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Properties of Cassava Bagasse and Polyvinyl Alcohol Biodegradable Foams

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Abstract The objectives of this work were to develop biodegradable foam trays based on cassava bagasse (CB) and polyvinyl alcohol (PVA) and to study the effects of these components on the microstructure, physicochemical and mechanical properties of the trays. The trays were produced by baking mixtures of 90–100 % (w/w) of CB and 0–10 % (w/w) of PVA. All of the formulations were able to form well-shaped and homogeneous trays with a good appearance. Incorporation of PVA improved the production yield and reduced the water sorption capacity of the produced materials, mainly at low levels. Moreover, the addition of PVA resulted in an increase in stress and strain at break values. The most positive effects of PVA were obtained when this polymer was used at lower levels (2.5 and 5.0 %).

Keywords Agro-industrial residue · Mechanical properties · New materials

Introduction

Expanded polystyrene (EPS) is classified as a chemically inert non-biodegradable plastic material and is largely used as food packaging, mainly for perishable products such as vegetables, fruits, sliced cheese and ham. Because of the difficulty of recycling this material, after its use, EPS packaging is deposited in sanitary landfills or garbage dumps. The use of biodegradable polymers as substitutes

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Department of Biochemistry and Biotechnology, CCE, State University of Londrina, PO Box 10.011, Londrina, PR 86057-970, Brazil e-mail: smali@uel.br for EPS appears to be an excellent alternative that will reduce negative effects on the environment, and this issue has received considerable attention from researchers [1–6].

Brazil is known to have a high diversity of renewable natural resources. Cassava (*Manihot esculenta*) is a root crop that is classified as the fifth most abundant starch crop produced in the world and the third most important food source for inhabitants of tropical regions. In 2,010, Brazil produced 26,595,000 t of cassava, representing 11 % of the world's production [7].

Cassava bagasse (CB) is the most important residue from industrial cassava starch extraction. This residue is mainly composed of water (70–80 wt%), residual starch and cellulose fibers, and its composition depends on the extraction process. The fibers content ranges between 15 and 50 wt% of the total solid residue, and the remainder of the solids is residual starch, ranging from 85 to 50 wt% [8, 9]. The final destination of CB is an important problem for the starch industry. The drying and transport process is expensive due to the high humidity of the material, and generally, this by-product is used in animal feed.

Several researchers have studied different uses for CB, such as the production of lactic acid by bacteria [10], production of ethanol [11] and development of biodegradable packaging [12, 13]. As is the case for all starchrich materials, CB packaging has poor mechanical properties and is susceptible to moisture when it is stored at high relative humidity. Some authors have reported that the resistance of starch materials to direct contact with water showed an improvement with the addition of polyvinyl alcohol (PVA). PVA is a synthetic polymer that is particularly well-suited for the formulation of blends with natural polymers [14–17].

Thus, the objectives of this work were to develop biodegradable trays based on CB and PVA and to study the effects of these components on the microstructure, physicochemical and mechanical properties of the trays.

Experimental

Materials

Cassava bagasse (CB) containing approximately 14 wt% fibers and 86 wt% starch was obtained from cassava roots as the liquid residue after laboratorial starch extraction [18]. The liquid residue was dried (50 °C/24 h) and milled to yield particles <0.35 mm. Glycerol, magnesium stearate and guar gum were purchased from Synth (Labsynth, São Paulo, Brazil). PVA (ACS grade, molecular weight 72,000 and degree of hydrolysis of 86.5–89.5 %) was purchased from Reagen (Quimibrás, Rio de Janeiro, Brazil).

Tray Production

The CB trays were manufactured using different formulations on the basis of previous results. Several concentrations of PVA ranging from 2.5 to 35 % w/w were previously tested, and the best range (2.5–10 % w/w) was chosen to be used in this work based on the appearance, cost and production yield. Formulations with more than 10 % w/w of PVA were very difficult to result in wellshaped trays.

