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Microencapsulated Jaboticaba Berry (*M. cauliflora*) Juice Improves Storage Stability and In Vitro Bioaccessibility of Polyphenols

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Abstract

Jaboticaba berry is a rich source of polyphenols with bioactive properties. However, polyphenols are known for their high reactivity under environmental conditions, which poses a challenge to producing stable, functional components for the food industry. This study investigated the storage stability and bioaccessibility of polyphenols in microencapsulated jaboticaba juice over 21 days at three storage temperatures: −20 °C, 4 °C, and 25 °C. Additionally, phenolic compounds and antioxidant capacity were evaluated before and after in vitro simulated gastrointestinal digestion. Microencapsulation was performed by spray drying at 160 °C using maltodextrin at different concentrations (10%, 12%, and 15%) as the wall material. The results showed that the stability of polyphenols during storage was significantly influenced by both temperature and the proportion of maltodextrin. Greater degradation of phenolic compounds was observed at 25 °C, particularly in the formulation with 10% maltodextrin. On the other hand, the bioaccessibility of polyphenols was significantly higher in microencapsulated juice after simulated gastrointestinal digestion compared to non-encapsulated jaboticaba juice ($p < 0.05$). These findings suggest that microencapsulation technique improved the bioaccessibility of phenolic compounds in jaboticaba and promoted better stability with the use of a higher concentration of maltodextrin. In conclusion, microencapsulation is a promising strategy for the development of functional food products enriched with natural bioactive compounds, providing greater protection and efficiency in delivering their health benefits.

Keywords: Brazilian berry; spray drying; berry; phenolic compounds; antioxidant capacity

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1. Introduction

In recent years, berries have been studied extensively due to the presence of bioactive compounds in their composition. Jaboticaba is a berry native from the Brazilian Atlantic Forest that has a dark purple rind with an astringent flavor and white pulp with a sweet taste [1,2]. The health benefits of jaboticaba berry have been associated with its high antioxidant capacity and high concentration of polyphenols [3]. Studies have shown the

potential benefits of jaboticaba in glycemic control [4–6], vascular function [7], and free radical scavenging [8–10].

Jaboticaba can be consumed fresh or used in the manufacture of food products as a natural coloring or as a functional ingredient. Currently, its addition is studied in the production of yogurts [11,12], bakery products [3], sausages [13], cookies [14] and gelatin [15], suggesting a relevant application of this fruit for the food industry.

However, jaboticaba is a seasonal fruit that has a limited harvest period during the year, is highly perishable (shelf life of 2 to 3 days in ambient conditions) and its bioactive compounds are easily degraded during storage and processing due to various factors, such as temperature, pH, and oxygen, altering its nutritional benefits [16–19]. In addition, the conditions of the gastrointestinal tract can promote the degradation of polyphenols due to the difference in pH throughout the stages of digestion, such as oral, gastric, and duodenal phases. Also, the presence of digestive enzymes that end up acting on the food can cause losses during digestion, compromising the expected effect of the polyphenols present in jaboticaba [20].

In a study conducted by Martins et al. (2021) [21], it was demonstrated that certain processes employed in the extraction of phenolic compounds can also result in their degradation. Similarly, Zhang et al. (2022) [22] emphasized that extraction parameters such as temperature, pH, and exposure to oxygen significantly influence the integrity and stability of phenolic compounds, often leading to their degradation if not properly controlled. In light of these considerations, it is evident that the utilization of technologies that can enhance the stability and bioaccessibility of bioactive compounds present in foodstuffs—such as food microencapsulation by spray drying—may serve as an effective technological approach to mitigate the degradation of polyphenols and optimize their bioactivity.

Spray-drying is one of the most widely used microencapsulation techniques in the food industry due to its low cost, processing speed, and scalability, particularly when compared to methods such as freeze-drying [23]. The choice of encapsulating agent plays a key role in the efficiency and stability of the encapsulated compounds. Among the various wall materials available, maltodextrin is frequently used owing to its high solubility, low viscosity, and cost-effectiveness [24,25].

Therefore, the aim of this study was to evaluate the stability of polyphenols from microencapsulated jaboticaba berry juice during different storage temperatures and at different proportions of maltodextrin. In addition, the in vitro bioaccessibility of phenolic compounds and antioxidant capacity were evaluated, both from microencapsulated and non-microencapsulated jaboticaba berry juice. Thus, this study is based on the hypothesis that higher proportions of maltodextrins may enhance the stability of polyphenols in microencapsulated jaboticaba juice. Furthermore, microencapsulation may contribute to increased polyphenol bioaccessibility and antioxidant capacity during simulated digestion, compared to non-microencapsulated juice.

2. Materials and Methods

2.1. Microencapsulation by Spray Dryer

The jaboticaba berries were purchased from a local supermarket in Macaé City, Rio de Janeiro, Brazil (22°22′15″ S, 41°47′13″ W), and were washed in water to remove dirt and sanitized. Subsequently, the whole Jaboticaba berries (i.e., peel, pulp, and seeds) were blended in a mixer, and the resulting juice was filtered using a nylon filter cloth (80 mesh) to remove solid particles.

Jaboticaba juice was microencapsulated using maltodextrin as the carrier material at three different concentrations, expressed as weight/volume (w/v): jaboticaba juice with 10% w/v maltodextrin (MJ 10%), 12% w/v (MJ 12%), and 15% w/v (MJ 15%). The mal-

todextrin concentrations selected for this study were based on previous research involving microencapsulation of jaboticaba [26], which demonstrated that these levels yielded optimal results for the encapsulation process. The maltodextrin used in this study (Adcel, Belo Horizonte, MG, Brazil) had a dextrose equivalent (DE) of approximately 17.0. The solutions were homogenized using a magnetic stirrer and used as feed in the spray-drying process. Maltodextrin was chosen as the wall material due to its favorable technological properties, such as low hygroscopicity, and its widespread use in the production of powdered food ingredients [27].

The microencapsulation process was carried out using a mini spray dryer (Model B-290, Büchi, Flawil, Switzerland) with a standard diameter nozzle of 1.0 mm and evaporation capacity of 1.0 L/h. The experiments were carried out using an inlet temperature of 160 °C, and outlet temperature of 76 °C, with a feed flow of 0.52 L/h and an air flow of 80% [28]. Inlet temperatures between 140 and 180 °C and outlet temperatures between 70 and 90 °C are widely used in spray drying processes, as they provide a good balance between drying efficiency and the preservation of phenolic compounds and antioxidant activity. Previous studies [29–31] demonstrate that these temperature ranges favor the obtaining of powders with low moisture content, good flow properties, and the high retention of bioactive compounds.

