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Low-pressure conductive thin film drying of açaí pulp

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ABSTRACT

Açaí has a high content of anthocyanins and unsaturated fatty acids, and drying parameters can affect the stability of these compounds. This study used a conductive thin-film dryer at a laboratory scale to mimic the (vacuum) drum drying of açaí pulp. The effect of drying chamber pressure on the drying kinetics and powdered product characteristics was investigated. Hot air drying and freeze-drying experiments were performed as references. A pilot-scale conductive dryer, namely a vacuum drum dryer, was used as well to dry açaí pulp. Conductive drying at low pressures led to lower boiling temperatures and shorter drying times but also more vigorous bubble formation. The anthocyanin content and antioxidant activity of contact-dried samples at low pressures were comparable to those of freeze-dried and significantly higher than those of hot air-dried. However, conductive drying caused greater lipid oxidation compared to freeze-drying and hot air drying. Oil loss was observed during vacuum drum drying except for the lowest pressure and the shortest drying time. Therefore, the results suggest that chamber pressure during conductive thin film drying should be carefully selected to obtain high-quality açaí powder.

1. Introduction

Drying is one of the widely applied processing methods in the food industry to reduce losses and extend the shelf life of products. Drying also facilitates the storage and transportation of foods by decreasing product weight and volume. In addition, it provides more product options for consumers. The quality of the dried product and the energy efficiency of the drying method are the two main concerns in the food drying process (Kumar, Karim, & Joardder, 2014). Hot air drying (HAD) is the most used technique for dehydrating fruits and vegetables because of its simplicity and low energy consumption. Nevertheless, this drying process results in nutritional and sensorial damage to the product due to the high air temperatures and long drying times. In contrast, freeze-drying (FD) has been found a very suitable drying method for producing high-quality dried fruits and vegetables since the process is conducted at low temperatures under a vacuum. Unfortunately, FD is a high-cost process because of the long drying time and high energy consumption, restricting its use on the industrial scale (Ratti, 2001; Xu, Zhang, Mujumdar, Duan, & Jin-cai, 2006). Hence, alternative drying techniques have been investigated to decrease drying time, increase energy efficiency, and still retain product quality as much as possible.

Conductive thin-film drying (CTFD) can potentially be used to produce high-quality dried products of fruits and vegetables with short drying times (Qiu, Acharya, Jacobs, Boom, & Schutyser, 2019; Simão et al., 2021). It is an energy-efficient process, with an energy consumption comparable to HAD and around half of FD (Bazyma et al., 2006). During CTFD, thermal energy is transferred mainly by conduction from hot water or steam condensation to a thin film of product spread over a flexible support or steel wall. Conductive drying may be done by refractance window drying or cast-tape drying employing mild drying conditions, i.e., heating medium with a temperature just below boiling point (usually at 90–98 °C) at atmospheric pressure and relatively short drying times (few minutes), which can be adequate for thermosensitive foods (Simão, Moraes, Carciofi, & Laurindo, 2020). Another option is atmospheric drum drying. One disadvantage of this drying method is that the product is exposed to high drying temperatures (commonly at 120–170 °C), resulting in quality loss. Therefore, drum drying is sometimes carried out under reduced pressure (vacuum drum drying), alleviating the thermal damage thanks to the lower boiling temperature, while lower oxygen concentration also can be an advantage (Qiu, Acharya, et al., 2019).

Açaí (*Euterpe oleracea* Mart.) is a Brazilian fruit with a high

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nutritional value, being rich in fibers, unsaturated fatty acids, and anthocyanins with high antioxidant capacity (Schauss et al., 2006). The interest in the consumption of açaí has grown worldwide due to the exotic flavor and benefits for human health, such as antioxidant, anti-proliferative, anti-inflammatory, and cardioprotective properties (Pacheco-Palencia, Duncan, & Talcott, 2009; Yamaguchi, Pereira, Lamarão, Lima, & Veiga-Junior, 2015). However, fresh açaí is highly perishable. Therefore, the drying of açaí pulp to produce açaí powder is an attractive alternative to prolong its shelf life. Furthermore, açaí powder may extend the consumption and commercialization of the fruit. The powdered product can be used as an ingredient in healthy drinks, yogurt, ice cream, baked goods, baby foods, desserts, and cereal bars. Pavan (2010, p. 88) compared the influence of refractance window drying, hot air drying, and freeze-drying on the quality attributes of açaí powder. Nevertheless, no systematic studies have been conducted on the CTFD of açaí pulp under reduced pressures to the best of our knowledge.

In this context, this study aimed to evaluate the effect of drying pressure on the quality of açaí powder produced by CTFD. The hypothesis was that drying under lower pressures would lead to a higher quality product. For comparison, reference açaí powders were also produced by HAD and FD. The product characterization was based on physical properties (moisture content, water activity, lipid content, and color) and quality markers (total monomeric anthocyanin content, antioxidant activity measured by DPPH assay, and lipid oxidation products quantified by ^{1}H NMR). Contact drying of açaí pulp was conducted at the laboratory and pilot scale.

2. Material and methods

2.1. Açaí pulp

Frozen açaí pulp bars (Açaí Diamante Negro, Pará, Brazil) were purchased from a local market in Amsterdam, the Netherlands. The total solids content of the pulp was 14.1 ± 0.3 g·100 g^{-1}, total soluble solids of 5.1 ± 0.2 °Brix, and pH 4.9 ± 0.1. Açaí pulp bars were kept frozen at −20 °C. For each test, a required amount of sample was taken from the frozen bar and thawed at room temperature until its temperature reached 10 °C.

