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ORIGINAL ARTICLE

Encapsulation of red araçá (*Psidium cattleianum* Sabine) peel extract: characterization and quantification of bioactive compounds

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Abstract

Red araçá is a fruit rich in bioactive compounds, mainly anthocyanins. This study aimed to encapsulate the anthocyanin extract from red araçá peel using maltodextrin and gum arabic as encapsulating agents in five formulations, and freeze-drying and spray-drying as drying methods. Freeze-drying with maltodextrin showed the highest retention efficiency (98.86%) and encapsulation of anthocyanins (38.63%), while spray drying resulted in lower retention and encapsulation values (80.87% and 33.45%, respectively). The addition of gum arabic reduced both encapsulation and retention efficiency. Freeze-drying extracts exhibited higher moisture and water activity compared to spray-drying extracts. Solubility was above 75% for all samples, and hygroscopicity was higher in spray-drying (16.57 to 19.33%). Color analysis revealed higher saturation and redder coloration in freeze-drying encapsulated extracts, especially in formulations with higher maltodextrin content. Freeze-drying also favored the retention of phenolic compounds. The antioxidant activity varied among the formulations, being higher in the samples containing Arabic gum, which showed greater DPPH inhibition percentages and lower IC_{50} values, regardless of the drying method. Results indicate that the choice of encapsulation method and agents can optimize both bioactive compound retention and antioxidant activity of the encapsulates.

Highlights

- Freeze-drying with maltodextrin achieved superior anthocyanin encapsulation efficiency.
- Spray-drying with gum arabic enhanced antioxidant activity and phenolic retention.
- Encapsulation improved physicochemical stability and functional properties of extracts.

Keywords Freeze-drying · Spray-drying · Red araçá · Maltodextrin · Gum arabic · Anthocyanin

Introduction

Native fruits are classified as such based on their natural origin, meaning they occur in specific regions without human intervention in their development. Over time, these fruits have adapted to unique ecosystems, playing a crucial role in maintaining local biodiversity (Brack et al. 2020). Brazil is the third-largest fruit producer globally, with an annual output of approximately 41.36 million tons; however, the potential of its wild fruits remains largely underexplored (Abrafrutas 2024).

The red araçá (*Psidium cattleianum* Sabine) is native to southern Brazil and belongs to the Myrtaceae family. It is characterized by its globose shape, slightly acidic juicy pulp, and numerous seeds. Rich in bioactive compounds,

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it contains high levels of phenolics, vitamin C, and unsaturated fatty acids (Pereira et al. 2018). These phytochemicals, particularly those concentrated in the fruit epicarp, contribute to health promotion and provide enhanced protection to plant tissues (Melo et al. 2020). Among them, anthocyanins stand out due to their relevance in disease prevention and food processing applications. However, anthocyanins are highly sensitive to light, pH variations, oxygen, and elevated temperatures (>70 °C), requiring preservation strategies to avoid degradation (Fang et al. 2020).

Encapsulation technology offers an effective approach to protect such phytochemicals, especially anthocyanins, by forming a physical barrier using wall materials or coatings. In addition to improving stability, encapsulation enables controlled release, increases bioavailability, masks undesirable flavors, and enhances bioaccessibility (Song et al. 2022). Freeze-drying is considered one of the most effective encapsulation methods, as freezing followed by sublimation minimizes water-mediated reactions and preserves both organoleptic and nutritional quality (Zhang et al. 2015). Spray-drying, in turn, is widely applied in the food industry due to its cost-effectiveness, operational simplicity, and ability to produce dry particles with good stability and low moisture content (Song et al. 2022).

Despite the nutritional potential and industrial relevance of red araçá, research on its cultivation, processing, and application in human nutrition remains limited (Mallmann et al. 2020). Existing studies have primarily focused on extraction and quantification of phenolic compounds, especially anthocyanins, and on evaluating the antioxidant properties of its peel (Meregalli et al. 2020). However, the optimal encapsulation strategy for preserving anthocyanins and phenolics from red araçá peel remains unknown. While extraction methods have been investigated, no study to date has systematically compared different drying methods and wall materials for stabilizing red araçá bioactive compounds.

Given this gap, this study specifically evaluates how different encapsulation strategies affect the preservation of anthocyanins from red araçá peel extract. Our hypothesis is that both the drying method (freeze-drying or spray-drying) and the choice of wall material (maltodextrin/gum arabic) significantly influence color preservation, retention efficiency, and encapsulation yield. Therefore, the objective of this work was to encapsulate and characterize red araçá peel extract using these two encapsulation methods and wall materials, clarifying which combination provides the most effective stabilization of bioactive compounds.

Table 1 Different formulations used in the encapsulation by freeze-drying (FD) and spray-drying (SD) of the anthocyanin extract from the Peel of red araçá (*Psidium cattleianum* Sabine)

Formulation	Maltodextrin - MD (%)	Gum arabic - GA (%)
FD1 and SD1	100	0
FD2 and SD2	70	30
FD3 and SD3	50	50
FD4 and SD4	30	70
FD5 and SD5	0	100

Materials and methods

Sample preparation and extraction

The red araçá fruits were harvested at coordinates −27.841692°, 52.302515°. A sample of the plant with branches, leaves, and fruits was incorporated into the Herbarium Pe. Balduíno Rambo at URI-Erechim, under the exsiccate registration number 12,609. The fruits were harvested between February and March 2023. The harvest point was established when the lower portion of the fruit showed a dark red color. The fruits were chosen and cleaned by submerging them in a 200 ppm NaClO solution for 15 min and frozen at −18 °C until analysis.

