



# Ultrasound processing to enhance drying of cashew apple bagasse puree: Influence on antioxidant properties and in vitro bioaccessibility of bioactive compounds



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## ABSTRACT

The present study has evaluated the effects of power ultrasound pre-treatment on air-drying and bioactive compounds of cashew apple bagasse. The sonication induced the disruption of cashew bagasse parenchyma, which resulted in lower resistance to water diffusion, less hysteresis, and increased rehydration rate. The processing did not affect the lignocellulose fibers or the sclerenchyma cells. For sonicated samples, water activity reached values below 0.4, after 2 h of drying, which is appropriate to prevent bacterial and fungi growth. The sorption isotherms of cashew apple bagasse presented sigmoid-shape for all samples and followed the type II according to BET classification. Sonicated cashew apple bagasse showed higher antioxidant activity, higher total phenolic compounds (TPC) and higher vitamin C content when compared to the non-sonicated sample. The increase in TPC and vitamin C contributed to the product antioxidant activity. A slight reduction on Vitamin C bioaccessibility was observed, but the TPC bioaccessibility has increased. Sonication reduced the quality loss of conventional drying treatments improving the quality of the dried product.

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## 1. Introduction

One of the main purposes of modern food technology is to maximize the retention of nutrients during processing and storage. Drying is a technology that promotes significant modifications in physical and chemical properties of food affecting the bioactive compounds and antioxidant activity. Pre-treatments can improve the drying process leading to high-quality products.

Ultrasound can improve mass and heat transfer phenomena [1]. In solids immersed in a fluid, ultrasound can accelerate the internal transport enhancing the mass transfer through the solid surface and the surrounding fluid [2]. The ultrasound effect on fruit tissue depends on the tissue structure and composition. Sonication might be beneficial to improve air-drying efficiency, with a consequent reduction in process costs [3].

Cashew apple is an important crop in Brazil, India, Indo-china and in some African countries, representing 1.1% of the world production of exotic fruits [4]. The cashew apple bagasse is the industrial waste of cashew apple peduncle processing. The residue has a dark yellow color, a fibrous aspect, and a typical astringent taste due to the presence of tannins. Despite edible and rich in nutrients, cashew apple bagasse is highly perishable and treated as waste by the food industry. Cashew apple bagasse can be applied as a raw material in several industrial processes such as second generation ethanol production and as a substrate for solid state fermentation to produce enzymes and xylitol, among other applications [5–7]. Fruit bagasse can be also used as food ingredients due to their bioactive compounds and high dietary fiber [8–10]. Some authors proposed the use of fruit bagasse as a partial substitute for wheat flour in bakery foods [11,12]. Cashew apple bagasse is rich in fibers, vitamins, and antioxidants, and thus this by-product is regarded as a possible food ingredient.

As drying is a way to preserve this by-product for further applications, this study has evaluated the effect of ultrasound

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processing on convective drying of ultrasound treated cashew apple bagasse puree. The study also evaluated the processing effect on cashew apple bagasse antioxidant compounds (phenolic compounds and Vitamin C).

## 2. Materials and methods

### 2.1. Sample preparation

The raw material was red cashew apples (*Anacardium occidentale L.*) harvested at commercial maturity stage in Ceará State, Brazil. After the fruit sanitization and nut removal, the cashew apple (peduncle) were reserved. The peduncles were not light protected to simulate the conditions that the fruit may be exposed during the postharvest period. The juice was extracted using an expeller press. The bagasse was packaged in polyethylene bags, vacuum sealed and stored at  $-18\text{ }^{\circ}\text{C}$  until use. Before the experiments, the cashew apple bagasse was thawed at  $4\text{ }^{\circ}\text{C}$ .

### 2.2. Sonication

The experiments with ultrasound application were carried out in 600 mL Becker flasks with a final sample volume of 200 mL (bagasse + water). Samples were processed in a 500 W ultrasound equipment (Unique<sup>®</sup> DES500, São Paulo, Brazil) with a 1.3 cm diameter probe tip without mechanical agitation or temperature control. The ultrasound frequency was 20 kHz, and the initial temperature was  $20\text{ }^{\circ}\text{C}$ .

The probe was submerged to a depth of 15 mm in the sample. The treatment was carried out in triplicate. The intensity of ultrasound power dissipated from the probe tip was calculated by Eq. (1) [13].

$$I = \frac{P}{\pi r^2} \quad (1)$$

where  $r$  is the radius of the titanium tip (cm), and  $P$  is the input power level (W). The input power was controlled through the amplitude setting, and the power level was adjusted to 20%, 60% and 100% of total input power (500 W). The calculated intensities were 75, 226 and  $373\text{ W/cm}^2$ , respectively.

### 2.3. Experimental design and data analysis

The effects of ultrasound processing were studied through a  $2^3$  face-centered central composite experimental design (CCD) with three central points to evaluate the process repeatability. US power intensity, bagasse:water ratio (g/g) and the processing time changed from 75 to  $373\text{ W/cm}^2$ , 1:2 to 1:4 and 2 to 10 min, respectively (Table 1). A control assay was carried out at  $51\text{ }^{\circ}\text{C}$ , which was the highest temperature recorded after the cashew apple bagasse sonication. Non-sonicated bagasse immersed in water with the same bagasse:water ratio was taken as the control samples.

### 2.4. Air-drying

After ultrasound processing, the samples were air-dried in a forced circulating air-drying oven (Marconi, model MA-035, Brazil). The drying temperature was  $60\text{ }^{\circ}\text{C}$ . The air moisture was 18% (determined by psychometry). The air velocity was 1 m/s, measured with an anemometer. The sonicated cashew apple bagasse was dried in a single-layer (2 mm) at the above conditions. The bagasse moisture (water content) during the air-drying period was measured weighting the samples every hour until constant weight.

The effective water diffusivity in cashew apple bagasse during air-drying was calculated using Fick's law. The equation used for the drying falling-rate period was based on the simplification of Fick's second law taking into account a long processing period [14]. The effective water diffusivity was adjusted using Eq. (2) and the Levenberg–Marquardt parameter estimation procedure.

$$\frac{dH}{dt} = -\frac{2\pi}{\delta^2} \cdot D \cdot (X - X_{eq}) \quad (2)$$

where  $D$  is the effective water diffusivity ( $\text{m}^2/\text{s}$ );  $X$  is the moisture content (g of water/g of dry matter);  $X_{eq}$  the equilibrium moisture content (g of water/g of dry matter),  $t$  is time (s), and  $\delta$  the bed height (or thickness) of the sample (m).

### 2.5. Water activity

Water activity was determined at  $25\text{ }^{\circ}\text{C}$  with a water activity meter (AquaLab, Decagon CX-2, Pullman, Washington, USA).

**Table 1**  
Experimental design and responses of the influence of ultrasonic processing on water activity and water diffusivity of cashew apple bagasse.

Assay	Power intensity ( $\text{W/cm}^2$ )	Time (min)	Bagasse:water ratio	Water diffusivity ( $10^{-10}\text{ m}^2/\text{s}$ )	
1	75	2	1:2	$1.06 \pm 0.00$	
2	75	10	1:2	$1.07 \pm 0.01$	
3	373	2	1:2	$1.18 \pm 0.00$	
4	373	10	1:2	$1.04 \pm 0.04$	
5	75	2	1:4	$2.14 \pm 0.01$	
6	75	10	1:4	$2.16 \pm 0.02$	
7	373	2	1:4	$1.50 \pm 0.01$	
8	373	10	1:4	$1.13 \pm 0.03$	
9	226	6	1:2	$1.04 \pm 0.03$	
10	226	6	1:4	$1.59 \pm 0.00$	
11	75	6	1:2	$1.18 \pm 0.00$	
12	373	6	1:3	$1.11 \pm 0.02$	
13	226	2	1:3	$1.20 \pm 0.04$	
14	226	10	1:3	$1.18 \pm 0.01$	
15 (C)	226	6	1:3	$1.66 \pm 0.01$	
16 (C)	226	6	1:3	$1.11 \pm 0.02$	
17 (C)	226	6	1:3	$1.24 \pm 0.00$	
Control					
				1:2	$1.06 \pm 0.00$
				1:3	$1.18 \pm 0.01$
				1:4	$1.66 \pm 0.01$

## 2.6. Determination of sorption isotherms

Vapor sorption isotherms of dried cashew apple bagasse were generated using a Vapor Sorption Analyzer-VSA (Aqualab, Decagon Devices, Pullman, USA) at 25 °C. The experimental data was fitted to the mathematical models of Brunauer, Emmet and Teller (BET); Guggenheim–Anderson–De Boer (GAB) and Double Log Polynomial (DLP) given by the following equations:

GAB

$$X_{eq} = \frac{X_m C k a_w}{(1 - k a_w)(1 - k a_w) + C k a_w} \quad (3)$$

BET

$$X_{eq} = \frac{X_m C a_w}{((1 - a_w)(1 - (C \ln(1 - a_w))))} \quad (4)$$

DLP

$$X_{eq} = b_3 \cdot chi^3 + b_2 \cdot chi^2 + b_1 \cdot chi + b_0 \quad (5)$$

where:

Xeq = equilibrium moisture content on dry basis (kg water/kg dry solid);

Xm = moisture content in molecular monolayer on dry basis (kg water/kg dry solid);

Aw = water activity (dimensionless);

C, k, a, b, b0, b1, b2 e b3 = empirical coefficients;

$$Chi = \ln(\ln(a_w)).$$

The parameters used to evaluate the fitted models were the coefficient of determination ( $R^2$ ) and the estimated error calculated by the Software VSA Downloader 1.0.967.