2.2. Characterization of the Microencapsulated Jaboticaba

2.2.1. Proximate Composition and Sugar Profile Analyses

In MJ 10%, MJ 12%, and MJ 15%, the analysis of moisture, total lipids, proteins, and ashes content was performed according to AOAC (2012). Total carbohydrates were assessed as described by Merrill and Watt (1973) [32].

Glucose, fructose, and sucrose were analyzed according to Duarte-Delgado et al. (2015) [33] using a high-performance liquid chromatography (HPLC) system. Briefly, 1 g of the samples were solubilized in 45 mL of the 10 mM sulfuric acid, mixed, and centrifuged at 10,000× *g* for 15 min. Then, 20 µL of the supernatant was injected into the HPLC system (Shimadzu, Kyoto, Japan), which was equipped with an Aminex HPX 87H column (300 × 7.8 mm, Bio-Rad, Hercules, CA, USA), guard column (30 × 4.6 mm, Bio-Rad (Hercules, CA, USA)), and a refractive index detector. Isocratic elution was carried out using a mobile phase solution consisting of 10 mM sulfuric acid at a flow rate of 0.6 mL/min.

2.2.2. Water Activity

The water activity (a_w) was measured using an electronic water activity meter (Aqualab Dew Point 4TEV, METER Group, Pullman, WA, USA). Prior to measurement, samples were stabilized at 25 °C for 15 min to ensure consistent readings.

2.2.3. Solubility

Solubility was evaluated according to the method described by Rezende, Nogueira, and Narain (2018) [34]. Briefly, 1 g of the microencapsulated jaboticaba berry was dissolved in 100 mL of distilled water and stirred on a magnetic stirrer for 30 min. The solution was then centrifuged at 3000× *g* for 5 min. After centrifugation, 25 mL of the supernatant was collected and transferred to a Petri dish. The dish was placed in an oven (NT513, Novatecnica, São Paulo, Brazil) at 105 °C for 5 h to dry. Solubility was calculated as the percentage ratio of the dried solution's weight to the initial weight of the sample.

2.2.4. Hygroscopicity

Hygroscopicity was determined using the methodology described by Rezende, Nogueira, and Narain (2018) [34]. One gram of the microencapsulated jaboticaba berry

was placed in a desiccator containing a saturated sodium chloride solution (75.3% NaCl) at 25 °C. After seven days, the samples were weighed, and hygroscopicity was expressed as the percentage (%) of moisture absorbed.

2.2.5. Colorimetric Analysis

A colorimetric analysis was performed using a CM 600D portable spectrophotometer (Konica Minolta Sensing, Inc., Osaka, Japan) equipped with a xenon flashlight source (with UV filter). Measurements were taken in diffuse illumination mode, 40 mm integrator sphere size, 8-degree viewing angle, and silicon photodiode matrix detector). The following color parameters were evaluated: luminosity (L^*, 100 = white, 0 = black), red-green (a*, +a = red, −a = green), blue-yellow (b*, +b = yellow, −b = blue), chroma (c*), and hue angle (h°). These measurements were conducted for both microencapsulated and non-microencapsulated jaboticaba berry juice with maltodextrin proportions of 10%, 12%, and 15%.

2.2.6. Fourier Transform Infrared Spectroscopy (FTIR)

An FTIR analysis of the microencapsulated jaboticaba powder was conducted using a Frontier FTIR spectrometer (Shimadzu, Kyoto, Japan) with the ATR (Attenuated Total Reflectance) method. Samples were placed between the ATR accessory and the diamond crystal, and spectra were recorded in transmittance mode over the range of 4000 to 400 cm^{-1}. The resolution was set at 4 cm^{-1}, with 64 scans per sample.

2.2.7. Particle Size

The Mastersizer 2000 equipment (Malvern Scirocco® 2000 Mastersizer, Malvern, Worcestershire WR14, UK) was used for a particle size analysis. The samples were diluted in ultrapure and deionized water at a ratio of 1:100 using an Ultraturrax disperser for 5 min, and then the measurements were carried out in triplicate at 25 °C.

2.3. Determination of Bioactive Compounds and Antioxidant Activity

2.3.1. Analysis of Total Polyphenols

Total polyphenols were determined using the Folin–Ciocalteu (F-C) reagent [35]. Briefly, 500 µL of the supernatant from the sample extraction was mixed with 300 µL of 1.5 M hydrogen peroxide. The sample was vortexed and underwent the F-C assay by diluting 50 µL of the sample mixture with 800 µL of ultrapure and deionized water and 50 µL of 0.25 N F-C reagent. After 3 min, 100 µL of 1 N sodium carbonate was added. The mixture was incubated for 2 h in the dark, and the absorbance values were determined at 765 nm. The outcome data were expressed as gallic acid equivalents in mg/100 g (mg GAE/100 g), and the analyses were performed in duplicate.

2.3.2. Analysis of Antioxidant Capacity

Total antioxidant capacity was evaluated using the Trolox equivalent antioxidant capacity (TEAC) assay [36]. The 2,2′-azino-bis (3-ethylbenzothiazoline-6-sulphonic acid) (ABTS) for the stock solution was prepared from 7 mmol/L ABTS and 2.45 mmol/L potassium persulfate in a volume ratio of 1:1,and then incubated in the dark at room temperature for 16 h and used within 2 days. A 50 µL of the supernatant from the microencapsulated samples extraction was mixed with a 950 µL ABTS working solution, and the absorbance was taken at 734 nm after 6 min of incubation at room temperature. The percentage of inhibition of absorbance at 734 nm was calculated, and the results were expressed as µmol of Trolox equivalents (µmol TE/100 g) and analyses were performed in duplicate.

2.3.3. Storage Stability Procedures of Total Polyphenols and Antioxidant Capacity

The samples (i.e., MJ 10%, MJ 12%, and MJ 15%) were divided into 50 mL conical tubes (polypropylene) for each day of analysis to avoid possible contamination. The samples were stored for 21 days under different conditions: room temperature (25 °C), refrigeration (4 °C), and freezing (−20 °C). Samples were taken from different temperatures for analysis at 0, 7, 14, and 21 days of storage. These temperatures were chosen to meet the different conditions of physical state of the samples, namely ambient temperature (25 °C), refrigerated (4 °C), and frozen (−20 °C), according to Corleto et al. (2018) [37], who evaluated the stability of bioactive compounds from beet at the same temperatures.