The extraction was carried out based on the methodology of Meregalli et al. (2020), where 10 g of red araçá peel were crushed and placed in an Erlenmeyer flask. Add 100mL of 90% ethanol, acidified with 0.1% HCl and adjust the pH to 1.5. Extraction took place in the dark, at 40±1 °C, for 120 min. The samples were centrifuged at 8000 rpm for 15 min at 4 °C. The supernatant was collected and the precipitate was washed twice with 5 mL of solvent, followed by another centrifugation. The extract was then vacuum filtered, the final volume was adjusted to 20 mL, followed by storage protected from light at a temperature of −18 °C.

Preparation of the anthocyanin complex: encapsulating agent

The anthocyanin–wall material complexes were prepared following Rezende et al. (2018), with minor modifications to improve reproducibility and standardize the feed solutions for both drying methods. Initially, 5 mL of the anthocyanin extract was transferred to a beaker, and 10 g of wall materials, maltodextrin DE 20 (MD), and gum arabic (GA), in the proportions described in Table 1, were added. Distilled water acidified to pH 1.5 was then added to complete a final volume of 100 mL. This procedure resulted in a feed solution with a final solids concentration of approximately 10% (w/v), which was intentionally standardized for use in both freeze-drying and spray-drying, ensuring comparability between the two encapsulation processes. The mixtures were homogenized in an orbital shaker at 280 rpm for 10 min at 25 °C, under light protection, until complete dissolution of the wall materials. The resulting solutions were immediately subjected to the respective drying processes.

Samples were frozen at −18 °C and lyophilized for 48 h (Modulyo, Edwards). The encapsulated material was crushed with a mortar and pestle, and stored in amber bottles at −18 °C. Spray-drying was carried out using a spray-dryer (Labmaq, MSD 1.0) equipped with a 0.5 mm diameter injection nozzle. The feeding was carried out by a peristaltic pump under the following conditions: drying air inlet temperature of 120 °C, drying air outlet temperature between 45 and 50 °C, sample atomization pressure between 0.07 and 0.12 bar and average sample feed flow to the dryer of 350 mL/h. The encapsulated material was collected in an amber glass container and stored at −18 °C.

The determination of anthocyanin retention efficiency (ER) for all tests was performed by evaluating the anthocyanin content retained inside the formed capsule (IFC), in mg, and on its outer surface (ES), in mg, and comparing them with the amount of anthocyanins total (TAC), in mg, provided in the encapsulation process according to Eq. 1. The masses, in mg, were calculated considering the concentrations of the analyzed solutions (mg/L), corresponding to the extracts used in the encapsulation and the preparation processes of the encapsulated samples, and their volumes (L). For the encapsulated samples, a correction was made by extrapolating the result to the total mass obtained in the encapsulation process.

$$\mathrm{ER}\ (\%) = \left(\frac{\mathrm{IFC} + \mathrm{ES}}{\mathrm{TAC}}\right) \times 100 \tag{1}$$

For ES quantification, 200 mg of microcapsules were added to 2mL of a 1:1 (v/v) ethanol: methanol mixture and stirred for 1 min, at room temperature and protected from light. The dispersion was then filtered through 0.45 µm Millipore and read in a spectrophotometer at 535 nm.

To break the complex formed and quantify IFC 200 mg of capsules were added to 2 mL of a mixture of methanol: water: acetic acid 50:42:8 (v/v/v) and stirred for 1 min, at room temperature and protected from light. The samples were placed in an ultrasonic bath at a frequency of 50 kHz, 99 W, and a temperature of 4 °C, for 30 min. The supernatant was centrifuged at 5,000 rpm for 10 min, filtered and read using a spectrophotometer. The result obtained, after subtracting the ES value, corresponds to the IFC. The encapsulation efficiency (EE) was determined by the ratio between the concentration of encapsulated anthocyanins (IFC) and the total concentration of anthocyanins present in the added free extract (TAC) (Eq. 2), according to the methodology used, which was that of Robert et al. (2010), with some modifications.

$$\mathrm{EE}\ (\%) = \frac{\mathrm{IFC}}{(\mathrm{TAC})} * 100 \tag{2}$$

Physicochemical characterization of encapsulated

To characterize the encapsulated extracts, moisture, water activity, higroscopicity, degree of caking, solubilit and color were determined. The moisture content was determined using a moisture analyzer. A 3 g sample of the encapsulated material was placed in the device, and the measurement was recorded as a percentage (%). For water activity (a_w), the samples were equilibrated at 27 °C for 30 min before being measured with a water activity analyzer.

Hygroscopicity was determined following Cano-Chauca et al. (2005) with minor modifications to adapt the protocol to the sample size and to standardize equilibration conditions. Briefly, 1.0 g of dry powder was placed in a small, hermetically sealed container together with a tray containing a saturated NaCl solution (≈75% relative humidity). The containers were kept at 25 °C for 7 days to reach equilibrium. After equilibration, samples were removed, quickly weighed, and the hygroscopicity was expressed as grams of water absorbed per 100 g of dry powder.

Caking degree (confirmation of agglomeration). The degree of caking was assessed according to Jaya and Das (2004), with adaptation of the sample mass to 1.0 g. This procedure was used as a confirmatory test for moisture-induced agglomeration. One gram of each powder was conditioned at 75% relative humidity and 40 °C for 90 min to promote moisture uptake and potential agglomeration. Samples were then dried to constant weight in a ventilated oven, cooled in a desiccator, and sieved through a 500 µm mesh for 5 min. The fraction retained on the sieve was quantified and reported as the degree of caking. Results were classified following the GEA Niro Research Laboratory criteria. This approach directly links the hygroscopic behavior to the physical occurrence of particle agglomeration under controlled environmental stress.

