

Casca de jaboticaba cristalizada: uma nova estratégia para industrialização de subprodutos

Crystallized jaboticaba peel: a novel strategy for by-product industrialization

Piel de jaboticaba cristalizada: una nueva estrategia para la industrialización de subproductos

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Resumo

O objetivo deste trabalho foi a aplicação integral da casca de jaboticaba na obtenção de uma casca de jaboticaba cristalizada por desidratação osmótica, cristalização e secagem por convecção a diferentes temperaturas. A cada dois meses durante um ano, o produto era submetido a determinações de antocianinas, compostos fenólicos, capacidade antioxidante,

características físico-químicas e determinação da cor. As medidas das cores e as determinações dos compostos bioativos foram significativamente influenciadas pela interação entre os fatores temperatura de secagem e tempo de armazenamento. A composição proximal não foi afetada pela temperatura de secagem, enquanto os sólidos solúveis totais, pH, acidez titulável e atividade da água apresentaram diferenças significativas. A casca de jabuticaba cristalizada é um produto de baixo custo e alternativa viável para minimizar as grandes perdas durante a safra.

Palavras-chave: Resíduo agroindustrial; Casca cristalizada; Desidratação osmótica.

Abstract

The aim of this work was the integral application of jabuticaba peel to obtaining a crystallized jabuticaba peel by osmotic dehydration, crystallization and convective drying at different temperatures. Every two months for a year, the product was subjected to determinations of anthocyanins, phenolic compounds, antioxidant capacity, physicochemical characteristics and determination of color. The color measurements and bioactive compounds determinations were significantly influenced by the interaction between the factors drying temperature and storage time. The proximal composition was not affected by drying temperature, whereas total soluble solids, pH, titratable acidity and water activity showed significant differences. Crystallized jabuticaba peel is a low cost product and viable alternative to minimize the large losses during the harvest season.

Keywords: Agroindustrial residue; Crystallized peel; Osmotic dehydration.

Resumen

El objetivo de este trabajo fue la aplicación integral de la piel de jabuticaba para obtener una piel de jabuticaba cristalizada mediante deshidratación osmótica, cristalización y secado por convección a diferentes temperaturas. Cada dos meses durante un año, el producto se sometió a determinaciones de antocianinas, compuestos fenólicos, capacidad antioxidante, características físico-químicas y determinación del color. Las medidas de color y las determinaciones de los compuestos bioactivos fueron influenciadas significativamente por la interacción entre la temperatura de secado y los factores de tiempo de almacenamiento. La composición proximal no se vio afectada por la temperatura de secado, mientras que los sólidos solubles totales, el pH, la acidez titulable y la actividad del agua mostraron diferencias significativas. La piel de jabuticaba cristalizada es un producto de bajo costo y una alternativa viable para minimizar las grandes pérdidas durante la cosecha.

Palabras clave: Resíduos agroindustriales; Piel cristalizada; Deshidratación osmótica.

1. Introduction

The interest in edible tropical fruits has increased in developed countries due to their potential health benefits (Clerici & Carvalho-Silva, 2011; Oliveira et al., 2012). Among the native species of importance in Brazil, stands out jabuticaba tree (*Myrciaria* sp) belonging to the Myrtaceae family, which was domesticated and incorporated into popular culture by indigenous Tupi (Danner et al., 2006).

The jabuticaba fruit, does not have high commercial value since the fruit has a shelf life of up to three days, when changes are observed in the appearance of the fruit, due to water loss, decay and fermentation of the pulp, which impairs their commercialization (Lima et al., 2008). However, many products made from fruit do not use the peel in their formulations, making it a co-product for the food industry, since peels and seeds reach about 50% of the total weight of the jabuticaba fruit (Lima et al., 2008).

The utilization of non-edible parts of tropical fruits, as well as being an innovative strategy to minimize the potential for environmental problems caused by improper disposal of by-products (Sousa & Correia, 2010), could be an alternative to increase the consumption of bioactive compounds since the peel and the seeds of jabuticaba contain significant amounts of bioactive compounds (antioxidants, anthocyanins, phenolics compounds and tannins) (Alejandro et al., 2013; Gurak et al., 2014; Vendramini & Trugo, 2004; Wu & Kennelly, 2013). The frequent intake of bioactive compounds from fruits and vegetables contributes to control parameters related to inflammation, lipid and glycemic profile, oxidative stress and free radicals.