2.3.4. Simulated In Vitro Gastrointestinal Digestion

Analyses of phenolic compounds and antioxidant capacity in MJ 10%, MJ 12%, and MJ 15% were performed before and after simulated in vitro digestion to estimate and compare bioaccessibility. In addition, an analysis was also performed for freeze-dried jaboticaba as a control (non-microencapsulated jaboticaba). To obtain freeze-dried jaboticaba, we used berries that were stored at −80 °C before the freeze-drying process. We used an L101 freeze dryer (Liobras, Campinas, Brazil). Jaboticaba berries were lyophilized for 48 h at −40 °C with a vacuum pressure of 0.05 mmHg. The dried samples were removed from the equipment flask, crushed with a blender, sifted through a domestic sieve with a 1 mm opening, and stored at −80 °C for further analysis. For the preparation of the control sample (non-encapsulated jaboticaba juice) the jaboticaba fruits were selected and sanitized, and the whole fruits (including peel, pulp, and seeds) were triturated. The resulting mixture was passed through a fine voile cloth to separate the liquid portion from the solid residue. The juice obtained was then freeze-dried under the same conditions as described above.

The in vitro digestion procedure was performed as previously described by Morgado et al. (2016) [38]. In the oral phase (OP), the samples were transferred to clean tubes and mixed with Hanks' balanced salt solution (with $NaCO_3$, without phenol red, sterile filtered, Sigma-Aldrich, Poole, UK) to create a final volume of 20 mL. In addition, the solution was incubated with 1 mL of alpha-amylase (240 KNU-T/g, Termamyl® 2x, Novozymes, Bagsværd, Denmark) at 37 °C for 10 min. An aliquot was stored at −20 °C. Then, the pH samples were adjusted to 2.0 with 1 mL of a porcine pepsin preparation (0.04 g pepsin in 1 mL 0.1 M HCl) and incubated at 37 °C in a shaking water bath at 90 rpm for 1 h for gastric phase (GP). An aliquot of each sample was removed and stored at −20 °C. After, 0.9 M sodium bicarbonate was added to adjust the pH for 5.3 followed by the addition of 200 µL of bile salts glycodeoxycholate (0.04 g in 1 mL saline), taurodeoxycholate (0.025 g in 1 mL saline), taurocholate (0.04 g in 1 mL saline), and 100 µL of pancreatin (0.04 g in 500 mL saline solution). The pH of each sample was increased to 7.4 with 1 M NaOH. Samples were incubated in a shaking water bath (95 rpm) at 37 °C for 2 h to complete the duodenal phase (DP). After the DP, a 500 mL aliquot of each sample was stored at −20 °C. The blank was prepared with identical chemicals but without a sample and underwent the same conditions.

2.4. Statistical Analysis

The normality of data was tested using the Shapiro–Wilk test, and the homogeneity of variances was examined with the Levene test. To identify differences in proximate composition, sugar profile, total polyphenols, total antioxidant capacity, and particle size between MJ 10%, MJ 12%, and MJ 15% samples, a one-way ANOVA was performed. To identify differences in the levels of total polyphenol and total antioxidant capacity during storage and before and after simulated in vitro digestion, a two-way ANOVA with repeated measures was performed. Additional post hoc tests with Bonferroni adjustment were

performed. Statistical significance was set at the 0.05 level of confidence. All analyses were performed using a commercially available statistics software package (IBM SPSS Statistics version 27 for Mac), and the results were expressed as means and standard deviations.

3. Results

3.1. Proximate Composition and Sugar Content of Microencapsulated Jaboticaba Berry Juice

Table 1 shows the values of centesimal composition and sugar profile of jaboticaba juice microencapsulated in different proportions of maltodextrin.

Table 1. Proximate composition and sugar profile from microencapsulated jaboticaba berry juice.

	MJ 10%	MJ 12%	MJ 15%
Proximate composition			
Moisture (%)	12.67 ± 0.26	9.50 ± 1.55	8.73 ± 0.27
Ash (%)	1.85 ± 0.01	1.45 ± 0.01	1.45 ± 0.04
Protein (%)	4.09 ± 0.23	2.78 ± 0.34	2.79 ± 0.24
Lipid (%)	0.65 ± 0.07	0.65 ± 0.07	0.6 ± 0.00
Carbohydrate (%)	80.75 ± 0.03	85.63 ± 1.82	86.43 ± 0.47
Sugars			
Sucrose (g/100 g)	0.014 ± 0.00	0.019 ± 0.001	0.024 ± 0.002
Glucose (g/100 g)	17.38 ± 0.90	20.08 ± 2.49	22.83 ± 3.35
Fructose (g/100 g)	16.29 ± 0.68	21.39 ± 4.91	26.94 ± 1.70

Data are expressed as means ± standard deviation. MJ 10%, microencapsulated jaboticaba berry juice with 10% maltodextrin; MJ 12%, microencapsulated jaboticaba berry juice with 12% maltodextrin; MJ 15%, microencapsulated jaboticaba berry juice with 15% maltodextrin. No statistical differences were found for any of the comparisons in this table.

Our results showed that the moisture content ranged from 8.73 ± 0.27% to 12.67 ± 0.26% (Table 1). One-way ANOVA revealed no statistical differences between the samples ($p > 0.05$), suggesting that the varying proportions of the wall agent had no influence on moisture content.

In the macronutrients, it was observed that the samples were composed mainly of carbohydrates (>80.75%), with low levels of proteins (<4.09%) and lipids (<0.65%). There was no significant difference in total lipids and protein content between the microencapsulated MJ 10%, MJ 12%, and MJ 15% ($p > 0.05$). MJ 10% had lower carbohydrate content than MJ 15% ($p = 0.041$). No significant difference was observed for carbohydrate content between MJ 10% and MJ 12% ($p = 0.062$) and between MJ 12% and MJ 15% ($p > 0.999$). The ash content for MJ 10% was higher compared to MJ 12% ($p = 0.002$) and MJ 15% ($p = 0.002$). No significant difference was observed in the ash content between MJ 12% and MJ 15% ($p > 0.999$).

Regarding the type and amount of sugars analyzed in the different samples of microencapsulated jaboticaba, no statistically significant difference was observed between the samples ($p > 0.05$), as can be seen in Table 1. However, we can observe an increase in sugar content as we increase the proportion of maltodextrin in microencapsulation, which may explain the higher amount of sugars present in the MJ 15% sample. In our analysis, a low glucose content was observed in MJ 15% ($p = 0.029$), with a significant difference in relation to the MJ 12% sample. On the other hand, sucrose levels were the lowest (<0.019 g/100 g) in all samples, followed by glucose and fructose, which is explained by the fact that the sample under analysis was a fruit.

