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Biopolymers to overcome challenges in açaí pulp drying: Processing and powder quality evaluation

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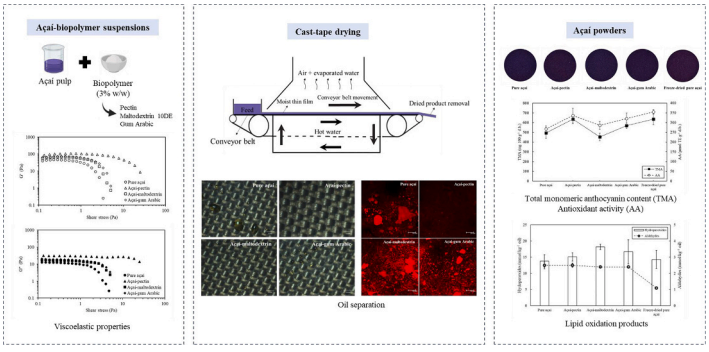
HIGHLIGHTS

- Açaí powder rich in anthocyanins and lipids from cast-tape drying is feasible.
- The release of oil from açaí pulp during drying was hindered by adding pectin.
- Adding pectin preserved anthocyanins and antioxidant activity in açaí powder.
- Formation of oxygenated α,β-unsaturated aldehydes in açaí powders has been identified.

GRAPHICAL ABSTRACT



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ABSTRACT

The production of stable açaí powder by affordable drying methods is challenging due to its high unsaturated lipids and anthocyanins content. This study assessed the effect of adding pectin, maltodextrin, and gum Arabic at a low concentration on the processing of açaí pulp and the powdered product quality obtained by cast-tape drying, an energy-efficient drying technique. The pectin-containing sample had the highest linear viscoelastic range and flow point, contributing to the film-forming during drying and the powder quality. Pectin hindered oil separation during drying, as shown in micrograph images and CLSM, and the dried açaí-pectin material was effortlessly removed from the support. FTIR spectra indicated physical rather than chemical interactions between açaí and pectin. Moreover, adding pectin resulted in a powder with anthocyanin content and antioxidant activity like freeze-dried products. This study demonstrates that a small biopolymer concentration is a technological solution for obtaining high-quality açaí powders by cast-tape drying.

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1. Introduction

Açaí (*Euterpe oleracea* Mart.) is one of the most worldwide known Amazonian fruits with significant production and socio-economic importance in the northern states of Brazil. This fruit, one of the so-called superfruits, has attracted the attention of industries and scientists due to its high nutritional value, which benefits human health. It is rich in fibers, proteins, unsaturated fatty acids, and anthocyanins with high antioxidant activity [1–3]. Nevertheless, açaí fruit and its fresh pulp are highly perishable, with few hours of pulp shelf life even under refrigeration. The drying of açaí pulp to produce açaí powder is an attractive approach to prolong its shelf-life. Furthermore, açaí pulp dehydration may reduce losses, facilitate commercialization, add value, and diversify consumption options. However, the drying technique and conditions can affect the stability of anthocyanins and lipids of açaí. Therefore, these must be carefully selected to retain the fruit's nutritional quality as much as possible.

Freeze-drying (FD) and spray-drying (SD) are commonly applied to produce açaí powder [4–6]. Even though FD results in high-quality dried products, this technology is costly because of the extended operation time and high energy consumption [7]. SD has the advantage of short drying times but has low energy efficiency and requires previous filtrating of açaí pulp to eliminate solids that can block the nozzle atomizer [8]. Filtrating the açaí pulp removes insoluble fibers and reduces lipid content, leading to significant nutritional losses for the resulting dried powder. In addition, traditional SD processes usually require a relatively large amount of biopolymers to reduce stickiness and protect the bioactive compounds [6,9].

Producing fruit powders with good nutritional properties by energy-efficient drying methods has been challenging. Cast-tape drying (CTD) or refractance window drying – a particular case of CTD – is a conductive thin-film drying technique that converts whole fruit or vegetable purees or juices (with no filtration) into films, flakes, or powders [10]. This technique operates at atmospheric pressure and uses hot water just below its boiling temperature as a heating medium to supply latent heat for water evaporation from a thin layer of product spread on a flexible support [11]. CTD can dry the fruit pulp in a few minutes, even at moderate product temperatures during drying, e.g., 70 °C, with retention of heat-sensitive compounds [12,13]. Moreover, this process consumes less energy than SD and FD [14]. Nevertheless, Pavan [15] and Souza [16] reported lower retention of anthocyanins in açaí powder produced by refractance window drying than in freeze-dried açaí powder. Furthermore, lipids in dried açaí powders are in the liquid state at room temperature and, therefore, more prone to oxidation [15].

A biopolymer (polysaccharide or protein) added at a low concentration to the pulp before drying can be a promising strategy to better protect the bioactive compounds without compromising the açaí powder's quality produced by CTD. Biopolymers containing hydrophilic and hydrophobic groups that create a polymer network may promote the protection of sensitive compounds from heat, oxygen, moisture, and light during processing and storage [17]. Maltodextrin and gum Arabic are commonly used biopolymers to prevent the degradation of bioactive compounds during the drying of fruit juices and purees, mainly due to their high solubilities, low viscosities, and high glass transition temperatures [6,18]. Pectin has also shown great potential for adequately protecting bioactive compounds because of its gelling, stabilizing, and emulsifying properties [19,20].

Therefore, it was hypothesized that a low concentration of biopolymer added to the açaí pulp before CTD would protect the food against oxidation and other possible reactions, leading to a powdered product with quality as good as freeze-dried pulp. In this way, the present study investigated the influence of only 3% (*w*/w) of pectin, maltodextrin, or gum Arabic added into the açaí pulp on the drying process and powdered product quality. It was assessed by color, lipid presence, anthocyanin retention, antioxidant activity, and formation of lipid oxidation products.

2. Material and methods

2.1. Materials

Frozen açaí pulp (Norfrutas Eireli, Belém, PA, Brazil) – obtained by macerating the açaí fruit and adding water and afterward frozen – was purchased from a local market in Florianópolis, SC, Brazil. The biopolymers used were pectin with a 68% degree of esterification (GENU® high methoxyl pectin type 106 BP, CPKelco, Limeira, SP, Brazil), maltodextrin DE10 (MOR-REX® 1910, Ingredion, São Paulo, SP, Brazil), and gum Arabic (Wifa Ingredientes, Palhoça, SC, Brazil). Nile Red, potassium bromide (KBr), 2,2-diphenyl-1-picrylhydrazyl (DPPH), and (±)-6-hydroxy-2,5,7,8-tetramethylchromane-2-carboxylic acid (Trolox) were purchased from Sigma-Aldrich (St. Louis, USA). Chloroform-*d* ($CDCl_3$) and dimethyl sulfoxide-d_6 (DMSO-d_6) were purchased from Euriso-top (Saint-Aubin, France). All the other chemical reagents used were of analytical grade.