The determination of solubility followed the methodology described by Cano-Chauca et al.(2005), with some adaptations. 1 g of the sample was added to 25 mL of water, and the mixture was shaken in an orbital shaker at 800 rpm for 5 min. The sample was centrifuged at 3,000 rpm for 5 min, and a volume of 20 mL of supernatant liquid was collected. This portion was then placed in an oven at 105 °C until constant weight. Solubility was calculated based on the relationship between the weight of the dry supernatant and the initial weight of the sample, expressed as a percentage.

Encapsulated extracts were analyzed using a Minolta colorimeter (model CR-400, Osaka, Japan), using the three-dimensional color coordinate system (L*a*b*). 1 g of encapsulated material was placed at the bottom of a small container, and the samples were then read. The chroma index (C*) was calculated using Eq. 3, and the Hue angle (hº), from Eq. 4:

$$C = \sqrt{(a^{*2}) + (b^{*2})} \quad (3)$$

$$\text{Hue}\,(\text{h}^{\circ}) = \arctan\ g\frac{b}{a} \quad (4)$$

Determination of bioactive compounds in encapsulates

The bioactive compounds determined were total anthocyanins, total phenolic compounds, flavonoids and antioxidant activity. For the encapsulated extract, the capsules were broken by adding 0.5 g in 5 mL of H_2O at pH 1.5. The samples were homogenized in a tube shaker for 1 min and placed in an ultrasound bath (100 Hz) for 5 min at a temperature of 4 °C. Centrifugation was then carried out at 3000 rpm for 5 min, and the supernatant was sent for analysis.

For the quantification of total anthocyanins (TA), the methodology used was that of Rosa Fetter et al. (2010), with absorbance reading at 535 nm in a spectrophotometer, using the solvent as a blank. Quantification was obtained through interpolation of the tangent linear regression to the standard curve of commercial cyanidin-3-glucoside.

The total phenolic content was determined following the methodology proposed by Singleton et al. (1999) with adjustments. 0.5 mL of the sample was added and then 2.5 mL of the Folin-Cicocalteu reagent at 0.2 N under stirring. The mixture remained at rest for 5 min, at room temperature and in the dark. Subsequently, 2 mL of 5% $NaCO_3$ and 50 µL of 10% LiCl were added to the mixture, incubating for 2 h in the dark and in a water bath at 25 °C. The absorbance measurement was carried out in a spectrophotometer at 760 nm. The results were expressed in Eq.mg of gallic acid.

The total flavonoid content was determined according to the methodology described by Zhishen et al. (1999). 0.25 mL of the sample was added to 1.25 mL of deionized water and 75 µL of 5% $NaNO_2$ solution. It was homogenized in a Vortex tube shaker, followed by resting for 6 min at 25 °C. 150 µL of a 10% $AlCl_3$ solution was added, stirred again and remained at rest for another 5 min at 25 °C. 0.5 mL of 1 mol/L NaOH solution and 2.5 mL of H_2O were then added while stirring (1200 rpm) and the measurement was taken on a spectrophotometer at 510 nm. The results were expressed in Eq.mg of catechin.

The antioxidant activity (AA) of the encapsulated extracts was determined by the DPPH (1,1-diphenyl-2-picrylhydrazyl) method Roesler et al. (2007; with adaptations) (Eq. 5). Samples (25 to 100 mg/mL; 400 µL) were mixed with 2000 µL of 0.1 mM DPPH in methanol and kept in the dark for 30 min. A blank was prepared with acidified H_2O and DPPH, and a control with sample and methanol. Absorbance was read at 515 nm, and results were expressed as % DPPH inhibition. IC_{50} values were obtained by linear regression.

$$AA\ (\%) = 100 - \frac{[(\text{ABS}_{\text{sample}} - \text{ABS}_{\text{blank}}) \times 100]}{\text{ABS}_{\text{control}}} \quad (5)$$

In which:

$\text{ABS}_{\text{control}}$ = 400µL of sample + 2000 µL of methanol.

$\text{ABS}_{\text{blank}}$ = 400 µL of acidified water + 2000 µL of DPPH.

Statistical analysis

All experiments were performed in triplicate. The results were subjected to statistical analysis using the STATISTICA program (version 5.0). Analysis of variance (ANOVA) was used to detect the significant difference between samples. The average values were compared with each other using the Tukey test at a 5% probability of error ($p \leq 0.05$).

Results and discussion

Encapsulation and retention efficiency

Figure 1 shows the visual appearance of the powders obtained by freeze-drying and spray-drying, highlighting clear differences in color preservation and particle morphology. The freeze-dried samples (FD) exhibited a more intense pink color, homogeneous structure, and porous appearance, features consistent with the reduced thermal degradation associated with freeze-drying. Because this process occurs under low-temperature conditions and removes water by sublimation, anthocyanin stability is preserved, minimizing oxidative and browning reactions during processing. In contrast, the spray-dried samples (SD) showed visibly paler coloration and more aggregated particles. This behavior is associated with the rapid and intense heating of the extract during atomization, which promotes oxidation and browning reactions, contributing to pigment discoloration (Ijod et al. 2024).

The composition of the wall materials also had a marked influence on color preservation. Formulations containing higher proportions of maltodextrin (FD1/SD1 and FD2/SD2) showed better pigment retention, whereas increasing the fraction of gum arabic resulted in more pronounced discoloration, especially in samples SD4 and SD5. This effect may be related to the greater ability of maltodextrin to form a continuous protective matrix around the anthocyanins, reducing their exposure to heat and oxygen. Gum arabic, although beneficial for properties such as emulsification and solubility, does not provide the same degree of thermal



Fig. 1 Visual comparison of encapsulated formulations containing different proportions of maltodextrin and gum arabic subjected to freeze-drying (FD) or spray-drying (SD). FD1 and SD1: 100% maltodextrin; FD2 and SD2: 70% maltodextrin and 30% gum arabic; FD3 and SD3: 50% maltodextrin and 50% gum arabic; FD4 and SD4: 30% maltodextrin and 70% gum arabic; FD5 and SD5: 100% gum arabic

protection, which becomes critical especially during spray-drying (Deng et al. 2023).