3.2. Water Activity (a_w)

The values of water activity showed minimal variation among the samples, with the lowest value observed for MJ 15% compared with the other samples, as detailed in Table 2. However, no statistically significant differences were found ($p > 0.05$).

Table 2. Solubilities, hygroscopicity, and water activity from microencapsulated jaboticaba berry juice.

	MJ 10%	MJ 12%	MJ 15%
Solubility (%)	75.00 ± 6.83	78.00 ± 5.16	85.00 ± 6.00
Hygroscopicity (%)	26.50 ± 0.71	25.00 ± 2.83	23.50 ± 2.12
Water Activity (a_w)	0.30 ± 0.01	0.31 ± 0.02	0.28 ± 0.01

Data are expressed as means ± standard deviation. MJ 10%, microencapsulated jaboticaba berry juice with 10% maltodextrin; MJ 12%, microencapsulated jaboticaba berry juice with 12% maltodextrin; MJ 15%, microencapsulated jaboticaba berry juice with 15% maltodextrin. No statistical differences were found for any of the comparisons in this table.

3.3. Solubility

Solubility is a critical factor influencing the reconstitution capacity of microencapsulated jaboticaba berries [29] and can affect the availability of microencapsulated compounds, which is essential for incorporation into food products. In the present study, solubility values ranged from 75% to 85% (Table 2). The highest solubility was observed in the microencapsulated jaboticaba berry juice with 15% maltodextrin, although the other samples showed very similar values with no statistically significant differences ($p > 0.05$).

3.4. Hygroscopicity

In our analysis, the hygroscopicity values ranged from 23.50% to 26.50% (Table 2). The results indicate that hygroscopicity decreased as the concentration of the wall-acting agent increased (i.e., 10%, 12%, and 15% of maltodextrin). Specifically, the lowest hygroscopicity was observed in jaboticaba juice microencapsulated with 15% maltodextrin (MJ 15%) but without a statistically significant difference.

3.5. Colorimetric Analysis

The color analysis was performed in microencapsulated and non-microencapsulated jaboticaba juice, and its results are summarized in Table 3. Significant differences ($p < 0.05$) were observed in all color parameters between microencapsulated and non-microencapsulated jaboticaba juice, indicating distinct color characteristics between the two forms.

Table 3. Instrumental color parameters (means standard deviation) of microencapsulated and no microencapsulated jaboticaba berry juice.

	Jaboticaba Berry Juice	MJ 10%	MJ 12%	MJ 15%
*L**	33.18 ± 0.92 [a]	65.74 ± 0.32 [a]	69.19 ± 2.30 [a]	76.34 ± 1.67
a*	40.81 ± 2.39	23.47 ± 0.17 [b]	19.23 ± 0.92 [b,c]	16.55 ± 0.11 [b,c]
b*	17.72 ± 1.53	12.40 ± 0.04 [a,b]	10.88 ± 0.16 [a,b]	8.61 ± 0.10 [b]
c*	44.49 ± 2.8	26.57 ± 0.13 [b]	22.10 ± 0.87 [b]	18.68 ± 0.15 [b]
h°	23.44 ± 0.6	27.85 ± 0.26 [b]	29.53 ± 0.91 [a,b]	27.73 ± 0.54 [b]

Data are expressed as means ± standard deviation. MJ 10%, microencapsulated jaboticaba berry juice with 10% maltodextrin; MJ 12%, microencapsulated jaboticaba berry juice with 12% maltodextrin; MJ 15%, microencapsulated jaboticaba berry juice with 15% maltodextrin. Comparisons were performed considering the treatment for each parameter. [a] indicates a significant difference ($p < 0.05$) from MJ 15%. [b] indicates a significant difference ($p < 0.05$) from Jaboticaba berry Juice. [c] indicates a significant difference ($p < 0.05$) from MJ 10%.

The luminosity parameter (*L**) showed that the powders of the microencapsulated jaboticaba juice exhibited a lighter shade in relation to the non-microencapsulated jaboticaba juice. Notably, among the microencapsulated powders, the MJ 15% sample showed significantly higher luminosity values than MJ 10% ($p < 0.001$) and MJ 12% ($p = 0.002$), indicating a lighter color in the MJ 15% sample.

For the red-green parameter (a*), the non-microencapsulated jaboticaba juice showed a higher value compared to the microencapsulated jaboticaba juice powders, reflecting a significant loss of red color during microencapsulation. Significant differences were also

observed between the MJ 10% and MJ 12% ($p = 0.023$) and between MJ 10% and MJ 15% ($p = 0.001$).

The blue-yellow parameter (b*) showed a significant reduction in non-microencapsulated jaboticaba juice in relation to microencapsulated jaboticaba juice powders ($p < 0.05$). In addition, MJ 15% presented lower values (b*) than MJ 10% ($p = 0.002$) and MJ 12% ($p = 0.041$).

The chroma values (c*) indicated a more vivid color in the non-microencapsulated jaboticaba juice compared to the microencapsulated jaboticaba juice powders ($p < 0.05$). The shade angle (h°) increased significantly in microencapsulated jaboticaba juice powders compared to non-microencapsulated juice ($p < 0.05$), with a notable reduction observed between MJ 12% and MJ 15% ($p = 0.046$).

3.6. Fourier Transform Infrared (FTIR) Spectroscopy

From the FTIR spectroscopy, it is possible to evaluate the secondary structure of proteins and changes in the secondary structure in the presence of ligands. Figure 1 shows the spectra of non-microencapsulated jaboticaba juice and microencapsulated jaboticaba juice powder in different proportions: MJ 10%, MJ 12%, and MJ 15%. The spectra revealed broadbands in the region of 3000–3500 cm^{-1}, corresponding to unsaturated O-H hydroxyl bonds. Bands close to 2900 cm^{-1} were attributed to the stretching vibrations of the C-H bonds. In addition, several peaks have been observed in the region of 1500–1750 cm^{-1} and wide peaks in the region between 1000 and 1500 cm^{-1}.

