

## Tablets containing açai (*Euterpe* spp.) dried extract for dietary supplementation: A study of functional properties

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### Abstract

Açai is a very popular functional food in the Amazon due to its nutritional properties, its high caloric value, and its high content of anthocyanins and phenolic compounds, which mainly confers antioxidant and anti-inflammatory activities. Thus, this study aimed at the development of immediate-release tablets containing açai dried extract by direct compression. The extract was subjected to physical evaluation of the powder, quantification of total polyphenol (TP) content (mg EAG/g), total anthocyanin content (mg C3G/100 g), cyanidin-3-rutinoside marker ( $\mu\text{g}$  C3R/g) by LC-MS, obtention of the tablet, and their physical-mechanical evaluation. Açai dried extract showed spherical shape particles, with smooth and rough surfaces and heterogeneous sizes, which are characteristic of dried powders when obtained by spray drying. In addition, the extract presented TP content ( $23.307 \pm 2.190$  mg EAG/g), total anthocyanins (10.52 mg C3G/100 g), and cyanidin-3-rutinoside ( $42 \mu\text{g}$  C3R/g). Tablets had good mechanical properties, good thermal stability under  $200^\circ\text{C}$ , and  $3.65 \mu\text{g}$  C3R/g of cyanidin-3-rutinoside content per tablet unit. The results obtained in this study showed the possibility of obtaining tablets containing açai dried extract with good mechanical properties and antioxidant activity aiming its use as a dietary supplement.

**Keywords:** drying technology; direct compression; cyanidin-3-rutinoside; LC-MS; spray drying; mechanical property.

**Practical Application:** Tablets containing açai dried extract were obtained by direct compression with antioxidant properties, which were characterized by the presence of anthocyanidins, in particular, cyanidin-3-rutinoside. The developed formulation had good mechanical properties, good thermal stability, and  $3.65 \mu\text{g}$  C3R/g of cyanidin-3-rutinoside content per tablet unit and may be applied as a promising food supplement.

## 1 INTRODUCTION

The species *Euterpe* spp. is a palm tree native to the Brazilian Amazon, adapted to high-temperature conditions, rainfall, and relative humidity (Carvalho et al., 2017). Its fruit called açai is one of the most popular foods in the Amazon, with significant socioeconomic importance for the region, as it is a food with high nutritional and caloric value and its processed pulp is marketed worldwide due to its nutritional and functional benefits (da Silveira et al., 2019; Tonon et al., 2010). Thus, it stands out in the growing dietary supplement market for presenting a variety of vitamins, minerals (Mn, Fe, Zn Cu, and Cr), carbohydrates, fibers, and proteins (da Silva Carvalho et al., 2016; Yamaguchi et al., 2015). In addition, it is a food with high energy value because it contains a high content of vegetable oil, about 53% on a dry basis, and is a source of essential fatty acids, predominantly monounsaturated fatty acids (68–71%) and polyunsaturated fatty acids (7.7–10.6%) (Loureiro Contente et al., 2020; Nascimento et al., 2008).

Plants of the genus *Euterpe* have been widely studied. Therefore, açai pulp stands out for its functional properties due to the antioxidant activity related to its content of anthocyanins

(responsible for the violet color of the fruit) and phenolic compounds (Schulz et al., 2021; Tonon et al., 2008; 2010). From the extract and oil, high concentrations of bioactive compounds are distinguishable and promote a series of biological properties in the plant species, such as antioxidant, anticarcinogenic, anti-inflammatory, and antimicrobial activity, acting in the prevention of oxidation of low-density protein (LDL), antinociceptive, antitumor, antileishmanial, antiaging, cardiovascular, and neuroprotective diseases (Magalhães et al., 2020; Park et al., 2014; Santamarina et al., 2019; Schulz et al., 2021).

In view of this, dietary supplements contain a concentrated form of a food-bioactive compound and can be used for the purpose of improving an individual's health. In this context, Brazil obtained a regulatory framework for dietary supplements, which aimed at contributing to the population's access to safe and quality food supplements, in order to reduce information asymmetry existing in the market, facilitate sanitary control and the risk management of these products, eliminate unnecessary obstacles to commercialization and innovation, and also simplify current regulatory stock. Therefore, Resolution No. 243/2018, which provides for the health requirements of food

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supplements, is supervised by the National Health Surveillance Agency (ANVISA). Such changes arose especially due to advances in research, development, and innovation in this category of products, as well as the growing demand of the population, which has been seeking healthier lifestyles.

Tablet production can be performed using three basic techniques: wet granulation, dry granulation, and direct compression. However, direct compression is considered the technique of choice for the production of tablets that contain thermolabile and moisture-sensitive active components (Cianchino et al., 2020), as is the case with açai pulp extract. Therefore, direct compression formulations consist of basic ingredients responsible for increasing the bulk and improving the flow of the blend, biodisponibility, and active ingredients. Therefore, the excipients have to compensate for poor flow and compression properties, which are often inherent to the active extract (Paul & Sun, 2018).

In this way, this study proposes the development of tablets containing açai dried extract of the pulp obtained by direct compression and to evaluate the anthocyanin content, in addition to their antioxidant activity and their physical-mechanical characteristics, aiming at its use as a dietary supplement in the population diet.

## 2 MATERIALS AND METHODS

### 2.1 Plant materials

The açai (*Euterpe* spp.) dried extract by spray drying (Lot: 025050), referred to from now on in this study as açai dried extract (ESAc), was acquired from Catedral Laboratory LTDA (Vespasiano, Minas Gerais).