2.2. Drying experiments

Three different drying methods were used to dry açaí pulp: conductive thin-film drying at the laboratory- and pilot-scale, hot air drying, and freeze-drying. Except for the contact drying at the pilot scale (vacuum drum drying), the samples were spread with a thickness of 0.5 mm over the drying supports, which was the minimum thickness possible to apply. The drying processes were stopped when the moisture content of dried açaí samples was below 5% (wet basis), following Brazilian legislation (MAPA–Ministério da Agricultura & Pecuáriae Abastecimento, 2018). Then, the dried samples were collected and stored in the freezer for further analysis.

2.2.1. Conductive thin-film drying (CTFD) at lab-scale

A custom-built experimental conductive dryer was used to mimic the (vacuum) drum drying at a laboratory scale, as described in Qiu, Kloosterboer, Guo, Boom, and Schutyser (2019). This device consists of a drying chamber equipped with an analytical balance (Sartorius, Cubis MSE, Germany) and two temperature sensors (National Instruments™, K-Type, the Netherlands), allowing simultaneous online monitoring of mass and temperatures during the drying process. The sample is uniformly spread on a steel plate (with a surface area of 0.0163 m^2), which is heated with an induction coil. Even though this system does not precisely mimic the conditions during the drum drying due to the difference in heat supply (our system operates at constant heat flux and drum drying at a constant temperature), it allows a better understanding of the conductive drying behavior and the effect of drying on product quality.

Films of açaí pulp were formed by carefully spreading 8.2 mL of the sample over the plate surface that was first coated with a vegetable oil-based spray (Dr. Oetker, Baking spray, Germany). The spray was applied to prevent pulp browning due to contact with the steel plate (Santos, Azevedo, Bastos, & Carvalho, 2016) and adhesion of the dehydrated pulp layer to the plate. Films were dried at a constant power input of 1300 W (heat flux of 80 kW m^{-2}), chosen based on preliminary experiments (data not shown). Three different chamber pressures (415, 700, and 1013 mbar) were used, for which the pure water boiling temperatures are 77, 90, and 100 °C, respectively. Mass and temperatures were recorded every second. The obtained data were used to calculate the evaporation rate (g_{water} s^{-1} m^{-2}). The experiments were performed in triplicate.

Table 1
Drying conditions used for dehydration of açaí pulp in the vacuum drum dryer.

Parameters	Drying conditions		
	1	2	3
Chamber pressure (mbar)	140	140	200
Wall temperature (°C)	100	100	100
Residence time (s)	30	60	60
Chamber temperature (°C)		65	
Film thickness (μm)		≈75	

Note: chamber temperature and film thickness were kept the same for all three drying conditions. Film thickness was estimated from the gap between the two drums.

2.2.2. Hot air drying (HAD) and freeze-drying (FD)

Açaí pulp was spread on polystyrene trays and dried by HAD and FD. These drying processes were considered referential cases for the study. The HAD was performed in a convective oven (Heraeus, Function Line T6, Germany) at 60 °C, with a relative humidity of 8%, for 2.5 h. The FD was conducted according to the following steps: first, the spread açaí pulp was frozen at −20 °C and then transferred into the freeze-dryer (Martin Christ, Epsilon 2-10D LSCplus, Germany). The chamber pressure was reduced to 1 mbar, at a shelf temperature of −20 °C, for 2 h. After this, the shelf temperature was increased to −10 °C and maintained at this temperature for 4 h. Subsequently, the shelf temperature was increased to 0 °C, which was maintained for 4 h. Finally, the shelf temperature was increased and kept at 20 °C for 1.5 h, the pressure was decreased to 0.01 mbar and maintained at this level for 1.5 h. This last drying step lasted for 11 h, and the total drying time was 29 h. All drying experiments were conducted in duplicate.

2.2.3. Vacuum drum drying (VDD)

A pilot-scale vacuum drum dryer (ANDRITZ Gouda, Waddinxveen, the Netherlands) was used for drying açaí pulp. The device consisted of two hollow steel chromium-plated drums with 0.20 m external diameter and 0.48 m length enclosed in a vacuum chamber. The chamber was maintained at 65 °C to avoid vapor condensation and was operated under two reduced pressures (140 and 200 mbar), resulting in pure water boiling temperatures of 52 and 60 °C, respectively. The drums were internally heated by steam with a pressure of 1.0 bar, which provided a hot wall temperature of 100 °C. The rotation speeds of drums were fixed at 0.5 and 1 rpm, giving residence times of 30 and 60 s, respectively. The sample was poured manually and fed evenly between the two drums into a hot feeding pool. The clearance between the two drums was fixed at 0.15 mm, allowing the formation of a thin layer adhered to the drum surface as the sample flowed through the gap of the drums. On the opposite side of the drum, the dried product was scraped from the drum surface by doctor blades. The dried sample collected was mixed together for further analysis from the same drying conditions. Table 1 shows the drying conditions applied in the VDD.