Figure 2 presents the retention and encapsulation efficiencies, quantifying the impact of the drying methods and formulations. The freeze-dried powders showed the highest retention efficiencies (92.15–98.86%), confirming the protective effect of lyophilization on thermosensitive compounds. Encapsulation efficiency was also superior in this method (25–39%), suggesting more effective formation of the encapsulating matrix under low-temperature conditions. In contrast, spray-drying resulted in substantially lower efficiencies, both for retention (57.39–80.87%) and encapsulation (20–33%), a behavior widely associated with thermal degradation and the limited time available for structural matrix organization (Xu et al. 2019). Moreover, the presence of sugars and organic acids in the extract may have favored agglomeration and partial particle collapse during atomization.

As also observed in Fig. 1, maltodextrin showed greater retention in both processes (FD1 and SD1), corroborated by the linear trends in Fig. 2, which reveal a strong positive correlation between increasing MD proportions and anthocyanin retention. In contrast, encapsulation efficiency (EE) showed low correlation with the MD/GA ratio in both drying methods, indicating that the wall material composition alone does not fully explain EE variability. Studies report that EE depends on specific interactions between bioactive compounds and wall materials, influenced by phenolic structure and reactivity, viscosity, and molecular organization of the matrix (da Rosa et al. 2014). EE is also affected by the core: wall ratio, droplet size, and drying conditions, rather than solely by the amount of wall agent (Zabot et al. 2022).

Furthermore, the literature shows that the synergy between maltodextrin (MD) and gum arabic (GA) is not linear: changes in GA concentration can alter the density and structure of the polymeric film formed, influencing retention in a non-proportional manner (Rahman Mazumder and Ranganathan 2020). GA, due to its branched structure and protein fractions, forms complex matrices that interact with the core but do not necessarily lead to progressive increases in encapsulation efficiency (Pérez-Pérez et al. 2024). While maltodextrin tends to form stable, low-hygroscopicity films that enhance anthocyanin protection, higher proportions of GA progressively reduced formulation performance. Intermediate mixtures of the two polymers (particularly FD3–FD4 and SD2–SD4) showed the lowest EE values, possibly due to the formation of less organized matrices with reduced capacity to entrap the bioactive compounds.