2.2. Preparation of açaí-biopolymer suspensions

The frozen açaí pulp was stored at −18 °C, and the required amount for each experiment was thawed at room temperature until 10 °C. The physicochemical composition of the açaí pulp is presented in Table 1. Proximate composition was carried out according to AOAC methods [21] for moisture content (method no. 930.15), proteins (method no. 977.02), lipids (method no. 930.09), crude fiber (method no. 978.10), and ash (method no. 930.05). Carbohydrates were calculated by difference. In addition, the soluble solids concentration was determined by a refractometer (Atago, PAL-BX/RI, Japan), and pH was measured using a pH meter (Testo, testo 205, Germany).

Four suspensions were prepared: (i) Açaí pulp without the addition of biopolymers (pure açaí pulp); (ii) Açaí pulp + pectin; (iii) Açaí pulp + maltodextrin; and (iv) Açaí pulp + gum Arabic. A low proportion of biopolymers was chosen to obtain as pure açaí powders as possible. Thus, all açaí + biopolymer suspensions were prepared using 3 g of biopolymer per 100 g of açaí pulp. It was the lowest biopolymer concentration in which the phase separation during drying was not visually observed. The homogenization was performed using a hand blender (Oster, FPSTHB2610R-057, Brazil) for 2 min at the minimum speed.

2.3. Rheological properties

Dynamic oscillatory measurements were performed using a rotational rheometer (Thermo Scientific™, HAAKE MARS, Germany) with a parallel plate geometry of 20 mm diameter and 2.5 mm gap. The açaí suspensions were evaluated at 11.2 ± 0.1 °C, which was the temperature when the samples were spread over the cast-tape dryer support. The amplitude stress sweep from 0.1 to 25 Pa was carried out at 0.1 Hz to define the linear viscoelastic (LVE) region. Moreover, the critical shear stress (flow point) was determined at the crossover point ($G' = G''$). Subsequently, the frequency sweep test (0.01–10 Hz) was conducted at a constant shear stress value within the LVE region to obtain the dynamic modulus: elastic or storage modulus (G') and viscous or loss modulus

Table 1
Composition of açaí pulp (*Euterpe oleracea* Martius).

Component	Mean ± SD
Moisture content (g 100 g^{-1} w.b.)	84.97 ± 0.25
Proteins (g 100 g^{-1} d.b.)	10.49 ± 0.20
Lipids (g 100 g^{-1} d.b.)	52.23 ± 0.83
Total fiber (g 100 g^{-1} d.b.)	7.02 ± 1.50
Carbohydrates (g 100 g^{-1} d.b.)	25.55 ± 0.27
Ash (g 100 g^{-1} d.b.)	4.97 ± 0.35
pH	5.01 ± 0.12
Total soluble solids (°Brix)	4.36 ± 0.40

Note: w.b.: wet basis; d.b.: dry basis.

(G″). The measurements were performed in independent triplicate.

2.4. Drying processes

2.4.1. Cast-tape drying (CTD)

CTD experiments were performed by casting the açaí suspensions on the conveyor belt of 0.25 mm-thick fiberglass film coated with Teflon® (Indaco, Lençol Armalon® Standard, Brazil) in a pilot-scale apparatus (Fig. 1). The dryer presented an effective drying area of 0.54 m^2 (2.00 m long x 0.27 m wide). The hot water temperature below the conveyor belt was controlled at 98 °C, producing vapor to heat the açaí suspensions by indirect contact during drying [22]. The belt coated with Teflon® is a hydrophobic, opaque surface with low surface energy. The casting used a doctor blade with a gap between 1 and 2 mm and a conveyor belt speed from 6.25 to 12.5 cm min^{-1}, resulting in a uniform suspension thickness of around 1.5 mm (Table 2). In order to obtain the same spreading thickness and allow the dryer to operate in a continuous regime, each pair of doctor-blade gap and belt velocity was selected from scanning the thickness of spread suspension in the function of belt velocity (3.33–26.7 cm min^{-1}) and spreader gap (1–3 mm) (data not shown). The thicknesses of spread samples were measured with a caliper (Mitutoyo Co., Japan) immediately after the passage of suspensions through the spreader.

The evaporated water during drying was removed by natural convection under ambient conditions: relative humidity of 50–73% and temperature of 25–29 °C, measured continuously. Drying was conducted until the product moisture content was less than 5% (wet basis), following Brazilian legislation [23]. The dried açaí suspensions were scraped off from the support at the end of the heating zone. After drying the açaí suspensions, micrographs of the fiberglass support coated with Teflon® were captured using an optical stereoscope (OptiCam, OPT 10000, Brazil). The images were analyzed by image processing software (TSview, Tucsen, China).

2.4.2. Freeze-drying (FD)

FD experiments were performed to obtain pure dehydrated açaí pulp to be a reference for this study. The FD process was conducted in a lab-scale freeze-dryer (Liobrás, Liotop L101, Brazil). The sample was dried to a moisture content below 5% (wet basis) in a chamber with an internal pressure of 0.06 mbar and an ice condenser temperature of −54 °C.

2.5. Characterization of the dehydrated product

After the CTD process, the dried açaí samples were removed from the belt as films/flakes. CTD and FD samples were ground using a knife mill (TECNAL, TE 631/2, Brazil) and sieved in a 20-mesh sieve to obtain açaí powders with 850 μm or fewer particle sizes. The açaí powders were hermetically packed into high-density polyethylene (HDPE) bottles with

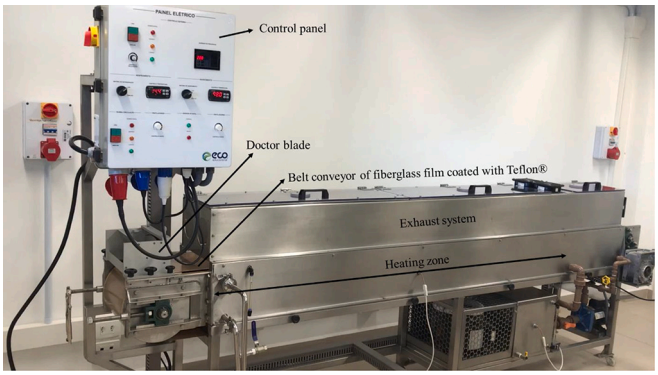

Fig. 1. Device of the continuous cast-tape drying.

Table 2
The doctor blade gap, conveyor belt velocity, and spreading thickness for each açaí suspension dehydrated by cast-tape drying (CTD).

Suspension samples	Spreader gap (mm)	Belt speed (cm min^{-1})	Suspension thickness (mm)
Pure açaí	1.0	12.5	1.41 ± 0.13
Açaí-pectin	2.0	6.25	1.40 ± 0.12
Açaí-maltodextrin	1.5	8.00	1.55 ± 0.11
Açaí-gum Arabic	1.5	8.00	1.50 ± 0.10

around 3/4 of their filled volume and stored in a freezer at −18 °C until further analysis. The açaí powder characterization used the grounded samples, except in the procedure of oil distribution in the dried samples (item 2.5.1).