All formulations are shown in Table 1. The CB content ranged from 90 to 100 % (w/w), and the PVA content ranged from 0 to 10.0 % (w/w). The baking process was performed according to Vercelheze et al. [2] with modifications. The CB, PVA, water and additives (1 % w/w magnesium stearate and 1 % w/w guar gum) were mixed for 5 min with a mechanical stirrer at 18.000 rpm (Fisatom, Brazil), and glycerol (10 % w/w) was then added. The magnesium stearate was added to prevent the starch foam sticking to the mold and the guar gum was added to prevent solid separation [15], and their levels were determined according to literature data [12]. After further stirring for 5 min, 40–43 g of each formulation was

 Table 1
 Compositions of the batters used to prepare the trays based on cassava bagasse (CB) and polyvinyl alcohol (PVA)

Samples	CB (g/100 g solids)	PVA (g/100 g solids)	Water (mL/100 g solids)	Batter amount (g)
PVA 0	100	0	210	43
PVA 2.5	97.5	2.5	210	40
PVA 5.0	95.0	5.0	210	40
PVA 7.5	92.5	7.5	210	40
PVA 10.0	90.0	10.0	210	40

homogeneously layered onto a Teflon mold (10×10 cm), and thermopressing was applied with a hydraulic press (JOMAQ, São Paulo, Brazil) for 7 min at 150 °C and 100 bar. Finally, the trays were removed from the press and stored for 7 days at 25 ± 0.5 °C and 58 % relative humidity before characterization.

Production Yield

The production yield is indicative of the efficiency of the process for each formulation. Trays of each formulation were produced separately over a period of 6 h, and the production yield was calculated as the percentage of complete trays produced [13].

Thickness and Density

The tray thickness was measured with a manual micrometer (Mitutoyo, Japan). Thickness measurements were performed using strips measuring 25 mm by 25 mm; the reported value is the average of 8 measurements of each tested sample.

The density was calculated as the relationship between weight and thickness [15]. The reported values are the averages of 8 determinations for each formulation.

Color

Tray color was determined using a colorimeter (CR 10, Minolta Chroma Co., Osaka, Japan). The color parameters range from L = 0 (black) to L = 100 (white), -a (greenness) to +a (redness), and -b (blueness) to +b (yellowness). The instrument was calibrated using a set of three Minolta calibration plates. The reported values are the averages of five determinations for each formulation.

Scanning Electron Microscopy (SEM)

SEM analyses were performed with a FEI Quanta 200 microscope (Oregon, USA). The foam pieces were mounted on bronze stubs using double-sided tape and then coated with a layer of gold (40–50 nm) for surface and cross-section visualization. To obtain the cross-sections, the samples were immersed in liquid nitrogen to prevent deformation during sectioning. All samples were examined using an accelerating voltage of 20 kV.

Crystallinity

The crystallinity of the foams was investigated by X-ray diffraction. The analysis was performed with a Panalytical X'Pert PRO MPD diffractometer (Netherlands), using K α copper radiation ($\lambda = 1.5418$ Å), at 40 kV voltage and

30 mA current. All assays were performed with ramping at 1°/min. The relative crystallinity index was calculated using Ruland's method [19] as follows: $CI = ((A_c)/(A_c + A_a))$, where A_c is the crystalline area, and A_a is the amorphous area.

Water Absorption Capacity (WAC)

The water absorption capacity was evaluated according to the Cobb method [20]. First, samples measuring 2.5 cm by 5 cm were weighed and soaked in 100 mL of distilled water for 1, 5, 10, 15, 20, 25 and 30 min. After removing the excess water with tissue paper, the samples were weighed again. The quantity of absorbed water was calculated according to the weight difference and expressed as the mass of absorbed water per mass of the original sample. The reported values are the means of five determinations for each formulation.

Water Sorption Isotherms

The water sorption isotherms were obtained using starch foam specimens $(2.5 \times 2.5 \text{ cm})$ that were pre-dried for 14 days over anhydrous calcium chloride and then placed over saturated salt solutions at 25 °C in separate desiccators with the desired levels of water activity (0, 0.11, 0.33, 0.11, 00.45, 0.58, 0.75 and 0.90) [21] for 7 days. The equilibrium moisture was determined by drying at 105 °C and was calculated from the increase in the mass of the dried sample after equilibration at a given RH. All tests were conducted in triplicate. The sample dimensions (width, length and thickness) were recorded at the initial and final incubation times (7 days) under each RH condition for the water sorption isotherm assays. The GAB (Guggenheim-Anderson-de Boer) model was used to fit the starch foam water sorption isotherm data, and monolayer values were calculated from the equations [22]. The GAB isotherm model can be expressed as follows: $M = m_0 C K a_w / (1 - m_0 C K a_w) / (1 - m_0 C$ Ka_w)(1 - Ka_w + CKa_w), where *M* is the equilibrium moisture content at a given water activity (a_w) , m₀ is the monolayer value (g water/g solids), and C and K are GAB constants. All tests were conducted in triplicate.

