Characterization of the jabuticaba residue dehydrated and lyophilizated

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Abstract

The world's population is increasingly interested in ready to eat, healthy, nutritious and functional foods. Brazil has a great biodiversity of native fruits and one of them is the jabuticaba. These native fruit is rich in fibers, mainly in the residue (skin and seed) of the jabuticaba processing, which is around 50% of the fruit weight. Thus, the objective of this research was characterize the residue of the jabuticaba Sabará (Plinia cauliflora) type dehydrated by two different methods: dehydration in a tray dryer with forced air circulation at 45 °C (R1) and lyophilization (R2). The characterization of the skin and seed (residue) of the jabuticaba processing by means of physic-chemical and microbiological parameters, bioactive compounds (phenolic, antioxidant and total anthocyanins content), particle size and instrumental color. The residue R1 presented values of total fibers (26.2±0.02 g/100 g), moisture (6.19 g/100 g) and pH (3.42) significantly (p<0.05) higher than residue R2. The residue R2 presented values of total acidity (110.3 mEq NaOH/100 g) and acidity in citric acid (7.06 g/100 g) significantly (p<0.05) higher than R1. Microbiological analyzes confirmed satisfactory sanitary hygienic conditions of the processing, the total coliform counts at 35 °C and thermotolerant coliforms at 45 °C were lower than 3.0 NMP/g for both residues, the were below the maximum limit permitted by the current Brazilian legislation. The mold and yeast counts of R1 was1.0x10³ CFU/g and R2 was 2.2x10³ CFU/g, both were below the maximum limit (10^4 CFU/g) permitted. There was no significant difference (p> 0.05) in the mean of the particle size of R1 (80.8 μ m) and R2 (80.7 μ m).

Keywords: jabuticaba, residue, dehydration, fiber, bioactive compounds.

Resumo

Caracterização do resíduo de jabuticaba desidratado e liofilizado

A população mundial está cada vez mais interessada em alimentos prontos para consumo, saudáveis, nutritivos e funcionais. O Brasil tem uma grande biodiversidade de frutas nativas e uma delas é a jabuticaba. Esta fruta nativa é rica em fibras, principalmente no resíduo (casca e semente) do processamento da jabuticaba, que é cerca de 50% do peso da fruta. Assim, o objetivo desta pesquisa foi caracterizar o resíduo da jabuticaba do tipo Sabará (*Plinia cauliflora*) desidratado por dois métodos diferentes: desidratação em um secador de bandeja com circulação de ar forçada a 45 °C (R1) e liofilização (R2). Caracterizar a casca e da semente (resíduo) do processamento de jabuticaba por meio de

parâmetros físico-químicos e microbiológicos, compostos bioativos (teor de compostos fenólico, capacidade antioxidante e teor de antocianinas totais), tamanho de partícula e cor instrumental. O resíduo R1 apresentou valores de fibras totais ($26,2\pm0,02$ g/100 g), umidade (6,19 g/100 g) e pH (3,42) significativamente (p < 0,05) maior do que o resíduo R2. O resíduo R2 apresentou valores de acidez total (110,3 mEq de NaOH/100 g) e acidez em ácido cítrico (7,06 g/100 g) significativamente (p < 0,05) superior a R1. As análises microbiológicas confirmaram as condições higiênicas sanitárias satisfatórias do processamento, as contagens de coliformes totais a 35 °C e os coliformes termotolerantes a 45 °C foram inferiores a 3,0 NMP/g para ambos os resíduos, ficando abaixo do limite máximo permitido pela legislação brasileira vigente. As contagens de bolores e leveduras de R1 foi de 1,0 x 10³ UFC/g e R2 foi de 2,2x10³ CFU/g, ambos estavam abaixo do limite máximo (10⁴ CFU/g) permitidos. Não houve diferença significativa (p > 0,05) na média do tamanho de partícula de R1 (80,8 µm) e R2 (80,7 µm).

Palavras-chave: jabuticaba, resíduo, desidratação, fibra, compostos bioativos.

Introduction

The jabuticaba of the species Sabará (*Plinia cauliflora*) is a native fruit from Brazil, that has high productivity and it has a great economic potential considering it can be transformed in different products due to its unique sensorial characteristics. It is a fruit widely appreciated; besides it has a potential functional claim (Junior et al., 2011).

Due to the high activity of water and high sugar content, jabuticaba has a very limited shelf life. Because of the perishability, usually it is transformed into different products, such as: jelly, jam, juice, frozen pulp and fermented products (liqueur, "wine" and vinegar).