2.5.1. Oil distribution in the dehydrated açaí samples

Dried açaí suspensions were stained with Nile Red dye and evaluated by confocal laser scanning microscopy (CLSM) to visualize oil droplet distribution in the samples after drying. In brief, a stock solution of Nile Red (0.8 mg mL^{-1}) in acetone was prepared and then diluted with distilled water in a 1:50 (v/v) ratio. Samples were cut into squares (5 mm x 5 mm) and stained with 200 μL of the Nile Red solution for 2 min. A confocal laser scanning microscope (Leica Microsystems, Leica TCS SP5, Germany) was used. The Nile Red fluorescence was detected using a He—Ne laser with an excitation wavelength of 543 nm and an emission filter at 605 to 640 nm (red). The micrographs of dried samples were obtained by converting a sequence of optical sections (z-stack) into a maximum intensity projection image (two-dimensional image) using Leica LAS AF Lite software.

2.5.2. Moisture content and water activity

Moisture content was determined by the gravimetric procedure using a vacuum oven (TECNAL, TE-395, Brazil) at 70 °C [21]. Water activity was measured with a water activity meter (Decagon Devices Inc., Aqualab 4TE, USA). Analyses were performed in triplicate.

2.5.3. Lipid content

The lipid content of the açaí powder samples was determined using petroleum ether as the solvent in a Soxhlet extractor, according to AOAC official method no. 930.09 [21]. The measurements were conducted in triplicate. The results were expressed as g of lipid per 100 g on dry basis. The mass of hydrocolloids was disregarded in order to have the same dry matter content at all açaí powders and, thereby, compare the oil loss of samples.

2.5.4. Color

A computer vision system was used to determine the color parameters of samples, as described in Simão et al. [24]. The color values were expressed as L^* ($L^* = 0$: black; $L^* = 100$: white), a^* ($-a^*$ = green; $+a^*$ = red), and b^* ($-b^*$ = blue; $+b^*$ = yellow). The color difference (ΔE^*) was calculated according to Eq. (1), using the FD sample as the reference value (L_0^*, a_0^*, and b_0^*). The measurements were in triplicate.

$$\Delta E^* = \sqrt{\left(L^* - L_0^*\right)^2 + \left(a^* - a_0^*\right)^2 + \left(b^* - b_0^*\right)^2} \quad (1)$$

2.5.5. Fourier transform infrared (FTIR) spectroscopy

FTIR spectra of the biopolymers and açaí powders produced by CTD were obtained using an FTIR spectrometer (Agilent Technologies, Cary 660, USA). Samples (2 mg) and KBr pellets (50 mg) were mixed, pressed, and then analyzed at room temperature using a scanning range from 400 to 4000 cm^{-1} and resolution of ±2 cm^{-1} at 21 scans min^{-1}.

2.5.6. Total monomeric anthocyanin content

The total monomeric anthocyanin content was determined by the

pH-differential method described by Giusti and Wrolstad [25]. Anthocyanins were extracted three times from 0.3 g of açaí powder using 10 mL of an HCl/water/ethanol solution (1:29:70, *v*/v/v). At each time, the mixture was sonicated for 5 min at room temperature and then centrifuged at 3400 rpm for 10 min. Supernatants were collected and combined. The extracts were separately diluted with 0.025 mol L^{-1} potassium chloride buffer (pH 1.0) and 0.4 mol L^{-1} sodium acetate buffer (pH 4.5). Total anthocyanin content was expressed in terms of mg of cyanidin-3-rutinoside equivalent per 100 g of dry extract, excluding the mass of the hydrocolloids. Absorbances were measured at 523 and 700 nm, using the molecular weight and molar absorptivity of cyanidin-3-rutinoside of 631 g mol^{-1} and 28,840 L mol^{-1} cm^{-1}, respectively. Cyanidin-3-rutinoside is the predominant anthocyanin in açaí [8,26]. Three independent replicates were evaluated.

2.5.7. Antioxidant activity

The antioxidant activity was determined in extracts obtained according to the method described by Larrauri et al. [27], with some modifications. Briefly, 0.5 g of açaí powder was extracted sequentially with 40 mL methanol/water (50:50, *v*/v) and 40 mL acetone/water (70:30, *v*/v) at room temperature for a total time of 120 min. After each extraction, the material was centrifuged at 4700 rpm for 15 min, and the supernatant was recovered. Methanol and acetone extracts were combined and made up to 100 mL with distilled water. The antioxidant activity was measured by DPPH radical scavenging activity assay according to Brand-Willians et al. [28], with minor adaptations. The reaction was carried out with 100 μL of açaí extract and 3.9 mL of 0.06 mmol L^{-1} DPPH methanolic solution for 60 min in the dark and at room temperature. Afterward, the absorbance was measured at 515 nm. The same analysis was performed on the Trolox methanolic solution in six dilutions ranging in concentration from 40 to 1000 μM, allowing the construction of a standard Trolox curve. The results were expressed as μmol of Trolox Equivalent (TE) per g of dried extract (disregarding the mass of hydrocolloid). The analysis was carried out in triplicate.

2.5.8. Lipid oxidation products

Primary and secondary lipid oxidation products (hydroperoxides and aldehydes, respectively) were quantified by proton nuclear magnetic resonance (^{1}H NMR) described by Merkx et al. [29]. The time interval between the completion of the samples' drying and the assessment of lipid oxidation products was about six months. The oil from the açaí powder samples was extracted for 60 min in a hot water bath at 45 °C with orbital agitation, using petroleum ether as the solvent in a 1:3 (*w*/w) sample:solvent ratio. The extracts were filtered under a vacuum, and the solvent was evaporated using a rotary evaporator (37 °C, 200 mbar) [30]. Then, 150 μL of oil was collected, and 450 μL 5:1 $CDCl_3$/DMSO-d_6 was added. The mixture was transferred to a 5-mm NMR tube and analyzed in a Bruker Avance III 600 MHz NMR spectrometer (Bruker BioSpin, Switzerland) equipped with a 5-mm cryoprobe at a temperature of 295 K. For each sample, a single pulse and a band-selective experiment were performed. The peaks of the glycerol backbone at δ 4.4 ppm were recorded from the single pulse experiment and were used to quantify the lipid oxidation products. From two band-selective excitations, the signals of hydroperoxides were obtained between δ 11.3 and 10.6 ppm and aldehydes between δ 9.8 and 9.4 ppm. Data were processed using Bruker TopSpin 4.0 software. Concentrations of hydroperoxides and aldehydes were expressed as mmol per kg of oil, and details of calculations were described by Merkx et al. [29]. The analysis was carried out in independent duplicates.

2.6. Statistical analysis

The experimental data were presented as mean ± standard deviation and statistically evaluated by one-way analysis of variance (ANOVA) and Tukey's test at a 95% confidence level ($\alpha = 0.05$). The statistical analyses were performed using the software Statistica 10.0 (StatSoft, Tulsa, USA).

3. Results and discussion

3.1. Viscoelastic properties of the açaí suspensions

Assessing the viscoelastic properties of the açaí suspensions provides essential information about the internal structure of these materials [31]. All the açaí suspensions were preliminary evaluated by the amplitude stress sweep test (Fig. 2). Two different regions, named linear viscoelastic (LVE) region, where G′ and G″ were almost constant and stress independent, and nonlinear (n-LVE) region, where G′ and G″ started to decrease, could be recognized. Within the LVE region, the elastic component values were always higher than viscous component values (G′ > G″), indicating a solid-like behavior of all açaí suspensions. The açaí-pectin sample exhibited the highest G′ value and the largest LVE region, suggesting the strongest and least deformed polymer network.