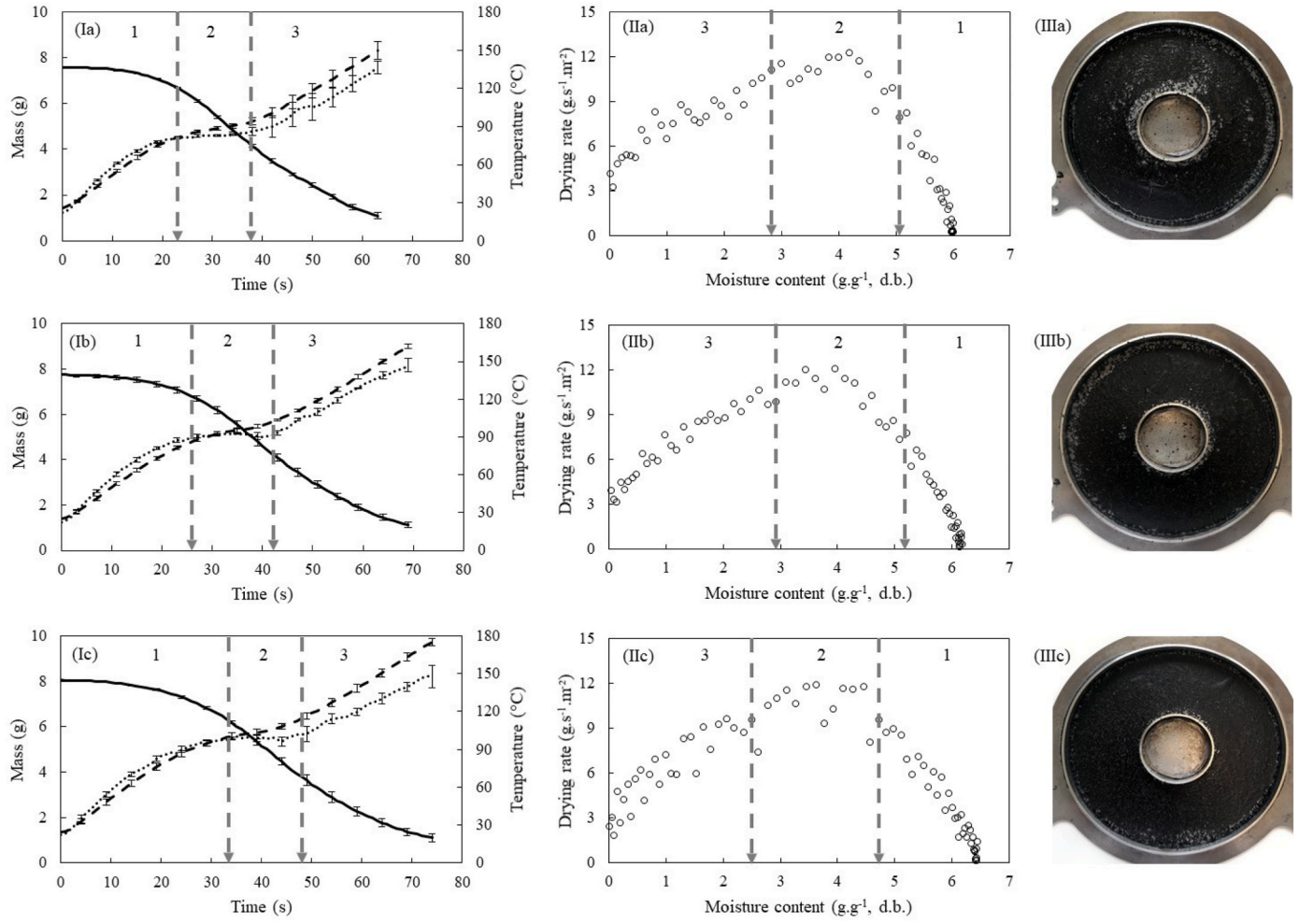

Fig. 1. (I) Mass (solid lines) and temperature profiles (dotted lines) of açaí pulp, and temperature profile of the heated pan (dashed lines); (II) drying rates versus moisture content; and (III) images of the dried açaí pulp at varying chamber pressure: (a) 415 mbar, (b) 700 mbar, and (c) 1013 mbar. The curves represent the average of three independent measurements. The error bars represent the standard deviation of the experimental data (n = 3).

2.3. Physical analysis of açaí powder

2.3.1. Moisture content and water activity

The moisture content of the powder samples was determined gravimetrically using a vacuum oven (BINDER, VD 53, Germany) at a pressure of 310 mbar and a temperature of 70 °C for 24 h. The water activity was measured using a water activity meter (Decagon Devices Inc., Aqualab TDL, USA) at 25 °C.

2.3.2. Lipid content

For lipid content determination, açaí samples were extracted with petroleum ether using an automatic Soxhlet extractor (Gerhardt, Soxtherm®, Germany). Results were expressed as g of lipid per 100 g of dry solids.

2.3.3. Color

The color parameters of samples were measured using a colorimeter (Konica Minolta, CR-400, Japan). The color values were expressed according to the CIELab scale as L^* ($L^* = 0$: black; $L^* = 100$: white), a^* ($-a^*$ = green; $+a^*$ = red) and b^* ($-b^*$ = blue; $+b^*$ = yellow). The total color differences (ΔE^*) between contact dried and reference samples were calculated using Eq. (1):

$$\Delta E^* = \sqrt{\left(L^*_{sample} - L^*_{ref}\right)^2 + \left(a^*_{sample} - a^*_{ref}\right)^2 + \left(b^*_{sample} - b^*_{ref}\right)^2} \quad (1)$$

2.4. Chemical analysis of açaí powder

2.4.1. Total monomeric anthocyanin content

The total monomeric anthocyanin content in the pulp and dried samples was quantified by the pH differential method (Giusti & Wrolstad, 2001). About 0.3 g of açaí powder was extracted with 10 mL of HCl/water/ethanol solution (1:29:70, v/v/v) using an ultrasonic bath (Elma, Elmasonic P, Germany) for 5 min at room temperature. The material was centrifuged at 3,400 rpm for 10 min, and the supernatant was filtered. The pellets were extracted twice more in the same fashion, and supernatants were combined. The extracts were diluted with two different buffer solutions (pH 1.0 and pH 4.5), and absorbances were recorded at 523 nm and 700 nm. Results were expressed as mg of cyanidin-3-rutinoside equivalent per 100 g of dry extract, using a molar absorptivity (ε) of 28,840 L mol^{-1} cm^{-1} and a molecular weight of 631 g mol^{-1}. Cyanidin-3-rutinoside is the major anthocyanin present in açaí (Pacheco-Palencia et al., 2009; Tonon, Brabet, & Hubinger, 2010).

2.4.2. Antioxidant activity

The antioxidant activity was determined using the DPPH free radical (DPPH•) scavenging method proposed by Brand-Williams, Cuvelier, and Berset (1995), with modifications. The extracts were prepared according to the method described by Larrauri, Rupérez, and Saura-Calixto (1997), with minor adaptations. In brief, 0.5 g of açaí powder was extracted with 40 mL methanol/water (50:50, v/v) at room temperature for 60 min.

