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Active compounds from the industrial residue of dry camu-camu

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Abstract

This paper evaluated the drying kinetics at different temperatures and the effect of the drying temperature on the physical-chemical properties and active compounds of the residue from the industrial processing of camu-camu. The drying temperatures of 40, 50, and 60 °C were evaluated. The residues dried at different temperatures were characterized in terms of their pH, water activity, anthocyanins, flavonoids, carotenoids, phenolic compounds and Fourier transform infrared spectroscopy. The parameters of the Page model showed the best adjustments to the experimental data. Drying affected the concentration of active compounds in the camu-camu residue. At 60 °C, the degradation of flavonoids, anthocyanins, and phenolic compounds was lower than in the dry residue, possibly because less time was required to achieve equilibrium in moisture content. Thus, in order to maintain higher concentrations of active compounds in the residue, drying at higher temperatures is recommended.

Keywords: by-product; fruits; anthocyanins; flavonoids; FTIR spectroscopy.

Practical Application: Determination of the best drying condition for the use of industrial residue.

1 Introduction

The food processing industry regularly generates a large amount of waste and by-products (Martins & Ferreira, 2017). In fruit processing, the residue volume can make up 20 to 50% of the processed fruits (Banerjee et al., 2017). In addition to the high volume, these residues have high humidity and microbial load; therefore, its improper treatment can cause serious environmental problems (Banerjee et al., 2017).

According to Martins & Ferreira (2017), the composition of fruit residues may have active compounds that are of interest in various applications. Moreover, the complete use of fruits by the industry can contribute to the reduction of waste in agribusiness and increase industrial profitability; the phytochemicals present can also be used by the food industry to assist in the stability and shelf life of food products (Ayala-Zavala et al., 2011). According to Kowalska et al. (2017) the food and pharmaceutical industries are interested in the use of compounds present in fruit by-products, as this can reduce the environmental impact of fruit waste and aid in the development of new products with different health benefits.

Camu-camu is a Brazilian fruit, consumed mainly in the north region of Brazil (Fujita et al., 2013), the different parts of which are rich sources of active compounds and have properties beneficial to health (Castro et al., 2018). Commercially, camu-camu was introduced to the world market starting in America and centers such as Europe and Asia, arousing the interest of local farmers in Brazil (Neves et al., 2015).

Camu-camu is used for the processing of juices, ice creams, concentrates, natural vitamin C nectar, and one of the main purposes is the production of drinks from the pulp, which corresponds to 50 - 55% of the fruit (Rodrigues et al., 2001). After processing camu-camu, the waste generated, consisting mainly of husks and seeds, which corresponds to 38 - 40% of the fruit (Rodrigues et al., 2001), can be a potential source of active compounds (Myoda et al., 2010).

The high content of active compounds in the camu-camu residue and their beneficial properties have been reported in the literature. Fidelis et al. (2020b) evaluated extracts (using different solvents) obtained from the seed of camu-camu and, under optimal conditions, found that the extract had different properties such as in vitro antioxidant activity and activity against cancer cells. Do Carmo et al. (2019) studied the hydroalcoholic extract obtained from camu-camu seeds and observed a high content of phenolic compounds, antioxidant activity, and cytotoxic effects against cancer cells; they also found it had antimutagenic potential. Fidelis et al. (2020a) reported a high content of phenolic compounds in the extract obtained from camu-camu seeds and reported its antioxidant, antimicrobial, anti-hypertensive, anti-hemolytic, and anti-hyperglycemic effects. Conceição et al. (2020) studied the active compounds present in the pulp and residue of camu-camu and observed a greater amount of phenolic compounds in the residue as compared to the pulp of the fruit.

Drying is one of the methods that can enable the use of residues from fruit processing. It can enable the use of various products, prolong the storage period, and reduce losses during the harvest (Nobrega et al., 2014). However, the drying process can have a significant impact on active compounds and other food components (Fujita et al., 2013). Thus, for the design, simulation,

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and optimization of the drying processes, data related to drying kinetics is of fundamental importance (Senadeera et al., 2003).

There are several studies on drying fruit residues. Silva et al. (2016) evaluated the effect of the drying process of acerola residue in different temperatures in a newly designed rotary dryer and reported considerable concentrations of phenolic compounds and flavonoids. Some authors have studied the drying of camu-camu. Azevêdo et al. (2014) studied the effect of drying (50 and 80 °C) on depulped camu-camu residue and, after the drying process, reported the presence of total anthocyanins, phenolics, and carotenoids. Fujita et al. (2013) evaluated the drying of camu-camu pulp in spouted bed drying and reported the presence of ascorbic acid, total phenolics, and proanthocyanidins, although reductions were observed as compared to fresh pulp. They also reported that the dry pulp showed antioxidant and antimicrobial activity in vitro. Silva et al. (2005) evaluated the drying of the camu-camu fruit (slices) in 50, 60, and 70 °C in a pilot scale vertical tray drier and observed superior retention of ascorbic acid in dried fruits at 50 °C. Studies of kinetics in residues obtained from the industrial processing of camu-camu are still incipient; however, they have great potential due to the antioxidant compounds present.

Thus, this paper aimed to study the drying temperature on the concentration of active compounds so as to use that on the active compounds (anthocyanins, flavonoids, carotenoids, and phenolic compounds). It also examines the chemical characterization of dry residues using the Fourier transform infrared (FTIR) technique and the physical-chemical properties (humidity, pH, Aw, color) of the residue from the industrial processing of camu-camu.

2 Materials and methods

2.1 Materials

The residue from the industrial processing of camu-camu for the production of camu-camu powders (resulting from the theft pulping operation, basically consisting of peels, seeds, and residual pulp) was supplied by the Doce Fruta industry (São Pedro do Turvo, SP, Brazil). The processed fruits used by the industry are the result of harvesting plantations located in Iguape (São Paulo, 24º42'29"S, 47º33'19"W) and Vitória do Xingu (Pará, 02º52'48"S, 52º00'36"W). The residue was fractionated, packed in plastic bags, and stored in a freezer until use. The following reagents were used: sodium carbonate, ethanol, chloridric acid, and hexane (Synth, SP, Brazil); quercetin, folin-ciocaulteu, gallic acid, and β-carotene (Sigma Life Science, St. Louis, Missouri); methanol (Dinâmica, SP, Brazil); aluminum chloride (Exodus scientific, SP, Brazil) and acetone (L. S. Chemicals, SP, Brazil).