At the crossover point (G′ = G″), G′ values of all suspensions suddenly decreased. The critical shear stress values depended on the sample, i.e., 2.8 Pa for açaí pulp, 3.3 Pa for açaí-maltodextrin suspension, 4.1 Pa for açaí-gum Arabic suspension, and 18.7 Pa for açaí-pectin suspension. After the crossover point, the viscous behavior dominated over the elastic behavior (G″ > G′) because the external shear stress disrupted the weak interactions in the suspensions.

For spreading suspensions in the CTD apparatus, the critical shear stress must be high enough to prevent inadequate flow and sedimentation of particles during drying but not too high for suspensions to flow under the shear conditions applied during passing through the spreader [31]. It is noted that the açaí-pectin suspension showed a high critical shear stress value. If the critical shear stress was even higher, this resulted in clogging of the gap used to prepare the film (data not shown).

The solid-like viscoelastic behavior of all suspensions was confirmed by the results of the frequency sweep test (Fig. 3), with a predominance of the storage modulus (G′) over the loss modulus (G″) and no crossover point within the experimental range of frequency. Higher G′ was found in the pectin-containing suspension, resulting in a stronger structure due to the formation of elastically effective intermolecular interactions, which agrees with the amplitude stress sweep test results.

3.2. Performance of the CTD process

All açaí suspensions were evenly spread as thin layers on the flexible support (Table 2). Incorporating biopolymer into açaí pulp influenced the drying time of the samples in CTD (Table 3). Açaí pulp without biopolymers had the lowest drying time (16 min). In contrast, the suspension added with pectin exhibited the longest drying time (32 min) to reach a moisture content of around 5% (wet basis). The hydrocolloids' water-binding properties may explain the higher drying time of açaí-biopolymer suspensions, leading to slower diffusion of water. Nevertheless, as already expected and found by Baeghbali et al. [14] and Durigon et al. [32], the CTD process times were much shorter than by FD.

At the end of the CTD process, the pure açaí pulp (without additives), açaí-maltodextrin, and açaí-gum Arabic samples could be removed from the drying support as flakes. On the other hand, the dried açaí-pectin suspension was removed as a film due to the more cohesive gel structure formed during drying and its higher resistance to the breakdown. Frabetti et al. [33] also reported removing the strawberry-pectin sample from cast-tape dryer support as a continuous film without breaking.

After drying and the samples' removal, the oil released from the suspensions to the fiberglass support coated with Teflon® was observed by optical micrographs (Fig. 4a), except for the dried pectin-containing suspension. This lower oil loss suggests the strongest internal network of the açaí-pectin suspension, reducing the mobility of oil droplets [19]. The oil loss was confirmed quantitatively by analyzing the lipid content.

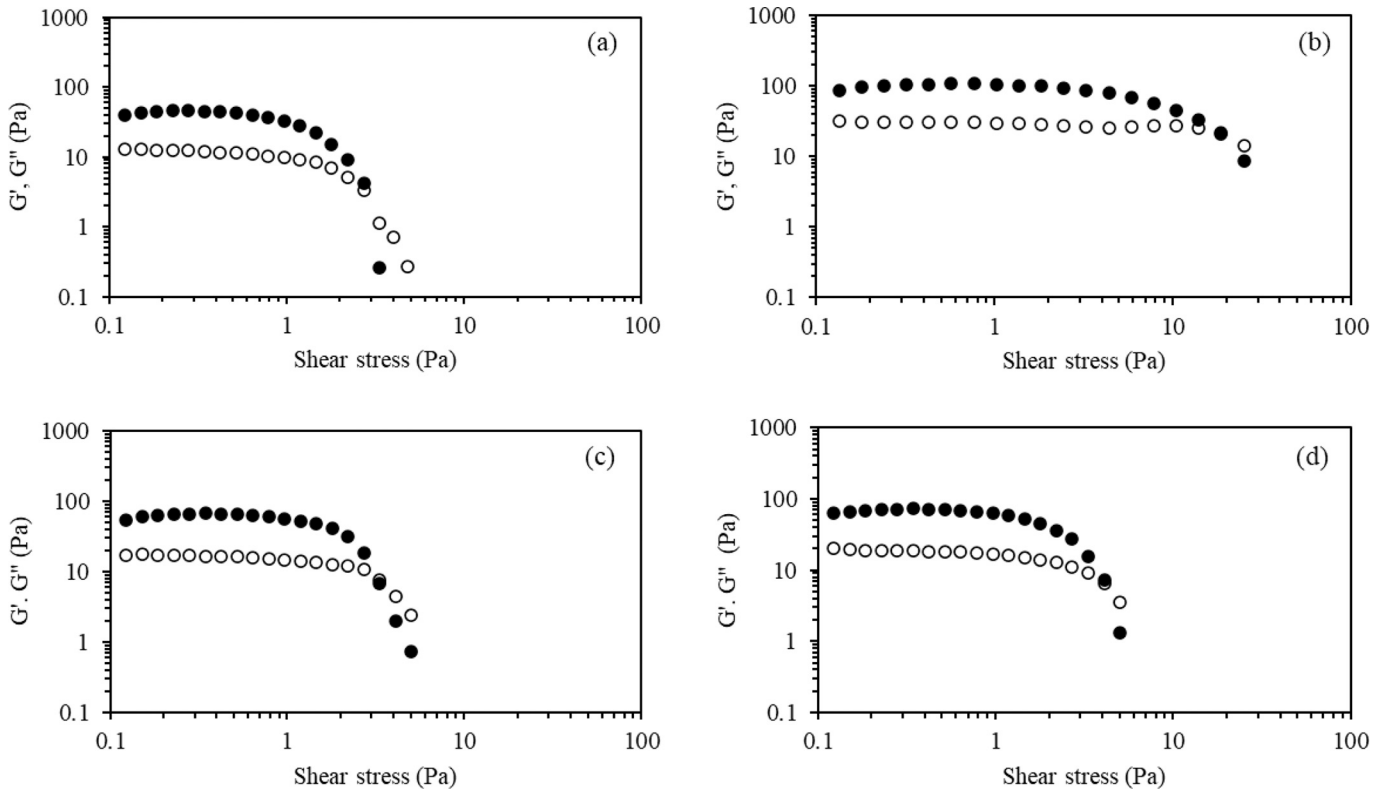

Fig. 2. Storage (G′, closed symbols) and loss (G″, open symbols) moduli as a function of shear stress of açaí suspensions: (a) pure açaí, (b) açaí-pectin, (c) açaí-maltodextrin, and (d) açaí-gum Arabic.

