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Properties of baked foams from oca (*Oxalis tuberosa*) starch reinforced with sugarcane bagasse and asparagus peel fiber

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Abstract

The aim of this work was evaluate the effect of the addition of sugarcane bagasse and asparagus peel fiber on the physical and mechanical properties of baked foams based on oca starch. Low concentrations of fiber reduce the density of the foams and the addition of fiber does not improve the flexural strength of the foams, but generates harder and deformable trays. High concentrations of sugarcane bagasse fiber generate more compact trays with a lower water absorption capacity than the control. Foams with asparagus peel fiber showed higher rates of thermal degradation than the control but not so extensive as to affect their applicability.

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Keywords: Oca starch; baked foam; new material; asparagus peel.

1. Introduction

Tray foam based on expanded polystyrene (EPS) are used to contain commodities such as food and pharmaceuticals, because of their low density, good thermal insulation property, high strength and low cost [1].

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However, the foams based on petroleum are no biodegradable, difficult to recycle and they can generate toxic products during their decomposition [2 – 4].

In this sense, the biodegradable polymers such as the starch has been used to produce biodegradable packing because of its low cost, renewable, , biodegradability, abundance and swelling properties [5].

Oca (*Oxalis tuberosa*) is a tuber native to the Peruvian Andes, producing 1.400 tons per year [6]. It has a high content of starch and has been used to make starch films [7], however, there is not record of use of oca starch to produce biodegradable baked foams. The thermoforming process has been used to produce baked foams by the cooking of starch-water mixtures heated in closed molds [8]. However, starch-based foam have limitations such as low process yield, poor mechanical property and high hydrophilicity [9]. An alternative for improving the mechanical properties and decrease the hydrophilicity of starch-based foams can be the addition of cellulose fibers. Vegetable fibers as reinforcements in biopolymers have been studied by several authors due to their specific properties [10]. Sugarcane bagasse fiber with cellulose content of 40-50% [11] has been used by several authors to improve the mechanical properties of foams made of starch [12 – 15]. Asparagus (*Asparagus officinalis* L.) is of great economic importance in Peru [16]. During its canning process, the peeling step generates a waste material, which could constitute about 40-50% of the fresh weight of asparagus, with a cellulose content of about 20-25% [17]. The use of asparagus peel fiber as a reinforcement in starch-based foam yet has not been reported.

The aim of this work was development and characterize baked foams based on oca starch reinforcement with sugarcane bagasse and asparagus peel fiber. Also was evaluated the effect of concentration of the fibers on the physical-chemical and mechanical properties of trays foam.

2. Materials and methods

2.1. Materials

Oca (*Oxalis tuberosa*, red variety) starch, sugarcane bagasse fiber (SB) and asparagus peel fiber (AP), was provided by Laboratory of Agro-industrial Process Engineering of the National University of Trujillo (Trujillo, Perú).

Starch contained 31.89 ± 2.20% amylose, 13.01 ± 0.27% moisture and 0.40 ± 0.05% protein. The SB fiber contained 8.05 ± 0.12% moisture, 23.69 ± 0.39 cellulose, 19.29 ± 1.34 hemicellulose, 1.50 ± 0.24 soluble lignin and 17.93 ± 1.08 insoluble lignin. The AP fiber contained 14.48 ± 0.08 moisture, 16.18 ± 0.67 cellulose, 20.33 ± 1.39 hemicellulose, 2.36± 0.09 soluble lignin and 19.35 ± 0.66 insoluble lignin (Data supplied by the provider). The fiber samples were ground in a knife mill and sieved through 50-mesh sieves (Tyler series, 300 μm).

2.2. Starch foam trays preparation by thermopressing

Table 1 shows the formulations used to prepare the oca starch-based baked foam reinforcement with sugarcane bagasse (SB) and asparagus peel fiber (AP). The proportions of starch, fibers and other ingredients were based on previous results (not published). Oca starch, sugarcane bagasse (starch/SB) and asparagus peel (starch/AP) ratios used to preparation of foams were 100/0, 95/5, 90/10, 85/15, 80/20, 70/30, and 60/40.

