

Article

Optimization of Parameters for Obtaining Microparticles Containing Açai Extract (*Euterpe precatoria* Mart.) by Spray Drying

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Abstract

Açai (*Euterpe precatoria* Mart.) is a fruit with tremendous bioactive potential, but its high perishability necessitates the use of technologies to improve its stability and further enhance its bioactivity, such as microencapsulation by spray drying. Maltodextrin (MD) and gum Arabic (GA) are commonly used encapsulants in spray drying, but their combination for the microencapsulation of açai extract has not been studied. This study aimed to optimize the microencapsulation of açai extract using spray drying, evaluating its effect on the physicochemical and functional properties of the microparticles (MPs). Optimization was performed using response surface methodology with a central composite rotatable design, considering inlet air temperature (IAT) and polymer ratio (GA:MD) as factors. The optimal conditions were 135 °C IAT and a 50:50 GA:MD, under which a process yield of 76.05%, total phenolic compounds of 2791.83 mg GAE/100 g, and hygroscopicity (HYG) of 7.28% were obtained. Solubility (WSI) and total anthocyanin content (TAC) were not optimized due to lack of fit ($p < 0.05$). The experimental validation showed precise agreement between predicted and experimental values, with relative errors of less than 5%. The MPs presented spherical forms with rough surfaces. The effectiveness of the microencapsulation process demonstrates the potential of açai MPs as possible functional ingredients for future food applications.

Keywords: Amazon fruits; bioactive compounds; microencapsulation; spray drying; response surface methodology



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1. Introduction

In recent years, the promotion of healthy lifestyles has gained significant global importance due to the increase in chronic non-communicable diseases [1,2], which has driven the

consumption of various foods rich in bioactive compounds [3]. In this context, fresh fruits stand out as food matrices of high nutritional value, as they provide vitamins, minerals, dietary fiber, and secondary metabolites such as phenolic compounds including flavonoids, as well as carotenoids, which have antioxidant, anti-inflammatory, and protective properties against oxidative stress caused by reactive oxygen species [4–6].

Among these matrices, açai, belonging to the Arecaceae family, is one of the most important Amazonian fruits, primarily represented by the species *Euterpe oleracea* Mart. and *Euterpe precatoria* Mart. Although both species share similar morphological and taxonomic characteristics, they differ in their total phenolic compounds (TPCs), total anthocyanin content (TAC) and unsaturated fatty acids [7]. Previous studies have reported that *Euterpe oleracea* contains up to 1405 mg GAE/100 g of TPCs [8] and 2247 mg/kg of TAC [9]. However, the açai variety *Euterpe precatoria* Mart. has been the subject of increasing interest due to its high content of TPCs (769 and 2414 mg GAE/100 g) [10] and flavonoids, including anthocyanins (3458 mg/kg) [9], in addition to its traditional use as a staple food rich in energy and its ethnomedicinal application for the treatment of fever, inflammation and cancer that are associated with a powerful antioxidant capacity that could be applied in the food industry [11–13]. However, the fresh fruits, as well as the pulp of this fruit, exhibit limited stability during processing and storage, attributed to their high moisture content [14], sugars [15], and the susceptibility of their bioactive compounds to factors such as temperature, oxygen, light, pH, and enzymatic activity, which can lead to their degradation and loss of functional quality [16,17].

Given this issue, spray drying has become established as an efficient technological alternative for processing perishable foods, as it allows for obtaining dry products by spraying liquid matrices into a stream of hot air. This rapidly reduces their water activity and, consequently, improves their stability as a final product [18,19]. However, in systems such as fruit juices, this process has inherent limitations, such as the high stickiness and adhesion of the matrix to the drying equipment surfaces. This reduces the recovery efficiency of microparticles (MPs) and promotes the degradation of thermolabile compounds. These effects are mainly associated with the low glass transition temperature (T_g) of low-molecular-weight sugars [20–22].

To overcome these limitations, encapsulating materials such as pectin, starch, maltodextrin (MD), and gum Arabic (GA) are used [23], which improve drying performance by increasing T_g and preventing the plasticizing effect. They also act as protective matrices for the bioactive compounds [20,24]. Previous studies on spray drying of açai, such as those by Tonon et al. [25], Valente et al. [26] and Vargas et al. [27], evaluated the effect of operating variables on the physicochemical stability of the encapsulating materials, focusing on MD (up to 15% total solids) as the sole encapsulant and wide or high temperature ranges (102 to 200 °C). However, the GA:MD binary relationship has not been reported using açai extract from *Euterpe precatoria*, highlighting a knowledge gap that this research seeks to address. Recently, it has been shown that combining MD and GA results in greater protection of bioactive compounds in the gastric phase, which is crucial to prevent the bioactivity loss of encapsulated compounds in the intestine [28]. Similarly, Salati et al. [29] demonstrated that the MD:GA combination significantly increases the encapsulation efficiency of TAC in sour cherry, reaching values between 97.27 and 98.92%, along with water solubility index (WSI) values exceeding 99.98%. Likewise, Kalušević et al. [30] reported WSI values between 80.3 and 94.3%, as well as process yield (PY) values of 63.7 to 77.0%, in MPs for TAC-rich extracts. Meanwhile, Mehrali et al. [31] observed hygroscopicity (HYG) values of 85 to 39% in MPs of tea flower pollen peptides, significantly improving flowability and decreasing the stickiness of the MPs. This behavior is attributed to the synergistic effect between both encapsulants, where GA contributes emulsifying properties and the ability

to form elastic MPs, while MD contributes high solubility and more precise control of the viscosity of the feed emulsion [32]. Therefore, the MD-GA mixture is a promising strategy for encapsulating açai.

For encapsulation, the process efficiency depends on operating conditions such as feed flow rate and inlet air temperature (IAT), as well as the system formulation, which involves the interaction between the bioactive of interest and the encapsulants [33]. Optimizing these parameters is necessary to obtain products with suitable physicochemical properties and optimal technological and bioactive functionality [34]. In this context, the objective of this study was to optimize, through spray drying, the IAT and the GA:MD polymer ratio in a feed with 8% total solids to obtain MPs from açai extract. Scientific evidence shows that the IAT is a determining factor that directly influences the thermal degradation of phenolic compounds [35,36], while the GA:MD ratio affects encapsulation capacity and oxidative stability [37,38]. Therefore, the aim is to establish optimal microencapsulation conditions that allow for high % PY, high % WSI, and low % HYG of MPs with good bioactive characteristics, such as mg C3G/100 g TAC and mg GAE/100 g TPCs.

2. Materials and Methods

2.1. Materials and Reagents

The materials used were fresh açai fruits (*Euterpe precatoria* Mart.), collected in the hamlet of Huambé, located in the city of Iquitos, Peru. Maltodextrin (dextrose equivalent (DE) of 10) and gum Arabic (Sigma-Aldrich®, St. Louis, MO, USA) were used as encapsulants. For the preparation of the açai ethanolic extract (AEE), double-distilled ethanol and Milli-Q water (99.8%; Merck®, Darmstadt, Germany) were used. Sodium carbonate (Na_2CO_3) and the Folin–Ciocalteu reagent with Trolox as a standard (Sigma-Aldrich®, St. Louis, MO, USA) were used for the determination of phenolic compounds. Potassium chloride (KCl), hydrochloric acid (HCl), and sodium acetate (CH_3COONa) were used to determine anthocyanins. All reagents were of analytical grade. Table 1 shows the percentage composition (g/100 g) of the açai fruit.