### 2.2 Reagents

Cyanidin-3-rutinoside standard, potassium chloride p.a., sodium acetate p.a., hydrochloric acid p.a., acetone p.a., formic acid p.a., methanol p.a., formic acid HPLC, acetonitrile HPLC-grade, Folin Ciocalteu reagent (2N), and standard gallic acid were purchased from Sigma & Aldrich.

### 2.3 Physical-chemical characterization of açai dried extract

#### 2.3.1 Water activity ( $A_w$ )

Approximately 1.0 g of ESAC was deposited in a sample holder and analyzed in Aqua Lab equipment (4TEV, Decagon Devices) at 25°C.

#### 2.3.2 Particle-size distribution

A quantity of 2.0 g of ESAC was weighed, and three drops of sodium pyrophosphate (chemical dispersant) were added. The sample was analyzed using laser diffraction equipment (Fritsch brand, model Analysette 22), with a reading range of 0.08–2000 µm. Particle-size variation of the extract (ESAC) was determined by parameters D10, D50, and D90 (Ghribi et al.,

2015). From these data and using Equation 1, the polydispersity index (PDI) was obtained for particle size:

$$IP = ((D90 - D10)/D50) \quad (1)$$

### 2.4 Determination of morphology

The ESAC was deposited in a sample holder with the aid of carbon adhesive tape and metalized with gold/palladium in metallizer (SC7620, Quorum Technologies) to allow necessary electrical conductivity in the image formation process. Electromicrographs were obtained using a scanning electron microscope (model VEGA3 SD, Tescan, USA) with an electron beam current of 85–90 µA and an acceleration voltage of 10.0 kV.

### 2.5 Analytical methods

#### 2.5.1 Extraction and quantification of total polyphenols in açai dried extract

Extraction of TP from ESAC was performed according to Rufino et al. (2010), with modifications. A quantity of 4.0 g of ESAC was weighed, and a water/methanol solution (50/50 v/v) (20 mL) was added, remaining at rest for 1 h at room temperature, protected from light. Then, the solution was centrifuged at 11,000 rpm for 20 min at 20°C, and the supernatant was filtered and stored in a volumetric flask (50 mL). Acetone/water solution (70/30 v/v) (20 mL) was added to the precipitate, repeating the same procedure as in the first extraction. The supernatant of the acetone/water solution was filtered and added to the supernatant of the water/methanol solution. The extract was kept in a freezer (-18°C) until analysis.

Quantification of TP followed the Folin Ciocalteu method, according to Aliakbarian et al. (2011). In test tubes, 2.4 mL of distilled water and 0.1 mL of ESAC (and standard gallic acid for standard curve construction) were added. Then 0.25 mL of Folin Ciocalteu reagent (2N), 0.5 mL of saturated sodium carbonate, and 1.75 mL of distilled water were homogenized, and after 60 min, reading was performed (725 nm) in a UV/Vis spectrophotometer (Shimadzu®, UV-1800). TP content was expressed in mg gallic acid equivalent (GAE)g<sup>-1</sup> of açai dried extract, which was obtained using an external standard curve of gallic acid at concentrations of 50–250 µg.mL<sup>-1</sup>.

#### 2.5.2 Extraction and quantification of total anthocyanins in açai dried extract

For extraction, ESAC (25.0 g) was placed in an amber flask, and 70% hydroethanolic solution (50 mL) acidified with 1% HCl was added, up to pH 1.0. The extraction time used was 24 h, at a temperature of 25°C. Subsequently, the solution was vacuum filtered to separate solid material and centrifuged at 11,000 rpm for 20 min in a centrifuge (Eppendorf, 5804R) (Chandrasekhar et al., 2012).

Quantification of anthocyanins was performed by differential pH method (Giusti & Wrolstad, 2001), using two buffer systems: potassium chloride pH 1.0 (0.025 M) and sodium acetate pH 4.5 (0.4 M). The supernatant was diluted to reach an absorbance between 0.3 and 0.8 at 510 nm. An aliquot of the

supernatant (1.0 mL) was diluted with 9.0 mL of the corresponding pH 1.0 and pH 4.5 buffer solutions, homogenized, and stored for 20 min in the absence of light. Subsequently, absorbance was measured at the maximum wavelength (700 nm), and a blank was prepared with distilled water. The final absorbance of the sample was calculated using Equation 2:

$$A = (A_{\lambda_{max,vis}} - A_{700nm})_{pH\ 1.0} - (A_{\lambda_{max,vis}} - A_{700nm})_{pH\ 4.5} \quad (2)$$

The concentration of total anthocyanins was calculated using Equation 3, and the results were expressed in cyanidin-3-glycoside content:

$$ATM = (A.PM.FD.1000)/(\epsilon.L) \quad (3)$$

where:

ATM: monomeric total anthocyanins (mg.100 g<sup>-1</sup> açai dried extract);

A: absorbance difference (Equation 2);

PM: molecular weight of cyanidin-3-glycoside (449.2 g.mol<sup>-1</sup>);

FD: dilution factor;

$\epsilon$ : molar absorptivity (26,900 L.mol<sup>-1</sup>.cm<sup>-1</sup>);

L: optical path (1.0 cm).