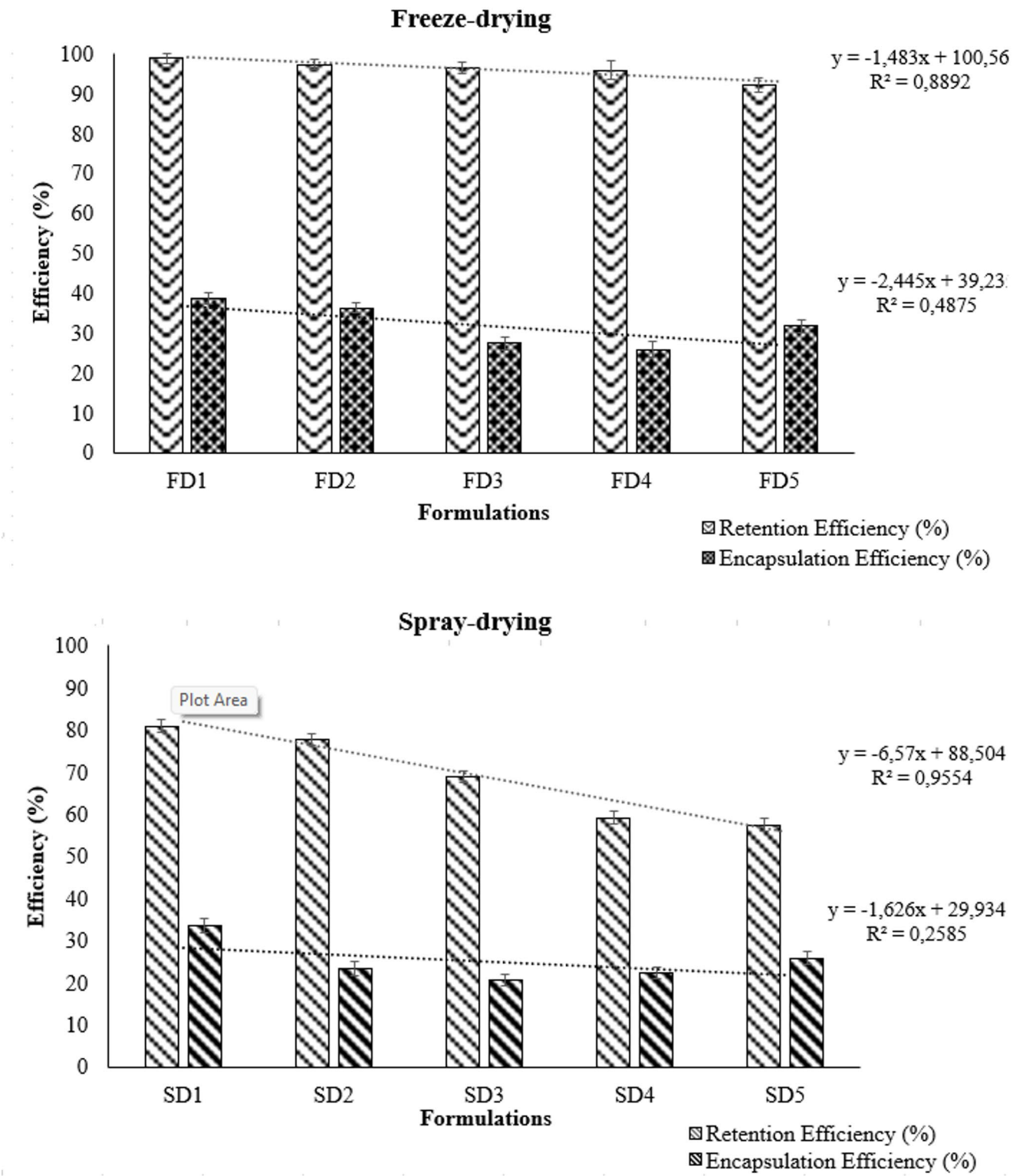


Fig. 2 Encapsulation efficiency (EE%) and retention efficiency (ER%) of anthocyanins from red araçá peel encapsulated by freeze-drying (FD) and spray-drying (SD) using different ratios of maltodextrin (MD) and gum arabic (GA). FD1 and SD1: 100% maltodextrin; FD2 and SD2: 70% maltodextrin and 30% gum arabic; FD3 and SD3: 50% maltodextrin and 50% gum arabic; FD4 and SD4: 30% maltodextrin and 70% gum arabic; FD5 and SD5: 100% gum arabic

Similar patterns have been reported for other anthocyanin-rich extracts encapsulated with maltodextrin (Bombana et al. 2023).

Physicochemical characterization of encapsulated

Table 2 presents the physicochemical characterization of extracts encapsulated with different proportions of MD and GA using freeze-drying and spray-drying, including measurements of moisture, water activity (aw), solubility, hygroscopicity, caking, and color. Freeze-dried extracts showed significantly higher ($p<0.05$) moisture (8.32–9.74%) and aw values (0.134–0.354) compared to spray-dried samples (1.88–4.11% moisture; 0.041–0.087 aw). These differences are related to the high temperature and rapid solvent evaporation in spray-drying, which result in lower residual moisture.

The low moisture content favors the stability of the encapsulated products, increasing their durability during storage, and water activity is equally important, as a food can be considered microbiologically stable with an aw of less than 0.6 (Negrão-Murakami et al. 2017).

When analyzing the different formulations with variation in MD and GA, it is clear that the greater the amount of MD, the greater the moisture content, regardless of the encapsulation method. MD, as it is a glucose polymer, can retain more water in its matrix due to the numerous hydroxyl groups (-OH), which form hydrogen bonds with water molecules. GA is a complex polysaccharide with a branched structure, which can limit the amount of water retained, resulting in lower moisture (Melo et al. 2020; Xiao et al. 2022).

Regarding solubility, all samples exhibited values above 75%, ranging from 75.42% to 89.61% for freeze-dried samples and from 79.80% to 85.62% for those obtained by spray-drying. Considering the wall materials, an increase in the proportion of maltodextrin in the formulations resulted in higher solubility of the extracts ($p<0.05$), regardless of the encapsulation method used. Although both maltodextrin (MD) and gum arabic (GA) produce highly soluble encapsulates, MD stands out due to its simplified linear molecular structure, lower molecular weight, and higher density of functional hydroxyl groups, which confer greater affinity for water. These characteristics enhance its dissolution capacity and consequently increase the solubility of the resulting formulations (Xiao et al. 2022).

Therefore, the choice between MD and GA as wall materials may be guided by the specific solubility requirements of the final product, as well as by the desired structural and physicochemical properties (Tonon et al. 2011). The high solubility of the encapsulated extracts is essential to ensure efficient reconstitution and homogeneous dispersion in the final applications. Moreover, it indicates that the particles

Table 2 Physicochemical characteristics of red araçá Peel extracts encapsulated by freeze-drying (FD) and spray-drying (SD) methods