Table 1. Centesimal composition of the açai fruit (*Euterpe precatoria* Mart.).

Parameters	Results (%)	Method of Analysis
Moisture	70.74 ± 0.28	AOAC (2023); method 950.46 [39]
Protein	4.48 ± 0.32	AOAC (2023); method 981.10 [39]
Lipids	5.97 ± 0.18	AOAC (2023); method 960.39 [39]
Ash	1.51 ± 0.02	AOAC (2023); method 920.153 [39]
Carbohydrates	15.30 ± 0.50	Calculated by difference

Mean values ± standard deviation (n = 3).

2.2. Methods

2.2.1. Preparation of Extract

The AEE was obtained following the methodology described by Pompeu et al. [40], with slight modifications. The açai pulp was macerated in a 99.8% hydroalcoholic solution in a 50:50 ratio with distilled water. This maceration was performed at a 1:4 ratio, comprising 1 kg of açai pulp in 4 L of solvent. The mixture was protected from light for 72 h at room temperature (30 ± 1 °C). Subsequently, the AEE was filtered through qualitative filter paper (14 µm pore size and 205 µm thickness) and concentrated in a rotary evaporator (ROVA-100, MRC Laboratory Equipment Ltd., Holon, Israel) at 50 °C for 20 min.

2.2.2. Total Phenolic Compounds

The TPCs of the AEE was determined using the Folin–Ciocalteu method described by Singleton et al. [41], with slight modifications. A 20 µL aliquot of AEE was diluted in 9980 µL of ethanol and thoroughly homogenized. For the reaction, 200 µL of this solution was mixed with 1500 µL of distilled water and 100 µL of Folin–Ciocalteu reagent. After 5 min of reaction at room temperature (30 ± 1 °C), 200 µL of Na₂CO₃ solution was added, and the mixture was incubated for 30 min in the dark for color development. The absorbance was then measured at 765 nm using a UV-Vis spectrophotometer (Thermo Scientific®, Madison, WI, USA; model GENESYS 150). The results were expressed as milligrams of gallic acid equivalents (GAE) per 100 g of AEE and were calculated from a calibration curve using the following equation:

$$\text{TPC (mg GAE/100 g)} = \frac{\text{Concentration } \left(\frac{\text{mg}}{\text{L}}\right) \times \text{dilution factor} \times \text{volume of extract (L)} \times 100}{\text{grams of extract (g)}} \quad (1)$$

2.2.3. Total Anthocyanins

The TAC of the AEE was determined using the differential pH method described by Lee et al. [42], with slight modifications. The 200 µL aliquots of the AEE were separately diluted in 9800 µL of KCl (0.025 M, pH 1.0) and CH₃COONa (0.4 M, pH 4.5) buffer solutions. The mixtures were allowed to stand for 15 min at room temperature (30 ± 1 °C), protected from light. Absorbances were then recorded at 520 and 700 nm using a UV-Vis spectrophotometer (Thermo Scientific®, Madison, WI, USA; model GENESYS 150). The results were expressed as milligram equivalents of cyanidin-3-glucoside (C3G) per 100 g of AEE and calculated using the following equation:

$$\text{TA} = (\text{A}_{520 \text{ nm}} - \text{A}_{700 \text{ nm}})_{\text{pH} 1.0} - (\text{A}_{520 \text{ nm}} - \text{A}_{700 \text{ nm}})_{\text{pH} 4.5} \quad (2)$$

$$\text{TAC (mg C3G/100 g)} = \frac{\text{Corrected absorbance} \times \text{molecular weight C3G} \times \text{dilution factor} \times \text{volume of extract} \times 100}{\text{Molar coefficient C3G} \times \text{cell length} \times \text{grams of extract (g)}} \quad (3)$$

2.2.4. Antioxidant Capacity

ABTS Radical Scavenging Activity

The antioxidant capacity of the AEE obtained from the pulp was determined by ABTS radical scavenging assays, following the methodologies described by Re et al. [43], with slight modifications. The ABTS•⁺ cation radical was generated by reacting ABTS (7 mM) with potassium persulfate (2.45 mM) and incubating in the dark for 12–16 h at room temperature. Subsequently, the solution was diluted with ethanol to obtain an absorbance of 0.70 at 734 nm. For the reaction, 30 µL of the AEE was mixed with 3000 µL of the ABTS•⁺ solution, and the absorbance was recorded at 734 nm after 6 min of reaction using a UV-Vis spectrophotometer (Thermo Scientific®, Madison, WI, USA; model GENESYS 150).

DPPH Radical Scavenging Activity

The antioxidant capacity of the AEE obtained from the pulp was determined by DPPH radical scavenging assays, following the methodologies described by Brand-Williams et al. [44], with slight modifications. An ethanolic DPPH solution (0.1 mM) was prepared and mixed with 100 µL of the AEE and 3900 µL of the radical scavenging solution. The mixture was incubated in the dark for 30 min at room temperature, and the decrease in absorbance was measured at 517 nm using a UV-Vis spectrophotometer (Thermo Scientific®, Madison, WI, USA; model GENESYS 150). In both assays, antioxidant capacity was quantified using a calibration curve with Trolox as the standard, and the results were expressed as µmol equivalents of Trolox per g of AEE, calculated according to the following equation:

$$\text{ABTS; DPPH } (\mu\text{mol ET/g}) = \frac{\text{Concentration } (\mu\text{mol/mL}) \times \text{volume of extract (mL)} \times \text{dilution factor}}{\text{grams of extract (g)}} \quad (4)$$

2.2.5. Production of Microparticles by Spray Drying

The production of MPs was carried out following the methodology described by Nogueira et al. [45], with slight modifications. The feed system was prepared with a total solids content of 8% (*w/w*), where the solid fraction was composed of 70% concentrated extract and 30% encapsulating material (dry basis), which were subjected to continuous stirring at room temperature (30 ± 1 °C) until a homogeneous solution was achieved.

The spray drying process was carried out in a laboratory-scale spray dryer (model SD-05, LabPlant UK Ltd., Filey, UK), equipped with a 1.5 mm diameter spray nozzle and a 500 × 215 mm drying chamber. The feed solution was kept under magnetic stirring at room temperature and introduced into the drying chamber using a peristaltic pump. The operating conditions included a drying air flow rate of 73 m³/h, a spray pressure of 0.06 MPa, and a feed flow rate of 10 mL/min. The experimental assays were designed based on the aforementioned methodology and preliminary tests, utilizing a central composite rotatable design (CCRD), with the IAT (125 to 145 °C) and biopolymer ratio (0 to 100%) as independent variables, as detailed in Table 2.

Table 2. Central composite rotational design and experimental results % PY, % WSI, % HYG, mg GAE/100 g TPCs and mg C3G/100 g TAC of spray-dried açai (*Euterpe precatoria* Mart.).