## 2.6 Obtaining tablets

Tablets were obtained by direct compression in a rotary press (Lemaq, model monopress LM 6000 C/H), using biconcave punches (11.5 mm) at a speed of 10 rpm. The formulation composition contained açai dried extract (50.15%), lactose (21.71%), microcrystalline cellulose (18.96%), talc (4.59%), and magnesium stearate (4.59%). Tablets were packed in amber glass bottles and properly identified for later chemical analysis and physical-mechanical evaluation.

## 2.7 Physical analysis of tablets

The determination of the uniformity of mass, as described in EP 2.9.5, was performed for all tablets. The established tolerance limit was no more than two tablets outside the  $\pm 3.0\%$  variation limit for tablets weighing more than 250 mg. As described in EP 2.9.7., the difference between the initial and final weight called friability was measured as a function of the percentage of powder loss, and the hardness of tablets was determined by the force required to crush or break them.

## 2.8 Determination of cyanidin-3-rutinoside in the dry extract and in tablets containing açai dried extract by ultra-performance liquid chromatography coupled to a mass spectrometer

### 2.8.1 Anthocyanin's extraction

The methodology described by Park et al. (2014) was used, with modifications. For anthocyanin extraction, 400 mg of

ESAc and 1.0 g of a sample regarding two ground tablets were weighed. Extract and ground tablets were solubilized separately in a water/formic acid solution (95/5 v/v) (2.0 mL). The solution was vigorously vortexed (Vixar, VM3000) for 5 min, subjected to an ultrasound bath (Kondentech, Digital Ultrasonic Cleaner) for 20 min, and centrifuged at 8,000 rpm for 15 min. Subsequently, the solution was filtered through a syringe filter (PVDF 0.22  $\mu$ m) and injected into a chromatograph (Agilent Technologies) UHPLC (model 1290) with column oven (G1311A), diode array detector (DAD), and pump (G4204A) coupled to a triple quadruple type spectrometer (QQQ) (model 6460C).

### 2.8.2 Identification and quantification of cyanidin-3-rutinoside

Identification and quantification analysis of cyanidin-3-rutinoside in ESc and tablets followed the methodology described by Fang and Bhandari (2011) with modifications. The content of cyanidin-3-rutinoside marker in dry extract and açai tablets was expressed in micrograms of cyanidin-3-rutinoside ( $\mu$ gC3R) and was obtained using a cyanidin-3-rutinoside external standard curve at concentrations of 1.5625 at 50  $\mu$ g.mL<sup>-1</sup>. Analysis was performed in an ultra-efficiency liquid chromatograph coupled to a mass spectrometer (LC-MS), in a Zorbax Eclipse plus C18 column (2.1 $\times$ 50 mm), with a diameter of 1.8  $\mu$ m, a heating temperature of 25°C, an injection volume of 5  $\mu$ L, and a flow of 0.25 mL.min<sup>-1</sup>. Mobile phase was (A) water acidified with formic acid (pH 3), (B) 80% acetonitrile in ultrapure water, elution profile of 0–1 min, 5% phase (B), 1–8 min, 5–25% phase (B), 8–12 min, 25–30% phase (B), 12–14 min, 30–55% phase (B), 14–16 min, 55% phase (B), 16–18 min, 55–5% phase (B), and 18–19 min, 5% phase (B).

The LC-MS features ESI positive ionization mode with a capillary voltage of 3,500 V, a nitrogen flow of 5.0 L.min<sup>-1</sup>, a fragmentation of 80 turns, an electrospray source temperature of 300°C, and monitored ions MS/MS from 595 to 449.287.

### 2.8.3 Thermoanalytical profile

ESAc and tablets were submitted to thermogravimetric analysis (TGA) using the thermal analyzer model DTG-60 (Shimadzu, Kyoto, Japan). Samples were weighed (2.5 mg) in a platinum crucible and analyzed under a nitrogen atmosphere flowing at a rate of 50 mL.min<sup>-1</sup>. The experiments were conducted at a temperature range of 25–600°C and a heating rate of 10°C.min<sup>-1</sup> (Silva da Costa et al., 2022).

## 3 RESULTS AND DISCUSSION

### 3.1 Physical analysis of açai dried extract

ESAc showed good microbiological stability, indicated by reduced water activity which is a measure of active water availability in a product (Table 1) (da Silva Carvalho et al., 2016). This result can be attributed to the drying efficiency of the extract as a function of the inlet air temperature of the spray dryer, resulting in dry powders with reduced water activity (Laokuldilok & Kanha, 2015), favoring greater heat transfer and efficient evaporation of water present in samples.

Furthermore, in this study, maltodextrin was used as a drying aid for açai extract, which can influence the water activity content of spray-dried extracts as the adjuvant used (Laokuldilok & Kanha, 2015). Studies carried out for the characterization of *Euterpe edulis* M. dry extract, which also used maltodextrin (10DE and 30DE) as a drying aid and an inlet air temperature of 160°C, showed reduced water activity values of  $0.245 \pm 0.019$  and  $0.314 \pm 0.021$ , confirming the efficient microbiological stabilization of extracts derived from fruits and dried by spray drying (da Silva Carvalho et al., 2016).

ESAc showed that particle-size distribution in dry extracts is related to the size of particles formed by the drying process (spray drying) (Table 1). Furthermore, powders with a particle size smaller than 100 µm exhibit low flow properties, so the knowledge of particle size is an important parameter during pre-formulation studies to obtain tablets, as this may affect the flowability, rehydration, solubility, and compaction of powders (Paim et al., 2016). According to these results, ESAC presented a PDI, characterizing the heterogeneous distribution of particles (Table 1), as a PDI lower than 2.0 is normally considered a narrow distribution of particles dried by spray drying (Fernandes et al., 2016).