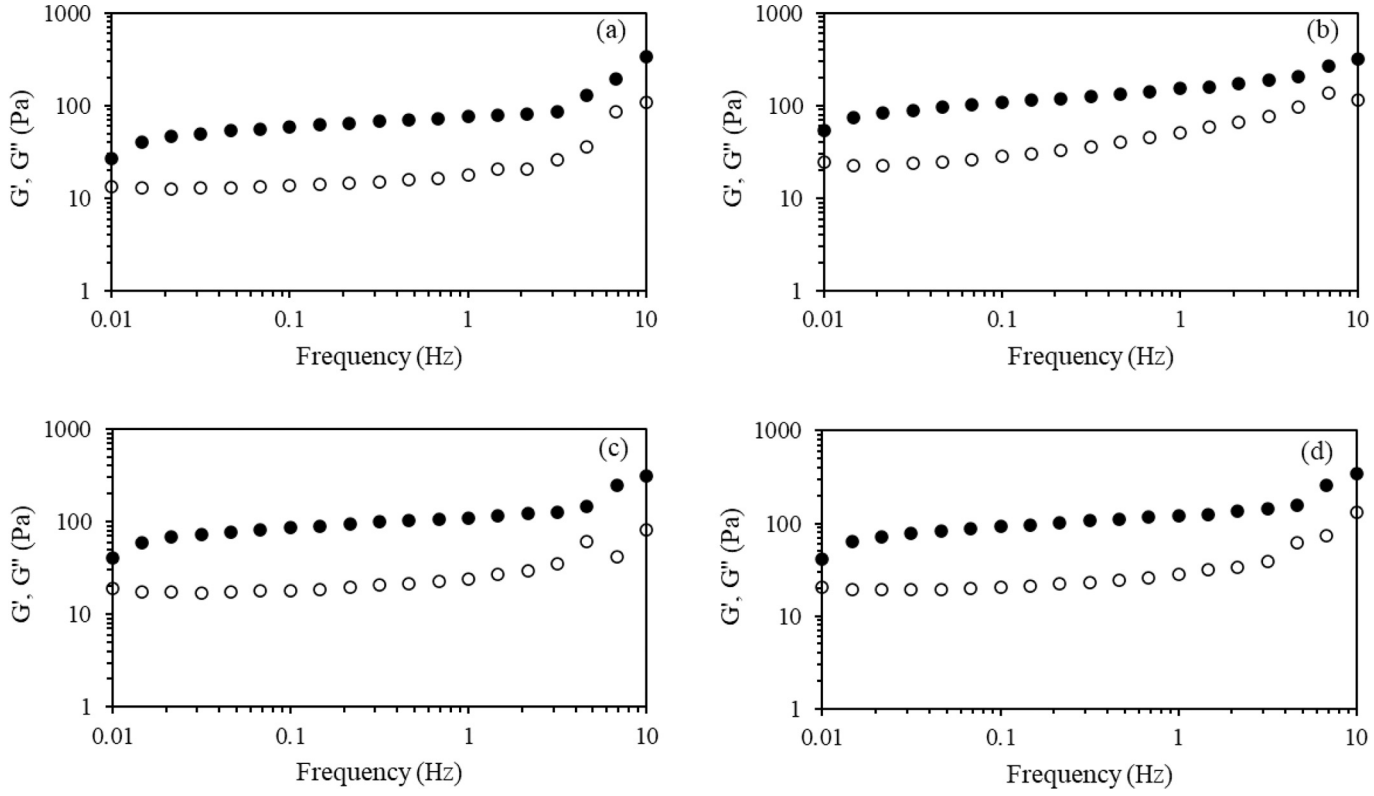

Fig. 3. Storage (G′, closed symbols) and loss (G″, open symbols) moduli as a function of the frequency of açaí suspensions: (a) pure açaí, (b) açaí-pectin, (c) açaí-maltodextrin, and (d) açaí-gum Arabic.

Table 3
Drying time, moisture content, water activity, lipid content, and color of the açaí powders produced by cast-tape drying (CTD) and freeze-drying (FD).

Parameters	Powder samples				
	Pure açaí	Açaí-pectin	Açaí-maltodextrin	Açaí-gum Arabic	Freeze-dried pure açaí
Drying time (h)	0.27	0.53	0.42	0.42	24
Moisture content (g g^{-1}, dry basis)	0.016 ± 0.009^{ab}	0.023 ± 0.006^{a}	0.016 ± 0.003^{ab}	0.013 ± 0.004^{b}	0.023 ± 0.005^{a}
Water activity	0.420 ± 0.033^{a}	0.412 ± 0.039^{a}	0.327 ± 0.033^{b}	0.242 ± 0.046^{c}	0.393 ± 0.050^{a}
Lipid content (g 100 g^{-1}, dry basis)	49.65 ± 0.64^{b}	55.67 ± 3.69^{a}	49.76 ± 0.78^{b}	50.45 ± 1.17^{b}	52.02 ± 0.20^{ab}
Color					
L^*	12.09 ± 0.92^{a}	12.69 ± 0.61^{a}	12.48 ± 0.71^{a}	12.21 ± 0.29^{a}	12.69 ± 0.74^{a}
a^*	3.64 ± 0.39^{b}	4.69 ± 0.25^{a}	3.92 ± 0.21^{b}	3.94 ± 0.12^{b}	4.79 ± 0.59^{a}
b^*	−3.95 ± 0.43^{a}	−3.86 ± 0.36^{a}	−3.79 ± 0.21^{a}	−3.78 ± 0.03^{a}	−3.65 ± 0.27^{a}
ΔE^*	1.33	0.24	0.90	0.99	–

Note: Means followed by different letters in the same row represent significant differences ($p \leq 0.05$), according to Tukey's test.

The oil released during drying causes product losses, in addition to the loss of its nutritional value. Moreover, it leads to operational problems, with frequent dryer downtime for cleaning [34].

3.3. Oil distribution in the dried açaí suspensions

Fig. 4b shows images obtained by confocal microscopy of the oil droplets' distribution in the samples dehydrated by CTD. Large oil droplets were irregularly distributed in the dried açaí suspensions, except for the açaí-pectin sample, in which tiny oil droplets were uniformly distributed. The pectin may have encapsulated the oil present in the açaí juice since high methoxylated pectin is highly hydrophobic and can interact with hydrophobic molecules, such as those from açaí oil [19,20].

3.4. Properties of açaí powders

3.4.1. Moisture content, water activity, lipid content, and color

The moisture content and water activity of açaí powder samples from CTD and FD are shown in Table 3. Even though there were statistical differences among samples ($p \leq 0.05$), moisture content and water activity were below 0.023 g g^{-1} (dry basis) and 0.420, respectively. These values are the same as those reported by Tonon et al. [6] for spray-dried açaí powder added with different carrier agents and Simão et al. [34] for pure açaí powder obtained by conductive thin-film drying at varying drying pressures.

Lipids are the main compounds in the açaí pulp, representing more than 50% of its dry matter (Table 1). As previously discussed, adding pectin to the açaí pulp hindered the oil separation during CTD. Thereby, the açaí-pectin powder showed the highest lipid content compared to the other cast-tape dried açaí powders (Table 3).