## 2.7. Microscopic analysis

### 2.7.1. Light microscopy

After sonication, small bagasse fragments were fixed for 4 h in an aqueous solution composed of glutaraldehyde (2.5%) and formaldehyde (4.0%) freshly prepared in cacodylate buffer (0.05 M, pH 7.2). Ethanol was used to dehydrate the material. After that, the sample was embedded in Histo-resin (Leica Histo-resin, Germany). The tissue blocks were cut using a Leica microtome (RM2255-Leica, Germany). Thin sections (approximately 3 μm) were stained with 1% toluidine blue pH 4.0. Photomicrographs of the cell structure were taken using an Eclipse 80i (Nikon, Japan) light microscope with digital image capture system.

### 2.7.2. Scanning electron microscopy

The samples fixed in glutaraldehyde and formaldehyde buffer, as previously described, were rinsed in cacodylate buffer and post-fixed for one hour at room temperature (20 °C) with 1.0% osmium tetroxide in 0.05 M cacodylate buffer (pH 7.2) and dehydrated with acetone. Acetone was partially removed with an absorbent paper, and the samples were fixed on stubs with conductive carbon tape. Afterward, the samples were air dried at room temperature (20 °C), sputtered coated with 10 nm gold (Quorum Q150TES) and observed with a scanning electron microscope (Inspect F50-FEI/Quanta 450 FEG-FEI).

## 2.8. Rehydration ability

Dried cashew apple bagasse samples were rehydrated by immersion in distilled water (solid-to-liquid ratio 1:50) at 20 °C

for 2 h, as described by Gamboa-Santos [15]. Water in excess was removed before the samples weighting. The rehydration ratio was calculated according to the following equation:

$$\text{Rehydration ratio} = \frac{m_r}{m_d} \quad (6)$$

where  $m_r$  is the mass of the rehydrated sample (g) and  $m_d$  is the mass of the dehydrated sample (g).

## 2.9. Color

The color of the cashew apple bagasse during drying was determined using a Minolta CR300 colorimeter (Tokyo, Japan). The colorimeter was calibrated using the illuminant D65, and the measurements were taken through an 8 mm port/viewing area. The following color parameters were determined: lightness ( $L^*$ ), C (chroma) and hue angle ( $h^\circ$ ). Color measurements were taken in quintuplicate.

## 2.10. Enzymatic assay

The enzyme extraction for peroxidase (POD, EC 1.11.1.7) and polyphenol oxidase (PPO, EC 1.14.18.1) were done mixing 10 g of the sample with 10 mL of potassium phosphate buffer (0.05 M pH 7.0) containing 1% (w/v) of polyvinylpyrrolidone (PVP). The mixture was centrifuged in a Sigma® 6K15 centrifuge (10,733g for 30 min at 4 °C). The supernatant was the enzyme source.

POD activity was monitored at 470 nm in a spectrophotometer (Spectrum® SP2000UV). The enzyme activity was measured as follows: 2.75 mL of a phosphate (sodium)-citrate (citric acid) buffer (0.1 M, pH 5.0) containing 1% (v/v) of guaiacol and 0.25 mL of hydrogen peroxide 3% (v/v) were added to 1.5 mL of enzyme extract. The assay mixture was incubated at 30 °C for 5 min. The reaction was interrupted by the addition of 1 mL of sodium bisulfite 30% (w/v). One unit of enzyme activity (1 UEA) was defined as the amount of enzyme that causes a change of 0.001 in the absorbance per minute [16–18].

The same enzyme extract was used to determine the PPO activity. The reaction mixture contained 0.3 mL of enzyme extract and 1.85 mL of a potassium phosphate buffer solution (0.1 M pH 6.0) containing catechol (0.1 M) and KCl (0.1 M). The reaction mixture was incubated at 30 °C for 30 min. The addition of 0.8 mL of perchloric acid 2 N stopped the reaction. One unit of enzyme activity (1 UEA) was defined as the amount of enzyme that causes a change of 0.001 in the absorbance (395 nm) per minute [16,18].

For ascorbate peroxidase (APX, EC 1.11.1.1) two grams of sonicated cashew apple bagasse were homogenized in 15 mL of 0.1 M potassium phosphate buffer (pH 7.0) containing EDTA 0.1 mM for 1 min, followed by centrifugation at 10,733g for 40 min at 4 °C. The supernatant fraction was used for the enzyme activity assay. APX activity was assayed according to the method of Nakano and Asada [19]. Enzyme activity was measured using the molar extinction coefficient for ascorbate (2.8 mM cm<sup>-1</sup>) and the results expressed in μmol H<sub>2</sub>O<sub>2</sub> mg<sup>-1</sup> P min<sup>-1</sup>, taking into account that 1 μmol of ascorbate is required for a reduction of 1 μmol H<sub>2</sub>O<sub>2</sub>.

## 2.11. Quantification of bioactive compounds

The main bioactive compounds presented in cashew apple bagasse are ascorbic acid (Vitamin C) and phenolics. Extracts for ascorbic acid analysis were obtained after the sample centrifugation at 10,733g for 10 min in a Sigma 6K-15 centrifuge (Sigma Centrifuges, Germany) to remove the insoluble matter.

The extracts for total phenolic compounds (TPC) and antioxidant activity were prepared according to the procedure developed by Larrauri et al. [20]. Samples were weighed (2 g) in centrifuge

tubes and extracted sequentially with methanol and acetone aqueous solution. An aliquot of 10 mL of methanol/water (50:50, v/v) was added to the sample at room temperature (20 °C). The mixture was kept statically for 1 h at room temperature, and the extraction was allowed for 1 h. The samples were centrifuged at 10733g (15 min/4 °C), and the supernatant was recovered. Then, 10 mL of acetone/water (70:30, v/v) was added to the solid residue at room temperature, extracted for 60 min and centrifuged. Methanol and acetone extracts were combined into a 25 mL volumetric flask, and the volume was completed with distilled water.

Ascorbic acid content was analyzed by high-performance liquid chromatography in an Agilent 1260 Infinity system equipped with four high-pressure pumps model Agilent G1311B, UV-VIS detector ProStar model 345, and a column oven (Agilent G1316A). Separations were done using a Biorad HPX 87 H (300 × 7.8 mm) column at 50 °C. The mobile phase was H<sub>2</sub>SO<sub>4</sub> 0.01 N at 0.6 mL/min. All samples were analyzed in triplicate. The software Agilent OpenLAB was used to acquire and handle the data.

Total phenolic compounds were determined using the Folin-Ciocalteu methodology [21]. The reaction mixture contained 250 µL of the phenolic extract, 500 µL of Folin-Ciocalteu reagent (Sigma–Aldrich, Germany), 500 µL of sodium carbonate and 500 µL of distilled water. The mixture was left in the dark for 30 min at 25 °C. The absorbance of the sample was measured at 700 nm. Gallic acid (HPLC grade, Sigma–Aldrich) was used as standard. Results were expressed as mg/100 of dry matter.

#### 2.12. Total antioxidant activity determinations

For ABTS<sup>+</sup> assay, the stock solutions were: 7 mM ABTS<sup>+</sup> (solution A) (Sigma®) and 140 mM potassium persulfate solution (solution B) (Sigma®). The working solution was prepared by mixing 5000 µL of the solution A and 88 µL of solution B and allowing them to react for 16 h at room temperature (≈20 °C) in the dark. The solution was diluted by mixing the ABTS<sup>+</sup> solution with methanol to obtain an absorbance of 0.700 ± 0.02 at 734 nm. The extracts (30 µL) were allowed to react with 3000 µL of the ABTS<sup>+</sup> solution for 6 min in a dark condition [22]. The level of radical scavenging was calculated according to the following equation:

$$\text{Scavenging rate(\%)} = \left(1 - \frac{A_i}{A_s}\right) \times 100 \quad (7)$$

where  $A_s$  is the absorbance of pure ABTS<sup>+</sup>,  $A_i$  is the absorbance of ABTS<sup>+</sup> in the presence of the sample.