2.2 Drying

Prior to the drying tests, residues from the industrial processing of camu-camu were thawed at room temperature (25 ± 3 °C, in the absence of light) for a period of 24 hours. Three dryings were carried out, on different days, in each of the temperatures evaluated, and in each of the dryings, two trays were used, thus totaling six points in each drying time. The drying tests were performed at temperatures of 40 °C (CCR40), 50 °C (CCR50), and 60 °C (CCR60), using an oven with forced ventilation (Marconi, MA 035/5, SP, BR). Aiming to achieve homogeneity of the mass and thickness of the material dried, the residues were dispersed (keeping the thickness of the layer constant at 6 mm) in aluminum trays that were 25 cm in diameter. The loss of mass of the system (tray + residue) as a function of time was recorded at intervals of 10 minutes during the first hour and 20 minutes in the remaining hours, until the system reached constant mass (mass change less than 0.1% in three consecutive weighings). The dry residues (dry to equilibrium humidity) were crushed (Mixer Oster, FPSTHB2610R-017, China) and standardized granulometry (16 mesh). The powders produced (CCR40, CCR50 and CCR60), were characterized.

2.3 Drying kinetics

For the evaluation of drying kinetics, the moisture ratio was determined according to Equation 1 as proposed by Akpinar et al. (2003).

$$RU = \frac{X - X_e}{X_i - X_e} \quad (1)$$

Where: RU = humidity ratio, X = humidity of the camu-camu residue at different drying times (g of water /g of residue), Xi = initial humidity of the camu-camu residue in natura (g of water /g of residue) and Xe = is equilibrium humidity of the dry residue of camu-camu (g of water /g of residue).

The experimental data (humidity ratio versus time) was adjusted to the parameters of the models listed in Table 1 using the Origin Pro software (2018).

The adjustments of the model parameters to the experimental data were evaluated considering the correlation coefficient (R^2), reduced chi-square values (x^2) Equation 2, and root mean square error (RMSE) Equation 3 as proposed by Sarsavadia et al. (1999).

$$X^2 = \frac{\sum_{i=1}^{n} (MR_{exp,i} - MR_{pre,i})^2}{N - n} \quad (2)$$

$$RMSE = \left[\frac{1}{N} \sum_{i=1}^{N} \left(MR_{exp,i} - MR_{pre,i} \right)^2 \right]^{1/2} \quad (3)$$

Where: $MR_{exp,i}$ = moisture ratio determined experimentally, $MR_{pre,i}$ = moisture ratio determined using the mathematical model, N = number of observations and n = number constant in the model.

2.4 Characterization of the residue from the industrial processing of camu-camu

pH

The pH values of fresh (after thawing) and dehydrated (CCR40, CCR50, and CCR60) residues were determined

Table 1. Mathematical models used to adjust the drying curves.

Model	Equation	Reference
Henderson Pabis	$RU = a\ \exp(-kt)$	Henderson & Pabis (1961)
Page	$RU = \exp\left(-kt^n\right)$	Page (1949)
Modified Page	$RU = \exp(-\left(kt\right)^n\right)$	Overhults et al. (1973)
Lewis	$RU = \exp(-kt)$	Lewis (1921)
Logarithmic	$RU = \mathrm{a}\exp(-kt) + c$	Yagcioglu et al. (1999)

Where: RU = humidity ratio, t = is drying time (min), k = drying constant (min^{-1}), a = model constant, c = model constant, n = model constants.

according to the AOAC methodology (Association of Official Analytical Chemistry, 2005). Residue samples (10 g) were dispersed in distilled water (100 mL, 25 ° C ± 0.1 °C) under agitation (30 min, shaker, Marconi, MA 420, SP, Brazil). After stirring, the dispersions were kept at rest for 10 min, and the pH was determined using a pH meter (WTW Com., WTW 3210, Weilheim, Germany).

Water content

The determination of the water content of the residues (in natura, CCR40, CCR50, and CCR60) was carried out according to the AOAC methodology (Association of Official Analytical Chemistry, 1998) at 105 °C (Fanem greenhouse, 515/4A, SP, Brazil).

Water activity

For the determination of water activity (in natura, CCR40, CCR50, and CCR60), Aqualab equipment (Decagon Devices, Pullman, WA, USA) was used.

Color parameters

Chroma (C*) and total color difference (ΔE*) were determined with a Miniscan XE colorimeter (hunterLab, Virginia, USA). For the realization, the D65 and 10° aperture were used as illuminants. The C* and ΔE* (the standard corresponds to the waste in natura) were determined using Equation 4 and Equation 5, respectively, according to ASTM D22-44 (American Society for Testing and Materials, 2005). For color analysis determination, samples were placed in a quartz crucible.

$$C^* = \sqrt{a^{*2} + b^{*2}} \tag{4}$$

$$\Delta E^* = \sqrt{\Delta a^{*2} + \Delta b^{*2} + \Delta L^{*2}} \tag{5}$$

Active compounds

The residues, in natura (after thawing, CCRIN) and dried at temperatures of 40 °C (CCR40), 50 °C (CCR50), and 60 °C (CCR60), were evaluated in terms of the total anthocyanins, carotenoids total, phenolic compounds, and flavonoids.

Anthocyanins

The determination of the concentration of anthocyanins was carried out as proposed by Francis (1982). The extraction of anthocyanins present in the residue from the industrial processing of camu-camu (in natura and dry) was carried out using the methodology proposed by Almeida et al. (2016) where the samples of the residues (1 g) were dispersed and homogenized (5000 rpm, Turrax T-25 Digital, IKA Labortechnick, Frankfurt, Germany) in ethanol solution (95%):HCl (1.5 mol/L) in the ratio 85:15 (30 mL, 2 minutes). Then, the solutions were diluted (50 mL) in 85 (ethanol):15 (HCl) solution, kept refrigerated in the absence of light for a period of 12 hours, and then filtered (Nalgon filter paper, 3 microns, 80 g/m2). The absorbances of the solutions were determined at 535 nm using Spectrum One spectrophotometer (Perkin-Elmer, Waltham, MA, USA), and the total anthocyanin content (mg/100 g) was determined according to Equation 6. as proposed by Fuleki & Francis (1968).

$$Total\ anthocyanins\left(\frac{mg}{100g}\right) = \frac{A\ Df}{98.2} \tag{6}$$

Where: A = absorbance (535 nm) and Df = dilution factor (5000).