To prepare each formulation the proportion of starch, fiber (SB or AP), water, glycerol (used as plasticizer) and magnesium stearate (used as release agent) were mixed at 1500 rpm for 10 min with a mechanic stirrer (Imaco, China). Then, 42 – 60 g of each formulation was homogeneously layered on a Teflon mold (27 cm × 20 cm × 25 mm, thickness of 3.0 mm) in a compression molding machine (RELES, Lima, Peru) at 140 °C for 18 min and 60 bar. Finally, the trays were removed, unmolded and stored for 4 days at 25 °C and 60% relative humidity before characterization.

2.3. Baked foams characterization

The thickness was measured using a manual micrometer (Stainless Hardened, 0-150 mm).). For each formulation, the reported thickness was the average of 36 values (three measurements taken from each of the 12 samples).

The density of foam tray (g cm^{-3}) was calculated according to Shogren et al. (1998) [18]. The samples were cut on rectangular strips measuring 100 mm by 25 mm. Each sample was weighed (g), and the volume (cm^3) was calculated by multiplying the length, width and thickness together; and the density was calculated as the relationship between weight and volume. The reported density values were the averages of 12 samples per formulation.

Table 1. Compositions of the batters used to prepare the baked foams made of oca starch, sugarcane and asparagus peel fiber.

Starch/fiber ratios	Water (g)	Batter amount (g)
100/0	100	50
95/5SB	100	50
90/10SB	100	50
85/15SB	105	50
80/20SB	105	50
70/30SB	110	60
60/40SB	122.5	60
95/5AP	100	42
90/10AP	100	45
85/15AP	100	45
80/20AP	102.5	47.5
70/30AP	105	60
60/40AP	112.5	60

The water absorption capacity was performed according to ABNT NBR NM ISO 535 (1999) [19]. Samples (2.5 cm x 5 cm) were weighted and soaked in distilled water for 30 s. The quantity of absorbed water was calculated as the weight difference and expressed as mass of absorbed water per mass of original sample. Reported values were the mean of five determinations for each formulation.

SEM analyses were performed with a Tecsan VEGA 3 LM with a gold coating system SPI 11430-AB (TESCAN USA, EE.UU). The foam pieces were mounted for cross-section visualization on bronze stubs using double-sided tape. Images were taken using an acceleration voltage of 20 kV in all cases.

The thermal decomposition of the starch foam was measured under a nitrogen atmosphere (100 mL min^{-1}) using a SETSYS Evolution TGA-DTA/DSC (SETARAM Instrumentation, France) equipment in the temperature range of 25 – 600 °C at a heating rate of 10 °C min^{-1} [3]. Sample masses: ∼6 mg. Sample pan type: alumina/referent pan: empty alumina.

A texture analyzer model TA.HDPlus (Stable Micro System, Surrey, UK) with a 10N load cell was used to determine the mechanical properties of the foam samples. Tensile tests were performed with strips measuring 100 mm by 25 mm, with an initial grip separation of 80 mm and with a crosshead speed of 2 mm/s. Stress–strain curves were recorded during extension, and stress and strain at break were determined [14]. Each formulation was assayed 12 times, and the reported values are the averages of these determinations. Mechanical properties of commercial expanded polystyrene (EPS) trays (2.53 mm of thickness and 0.041 g/cm^3 of density) were performed at the same conditions described for foam trays.

2.4. Statistical analysis

Analysis of variance (ANOVA), Tukey's test to compared the formulations (ratios starch/fiber) and Tukey's test to compared both types of fiber in baked foams ($p \leq 0.05$) were performed with the Statistica software version 7.0 (Statsoft®, USA).

3. Results and discussion

3.1 Physical properties

The thickness and density of the oca starch baked foam as a function of fiber concentration and type of fiber are shown in Table 2. All trays had an average thickness between 2.485 and 2.613 mm and an average density between 0.144 and 0.291 g cm^{-3}.