Jabuticaba skin has been extensively studied in recent years due to the high content of fibers, pectin and anthocyanins. It has different compounds, substances that control the blood sugar and cholesterol levels, helping to reduce the incidence of heart disease and gallstones (Ferreira et al., 2012). However, in general the residue (skin and the seeds representing around 50% of the initial mass of the whole fruit) of jabuticaba is discarded or it is used for animal feed.

This residue, mainly the skin, has a unique phytochemical composition with the presence of compounds of great interest, such as the different polyphenols, tannins, anthocyanins, cyanidin-3-glucoside, delphinidin-3-glycoside, depsides, gallotannins, elagitaninos and others. Moreover, the flour from the jabuticaba residue has around 15% of fibers and it is rich in pectin.

Different studies were carried out with the application of jabuticaba skin in different food products. It was used as a flour (by convective dehydration or freeze-dried) or as an extract (liquid or dehydrated). The ice cream was formulated with the flour jabuticaba skin, the physical-chemical and sensorial characteristics of the product were evaluated. The study concluded that the addition of up to 5% of jaboticaba of skin flour provided an increase in nutritional value of the ice cream without affecting the sensorial characteristics (Lamounier et al., 2015).

This research aims to characterize the jabuticaba residue in order to find out how the dehydration method affects the composition and content of bioactive compounds.

Material and Methods

The jabuticaba species (*P. cauliflora*) were purchase from the Boa Vista Farm, in Lagoa Branca, in Casa Branca municipality, State of São Paulo.

The jabuticaba fruits were transported to Embrapa Agroindustry of Food in Rio de Janeiro where it have had been washed. The sanitization of the fruits was by immersion in chlorinated water (200 ppm for 15 minutes). And then the jabuticaba fruits were processed in order to separate the skin and seeds (residue) from the pulp (Bonina 0.25 df).

The jabuticaba residue was divided into two parts for dehydration. Half of it was dried in a convective tray dryer with forced air circulation at 45 °C until reaching constant weight (this process last 36 hours). In the freeze-drying process, the residue was frozen at

18 °C for 12 hours. The lyophilization carried out with a lyophilizer (Edwards Pirani 50I), at a pressure of -1 atm and a temperature of -50 °C for 30 hours, reaching the maximum temperature of 40 °C.

The dehydrated jaboticaba residue was ground in a hammer mill and packaged in vacuum metallized polyethylene packs and then it was stored in a cold chamber at -18 $^{\circ}$ C until use.

The jabuticaba residue flour was characterized by physico-chemical, microbiological parameters, phenolic compounds content, antioxidant capacity, total anthocyanins content, particle size and instrumental color.

Total dietary fiber, moisture, pH, acidity (% of citric acid) and total acidity analyses were performed according to the methodologies described by AOAC (2010). To determine the water activity, the AquaLab apparatus (Washington, USA) was used according to the manufacturer's instructions.

Microbiological analyzes of total coliforms, thermotolerant coliforms and molds and yeasts were counted according to APHA (2001).

Total phenolic compounds determination was performed according to the spectrophotometric method proposed by Singleton & Rossi (1965), modified by Georgé et al. (2005) and the results were expressed as Gallic acid equivalent.

Antioxidant activity determination was evaluated by two methods, by ABTS and by ORAC. Following the ABTS method, the results were expressed in Trolox Equivalent Antioxidant Capacity (TEAC), the extracts were obtained according to the methodology described by Rufino et al. (2007) and the quantification was performed according to Re et al (1999). Using the Oxygen Radical Antioxidant Capacity (ORAC) method, the results were expressed in Trolox Equivalent Antioxidant Capacity (TEAC) based on the methodology described by Zuleta et al. (2009). The differential pH methodology was used the evaluated the anthocyanin content by according to Giusti and Wrolstad (2001) and adapted by Cruz (2013).

The particle size distribution of the jabuticaba flours was analyzed using a laser particle analyzer (Microtrac Inc., Montgomery Ville, USA). In the analysis was used distilled water as a dispersant and the refractive index of 1.33 (AACC, 2010).

Instrumental color analysis was performed by reflectance using in the equipment Color Quest XE, CIELAB and CIELCh scale. It was used a 25 mm diameter aperture, with a D65/10 illuminant and it measured the parameters: L* (the brightness); a* (-80 to zero = green, from zero to +100 = red); b* (-100 to zero = blue, from zero to +70 = yellow) and C* (Chroma) that defines the intensity or the saturation of color (Fernandes et al., 2010).