Total carotenoids

The determination of the total carotenoid concentration was performed according to the proposal by Neves et al. (2015) To extract the carotenoids from the camu-camu residue in natura and subject them to drying at 40, 50, and 60 °C, samples of the residues (0.2 g) were dispersed in hexane:acetone solution (6:4, 10 mL) and homogenized for 1 minute (vortex shaker, IKA-V1, Germany). After a period of 9 minutes (rest), the solutions were filtered (Nalgon filter paper, 3 microns, 80 g/m^2). The absorbances of the extracts were determined at 450 nm (Spectrophotometer Spectrum One-Perkin-Elmer, Waltham, MA, USA). A standard β-carotene was used as an external standard (Sigma Life

Science, concentrations in the range 0 to 4.0 μg/mL), and the concentration of carotenoids was expressed in mg equivalent of β carotene/100 g on a dry basis.

Phenolic compounds

The phenolic compounds were extracted from the residues of camu-camu in natura and to those submitted to drying at 40, 50, and 60 °C, according to the proposal by Fracassetti et al. (2013). Thus, samples of the residues (1.0 g) were dispersed in methanol solution (50%, 25 mL), homogenized for 2 minutes (Vortex IKA-V1, Germany), and maintained in an ultrasonic bath (Unique, USC-1400A, Brazil) for a period of 15 min. The extract (supernatant) was obtained after centrifugation (Eppendorf centrifuge, 5430R, Germany, 15 min) at 3000 rpm at a temperature of 4 ° C. The total concentration of phenolic compounds was determined according to Singleton et al.(1998) using 0.5 mL aliquots of the extracts. The absorbance was determined at 740 nm (Spectrum One spectrophotometer, Perkin-Elmer, Waltham, MA, USA). To determine the concentration of phenolic compounds, gallic acid was used as an external standard (gallic acid concentrations in the calibration curve between 0 and 0.0640 mg gallic acid/mL), and the results were expressed in mg equivalent gallic acid (GAE)/100 g of extract on dry basis.

Flavonoids

The determination of flavonoids in residues from the industrial processing of camu-camu was carried out according to the methodology proposed by Neves et al. (2015). The samples of the fresh and dehydrated residues (0.5 g) were dispersed in methanol (20 mL) and homogenized (1 min, vortex, IKA-V1, Germany); they were then stored in the absence of light under refrigeration for a period of 24 hours. After this period, the dispersions were filtered (Nalgon filter paper, 3 microns, 80 g/m^2). To aliquots of the obtained extracts (3 mL), a 5% solution of aluminum chloride in methanol (2 mL) was added. The solutions were homogenized (vortex, IKA-V1, Germany) and kept at rest for 30 minutes in the absence of light. Then, the absorbance was determined at 441 nm (Spectrum One spectrophotometer, Perkin-Elmer, Waltham, MA, USA). Quercetin (concentrations between 2 and 20 μg/mL) was used as an external standard.

FTIR spectroscopy

The samples of the dry residues (temperatures of 40, 50, and 60 °C) were analyzed on an FTIR spectrometer (Vertex 70, Bruker-Germany) with an attenuated total reflectance accessory (ATR). Thirty-two scans (4 cm^{-1} resolution) were performed. Spectrum analyses (qualitative and quantitative) were conducted according to the steps described in Garcia et al. (2019), using the origin Pro 9 software.

Statistical analysis

For the characterization of residues (pH, water content, water activity, color parameters, anthocyanins, total carotenoids, phenolic compounds, and flavonoids), the analyses were performed in triplicate. The difference between the means (95% confidence interval) was determined by the Duncan test with the aid of the software SAS (Versão 9.2, SAS, Inc.).

3 Results and discussion

3.1 Drying kinetics

As reported in the literature, the moisture ratio of the residue from the industrial processing of camu-camu showed an exponential reduction in the temperatures evaluated due to the increase in drying time (Figure 1); similarly, the drying time decreased with the increase in temperature. Similar behavior was observed by Silva et al. (2005) when drying slices of the camu-camu fruit using a vertical tray dryer with an air speed of 1.5 m/s, at temperatures of 50, 60, and 70 °C; they observed, from the drying curves, a reduction in the moisture content in the first two hours of drying for all the temperatures studied, with the moisture ratio reduction being more intense at 70 ° C.

Considering the drying kinetics, it was found that the parameters of the evaluated models adjusted to all models (Figure 1), regardless of the drying temperature, since the values of the correlation coefficients (R^2) were in the 0.992 – 0.999 range (Table 2). Regarding the values of x^2 and RMSE, the lower these values, the better the adjustment of the model parameters to the experimental data (and the smaller the deviation between the values predicted by the model and the experimental data) (Avhad & Marchetti, 2016; Gunhan et al., 2005). Thus, in Table 2, it can be seen that the lowest values of x^2 and RMSE, at all temperatures studied, were observed for the Page model (Table 2).

Da Silva et al. (2005) also found that the parameters of the Page model fit the experimental data best, in studies of the drying kinetics of the camu-camu fruit (slices) submitted drying at temperatures of 50, 60 and 70 °C, using a tray dryer. Haas et al. (2017) in studies of the drying kinetics of purple grape residue, also found that the Page model was the most appropriate to represent the variation in moisture ratio as a function of drying time. Regarding the values of the constants in the Page model, the value of the constant "k" increased with the increase in temperature (Table 2). Other authors, such as Silva et al. (2005) report similar behavior regarding the effect of temperature on the value of the constant "k", attributing this increase to the speed of water diffusion in the material. Similarly, there was an increase in the value of the constant "n" (Page model) due to the increase in temperature. This was also observed by Haas et al. (2017).