#### 2.13. In vitro digestion

The evaluation of the phenolic compounds and vitamin C bioavailability was done according to the method described by Chen et al. [23]. The analysis consisted of two sequential phases: gastric and enteric digestion. In the gastric phase, the pH of samples (10 g) was adjusted to 2.0 with HCl 6 M, after that 10 mL of a pepsin solution 300 U/mL was added. The mixture was incubated at 37 °C and 100 rpm during 2 h. In the enteric phase, the pH of samples was increased to 6.0 using an alkaline solution (NaOH 1 M). Bile and pancreatin were added to reach a concentration of 10 g/L and of 1 g/L, respectively. The samples were then incubated at 37 °C for 2 h under agitation (enteric phase 1). After that, the pH was increased to 6.7–7.5 using the same alkaline solution (NaOH 1 M) and more bile (10 g/L) and pancreatin (1 g/L) were added to the sample. The samples were incubated at 37 °C for more 2 h under agitation (enteric phase 2). The complete assay (gastric phase + enteric phase 1 + enteric phase 2) lasted 6 h. Immediately after the enteric phase, 2 mL of each sample was extracted and

analyzed for TPC and vitamin C concentrations as described above. All reagents were purchased from Sigma–Aldrich®.

#### 2.14. Statistical analysis

Except for color determination, which was taken in quintuplicate, all other assays were carried out in triplicate. Results were expressed as mean ± SD. *F*-test and ANOVA analysis were used as significant criteria for the fitted models. Tukey's test was used to determine the significant differences among means ( $p < 0.05$ ). Pearson's correlation coefficients were calculated at 5% of probability using the Student's test for all variables. Statistical analysis of the experimental data was carried out using the software Statistica 7.0 (StatSoft). All results were expressed in dry basis.

### 3. Results and discussion

#### 3.1. Sonication and drying

The cashew apple bagasse was sonicated according to the experimental planning presented in Table 1. The high potency ultrasound fragmented the bagasse resulting in a puree, which was transferred to Petri dishes and dried in film layer.

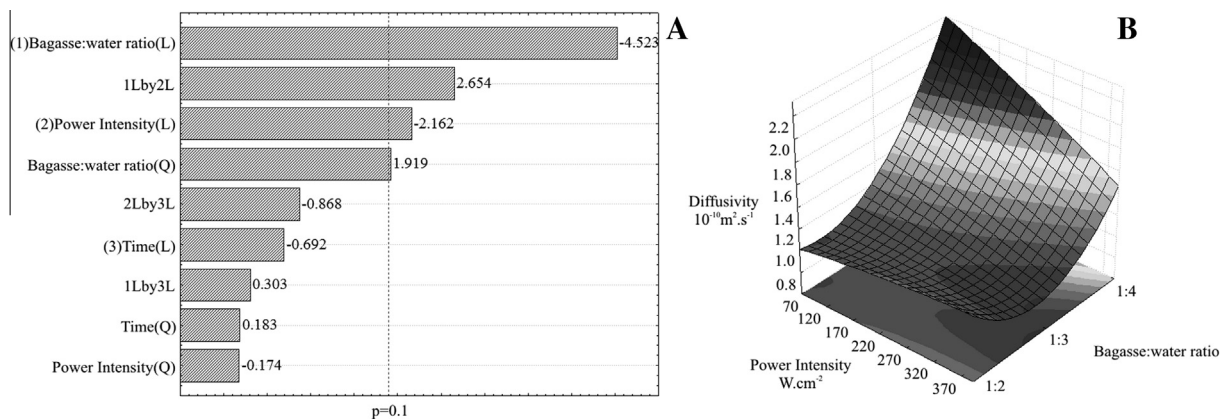
The drying curves followed the expected pattern observed in other fruits and vegetables, and the Fick's law, (Eq. (2)) was used to calculate the effective diffusivity. The application of ultrasound in different levels affected the effective water diffusivity of cashew apple bagasse puree during the air-drying process (Table 1). The maximum values observed were:  $2.16 \times 10^{-10} \text{ m}^2/\text{s}$  in assay 6 (75 W/cm<sup>2</sup>; 10 min and 1:4 bagasse:water ratio) and  $2.14 \times 10^{-10} \text{ m}^2/\text{s}$  in assay 5 (75 W/cm<sup>2</sup>; 2 min and 1:4 bagasse:water ratio). The increase of the mass transfer due to ultrasound processing is attributed to the reduction of the boundary layer thickness produced by pressure variations, oscillating velocities and microstreaming [24]. Fuente-Blanco et al. [25] also reported that cavitation produced by ultrasound is beneficial for the removal of moisture strongly attached.

Fig. 1A depicts the estimated effects of the independent variables on water diffusivity. The Pareto chart reveals that a linear increase of bagasse:water ratio and power intensity decreases the water diffusivity. However, the interaction effect between the bagasse:water ratio and power intensity were significant ( $p < 0.1$ ) and positive. Thus, a simultaneous increase of US power intensity and bagasse:water ratio favored the increase in water diffusivity.

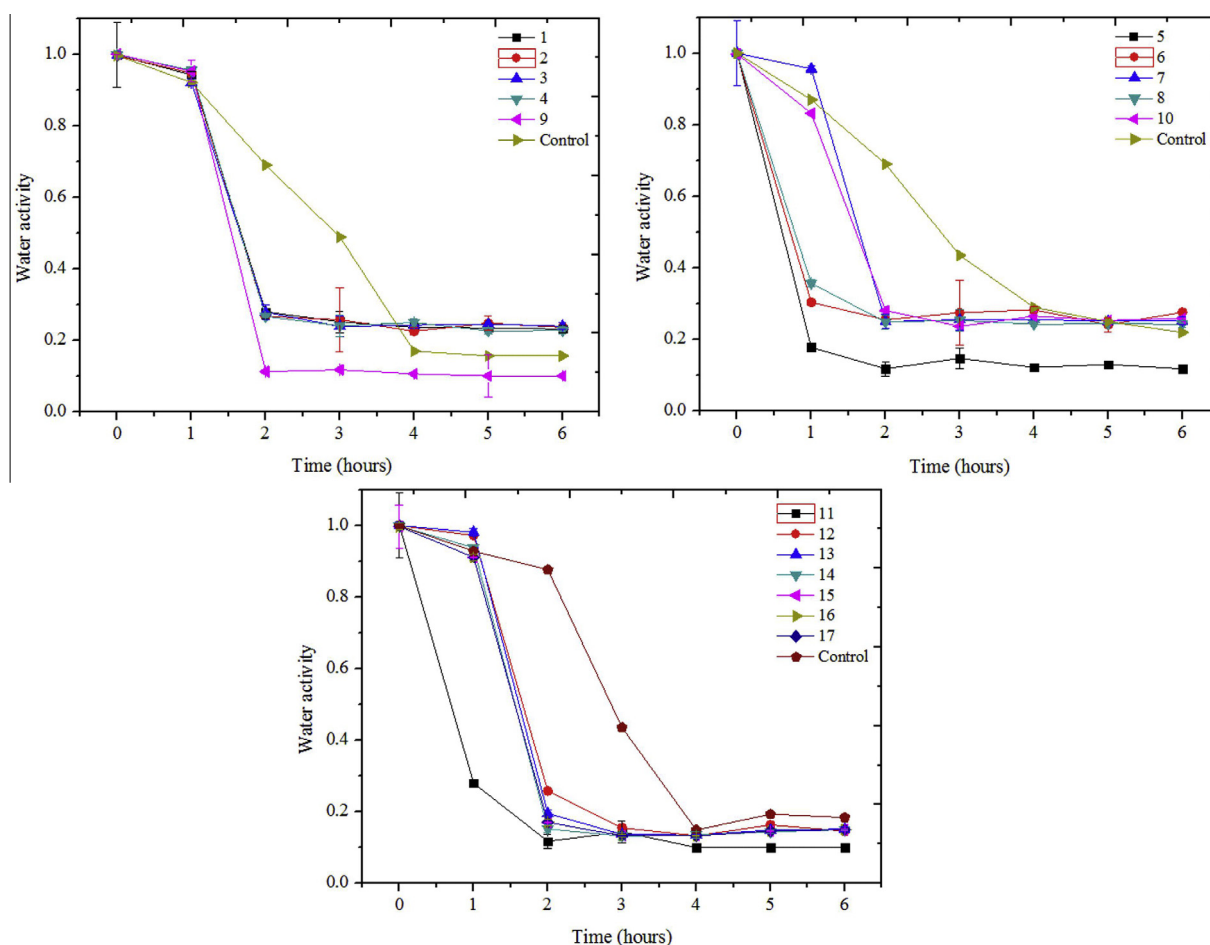
Water diffusivity data was fitted to the quadratic model given in Eq. (8). *F*-test and ANOVA analysis were used as significance criteria for the fitted models. The model was statistically significant at 90% of confidence interval since the calculated *F* value (4.05) was higher than the listed *F* value ( $F_{9,7} = 2.72$ ). Good correlation coefficients were obtained ( $R^2 = 0.84$ ).