The experiment followed a completely randomized design and all the analysis were performed in triplicate. The results were submitted to variance analysis and to the Student test for comparison of means, at the 5% probability level using statistical program $SAS^{\ensuremath{\mathbb{R}}}$ version 8.1.

Results and Discussion

The jabuticaba flour consists mainly of dietary fibers (Table 1). According to Brazilian regulation (Resolution RDC N° 54/2012), food products with a minimum of 6% of fibers can be classified as "high fiber content" (Brasil, 2012). Therefore, the dehydrated jabuticaba flours R1 and R2 can be considered as an ingredient with high fiber content, R1 had a significantly (p<0.05) higher total fiber content than R2.

Ferreira et al. (2012) found lower total fiber values (15.25%) than those observed in this study, they used the flour of the jabuticaba skin in the formulation of cookies. Leite-Legatti et al. (2012) studied the freeze-dried jabuticaba skin flour and found total fiber contents of 25% higher than that what was found in R2 using the same dehydration method.

According to Brazilian legislation, flours may contain maximum moisture content of 15% (BRASIL, 2005). The jabuticaba flours analyzed were in conformity with the legislation. The moisture found in jabuticaba flour R1 (6.61%) was significantly (p<0.05) lower than what was found in R2 (7.06%). Other researchers observed moisture values higher than this study. Zago (2014) characterized the jabuticaba skin flour found 11.1% of moisture; Boari et al. (2008) found 12.05% and Leite-Legatti et al. (2012) observed 15.33% of moisture in the jabuticaba skin. Alves et al. (2014) studied the moisture behavior of a dehydrated jabuticaba skin flour during 12 months of storage and they found a significant increase in the moisture content from 9.28% to 14.01%.

The R1 (0.40) and R2 (0.23) flour analyzed showed a low water activity, there was a significant difference (p<0.05) between them. These values are within the maximum limit to be safe, to avoid multiplication of microorganisms (Chiste et al., 2006).

The pH values of the jabuticaba flour allow to classify them as an acidic food, the pH of R1 (3.42) was significantly (p<0.05) higher than the R2 (3.32). Zago (2014) and Boari et al. (2008) in their studies with jabuticaba skin flour found values of 3.90 and 3.47, respectively. These values were very close to those observed in this study. Lima (2009) analyzing the pH of the skin (3.39) and the seed (3.97) of the jabuticaba Sabará also found similar values.

Total acidity and citric acid acidity analyzes in R2 (7.06% and 110.3) were significantly (p<0.05) higher than the values found in R1 (6.61% and 103.4). The values of acidity in citric acid found in this study were higher than what was found by Zago (2014) and Boari et al (2008), it was 1.55% and 1.37%, respectively.

The acidity values observed in this research are in agreement with the pH values. According to Ferreira et al (2013), low values of titratable acidity may be associated with the transformation of organic acids between carbon dioxide and water during storage.

The results of thermotolerant coliforms (<3 CFU/g) and total coliforms (<3 CFU/g), in the jabuticaba residues flour R1 and R2 confirm the good hygienic sanitary conditions of processing. The molds and yeasts values of R1 ($1.0x10^3$ CFU/g) and R2 ($2.2x10^3$ CFU/g) are below the maximum limit (10^4 CFU/g) allowed by Brazilian legislation (Brasil, 2001). Thus, it can be stated that the pre-processing and dehydration conditions of the both flours were adequate.

The jabuticaba flours R1 and R2 presented high levels of phenolic compounds, of total anthocyanins and high antioxidant activity by $ABTS^+$ and by ORAC methods (Table 2). These high levels of phenolic compounds and anthocyanins are compatible with the high antioxidant activity. There was no significant difference between treatments applied to phenolic compounds, total anthocyanins and antioxidant activity by the $ABTS^+$.

Araújo et al. (2013) evaluated the extraction of the phenolic compounds from the jabuticaba skin dehydrated at 45 °C observed lower values when compared to this study of 403 mg of gallic acid in 100 g of sample. These authors, when evaluating the antioxidant capacity of these samples using the $ABTS^+$ method, found similar values to the observed in this research of 1017.8 µmol trolox in each gram of sample (Araújo et al, 2013).