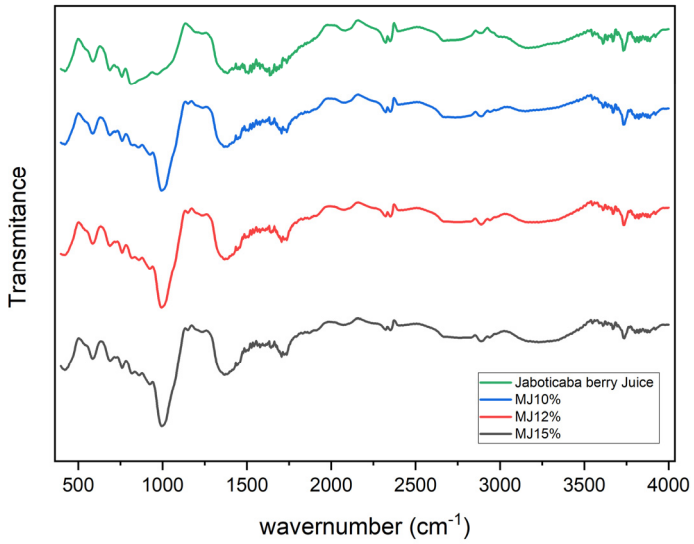


Figure 1. Fourier transform infrared analysis of microencapsulated and non-microencapsulated jaboticaba berry juice.

3.7. Particle Size

The microcapsules presented particle sizes with values of MJ 10% = 5.59 ± 0.04, MJ 12% = 7.08 ± 0.03, and MJ 15% = 7.35 ± 0.11 μm, where higher concentrations of maltodextrin led to the production of larger particles. A statistical analysis showed a significant difference between the three samples ($p < 0.05$).

3.8. Total Bioactive Compounds and Antioxidant Activity of Microencapsulated Jaboticaba Berry Juice

The non-microencapsulated jaboticaba juice, the pure fruit juice without the addition of maltodextrin, showed a total polyphenol content of 820.70 mg GAE/100 g, and was used as the reference sample to evaluate the effects of the microencapsulation process. This value was determined prior to spray-drying, using the original juice solution. Based on this refer-

ence, the total polyphenol content of the microencapsulated jaboticaba juice (MJ) prepared with different concentrations of maltodextrin was as follows: MJ 10% = 1370.92 ± 1.89, MJ 12% = 1300.00 ± 4.50, and MJ 15% = 1168.18 ± 45.45 mg GAE/100 g. The total antioxidant capacity of the non-microencapsulated jaboticaba juice was 3112.55 mM TE/100 g, whereas the microencapsulated samples presented values of MJ 10% = 11,863.77 ± 267.59, MJ 12% = 12,198.76 ± 125.17, and MJ 15% = 12,261.98 ± 35.76 mM TE/100 g. No statistically significant differences ($p > 0.05$) were observed in the total polyphenol content or antioxidant capacity among the microencapsulated samples.

3.9. Storage Stability of Microencapsulated Jaboticaba Berry Juice

3.9.1. Total Phenolic Content

At 25 °C, the analysis of the main effect of time revealed a significant reduction in total phenolic content in the MJ 10% sample on days 14 and 21 compared to day 0 ($p < 0.05$). Regarding the interaction effect, it was observed that the MJ 15% and MJ 12% samples exhibited higher phenolic compound concentrations on day 21 compared to MJ 10%, although without statistical significance ($p > 0.05$). Additionally, no significant differences were identified in the total phenolic content among the samples on days 0, 7, and 14 of storage.

On the other hand, the samples stored at 4 °C and −20 °C for 21 days showed no significant variations in total phenolic content ($p > 0.05$) (Figure 2).

3.9.2. Total Antioxidant Capacity

The changes in the antioxidant capacity of microencapsulated jaboticaba juice with different maltodextrin concentrations over 21 days of storage are presented in Figure 3. At 25 °C, a significant variation in antioxidant capacity was observed in the MJ 15% sample ($p < 0.05$) over time, with a reduction on day 14 compared to days 7 ($p = 0.004$) and 21 ($p = 0.026$). At the same temperature, comparing the samples, a significant reduction in antioxidant capacity was noted in the MJ 15% sample on day 14 compared to MJ 10% ($p = 0.011$) and MJ 12% ($p = 0.004$). At 4 °C, no significant differences were observed over time or between the samples during the 21 days of storage ($p > 0.05$). At −20 °C, there was a significant main effect for time ($p = 0.001$) and interaction effect ($p < 0.001$) throughout the stability analysis. At this temperature, there was a reduction in antioxidant capacity in the MJ 10% sample on day 21 compared to day 0 ($p = 0.009$). There was no significant difference in total antioxidant capacity for MJ 12% and MJ 15% over the 21 days of storage ($p > 0.05$). Regarding the interaction effect, on day 14, a significant reduction in antioxidant capacity was observed in the MJ 15% sample compared to the MJ 10% ($p = 0.035$) samples and MJ 12% ($p = 0.035$) samples. Furthermore, on day 21, the MJ 10% sample showed a reduction compared to the other samples MJ 12% ($p = 0.003$) and MJ 15% ($p = 0.003$) (Figure 3).

3.10. Simulated In Vitro Digestibility

3.10.1. Effects on Total Phenolic Content

Simulated gastrointestinal digestibility led to changes in the total phenolic content of jaboticaba juice, which can be seen in Figure 4. There was a significant main effect for time ($p < 0.001$) about the content of phenolic compounds, but no interaction effect ($p > 0.005$) between the samples. In relation to the main effect for time, the content of phenolic compounds increased after the in vitro digestion phases for MJ 10%, MJ 12%, and MJ 15%. However, there was no significant change in total phenolic content in non-microencapsulated jaboticaba juice after all phases of simulated digestion ($p > 0.05$). Before simulated in vitro digestion, the non-microencapsulated sample had a higher content of phenolic compounds than the MJ 10% ($p = 0.006$), MJ 12% ($p = 0.007$), and MJ 15% ($p = 0.009$)

samples. There was no significant difference between all the samples after OP, GP, and DP ($p > 0.05$).