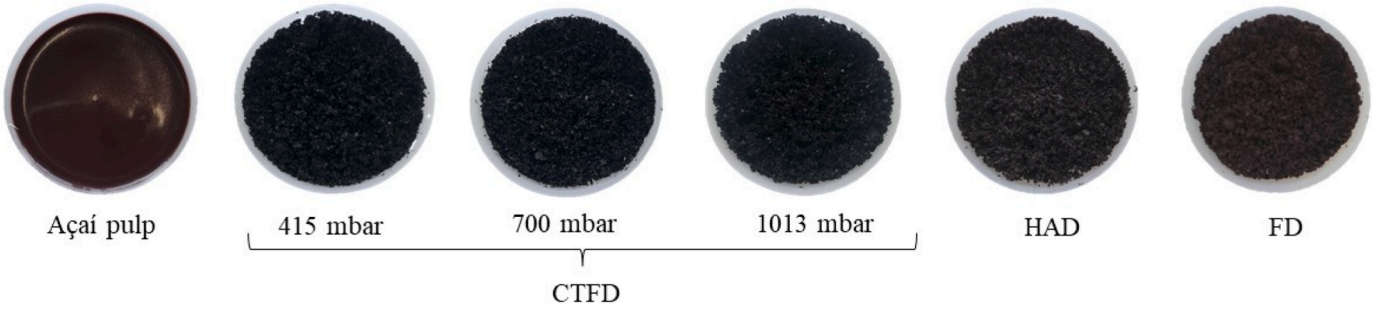

Fig. 2. Images of açaí pulp and powders produced by lab-scale conductive thin-film drying (CTFD) at 415, 700, and 1013 mbar, hot air drying (HAD), and freeze-drying (FD).

The material was centrifuged at 4,700 rpm for 15 min, and the supernatant was recovered. Then, the residue was re-extracted with 40 mL acetone/water (70:30, v/v) at room temperature for 60 min and centrifuged. Methanol and acetone extracts were pooled and diluted to a final volume of 100 mL with distilled water. Afterward, aliquots of 100 μL of açaí extract were added to 3.9 mL of 0.06 mmol L^{-1} DPPH methanolic solution. The absorbance was measured at 515 nm after 60 min of reaction at room temperature and in the dark. A standard Trolox curve (40–1000 μM) was prepared, and results were expressed as μmol of Trolox Equivalent (TE) per g of dry extract.

2.4.3. Lipid oxidation products

The proton nuclear magnetic resonance (^{1}H NMR) method, as proposed by Merkx, Hong, Ermacora, and van Duynhoven (2018), was applied to quantify hydroperoxides and aldehydes, which are primary and secondary lipid oxidation products, respectively. Firstly, the extraction of oil present in the açaí powder samples was performed as reported by Vargas-Ortiz, Servent, Salgado-Cervantes, and Pallet (2017). Subsequently, 150 μL of oil was collected, and 450 μL of 5:1 $CDCl_3$/DMSO-d_6 (Euriso-top, France) was added. After vortexing, the solution was transferred to a 5-mm NMR tube. ^{1}H NMR spectra were recorded on a Bruker Avance III 600 MHz NMR spectrometer (Bruker BioSpin, Switzerland) equipped with a 5-mm cryoprobe at a temperature of 295 K. A single pulse experiment was performed to record the peaks of the glycerol backbone at δ 4.4 ppm, which were used as internal calibration for the quantification of the lipid oxidation products. The peaks of hydroperoxides (δ 11.2–10.7 ppm) and aldehydes (δ 9.8–9.4 ppm) were obtained using two band-selective experiments. Data were processed using Bruker TopSpin 4.1 software. The concentrations of hydroperoxides and aldehydes were expressed as mmol per kg of oil.

2.5. Statistical analysis

All physicochemical analyses were performed at least in duplicate, and results are presented as mean ± standard deviation. One-way analysis of variance (ANOVA) and Tukey's test at a 95% confidence level ($\alpha = 0.05$) were applied to evaluate the experimental data. The statistical analyses were carried out using Statistica 10.0 (StatSoft, Tulsa, USA).

3. Results and discussion

3.1. Impact of varying pressure at lab-scale CTFD on drying behavior

Monitoring mass and temperature profiles during drum drying operation at a pilot or industrial scale is challenging. Thus, a laboratory-scale conductive thin-film dryer was used to provide relevant insights for drum drying, to study the impact of drying pressure on the drying kinetics of açaí pulp and the quality of the final product. Fig. 1(I and II) shows the mass and temperatures' evolutions throughout drying time and drying rate curves of açaí pulp during the lab-scale CTFD process, applying three different drying pressures. During drying, three distinct periods were identified: heating period (period 1), boiling or constant rate period (period 2), and conductive drying or declining rate period (period 3). These three drying periods were also observed by Qiu, Kloosterboer, et al. (2019) for the CTFD of maltodextrin and starch suspensions with thicknesses of 1 and 2 mm, using the same experimental system.

As expected, the decrease in the drying pressure reduced the boiling temperature of the samples. The corresponding boiling temperatures for 415, 700, and 1013 mbar were around 82, 91, and 100 °C, respectively. These temperatures were slightly above pure water boiling temperatures due to boiling point elevation. Decreasing the chamber pressure resulted as well in shorter drying times because the boiling (period 2) started earlier. The drying times were 63, 69, and 74 s for the chamber pressures of 415, 700, and 1013 mbar, respectively. Drying at the different pressures showed a similar maximum constant drying rate being approximately 10.5 $g_{water}\ s^{-1}\ m^{-2}$. This value was at least 1.4 times lower than those reported by Qiu, Kloosterboer, et al. (2019) for the drying of maltodextrin and starch suspensions, which may be explained by the presence of more oil in açaí pulp influencing the drying kinetics. Oil has a lower thermal conductivity than water and can act as a physical barrier to moisture migration (Cyprian et al., 2016). Furthermore, at the end of açaí pulp drying, it was found that with decreasing chamber pressure, bubble formation was more vigorous (Fig. 1III). A higher growth rate and larger size of bubbles are related to the lower vapor density at lower pressure (Surtaev, Serdyukov, & Malakhov, 2020).