3.2 Characterization of camu-camu industrial residue

pH

The pH values of the residue from the industrial processing of camu-camu in natura and after the drying process are in the range of 2.8 to 3.1, being classified as very acidic. According to Jay et al. (2005), in terms of acidity, food items can be classified as having low acidity (pH> 4.6), acidity (pH 3.7 - 4.0 to 4.6), and high acidity (pH <4.0 - 3.7). The reduced pH observed in some

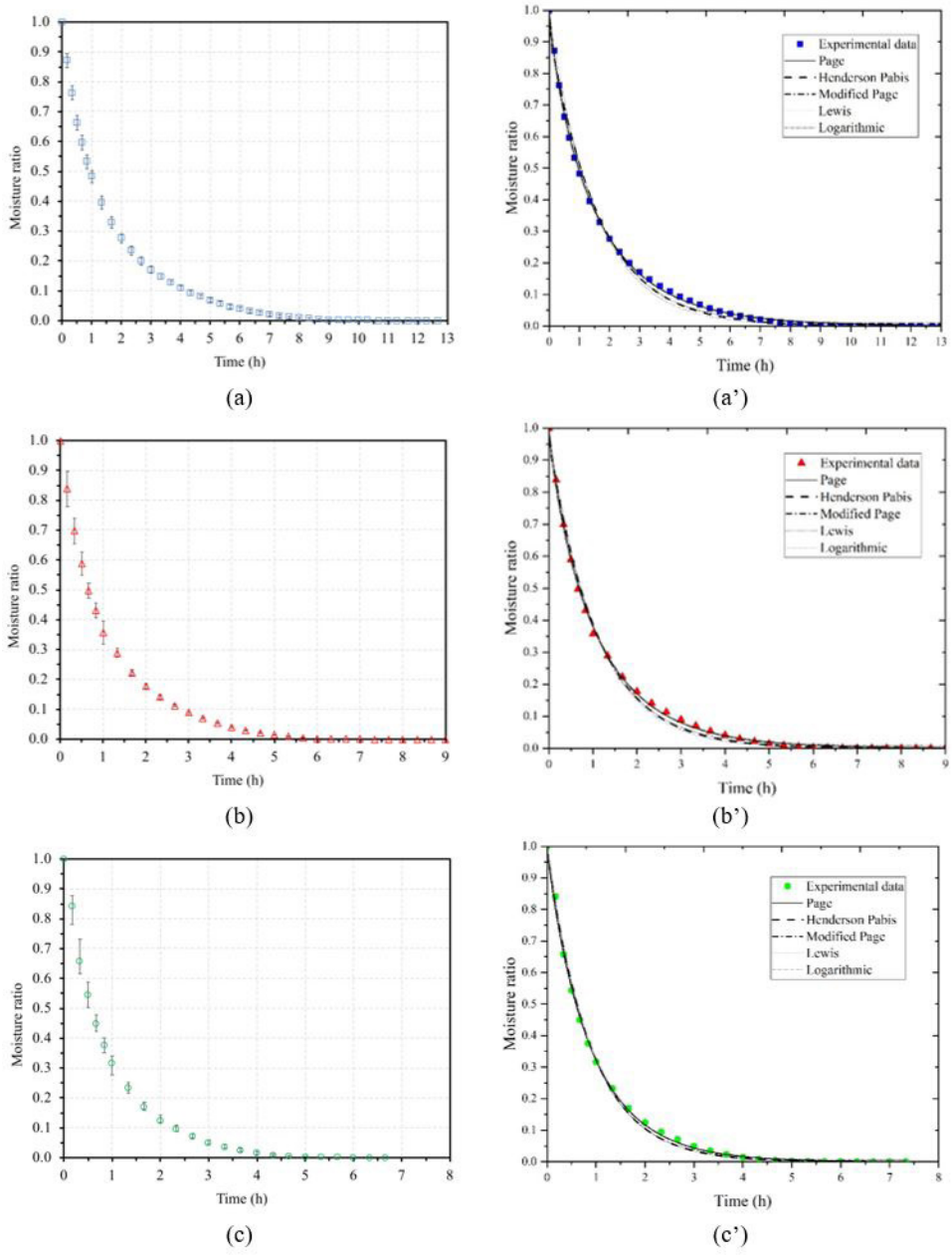

Figure 1. Moisture ratio as a function of drying time (hours) and adjustments of mathematical models (') for the residue from industrial processing of camu-camu at different temperatures: (a) 40 °C, (b) 50 °C and (c) 60 °C.

fruits and vegetables may be related to the organic acids present, which, in addition to contributing to their characteristic flavor, may be responsible for the reduction of pH values (Nawirska-Olszańska et al., 2014).

In general, the drying process caused a small reduction in the pH values of the dry residues when compared to the in natura residue, which may be related to the higher concentration of organic acids after the removal of water in the drying process, as observed by Rolle et al. (2012) in dehydrated grapes.

Azevêdo et al. (2014) reported that the pH of the camu-camu residue in natura and dehydrated (temperatures of 50 ° C and 80 °C) varied from 4.2 (in natura) to 3.3 (50 °C) and 3.8 (80 °C). Other works report that the pH of the camu-camu pulp is approximately 2.5 (Maeda et al., 2007). This pH difference may be related to the use of a heterogeneous material from fruits with different degrees of maturation.

Water content

As expected, the drying process reduced the water content of the waste by more than 80% (Table 3). On the other hand, the increase in the drying temperature caused a significant reduction in the water content, indicating a higher removal of free water.

Table 2. Values of the drying constant (*k*), constant of the models (*a, c, n*), correlation coefficient (R^2), reduced chi-square (x^2) and root mean square error (RMSE), of the different mathematical models adjusted to the data experiments on the drying kinetics of the industrial residue of camu-camu submitted to drying at different temperatures (40, 50 and 60 °C).