Formulation	Moisture (%)	Aw	Solubility (%)	Higroscopicity (g/100 g)	Degree of caking (%)	Color parameters				
						L*	a*	b*	Croma	Hue°
Freeze-drying										
FD1	$9.74^{Ba}\pm0.43$	$0.354^{Ba}\pm0.08$	$89.61^{Aa}\pm0.79$	$13.58^{Ba}\pm1.54$	$8.35^{Ba}\pm0.14$	$95.38^{Aa}\pm0.77$	$7.39^{Ca}\pm0.15$	$12.31^{Ba}\pm0.14$	14.36^{Ba}	59.02
FD2	$9.51^{Ba}\pm0.24$	$0.315^{Ba}\pm0.04$	$83.14^{Ab}\pm1.02$	$11.83^{Bb}\pm1.95$	$6.56^{Bb}\pm0.21$	$89.88^{Bb}\pm0.25$	$5.42^{Ab}\pm0.29$	$14.57^{Cb}\pm0.22$	15.55^{Ba}	69.59
FD3	$9.37^{Ba}\pm0.43$	$0.163^{Bb}\pm0.01$	$81.27^{Ab}\pm0.99$	$11.55^{Bb}\pm1.25$	$6.35^{Bb}\pm0.18$	$93.44^{Aa}\pm1.32$	$3.49^{Ac}\pm0.65$	$14.07^{Cb}\pm0.91$	14.50^{Ba}	76.07
FD4	$8.75^{Bb}\pm0.21$	$0.158^{Bb}\pm0.02$	$77.79^{Bb}\pm0.35$	$7.32^{Cc}\pm0.94$	$4.75^{Bc}\pm0.32$	$88.5^{Bb}\pm0.66$	$3.51^{Ac}\pm0.63$	$16.85^{Db}\pm0.59$	17.21^{Cb}	78.23
FD5	$8.32^{Bb}\pm0.11$	$0.134^{Bb}\pm0.06$	$75.42^{Bb}\pm0.013$	$6.81^{Cc}\pm1.13$	$3.94^{Bc}\pm0.15$	$86.11^{Bb}\pm0.72$	$3.56^{Ac}\pm0.47$	$18.04^{Dc}\pm0.34$	18.39^{Cb}	78.84
Spray-drying										
SD1	$4.11^{Ab}\pm0.18$	$0.087^{Aa}\pm0.01$	$85.62^{Aa}\pm0.23$	$19.33^{Aa}\pm1.34$	$72.71^{Aa}\pm0.03$	$99.23^{Aa}\pm0.23$	$4.13^{Aa}\pm0.71$	$9.68^{Aa}\pm3.14$	10.52^{Aa}	66.89
SD2	$2.46^{Ab}\pm0.06$	$0.062^{Ab}\pm0.01$	$83.47^{Aa}\pm1.58$	$18.41^{Ab}\pm1.02$	$67.65^{Aa}\pm0.01$	$99.03^{Aa}\pm0.51$	$2.94^{Ba}\pm0.25$	$9.72^{Aa}\pm0.17$	10.16^{Aa}	73.17
SD3	$2.28^{Ab}\pm0.20$	$0.057^{Ab}\pm0.01$	$82.36^{Ab}\pm2.22$	$18.18^{Ab}\pm2.15$	$62.81^{Ab}\pm0.03$	$100.98^{Aa}\pm0.56$	$0.81^{Bb}\pm0.11$	$10.18^{Aa}\pm1.29$	10.21^{Aa}	85.45
SD4	$2.37^{Ab}\pm0.12$	$0.047^{Ac}\pm0.01$	$81.44^{Ab}\pm0.98$	$16.57^{Ac}\pm2.48$	$59.92^{Ab}\pm0.03$	$99.25^{Aa}\pm0.20$	$-0.735^{Bc}\pm0.11$	$11.10^{Ba}\pm1.23$	10.12^{Aa}	93.79
SD5	$1.88^{Ac}\pm0.09$	$0.041^{Ac}\pm0.02$	$79.80^{Bc}\pm1.35$	$16.90^{Ac}\pm2.08$	$54.22^{Ac}\pm0.02$	$96.6^{Ab}\pm0.23$	$-1.00^{Ac}\pm0.37$	$12.37^{Bb}\pm1.09$	12.41^{Aa}	94.62

In the same encapsulation method, means ± standard deviations with different superscript lowercase letters indicate significant differences ($p<0.05$) between samples. In the same column, means ± standard deviations with different superscript capital letters indicate significant differences ($p<0.05$) between encapsulation methods. Formulations: FD1 and SD1: 100% MD; FD2 and SD2: 70% MD:30% GA; FD3 and SD3: 50% MD, 50% GA; FD4 and SD4: 30% MD, 70% GA, FD5 and SD5: 100% GA

obtained are small and possess a large surface area, which favors water penetration and incorporation into the matrix (Mehta et al. 2022).

Between the evaluated processes, a distinct behavior was observed, likely associated with the composition of the wall materials. In the freeze-drying process, only the encapsulates composed of 100% maltodextrin exhibited higher solubility ($p<0.05$) compared to those obtained by spray-drying. However, when GA was used alone or in combination with MD, the opposite trend was observed, with spray-dried encapsulates presenting higher solubility than the freeze-dried ones. This suggests that the composition of the wall material directly influences the solubilization behavior of the encapsulates.

Other factors that may contribute to the observed differences in solubility include the residual moisture content and the morphological characteristics of the encapsulated materials, both directly related to the degree of agglomeration formed during processing. Morphologically, spray-drying tends to produce smaller particles with higher specific surface area, which favors both dissolution in aqueous media and agglomerate formation, as shown in Fig. 1. Regarding moisture (Table 2), samples with lower residual content generally exhibit a more porous and amorphous structure, increasing their ability to absorb water rapidly upon contact with the solvent and thus favoring solubility even in systems with higher agglomeration.

In this context, the results suggest a combined influence of formulation composition (MD and GA), processing conditions, and particle morphology on the final solubility. This variability is consistent with previous reports (Tonon et al. 2011; Mehta et al. 2022), which emphasize that, depending on the matrix and the type of encapsulated compound, the spray-drying process may produce particles with solubility equal to or even higher than that obtained by freeze-drying.

Hygroscopicity is significantly higher ($p<0.05$) in samples obtained by spray-drying, ranging from 16.90 to 19.3%, compared to freeze-dried samples, which range from 6.32 to 13.58%. This phenomenon occurs mainly because the structure of the capsules formed in the atomization process is more porous than the freeze-dried ones, resulting in greater exposure of the encapsulated particles, which contributes to increased hygroscopicity (Herrera-Balandrano et al. 2021). This parameter decreases significantly ($p<0.05$) with increasing GA concentration in each formulation. Due to its complex and branched molecular structure, GA can limit the availability of functional groups capable of interacting strongly with water (Tonon et al. 2011).

Furthermore, according to the classification of the Gea Niro Research Laboratory, (2024), all encapsulates obtained by spray-drying are hygroscopic. On the other hand, freeze-dried products with a higher maltodextrin content (FD1, FD2 and FD3) (50–100%) are slightly hygroscopic, while those composed mainly of GA (FD4 and FD5) are classified as non-hygroscopic. The degree of caking is significantly lower ($p<0.05$) in freeze-dried samples, ranging from 3.94 to 7.75%. For encapsulated products produced in spray-drying, the variation was from 54.22 to 72.71%, a fact directly related to hygroscopicity and solubility. According to the Gea Niro Research Laboratory, (2024) classification, samples can be classified as: no caking formation for freeze-dried encapsulates and with a lot of caking formation for spray-drying encapsulates.