The values of the color parameters (L^*, a^*, and b^*) and total color difference (ΔE^*) between the açaí powders produced by CTD and FD are presented in Table 3. The addition of pectin resulted in a significantly higher a^* value (redness) in comparison with the other powder samples produced by CTD ($p \leq 0.05$), which could be attributed to the encapsulation of oil by pectin since the açaí oil color is dark green [35], as well as to the highest concentration of anthocyanins (as discussed in the section 3.4.3). There was no statistical difference between the color of açaí-pectin powder and freeze-dried pure açaí ($p > 0.05$). On the other hand, higher values of color differences were observed between the other powder samples produced by CTD and freeze-dried powder. However, these color differences are slightly noticeable to consumers as ΔE^* is between 0.5 and 1.5 [36]. Murillo-Franco et al. [37] encapsulated açaí pulp by vacuum drying with three wall materials (maltodextrin DE20, gum Arabic, and citrus pectin at a 94/6 pulp/biopolymer weight ratio), and freeze-dried pure pulp was used as control. The authors found a color difference between encapsulated açaí powders and freeze-dried açaí powder.

3.4.2. Fourier transform infrared (FTIR) spectroscopy analysis

FTIR spectroscopy analysis was used to evaluate the molecular structure of açaí powders obtained from CTD and the possible interactions between the açaí components and each biopolymer. Fig. 5 shows the spectra of açaí powders with and without biopolymers and the

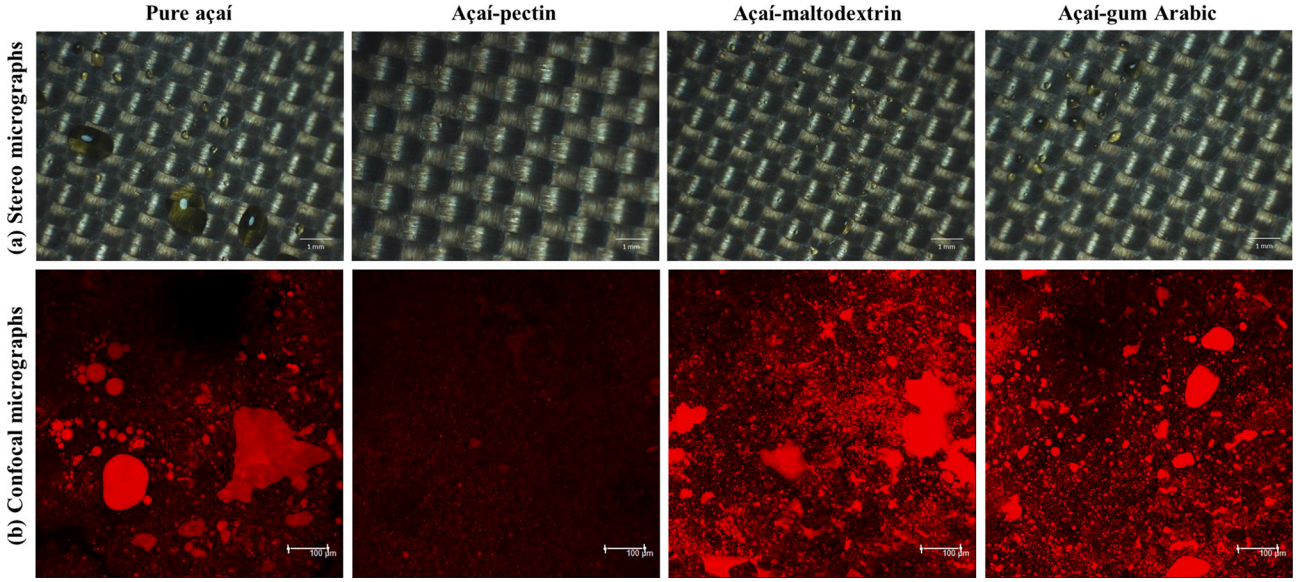

Fig. 4. (a) Stereo micrographs of fiberglass support coated with Teflon® and (b) confocal analysis of the açaí samples after the cast-tape drying (CTD) process.

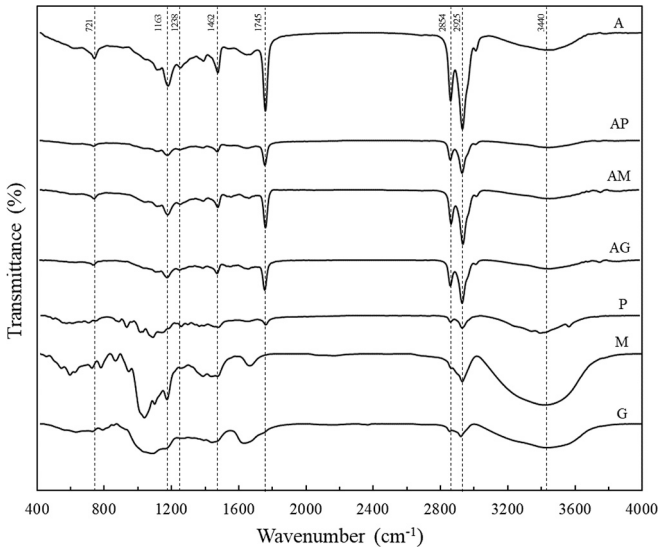

Fig. 5. FTIR spectra of açaí powders produced by cast-tape drying (CTD): pure açaí (A), açaí-pectin (AP), açaí-maltodextrin (AM), and açaí-gum Arabic (AG); and biopolymers: pectin (P), maltodextrin (M), and gum Arabic (G). The dashed lines are added to guide the eyes.

spectrum of biopolymers. Characteristic bands of fatty acids, flavonoids, and polysaccharides functional groups were observed in the FTIR spectrum of pure açaí powder. The broad peak at 3440 cm^{-1} was associated with the hydroxyl group's O—H stretching. Bands around 2925 cm^{-1} and 2854 cm^{-1} were attributed to asymmetric and symmetric CH_2 stretching, respectively, while the band at 1745 cm^{-1} was associated with C═O stretching vibrations of esterified carboxyl groups in fatty acids. The absorption peak at 1462 cm^{-1} was assigned to the C═C stretching vibrations of the aromatic ring in flavonoids and the bending (scissoring) vibrations of the CH_2 and CH_3 aliphatic groups in lipids. Bands at 1238 cm^{-1} and 1163 cm^{-1} were related to C—O stretching vibration in polysaccharides. The peak at 721 cm^{-1} was associated with the C—H bending (rocking) vibrations in fatty acids [38,39]. The typical absorption bands for pectin, maltodextrin, and gum Arabic are summarized in Table S1 (Supplementary data). The FTIR spectra of the açaí powders added with any biopolymers displayed similar peak positions to pure açaí powder, indicating physical rather than chemical interactions between açaí pulp components and biopolymers.

3.4.3. Total monomeric anthocyanin content and antioxidant activity

Açaí is rich in anthocyanins, the compounds responsible for the fruit's dark purple color and the primary antioxidant activity of açaí [3]. The total monomeric anthocyanin content and antioxidant activity determined by the DPPH assay in the açaí powders from CTD and FD, calculated on a defatted-and-dry basis, are presented in Fig. 6a and b, respectively.