Model	Constants	Temperatures (°C)		
		40	50	60
Handerson Pabis	*k*	0.60554	0.90867	1.11766
	a	0.93929	0.96287	0.98561
	R^2	0.995	0.996	0.997
	x^2	4.944E-6	7.971E-4	3.980E-3
	RMSE	0.0182	0.0700	0.1090
Page	*k*	0.71587	0.96743	1.13103
	n	0.83988	0.87316	0.92405
	R^2	0.999	0.999	0.999
	x^2	8.032E-8	6.405E-4	3.406E-3
	RMSE	0.0065	0.0663	0.1049
Modified Page	*k*	0.67167	0.96279	1.14252
	n	0.83989	0.87316	0.92406
	R^2	0.999	0.999	0.999
	x^2	1.766E-6	1.412E-3	5.941E-3
	RMSE	0.0141	0.0808	0.1205
Lewis	*k*	0.65752	0.95425	1.1378
	R^2	0.992	0.995	0.998
	x^2	1.325E-5	8.261E-4	3.933E-3
	RMSE	0.0234	0.0713	0.1098
Logarithmic	*k*	0.62699	0.93655	1.13874
	a	0.93712	0.96014	0.98323
	c	0.00833	0.00801	0.00549
	R^2	0.995	0.996	0.998
	x^2	3.940E-6	7.297E-4	3.645E-3
	RMSE	0.0173	0.0691	0.1078

Where: *k* = drying constant (min^{-1}), *a* = model constant, *c* = model constant, *n* = model constant.

Table 3. Water content (W_c) and water activity at the equilibrium humidity of the industrial residue of dry camu-camu in natura (CCRIN) and drying at temperatures of 40 (CCR40), 50 (CCR50) and 60 °C (CCR60).

Sample residue	W_c (g/100 g dry residue)	Water activity
CCRIN	83.82 ± 0.16[a]	0.989 ± 0.001[a]
CCR40	9.42 ± 0.06[b]	0.279 ± 0.004[b]
CCR50	8.47 ± 0.10[c]	0.213 ± 0.002[c]
CCR60	6.54 ± 0.33[d]	0.184 ± 0.002[d]

Lowercase letters in the same column indicate significant differences ($p < 0.05$).

Azevêdo et al. (2014) also reported a significant reduction in the moisture levels of fresh camu-camu residue (from 86.0 to 5.9%) dried at 50 and 80 °C.

Water activity

Camu-camu residues that were dried at higher temperatures showed lower water activity (Table 3). On the other hand, in the studied temperature range, the activity values were lower than 0.3, which is a desirable characteristic. Azevêdo et al. (2014) who evaluated the effect of drying the residue of camu-camu pulp at temperatures of 50 and 80 °C, observed water activity values of 0.268 and 0.183, respectively.

Color parameters

The drying process caused an increase in the L*, a*, and b* parameters of the dry residues as compared to the in natura residue (Table 4). Higher values of luminosity (L*) were observed in the dry residues (CCR40, CCR50, and CCR60) than the waste in natura (CCRIN). Similar results were observed in the study of drying dried camu-camu residue at temperatures of 50 and 80 °C conducted by Azevêdo et al. (2014). Horuz et al. (2017) also observed that L* values for cherry dried in a conventional oven (50, 60 and 70 °C) were higher than that of fresh fruit.

The higher values of the parameters a* and b* of the dry residues indicate a more intense reddish color. Stamenković et al.

(2019) also observed an increase in the values of a* and b* in raspberry convection drying; according to these authors, an increase in the values a* and b* is positive and indicates a more saturated color.

For C*, a significant increase was observed in the dry samples, as compared to the fresh sample, at different temperatures. As this parameter is related to color intensity, the results indicate that the drying process caused an increase in color intensity, which is possibly related to the concentration of pigments present in the residue. On the other hand, the increase in temperature did not significantly affect the C* values, indicating that the temperature range studied probably did not favor the degradation of the pigments present in the residue.

The results obtained for the color difference (ΔE*) indicate that the dry residue at different temperatures showed different colors (ΔE > 3) from the in natura residue. According to Adekunte et al. (2010), the color difference can be classified as very distinct (ΔE > 3), distinct (1.5 < ΔE < 3), and a little distinct (ΔE < 1.5).

3.3 Active compounds

Anthocyanins

There was a significant reduction in the concentration of anthocyanins (Table 5) of the dried residues as compared to the residue from the industrial processing of fresh camu-camu. According to Markakis & Jurd (1974) the rate of degradation of the anthocyanins increases with the increase in temperature, and therefore, the reduction in the concentration of anthocyanins in the dry residues was expected. Azevêdo et al. (2014) also reported a reduction in the anthocyanin content of the dry camu-camu residue compared to in natura. The anthocyanin content of the waste in natura was similar to that observed by Fracassetti et al. (2013) for camu-camu powder pulp (19.63 mg/100 g) and lower than that observed by Neves et al. (2015) and Zanatta et al. (2005) for camu-camu bark (24 and 30 mg/100 g, respectively).

However, it was found that the residues dried at a higher temperature (Table 5; CCR50 and CCR60) showed higher concentrations of anthocyanins, indicating that the drying time also had an influence on the concentration of anthocyanins. Haas et al. (2017) also observed a higher concentration of anthocyanins in dry grape residue at higher temperatures due to the reduction in the time needed for drying and, according to the authors, the drying process improves the stability of the product during storage; however, the drying time may also affect the concentration of active compounds in the product.

We must also consider the heterogeneity of the residue from the industrial processing, i.e., the composition of its bark (different degrees of maturity), seeds, and residual pulp, all of which can imply inherent variations in the concentration of anthocyanins since they are found in different concentrations in different parts of the fruit. According to Maeda et al. (2006) anthocyanins are predominantly present in camu-camu bark (181.38 mg/100 g anthocyanins) and are also responsible for the formation of its characteristic color (Gonçalves et al., 2010).

Total carotenoids

A ~50% reduction was observed (Table 5) in the total carotenoid content of the waste in natura in comparison with the dry residue at different temperatures; possibly, this reduction is related to the enzymatic oxidation and thermal degradation of the carotenoids. According to Rodriguez-Amaya et al. (2008) high temperatures and greater exposure to sunlight increase carotenogenesis in fruits and may also promote carotenoid

Table 4. Brightness (L*), chroma a*, chroma b*, chroma C* and color difference (ΔE*), from the residue of the industrial processing of camu-camu in natura (CCRIN) and after drying at temperatures of 40 (CCR40), 50 (CCR50) and 60 °C (CCR60).