Crystallization of jabuticaba peel is an alternative for utilization since it increases the fruit shelf-life. This process consists essentially in osmotic exchange between solids contained in high sugar concentrated syrups, at levels that prevent deterioration. Thus, with osmotic dehydration followed by drying, products such as candied fruit may be obtained, making it an alternative for the use of these co-products (Morita et al., 2005). The aim of this work was the novel industrial application of jabuticaba peel by the technically fruits crystallization to obtain a crystallized jabuticaba peel.

2. Material and methods

It is a quantitative study, in which part was carried out in the field (collection of fruits and preparation of crystallized jabuticaba peel) and part in the laboratory (determination of compounds and data analysis) (Perreira et al., 2018).

2.1 Raw material

Fruits of *Myrciaria jabuticaba* (Vell) Berg harvesting season 2012 were harvested at the Fazenda & Vinícola Jabuticabal in Nova Fátima, district of Hidrolândia, located at 16° 55' 32.35" South latitude and 49° 21' 39.76" West longitude, in the State of Goiás, Brazil. The fruits were selected, washed with clean water, sanitized with sodium hypochlorite 100 µL L⁻¹ for 15 min (Brazil, 2001). Peels were obtained in electrical depulper (Itametal, 0:25 Bonina df), pulp and seeds were separated for the preparation of other products.

2.2 Preparation of crystallized jabuticaba peel

Crystallization of jabuticaba peel was performed according to our patent registration number BR 1020130233510 in three steps: osmotic dehydration, crystallization and convective drying. Osmotic dehydration was carried out in thermostatic bath at 60 °C for 6 hours, at shaking frequency of 80 rpm, using the ratio: 1:4 peel/solution (w/w) using sucrose solution at 70 °Brix. The temperature and the concentration of sucrose solution were determined through preliminary tests (data not shown). After osmotic dehydration, jabuticaba peels were subjected to crystallization process which consisted in transferring them to a solution composed 20% of impalpable sugar (containing 1.5% of corn starch in order to remove moisture from the sugar) and drinking water at 80% (w/w). Peels at the ratio of 1:4 peel/solution (w/w) remained immersed for 30 minutes at 28 °C by thermostatic bath controlling.

They were dried at temperatures of 60, 70 and 80 °C in a convective tray dryer pilot (1.90 m long x 0.80 m wide with a capacity of five metal trays of 0.055 m x 0.057 m) at air flow rates of 0.0206 m³ of air per kg of crystallized jabuticaba peels per second to achieve final moisture between 20 and 25% and water activity between 0.5 and 0.6 (60°C/6 hours; 70°C/4.5 hours; 80°C/3 hours). After drying, about 40 g of crystallized peels in pieces were

transferred to metallized package (polyester-aluminum-polyethylene) of 12 cm long x 8 cm wide, in which vacuum was applied. The samples were stored at room temperature (28 ± 1 °C) and analyzes were conducted for 12 months at 2-month intervals.

2.3 Physicochemical characterization of crystallized jabuticaba peel

The moisture content was determined by drying at 105 °C to constant weight; ash was performed by gravimetry after incineration in a muffle at 550 °C; total nitrogen by the Kjeldahl method considering the conversion factor of 6.25 for crude protein according to *Association of Official Analytical Chemists* (AOAC, 2010). Total lipid content was determined by the method of Bligh and Dyer (1959). Carbohydrate content was calculated by difference, subtracting from one hundred the values of moisture, ash, protein and lipids. The energy value was estimated by the coefficients of ATWATER (carbohydrates and proteins = 4.0 kcal g⁻¹ and lipids = 9.0 kcal g⁻¹) (Merril & Watt, 1973). The levels of reducing and total sugars were determined by the 3,5-dinitrosalicylic acid method, according to the methodology proposed by Miller (1959), for the determination of total sugar prior hydrolysis was carried out. The non-reducing sugars were determined by difference. Total soluble solids were determined using a digital refractometer (Atago N-1D) and expressed in °Brix of the sample at 20 °C, according to AOAC (2010). The pH measurements were performed on digital potentiometer (pH meter HI-9224). Total acidity was determined by titration the sample with 0.1 N NaOH. Both analyses were performed according to AOAC (2010) and water activity was determined at 25 °C in Aqualab device (Aqualab CX-2). All the analysis were performed in tree replicates and average values have been reported.