Açaí powder with no biopolymers exhibited lower anthocyanin content than freeze-dried açaí powder, as previously reported by Pavan [15] and Souza [16]. The addition of maltodextrin did not protect anthocyanins during CTD, i.e., the açaí-maltodextrin powder did not display a statistical difference from cast-tape dried pure açaí pulp ($p > 0.05$). In contrast, the anthocyanin contents of açaí-gum Arabic and açaí-pectin powder were significantly higher than that of pure açaí powder produced by CTD ($p \leq 0.05$). Furthermore, açaí-pectin powder did not significantly differ from that of freeze-dried açaí powder ($p > 0.05$). These results may be related to the biopolymers' chemical

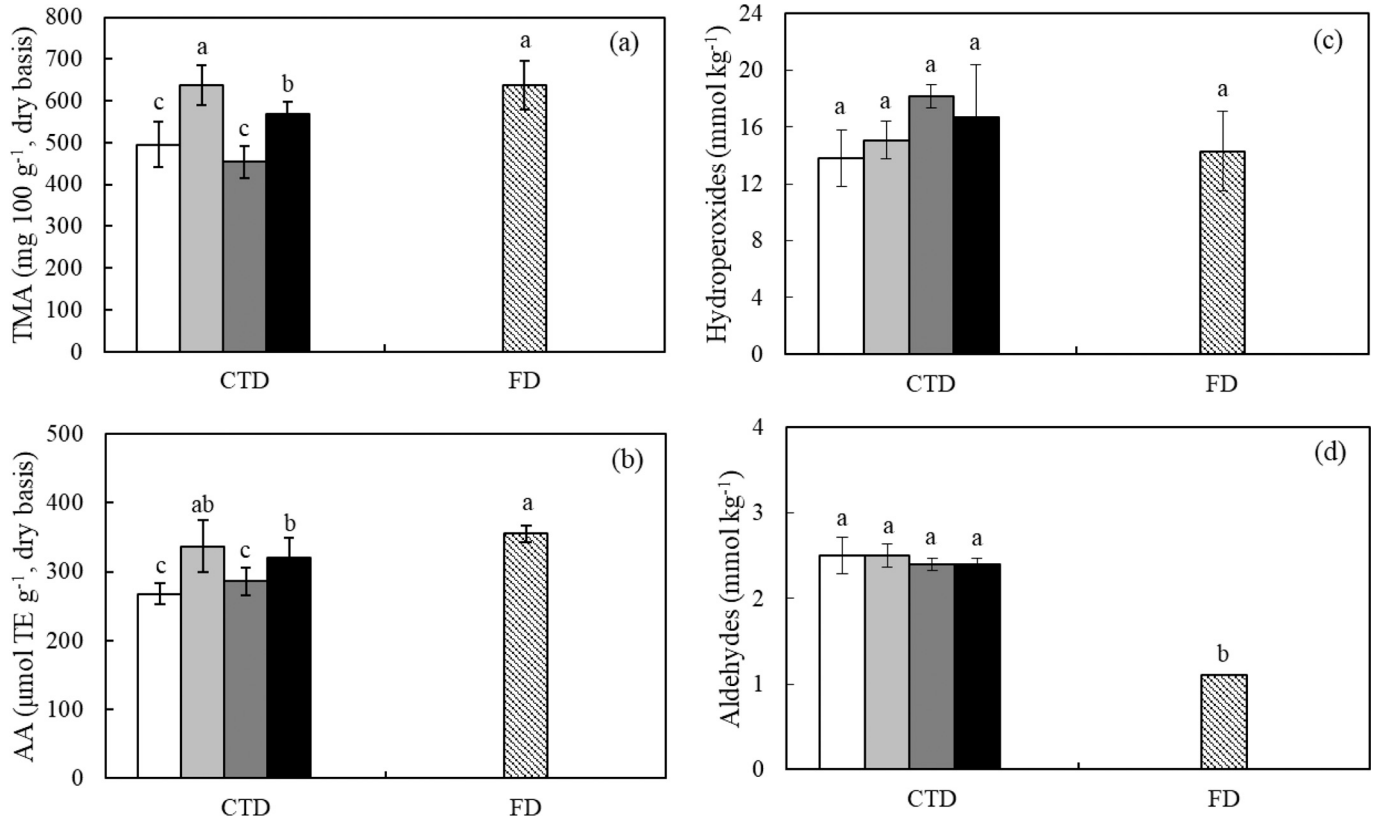

Fig. 6. (a) Total monomeric anthocyanin content (TMA), (b) antioxidant activity (AA), and concentration of (c) hydroperoxides and (d) aldehydes in açaí powders produced by cast-tape drying (CTD): pure açaí (white column), açaí-pectin (light grey column), açaí-maltodextrin (dark grey column), and açaí-gum Arabic (black column); and pure açaí powder from freeze-drying (FD). The different letters represent significant differences ($p \leq 0.05$).

structure and their interactions with anthocyanins. Lower DE maltodextrins contain many long chains and cannot create a dense and less oxygen-permeable network, resulting in poor protection of oxygen-sensitive compounds, such as anthocyanins [37,40]. Conversely, the protection of anthocyanins by gum Arabic has been attributed to the interaction between anthocyanins and glycoprotein fractions of gum Arabic through hydrogen bonding [41]. Pectin has been demonstrated to interact with anthocyanins through electrostatic interactions, hydrogen bonding, and hydrophobic interactions; the structural flexibility of this biopolymer influences these interactions [41,42].

The antioxidant activity of samples followed a similar tendency to the anthocyanin content. The addition of pectin resulted in an açaí powder with significantly higher antioxidant activity in comparison with the cast-tape dried pure açaí sample and comparable to that of freeze-dried powder. Other authors have already reported a relationship between anthocyanin content and antioxidant activity in açaí products [8,26].

3.4.4. Lipid oxidation products

Açaí contains a high unsaturated fatty acids content, representing approximately 74% of all fatty acids. Oleic, linoleic, and linolenic acids constitute around 56%, 13%, and 1% of the total fatty acids in açaí, respectively [1]. Açaí is potentially prone to lipid oxidation during processing and storage, a chemical reaction responsible for forming primary and secondary oxidation compounds. The formation of hydroperoxides and aldehydes during drying (primary and secondary lipid oxidation products, respectively) was determined from açaí powder samples by ^{1}H NMR. Fig. 7 shows ^{1}H NMR spectra of the açaí oils extracted from powdered samples produced by CTD and FD; the assignment of hydroperoxide and aldehyde signals, according to Merkx et al. [29], is given in Table S2 (Supplementary data). The oxidation process of linolenic acyl groups formed cyclized hydroperoxides (peak 1), 12- and 13-hydroperoxide isomers (peak 2), and 9- and 16-hydroperoxide isomers (peak 3). The cyclized hydroperoxides are generated by the internal cyclization of 12- and 13-hydroperoxides [43]. Two conjugated diene hydroperoxides (peak 4), i.e., 9-hydroperoxy-trans-10, trans-12-octadecadienoate (trans,trans-9-OOH) and 13-hydroperoxy-trans-9,trans-11-octadecadienoate (trans,trans-13-OOH), were formed from the oxidation of linoleic acyl groups. The formation of 8-hydroperoxy-cis-9-octadecenoate (cis-8-OOH) and 11-hydroperoxy-cis-9-octadecenoate (cis-11-OOH) isomers (peak 5) resulted from the degradation of oleic acyl groups [29,43]. Although a difference in the intensities of signals in the hydroperoxide region can be observed when comparing the spectra of açaí powders (Fig. 7a), the concentration of the primary lipid oxidation products did not show a statistically significant difference among samples ($p > 0.05$) (Fig. 6c).