Assay	Coded Values		Decoded Values		% PY	% WSI	% HYG	TPC	TAC
	X ₁	X ₂	IAT	GA:MD Ratio					
1	−1	−1	127.90	10:90	66.38	92.08	4.36	2617.31	64.55
2	−1	+1	127.90	90:10	68.20	91.99	5.23	2638.77	69.67
3	1	−1	142.10	10:90	68.04	93.59	8.25	2252.50	60.76
4	1	+1	142.10	90:10	69.45	93.52	8.02	2417.02	70.7
5	−1.41	0	125.00	50:50	70.75	91.03	3.07	2653.07	90.04
6	+1.41	0	145.00	50:50	73.42	94.29	7.81	2202.43	72.12
7	0	−1.41	135.00	0:100	63.95	93.07	5.93	2359.80	60.44
8	0	+1.41	135.00	100:0	67.75	93.65	5.31	2510.01	63.67
9	0	0	135.00	50:50	76.22	93.23	7.12	2803.29	83.50
10	0	0	135.00	50:50	75.82	93.15	7.58	2781.83	84.23
11	0	0	135.00	50:50	77.47	93.27	7.14	2760.37	82.08

X₁: IAT °C (inlet air temperature); X₂: GA:MD ratio (gum Arabic: maltodextrin); % PY: process yield; % WSI: water solubility index; % HYG: hygroscopicity; TPC: mg GAE/100 g total phenolic compounds; TAC: mg C3G/100 g total anthocyanin compounds.

2.2.6. Process Yield

The % PY of the MPs was determined following the methodology described by Domínguez-Valencia et al. [46]. This parameter was calculated as the ratio between the mass of the MPs recovered after spray drying and the mass of total solids in the feed solution, expressed as a percentage, according to the equation:

$$\text{PY (\%)} = \frac{\text{grams of dry microparticles (g)}}{\text{grams of total solids in the feed (g)}} \times 100 \quad (5)$$

2.2.7. Water Solubility Index

The % WSI of the MPs was determined following the methodology described by Pieczykolan and Kurek [47], with slight modifications. In beakers, 1 g of MPs was dispersed in 100 mL of distilled water and stirred for 5 min. Subsequently, the suspension was

centrifuged at $3000 \times g$ for 15 min (Hettich, model 320R, GmbH & Co. KG, Tuttlingen, Germany). After centrifugation, 25 mL of the supernatant was transferred to a pre-weighed beaker and dried in an oven at $105 \pm 1^\circ\text{C}$ for 24 h. Finally, the WSI was expressed as a percentage of dissolved solids and calculated using the following equation:

$$\text{WSI (\%)} = \frac{\text{grams of dry residue (g)}}{0.25} \times 100 \quad (6)$$

2.2.8. Hygroscopicity

The % HYG of the MPs was determined following the methodology of Cai and Corke [48], with slight modifications. A modified environment with a relative humidity of 81% and a temperature of $25 \pm 1^\circ\text{C}$ was created using a saturated sodium sulfate (Na_2SO_4) solution in desiccators. Two grams of MPs was placed in Petri dishes inside the desiccator and maintained in equilibrium for 7 days. Finally, the HYG was expressed as the percentage of water absorbed, based on the increase in mass of the MPs after exposure to a controlled environment, and was calculated according to the following equation:

$$\text{HYG (\%)} = \frac{\text{Final grams of microparticles (g)} - \text{Initial grams of microparticles (g)}}{\text{Initial grams of microparticles (g)}} \times 100 \quad (7)$$

2.2.9. Extraction and Analysis of Bioactive Compounds in Microparticles

The preparation of the AEE obtained from the MPs was carried out following the methodology described by Rufino et al. [49], with slight modifications. In Falcon tubes, 1 g of MPs was mixed with 5 mL of a hydroalcoholic solution (1:1, v/v) for 1 h at room temperature ($30 \pm 1^\circ\text{C}$) in the absence of light. Subsequently, the mixture was centrifuged at 3000 rpm for 10 min at $25 \pm 1^\circ\text{C}$, and the supernatant was recovered and filtered. The residue was subjected to a second extraction with 10 mL of the same solvent under the same conditions, and both AEEs were combined for analysis. For the TAC, the same methodology was followed, acidifying the hydroalcoholic solution with HCl to pH 1.0. The phytochemical characterization, which included the determination of TPCs and TAC, was carried out following the methodologies described in Sections 2.2.2 and 2.2.3.

2.2.10. Morphology of Microparticles

The morphology of the MPs was evaluated following the methodology described by Ibrahim Silva et al. [50]. Morphological analysis was performed using scanning electron microscopy (SEM) with a Nova NanoSEM 200 instrument (FEI, Hillsboro, OR, USA). The MPs were fixed to metal supports with carbon tape and coated with a conductive gold layer by sputtering (model FDU-010, Balzers Union AG, Balzers, Liechtenstein). Finally, micrographs were obtained under high vacuum conditions, using an accelerating voltage of 5 to 15 kV and magnifications of $1000\times$ and $2500\times$.

2.3. Statistical Analysis and RSM Optimization

A CCRD was applied, which integrated a 2^2 full factorial arrangement that included 4 assays under axial conditions and 3 replicates at the center point, totaling 11 experimental assays. The response variables evaluated were PY, HYG, WSI, TPCs, and TAC. The results obtained from the CCRD were fitted to a second-order polynomial model, and the corresponding regression coefficients were estimated. The normality of the experimental data was verified using the Shapiro–Wilk test. The models were evaluated using analysis of variance (ANOVA), considering the significance of the terms, lack of fit, and the coefficient of determination (R^2). The optimization of the response variables was performed using response surface methodology (RSM). A comparison of the TPCs and antioxidant capacity of the açai pulp and AEE was also carried out using Student's *t*-test at a 5% significance

level. Statistical analysis was performed using Statistica software (version 12.0, StatSoft Inc., Tulsa, OK, USA). Finally, the validation of the predictive model was performed by experimentally obtaining MPs under optimal conditions in triplicate.

3. Results and Discussion

3.1. Phenolic Compounds and Antioxidant Capacity of Açai Pulp and Extract

The results of the phytochemical characterization of the açai pulp and AEE are presented in Table 3.

Table 3. Phenolic compounds and antioxidant capacity of açai pulp and extract.

Sample	TPC	TAC	ABTS	DPPH
Pulp	241.45 ± 3.10 ^b	8.34 ± 0.25 ^b	1073.47 ± 2.35 ^b	831.33 ± 1.96 ^b
Extract	3964.32 ± 2.33 ^a	108.17 ± 0.75 ^a	21,057.73 ± 4.32 ^a	9487.80 ± 3.83 ^a

Mean values ± standard deviation (n = 3). TPC: total phenolic content (mg GAE/100 g); TAC: total anthocyanin content (mg C3G/100 g); ABTS: 2,2'-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) (μmol TE/g); DPPH: 2,2-diphenyl-1-picrylhydrazyl (μmol TE/g). Different letters in the same column differ from each other ($p < 0.05$) according to Student's *t*-test.