2.4 Color

Color determination was performed by reading the three parameters defined by the CIELAB system. The color instrumental parameters (L*, a*, b*) were determined with a colorimeter diffuse reflectance (ColorQuest II Sphere), with geometrical optical sensor ball, performing reading by reflection, using angle to observation 10, main illuminant D65 and specular reflection excluded (RSEN). The L* value defines lightness (L* = 0 black and L* = 100 white) while a* and b* define the chromaticity (+a* red and -a* green, +b* yellow and -b* blue), and Chroma was estimated by the equation 1.

$$\text{Chroma} = \sqrt{a^2 + b^2} \quad (1).$$

2.5 Bioactive compounds

2.5.1 Total anthocyanins

The total anthocyanin content was estimated by the method of Lees and Francis (1972), adapted by Barcia et al. (2012). For the extraction of anthocyanin compounds we used 1 g of sample, which were added 25mL of ethanol solution of pH 1.0, and incubated for one hour at room temperature. Readings were performed on spectrophotometer (SP-220 Biospectro) at 520 nm, representing the absorption spectrum of anthocyanins present in crystallized jaboticaba peel. The quantification of anthocyanins was based on the of cyanidin-3-glucoside, which is the major anthocyanin present in jaboticaba peel (Equation 2).

$$A = \varepsilon \cdot C \cdot l \quad (2)$$

where A is the absorbance; ε is the molar extinction coefficient ($98.1 \text{ L mol}^{-1} \text{ cm}^{-1}$) (Barcia et al., 2012); C is the concentration (mol L^{-1}) and l is the path length (cm). The results were expressed as milligrams of cyanidin-3-glucoside per 100 grams of sample (dry basis).

2.5.2 Total phenolic compounds

Phenolic compounds were determined by spectrophotometry (Biospectro SP-220) at 750 nm, using the Folin-Ciocalteu reagent (Waterhouse, 2002). Phenolics were extracted in water, and the quantification was based on the standard curve for gallic acid, in the range 5 to 50 mg of gallic acid L^{-1} . The results were expressed in mg of gallic acid equivalents (GAE) per 100 grams of sample (dry basis).

2.5.3 Antioxidant activity

The antioxidant activity was measured by the DPPH (2,2-diphenyl-1-picrylhydrazyl) method, according to Brand-Williams *et al.* (1995), with modifications of Borguini and Torres (2009). The discoloration of DPPH was read at 517 nm in spectrophotometer (Biospectro SP-220) in aqueous extracts and the results were expressed as percentage of

discoloration. The absorbance readings were performed after 20 minutes of reaction. Calculations were carried out with the aid of the equation 3.

$$\% \text{ discoloration DPPH} = \left(1 - \left(\frac{\text{Abs sample} - \text{Abs blank}}{\text{Abs control}} \right) \right) * 100 \quad (3)$$

where, Abs sample is the absorbance of the sample; Abs blank is the absorbance of the blank; Abs and control is the control absorbance (750 μ L of methanol + 1.5 mL of DPPH).

2.6 Statistical Analysis

Analysis of variance (ANOVA) and Tukey test were performed by Statistical SISVAR software at 5% of probability.

3. Results and Discussion

The moisture, ash, protein, lipid, carbohydrate contents and energy value of crystallized jabuticaba peel were not affected by the drying temperature ($p > 0.05$) (Table 1).

Table 1 – Proximal composition of crystallized jabuticaba peel.

Proximal composition (g . 100 g ⁻¹)	Averages \pm standard deviation
Moisture	22.47 \pm 0.179
Protein	1.57 \pm 0.162
Lipids	0.31 \pm 0.002
Ash	0.17 \pm 0.001
Carbohydrate	75.48 \pm 0.333

Source: Author (2019).

The energy value was 310.98 \pm 0.45 kcal (100 g⁻¹) due almost exclusively to sugars from the peel incorporated by osmotic dehydration, once the product presented low values for protein and lipids.

Total sugar content, expressed by % glucose, reducing and non-reducing sugars were not affected ($p > 0.05$) by the drying temperature, whereas total soluble solids, pH, titratable acidity and water activity were significantly influenced ($p \leq 0.05$) as shown in Table 2.

Table 2 – Total sugar (% glucose), reducing and non-reducing sugar contents, total soluble solids, pH, titratable acidity and water activity of crystallized jabuticaba peel, osmotically dehydrated and subjected to different drying temperatures.