### 3.2 Morphology

Açai dried extract particles showed a spherical shape with a predominance of rough surfaces and different size particles, corroborating the result of particle-size distribution with a tendency to agglomeration and low fluidity of powders (Figure 1), which tend to decrease the flow. The external microstructure of microparticles with irregular surfaces, spherical shapes, and heterogeneous sizes is a very common feature of spray-dried samples (da Silva Carvalho et al., 2016; Valente et al., 2019).

Meanwhile, the morphology of particles can be affected by the type of adjuvant concentration, temperature, or drying

speed (Simon-Brown et al., 2016). In the study of *Euterpe oleracea* M. dry extract using maltodextrin 10 DE as a drying aid in the temperature range from 138 to 202°C, particles with different shapes and sizes were observed in the extract. Therefore, the authors suggested that a higher drying temperature would mean a shorter contact time, which would lead to faster powder formation and prevent particles from affecting the texture of a food matrix, as long as the threshold diameter would be less than 100 µm shrinkage. However, higher drying temperatures can also lead to anthocyanin degradation, which is undesirable (Valente et al., 2019).

### 3.3 Determination of total polyphenol and anthocyanin content in açai dried extract

ESAc showed a TP value of  $23.307 \pm 2.190 \text{ mg}_{\text{GAE}} \cdot \text{g}^{-1}$  and a total anthocyanin content of 10.52 mg of cyanidin-3-glycoside.100 g<sup>-1</sup>. A dry extract of *Euterpe edulis* M. juice, used as a probiotic, with maltodextrin 10 DE as a drying adjuvant, showed values of  $12.294 \pm 0.235 \text{ mg}_{\text{GAE}} \cdot \text{g}^{-1}$  and  $574.7 \pm 39.4 \text{ mg}$  of cyanidin-3-glycoside.100 g<sup>-1</sup>, respectively (Paim et al., 2016). A dry extract of *Euterpe oleracea* M., with drying agents maltodextrin 10 DE and maltodextrin 20 DE, obtained values of  $137.96 \pm 1.71$  and  $135.42 \pm 1.24 \text{ mg}_{\text{GAE}} \cdot \text{g}^{-1}$ , respectively, and  $269.38 \pm 2.15 \text{ mg}$  of cyanidin-3-glycoside.100 g<sup>-1</sup> to anthocyanin (Costa et al., 2015; Tonon et al., 2009).

TP content and the total anthocyanin content of ESAC may be related to the storage period of the dry extract and different growth conditions, as well as seasonal, genetic, and agronomic factors of the fruit, which considerably influence phenolic composition content in plant tissues (Borges et al., 2011; Tonon et al., 2010).

Another factor is the use of maltodextrin with a higher equivalent dextrose unit, which is more sensitive to high temperatures during the drying process, as it contains shorter chains, can lead to structural deformations during heating processes, and can also cause variations in bioactive compounds content (Franceschinis et al., 2014).

### 3.4 Obtaining the formulation

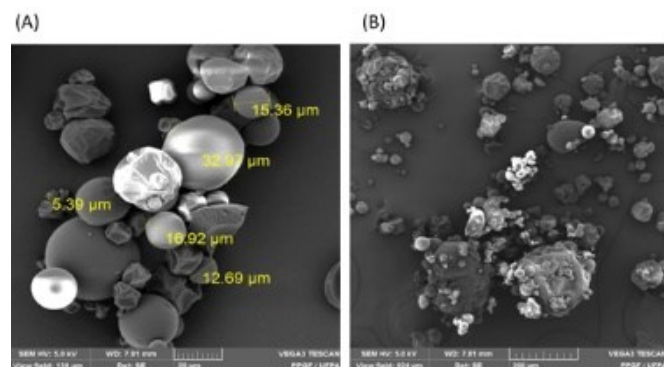
#### 3.4.1 Physical mechanical evaluation of tablets containing açai dry extract

Tablets containing açai dried extract showed characteristic color, smooth surface, and circular and grooved shape, with an average weight of  $550.5 \pm 1.7 \text{ mg}$ . The prepared tablets were evaluated for uniformity of weight, hardness, and friability. The batch of the manufactured tablet was acceptable according to the European Pharmacopeia, and the variation in weight was less than 3.0%. This parameter confirmed the consistency of dosage units during compression. As soon as to friability, the formulation had a loss weighing less than 1% ( $0.86 \pm 0.2$ ), which would determine a higher resistance to abrasion. The hardness of the tablet was also considered acceptable ( $4.45 \pm 0.1 \text{ kg/cm}^2$ ), and it is an important parameter to estimate the disintegration time, as the resistant tablet does

**Table 1.** Physical properties of açai dried extract.

Parameters	Results
Water activity (Aw)	$0.302 \pm 0.012^*$
Particle size (µm)	< 157.3
Polydispersity index (PDI)	3.56

\*Result is expressed by means ± standard deviation.



**Figure 1.** SEM of açai spray-dried extract particles. (A) Viewed at 1,490x. (B) Viewed at 225x.

not disintegrate in the time required to satisfy the dissolution specifications (Cianchino et al., 2020).