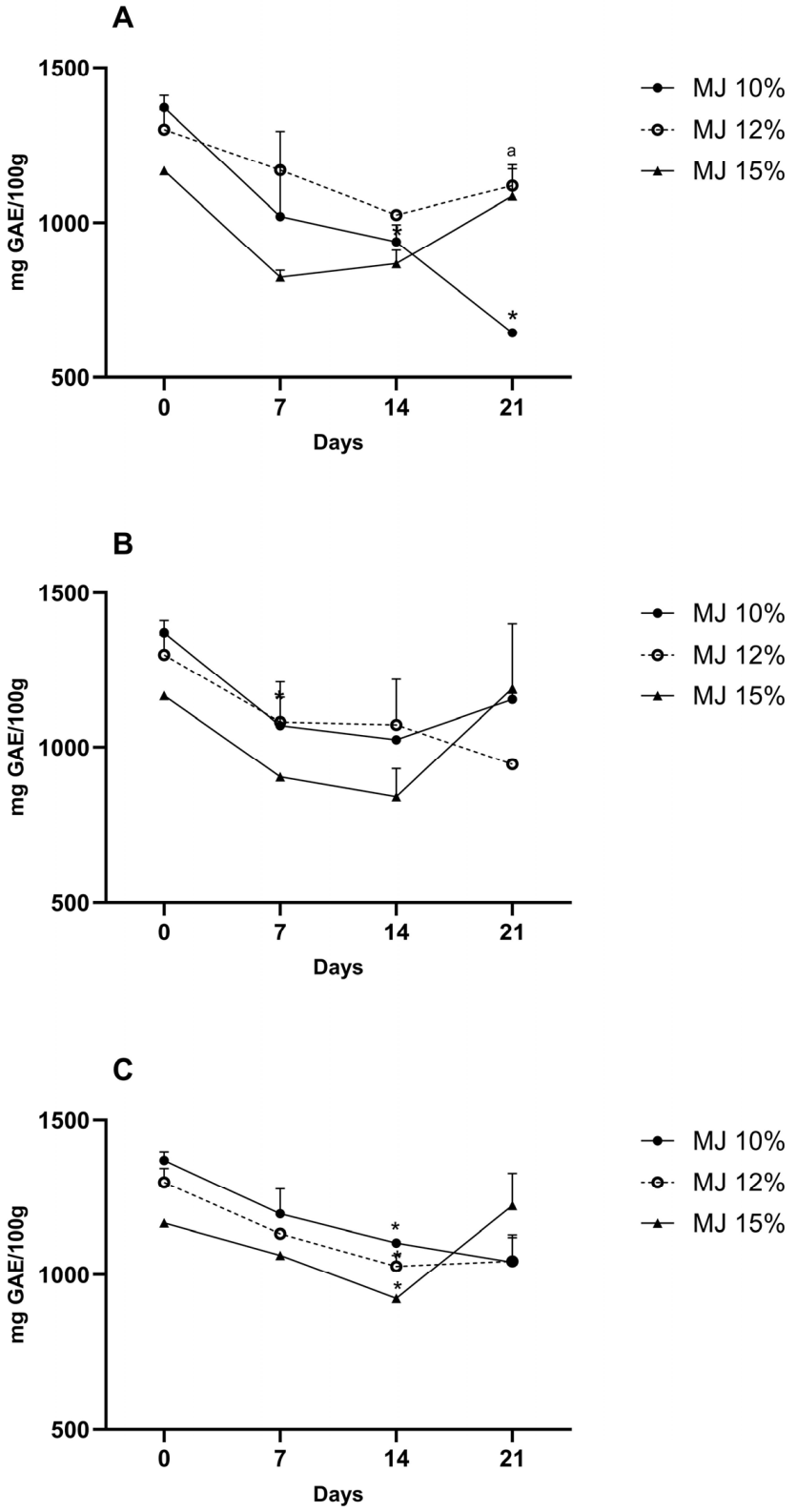


Figure 2. Stability analysis of the phenolic compounds—(**A**) 25 °C; (**B**) 4 °C; (**C**) −20 °C; MJ 10%: microencapsulated jaboticaba—10% maltodextrina; MJ 12%: microencapsulated jaboticaba—12% maltodextrina; MJ 15%: microencapsulated jaboticaba—15% maltodextrina. GAE: gallic acid equivalent. * Significant difference ($p < 0.05$) in relation to time. [a] indicates a significant difference ($p < 0.05$) between the samples.