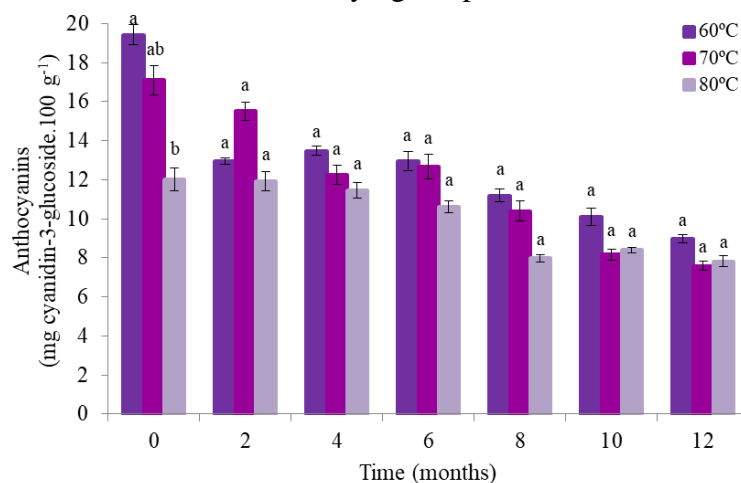
Physical and chemical analyzes	Drying temperature*		
	60 °C	70 °C	80 °C
Total sugars (g . 100 g ⁻¹)	73.937 ^a ± 0.089	74.335 ^a ± 0.084	74.340 ^a ± 0.087
Reducing sugars (g . 100 g ⁻¹)	57.168 ^a ± 0.197	56.988 ^a ± 0.186	57.141 ^a ± 0.175
Non-reducing sugars (g . 100 g ⁻¹)	16.768 ^a ± 0.228	17.347 ^a ± 0.263	17.199 ^a ± 0.235
Total soluble solids (°Brix)	74.571 ^{ab} ± 0.218	75.048 ^a ± 0.309	74.048 ^b ± 0.282
pH	3.670 ^a ± 0.002	3.673 ^a ± 0.002	3.633 ^b ± 0.002
Titratable acidity (g . 100 g ⁻¹)	6.083 ^c ± 0.087	6.770 ^b ± 0.074	7.449 ^a ± 0.113
Water activity	0.592 ^a ± 0.001	0.583 ^b ± 0.002	0.595 ^a ± 0.001

*Means followed by the same letter in the same line represent statistical similarities among drying temperatures, at 5% of probability by the Tukey test. Source: Author (2019)

The addition of sucrose in the osmotic dehydration process, concentrations of reducing sugars were higher than non-reducing, as seen in Table 2. This occurred due to the dissolution of sucrose in water for the osmotic dehydration process. According Bobbio and Bobbio (2003), sucrose is a non-reducing disaccharide, which in aqueous solution and acid medium under heating, is easily hydrolyzed in reducing D-glucose and D-fructose monosaccharides. Total soluble solids range from 74 to 75 °Brix, whose major component is represented by the total soluble sugars and then by organic acids.

Increasing the drying temperature resulted in higher loss of anthocyanin of crystallized jabuticaba peel, which could be related to the thermal sensibility of this pigment (Figure 1).

Figure 1 – Mean values and standard deviation of anthocyanin contents, expressed as mg cyanidin-3-glucoside . 100 g⁻¹ (dry basis) of crystallized jabuticaba peel, under different convective drying temperatures.

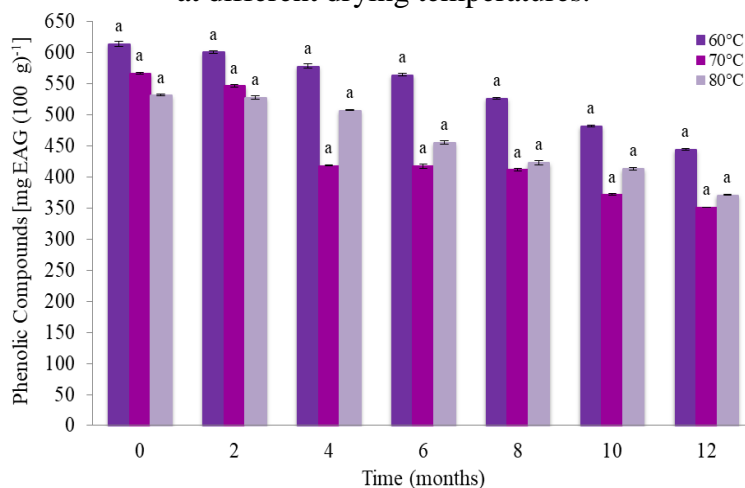


Note: Same letters at the same time do not differ at a significance level of 95% ($p \leq 0.05$), by Tukey test. Source: Author (2019).

