that increased the WAC in the first minutes. The PBAT-coated samples with increased water resistance can be suitably used as boxes for the distribution packaging of highly respiring fruits or vegetables, for example. Similarly, the water resistance of Kraft paper was dramatically down after coating with PBAT [27] and TPS foams with 6% w/v PLA coating exhibited an excellent reduction of 225% in water absorption when compared to TPS foam [39].

A poor water resistance of semi rigid starch samples was reported by Machado et al. [18] who found values ranging from 22% (1 min) to 154% (30 min) and Debiagi et al. [40]. Spada et al. [6] verified a large WAC reduction when 20, 40 and 60% of rice husk were added to starch foams. When 60% of rice husk was added to the starch foam, the water uptake capacity varied from $120 \pm 5\%$ to $26 \pm 8\%$ after 20 min in water, corresponding to a reduction of 79% associated with the presence of lignin that contains crystalline portions resistant to water molecules.

Flexural Mechanical Properties

Table 3 shows the mechanical properties of the composites with and without coating. The incorporation of 80% rice husk resulted in values equal to that reported by Spada et al. [41] for samples with 40% rice husk. When compared to sample with 60%, it presented lower values of maximum flexural stress and modulus of elasticity, i.e., the material stiffness was altered incorporating more rice husk, weakening the composite structure. The low value of elongation at break was expected and has been reported by many researchers who work with rice husk [42–44]. The presence of silica and fibers could enhance some physical properties as commented above but decrease the flexibility of composites.

The PLA and PBAT leads to significant statistical differences in the flexural mechanical properties, as shown in Table 3. While the maximum flexural strength of the coated composites with PLA was increased, the modulus of elasticity with PBAT was reduced. Despite of this, the strain at break remained statistically equal. The tensile strength and modulus of elasticity of foams based on potato starch was increased after coating with PLA (2%, 4% and 6% w/v) as commented by Bergel et al. [39].

The results with PLA can be explained by the voids reduction in the composites as observed in Fig. 2, i.e., the polymer can have permeated inside the samples. Rhim et al. [45] and Zhang et al. [46] also found similar behavior in the mechanical properties of materials with PLA coating. Differently from PLA, PBAT is a polymer of high flexibility.

Conclusion

Composites with good properties were prepared by coating the material with PBAT and PLA using a solution method. The coated composites had significantly lower water absorption with increased hydrophobicity. Also, the composites coated with PBAT showed lower rigidity. The double coating with PLA showed more promising results for the industrial scale production when a water resistance is required for packaging of foods or highly respiring fruits and vegetables.

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