$$D = 5.90 - 19.65x + 19.22x^2 - 0.003y - 0.000001y^2 - 0.02z - 0.0015z^2 + 0.01xy + 0.04xz - 0.0001yz \quad (8)$$

The response surface plot, built with Eq. (8), showing the effect of the power intensity and the bagasse:water ratio on water diffusivity is presented in Fig. 1B. The highest water diffusivity was observed at higher bagasse:water ratio (Table 1). The increase in water diffusivity implies in shorter processing times. As air-drying is an expensive processing, the decrease in the drying time means less energy consumption and thus a cheaper process. To increase the effective diffusivity only by heating, an increase in the air drying temperature from 40 to 65 °C is necessary. However, high drying temperatures are associated with the degradation of



**Fig. 1.** Pareto chart for water diffusivity of cashew apple bagasse in air-drying process after sonication. Linear (L) and quadratic (Q) responses; 1L by 2L interaction between processing time and power intensity; 1L by 3L interaction between bagasse:water ratio and processing time; 2L by 3L interaction between power intensity and processing time (1A); Response surface plot showing the effects of bagasse:water ratio and ultrasound power intensity on water diffusivity plotted for a constant processing time of 6 min (1B).

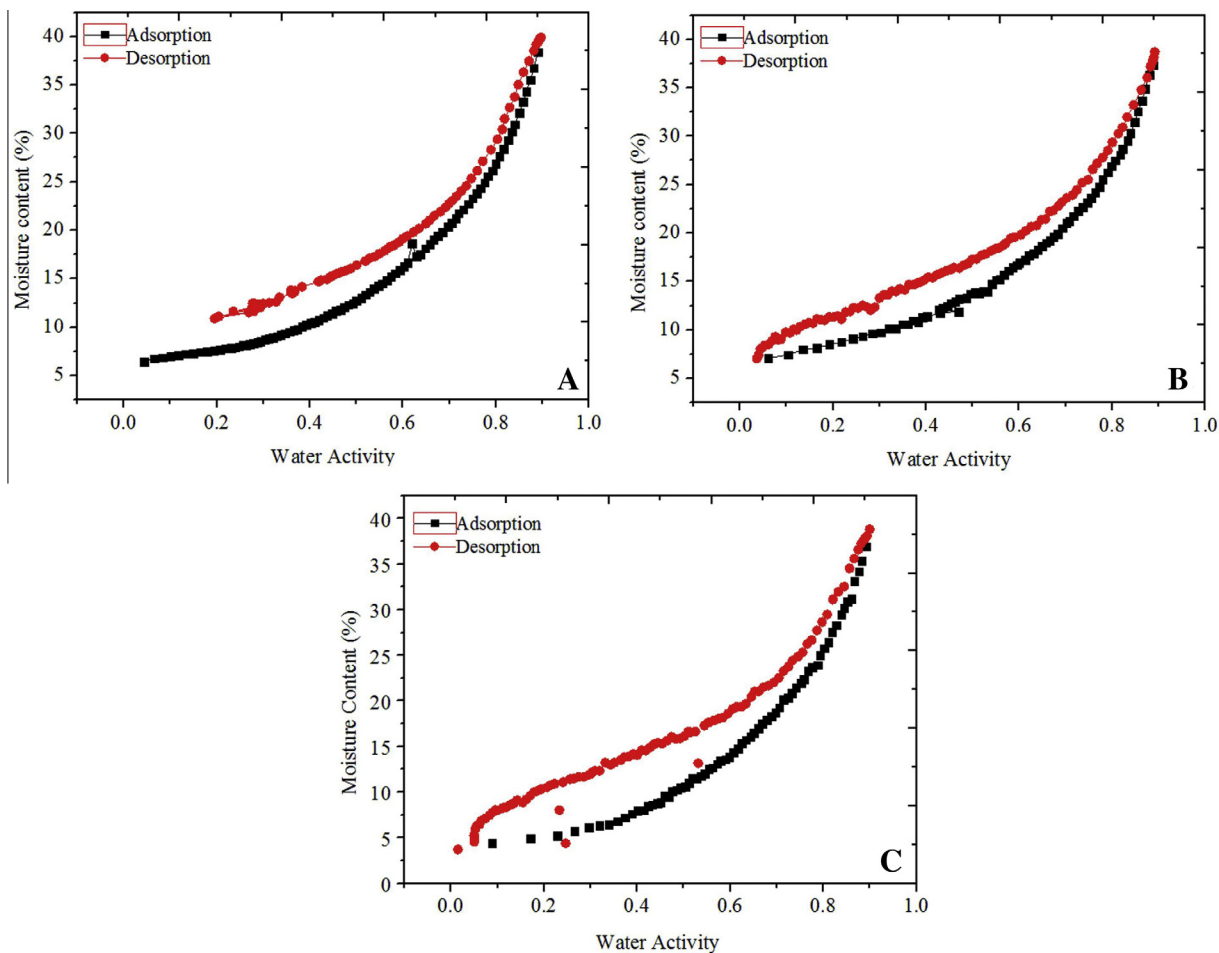


**Fig. 2.** Water activity during drying of sonicated cashew apple bagasse.

heat sensitive compounds. On the other hand, the ultrasonic effects were dependent on the applied power and the higher the ultrasonic power, the higher is the effective diffusivity values [24].

Water activity ( $A_w$ ) is one of the most important parameters in dried products and, it is related to the microbial spoilage. Fig. 2 shows the water activity decrease during drying. Assays 5, 6, 8 and 11 presented the fastest  $A_w$  decrease. In assay 5 (1:4 bagasse: water ratio,  $75 \text{ W/cm}^2/2 \text{ min}$ ) the decline in water activity was

more intense in the first hours of drying. For sonicated samples, in the first 2 h of drying, water activity reached values below 0.4, which confers microbial stability since the microbial spoilage is avoided at low  $A_w$  values. For the subsequent drying optimization, the experimental assays 5 and 6 (1:4 bagasse: water ratio;  $226 \text{ W/cm}^2$ , 6 min) were chosen due to their high water diffusivity values. Fig. 3 presents the moisture content as a function of water activity (sorption isotherms).



**Fig. 3.** Sorption isotherms at 30 °C: dried cashew apple bagasse sonicated at 226 W/cm<sup>2</sup> using bagasse:water ratio of 1:4 during 2 min (3A) and 10 min (3B). Fig. 3C shows the sorption isotherm for control sample.

**Table 2**

Desorption isotherm parameters of cashew apple bagasse sonicated and their coefficients of determination ( $R^2$ ).