The contents of anthocyanins were significantly influenced by the interaction between the factors drying temperature and storage time ($p \leq 0.05$), as seen in Figure 1. Temperature is an important factor in the stability of natural pigments. The degradation of anthocyanins in solution is higher at temperatures over 25 °C, even when complexed with tannic acid, and this degradation is still more pronounced when the pH of the medium increases (Stringheta, 1991).

The phenolic compounds of crystallized jabuticaba peel decreased during the storage period, showing differences among drying temperatures ($p \leq 0.05$) (Figure 2).

Figure 2 – Mean values and standard deviation of phenolic compounds of crystallized jabuticaba peel, expressed in milligrams of gallic acid equivalents (GAE) (100 g^{-1}) (dry basis) at different drying temperatures.



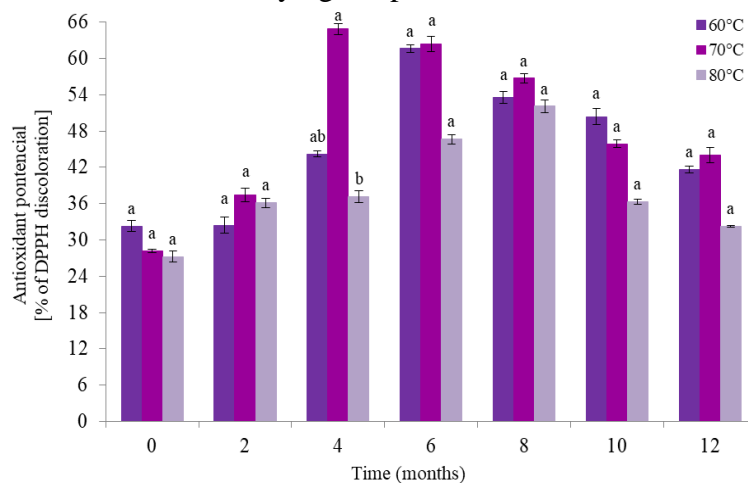
Note: Same letters at the same time do not differ at a significance level of 95% ($p \leq 0.05$), by Tukey test. Source: Author (2019).

During 12 months of storage there were losses due to the effects of weather, but the dried crystallized jabuticaba peel 60 °C were suffering less reduction, as seen in Figure 2, indicating that this is the most appropriate drying temperature for preservation of phenolic compounds.

This reduction in the concentration of phenolic compounds is explained by the low thermal stability at temperatures above 23 °C, corroborated by studies conducted by Chang et al. (2006) that evaluated the effect of storage temperature on the stability of phenolic compounds in fruits.

The antioxidant potential of crystallized jabuticaba peel in aqueous extracts were significantly influenced by the interaction between the factors drying temperature and storage time ($p \leq 0.05$), as shown in Figure 3.

Figure 3 – Mean values and standard deviation of antioxidant potential of crystallized jabuticaba peel, expressed as % of DPPH discoloration (dry basis), at different convective drying temperatures.



Same letters at the same time do not differ at a significance level of 95% ($p \leq 0.05$), by Tukey test. Source: Author (2019).

The increase in antioxidant capacity of crystallized jabuticaba peel (Figure 3) could have its origin in the Maillard reaction products, such as amino-reductans, which have antioxidant effects (Fennema, 2000). These reaction products are formed during prolonged storage of foods containing reducing sugars and aminoacids. The Maillard reaction occurs mainly during thermal processing of foods containing amino acids and reducing sugars, but it can also occur during storage, being more significant in foods with intermediate moisture, which have water activity in the range of 0.5 to 0.8 (Azeredo, 2012), as in the case of the present work. However, the reduction at the end of 12 months, as seen in Figure 3, is explained possibly by degradation of anthocyanins as a function of storage temperature above 20 °C, according to a study by Wicklund et al. (2005) which evaluated antioxidant capacity and color of strawberry jam during storage. Nevertheless, it is noteworthy that even decreasing at the end of storage, antioxidant capacity presented highest levels relative to the start of the experiment.