Thus, these results guarantee their physical integrity and allow adequate support to mechanical shocks from manufacturing processes, such as coating, dredging, blistering, packaging, transport, storage, and handling. Hence, tablets containing açai dried extract show themselves as a promising product from a technological point of view, as they presented good mechanical resistance during their acquisition, which could be extended to user's handling.

### 3.5 Characterization of formulation

#### 3.5.1 Determination of cyanidin-3-rutinoside marker in açai dried extract and in their tablets by ultra-performance liquid chromatography coupled to mass spectrometry

Identification of the marker (cyanidin-3-rutinoside) was made by comparing retention time (RT) using UV-Vis spectrum and MS/MS analyses of ESAC chromatogram (Figure 2A) and tablets containing the extract (Figure 2B) with cyanidin-3-rutinoside standard chromatogram (Figure 2C). MS/MS analysis of ESAC and tablets showed precursor ions at  $m/z$  corresponding to the molar mass of cyanidin-3-rutinoside and the product ions at  $m/z$  corresponding to the molar mass of cyanidin-3-glycoside and cyanidin aglycone (Table 2). These results confirmed that açai dried extract presented 42  $\mu\text{g}$  of cyanidin-3-rutinoside.g<sup>-1</sup> and the two analyzed tablets showed a value of 7.30  $\mu\text{g}$  of cyanidin-3-rutinoside, with a concentration of 3.65  $\mu\text{g}$  cyanidin-3-rutinoside.g<sup>-1</sup> for each tablet.

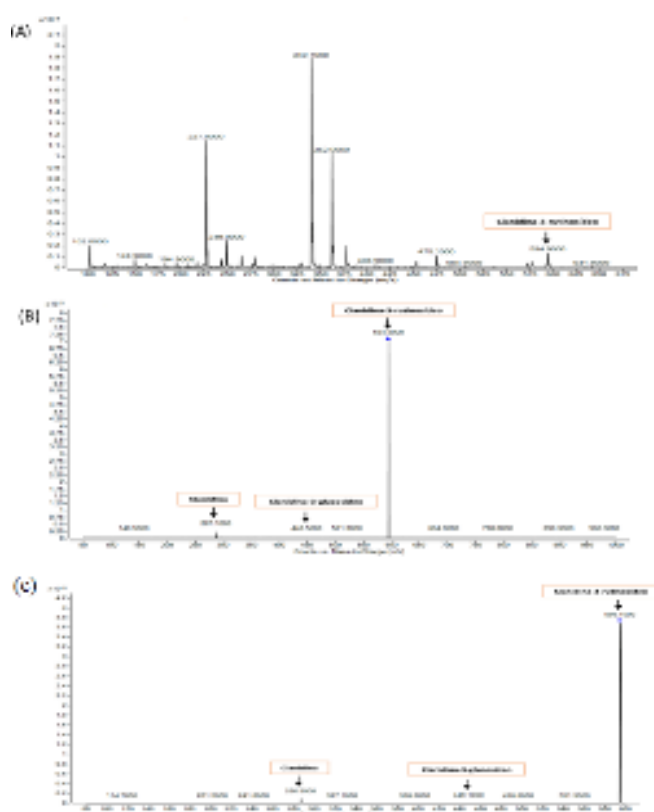
### 3.6 Thermoanalytical profile

Thermoanalytical studies of ESAC and tablets were performed to analyze the thermal resistance in which the product will be submitted during the production stages.

TG/DTG curves of the extract showed two mass loss events (Figure 3A). The first event can be attributed to water evaporation and the presence of volatile compounds and the second event where the greatest mass loss occurred, and degradation can be probably attributed to pyrolytic decomposition as a result of carbonization of organic compounds present in the extract, such as starch, fibers, and residual lipid material, as well as maltodextrin, used as adjuvant aid for açai extract (Table 3). Such values corroborate the results found for other tropical fruit derivatives, such as microencapsulated cupuassu seed by-product, which also used maltodextrin (16.5–19.5 DE) as drying adjuvant and wall material for the encapsulation process, showing decomposition temperatures of 198, 41–237, and 47°C

and  $\Delta m = 28.95\%$  (Silva da Costa et al., 2022). Thus, it was found that the use of maltodextrin as an encapsulation agent does not change qualitatively the nature of thermal events in tropical fruit extract.