Freeze-drying forms particles with lower hygroscopicity and a less porous structure, resulting in a reduced tendency to agglomerate. On the other hand, the particles formed by spray-drying are more porous and have greater surface exposure, which increases hygroscopicity and, consequently, the probability of agglomeration due to greater moisture absorption (Tonon et al. 2011; Mehta et al. 2022).

Regarding color parameters, spray-drying produced encapsulated extracts with higher luminosity values (L*), ranging from 96.6 to 100.98, compared to freeze-dried extracts, which ranged from 86.11 to 95.38, indicating that they are lighter ($p<0.05$). The higher percentage of moisture in the freeze-dried samples can contribute to the lower luminosity, since the remaining water can act as a medium that alters the reflection of light on the particles. The freeze-drying process can also cause a concentration of solutes on the surface of the particles, altering the perception of color (Różyło 2020; Xiao et al. 2022).

For a* values, freeze-dried samples presented significantly higher values ($p<0.05$) compared to spray-drying, confirming the visual aspect that also presents a greater tendency to red. The greater tendency to red in freeze-dried samples may be due to the process, which is less aggressive in terms of temperature and can protect the chromatic properties of the encapsulated compounds (Różyło 2020).

For parameter b*, samples whose encapsulating agent is GA present slightly higher values, significant ($p<0.05$) from 50% of the formulation of each encapsulate, indicating a slightly more intense yellowish tone. This fact is mainly due to the color of GA, which is naturally yellowish (Ashour et al. 2022).

The Chroma index, which represents color saturation, is a critical indicator of the chromatic intensity of samples. In freeze-dried samples, Chroma ranged from 13.19 to 16.30, while in samples obtained via spray-drying, values ranged from 9.68 to 12.41. The greater saturation in the freeze-dried samples can be attributed to the more effective preservation of natural pigments during the Freeze-drying process, which minimizes the thermal and chemical degradation of the encapsulated compounds (Xiao et al. 2022). Additionally, samples with a higher ($p<0.05$) percentage of gum arabic

Table 3 Bioactive compounds from encapsulated extracts of red araçá Peel obtained by freeze-drying (FD) and atomization processes via spray-drying (SD) using maltodextrin and gum Arabic as encapsulating agents

	Freeze-drying		Spray-drying	
	FD1	FD5	SD1	SD5
Total Anthocyanins (Eq. mg of cyanidin-3-glucoside /100 g of encapsulated extract)	$12.27^{A} \pm 0.16$	$8.68^{C} \pm 0.53$	$10.63^{B} \pm 0.39$	$5.89^{C} \pm 0.22$
Total Phenolics (Eq. µg of GAE/ 100 g of encapsulated extract)	$24.62^{C} \pm 0.19$	$41.1^{A} \pm 0.26$	$23.57^{C} \pm 0.16$	$31.56^{B} \pm 0.42$
Flavonoids (Eq. µg de catechin/ 100 g of encapsulated extract)	$23.65^{C} \pm 0.85$	$30.42^{B} \pm 0.19$	$18.11^{D} \pm 0.35$	$37.38^{A} \pm 0.29$
Antioxidant activity (% DPPH inhibition*)	$74.86^{D} \pm 0.14$	$85.76^{B} \pm 0.92$	$83.35^{C} \pm 0.12$	$89.52^{A} \pm 0.55$
Antioxidant activity IC_{50} (mg/g)	$26.55^{A} \pm 0.32$	$15.66^{D} \pm 0.22$	$18.51^{B} \pm 0.16$	$14.83^{C} \pm 0.41$

Means ± standard deviations with different superscript capital letters indicate significant differences ($P < 0.05$) between encapsulation methods. FD1 and SD1: 100% maltodextrin; FD5 and SD5: 100% gum arabic

(GA) in the formulation also present greater saturation. This is driven by the b* parameter, which reflects the intensity of the yellowish tone. Gum Arabic, being naturally yellowish, contributes to raising the b* value, resulting in greater color saturation.

The Hue angle, which reflects the tone or hue of the samples, presented values ranging from 59.02 to 94.62°. Lower Hue values (59.02 to 66.89°) obtained in isolated maltodextrin samples indicate shades closer to red, suggesting a greater amount of anthocyanins in the samples. The highest values ($p < 0.05$) were found for samples with gum arabic, showing shades closer to yellow, which can also be explained by the influence of the encapsulating agent.

Therefore, the presence of GA in the formulation not only influences the tone, but also increases the chromatic intensity of the encapsulates (Ashour et al. 2022). From the detailed analysis of the encapsulated characterization data, obtained using different encapsulation methods and different proportions of wall materials, it was determined that subsequent tests would be conducted exclusively with the variation of processing methods (freeze-drying and spray-drying) and with the use of homogeneous wall materials (100% maltodextrin or 100% gum arabic - FD1, SD1, and FD5, SD5, respectively).

Quantification of bioactive compounds

The bioactive compounds and antioxidant activity of formulations F1 and F5, encapsulated by freeze-drying and spray-drying, are shown in Table 3. Total anthocyanin (TA) content, expressed as mg cyanidin-3-glucoside/100 g, varied significantly ($p < 0.05$) according to the encapsulation method and wall material. In freeze-drying, FD1 showed the highest TA content (12.27 mg), while FD5 presented a lower value (8.68 mg). Similarly, spray-dried samples had higher TA ($p < 0.05$) in the 100% MD formulation (10.63 mg) compared to 100% GA (5.89 mg). These findings indicate that MD is more effective in retaining anthocyanins in both techniques, while freeze-drying proved superior to spray-drying. This behavior can be attributed to the molecular structure of MD, which enables the formation of a more stable protective matrix for anthocyanins during encapsulation.