During storage, the anthocyanins (Figure 1) can undergo two basic changes, as their color become gradually less intense and/or change their tone by the formation of degradation compounds, resulting in different colors of the original (Silva et al., 2010), and these changes can be observed through the values of L^* , a^* , b^* (Table 3).

Table 3. Mean values of determination of the parameters a*, b*, L* and chroma of crystallized jabuticaba peel under different convective drying temperatures.

Parameters	Time (month)	60 °C	70 °C	80 °C
L*	0	22.380 ^c ± 0.096	24.993 ^a ± 0.229	25.540 ^b ± 0.110
	2	23.997 ^c ± 0.112	26.630 ^a ± 0.550	25.570 ^b ± 0.069
	4	26.093 ^b ± 0.067	27.213 ^a ± 0.208	25.830 ^b ± 0.157
	6	28.490 ^a ± 0.159	27.257 ^b ± 0.071	26.133 ^c ± 0.530
	8	28.687 ^a ± 0.031	27.650 ^c ± 0.175	28.240 ^b ± 0.358
	10	29.000 ^c ± 0.165	31.687 ^a ± 0.021	29.653 ^b ± 0.121
	12	31.553 ^b ± 0.035	32.470 ^a ± 0.195	30.687 ^c ± 0.021
a*	0	6.480 ^a ± 0.044	5.480 ^b ± 0.044	2.177 ^c ± 0.081
	2	1.013 ^{ab} ± 0.153	1.227 ^a ± 0.159	0.777 ^b ± 0.112
	4	0.697 ^a ± 0.099	0.930 ^a ± 0.151	0.667 ^a ± 0.047
	6	0.563 ^a ± 0.402	0.490 ^a ± 0.108	0.653 ^a ± 0.040
	8	0.170 ^b ± 0.092	0.480 ^a ± 0.141	0.597 ^a ± 0.114
	10	0.133 ^b ± 0.070	0.483 ^a ± 0.076	0.540 ^a ± 0.108
	12	0.077 ^a ± 0.119	0.340 ^{ab} ± 0.193	0.460 ^a ± 0.036
b*	0	-0.640 ^a ± 0.044	-1.330 ^b ± 0.104	-0.807 ^a ± 0.115
	2	-0.507 ^a ± 0.129	-1.253 ^b ± 0.122	-0.567 ^a ± 0.081
	4	-0.287 ^a ± 0.123	-1.227 ^b ± 0.045	-0.310 ^a ± 0.010
	6	0.067 ^a ± 0.117	-1.213 ^c ± 0.083	-0.210 ^b ± 0.080
	8	0.113 ^a ± 0.015	-0.943 ^c ± 0.040	-0.210 ^b ± 0.115
	10	0.137 ^a ± 0.116	-0.770 ^b ± 0.066	0.017 ^a ± 0.185
	12	0.180 ^c ± 0.010	1.053 ^a ± 0.047	0.853 ^b ± 0.127
Chroma	0	6.512 ^a ± 0.048	5.640 ^b ± 0.019	2.324 ^b ± 0.066
	2	1.134 ^b ± 0.187	1.757 ^a ± 0.150	0.962 ^b ± 0.130
	4	0.760 ^b ± 0.103	1.543 ^a ± 0.085	0.735 ^b ± 0.047
	6	0.577 ^b ± 0.397	1.313 ^a ± 0.043	0.689 ^b ± 0.044
	8	0.208 ^c ± 0.079	1.062 ^a ± 0.099	0.642 ^b ± 0.088
	10	0.201 ^c ± 0.110	0.911 ^a ± 0.070	0.563 ^b ± 0.091
	12	0.213 ^b ± 0.061	1.116 ^a ± 0.095	0.971 ^a ± 0.119

*Same letters in the same line do not differ at a significance level of 95% ($p \leq 0.05$), by Tukey test. Source: Author (2019).

It was noted that the levels representing a* values, as seen in Table 3, showed the same pattern of the curves of anthocyanins (Figure 2), decreasing during storage, tending to yellow. In addition, crystallized jabuticaba peels dried at 80 °C showed significant differences in relation to the others. The increased brightness indicates whitening of the product, therefore, may be noted that, throughout the storage time, the samples became paler.

4. Conclusion

The use of co-product of jaboticaba processing industry is a viable alternative to reduce the wastage caused by large losses during harvest and shows that the industrialization of these materials is a possible alternative for food diversification. Moreover, crystallized jaboticaba peel is a product having considerable amounts of anthocyanins, phenolic and antioxidants compounds.

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