Residue	L*	a*	b*	C*	ΔE*
CCRIN	51.80 ± 0.02^{a}	11.22 ± 0.03^{a}	23.14 ± 0.07^{a}	25.72 ± 0.07^{a}	NC
CCR40	59.19 ± 0.17^{b}	11.74 ± 0.02^{b}	25.96 ± 0.09^{b}	28.49 ± 0.09^{b}	7.92 ± 0.16^{a}
CCR50	56.59 ± 0.20^{c}	12.11 ± 0.09^{c}	26.77 ± 1.76^{b}	29.38 ± 1.65^{b}	6.18 ± 1.03^{b}
CCR60	59.28 ± 0.09^{b}	11.73 ± 0.05^{b}	25.18 ± 0.04^{b}	27.78 ± 0.06^{b}	7.77 ± 0.07^{a}

Lower case letters in the same column indicate significant differences ($p < 0.05$). NC: not calculated

Table 5. Concentrations of anthocyanins, carotenoids, phenolic compounds and flavonoids from the residue of industrial processing of camu-camu in natura (CCRIN) and subjected to drying at temperatures of 40 (CCR40), 50 (CCR50) and 60 °C (CCR60).

Sample	Anthocyanins (mg/100 g DB)	Carotenoids (mg equivalent β carotene/100 g DB)	Phenolic compounds (mg GAE /100 g DB)	Flavonoids (mg quercetin equivalent /100 g DB)
CCRIN	21.10 ± 0.94^{a}	14.34 ± 0.72^{a}	10011.06 ± 585.26^{a}	32.28 ± 3.13^{a}
CCR40	12.60 ± 0.19^{b}	7.57 ± 0.62^{b}	3673.26 ± 122.30^{b}	17.93 ± 1.38^{b}
CCR50	14.20 ± 0.28^{c}	7.56 ± 0.39^{b}	4341.36 ± 183.84^{c}	20.56 ± 2.90^{b}
CCR60	16.35 ± 0.44^{d}	7.13 ± 0.14^{b}	5691.35 ± 254.70^{d}	27.20 ± 2.49^{c}

GAE: gallic acid equivalent; DB: dry basis. Lower case letters in the same column indicate significant differences ($p < 0.05$).

photodegradation; however, degradation of these compounds may occur due to increased surface area or porosity, alteration of the cell structure, processing, and the temperature conditions in storage.

However, regardless of the temperature used, no significant differences were observed in the total carotenoid content of the dry residue. The same effect was observed by Azevêdo et al. (2014) who reported values of 5.69, 1.08, and 0.74 mg/100 g DW for the camu-camu residue in natura, dried at 50 °C, and dried at 80 °C, respectively, indicating that the main cause of the degradation of carotenoids is oxidation, enzymatically or not enzymatically. According to Rodriguez-Amaya et al. (2008) carotenoid composition can vary depending on factors such as cultivar or variety, harvest maturity, climate or geographical location, season, part of the plant used, conditions during agricultural production, post-harvest handling, processing, and storage conditions.

Phenolic compounds

When comparing the values of the concentration of phenolic compounds in the camu-camu residue in natura with those dried at different temperatures, a significant reduction ($p < 0.05$) was observed (Table 5), possibly due to the degree of oxygen exposure and the thermal deterioration of the phenolic compounds. Wojdyło et al. (2014) carried out the vacuum drying of cherry at different temperatures and found that prolonged exposure to temperature can result in irreversible oxidative processes, causing the thermal degradation of phenolic compounds. Azevêdo et al. (2014) also observed that drying temperature had an impact on the content of active compounds in the dry camu-camu residue at 80 °C a reduction of 63.9% was observed in the content of phenolic compounds as compared to the fresh residue.

On the other hand, there was an increase in the concentration of phenolic compounds in the waste dried in increasing temperatures (Table 5). The observed results are possibly related to the drying time, i.e., the amount of time the residue was exposed to the drying temperature. However, the temperature range used in the drying processes is also an important parameter in the concentration of phenolic compounds. Azevêdo et al. (2014) verified the following values regarding the concentration of phenolic compounds: 3738.0 mg GAE/100 g for fresh camu-camu residue, 1843.6 mg GAE/100 g for residue dried at 50 °C, and 1349.4 mg GAE/100 g for residue dried at 80 °C.

Flavonoids

The flavonoid content of the residue was higher in natura than in the dry residue; on the other hand, similar to the phenolic compounds, the residues dried at higher temperatures present higher concentrations of flavonoids, and the highest concentration was observed for the residue dried at 60 °C, which is possibly related to the long drying time at lower temperatures.

According to Erbay & Icier (2009) the drying temperature is not the only factor that can cause damage to the active compounds; the heat treatment time, the degree of ripeness, and the different parts of the fruit all influence the content of active compounds in the material. In accordance to Genovese et al. (2008) flavonoids comprise a group of phenolic compounds widely distributed in plants, and their distribution depends on several factors, including variety and degree of exposure to light. The authors also observed that the flavonoids present in greater quantity in camu-camu are anthocyanins. Chirinos et al. (2010) evaluated the antioxidant capacity of the edible portion of the camu-camu fruit (the peel and the flesh) at different stages of maturation (full green and green–reddish and red) and reported that after ripening, the total flavonoids increased from 7.7 to 13.3 mg QE/100 g FW, indicating that the antioxidant potential of camu-camu is not only a result of the high concentration of ascorbic acid but also its phenolic content, including flavonoids.

Chemical characterization of dry residue by FTIR Spectroscopy

On analyzing the spectra in the IR region of the dried residues (Figure 2a), bands were found in the region between 1200 and 900 cm^{-1}, predominantly characteristic of the C-O-C and angular C-O-C and C-H stretch vibrations of structural polysaccharides, such as cellulose, hemicellulose, lignin, and pectin (Türker-Kaya & Huck, 2017; Xu & Wang, 2015). Due to this, the intensity of the spectra was normalized by the intensity of the band at approximately 1000 cm^{-1}.