Mechanical Properties

A Brookfield CT3 texture analyzer (USA) with a 25-N load cell was used to determine the mechanical properties of the foam samples. Tensile tests were performed using strips measuring 80 mm by 25 mm, an initial grip separation of 80 mm and a crosshead speed of 2 mm/s. Stress–strain curves were recorded during extension, and stress and strain at break were determined. The reported values are the averages of 6 assays.

Statistical Analysis

Analyses of variance (ANOVA) and Tukey's mean comparison test ($p \le 0.05$) were performed using Statistica software version 7.0 (Statsoft, OK, USA).

Results and Discussion

Production Yield

All of the tray formulations produced in this work were able to form well-shaped, homogeneous trays with good appearance, and no evident cracks were observed (Fig. 1). Production yields ranged from 90 to 100 % (Table 2); only the PVA 0 sample (produced with 100 % CB) had 90 % production yield, and the addition of PVA at all levels (2.5-10 %) increased the production yield to 100 %, improving the foaming ability of the CB under the studied conditions (Table 2). Cassava starch and PVA are compatible materials that interact by hydrogen bonding [23–26], which can reinforce the polymeric matrices and result in more expandable trays.

Thickness and Density

The thickness of the trays ranged from 1.62 to 2.04 mm (Table 2), and the density ranged from 0.52 to 0.82 g/cm³ (Table 2); the PVA 0 sample had the lowest thickness and the highest density values. PVA incorporation at all levels significantly (Tukey test, $p \le 0.05$) increased the thickness and decreased the density of the samples. The processing of starches results in stiff materials that do not support air cell growth in their foams [27], and PVA acted as a reinforcing agent that improved the foaming ability of the starch pastes, resulting in more expandable materials with higher thickness and lower density.

Color

The color parameters (L*, a* and b*) of the trays are presented in Table 2. The luminosity (tendency to white color) ranged from 65.23 to 79.03, and the addition of PVA increased the luminosity; the whitest samples were PVA 5.0 and PVA 7.5. Our results agreed with those of Mali et al. [17], who reported that PVA incorporation in starch materials results in whiter samples.

An increase in the b* parameter indicates a tendency to a yellow color [3], and our results for this parameter ranged from 13.32 to 15.90 (Table 2), indicating that our samples had a yellowish aspect, as visually observed in Fig. 1. The incorporation of PVA did not significantly affect the b* parameter. An increase in the a* parameter is indicative of Fig. 1 Appearance of

cassava bagasse and PVA:





Table 2 Production yield, thickness, density, color parameters and crystallinity index (CI) of biodegradable trays based on with cassava bagasse and polyvinyl alcohol

Samples	Production yield (%)	Thickness (mm)	Density (g/cm ³)	Color parameters			CI (%)
				L*	a*	b*	
PVA 0	90	$1.62 \pm 0.19b$	$0.82\pm0.06a$	$65.23 \pm 2.99c$	$1.72\pm0.62a$	$14.62 \pm 1.99a$	4.52
PVA 2.5	100	$1.99 \pm 0.10a$	$0.57\pm0.19\mathrm{b}$	$76.61 \pm 4.86b$	$1.88 \pm 1.44a$	$15.90 \pm 2.14a$	5.10
PVA 5.0	100	$1.98 \pm 0.18a$	$0.52\pm0.14\mathrm{b}$	$79.02 \pm 1.13a$	$1.67\pm0.87a$	$15.02\pm2.61a$	5.30
PVA 7.5	100	$1.84\pm0.06a$	$0.60\pm0.04\mathrm{b}$	$79.03 \pm 1.98a$	$1.14 \pm 0.25a$	$14.19 \pm 1.05a$	6.12
PVA 10.0	100	$2.04\pm0.15a$	$0.55\pm0.07b$	$75.76\pm2.41b$	$0.98\pm0.37a$	$13.32\pm1.56a$	6.15