The results obtained reveal significant differences in the content of bioactive compounds between fresh pulp and the extract of açai, reflecting both the nature of the matrix and the efficiency of the extraction process used. Fresh açai pulp contained 241.45 mg GAE/100 g of TPCs and 8.34 mg C3G/100 g of TAC. Comparing these values with those reported by Garzón et al. [51], who found 607 mg GAE/100 g of TPCs and 57 mg C3G/100 g of TAC in açai pulp (*Euterpe oleracea*) from Colombia, the values in the present study are approximately 2.5 times lower for TPCs and 6.8 times lower for TAC. This variation can be attributed to multiple intrinsic and extrinsic factors that affect the phytochemical composition of the fruits [52,53]. Madalão et al. [54] identified that the stage of ripeness, geographic origin, and harvest are critical determinants of TPCs and TAC in species of the genus *Euterpe*. In their study with juçara pulp (*Euterpe edulis*), they reported 1226.39 mg GAE/100 g of TPCs and 298.86 mg C3G/100 g of TAC, values that exceed those of the present study, highlighting the need to characterize the fruits individually. Additionally, post-harvest conditions play a critical role in the stability of these thermolabile and photosensitive compounds, particularly when exposure to light, oxygen and high temperatures promotes their degradation through oxidation, polymerization and flavylum ring rupture reactions, reducing their content in the plant matrix [55].

In contrast to fresh pulp, the AEE obtained through maceration showed a significant increase in both parameters, reaching 3964.32 mg GAE/100 g of TPCs and 108.17 mg C3G/100 g of TAC. This 16- and 13-fold increase in TPCs and TAC, respectively, demonstrates the high efficiency of the extraction method used to concentrate these bioactive compounds. These values are comparable to those reported by Da Silva et al. [56] in açai juice processed by high hydrostatic pressure at 400 and 600 MPa for 5 min at 20 °C, and thermal pasteurization at 85 °C for 1 min, who obtained values between 3166 and 3797 mg GAE/100 g of TPCs.

The efficiency of the ethanol: water system in the extraction of bioactive compounds can be explained by the fundamental principles of polarity and solubility that govern solute–solvent interactions. Wu et al. [57] demonstrated that adding up to 50% water to organic solvents significantly increases the recovery of total phenols and flavonoids from plant matrices. This is because glycosylated anthocyanins and highly polar phenolic acids are better solubilized in hydroalcoholic mixtures, while aglycone flavonoids and higher-molecular-weight compounds require solvents of intermediate polarity for optimal extraction. The presence of water in the extraction system facilitates solvent penetration

into the plant matrix by increasing the contact area between the solvent and the bioactives, thus improving mass transfer efficiency and extraction kinetics [58,59].

Extraction efficiency does not depend exclusively on the solvent system [40]. Hanula et al. [60] reported TPCs of up to 35.26 and 35.95 mg GAE/g in açai extract obtained by ultrasound at 45 °C for 25 and 45 min, values lower than the AEE of the present study. Therefore, the selection of the extraction method should consider not only quantitative yield but also the preservation of the integrity and biological functionality of the bioactives, as well as economic factors, scalability, and sustainability of the process.

From a functional and nutraceutical perspective, *Euterpe precatoria* has been reported as a relevant source of bioactive compounds with high antioxidant capacity. Kang et al. [61] noted that this species exhibits superior antioxidant activity within the Arecaceae family. In this regard, the observed increase in antioxidant capacity values measured by ABTS (21,057.73 µmol TE/g) and DPPH (9487.80 µmol TE/g) in the AEE is associated with the increased content of phenolic compounds and anthocyanins. At the biochemical level, these compounds act as electron or hydrogen donors in in vitro assays, neutralizing free radicals, reactive oxygen species, and pro-oxidant metal ions [62].

Clinical studies have suggested that the consumption of *Euterpe*-derived products may be associated with improvements in markers of systemic oxidative stress and vascular function, although these studies are still limited. Reduction in oxidative stress has been implicated in the pathophysiology of chronic non-communicable diseases, including cardiometabolic, neurodegenerative, inflammatory, and renal disorders [63], and in this context, açai has shown antioxidant and anti-inflammatory properties, as well as modulatory effects against metabolic alterations and vascular dysfunction [64].

3.2. Optimization of Microparticles Production

The levels of the independent variables, expressed in coded and decoded terms, as well as the experimental responses derived from the CCRD, are presented in Table 2.

The ANOVAs for the response variables are shown in Table 4. The WSI and TAC responses were discarded due to a lack of fit ($p < 0.05$), indicating that the second-order polynomial model did not adequately describe the experimental variability for these responses. Nevertheless, the ANOVA revealed significant effects of IAT and the GA:MD ratio on TAC, highlighting a significant quadratic effect of X_2 , which suggests a non-linear relationship between carrier composition and TAC. In contrast, WSI was mainly influenced by IAT, whose significant linear and quadratic effects ($p < 0.05$) indicate that higher IAT favored the formation of more soluble structures. Meanwhile, the GA:MD ratio did not show a significant effect on WSI. % PY was primarily determined by the polymer ratio (GA:MD), while % HYG depended on the IAT and the type of encapsulant, and TPCs (mg GAE/100 g) were dominated by the IAT (Table 4). Regression analysis allowed for the fitting of the second-order polynomial models, which were used to describe and optimize the response variables according to the following equations with coefficients of determination (R^2) between 0.90 and 0.98:

$$\% \text{ PY} = -706.864 + 11.295X_1 - 0.041X_1^2 + 48.887X_2 - 41.214X_2^2 - 0.036X_1X_2 \quad (8)$$

$$\% \text{ HYG} = -258.330 + 3.636X_1 - 0.012X_1^2 + 16.727X_2 - 3.702X_2^2 - 0.097X_1X_2 \quad (9)$$

$$\text{mg GAE/100 g TPC} = -49045.353 + 790.391X_1 - 3.031X_1^2 - 431.525X_2 - 1137.435X_2^2 + 12.593X_1X_2 \quad (10)$$

Table 4. Analysis of variance of the quadratic model for % PY; % WSI, % HYG, mg GAE/100 g TPCs, and mg C3G/100 g TAC of spray-dried açai (*Euterpe precatoria* Mart.).

Factor	Sum of Squares	Degree of Freedom	Mean Square	F-Value	p-Value
% PY					
X_1	5.58	1	5.58	7.54	0.11
X_1^2	25.11	1	25.11	33.89	0.03
X_2	8.94	1	8.94	12.06	0.07
X_2^2	175.98	1	175.98	237.54	0.00
X_1X_2	0.04	1	0.04	0.06	0.83
Lack of fit	1.52	3	0.51	0.68	0.64
Pure error	1.48	2	0.74		
Total SS	197.73	10			
% WSI					
X_1	7.31	1	7.31	1757.98	0.00
X_1^2	0.67	1	0.67	179.54	0.01
X_2	0.04	1	0.04	12.00	0.07
X_2^2	0.00	1	0.00	0.00	0.97
X_1X_2	0.00	1	0.00	0.03	0.89
Lack of fit	0.54	3	0.18	47.88	0.02
Pure error	0.01	2	0.00		
Total SS	8.60	10			
% HYG					
X_1	22.39	1	22.39	331.20	0.00
X_1^2	2.26	1	2.26	33.49	0.03
X_2	0.00	1	0.00	0.04	0.86
X_2^2	1.42	1	1.42	21.01	0.04
X_1X_2	0.30	1	0.30	4.47	0.17
Lack of fit	2.64	3	0.88	13.00	0.07
Pure error	0.14	2	0.07		
Total SS	28.49	10			
mg GAE/100 g TPC					
X_1	187,197.05	1	187,197.05	406.48	0.00
X_1^2	134,993.99	1	134,993.99	293.13	0.00
X_2	19,604.70	1	19,604.70	42.57	0.02
X_2^2	134,032.81	1	134,032.81	291.04	0.00
X_1X_2	5116.54	1	5116.54	11.11	0.08
Lack of fit	17,694.40	3	5898.13	12.81	0.07
Pure error	921.06	2	460.53		
Total SS	449,699.58	10			
mg C3G/100 g TAC					
X_1	98.40	1	98.40	82.32	0.01
X_1^2	15.96	1	15.96	13.35	0.07
X_2	51.19	1	51.19	42.82	0.02
X_2^2	844.42	1	844.42	706.45	0.00
X_1X_2	5.81	1	5.81	4.86	0.16
Lack of fit	82.79	3	27.60	23.09	0.04
Pure error	2.39	2	1.20		
Total SS	1092.21	10			

X_1 : IAT °C (inlet air temperature); X_2 : GA:MD ratio (gum Arabic: maltodextrin); % PY: process yield; % WSI: water solubility index; % HYG: hygroscopicity; TPC: mg GAE/100 g total phenolic compounds; TAC: mg C3G/100 g total anthocyanin compounds.