3.2. Impact of varying pressure at lab-scale CTFD on açaí powder properties

3.2.1. Moisture content and water activity

The moisture content and water activity of açaí powders produced by lab-scale CTFD at different drying pressures, HAD, and FD ranged from 0.015 g g^{-1} to 0.026 g g^{-1} (dry basis) and from 0.163 to 0.500, respectively. Moisture content values were close to those reported by Pavan (2010, p. 88) for açaí powder produced by refractance window drying, hot air drying, and freeze-drying. Water activity values below 0.5 prevent microbial growth and reduce rates of other deteriorative reactions, including nonenzymatic browning and enzymatic activity (Taoukis, Labuza, & Saguy, 1997).

3.2.2. Color

The açaí pulp had a dark purple color, as shown in Fig. 2, due to the high concentration of anthocyanins (Bichara & Rogez, 2011). Anthocyanins are plant pigments responsible for blue, purple, red, and orange colors (Schwartz, Cooperstone, Cichon, von Elbe, & Giusti, 2017). The different drying methods resulted in different product colors. From visual analysis (Fig. 2), the contact-dried and hot air-dried samples showed a darker color compared to the freeze-dried açaí powders.

The instrumental color parameters (L^*, a^*, and b^*) and the total color differences (ΔE^*) of the açaí powders, considering HAD and FD as

Table 2
Color parameters (L^*, a^*, and b^*) and total color differences (ΔE^*) of açaí powders.

Drying process	L^*	a^*	b^*	ΔE^* (HAD ref)	ΔE^* (FD ref)
CTFD (415 mbar)	30.15 ± 0.23^{b}	0.26 ± 0.01^{c}	0.16 ± 0.05^{d}	1.26	3.23
CTFD (700 mbar)	30.38 ± 0.16^{b}	0.30 ± 0.04^{c}	0.22 ± 0.03^{cd}	1.16	3.10
CTFD (1013 mbar)	30.19 ± 0.17^{b}	0.28 ± 0.01^{c}	0.27 ± 0.07^{c}	1.18	3.16
HAD	30.47 ± 0.14^{b}	1.33 ± 0.07^{b}	0.74 ± 0.02^{b}	–	–
FD	31.33 ± 0.34^{a}	3.09 ± 0.08^{a}	1.19 ± 0.04^{a}	–	–

Note: Drying processes: conductive thin-film drying (CTFD), hot air drying (HAD), and freeze-drying (FD). HAD ref and FD ref are hot air-dried and freeze-dried reference samples, respectively. Color parameters are expressed as average ± standard deviation (n = 6). For each drying process, açaí powder was prepared in two independent experiments, and samples were evaluated in triplicate. Means followed by different letters in the same column represent significant differences ($p \leq 0.05$), according to Tukey's test.

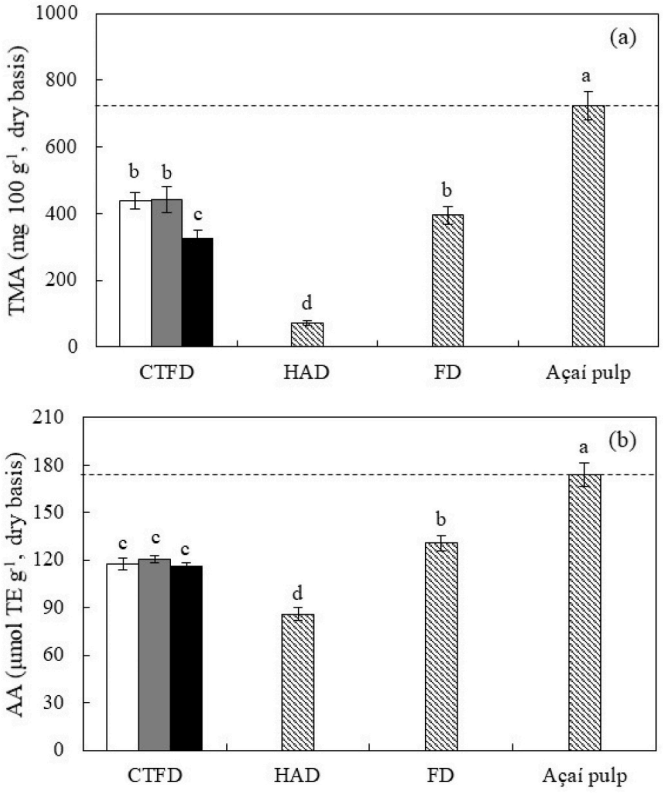

Fig. 3. (a) Total monomeric anthocyanin content (TMA) and (b) antioxidant activity (AA) in the açaí pulp and powders produced by lab-scale conductive thin-film drying (CTFD) at 415 mbar (white column), 700 mbar (grey column), and 1013 mbar (black column), hot air drying (HAD), and freeze-drying (FD). For each drying process, açaí powder was prepared in independent duplicate, and the measurements were carried out in triplicate. The error bars represent the standard deviation of the experimental data (n = 6). The different letters represent significant differences ($p \leq 0.05$). The dashed lines are added to guide the eyes.

references, are shown in Table 2. The chamber pressure during lab-scale CTFD did not significantly influence dried samples' luminosity (L^* value) and redness (a^* value). Nevertheless, decreasing the drying pressure decreased the b^* value, indicating a slight tendency to blue. Comparing each drying method, the L^* values of açaí pulp dried by