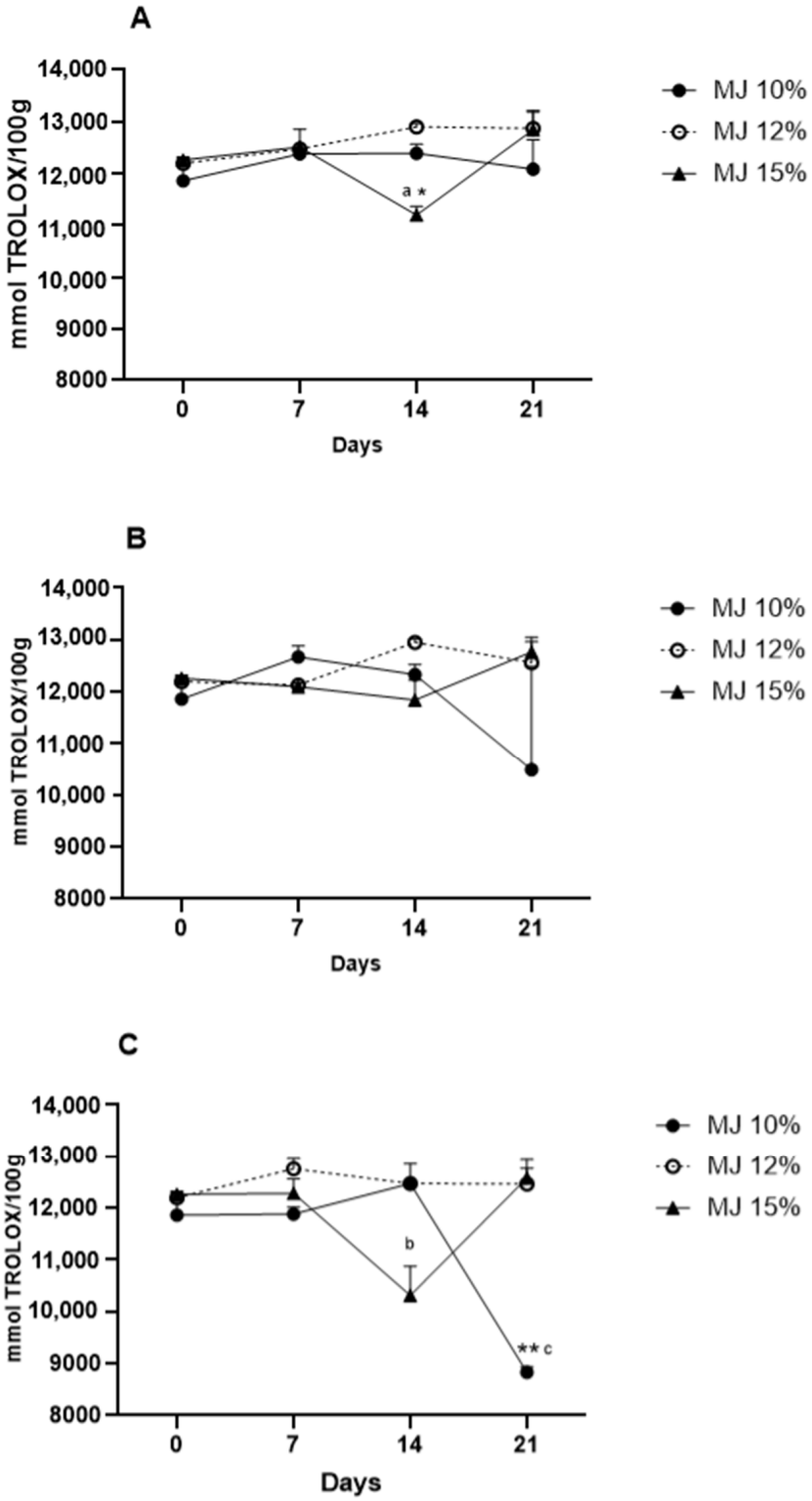


Figure 3. Stability analysis of the antioxidant capacity: (**A**) 25 °C, (**B**) 4 °C and (**C**) −20 °C; MJ 10%: microencapsulated jaboticaba—10% maltodextrina; MJ 12%: microencapsulated jaboticaba—12% maltodextrina; MJ 15%: microencapsulated jaboticaba—15% maltodextrina. * Indicates a significant difference ($p < 0.05$) in the MJ 15% sample in relation to time. [a] indicates a significant difference ($p < 0.05$) in the MJ 15%D sample compared to the other samples. ** ($p < 0.05$) indicates a significant difference in the MJ 10%D sample in relation to time. [a,b] indicates a significant difference ($p < 0.05$) between the samples; [c] indicates a significant difference ($p < 0.05$) in the MJ 10%D sample in relation to the other samples.

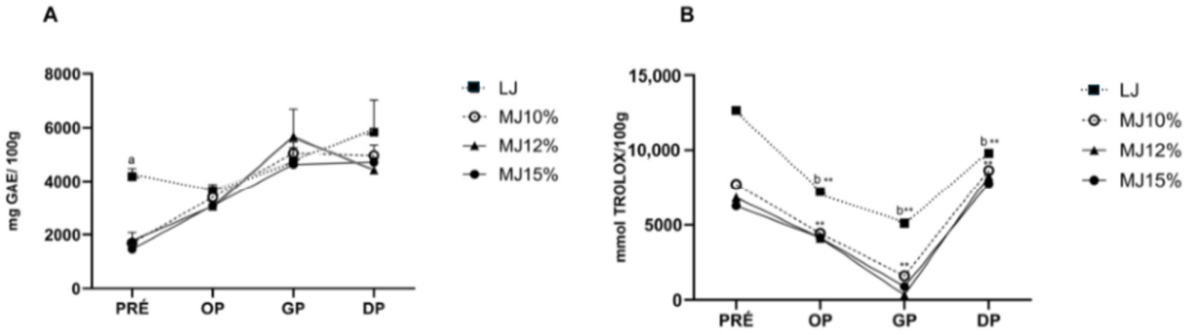


Figure 4. Simulated In Vitro Digestibility analysis: (**A**) Phenolic compound and (**B**) antioxidant capacity. PRE = phase before digestion begins; OP = oral phase; GP = gastric phase; DP = duodenal phase; MJ 10%: microencapsulated jaboticaba—10% maltodextrina; MJ 12%: microencapsulated jaboticaba—12% maltodextrina; MJ 15%: microencapsulated jaboticaba—15% maltodextrina. GAE: gallic acid equivalent. LJ: freeze-dried jaboticaba. ** ($p < 0.05$) denotes a significant difference in relation to the effect of time. [a] Denotes a significant difference in relation to the other samples. [b] Denotes a significant difference ($p < 0.05$).

3.10.2. Effects of Total Antioxidant Capacity

The microencapsulated jaboticaba juice underwent changes in total antioxidant capacity throughout the simulated gastrointestinal digestibility as can be seen in Figure 4, a significant main effect was observed for time ($p < 0.001$) and interaction ($p < 0.001$). Regarding the effect of time, the total antioxidant capacity in MJ 10%, MJ 12%, and MJ 15% was reduced in the OP and GP ($p < 0.05$), while the non-microencapsulated jaboticaba juice showed reduced total antioxidant capacity after all the in vitro digestibility phases. Regarding the interaction effect, the non-microencapsulated jaboticaba juice showed a higher total antioxidant capacity compared to the other microencapsulated samples, being MJ 10% ($p = 0.003$), MJ 12% ($p = 0.002$), and MJ 15% ($p = 0.001$). No significant difference was observed between MJ 10%, MJ 12%, and MJ 15% samples ($p > 0.05$). After the DP, no significant difference was observed in the samples during the simulated digestion ($p > 0.05$) (Figure 3).

4. Discussion

Our study demonstrated that microencapsulation of jaboticaba juice using maltodextrin, particularly at the highest concentration tested (MJ 15%), significantly enhanced the stability of phenolic compounds and antioxidant capacity. This effect was most pronounced during storage at −20 °C and throughout the in vitro digestion process. These findings support the protective role of maltodextrin as a wall material against enzymatic degradation and environmental stressors, contributing to improved bioaccessibility of jaboticaba's bioactive compounds. A summary of the key findings and their implications is presented in Figure 5.

The centesimal composition varied across formulations. The MJ 15% sample had the lowest moisture content, consistent with previous studies showing that increasing maltodextrin concentration promotes water reduction and improves the drying efficiency [27]. Moisture content and water activity are critical for powder stability. Our water activity values remained below 0.30, meeting industry standards for spray-dried powders [29,39], which supports their long-term stability. Higher ash and protein contents were observed in MJ 10%, likely due to a lower dilution effect from the smaller amount of wall material [40]. Similarly, higher sugar concentrations in MJ 10% may reflect this lower dilution, rather than differences in sugar content derived from the juice. This observation aligns with findings from Inada et al. (2015) [1] and Alezandro et al. (2013) [41], who reported that

microencapsulation with reduced wall material can concentrate intrinsic juice compounds due to lower matrix volume.