3.2.1. Process Yield

The % PY is a crucial variable for industries, as it is directly related to production costs and efficiency [65]. Optimizing % PY in spray drying is critical for the microencapsulation of bioactive compounds, especially in binary systems (mixture of encapsulants), where the balance between contrasting physicochemical properties determines product recovery. In the present study, the % PY ranged from 63.95 to 77.47%, reaching its maximum value under the central conditions of the experimental design (assay 11, Table 2), with an intermediate temperature (135 °C) and a polymer ratio (GA:MD) of 50:50. In contrast, the lowest % PY was also observed at an intermediate IAT (135 °C) but in the absence of GA (GA:MD; 0:100), i.e., pure MD (assay 7), which suggests that the lack of GA reduces the efficiency of the process and favors the adhesion of the material to the dryer walls.

The superiority of the GA:MD system over pure encapsulants lies in its differential effect on the glass transition temperature (T_g) of the resulting MPs and, consequently, on their tendency to exhibit sticky behavior during and after drying. T_g defines the transition from a rigid to a rubbery state, increasing molecular mobility and the tendency to adhere [66–68]. GA, a branched polysaccharide with high molecular weight (250 to 1500 kDa) and high solubility, promotes the stability of the feed emulsion by significantly raising the T_g of the system [69,70]. In contrast, MD, a low-molecular-weight starch hydrolysate (1.6 to 1.9 kDa), exhibits high solubility and low viscosity, which favors atomization. However, its limited ability to increase T_g according to its dextrose equivalent (DE) reduces its effectiveness in controlling system stickiness [71–73], especially considering that açai contains low-molecular-weight sugars [74].

This behavior is consistent with Santana et al. [75], who demonstrated that the incorporation of high-molecular-weight encapsulating materials such as GA, whey protein concentrate, and soy protein isolate increases the T_g of *Euterpe edulis* MPs, reducing stickiness and improving % PY between 33.88 and 76.55%. This behavior was statistically observed in the present study ($p < 0.05$). Similarly, Arumugham et al. [38] reported that, in binary systems (GA:MD) applied to date extract, a carrier concentration of 45% optimizes the balance between a high T_g and adequate viscosity, while higher concentrations increase viscosity and reduce process efficiency.

In this regard, the adhesion of the atomized material in the dryer reduces the % PY, decreases operational efficiency, and deteriorates the quality of the raw materials, affecting the profitability of the process [76,77]. While GA increases the T_g of the system, its concentration must avoid excessive increases in feed viscosity, which affect atomization by generating larger droplets, prolonging drying time, and promoting moisture retention [78,79]. This behavior agrees with that reported by Tonon et al. [80], who showed that, in the spray drying of açai pulp, increasing the MD (10 to 30%) raises the feed viscosity, negatively affecting atomization and reducing the % PY. In contrast, they observed that higher IATs (138 to 202 °C) improved heat and mass transfer efficiency, accelerated evaporation of surface water (humidity), and decreased the residence time of atomized droplets, leading to greater product recovery.

In fact, increased IAT favors MPs recovery, a behavior also statistically evidenced in the present study ($p < 0.05$). This finding is consistent with the results of Decker et al. [81], who reported that increases in IAT from 120 to 140 °C increased % PY. Similarly, Arumugham et al. [38] observed that higher IATs within the studied range (150 and 170 °C) promoted greater heat and mass transfer, resulting in higher % PY (45.35 to 48.84%), although this positive effect must be balanced against the potential thermal degradation of bioactive compounds. Likewise, Vargas et al. [27] reported that both the IAT and the proportion of the encapsulating material directly influence the % PY of encapsulation of açai pulp (*Euterpe oleracea* Mart.), identifying an optimal IAT of 155 °C that maximizes the % PY of the

MPs (>88%) without significantly compromising its nutritional quality. However, Rosales-Chimal et al. [82], in the microencapsulation of anthocyanin extracts microencapsulated with taro starch, observed an increase in % PY (up to 70.1%) when the IAT was increased from 90 to 125 °C, followed by a decrease at higher IATs such as 125 and 160 °C. In general, the effect of IAT on % PY is one of the most determining operational factors in the efficiency of heat and mass transfer during the atomization and dehydration of the atomized droplets that will ultimately become MPs [83]. However, in the present study, its role emerges as a modulator rather than a determining factor. The fact that both the maximum % PY (assay 11) and the minimum % PY (assay 7) were obtained at the same IAT of 135 °C demonstrates that, within the evaluated experimental range and under the specific operating conditions employed, the composition of the binary system exerts a dominant effect on % PY, exceeding the magnitude of the IAT effect. This finding does not contradict the importance of IAT documented in the literature, but rather underscores that when IAT is kept constant, the physicochemical properties of the carrier system, particularly its ability to modulate the Tg of the raw materials and the viscosity of the feed mixture, emerge as the determining factors of process efficiency.

3.2.2. Hygroscopicity

The HYG is another critical variable and an important physicochemical property to consider in the industry, due to its influence on the stability of dehydrated foods, particularly during storage [84]. % HYG ranged from 3.06% to 8.24%, with the lowest value (assay 5) recorded at low temperature (125 °C) and a polymer ratio (GA:MD) of 50:50, while the highest (assay 3) was observed at high temperature (142.1 °C) in the near absence of GA (GA:MD; 10:90). These results suggest that MD, as an encapsulating material, generates more amorphous polymers than GA with a greater affinity for moisture, especially under elevated thermal conditions.

As expected, in the present study, the IAT exerted a statistically significant influence on the % HYG ($p < 0.05$). Tonon et al. [80] indicated that IAT regulates HYG by modifying the amorphous structure of the matrix and its Tg. In particular, high thermal conditions (>138 °C) favor matrix amorphization and reduce moisture, increasing the hygroscopic gradient and thus the tendency for water adsorption from the medium. This behavior agrees with that reported by Bednarska & Janiszewska-Turak [85], who, in the microencapsulation of blackberry pulp (*Rubus fruticosus*) under IAT between 100 and 150 °C, reported % HYG values between 10.93 and 15.31%, identifying IAT as the variable with the greatest influence on the HYG of the final product. Similarly, Negrão-Murakami et al. [86] established a direct correlation between IAT and HYG mediated by the size of aronia MPs, observing that higher IAT (200 °C) produce smaller MPs with a larger exposed surface area, and that under water activity conditions above 0.5, the type of encapsulant had no significant impact ($p > 0.05$).