The data are the means of replicate determinations \pm standard deviation

Different letters in the same column indicate significant differences ($p \le 0.05$) between means (Tukey test)

the red color of a sample, which was not observed in this work once the a* values reached values close to zero. According to Famá et al. [28], in samples containing lignocellulosic fibers, the redness aspect is related to the lignin content, and as CB is rich in hemicelluloses and cellulose [29] redness is not characteristic of the material.

Scanning Electron Microscopy (SEM)

Figure 2 shows scanning electron micrographs of the produced trays. It was observed that the trays had a sandwichtype structure with dense outer skins and large cells in the interior, such as those reported by other authors working with baked starch trays [2–5]. The samples with incorporated PVA showed more expanded structure that can be observed by the presence of large air cells in the interior, and these results are in accord with the thickness and density data.

Crystallinity

As observed in the X-ray diffraction patterns (Fig. 3), the raw materials (CB and PVA) showed semicrystalline structure. CB presented characteristic peaks at $2\theta = 15.3^{\circ}$; 17.3° ; 18.3° and 23.5° , and according to data in the literature [30, 31], these peaks are related to cassava starch present in the bagasse, which has C-type crystallinity. Furthermore, the crystallinity indexes of CB and PVA were calculated as 21.08 % and 25.44 %, respectively. According to data in the literature [29], cellulose from CB has a characteristic peak at $2\theta = 22.3^{\circ}$, which occurred in a close angle to the 23.5° related to the starch, which is



Fig. 3 X-ray diffraction patterns of cassava bagasse, PVA and biodegradable trays based on cassava bagasse and PVA



Fig. 2 Cross-sections (SEM) of biodegradable trays based on cassava bagasse and PVA: a PVA 0; b PVA 2.5; c PVA 5.0; d PVA 7.5 and e PVA 10

present in a large proportion in the bagasse. PVA presented characteristic peaks around $2\theta = 19.5^{\circ}$ and $2\theta = 23^{\circ}$. These peaks have been reported by other authors [32–34].

As observed in the X-ray diffraction patterns (Fig. 3), the trays showed an amorphous structure, mainly as a consequence of the starch gelatinization during the trays' processing. The crystallinity index of the trays ranged from 4.52 to 6.15 % (Table 2). During the thermal processing of native starch, the granular structure is completely or partially destroyed, resulting in an amorphous matrix [35]. Our results are consistent with those of Vercelheze et al. [2] and Matsuda et al. [4], who related the loss of the crystalline structure in the thermal processing of starch materials.

Water Absorption Capacity (WAC)

The water absorption capacity of the trays is displayed in Fig. 4. In general, the samples presented similar behavior, with increasing WAC with the increase of the immersion time. When immersed from 1 to 30 min, the PVA 0 sample presented significantly higher ($p \le 0.05$) WAC values (50.38–305.06 %) than samples incorporating PVA (Fig. 4). These results indicate that the presence of PVA modifies the water sensitivity of the starch matrix. Cinelli et al. [5] also reported a similar trend for foams based on potato starch, corn fibers and PVA. The trays with PVA absorbed less water because this polymer replaces, at least

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partially, interactions between starch matrix and water with hydrogen bonds.

Of the samples incorporating PVA (Fig. 4), the PVA 2.5 sample had lower WAC values (36.92–199.44 %), while PVA 10.0 presented higher values (80.86–337.55 %). PVA is less hygroscopic than starch, so there is a reduction in WAC with the incorporation of PVA in starch matrices. Nevertheless, the results reported in this work showed that increasing the PVA content to higher levels (7.5 and 10 %) results in a significant WAC increase. Possibly, when the PVA content exceeds its capacity to interact with the starch matrix, an increase of the WAC may occur; therefore, the addition of PVA must be controlled.