Table 2. Thickness and density of composite baked foams based on oca starch, sugarcane bagasse fiber (SB) or asparagus peel (AP) fiber.

Starch/fiber ratio	Thickness (mm)		Density (g cm^{-3})	
	SB	AP	SB	AP
100/0	2.570 ± 0.004^{b}	2.570 ± 0.004^{b}	0.171 ± 0.002^{c}	0.171 ± 0.002^{d}
95/5	2.570 ± 0.025^{abB}	2.613 ± 0.013^{aA}	0.157 ± 0.014^{cA}	0.144 ± 0.016^{eA}
90/10	2.577 ± 0.054^{abA}	2.575 ± 0.093^{bA}	0.158 ± 0.015^{cA}	0.161 ± 0.022^{deA}
85/15	2.579 ± 0.027^{bA}	2.572 ± 0.028^{bA}	0.175 ± 0.022^{cB}	0.203 ± 0.014^{cA}
80/20	2.587 ± 0.013^{abA}	2.582 ± 0.044^{bA}	0.221 ± 0.010^{bA}	0.200 ± 0.005^{cB}
70/30	2.602 ± 0.016^{aA}	2.528 ± 0.076^{cB}	0.234 ± 0.019^{baB}	0.267 ± 0.011^{bA}
60/40	2.597 ± 0.019^{abA}	2.485 ± 0.099^{dB}	0.272 ± 0.022^{aB}	0.291 ± 0.005^{aA}

Data are the means of replicate determinations ± standard deviation. Different small letters in the same column indicate significant differences ($p \leq 0.05$) between means (Tukey's test), and different capital letters in the same line indicate significant differences ($p \leq 0.05$) between means (Tukey's test).

At lower fiber concentrations (<20%), the thickness of starch/fiber foam tray did not vary significantly when the baked foams was reinforced with SB fiber. A concentration of 5% AP fiber causes an increases in the thickness of baked foam. When the fiber concentration was increased to 30% and 40% the thickness of starch/SB tray increased whereas for starch/AP decreased. This tendency indicated that in presence of high SB concentration the starch tray foam is more porous than in presence of AP fiber. Nevertheless, the density of both starch/fiber foam increased significantly when high SB or AP fibers concentration (15 - 40%) were added in the mixture. In addition, the starch/AP trays are denser than the starch/SB trays, which may be the result of a larger thickness, increasing the foaming capacity of the baked foam [5]. However, at low fiber concentration the density of starch/fiber foam tray decreased when compared with the control (100/0). These results are similar to those found for cassava starch trays and malt bagasse fiber [5] and for cassava starch baked foams reinforced with sugarcane bagasse fiber and montmorillonite [14]. The authors found a reduction of the density of their foams with the incorporation of fiber up to 20%.

The density values recorded in the present study were high compared to expanded polystyrene (EPS) (2.53 mm of thickness and 0.041 g cm^{-3} of density). However, the values found in this study (0.144 - 0.291 g cm-3) are below that reported by other authors for baked foams made from wheat starch, cassava, potato and maize (0.07 - 0.41 g cm^{-3}) [10, 14, 20, 21].

The micrograph of cross section of oca starch foams reinforced with SB fiber (95/5), AP fiber (95/5) and without addition of fiber (100/0) show a heterogeneous porous structure being larger when the fiber was added in the starch matrix (Figure 1). The fiber distribution through the cellulose matrix was different for both SB and AP fiber. Cells larger and with thinner layer were observed for trays with SB fiber than AP fiber, which may be due to less interference with starch expansion during thermoforming of the tray [22]. These air cells are responsible for the porosity of the starch trays. This can explain the lower density of starch/SB foam tray when compared with starch/AP fiber, as was shown in Table 3.