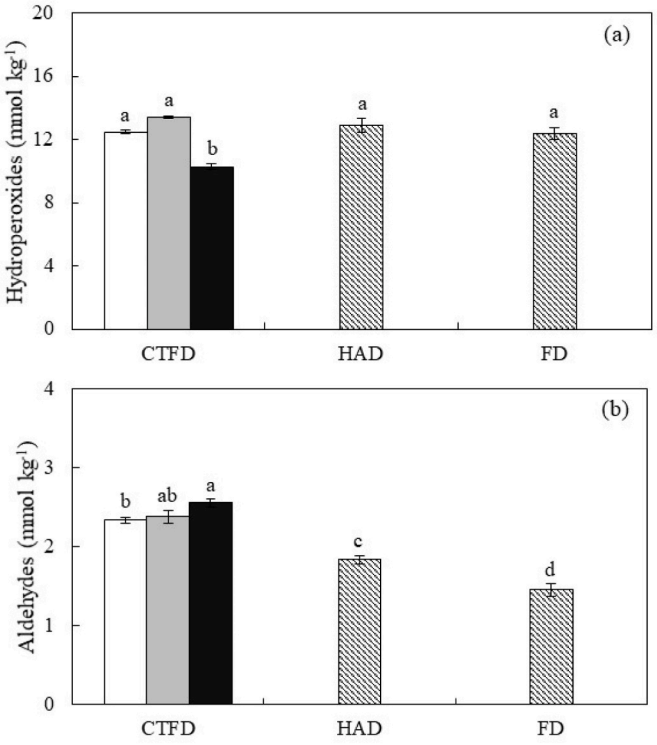

Fig. 4. The concentration of (a) hydroperoxides and (b) aldehydes in açaí powders produced from lab-scale conductive thin-film drying (CTFD) at 415 mbar (white column), 700 mbar (grey column), and 1013 mbar (black column), hot air drying (HAD), and freeze-drying (FD). For each drying process, açaí powder was prepared in two independent experiments, and the samples were analyzed in duplicate. The error bars represent the standard deviation of the experimental data (n = 4). The different letters represent significant differences ($p \leq 0.05$).

CTFD were similar to that from HAD and significantly lower than the FD samples, suggesting a darker color. Moreover, higher ΔE^* values were observed between CTFD and FD powders. These results reassert the visual analysis. The fewer color changes between CTFD and HAD samples can be attributed to the drying temperatures during CTFD and extended exposure to oxygen during HAD, which possibly accelerated nonenzymatic browning (Maillard reaction) and anthocyanin degradation, respectively.

3.2.3. *Total monomeric anthocyanin content and antioxidant activity*

The anthocyanin content and antioxidant activity measured by the DPPH assay of açaí pulp and CTFD, HAD, and FD powders are presented in Fig. 3. Açaí powders obtained by CTFD under reduced pressures showed anthocyanin retention (61%) comparable to that of freeze-dried samples (54%) and significantly greater than that of hot air-dried powders (10%). Anthocyanins are sensitive to high temperature, oxygen, and light, among other factors (Tonon et al., 2010). Therefore, the high anthocyanin retention during CTFD at low pressures may be explained by the short drying times at moderate boiling temperatures and low oxygen concentrations. This result is supported by Zhou, Chen, Bi, Wang, and Wu (2017), who reported the positive effect of low temperature and low oxygen atmosphere on anthocyanin retention during mulberry fruit drying.

Conversely, the antioxidant activity of CTFD samples was significantly lower than that of freeze-dried samples, with no significant difference among drying pressures applied in CTFD. Even though anthocyanins account for approximately 90% of the antioxidant activity of açaí (Pacheco-Palencia et al., 2009), two hypotheses can justify the antioxidant activity behavior of the samples (Tonon et al., 2010): (1) the

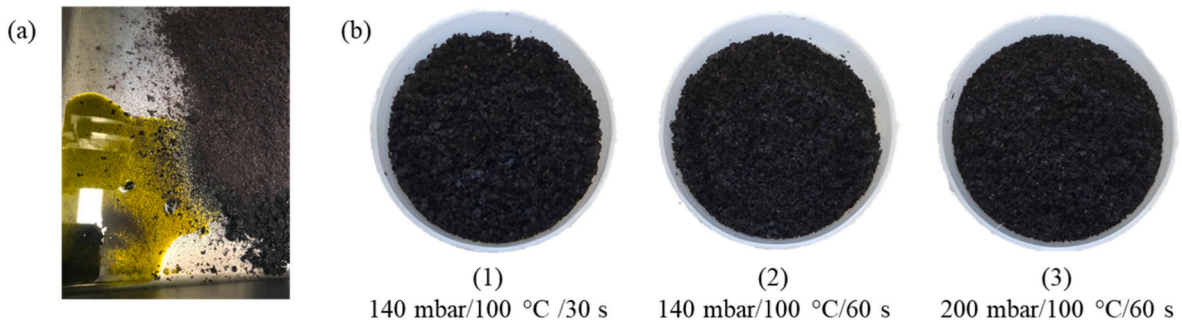

Fig. 5. (a) Representative image of oil release from the matrix during vacuum drum drying (VDD) of açaí pulp; and (b) Images of vacuum drum-dried açaí powders at the different drying conditions.

freeze-drying may have increased the bioavailability of non-identified compounds, which contribute to the antioxidant activity; and (2) the increase in boiling temperature in CTFD, due to the increase in the chamber pressure, may have provided the formation of Maillard reaction products with antioxidant activity.

3.2.4. Lipid oxidation products

Açaí has a high unsaturated fatty content, being susceptible to lipid oxidation during processing and storage (Schauss et al., 2006). In the lipid oxidation process, primary oxidation products are formed (peroxides, especially hydroperoxides), which further decompose to secondary oxidation products (e.g., aldehydes, ketones, alcohols, and acids) that are responsible for negative quality changes in foods, such as deterioration of flavor and reduction in nutritional value (Barriuso, Astiasarán, & Ansorena, 2013). After the drying processes, the concentration of hydroperoxides and aldehydes in the lipid fraction (oil) of açaí powders was measured (Fig. 4).