Water Sorption Isotherms

The trays presented similar isotherm patterns (Fig. 5) that were classified as type 2 sigmoid isotherms, which are typical of hydrophilic products [37]. In general, the equilibrium moisture content of the samples increased with increasing water activity.

Trays containing PVA (PVA 2.5, PVA 5.0, PVA 7.5 and PVA 10.0) showed lower equilibrium moisture contents at all water activity levels (Fig. 5). The GAB parameters for the tray samples are shown in Table 3. PVA 2.5 and PVA 5.0 showed the lowest monolayer values, 0.0678 and 0.0654 g/g, respectively, while PVA 7.5 and PVA 10 showed the highest values, 0.0881 and 0.087 g/g, respec-

Fig. 4 Water absorption capacity of biodegradable trays based on cassava bagasse and PVA





Fig. 5 Water sorption isotherms of biodegradable trays based on cassava bagasse and PVA: (*filled square*) PVA 0; (*filled circle*) PVA 2.5; (*filled triangle*) PVA 5.0; (*asterisk*) PVA 7.5 and (*filled diamond*) PVA 10

tively (Table 3). The monolayer value indicates the maximum amount of water that can be adsorbed in a single layer per gram of dry matter; that is, the number of sorbing sites [38]. This result indicated that the incorporation of PVA at certain levels (2.5 and 5.0 %) resulted in decreased hygroscopicity of the CB trays, but higher amounts were not effective.

Furthermore, none of the samples showed dimensional changes during storage at all conditions. Starch foams tend to gain weight, thus leading to changes in their dimensions, when stored at high RH [27]; this was not observed in our work.

Mechanical Properties

The PVA 0 sample showed the lowest stress and strain at break values (Table 3), and these values significantly

increased (Tukey test, $p \le 0.05$) with PVA incorporation, except for the PVA 10 sample, which showed similar strain at break values to those of the PVA 0 sample. The stress at break values increased approximately 7–10 times with the addition of PVA, and these results are related to the higher compatibility among starch, cellulose and PVA, which can interact by hydrogen bonding [5, 36, 37, 39].

Of the samples with PVA, the PVA 10 sample showed the lowest stress and strain at break values (Table 3). According to Iriani et al. [40], PVA can act as a reinforcing agent only at intermediate concentrations because when used in high amounts, it is difficult to obtain a homogeneous matrix. These results are consistent with the WAC and isotherm data, which showed that the most positive effects of PVA were obtained when this polymer was used at lower levels (2.5 and 5.0 %).

The stress and strain at break values of our trays are higher than those reported for commercial expanded polystyrene trays used in supermarkets, which have stress and strain values of approximately 2.0 MPa and 2.0 % at break, respectively, as reported by Schmidt and Laurindo [41].

Conclusions

In this work, biodegradable trays based on CB and PVA were produced by a baking process, resulting in amorphous polymeric matrices. The produced trays had a good appearance, and the addition of PVA at all levels resulted in 100 % production yield, while the sample produced without PVA presented a 90 % yield. PVA addition decreased the density and increased the thickness of the trays. The water sorption capacity of the trays decreased with the incorporation of PVA, and the best results were achieved when 2.5 and 5.0 % PVA were employed. The stress at break values increased approximately 7–10 times

Table 3 GAB model parameters and mechanical properties of biodegradable trays based on with cassava bagasse and polyvinyl alcohol

Stress at break (MPa) Strain at break (%
9 $19.49 \pm 3.54c$ $2.60 \pm 1.87b$
9 167.82 ± 34.00 a,b 5.81 ± 1.12 a
9 164.72 ± 27.45 a,b 6.02 ± 1.05 a
9 $206.89 \pm 23.28a$ $5.97 \pm 1.01a$
9 $141.17 \pm 28.42b$ $2.50 \pm 0.30b$
-)))))

Different letters in the same column indicate significant differences ($p \le 0.05$) between means (Tukey test)

^a $M = m_0 C K a_w / (1 - K a_w) (1 - K a_w + C K a_w)$, where M is the equilibrium moisture content at a water activity (a_w), m_0 is the monolayer value (g water/g solids), and C and K are the constants

with PVA addition, and in general, the most positive effects of PVA were obtained when this polymer was used at lower levels (2.5 and 5.0 %).

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