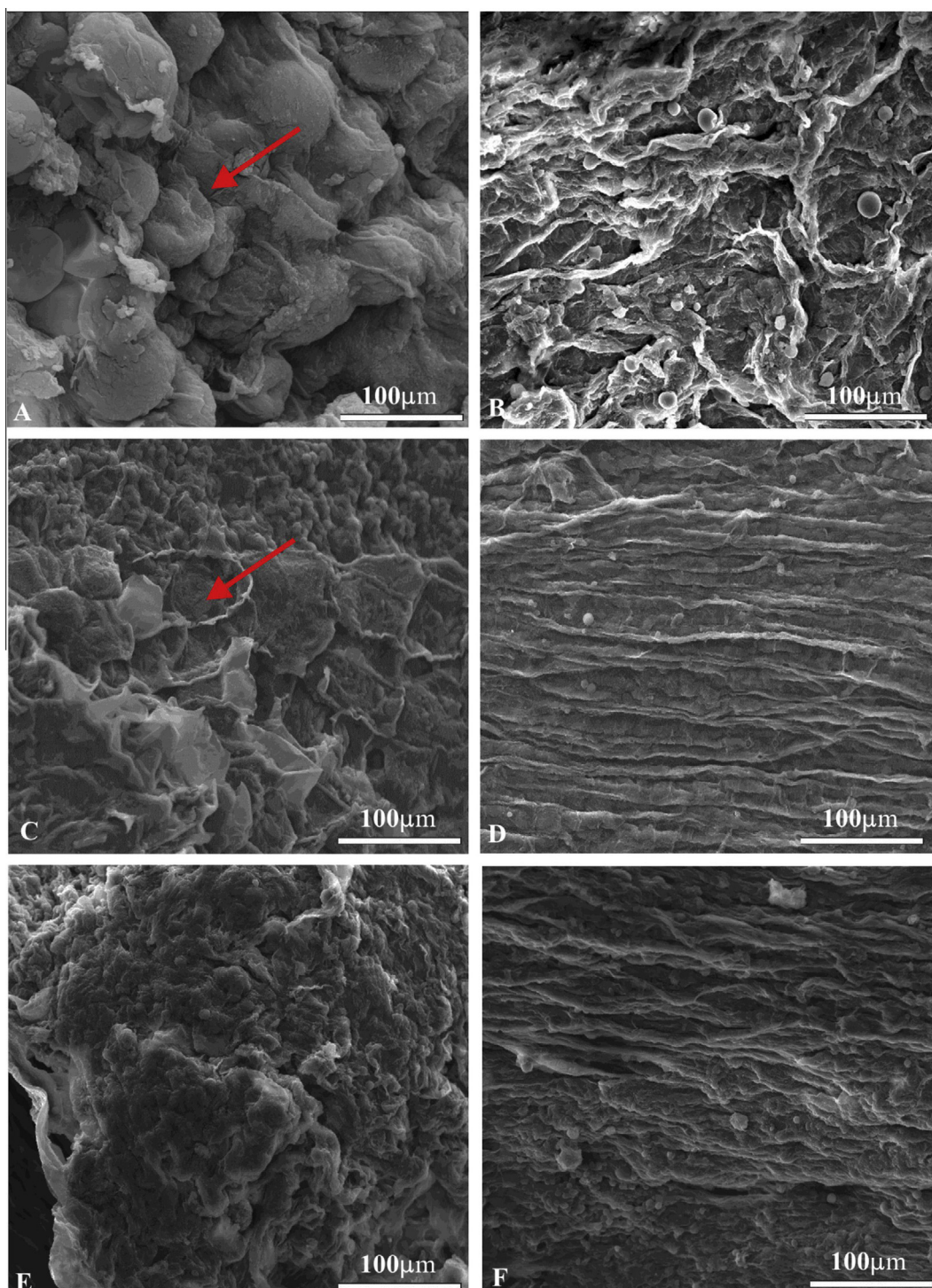
Parameters					$R^2$	$E$
<i>GAB</i>						
Assay 5	Xm	C1	$k$		0.998	0.335
	9.23	$1.06 \times 10^4$	0.86			
Assay 6	Xm	C1	$k$		0.998	0.32
	10.16	82.92	0.82			
Control	Xm	C1	$k$		0.997	0.57
	8.73	35.10	0.86			
<i>BET</i>						
Assay 5	Xm	C			0.976	0.223
	12.38	14.97				
Assay 6	Xm	C			0.965	0.52
	12.01	34.25				
Control	Xm	C			0.972	0.49
	10.93	17.60				
<i>DLP</i>						
Assay 5	$b_0$	$b_1$	$b_2$	$b_3$	0.998	0.322
	13.54	-6.35	3.08	0.19		
Assay 6	$b_0$	$b_1$	$b_2$	$b_3$	0.999	0.23
	14.45	-7.05	1.45	-0.20		
Control	$b_0$	$b_1$	$b_2$	$b_3$	0.997	0.50
	12.41	-6.94	1.32	-0.54		

Assay 5 sonicated at 75 W/cm<sup>2</sup> during 2 min at 1:4 of bagasse:water ratio; Assay 6 sonicated at 75 W/cm<sup>2</sup> during 10 min at 1:4 of bagasse:water ratio; Control non-sonicated immersed in water by 10 min at 1:4 of bagasse:water ratio. Xm = moisture content in molecular monolayer on dry basis (kg water/kg dry solid); C, k,  $b_0$ ,  $b_1$ ,  $b_2$  e  $b_3$  = empirical coefficients.

The sorption isotherms of cashew apple bagasse presented sigmoidal-shape for all samples and followed the type II of BET classification (Fig. 3). The isotherm shape reflects the way the water binds to the food matrix. The BET type II isotherm takes into account the existence of multilayers at the internal surface of the material [26,27]. The isotherms presented in Fig. 3 evidence the hysteresis phenomena, which happens when the adsorption isotherm present lower values than the desorption isotherm. Sonicated samples (3A and 3B) resulted in the less hysteresis effect due to the closer adsorption and desorption

curves compared to control (Fig. 3C). Caurie [28] reported that hysteresis is an index of food quality. The higher the hysteresis, the lower is the dried food stability. Thus, sonication can improve the stability of dried food due to the hysteresis phenomena decrease.

Table 2 shows the isotherms parameters of sonicated cashew apple bagasse and control sample (non-sonicated). The sonicated samples showed higher monolayer values ( $X_m$ ) compared to control. The monolayer moisture ( $X_m$ ) value reflects the amount of water strongly adsorbed on specific locations



**Fig. 4.** Scanning electron micrographs of cashew apple bagasse after 2 min of sonication at  $75 \text{ W/cm}^2$ : (5A and 5B); bagasse after 10 min of sonication at  $75 \text{ W/cm}^2$  (5C and 5D); and raw bagasse (5E and 5F).  $1000\times$ .

of the food, and it is considered the best value to assure its stability [29].

The increase on  $X_m$  and the hysteresis reduction observed for sonicated samples are in agreement to the reported for sonicated banana starch [30]. Sonicated banana starch presented higher monolayer values ( $X_m$ ) and lower hygroscopicity compared to the control (non-sonicated sample). Thus, sonication may increase  $X_m$  and reduce the product hysteresis (Table 2).

The GAB constants C1 and K are indicative of the isotherm type [31]. Observing the parameters C1 and K in Table 2, it is possible to note that  $k < 1$  and  $C > 2$  were obtained in all assays. According to Blahovec [32], the values obtained for these isotherms are type II isotherms as can be observed in Fig. 3 (sigmoidal-shape).

According to the  $R^2$  values, all models were well adjusted to the experimental data. However, to evaluate the best fitting, the lower error ( $E$ ) was considered. Therefore, the DLP equation was the best-fitted model for all samples evaluated, followed by GAB and BET, respectively. DLP is a model developed by Decagon Devices® and it is considered superior to the others models for complex isotherms because it provides a better adjust in a broad range of  $A_w$  [33].

### 3.2. Tissue structure

Fig. 4 shows the scanning electron microscopy (SEM) of sonicated and non-sonicated cashew apple bagasse. Sonication waves

caused severe structural damage to cells with visible rupture of parenchyma cells compared to the non-sonicated bagasse (Fig. 4E) as seen in the scanning electron micrographs (Fig. 4C; assay 5). Although no cell rupture was found for assay 6 (Fig. 4A), it is possible to note the plasmolysed aspect of cells. Parenchyma cell walls are composed almost entirely of cellulose fibrils and play a role in the water movement and transport in plants [34].

Sonication did not affect the lignocellulosic fibers, or sclerenchyma cells (Fig. 4B, D). The main feature of sclerenchyma cells is the presence of thickened and often lignified secondary walls. Due to the presence of these walls, sclerenchyma cells are essential elements in the strength and plant support [35].

Fig. 5 evidence the micro-channels formation and the cell collapse due sonication. This result is consistent with previously published studies [25–27]. Ultrasonic pretreatment affects the fruit tissue, making the water diffusion easier during air-drying. The microscopic channels formed due to the US processing contributes to the water diffusivity increase [36–38]. Rodrigues et al. [3] also found disruption and breakdown of cells with elongation of parenchyma cells in sapota after ultrasound treatment (25 kHz; US intensity of  $48.70 \text{ W/cm}^2$  for 10 min). In this work, higher ultrasound intensities were applied what can explain the intensity of damages observed in cashew apple cells. As the authors suggested [3], the effect of ultrasound on fruit tissue depends on the tissue structure and composition, but also depends on the processing parameters.