This mechanism is consistent with the results of the present study, where the higher % HYG observed can likely be attributed to the generation of smaller and finer MPs with a larger surface area. However, the highest % HYG values (assay 3) were recorded when using a higher proportion of MD, an effect that was statistically significant ($p < 0.05$). Vargas et al. [27] highlighted that a higher proportion of MD in the spray drying of açai (*Euterpe oleracea* Mart.) exhibits a high Tg that minimizes structural changes, and that moderate IATs (110 °C) optimize this parameter, resulting in a minimum HYG of 4.42%.

The DE of the MD is a key determinant of the HYG, defining the molecular weight, chain length, and availability of hydroxyl groups capable of interacting with water [71,87]. In this regard, Tonon et al. [88] demonstrated that MD with 30 DE, especially in combination with GA, exhibits a higher % HYG than those formulated with MD with 10 DE, and that

a gradual increase in the proportion of GA led to a progressive increase in % HYG (MD 10 DE: 12.12%; GA: 14.33%; GA:MD 50:50: 12.96%), probably due to the presence of protein and its polar groups. These results differ from those obtained in the present study. Although experiments 1 and 2 showed a higher % HYG for GA, the overall trend revealed a slightly predominant effect of MD in all experiments, probably because it has a density of 10 DE. However, the GA:MD binary combination showed intermediate and constant % HYG values in the MPs, likely due to the formation of a matrix with a higher Tg, which possesses microstructural stability against caking and crystallization of amorphous sugars [89]. Nevertheless, its effect depends on the formulation and processing conditions.

In general, the % HYG obtained in this study is below the critical threshold of 20% proposed by Arumugham et al. [38]. Furthermore, according to the GEA Niro classification cited by Cano-Chauca et al. [90], all the raw materials fall within the non-hygroscopic category (<10%), which is desirable as it contributes to physical stability, reduces agglomeration, and preserves product flowability [91,92]. This behavior suggests a positive effect on the shelf life of the MPs during storage, as the combination of low HYG and temperature control may reduce moisture absorption and preserve physicochemical stability. Nayak et al. [93] reported HYG values between 2.32% and 5.07% in sachinchi oil microcapsules, associated with low moisture contents (1.64 and 1.76%) and improved storage stability. Furthermore, Rascón et al. [94] reported that anthocyanin MPs obtained by spray drying using MD DE 21, GA, and tricalcium phosphate exhibited a HYG value of 4.38%, significantly below the 10% threshold. This low HYG was directly correlated with higher anthocyanin retention (485 mg/100 g) and antioxidant activity (69.90%), demonstrating that minimizing moisture adsorption helps preserve the chemical integrity of bioactive compounds.

This finding highlights the importance of maintaining the MPs below their Tg and under low-water-activity conditions in order to minimize molecular mobility and degradation reactions [95]. Therefore, the low HYG observed in this study could favor the incorporation of the MPs into dry or intermediate-moisture food systems by reducing caking and preserving the functional properties of the encapsulated compounds. However, further long-term storage studies are required to confirm their technological stability and shelf life.

3.2.3. Total Phenolic Compounds

Phenolic compounds are high-biological-value antioxidants which are of interest to the functional food industry [96]. This parameter indicates the amount of this compound retained in the raw materials after spray drying, which is a key indicator of increased nutritional, functional, and commercial value. The TPCs obtained in this study ranged from 2202.43 to 2803.29 mg GAE/100 g, reaching its maximum value (assay 9) at an intermediate temperature (135 °C) and a polymer ratio (GA:MD) of 50:50, while the minimum value (assay 6) was reached at a higher temperature (145 °C) with the same polymer ratio. After spray drying, the retention percentage of TPCs ranged from 55.56 to 70.71%. These results show that, although the encapsulation matrix partially protects the TPCs and the combined use of GA:MD favors greater retention, an excessive increase in IAT can induce their degradation.

The thermal degradation observed in the present study is consistent with the findings of Do & Nguyen [68], who investigated the influences of the thermal intensity (120 to 160 °C) and the proportions of GA and microcrystalline cellulose in blackberry juice raw materials. Their findings showed that increasing the thermal intensity from 120 °C to 150 °C reduced the TPCs from 36.99 to 34.35 mg GAE/g. However, raw materials dried at 160 °C exhibited the highest TPCs (38.32 mg GAE/g), suggesting a possible effect of polyphenol polymerization at extremely high IAT.

This behavior is consistent with the recognized thermolability of phenolic compounds, whose exposure to severe thermal conditions favors oxidation, isomerization, and glycosidic bond cleavage reactions, reducing both their concentration and biological activity [97,98]. Tolun et al. [99] noted that thermal processing can result in thermal and oxidative degradation with a significant loss of natural antioxidants. In this regard, Cao et al. [100], in the microencapsulation of grape (*Vitis vinifera* L.) polyphenols using MD and GA, observed that the IAT and the concentration of the encapsulating material significantly influence ($p < 0.05$) the TPCs. They reported that TPCs decreased with increasing IAT, although the decrease was not statistically significant when increasing the IAT from 120 °C to 140 °C, while it was significant when the IAT was set at 160 °C. Interestingly, these authors observed an inverse trend in the TPCs of the microencapsulated products obtained at 180 °C, attributed to the polymerization of polyphenols at high IAT. This suggests that there is a critical thermal threshold beyond which degradation mechanisms can be partially compensated by polymerization reactions that increase TPC values. On the other hand, Vargas et al. [27] evaluated the effect of the spray drying process on bioactive compounds in açai pulp (*Euterpe oleracea* Mart.), confirming that IAT between 110 and 70 °C affected the retention of TPCs, with greater degradation at higher IATs, and that prolonged thermal exposure during drying compromises the structural integrity of the polyphenols.

Polyphenols are compounds sensitive to external conditions, particularly high temperatures where thermal decomposition, polymerization, and transformation reactions can occur [101], making the selection of encapsulating materials with complementary protective capacity essential. In this study, the composition of the encapsulating material was statistically significant ($p < 0.05$) and constituted the second determining factor for phenolic retention. Experiment 9, formulated with a polymer ratio (GA:MD) of 50:50, achieved the maximum TPC content. Rigon & Zapata Noreña [102], in their study on the drying of phenolic compounds from cranberry (*Vaccinium macrocarpon*) juice, reported that the TPCs in the MPs ranged from 5416 to 8219 µg GAE/g, and that those containing GA exhibited greater phenolic retention. They attributed this effect to the fact that GA contains 2% protein covalently bound to the carbohydrate chain through hydrophobic interactions, which acts as a protective physical barrier. Furthermore, they noted that the MD forms complexes that improve the stability of the polyphenols. In this regard Gaćina et al. [103] confirm the effect of the polymer mixture. These authors reported TPC values between 2106.56 and 2429.22 mg GAE/100 g in blackberry (*Rubus fruticosus*) MPs with encapsulant concentrations of 10% and 15% using GA at 140 °C, observing that the values decreased at lower IATs, which suggests an optimal IAT window where GA exerts its maximum protective capacity without suffering significant degradation. Borges et al. [104] evaluated IAT of 120, 150 and 180 °C in the microencapsulation of blackthorn flowers (*Prunus spinosa*) and reported a TPC retention of 4060.07 mg GAE/100 g (87.87%) under optimized parameters, achieving the highest concentrations of phenolic acids with the addition of 25% GA in MD, which confirms that the proportion of the encapsulant determines the maximization of this parameter.