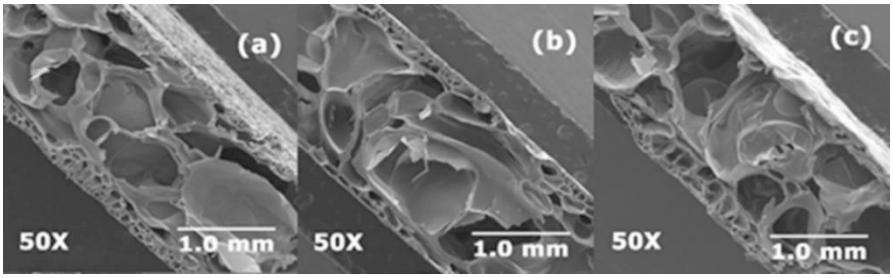

Fig. 1. Images of baked foam: (a) Starch/fiber 100/0, (b) Starch/SB 95/5, and (c) Starch/PA 95/5.

Table 3. Water absorption capacity (WAC) of composite foam based on native starch and fiber.

Starch/fiber ratio	WAC (g water/g m.s.)	
	SB	AP
100/0	78.88 ± 9.23^{a}	78.88 ± 4.23^{a}
95/5	94.69 ± 9.71^{aA}	79.21 ± 6.23^{aB}
90/10	98.37 ± 10.80^{aA}	88.14 ± 1.89^{aB}
85/15	95.88 ± 9.46^{aA}	90.81 ± 3.98^{aB}
80/20	77.65 ± 4.20^{aB}	93.81 ± 3.58^{aA}
70/30	74.48 ± 8.03^{abB}	85.84 ± 9.25^{aA}
60/40	64.87 ± 7.05^{bB}	84.05 ± 8.38^{aA}

Data are the means of replicate determinations ± standard deviation. Different small letters in the same column indicate significant differences ($p \leq 0.05$) between means (Tukey's test), and different capital letters in the same line indicate significant differences ($p \leq 0.05$) between means (Tukey's test).

The WAC for each formulation of baked foam is shown in Table 2 as a function of fiber concentration and fiber type (SB and AP). The addition of low concentrations (5%, 10% and 15%) of SB fiber increased the water absorption capacity (WAC) of starch-based foam tray, meanwhile the addition of high SB fiber concentration (>20%) decreased the WAC of trays even when compared to control (100/0). However, for starch/AP fiber foam tray, the addition of high concentration of fiber (30 and 40%) yielded trays with lower WAC than at low AP fiber concentrations (5 to 20%), but these trays had greater WAC than both control (100/0) and starch/SB foam tray (30 and 40% SB fiber) . On the other hand, in SB fiber concentrations up to 15%, the starch/SB foam tray showed greater WAC than starch/AP foam tray. The WAC of starch-based foam stray is dependent on the porosity of the trays and on the capacity of absorbing water of starch and the fibers [23]. The addition of low SB fiber concentrations probably yielded trays more porous with larger diameters of cellules that facilitated the entry of water, whereas high SB concentrations decreased of starch mass in the mixture decreasing the foaming of starch yielding a more compact structure (Figure 1) with greater density and least WAC.

The addition of AP fiber yielded starch-based trays with a more compact structure and small diameter pores (Figure 1) the WAC of these trays were lesser than starch/SB foam tray at low fiber concentrations (<15%). When the proportion of fiber (crystalline phase) increases in a starch-based material, the cellulose present in the fiber causes a reduction in WAC [24]. This explains why trays with SB fiber (23.69 % cellulose) are able to reduce WAC when compared to control and have a lower WAC than starch/AP trays (16.18 % cellulose) at high fiber concentrations.

The water absorption capacity (WAC) of the oca starch foams ranged from 64.87 to 98.37 (g water 100g d.b.$^{-1}$) at 30 seconds of immersion in Water. The water absorption values of these materials were higher than those reported for trays obtained in other studies [9, 10, 25]. However, a significant reduction at high concentrations of SB fiber extends the possibility of using these materials.

3.2 Mechanical properties

Table 4 shows the values of flexural strength and strain at break of trays with sugarcane bagasse fiber (starch/SB) and asparagus peel fiber (starch/AP). The addition of fiber (SB or AP) decreases the flexural strength of the oca trays (Tukey's test, $p \leq 0.05$).