The US Department of Agriculture published a list of 277 foods based on the antioxidant activity by the ORAC method (Haytowitz et al., 2010). The antioxidant activity determined by the ORAC method of R1 was significantly (p<0.05) higher than R2. The high values of total phenolic compounds are responsible for the high antioxidant capacity of the fruits (Bahramikia et al., 2009; Sahreen et al., 2010; Barros et al., 2012).

Alves et al (2011) analyzed the total anthocyanins content in the jabuticaba skin flour dehydrated at 45 °C and found values of 646 mg/100 g, close to what was found in this research, and 205.7 mg/100 g in the freeze-dried, lower value than found in this research.

In the particle size distribution analysis the flour R1 and R2 there was no significant difference (p> 0.05), it was 80.8 μ m and 80.7 μ m, respectively. In R1 flour sample about 60% of the particles passed through a 79.63 μ m aperture, while in the R2 about 80% of the particles passed through a 65.16 μ m aperture. Since R1 and R2 have particles smaller than 500 μ m, they are classified as powder.

On the chromatographic coordinates a^* and b^* of the jabuticaba flour, it can be observed that R2 presented values significantly higher than those found in R1, as well as in the value of L* (Table 3). Due to the values of R1 and R2, they were classified as purplish color.

The coordinate a^* , in R1 and R2 are represented by positive values, indicate shades that tend to red, while the coordinates of b^* indicate a tendency toward yellow. Observing the values of the intensity of red (+ a^*) and intensity of yellow (- b^*), it was verified that there was predominance of the intensity of red on the intensity of yellow.

Alves et al (2014) found L^* values (37.10) lower than those found in this study. The drying temperature of the samples, favoring the darkening, and it can influence the luminosity.

The low dehydration temperatures of 45 $^{\circ}$ C (R1) and 40 $^{\circ}$ C (R2) contributed to maintain of high phenolic compounds, total anthocyanins and antioxidant capacity. According to Leonid et al. (2006), nutrients, bioactive compounds and pigments are sensitive to thermal processing.

Conclusions

The jabuticaba flour R1 presented higher concentration of dietary fiber, phenolic compounds and antioxidant capacity, whereas the R2 showed a higher concentration of anthocyanins.

Both dehydrated jabuticaba flours presented satisfactory sanitary hygienic conditions and confirmed the effectiveness of the pre-processing steps and the dehydration processing employed, ensuring the microbiological quality.

Therefore, both flours R1 and R2 studied are interesting ingredients to apply in the food industry in different products to provide color and to increase the fiber content. Besides, they showed a high antioxidant activity due to the presence of phenolic compounds and anthocyanins that can possible bring health benefits to consumers.

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Table 1 - Physico-chemical results of jabuticaba flour R1 and R2. Different letters in the columns indicate significant difference (p < 0.05).

Jabuticaba flour	Dietary fibers	Moisture	Acidity in citric acid	Total acidity	рН	Water activity
		$(g.100 g^{-1})$		(meq NaOH .100 g ⁻¹)		
R1	26.20 ^a	6.19 ^a	6.61 ^b	103.41 ^b	3.42 ^a	$0,400^{a}$
R2	17.34 ^b	5.30 ^b	7.06 ^a	110.30 ^a	3.32 ^b	0,228 ^b

Table 2 - Phenolic compounds, total anthocyanins and antioxidant capacity by the $ABTS^+$ and ORAC methods. Different letters in the columns indicate significant difference (p<0.05).

Jabuticaba flour	Phenolic compounds	Total	Antioxidant capacity		
	(mg gallic acid.100 g ⁻¹)	anthocyanins (mg100 g ⁻¹)	(µmol tro ABTS ⁺ (TEAC)	ORAC	
R1	544,37±6,84 ^a	601,88±15,61 ^a	1084,12±61,55 ^a	438,05±7,43 ^a	
R2	458,37±9,45 ^a	822,45±30,99 ^a	$955,85\pm58,09^{a}$	421,43±19,61 ^b	

Table 3 -. Luminosity components (L*), chromatographic coordinates (a* and b*) and h_{ue} angle evaluated in the jabuticaba residue flours R1 and R2. Different letters indicate significant differences between treatments (p<0.05).

Jabuticaba flour	L*	a*	b*
R1	44.97 ± 0.30^{b}	9.27 ± 0.28^{b}	1.68 ± 0.11^{b}
R2	48.46 ± 0.69^{a}	13.30 ± 0.57^{a}	2.69 ± 0.18^{a}