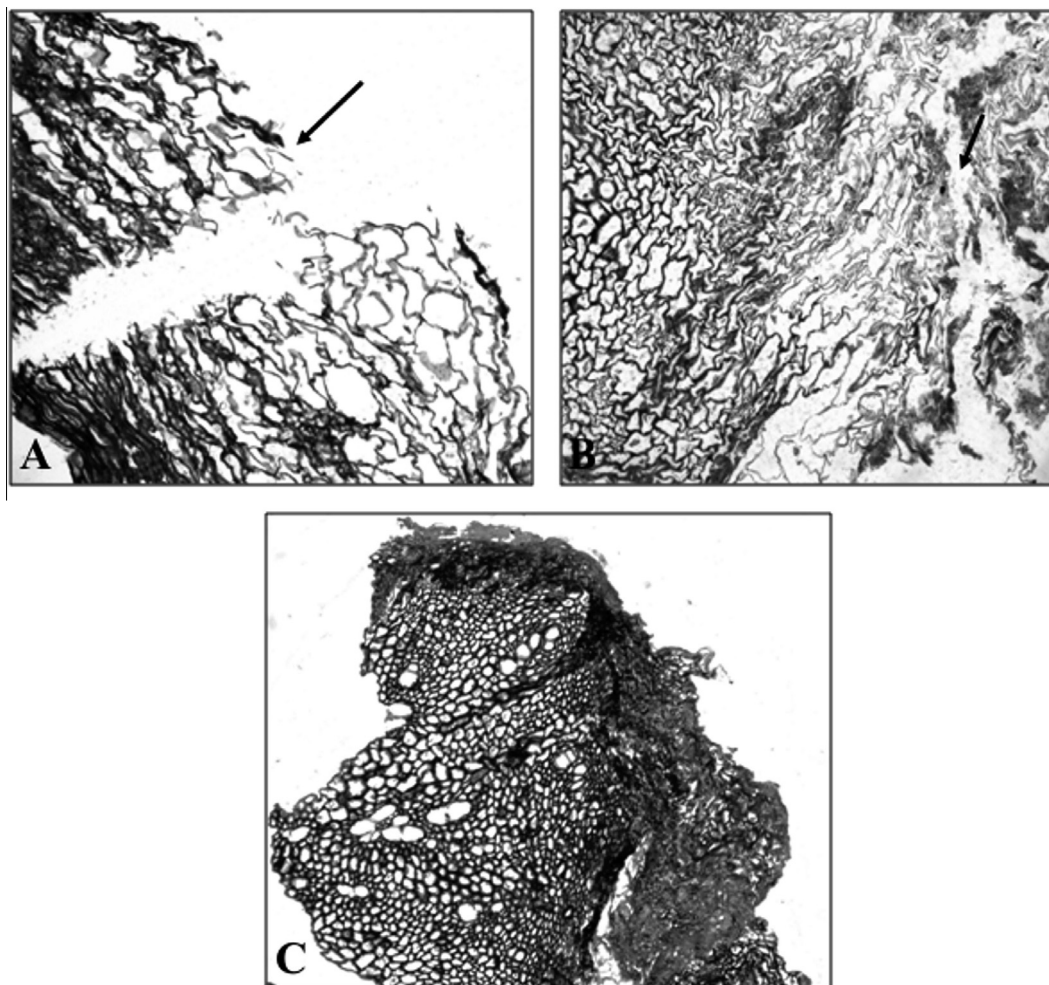


Fig. 5. Photomicrographs of cashew apple bagasse after 2 min of sonication at  $75 \text{ W/cm}^2$ : (A), region with collapsed cells; and bagasse after 10 min of sonication at  $75 \text{ W/cm}^2$  (B), region with wide microscopic channels. Magnification:  $20\times$ . Control sample (C). Magnification:  $10\times$ .



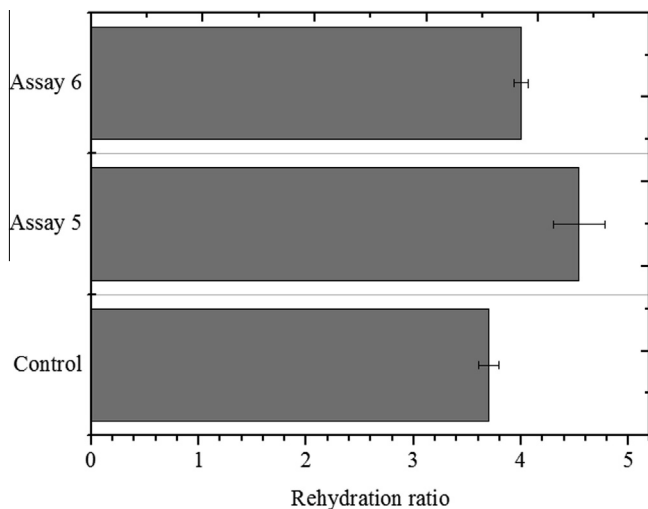


Fig. 6. Rehydration ratio of convective dried cashew apple bagasse.

The structural changes observed for sonicated samples significantly affected the rehydration capacity of cashew apple bagasse (Fig. 6). Rehydration rate increased 22% (assay 5) and 8% (assay 6) compared to control. This result may be attributed to the structural changes caused by ultrasound such as the formation of microchannels, loss of cellular adhesion and rupture of the cell walls [15]. High rehydration rates facilitate the use of the dried product as food ingredients. Dried ingredients must be fast rehydrated and well mixed in the food matrix.

### 3.3. Effects on enzymes

The most important factors that determine the rate of the enzymatic browning of fruits and vegetables are the concentrations of polyphenoloxidase (PPO) enzyme, phenolic content, pH, temperature, and oxygen availability [18]. Fig. 7A depicts the PPO activity during drying. Drying reduced the PPO activity in sonicated cashew apple bagasse resulting in about 20 and 12% enzyme activity loss for assays 5 and 6, respectively (Fig. 7A). However, PPO activity increased  $\approx 20\%$  in control sample after 2 h of drying. Piga et al. [39] reported that PPO activity remains high for long periods when the drying temperature was 55 °C. At temperatures above 75 °C only a moderate activity was observed. Terefe et al. [40] reported that the thermostability of PPO depends on the species as well as on the plant cultivar.

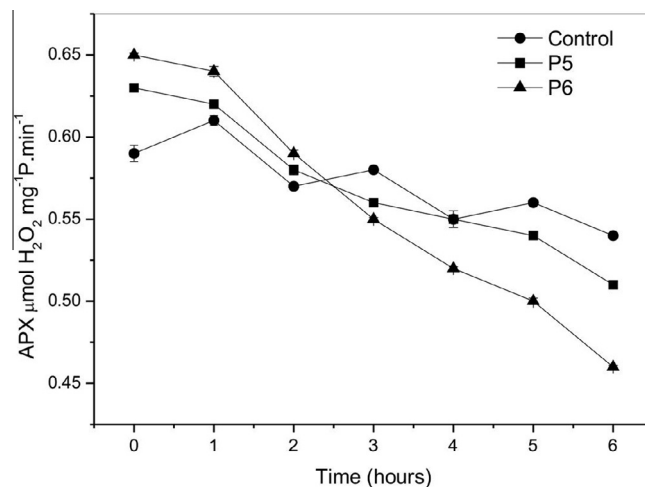
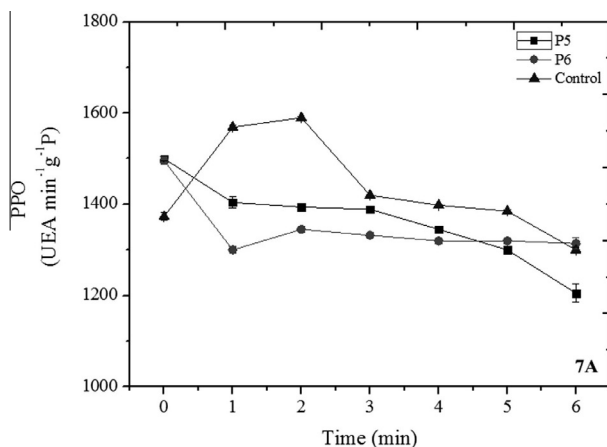


Fig. 8. APX activity of cashew apple bagasse sonicated at 75 W/cm<sup>2</sup> during 2 min (Assay 5) and 10 min (Assay 6) using a bagasse:water ratio of 1:4 along drying.

Fig. 7B shows the POD activity of sonicated cashew apple bagasse along drying. POD activity did not decrease, showing that drying unaffected POD activity. However, ultrasound pretreatment irreversibly inactivated the enzyme decreasing its initial activity by 75% compared to the control sample. The enzyme activity in the control increased about 9% after 6 h of drying (Fig. 7B).

Fig. 8 shows that drying decreased the APX activity in sonicated bagasse. The enzyme loss was about 20% for assay 5 and 30% for assay 6. A slight increase was observed in control at the beginning of drying, followed by a less sharp drop compared to the treated samples. The reduced activity of PPO, POD and APX in sonicated and dried cashew apple bagasse is relevant of the final product quality.

### 3.4. Effects on color

Despite the high activity of PPO, no browning was observed in sonicated samples (Fig. 9). Non-enzymatic browning reactions were typically found after extended drying periods due to the low water activity [41]. A higher decrease in all color parameters evaluated was observed for the control sample, which denotes a more intense browning of the product surface. The results clearly reveal that sonication could effectively minimize browning because the chroma and hue values were much more stable in sonicated samples.