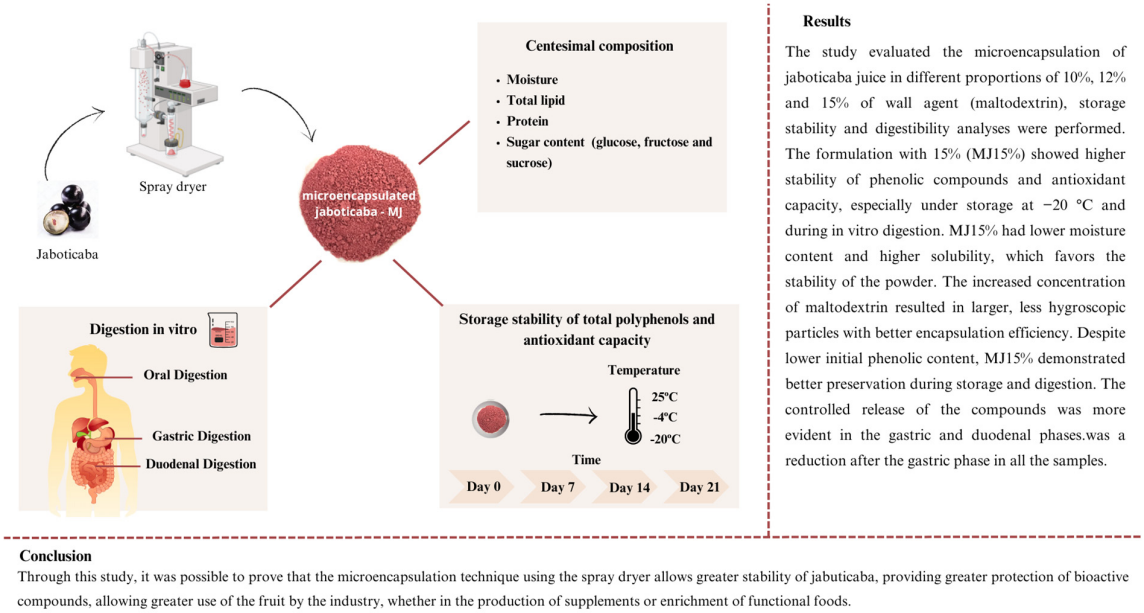


Figure 5. Summary graph of the effects of jaboticaba juice microencapsulation with different maltodextrin concentrations. The 15% formulation (MJ 15%) showed greater stability, improved release during digestion, and superior physicochemical properties.

The MJ 15% sample exhibited the highest solubility (85%), in agreement with other studies using maltodextrin for encapsulation [42]. The hydrophilic nature of maltodextrin and its ability to form a homogeneous matrix facilitate better dispersion in aqueous media. In contrast, MJ 10% showed higher hygroscopicity, which correlates with its higher moisture content and smaller particle size, increasing surface area exposure [29,43].

Particle size increased with maltodextrin concentration, forming larger microparticles. Particles demonstrated in the present study (5.59–7.35 µm) fall within the microparticle range defined by Silva et al. (2013) [44]. Larger particles contribute to reduced hygroscopicity and improved encapsulation efficiency, ultimately supporting controlled compound release during digestion.

Color parameters (L^*, a*, b*) confirmed visual differences between microencapsulated and non-encapsulated powders. Increased maltodextrin content resulted in lighter powders (higher L^*), due to pigment encapsulation within thicker wall layers [41]. An FTIR analysis revealed compositional changes attributed to maltodextrin. The region near 1000 cm^{-1}, associated with carbohydrate vibrations, was more prominent in microencapsulated powders. Bands between 1100 and 1400 cm^{-1} corresponded to amines [45], and phenolic-related peaks appeared between 1500 and 1750 cm^{-1} [46], with greater intensity in encapsulated samples.

The MJ 15% sample, despite showing lower initial total phenolic content, demonstrated better preservation during digestion and storage. Phenolic concentrations in our samples fall within the "medium" classification, according to Rufino et al. (2010) [47]. Antioxidant capacity was better retained in MJ 15%, especially under cold storage (4 °C). Although no statistical significance was observed, these differences may indicate that thicker wall layers may help shield compounds from oxidative degradation [46,48]. Phenolic content fluctuated across the 21-day storage period. At 25 °C, degradation was more pronounced, especially in MJ 10%, which had the thinnest wall layer. At −20 °C, MJ 15% showed an increase in phenolic content by day 21. This increase may be attributed

to enzymatic activity that promotes the degradation of plant cell wall structures and the release of phenolics previously bound to the matrix. Enzymes such as cellulases, pectinases, hemicellulases, β-glucosidases, and esterases (e.g., feruloyl esterase) can break down polysaccharide structures or hydrolyze glycosidic and ester bonds, thereby releasing phenolic compounds in simpler and more detectable forms. Corleto et al. (2018) [37], in their study on arugula, reported an increase in phenolic compounds after 30 days of storage, suggesting that enzymatic action may contribute to the rise in total phenolics even under post-processing conditions.

Microencapsulated jaboticaba samples consistently outperformed the non-microencapsulated sample during in vitro digestion. A progressive release of phenolic compounds was observed throughout the digestive phases, with significant increases during the gastric and duodenal stages. Antioxidant capacity followed a similar trend—declining in the oral and gastric phases but increasing in the duodenal phase. This suggests that microencapsulation supports controlled release, with maltodextrin offering effective compound protection along the gastrointestinal tract [46].

One limitation of the study was the lack of analyses to identify and characterize the specific phenolic compounds in jaboticaba. Additionally, the stability assessment was conducted only up to 21 days after preparing the microencapsulated jaboticaba powder. Future studies should extend this evaluation over a longer period, including a shelf-life analysis. For instance, Moser et al. (2017) [49] observed an increase in phenolic compounds in microencapsulated BRS Violeta grapes during the first 60 days of storage at 25 °C, followed by a gradual decline, highlighting the need for long-term stability studies. Moreover, the digestibility analysis of microencapsulated jaboticaba did not include the colon stage, ending at the duodenal phase. Future research could investigate the remaining digestive stages that were not covered in this study.

5. Conclusions

This study demonstrated that microencapsulation technology significantly increased the total phenolic content of jaboticaba juice compared to non-microencapsulated juice. Maltodextrin, used as a wall material, effectively protected these compounds from degradation during the spray-drying process. The highest maltodextrin concentration (MJ 15%) led to an increase in particle size while reducing moisture levels, water activity, and hygroscopicity. Additionally, the solubility of the microcapsules was directly influenced by maltodextrin concentration, while color parameters remained stable, indicating a protective effect against heat of the drying process. Microencapsulation effectively preserved the stability of phenolic compounds and the antioxidant capacity of jaboticaba over time and at varying temperatures compared to freeze-dried jaboticaba without maltodextrin. Moreover, the technique significantly increased the stability of jaboticaba's bioactive compounds throughout all stages of in vitro digestion, reinforcing its potential for functional ingredients development. These findings highlight microencapsulation with maltodextrin as a promising strategy for protecting the bioactive components of jaboticaba, improving its applicability in functional food formulations, and ensuring greater stability during refrigerated storage. Future research should explore the health benefits of consuming microencapsulated jaboticaba, along with its sensory properties and commercial viability. The microencapsulation technique presents opportunities for incorporating of jaboticaba into a wide range of food products, such as yogurts, cakes, and cookies, expanding its applications in the food industry.

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