The CTFD at atmospheric pressure resulted in the lowest formation of hydroperoxides, which can be due to the fastest decomposition of these compounds to secondary lipid oxidation products, as illustrated by the highest aldehydes concentration (Fig. 4b), which was caused by the highest boiling temperature during drying (Hoppenreijs, Berton-Carabin, Dubbelboer, & Hennebelle, 2021). The formation of aldehydes during CTFD decreased as drying pressure was reduced, which can be associated with the reduction in the boiling temperature, oxygen concentration, and drying time. Although HAD showed a longer air product exposure time than CTFD, a lower degree of aldehydes formation was observed for hot air-dried samples. This result is probably related to lower temperatures in HAD compared to CTFD, suggesting a dominant effect of temperature during the drying of açaí pulp for accelerating lipid oxidation.

According to Codex Alimentarius (1999), the recommended maximum value of hydroperoxides for edible oils is 10 milli-equivalents O_2 kg^{-1}. However, for all açaí powders in this study, irrespective of the drying method used, the hydroperoxides concentrations exceeded this recommended value and varied from 10.3 to 13.4 mmol hydroperoxides kg^{-1} (or 20.6 to 26.8 milli-equivalents O_2 kg^{-1}). Lucas, Zambiazi, and Costa (2018) found lower hydroperoxides concentration in freeze-dried açaí powder (6.34 milli-equivalents O_2 kg^{-1}). The use of different oil extraction methods contributes to the differences in the concentration of primary lipid oxidation products since this process affects the oil quality (Santana, dos Reis, Torres, Cabral, & Freitas, 2015). Extraction with petroleum ether at a temperature of 45 °C was used in the present study, whereas Lucas et al. (2018) used extraction with chloroform, methanol, and water at room temperature.

3.3. Performance of the VDD for production of açaí powder

A partial limitation of the experimental system (CTFD at lab-scale) is its operation at constant power, while (vacuum) drum drying operates at a constant wall temperature. In this way, the drying of açaí pulp was also performed in VDD at the pilot scale. The effect of pressure in the vacuum chamber and residence time on the quality of açaí powder were assessed.

The original açaí pulp (86%, wet basis) could be directly fed into the dryer. During VDD, most of the moisture in the sample was evaporated during boiling in the feeding pool between the drums (period 2), and a conductive drying (period 3) occurred on the drums after the açaí pulp was cast as a thin film (Qiu, Acharya, et al., 2019). The spreading, drying, and collection of the dried açaí as small flakes were observed for all applied drying conditions. The drying process took between 30 and 60 s. However, oil separation was visually observed (Fig. 5a), except for condition 1 (140 mbar/100 °C/30 s), which was the mildest drying condition. This visual result was confirmed by quantitative analysis of lipid content, as discussed in the following section.

Table 3
Moisture content, water activity, lipid content, and color of vacuum drum-dried açaí powders.

Parameters	Drying conditions		
	(1) 140 mbar/ 100 °C/30 s	(2) 140 mbar/ 100 °C/60 s	(3) 200 mbar/ 100 °C/60 s
Moisture content (g g^{-1}, dry basis)	0.015 ± 0.000^{a}	0.010 ± 0.001^{b}	0.014 ± 0.001^{a}
Water activity	0.232 ± 0.002^{a}	0.143 ± 0.004^{c}	0.216 ± 0.003^{b}
Lipid content (g 100 g^{-1}, dry basis)	45.1 ± 0.6^{a}	39.6 ± 0.2^{b}	35.2 ± 1.2^{c}
Color			
L^*	30.38 ± 0.10^{a}	30.32 ± 0.15^{a}	30.59 ± 0.09^{a}
a^*	1.07 ± 0.06^{b}	0.94 ± 0.02^{c}	1.20 ± 0.06^{a}
b^*	0.38 ± 0.02^{a}	0.38 ± 0.03^{a}	0.39 ± 0.05^{a}

Note: Parameters are expressed as average ± standard deviation (n = 3). For each drying condition, the measurements were performed in triplicate. Means followed by different letters in the same row represent significant differences ($p \leq 0.05$), according to Tukey's test.

3.4. Physicochemical properties of vacuum drum-dried açaí powder

The moisture content, water activity, lipid content, and color parameters of the vacuum drum-dried açaí powders at the different drying conditions are presented in Table 3. The VDD of açaí pulp resulted in powders with moisture content and water activity close to those obtained by other drying methods that require a much longer drying time (Oliveira et al., 2020; Pavan, 2010, p. 88).

Lipids are the major compounds of açaí pulp (46.7 ± 0.3 g 100 g^{-1}, on dry basis). During the VDD, the increase in the residence time and boiling temperature (conditions 2 and 3) resulted in a significant decrease in the lipid content of dried samples because of undesired oil release from the matrix. The oil loss during drying leads to a significant reduction in the product's nutritional value, besides operational problems, such as frequent dryer downtime for cleaning.