Therefore, the TPC values obtained are consistent and demonstrate that process optimization requires a careful balance between the IAT and the encapsulant composition, which underlines that the protective capacity of the encapsulation matrix has thermodynamic limits that cannot be overcome solely by modifying the encapsulant.

The three-dimensional surface graphs (Figure 1) show the interaction effects of IAT and GA:MD polymer ratio on % PY, % HYG, and mg GAE/100 g TPCs of AEE MPs.

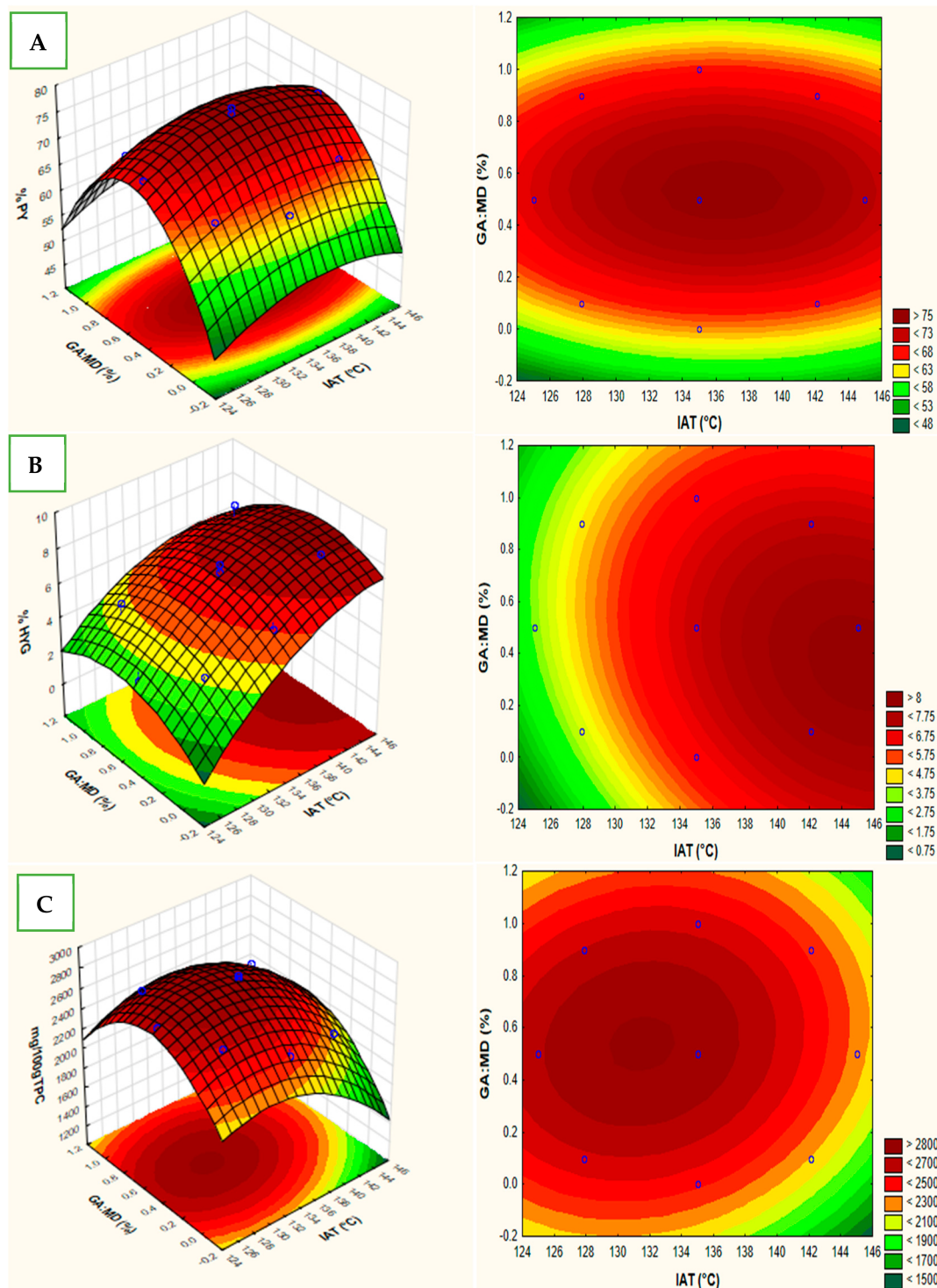


Figure 1. Three-dimensional response surface and contour plots showing the combined effects of inlet air temperature (IAT) and gum Arabic: maltodextrin ratio (GA:MD) on (A) powder yield (% PY), (B) hygroscopicity (% HYG) and (C) total phenolic content (mg GAE/100 g TPCs) of spray-dried açai (*Euterpe precatoria* Mart.).

3.3. Experimental Validation

The validation of the model was performed using confirmatory tests under conditions representative of the experimental design (assays 5, 6, and 9), comparing the values predicted by the regression equations with the experimental results for the response variables % PY, % HYG, and mg GAE/100 g TPCs (Table 5). Agreement was assessed using relative error, demonstrating the models' adequate predictive capacity.

Table 5. Comparison of predicted and experimental values of the response variables (% PY, % HYG and mg GAE/100 g TPCs) in the validation of the microencapsulation model.

Responses	IAT	GA:MD Ratio	Predicted Values	Experimental Values	% Error
% PY	125 °C	50:50	71.15	69.98 ± 1.05	1.67
	135 °C	50:50	76.46	76.05 ± 0.76	0.54
	145 °C	50:50	73.50	73.79 ± 1.09	0.39
% HYG	125 °C	50:50	3.58	3.47 ± 1.21	3.17
	135 °C	50:50	7.18	7.28 ± 0.93	1.37
	145 °C	50:50	8.30	8.13 ± 1.35	2.09
mg GAE/100 g TPC	125 °C	50:50	2686.49	2653.07 ± 2.82	1.26
	135 °C	50:50	2773.66	2791.83 ± 1.49	0.65
	145 °C	50:50	2254.71	2222.43 ± 2.63	1.45

The validation of the microencapsulation model developed in this study showed adequate agreement between the predicted and experimental values for the evaluated response variables (% PY, % HYG, and mg GAE/100 g TPCs), with relative errors less than 5% under all analyzed conditions (125, 135, and 145 °C) at a polymer ratio of 50:50. These results confirm the predictive capacity of the model within the experimental range, in accordance with spray-drying processes of fruit extracts, where low errors between predicted and experimental values are indicative of robust and applicable models [80,105–108].

% PY and mg GAE/100 g TPCs showed the lowest relative errors (0.39 to 1.67% and 0.65 to 1.45%, respectively), demonstrating a highly satisfactory model for these responses. In particular, the 135 °C IAT condition stood out for combining the highest experimental value of TPCs (2791.83 mg GAE/100 g) with one of the lowest prediction errors, supporting its suitability as an optimal condition. This validates the system's protective efficacy against thermal and oxidative degradation of phenols, consistent with studies highlighting microencapsulation as a barrier against volatility and reactivity, achieving efficiencies > 90% in tropical fruit polyphenols. In contrast, % HYG showed greater relative variability, with a maximum error of 3.17% at an IAT of 125 °C, suggesting a greater sensitivity of this property to microstructural changes in the system. This behavior can be attributed to the greater plasticization of the matrix at lower IATs, associated with higher moisture content and greater availability of hydrophilic sites, since at higher IATs (135 to 145 °C), the reduction in error (1.37 to 2.09%) indicates a better fit to the model, possibly due to the formation of denser structures with lower molecular mobility.