Abbas et al. (2017), in a study using midi-infrared in patterns of phenolic compounds, found that the bands in the region between 2500 and 1800 cm^{-1} are related only to the absorption bands referring to CO_2, without complementary data on the chemical composition of the samples. Thus, in order to obtain qualitative and quantitative information on the main components of the waste, deconvolution and adjustment of the absorption signals ("curve-fitting") were carried out in the regions from 3700 to 2400 cm^{-1} (Figure 2b) and from 800 to 1800 cm^{-1} (Figure 2c). Thus, as seen in Table 6, by specifying the spectral ranges of 3700 to 2400 cm^{-1} and 800 to 1800 cm^{-1}, the area values of the absorption peaks present in the spectra FTIR of the residues dried different temperatures (CCR40, CCR50, and CCR60) as well as the possible vibrations responsible for each signal were obtained.

Using the area of the bands of the CCR40 residue as a reference, a reduction of the area of 4.10 and 4.70% can be seen for the CCR50 and CCR60 samples, respectively. A small decrease in absorption occurred in nearly the entire spectral region of the CCR50 and CCR60 residues, indicating a reduction in a range of volatile components with an increase in drying temperature. An example of this reduction is found in Table 5, which shows the decrease in the concentration of β-carotene in samples subjected to higher drying temperatures. Moreover, the drying process also removes the layer of free water on the surface of the samples; however, a small amount that is bound more strongly may remain. The residues dried at temperatures of 50 and 60 °C showed lower values of water activity (Table 3), indicating a reduction in the amount of water present.

Quijano & Pino (2007) in studies using different extraction methods, identified about 138 volatile compounds in camu-camu; the authors reported that the most abundant were limonene and α-pinene. The volatilization of components, such as alcohols,

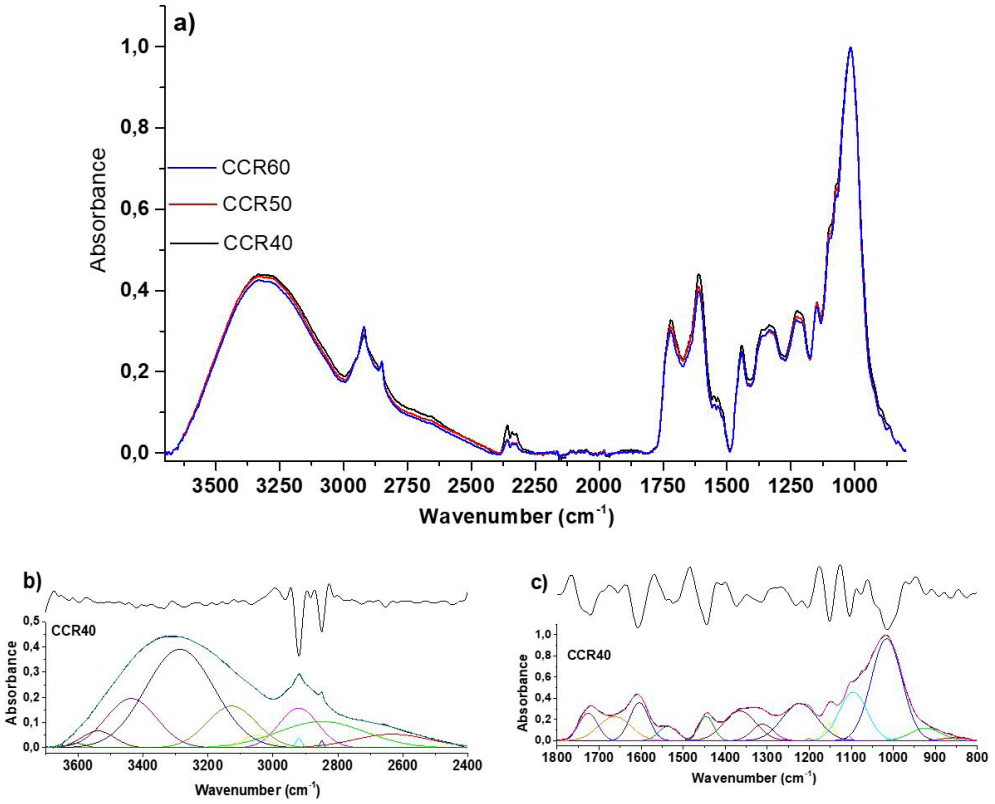

Figure 2. Spectra in the IR region of residues from industrial processing of camu-camu subjected to drying at 40 (CCR40), 50 (CCR50) and 60 °C (CCR60): a) scanning in the 4000 to 400 cm^{-1} region. b) spectrum with second derivative and analysis of signal adjustment ("fitting") of the absorbance bands of the CCR40 sample.

ketones, acids, and terpenes, along with the decrease in free and bound water contribute to the reduction observed in the absorption bands in the FTIR spectra.

The FTIR spectra of the camu-camu residues show signs characteristic of the biomolecules that make up a biological sample (Rana et al., 2018; Szymanska-Chargot et al., 2015; Szymanska-Chargot & Zdunek, 2013; Thumanu et al., 2015) with the absorption vibrations attributed to the macromolecules (proteins, polysaccharides, lipids, and nucleic acids) predominating. In the spectral region ranging between 3700 and 2400 cm^{-1}, the bands are mainly related to the vibrational stretch bands (ν) O-H and N-H (connected or free) between 3700 and 3100 cm^{-1} and C-H between 3100 and 2800 cm^{-1}. The vibrations of ν(O-H and N-H) links (hydrogen bonds) result in a wide and intense band normally associated with proteins, nucleic acids, and carbohydrates and, to a lesser extent, with alcohols, acids, esters, water, and phenolic compounds, among others (Largo-Gosens et al., 2014; Wilson et al., 2000). The reduction of moisture and water activity in the residues due to the increase in the drying temperature (Table 3) results in the reduction of free and bound water in hydrogen bonds. This reduction in humidity also contributes to the reduction of the broad band centered in the 3300 cm^{-1} region in the FTR spectra of CCR50 and CCR60 (Figure 2).