Secondary oxidation compounds were also generated during the drying process of açaí samples due to the extended exposure of hydroperoxides to oxidation conditions. Aldehydes were the secondary oxidation products detectable by ^{1}H NMR with signals between δ 9.8 and 9.4 ppm (Fig. 7b). n-alkanals (peak 6), 4-hydro(pero)xy-trans-2-alkenals (peak 7), and trans,trans-2,4-alkadienal and 4,5-epoxy-trans-2-alkenals (peak 8) were the aldehydes found in the samples. Similar compounds were reported by Guillen and Goicoechea [44], who evaluated corn oil, an oil rich in polyunsaturated groups, stored at room temperature. There was no statistical difference among the concentrations of total aldehydes in the CTD samples ($p > 0.05$) (Fig. 6d). However, the concentration of aldehydes was significantly lower in the açaí oil from freeze-dried açaí ($p \leq 0.05$), possibly due to less formation of 4-hydro(pero)xy-trans-2-alkenals, trans,trans-2,4-alkadienal, and 4,5-epoxy-trans-2-alkenals, explained by the lower temperature and oxygen exposure during drying. The formation of 4-hydro(pero)xy-trans-2-alkenals and 4,5-epoxy-trans-2-alkenals, which are oxygenated α,β-unsaturated aldehydes, requires special attention since they have been considered possible causative agents of degenerative diseases [45,46]. The occurrence of these compounds has also been observed in different dried foods rich in unsaturated fatty acids, such as dry nuts [47], dried seaweeds [48], and linolenic acid-enriched egg yolk powder [49].

To the best of our knowledge, the present study reports for the first time the nature and proportions of these different aldehydes generated in the açaí oil during pulp processing.

4. Concluding remarks

Adding biopolymers at a low concentration to açaí pulp plays an essential role in CTD processing and improves the dried pulp's characteristics. Adding 3% of pectin was beneficial for the film-forming during drying. It resulted in a stronger and less brittle dried film structure than pure açaí, açaí-maltodextrin, and açaí-gum Arabic suspensions. The addition of different biopolymers demands adjustments of the spreading gap, influencing the drying time that requires adjustment of the belt velocity. The pectin-containing sample exhibited the longest drying

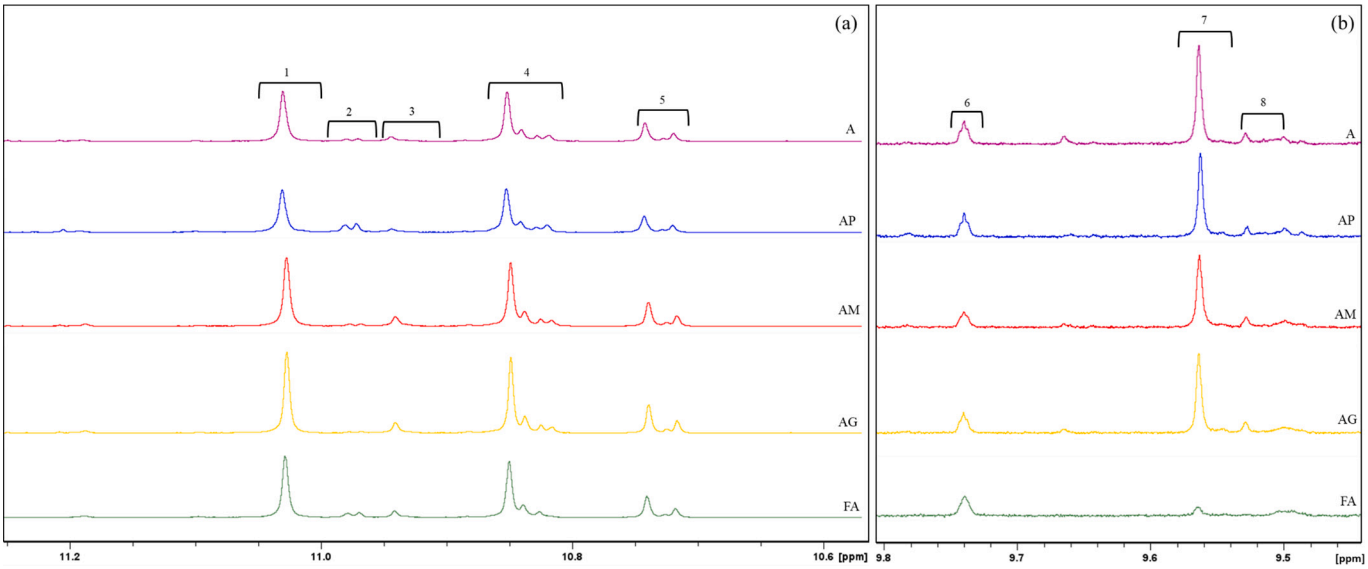

Fig. 7. ^{1}H NMR spectra of the (a) hydroperoxide region (δ 11.25–10.6 ppm) and (b) aldehyde region (δ 9.81–9.44 ppm) of the açaí powders produced by cast-tape drying (CTD): pure açaí (A), açaí-pectin (AP), açaí-maltodextrin (AM), and açaí-gum Arabic (AG); and freeze-dried pure açaí powder (FA).

time; however, adding only 3% of pectin hindered the oil release from the pulp and formed a cohesive structure during drying, which helped the removal of the dried material as a film from the cast-tape dryer belt. Furthermore, adding pectin to açaí pulp protected the anthocyanins (retention of 97%) and antioxidant activity that were similar to those of freeze-dried açaí pulp; also, these dried samples stood out for their attractive color. Finally, we showed that adding 3% of pectin, maltodextrin, or gum Arabic to açaí pulp was not effective in slowing down lipid oxidation during CTD. In other words, there was a production of aldehydes (secondary lipid oxidation compounds) in açaí powders from CTD higher than that observed in FD powder. Therefore, additional studies that assess sensory properties and the benefits/risks of consumption of these powders are recommended. Further research can potentially transform the production of açaí powder, rendering it more accessible using CTD while concurrently preserving its bioactive properties.

CRediT authorship contribution statement

Raquel S. Simão: Conceptualization, Formal analysis, Investigation, Methodology, Validation, Writing – original draft, Writing – review & editing. **Jaqueline O. de Moraes:** Conceptualization, Methodology, Supervision, Writing – review & editing. **Lu Zhang:** Resources, Supervision, Writing – review & editing. **Anja Schröder:** Formal analysis, Investigation, Methodology. **Bruno A.M. Carciofi:** Conceptualization, Methodology, Resources, Writing – review & editing. **Maarten A.I. Schutyser:** Resources, Supervision, Writing – review & editing. **João B. Laurindo:** Conceptualization, Funding acquisition, Methodology, Resources, Supervision, Writing – review & editing.

Declaration of competing interest

The authors declare no conflict of interest.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.powtec.2024.119424.

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