Trays with addition of fibers showed a less compact structure and with distribution no homogenous of pores when compared to the control (100/0), causing a lower resistance of the material. Since in the formulations when the starch content decrease and the fiber agglomerates interfering with the starch-starch interactions, generating trays with less mechanical resistance [25].

When comparing fiber type (SB or AP), the starch/SB trays in concentrations 40% had higher values of flexural strength than starch/AP trays at the same fiber concentration. This behavior may be due to the fact that at high fiber concentrations, the interactions between the starch are lower, so under stress, the force is transmitted to the fiber and its strength depends on the characteristics of the reinforcing material [26]. Thus, SB fiber with a higher cellulose content (23.69 %) than PA fiber (16.18 %) produces trays that are more resistant to stress.

Table 4. Mechanical properties (measured by tensile tests) of the trays based on native starch and fiber.

Starch/fiber ratio	Flexural strength (MPa)		Strain at break (%)	
	SB	PA	SB	PA
100/0	0.65 ± 0.08^{b}	0.65 ± 0.08^{a}	1.10 ± 0.02^{a}	1.10 ± 0.02^{b}
95/5	0.61 ± 0.04^{bA}	0.62 ± 0.04^{aA}	0.90 ± 0.02^{bA}	0.90 ± 0.01^{bA}
90/10	0.52 ± 0.08^{cA}	0.51 ± 0.04^{bA}	0.60 ± 0.01^{bA}	0.80 ± 0.01^{bcB}
85/15	0.53 ± 0.05^{cA}	0.51 ± 0.04^{bA}	0.60 ± 0.01^{bA}	0.70 ± 0.01^{bcB}
80/20	0.57 ± 0.07^{bcA}	0.53 ± 0.02^{bB}	0.60 ± 0.02^{bA}	0.80 ± 0.02^{bcB}
70/30	0.58 ± 0.09^{bcA}	0.55 ± 0.04^{abB}	0.60 ± 0.01^{bA}	1.60 ± 0.03^{aB}
60/40	0.79 ± 0.04^{aA}	0.55 ± 0.04^{abB}	0.60 ± 0.01^{bA}	1.50 ± 0.01^{abB}

Data are the means of replicate determinations ± standard deviation. Different small letters in the same column indicate significant differences ($p \leq 0.05$) between means (Tukey's test), and different capital letters in the same line indicate significant differences ($p \leq 0.05$) between means (Tukey's test).

The strain at break (elongation, %) significantly decreased with the addition of SB fiber. These results differ from those found in cassava foams with sugarcane bagasse fiber, where an addition up to 20% results in a 40% increase in the elongation of the trays [14]. The AP fiber in a starch/fiber ratio of 70/30 and 60/40 significantly increases the elongation of the trays (Tukey's test, $p \leq 0.05$). This may be because baked foams with AP fiber in high concentrations (20% - 40%) have greater water absorption capacity (Table 2); forming a structure that retains water, being that the water acts as plasticizer [27], an improvement in the elongation is justified. A similar behavior to the 70/30 and 60/40 starch/AP trays was found in other studies, where the presence of fiber reduces the cohesive forces in the polymer matrix, decreasing the brittle characteristics and increasing its resistance to deformation [10, 28].

The same test used to evaluate the trays was used to evaluate the mechanical properties of expanded polystyrene (EPS) (0.83 ± 0.11 MPa, 2.82+ 0.38%). The results of the present study suggest that baked oca starch foams may be a viable alternative to EPS trays.