The bands in the region between 3100 and 2800 cm^{-1} are related to symmetric and asymmetric ν(C-H) vibrational modes, which are attributed mainly to lipids and, in some cases, proteins, carbohydrates, and nucleic acids (Ammawath & Man, 2010; Türker-Kaya & Huck, 2017; Xu & Wang, 2015). On the other hand, according to Quijano & Pino (2007), limonene and α-pinene are the most abundant volatile compounds determined in the fruit of camu-camu, and these terpenes show signs of being abundant in the region of ν(C-H). The reduction in carotenoids (Table 5) as well as terpenes (Quijano & Pino, 2007) also contribute to the decrease of ν(C-H) signs. The spectral region between 1800 and 800 cm^{-1} contains the largest amount of information about the analyzed sample and is therefore called the fingerprint region (Abbas et al., 2017; Türker-Kaya & Huck, 2017).

The band observed at 1724 cm^{-1} is related to the vibrational way of stretching the C = O bond of esters present in phospholipids, cellulose, pectin, and hemicellulose (Türker-Kaya & Huck, 2017; Xu et al., 2013; Xu & Wang, 2015). Phenolic acids and ascorbic acid also show signs of C = O around 1660 cm^{-1} as reported by Panicker et al. (2006) and Yang & Irudayaraj (2002).

The reduction of the band areas in the region from 1800 to 800 cm^{-1} is also due to the volatilization of smaller compounds, such as essential oils, that show characteristic signs of δ(C = O in ~1730 cm^{-1}, ν(C-H) at ~1446 cm^{-1}, ν(C-C) at ~1097 cm^{-1}, and ν(C-O) at ~1151 cm^{-1} as reported by Micu et al. (2015).

Table 6. Area values and assignment of the absorption bands present in the FTIR spectra of the CCR40, CCR50 and CCR60 samples.

Region (cm^{-1})	CCR40	CCR50	CCR60	Assignment	
	Area	Area	Area	Functional Group	Substance
893 a 875	7.62	5.05	5.03	ν(N-H); ν(C-O)	Protein; glycosidic linkage of hemicellulose; (Xu & Wang, 2015)
936 a 926	5.06	4.78	4.34	δ(C-H) e (O-H)	Cellulose, hemicellulose; (Xu & Wang, 2015)
1015 a 1023	86.06	82.48	88.91	ν(C-O); ν(C-C); δ(O-C-H)	Cellulose, hemicellulose; lignin; (Xu & Wang, 2015) pectin; glycogen; (Largo-Gosens et al., 2014)
1096 a 1091	40.76	43.43	35.97	ν_a(C-O-C) $\nu_s(PO_2^-)$	Cellulose, hemicellulose; lignin;[51] pectin; glycogen;[59] essential oils[66] Polysaccharides; nucleic acids;
1106 a 1104	0.67	0.54	0.54	ν_s(P-O-C); ν_s(C-O-C)	Carbohydrates; (Largo-Gosens et al., 2014)
~1151	4.78	4.81	4.95	ν_a(C-O-C)	Cellulose, hemicellulose;[51] essential oils;[60]
~1200	0.33	0.32	0.24	δ(O-H)	Cellulose, hemicellulose; (Xu & Wang, 2015)
~1222	36.78	35.09	36.32	ν(C-O) ν(C=N e N-H)	lignin; Xylan;[52] phenolic compounds; (Abbas et al., 2017) Protein Amide IV;
~1310	10.23	9.01	7.86	ν(C-N)	Protein Amide III; (Abbas et al., 2017)
1366 a 1362	26.66	26.08	26.66	δC-H)	Cellulose and hemicellulose; (Türker-Kaya & Huck, 2017)
~1446	10.08	9.23	9.46	ν(C-C) de aromático; $\delta_a(CH_2)$; $\delta_a(CH_3)$	Phenolic compounds; polysaccharides; lipids and proteins;[52] essential oils;[60]
~1536	7.48	6.23	6.44	ν(C = N); νN-H)	Protein Amide II; (Türker-Kaya & Huck, 2017)
~1604	20.46	18.97	18.65	ν(C = O) de aromáticos; ν(C=C) de aromáticos;	Lignin and alkaloid; (Türker-Kaya & Huck, 2017) Phenolic compounds;[53, 60]
1667 a 1663	21.99	21.40	20.36	ν(C = O);	Protein amide I; alkaloid; pectin, water associated with cellulose;[52] or lignin;[51, 52]
~1726	14.62	13.73	12.81	ν(C = O)	Phospholipid; pectin; lignin; cutin;[52, 60] Hemicellulose;[51, 52] flavonoids; essential oils;[60]
2659 a 2630	15.68	12.83	15.12	ν(N-H) de NH_3^+	Mainly proteins; (Abbas et al., 2017)
3058 a 2849	64.67	62.18	59.04	ν_s(C-H); ν_a(C-H)	Mainly lipids and less contribution of carbohydrates, proteins and acids. nucleic acids;[52] lignin;[51] wax;[60] essential oils[60]
3635 a 3127	182.52	177.50	177.59	ν(O-H) e ν(N-H) associada e livre	Water; lignin;[51] carbohydrates, proteins (amide A and B); nucleic acids;[53] alcohols and phenolic compounds;[52] cutin;[60] alcohols and volatile acids
$Total_{area}$ =	**556.45**	**533.66**	**530.29**		
% $Total_{area}$ =	**100**	**95.90**	**95.30**		
% Reduced $total_{area}$ =	**0**	**4.10**	**4.70**		

ν = stretch vibration; δ = angular vibration; a = asymmetric; s = symmetr.

4 Conclusion

It was found that the Page model is most suited to represent the variation in the drying kinetics of the camu-camu residue in the studied temperature range. The dry residue had low humidity and water activity, contributing to delayed deterioration reactions and increased shelf life. On the other hand, the results indicate that even after the drying process, the residues contained active compounds (phenolic compounds, flavonoids, anthocyanins, and carotenoids). The residues submitted to drying at 60 °C generally presented higher values due to the shorter drying time and the consequent exposure of the material to conditions favorable to their degradation. Therefore, the use of this industry by-product is a promising option for obtaining products with functional properties and can contribute to reducing the environmental impact of fruit waste.

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