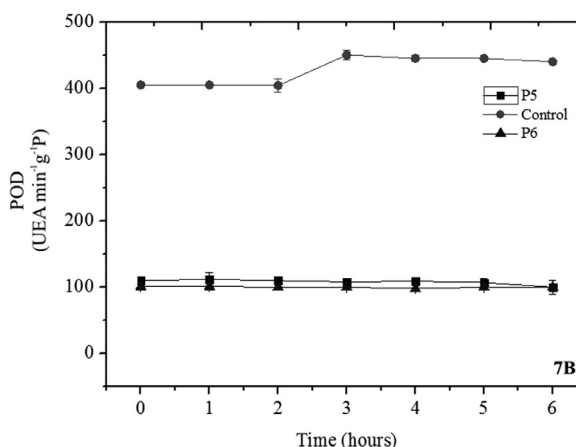
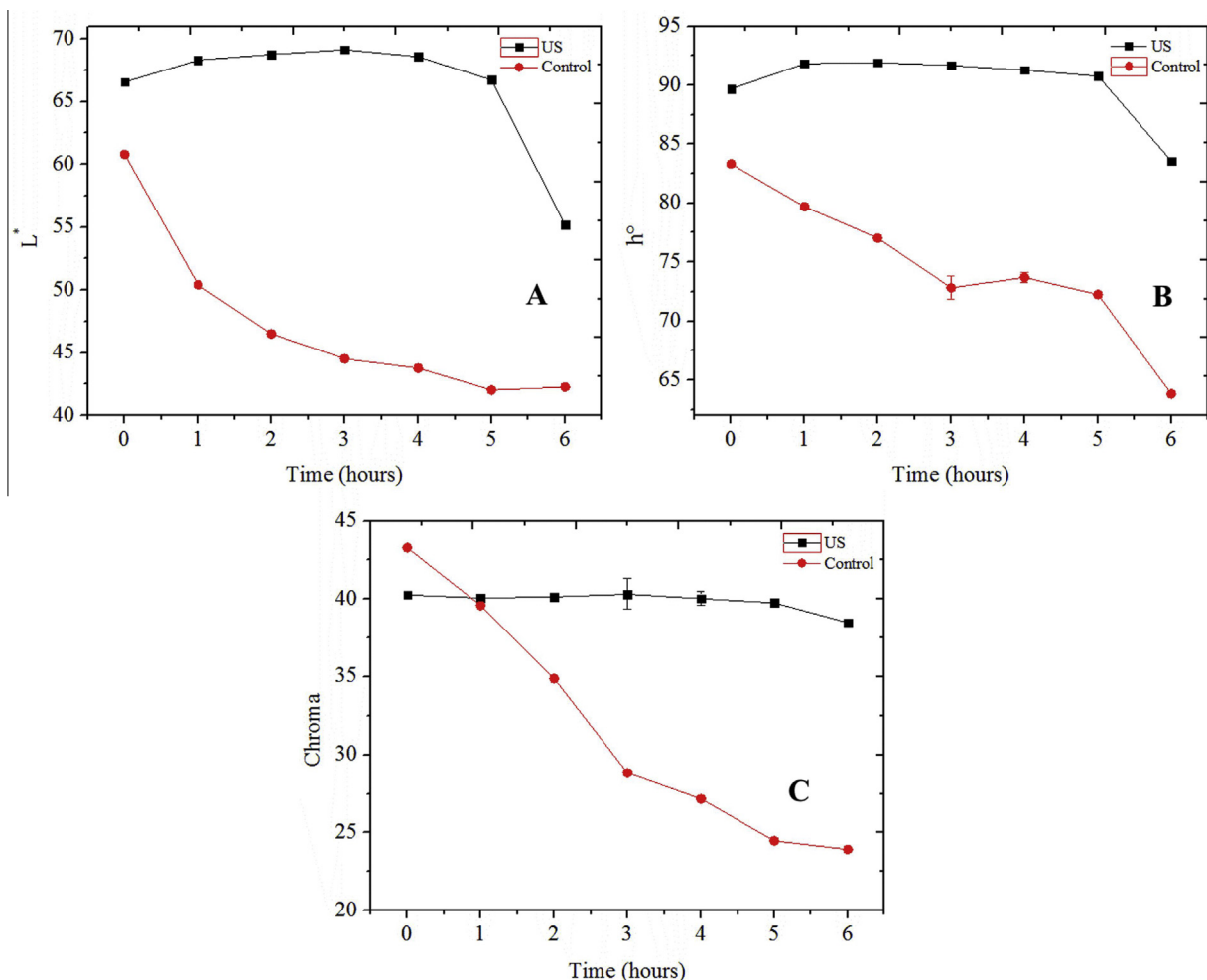


Fig. 7. PPO activity of cashew apple bagasse sonicated at 75 W/cm<sup>2</sup> during 2 min (Assay 5) and 10 min (Assay 6) using a bagasse:water ratio of 1:4 along drying (7A); POD activity of cashew apple bagasse sonicated at 75 W/cm<sup>2</sup> during 2 min (Assay 5) and 10 min (Assay 6) using a bagasse:water ratio of 1:4 (7B).

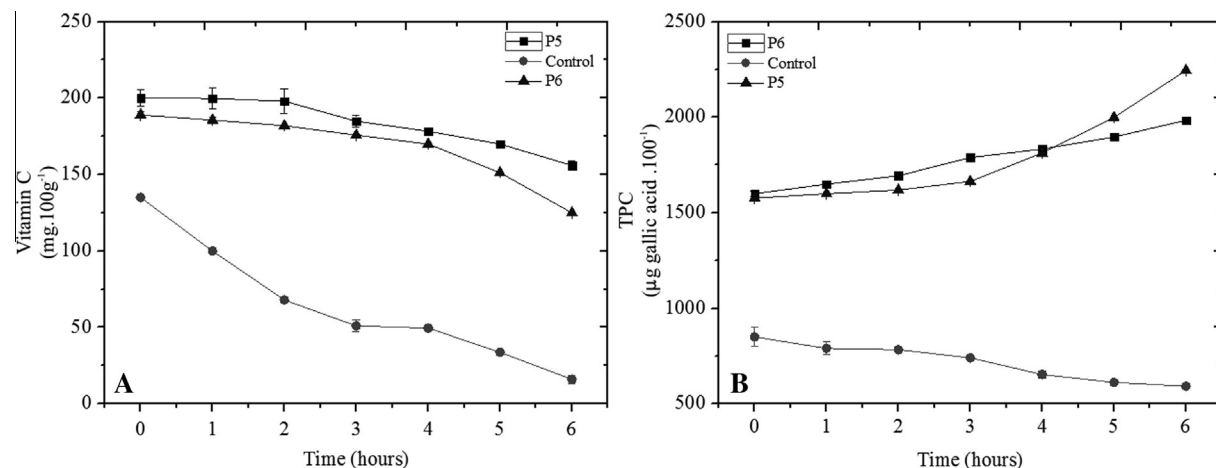


**Fig. 9.** Color parameters  $L^*$ (A),  $h^\circ$  (B) and chroma (C) along drying of cashew apple bagasse sonicated at  $75 \text{ W/cm}^2$  during 2 min (Assay 5) and 10 min (Assay 6) using a bagasse:water ratio of 1:4. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

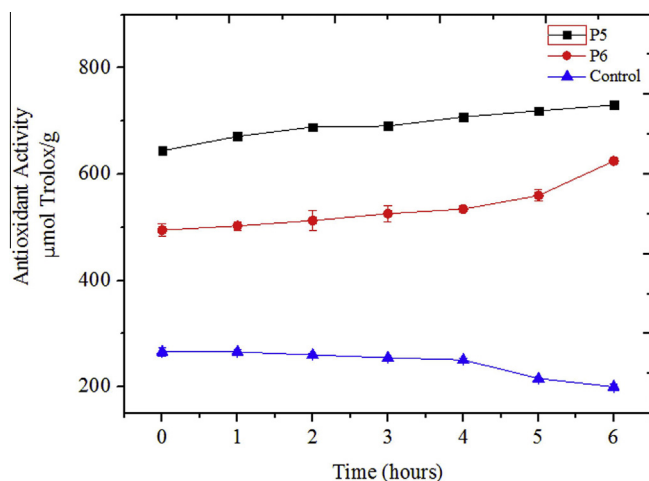
### 3.5. Effects of sonication on the functional compounds of dried cashew apple bagasse

In several studies, ascorbic acid (Vitamin C) has been taken as an index of nutrient quality of foods. Ultrasound is a technology reported as to have a minimal effect on the quality of food that

contain heat labile vitamins [42]. Fig. 10A shows that vitamin C content decreased progressively with the drying time, as expected, due to the heat sensitive nature of ascorbic acid. The initial vitamin C content was  $200 \text{ mg } 100 \text{ g}^{-1}$  and  $189 \text{ mg } 100 \text{ g}^{-1}$  for sonicated samples in assays 5 and 6, respectively, and  $135 \text{ mg } 100 \text{ g}^{-1}$  for the control sample. Sonication increased the vitamin C content



**Fig. 10.** Vitamin C of cashew apple bagasse sonicated at  $75 \text{ W/cm}^2$  during 2 min (Assay 5) and 10 min (Assay 6) using a bagasse:water ratio of 1:4 (9A); Total phenolic compounds of cashew apple bagasse sonicated at  $75 \text{ W/cm}^2$  during 2 min (Assay 5) and 10 min (Assay 6) using a bagasse:water ratio of 1:4 (9B).