3.3 Thermal properties of the starch foams

The behavior during the thermal degradation of baked foams of oca starch without addition of fiber (100/0), bagasse fiber (95/5SB) and asparagus peel fiber (95/5AP) are detailed through of the TG and DTG curves (Figure 2). The decomposition of biopolymers occurs in three stages [29]. The first stage up to around 200 ° C was associated with the loss of free and bound water absorbed by the foams [30] (Figure 2A), similar to that reported in trays of cassava starch and citric acid-modified cassava starch [3]. The decomposition of the glycerol and starch rich

phase occurs in the second stage (185 °C - 325 °C), with the oxidant reaction of the main organic compounds (decomposition of lignin, hemicellulose and mainly cellulose) [27], with a mass loss of about 65%. Trays with PA fiber showed greater resistance to thermal degradation than trays with SB fiber and trays without fiber (100/0), probably to a higher lignin content in the fiber (Figure 2A). In the third stage, it shows the oxidation of partially decomposed starch, which generates solid wastes such as ash and inorganic material (about 23% of the initial mass) [15].

The loss of mass between 200 and 400 ° C is caused by the degradation of cellulose due to dehydration, depolymerization or decomposition of glycosyl units, followed by the formation of a carbonized residue [27]. As shown in Figure 2, a larger mass loss occurs for the tray with SB fiber (95/5SB), due to a higher content of cellulose in its composition. While the thermal stability of the tray with AP fiber (95/5PA) can lead to a higher lignin content (the thermally stable composite that makes up the fiber) [27].

As shown in Figure 2B, two basic peaks were observed in the DTG curves of the oca starch trays with fiber. By incorporating fiber into the starch matrix, the overall thermal stability of the trays increased. (1) a higher rate of evaporation of water occurs in the trays with fiber and (2) a slight increase in the degradation rate of fiber trays (95/5SB and 95/5AP) when compared to (100/0) is shown in peak 2. This increase in the rate of degradation of fiber-reinforced trays refers to weaker interactions in the mixture [3] and is in accordance with the physical and mechanical properties and trays.

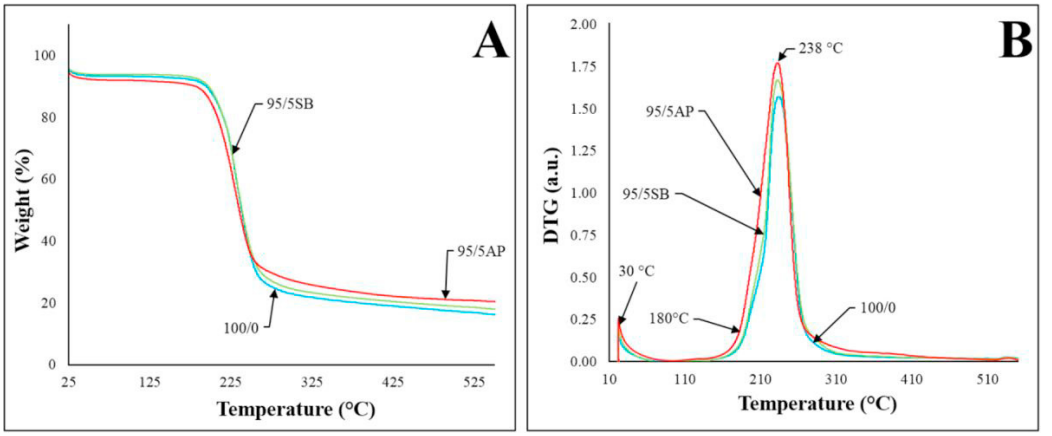

Fig. 2. Thermogravimetric curves of foam trays 100/0, 95/5PA and 95/5SB: (A) TG curves and (B) DTG curves.

4. Conclusion

The starch/SB and starch/AP baked foams produced in this study had good appearance and adequate expansion. The trays with starch/fiber ratios of 95/5SB and 95/5AP and the control tray had greater physical properties. All the trays obtained here had higher density and thickness than polystyrene trays. The addition of SB fiber in concentrations above 20% decreases the water absorption capacity of the baked foams. The addition of the SB or AP fiber did not improve the mechanical properties of the trays, suggesting a poor distribution of the fiber in the polymer matrix. The addition of fibers to the polymer matrix did not improve the thermal stability of the foams, which in concordance with mechanical properties. These trays can be used to pack dry foods with short shelf life, and they can be a viable and sustainable alternative to replace traditional packaging, thereby reducing the consumption of petroleum.

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