In contrast to what was observed in the lab-scale conductive drying,

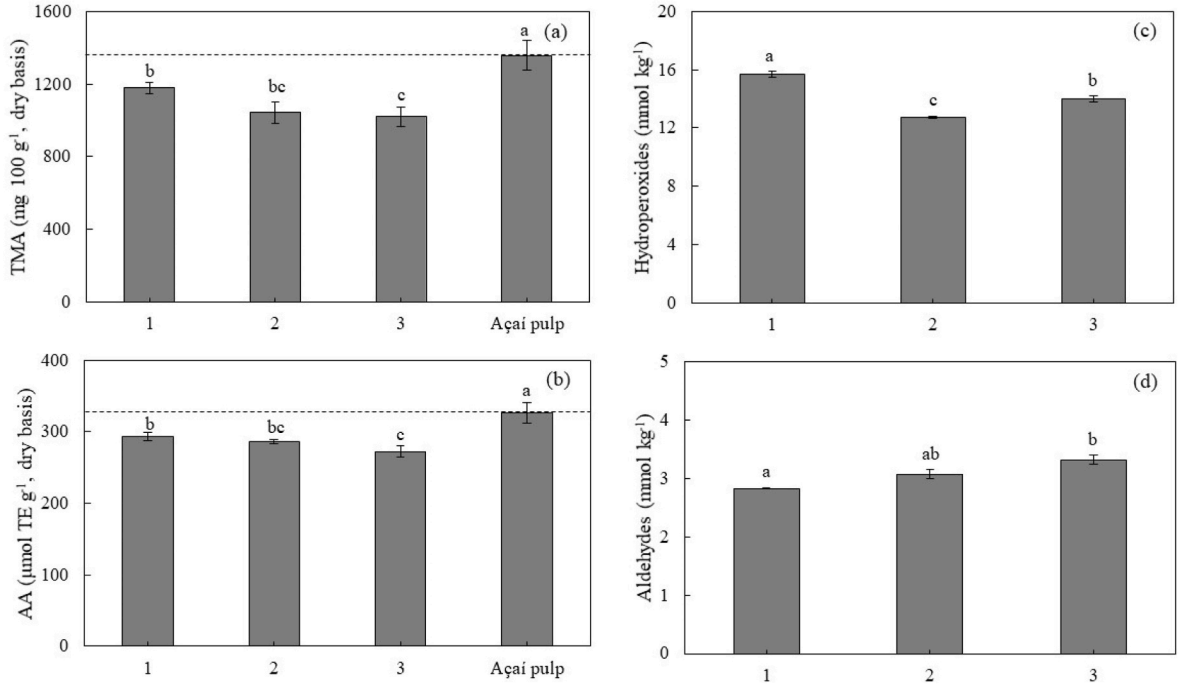

Fig. 6. (a) Total monomeric anthocyanin content (TMA), (b) antioxidant activity (AA), and concentration of (c) hydroperoxides and (d) aldehydes in vacuum drum-dried açaí powders at different drying conditions: (1) 140 mbar/100 °C/30 s; (2) 140 mbar/100 °C/60 s; and (3) 200 mbar/100 °C/60 s. For each drying condition, the measurements were performed in duplicate The error bars represent the standard deviation of the experimental data (n = 2). The different letters represent significant differences ($p \leq 0.05$). The dashed lines are added to guide the eyes.

drying conditions used in VDD affected the a^* value (greenness/redness), probably due to the loss of oil during drying since the açaí oil has a dark green color (Silva & Rógez, 2013). Fig. 5b shows the açaí powders produced by VDD.

The anthocyanin content and antioxidant activity of samples produced by VDD (Fig. 6a and b) were calculated by excluding the lipid content of samples to have the same dry matter content at all drying conditions. The VDD decreased the anthocyanin content and antioxidant activity of powders, by an average of 13–25% and 10–16%, respectively, from the anthocyanin content and antioxidant activity of açaí pulp. Similar results were reported by Tonon et al. (2010) for powders produced by spray drying of the filtered açaí pulp added with carrier agents. As observed in the lab-scale CTFD, increasing boiling temperature under vacuum conditions did not influence the anthocyanin content and antioxidant activity in açaí powders. However, the product's anthocyanin content and antioxidant activity were negatively affected by the simultaneous increase in boiling temperature and residence time (condition 3).

Concerning oxidative stability of açaí oil in the vacuum drum-dried samples (Fig. 6c and d), the concentration of primary lipid oxidation products ranged from 12.7 to 15.7 mmol hydroperoxides kg^{-1} (or 25.5 to 31.4 milli-equivalents O_2 kg^{-1}). These high values can be again related to the oil extraction method. Although condition 1 showed the highest hydroperoxides concentration, the lowest aldehydes concentration was observed, indicating few changes in flavor and odor of powders. This result agrees with that from the lab-scale drying, i.e., a low boiling temperature, low oxygen concentration, and short drying time during contact drying resulted in a slow oxidative breakdown of primary products to form secondary products.

4. Conclusion

The conductive thin-film drying process at the laboratory and pilot scale allowed producing açaí powders with low moisture contents and water activities with short drying times. High retention of anthocyanins and antioxidant activity was achieved by drying at low pressures due to low boiling temperatures, low-oxygen environments, and short process times. Moreover, decreasing the drying pressure resulted in slow decomposition of primary lipid oxidation products to form secondary lipid oxidation products, suggesting smaller undesirable sensorial and nutritional effects in açaí powders. However, contact drying resulted in less oxidative stability of açaí oil than hot air drying and freeze-drying. In addition, oil release is a risk during pilot-scale drying but could be mitigated by selecting mild low-pressure drying conditions. Vacuum conductive thin film drying is, therefore, a good alternative for producing açaí powder, although further process optimization is still required. The assessment of functional (such as dispersion time and solubility) and sensorial properties is also recommended to obtain detailed information on the rehydration behavior and consumers' acceptability of açaí powders.

CRediT authorship contribution statement

Raquel da Silva Simão: Conceptualization, Methodology, Formal analysis, Investigation, Writing – original draft, Writing – review & editing. **Lu Zhang:** Conceptualization, Supervision, Writing – review & editing. **Jaqueline Oliveira de Moraes:** Conceptualization, Supervision. **Anja Schröder:** Methodology, Formal analysis, Investigation. **João Borges Laurindo:** Conceptualization, Supervision, Writing – review & editing, Funding acquisition. **Maarten A.I. Schutyser:** Conceptualization, Supervision, Writing – review & editing, Resources.

Declaration of competing interest

The authors declare no conflict of interest.

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