**Fig. 11.** Changes in antioxidant activity of cashew apple bagasse sonicated at 226 W/cm<sup>2</sup> during 6 min using a bagasse:water ratio of 1:4 during drying.

**Table 3**

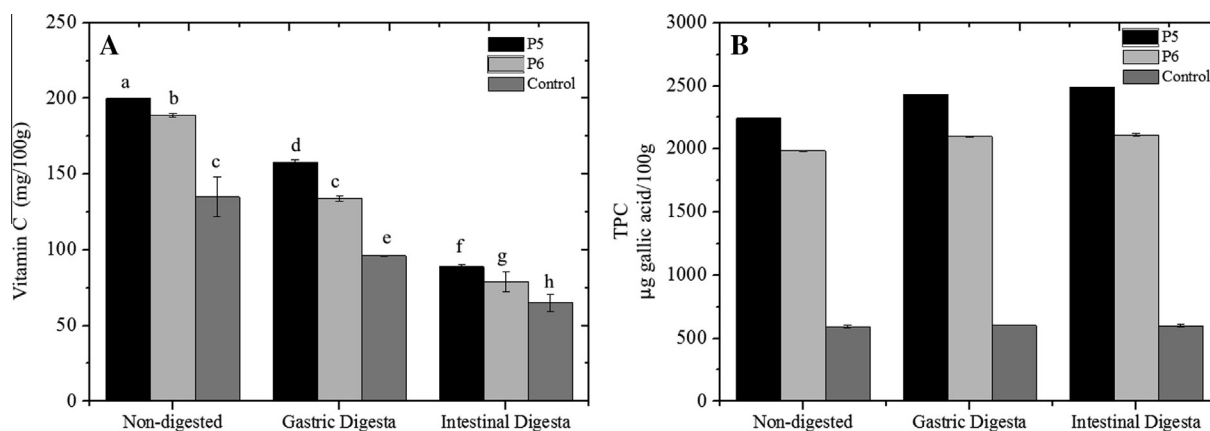
Pearson's correlation coefficients (*R*) between antioxidant capacity and antioxidant compounds of dried sonicated bagasse.

Assay 5			Assay 6		
<i>R</i>	TPC	Vitamin C	<i>R</i>	TPC	Vitamin C
ABTS	0.87*	−0.91*	ABTS	0.93*	−0.96*
Vitamin C	−0.96*		Vitamin C	−0.98*	

\* Significant at  $p < 0.05$ .

due to the cell disruption and the release of the intracellular content. Thus, the initial vitamin C content of sonicated samples were higher than in the control sample.

The stability of vitamin C during drying is affected by several variables such as the drying conditions and the sample moisture content [43]. As shown in Fig. 10A, for sonicated samples there was a slow initial rate of vitamin C loss at relatively higher moisture contents (beginning of drying), followed by a period of faster degradation rates as the moisture content decreased (end of drying). The maximum losses observed after 6 h of air-drying were 20% and 34% for sonicated samples (assays 5 and 6, respectively) and 88% for control samples (Fig. 10A). A similar trend was observed for strawberries dehydration assisted by power ultrasound. Sonicated strawberry samples, even after the most severe ultrasound processing conditions (70 °C and 60 W), presented higher vitamin C retention compared to thermally treated samples [15].



**Fig. 12.** Vitamin C concentration during in vitro gastrointestinal digestion of sonicated and dried (6 h) cashew apple bagasse. Different lower case letters indicate significant differences ( $p < 0.05$ ) (11A); Total phenolic concentration during in vitro gastrointestinal digestion of sonicated and dried (6 h) cashew apple bagasse. Different lower case letters indicate significant differences ( $p < 0.05$ ) (11B).

Fig. 10B shows the concentration of TPC during the drying process. Sonicated samples showed a significant increase ( $p < 0.05$ ) in TPC along drying, ranging from 1576 to 2244 mg GAE/100 g for assay 5 and 1600–1984 mg GAE/100 g for assay 6. For control samples, it was observed a gradual decrease in TPC concentration (30%) (Fig. 10B). The higher concentrations of vitamin C and phenolics in sonicated samples are due to the cell disruption promoted by the ultrasound treatment, which allows the release of the intracellular compounds. The reduction in the enzyme activity of oxidoreductases such as POD, PPO and APX (ascorbate peroxidase) also contributed to the preservation of TPC and vitamin C.

Cashew apple presents high antioxidant activity, high TPC and vitamin C values [22]. As can be seen in Fig. 11, the antioxidant activity tended to increase ( $p < 0.05$ ) during drying. The increase in phenolic compounds and the high final amount of vitamin C contributed to the enhancement of the antioxidant activity in sonicated samples. Piga et al. [39] studied the influence of drying parameters on the phenolic compounds and antioxidant activity of prunes and found higher antioxidant values after drying. The authors have attributed the result to the formation of new compounds with higher antioxidant activity, for example, in Maillard reaction, which creates several products with markedly higher antioxidant power.

In the present study, the total phenolic contents showed a positive correlation with antioxidant activity ( $R = 0.87$ ; assay 5 and  $R = 0.93$ ; assay 6) as shown in Table 3. The positive correlation indicates that phenolic compounds are the major contributors to the antioxidant properties of this product. A significant and positive correlation was also reported for phenolic compounds and antioxidant activity by other authors [22,44,45].

### 3.6. In vitro bioaccessibility of vitamin C and phenolic compounds

Bioaccessibility is defined as the amount of an ingested nutrient that is available for absorption in the gut after digestion [46]. Kamiloglu et al. [47] emphasized the importance to evaluate the availability of antioxidants after digestion due to evidence of poor bioavailability of certain antioxidants, which would, in turn, have a limited effect on health. Fig. 12A depicts the effect of sonication on bioaccessibility of vitamin C of dried cashew apple bagasse.

The vitamin C concentration of sonicated samples was reduced by 21% (assay 5) and by 29% (assay 6) in the gastric digesta, compared to non-digested sonicated cashew apple bagasse. After intestinal digestion, greater losses of vitamin C were observed (55% for assay 5 and 58% for assay 6). Similar behavior was observed for control samples (52%). The ultrasound processed

sample showed a better vitamin C bioaccessibility compared to the non-sonicated sample (control). Despite the observed losses, the daily requirement of vitamin C (40 and 45 mg per day according to the FAO/WHO) is achieved in  $\approx 50$  g of dried cashew apple bagasse.

Similar recovery of vitamin C in gastric digesta (67.7%) and intestinal digesta (47.3%) were found for blended fruit juice [48], showing that the loss is expected after digestion. The vitamin C oxidation in the gastrointestinal tract is due to its action as electron donor maintaining ions in the reduced state and regenerating the active form of other dietary constituents.

Fig. 12B shows the effect of sonication in bioaccessibility of total phenolic compounds of cashew apple bagasse.

The total phenolic concentration increased 8% (assay 5) and 6% (assay 6) in the gastric digesta, compared to the non-digested cashew apple bagasse (Fig. 12B). Likewise, TPC increased 2% (assay 5) and 1% (assay 6) in the intestinal digesta. No significant differences were found for gastric and intestinal digesta of control samples.

During the gastric phase, the extraction of phenolics from cashew apple bagasse was more efficient. The release of polyphenols from cashew apple bagasse in simulated gastro-intestinal digestion is mainly achieved during the gastric phase. These results are in agreement with Palafox-Carlos [49], who reported that phenolic acids in the aglycone form are absorbed in the upper part of the gastrointestinal tract, explaining the rapid absorption of these compounds ranging from 1 to 2 h after intake of fruits and vegetables.

Few studies on the effects of ultrasound on bioaccessibility of bioactive compounds have been done. Sonication caused loss of cell integrity and decrease the degree of pectin esterification of tomato pulp [50]. The changes on the gel-like properties resulted in a reduction of the lycopene in vitro bioaccessibility. The authors attributed this results to the presence of a stronger network that make lycopene less available to the digestion. No other previous publications were found on the effect of sonication on the bioaccessibility of bioactive compounds in foods.

#### 4. Conclusions

This study showed that ultrasound processing had significant impact on the drying rates of cashew apple bagasse due to the increase of water diffusivity in most cases. This phenomenon may occur due to the formation of microchannels as a result of ultrasound application. Moreover, samples pretreated with ultrasound retained more vitamin C and total phenolic compounds compared to conventional dried samples. This process can be applied to produce dried fruits products in reduced time with high nutritional value. Ultrasound processing also reduced the rehydration rates of the dried product. Better rehydration rates facilitate the use of dried bagasse as food ingredient due to the better mixing in the food matrix.

Our findings confirm that ultrasound processing increased the bioaccessibility of vitamin C and phenolic compounds compared to control samples (non-sonicated). The results obtained are promising for industrial application. The sonicated dried cashew apple bagasse showed better nutritional quality compared to the non-sonicated sample. The dried product may be applied as food ingredient in ice-creams, deserts and others formulations.

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