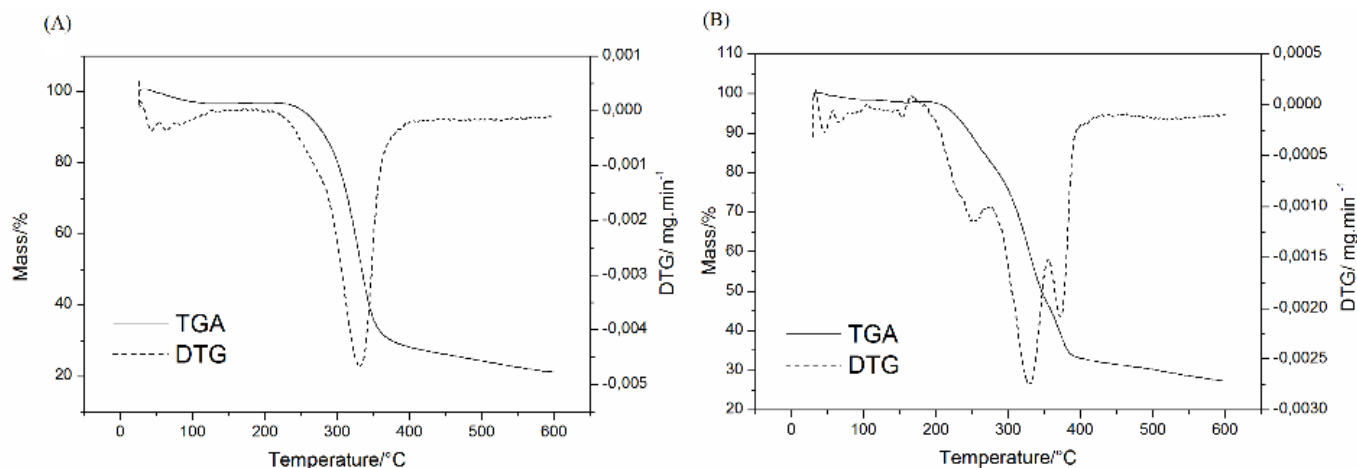
Comparing maltodextrin used in drying processes of other fruit extracts, a greater mass loss was observed at a temperature above 200°C. *Rubus glaucus* B. dry extract obtained with maltodextrin (20 DE) also presented a thermogravimetric profile with greater mass loss at temperatures above 200°C, which was attributed to polysaccharides present in sample degradation. In this way, *Bactris guineensis* dry extract obtained with maltodextrin (19–20 DE) was evaluated at a temperature range from 0 to 200°C and showed  $\Delta m = 70\%$ , at a temperature above 180°C, corresponding to thermal degradation of maltodextrin and other organic components of the extract (Osorio et al., 2011). Furthermore, it is important for the food industry that samples containing maltodextrin exceed 100°C without significant mass loss because this temperature is used in food sterilization processes (Osorio et al., 2011).



**Figure 2.** LC-MS/MS chromatograms. (A) MS/MS spectrum of açai dried extract. (B) MS/MS spectrum of açai tablet. (C) MS/MS spectrum of the cyanidin-3-rutinoside standard.

**Table 2.** Identification of cyanidin-3-rutinoside in açai dried extract and tablets containing the extract.

	RT (min)	$\lambda$ (nm)	Precursor ion [M] <sup>+</sup> (m/z)	Product ion MS/MS (m/z)
Dry extract	7.20	510	594.9	-
Tablet	7.45	510	594.8	448.5/287.1
Cyanidin-3-rutinoside	7.14	510	595.1	449.1/286.9



**Figure 3.** TG/DTG curves obtained in the temperature range from 25 to 600°C, at 10 °C.min<sup>-1</sup> under an N<sub>2</sub> atmosphere and a flow rate of 50 mL.min<sup>-1</sup>. (A) Açai dried extract. (B) Tablet containing açai dried extract.

**Table 3.** Thermoanalytical profile (TG/DTG) of açai dried extract and tablet containing the extract, analyzed under N<sub>2</sub> atmosphere, a flow rate of 50 mL.min<sup>-1</sup>, a temperature range of 25–600°C, and a heating rate of 10°C.min<sup>-1</sup>

Samples	Event	TG/DTG	
		(T <sub>on set</sub> –T <sub>end set</sub> /°C)	Δm/%
ESAc	1	30–110	1.4
	2	238.21–352.20	70.14
Tablet	1	225.06–259.29	13.7
	2	304.92–348.35	26.37
	3	318.75–367.82	28.16
	4	275.17–368.46	69.06

ESAc: açai dried extract; TG/DTG: thermogravimetric/derivate thermogravimetric; Δm/%: mass lost.

The TG/DTG curve of the tablet (Figure 3B) shows the presence of four mass-loss events (Table 3). Thus, it was found that all mass loss events observed in TG/DTG curves of the tablets showed temperatures above 200°C, which may be related to thermal degradation suffered by pharmaceutical adjuvants (cellulose, lactose, and magnesium stearate) as well as maltodextrin, due to its polysaccharide composition. This result corroborates the thermal degradation of each adjuvant separately, with the occurrence of thermal degradation processes starting at 200°C (data not shown).

## 4 CONCLUSION

Açai dried extract was submitted to different parameters to investigate processes in the development of tablets with the purpose of dietary supplementation. The extract and tablet proved to be thermally stable at the temperature range commonly used for dietary products, as they did not present significant mass variations or losses that would prevent their use as a nutritional integrator. In this sense, quality control evaluation of tablets containing açai dried extract, produced by direct compression, considered it a product with good mechanical

properties. Thus, this study allowed us to establish ways to show tablet preparation with good technological properties from açai dried extract aiming at their use as a dietary supplement in the population diet, due to their antioxidant potential and presence of phenolic compounds, mainly anthocyanins, with 3.65 µg of cyanidin-3-rutinoside.g<sup>-1</sup> per dose. However, tests such as disintegration time, dissolution profile, and dose uniformity must be performed for the complementary evaluation of these tablets.