Studies indicate that MD can better protect phenolic compounds from oxidative and thermal degradation, thus preserving the concentration of total anthocyanins. Deng et al. (2023), concluded that MD used as an encapsulating agent promoted a higher TA content in the encapsulated purple corn. On the other hand, GA has a more complex and branched structure that, although useful in certain encapsulation contexts, may be less efficient in protecting anthocyanins against degradation due to its lesser ability to form a uniform physical barrier around the encapsulated molecules (Saifullah et al. 2019). GA demonstrated greater efficiency in retaining phenolic compounds in both freeze-drying and spray-drying encapsulation.

In freeze-drying, the concentration of total phenolics was 41.1 µg GAE/100 g for GA, compared to 24.62 µg GAE/100 g for MD. In spray-drying, GA retained 31.56 µg GAE/100 g, while MD retained 23.57 µg GAE/100 g. This data shows that, regardless of the method used, GA tends to retain a greater amount of phenolic compounds than MD because, in its complex structure, GA has proteins and other polysaccharides that can interact with phenolic compounds, forming associations that stabilize these compounds and reduce their degradation during the encapsulation process (Ashour et al. 2022). This additional interaction can better protect phenolic compounds, resulting in greater retention compared to MD.

Comparison between spray-drying and freeze-drying encapsulation methods reveals that freeze-drying is more

efficient in retaining total phenolics for both encapsulating agents, as freeze-drying involves very low temperatures and a water removal process by sublimation, which minimizes thermal and oxidative degradation of heat-sensitive phenolic compounds (Yamashita et al. 2017). This method better preserves the integrity of bioactive compounds, resulting in higher retention of total phenolics. On the other hand, spray-drying operates at higher temperatures which, even remaining under this condition for a short time, may cause degradation of phenolic compounds during the encapsulation process (Huang et al. 2023).

The formulation obtained by spray-drying with 100% gum arabic (SD5) exhibited the highest antioxidant capacity ($p<0.05$) (89.52% DPPH inhibition) and the lowest IC_{50} value (14.83 mg/g), despite showing lower anthocyanin contents compared to the other formulations and lower total phenolics compared to the formulation containing 100% maltodextrin (Table 3). This result indicates that the observed antioxidant activity does not derive solely from the bioactive compounds originally present in the extract but also from the possible formation of new antioxidant molecules during thermal processing.

Although gum arabic is primarily composed of polysaccharides, it contains a relevant fraction of glycoproteins, a characteristic that distinguishes it from maltodextrin. This protein fraction enables GA to participate in Maillard reactions under intense heating conditions such as those occurring during spray-drying. Studies have shown that, in systems containing proteins and reducing sugars, free amino groups ($–NH_2$) can react under heating, forming intermediates such as Amadori compounds, which subsequently evolve into advanced products, including melanoidins (Kalušević et al. 2017). These melanoidins, besides contributing to matrix browning, are known to exhibit strong antioxidant activity, acting as radical scavengers, reducing agents, and metal chelators (Langner and Rzeski 2013).

In spray-drying systems containing proteins and carbohydrates, decreases in available lysine, development of brown pigments, and increases in fluorescence have been reported, all indicative of Maillard reaction product formation. These products may exhibit antioxidant activity similar to or even higher than that of the original phenolics, contributing significantly to the final antioxidant activity of the microparticles (Bolchini et al. 2025; Zhou and Langrish 2022; Liu et al. 2020).

Furthermore, the glycoprotein content of GA may favor an additional mechanism: partial denaturation of these molecules during heating can expose sulfhydryl or thiol (–SH) groups, which also possess antioxidant capacity, as reported in recent studies (Drozłowska et al. 2023). Thus, the antioxidant effect observed for SD5 likely results from the combination of the remaining bioactive compounds in the extract and the newly formed antioxidant compounds generated during thermal processing.

Although gum arabic did not show the highest retention efficiency for anthocyanins or phenolics, its active participation in melanoidin formation and the exposure of antioxidant functional groups appears to compensate for these losses in the context of antioxidant activity, particularly in spray-drying. In freeze-drying, where exposure to high temperatures does not occur, such reactions are not expected; nevertheless, the FD5 formulation exhibited high antioxidant activity ($p<0.05$) (85.76%), suggesting that the polymeric structure of GA also contributes to phenolic stabilization at low temperatures.

It is important to emphasize that the measured antioxidant activity reflects the combined effect of the original compounds and the newly formed compounds generated during processing. However, the present study did not directly quantify Maillard reaction products such as melanoidins, absorbance at 420 nm, fluorescence, or available lysine. Therefore, although the evidence strongly supports this mechanism, future studies are recommended to confirm this hypothesis.

Conclusion

Freeze-drying stood out as the most effective encapsulation method, reaching efficiencies above 90% and surpassing spray-drying, whose maximum efficiency was 81%. Among the wall materials evaluated, maltodextrin was the most effective, ensuring high encapsulation efficiency and improved extract solubility due to its ability to form a protective matrix that preserved anthocyanins by minimizing oxidative and thermal degradation. In contrast, gum arabic promoted better retention of phenolic compounds and higher antioxidant activity, particularly in spray-dried samples, likely due to the formation of melanoidins during the process. Overall, freeze-drying remained superior to spray-drying, especially regarding the retention of anthocyanins and phenolic compounds.

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Data availability Data will be made available upon request.

Declarations

Conflict of interest The authors declare that they are not aware of any conflicts of financial interest or personal relationships that may have influenced the work reported in this article.

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