Therefore, it is confirmed that the optimal drying conditions are an IAT of 135 °C and a GA:MD ratio of 50:50. The results obtained in this study are consistent with the multi-objective optimization approach widely applied in the functional food industry, where experimental validation of the model is a key step for process scalability, allowing the definition of reliable operating conditions that guarantee the quality and stability of the microencapsulated product.

3.4. Morphology

Scanning electron microscopy (SEM) images of the MPs were taken at the optimal point, revealing a predominantly spherical morphology with a size ranging from 1.19 to 24.94 µm, with smooth to slightly wrinkled surfaces. Furthermore, no deep cracks or significant fractures were evident, and the presence of pores was limited. Figure 2 shows SEM micrographs of the optimized açai extract MPs.

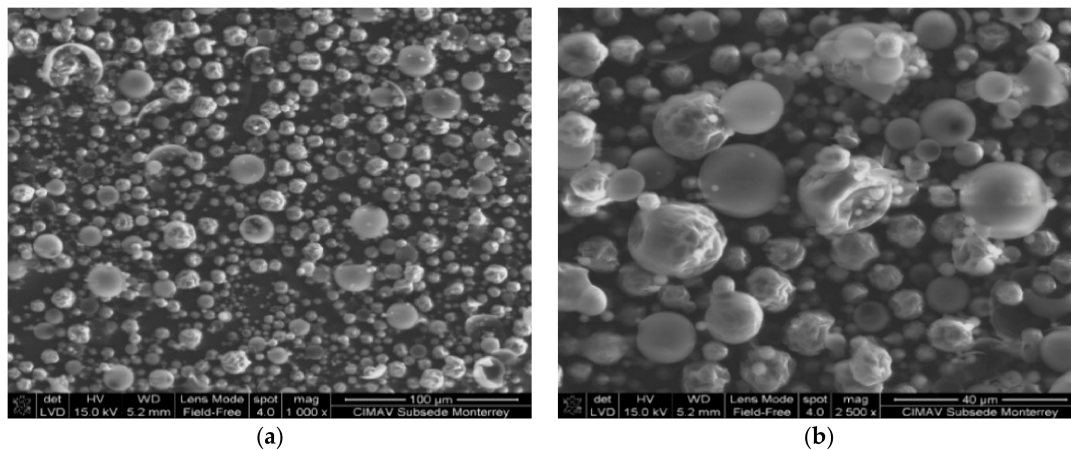


Figure 2. Scanning electron microscopy (SEM) images of spray-dried açai extract microparticles (MPs) produced under optimal conditions at magnifications of 1000 $\times$ (a) and 2500 $\times$ (b).

These results suggest an efficient microencapsulation process, which can be corroborated by the moderate degree of agglomeration between the particles, possibly associated with surface cohesive forces and the hygroscopic nature of the encapsulants and the phenolic compounds of the extract [109]. This behavior has been previously reported by Kalušević et al. [30] and Otálora et al. [110] in MPs obtained by spray drying with maltodextrin and gum Arabic, where the intermediate cohesiveness of the particles was described as a characteristic feature of the system and not as indicative of poor microencapsulation. In this sense, the spherical morphology and the absence of pronounced cracks could contribute to reducing permeability to oxygen and moisture, favoring the stability of bioactive compounds during storage [111,112], as reported by Tonon et al. [16].

These results are consistent with previous observations in spray-drying processes, such as those reported by Bastías-Montes et al. [113], who observed spherical shapes with smooth surfaces in pineapple (*Ananas comosus*) MPs, with diameters ranging from approximately 1.3 to 18.2 μm , except for the formulation containing inulin. They also observed a tendency toward agglomeration in all the compositions in their study, especially when using inulin. Similarly, Estupiñan-Amaya et al. [114] observed spherical morphologies in maqui (*Aristotelia chilensis*) extract under spray drying, where increasing the IAT resulted in powders with a larger average diameter.

On the other hand, regarding the size range, Valente et al. [26] characterized açai (*Euterpe oleracea* Mart.) MPs with uniform morphology and spherical MPs with a mean diameter less than 10 μm , while Negrão-Murakami et al. [86] reported spherical and wrinkled MPs from aronia juice with diameters of 24.8 μm , confirming that higher IAT increases the MP diameter. In particular, the MP sizes obtained in the present study agree with those reported by Estupiñan-Amaya et al. [114] in the microencapsulation of wild blueberry juice, who recorded MPs with average diameters of 7.09 μm using GA:MD, attributing the larger size to the high viscosity of GA which reduces the deformability of the drops during atomization; this value is within that reported by the present study.

In general, the observed spherical morphologies are characteristic of systems in which surface tension predominates during spray drying, favoring the minimization of surface energy. The GA:MD combination used in this study, while improving wall stability, generates morphological heterogeneity as a result of the competition between the rapid film formation provided by the MD and the structural strength conferred by the GA, leading to a mixed microstructure with differentiated properties. Likewise, the partial collapse observed in some MPs is attributed to the differential contraction between the still-wet core

and the rigid surface crust during the drying process, a phenomenon that occurs when the evaporation rate exceeds the capacity of the polymer matrix.

4. Conclusions

Spray drying proved to be an effective technique for the microencapsulation of açai extract using GA and MD at 8% total solids in the feed. The temperature at which the açai extract was absorbed had a predominant effect on the responses, followed by the GA:MD ratio. The polymer ratio primarily influenced the percentage of process yield, while the inlet air temperature determined the hygroscopicity (HYG) and the content of total phenolic compounds (TPCs). After spray drying, the retention percentage of total phenolic compounds ranged from 55.56% to 70.71%, demonstrating an adequate preservation of bioactive compounds during the microencapsulation process. In this regard, optimization allowed for the establishment of a temperature of 135 °C and a GA:MD ratio of 50:50, achieving a suitable balance between drying efficiency, physicochemical stability, and retention of phenolic compounds. Furthermore, experimental validation showed precise agreement between predicted and experimentally observed values, with relative errors less than 5%. In this context, the microparticles (MPs) exhibited a predominantly spherical morphology, with a wide particle size distribution and rough surfaces, demonstrating the efficiency of the microencapsulation process. Furthermore, the results suggest the potential of açai MPs for application as functional ingredients in food systems; however, additional studies, into such properties as water activity (A_w), glass transition temperature (T_g), and shelf life, are required to confirm their technological applicability and stability at a multiscale level.

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Abbreviations

The following abbreviations are used in this manuscript:

MD	Maltodextrin
GA	gum Arabic
MPs	microparticles

IAT	inlet air temperature
IATs	inlet air temperatures
Tg	glass transition temperature
% PY	process yield
% WSI	water solubility index
% HYG	Hygroscopicity
TAC	total anthocyanin content
TPCs	total phenolic compounds
GAE	gallic acid equivalents
C3G	cyanidin-3-glucoside
CCRD	central composite rotational design
AEE	açaí ethanolic extract
ANOVA	analysis of variance
R ²	coefficient of determination
RSM	response surface methodology

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