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## REFERENCES

- Agência Nacional de Vigilância Sanitária (ANVISA) (2018). Resolução da Diretoria Colegiada - RDC n° 243, de 26 de julho de 2018. Dispõe sobre os requisitos sanitários dos suplementos alimentares. *Diário Oficial da União*, (144).
- Aliakbarian, B., Casazza, A. A., & Perego, P. (2011). Valorization of olive oil solid waste using high pressure-high temperature reactor. *Food Chemistry*, 128(3), 704-710. <https://doi.org/10.1016/j.foodchem.2011.03.092>
- Borges, G. D. S. C., Vieira, F. G. K., Copetti, C., Gonzaga, L. V., & Fett, R. (2011). Optimization of the extraction of flavanols and anthocyanins from the fruit pulp of *Euterpe edulis* using the response surface methodology. *Food Research International*, 44(3), 708-715. <https://doi.org/10.1016/j.foodres.2010.12.025>
- Carvalho, A. V., Ferreira Ferreira da Silveira, T., Mattietto, R. de A., Padilha de Oliveira, M. do S., & Godoy, H. T. (2017). Chemical composition and antioxidant capacity of açai (*Euterpe oleracea*) genotypes and commercial pulps. *Journal of the Science of Food and Agriculture*, 97(5), 1467-1474. <https://doi.org/10.1002/jsfa.7886>

- Chandrasekhar, J., Madhusudhan, M. C., & Raghavarao, K. S. M. S. (2012). Extraction of anthocyanins from red cabbage and purification using adsorption. *Food and Bioprocess Technology*, 90(4), 615-623. <https://doi.org/10.1016/j.fbp.2012.07.004>
- Cianchino, V. A., Favier, L. S., Ortega, C. A., Peralta, C., & Cifuentes, D. A. (2020). Formulation development and evaluation of Silybum marianum tablets. *Rodriguésia*, 71. <https://doi.org/10.1590/2175-7860202071044>
- Costa, R. G., Andreola, K., de Andrade Mattietto, R., de Faria, L. J. G. & Taranto, O. P. (2015). Effect of operating conditions on the yield and quality of açai (*Euterpe oleracea* Mart.) powder produced in spouted bed. *LWT - Food Science and Technology*, 64(2), 1196-1203. <https://doi.org/10.1016/j.lwt.2015.07.027>
- da Silva Carvalho, A. G., da Costa Machado, M. T., da Silva, V. M., Sartoratto, A., Rodrigues, R. A. F., & Hubinger, M. D. (2016). Physical properties and morphology of spray dried microparticles containing anthocyanins of jussara (*Euterpe edulis* Martius) extract. *Powder Technology*, 294, 421-428. <https://doi.org/10.1016/j.powtec.2016.03.007>
- da Silveira, T. F. F., Cristianini, M., Kuhnle, G. G., Ribeiro, A. B., Filho, J. T., & Godoy, H. T. (2019). Anthocyanins, non-anthocyanin phenolics, tocopherols and antioxidant capacity of açai juice (*Euterpe oleracea*) as affected by high pressure processing and thermal pasteurization. *Innovative Food Science and Emerging Technologies*, 55, 88-96. <https://doi.org/10.1016/j.ifset.2019.05.001>
- Fang, Z., & Bhandari, B. (2011). Effect of spray drying and storage on the stability of bayberry polyphenols. *Food Chemistry*, 129(3), 1139-1147. <https://doi.org/10.1016/j.foodchem.2011.05.093>
- Fernandes, R. V. de B., Botrel, D. A., Silva, E. K., Borges, S. V., Oliveira, C. R. de, Yoshida, M. I., Feitosa, J. P. de A., & de Paula, R. C. M. (2016). Cashew gum and inulin: New alternative for ginger essential oil microencapsulation. *Carbohydrate Polymers*, 153, 133-142. <https://doi.org/10.1016/J.CARBPOL.2016.07.096>
- Franceschinis, L., Salvatori, D. M., Sosa, N., & Schebor, C. (2014). Physical and Functional Properties of Blackberry Freeze- and Spray-Dried Powders. *Drying Technology*, 32(2), 197-207. <https://doi.org/10.1080/07373937.2013.814664>
- Ghribi, A. M., Gafsi, I. M., Blecker, C., Danthine, S., Attia, H., & Besbes, S. (2015). Effect of drying methods on physico-chemical and functional properties of chickpea protein concentrates. *Journal of Food Engineering*, 165, 179-188. <https://doi.org/10.1016/j.jfoodeng.2015.06.021>
- Giusti, M. M., & Wrolstad, R. E. (2001). Characterization and Measurement of Anthocyanins by UV-Visible Spectroscopy. *Current Protocols in Food Analytical Chemistry*. <https://doi.org/10.1002/0471142913.faf0102s00>
- Laokuldilok, T., & Kanha, N. (2015). Effects of processing conditions on powder properties of black glutinous rice (*Oryza sativa* L.) bran anthocyanins produced by spray drying and freeze drying. *LWT - Food Science and Technology*, 64(1), 405-411. <https://doi.org/10.1016/J.LWT.2015.05.015>
- Loureiro Contente, D. M., Pereira, R. R., Rodrigues, A. M. C., da Silva, E. O., Ribeiro-Costa, R. M., & Carrera Silva-Júnior, J. O. (2020). Nanoemulsions of Acai Oil: Physicochemical Characterization for the Topical Delivery of Antifungal Drugs. *Chemical Engineering and Technology*, 43(7), 1424-1432. <https://doi.org/10.1002/ceat.201900627>
- Magalhães, T. S. S. de A., Macedo, P. C. de O., Converti, A., & de Lima, Á. A. N. (2020). The use of euterpe oleracea mart. As a new perspective for disease treatment and prevention. *Biomolecules*, 10(6), 813. <https://doi.org/10.3390/biom10060813>
- Nascimento, R. J. S. do, Couri, S., Antonias, R., & Freitas, S. P. (2008). Composição em ácidos graxos do óleo da polpa de açai extraído com enzimas e com hexano. *Revista Brasileira de Fruticultura*, 30(2), 498-502. <https://doi.org/10.1590/S0100-29452008000200040>
- Osorio, C., Forero, D. P., & Carriazo, J. G. (2011). Characterisation and performance assessment of guava (*Psidium guajava* L.) microencapsulates obtained by spray-drying. *Food Research International*, 44(5), 1174-1181. <https://doi.org/10.1016/J.FOODRES.2010.09.007>
- Paim, D. R. S. F., Costa, S. D. O., Walter, E. H. M., & Tonon, R. v. (2016). Microencapsulation of probiotic jussara (*Euterpe edulis* M.) juice by spray drying. *LWT*, 74, 21-25. <https://doi.org/10.1016/J.LWT.2016.07.022>
- Park, S., Valan Arasu, M., Lee, M. K., Chun, J. H., Seo, J. M., Lee, S. W., Al-Dhabi, N. A., & Kim, S. J. (2014). Quantification of glucosinolates, anthocyanins, free amino acids, and vitamin C in inbred lines of cabbage (*Brassica oleracea* L.). *Food Chemistry*, 145, 77-85. <https://doi.org/10.1016/J.FOODCHEM.2013.08.010>
- Paul, S., & Sun, C. C. (2018). Systematic evaluation of common lubricants for optimal use in tablet formulation. *European Journal of Pharmaceutical Sciences*, 117, 118-127. <https://doi.org/10.1016/J.EJPS.2018.02.013>
- Rufino, M. do S. M., Alves, R. E., de Brito, E. S., Pérez-Jiménez, J., Saura-Calixto, F., & Mancini-Filho, J. (2010). Bioactive compounds and antioxidant capacities of 18 non-traditional tropical fruits from Brazil. *Food Chemistry*, 121(4), 996-1002. <https://doi.org/10.1016/J.FOODCHEM.2010.01.037>
- Santamarina, A. B., Jamar, G., Mennitti, L. V., Ribeiro, D. A., Cardoso, C. M., Vera De Rosso, V., Oyama, L. M., Pisani, L. P., & Maione, F. (2019). Polyphenols-Rich Fruit (*Euterpe edulis* Mart.) Prevents Peripheral Inflammatory Pathway Activation by the Short-Term High-Fat Diet. *Molecules*, 24(9), 1655. <https://doi.org/10.3390/molecules24091655>
- Schulz, M., Tischer Seraglio, S. K., Gonzaga, L. V., Costa, A. C. O., & Fett, R. (2021). Phenolic Compounds in Euterpe Fruits: Composition, Digestibility, and Stability—A Review. *Food Reviews International*, 39(1), 369-396. <https://doi.org/10.1080/87559129.2021.1909060>
- Silva da Costa, R., Pinheiro, W. B. de S., Arruda, M. S. P., Costa, C. E. F., Converti, A., Ribeiro Costa, R. M., & Silva Júnior, J. O. C. (2022). Thermoanalytical and phytochemical study of the cupuassu (*Theobroma grandiflorum* Schum.) seed by-product in different processing stages. *Journal of Thermal Analysis and Calorimetry*, 147(1), 275-284. <https://doi.org/10.1007/s10973-020-10347-0>
- Simon-Brown, K., Solval, K. M., Chotiko, A., Alfaro, L., Reyes, V., Liu, C., Dzandu, B., Kyereh, E., Goldson Barnaby, A., Thompson, I., Xu, Z., & Sathivel, S. (2016). Microencapsulation of ginger (*Zingiber officinale*) extract by spray drying technology. *LWT - Food Science and Technology*, 70, 119-125. <https://doi.org/10.1016/j.lwt.2016.02.030>
- Tonon, R. v., Brabet, C., & Hubinger, M. D. (2008). Influence of process conditions on the physicochemical properties of açai (*Euterpe oleracea* Mart.) powder produced by spray drying. *Journal of Food Engineering*, 88(3), 411-418. <https://doi.org/10.1016/J.JFOODENG.2008.02.029>
- Tonon, R. v., Brabet, C., & Hubinger, M. D. (2010). Anthocyanin stability and antioxidant activity of spray-dried açai (*Euterpe oleracea* Mart.) juice produced with different carrier agents. *Food Research International*, 43(3), 907-914. <https://doi.org/10.1016/J.FOODRES.2009.12.013>

- Tonon, R. v., Brabet, C., Pallet, D., Brat, P., & Hubinger, M. D. (2009). Physicochemical and morphological characterisation of açai (*Euterpe oleracea* Mart.) powder produced with different carrier agents. *International Journal of Food Science and Technology*, *44*(10), 1950-1958. <https://doi.org/10.1111/J.1365-2621.2009.02012.X>
- Valente, M. da C. da C., do Nascimento, R. A., Santana, E. B., Ribeiro, N. F. da P., Costa, C. M. L., & de Faria, L. J. G. (2019). Spray drying of extract from *Euterpe oleracea* Mart.: Optimization of process and characterization of the açai powder. *Journal of Food Process Engineering*, *42*(8), e13253. <https://doi.org/10.1111/jfpe.13253>
- Yamaguchi, K. K. D. L., Pereira, L. F. R., Lamarão, C. V., Lima, E. S., & da Veiga-Junior, V. F. (2015). Amazon acai: Chemistry and biological activities: A review. *Food Chemistry*, *179*, 137-151. <https://doi.org/10.1016/J.FOODCHEM.2015.01.055>