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# Evaluation of freeze-dried phenolic extract from cashew apple by-product: Physical properties, *in vitro* gastric digestion and chemometric analysis of the powders

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ABSTRACT

The aim of this study was to produce powders from the phenolic extract of the cashew by-product using maltodextrin and gum arabic as encapsulating agents to preserve these bioactive compounds and their antioxidative activity. Extraction was assisted by an ultrasound bath to increase the release of the bioactive compounds, resulting in the hydroalcoholic extract from cashew bagasse. The powders were physically and morphologically characterized, and their total phenolics, antioxidant activity and bioaccessibility were evaluated. All parameters were analyzed by chemometrics. In addition, UPLC-HRMS analysis was used to evaluate the phenolic profile of the extracts, revealing that the powders were able to protect some of the original compounds of the extract, such as catechin, the myricetin fraction and quercetin. The powders showed high total phenolic retention capacity, especially maltodextrin (2893.34 ± 20.18 mg GAE/100 g (DW)), which was the encapsulant that preserved the highest content of polyphenols and antioxidant activity after bioaccessibility in comparison to the unencapsulated extract. The powders showed low water activity (<0.2), low moisture (<8%), high solubility (>60 %) and low hygroscopicity (<4%). The SEM analysis showed that lyophilized extract samples resembled broken glass, which is characteristic of the lyophilization process, and in addition to a predominantly amorphous structure as demonstrated by the X-ray diffraction. The extraction and encapsulation of phenolic compounds from the cashew by-product through lyophilization and using maltodextrin and gum arabic as encapsulants enabled their preservation and potential use of these compounds by the nutraceutical or food industry, and can be used as food additive in order to enrich the content of compounds and the antioxidant activity of numerous products.

## 1. Introduction

Cashews (*Anacardium occidentalle L.*) are one of the most economically important crops in Northeast Brazil. Cashew apples are rich in antioxidant compounds such as carotenoids (de Abreu et al., 2013), vitamin C (Rodríguez, Gomes, Rodrigues, & Fernandes, 2017) and phenolic compounds (de Brito, Pessanha de Araújo, Lin, & Harnly, 2007), including gallic acid, ellagic acid, quercetin 3-*O*-rhamnoside, myricetin, quercetin, and anacardic acid (Lopes et al., 2018). They are found in both the pulp/fibers and in the bark of the peduncle (Schweiggert et al., 2016). A comprehensive characterization of cashew apple phenolics was performed by Michodjehoun-Mestres et al. (2009a, 2009b), extracting both tannins and monomeric phenols, most abundantly found in the bark. The content and antioxidant capacity of alkylphenols in different cashew products (cashew apple, raw and roasted nuts and cashew nutshell liquid) were also reported, noting that cashew apple and its fiber exclusively contained anacardic acids, while cashew nuts contained both anacardic and cardol acids. Anacardic acids showed greater antioxidant capacity than cardols and cardanols (Trevisan et al., 2006). Thus, cashew apple by-products can constitute a raw

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material which is rich in antioxidant compounds to be used by the food production industry, reducing the large amount of agro-industrial residues currently generated in Brazil which are usually discarded into the environment or used as animal feed.

By-products from tropical fruit processing are considered excellent sources of bioactive compounds because of their antioxidant properties which are beneficial to human health (Ballesteros et al., 2017; da Fonseca Machado et al., 2018; Fonteles et al., 2016). In this regard, phenolic compounds (Fonteles et al., 2016; Rezende, Nogueira, & Narain, 2018) stand out due to their high antioxidant activity. However, such compounds are sensitive to light, oxygen and moisture (Ballesteros et al., 2017), so it is necessary to combine the extraction of these compounds with those that protect and stabilize them. Encapsulation is a widely studied alternative for this purpose which uses a wall material that protects the core, meaning the compounds of interest associated with a drying technique to produce a stable particle with good physical characteristics. Research on the use of freeze-drying to promote the retention of these antioxidants has been conducted on compounds extracted from acerola (Rezende, Nogueira & Narain, 2018), on anthocyanins from saffron petals (Mahdavee Khazaei, Jafari, Ghorbani, & Hemmati Kakhki, (2014) and even phenolics extracted from used coffee grounds (Ballesteros et al., 2017).

In addition, the choice of encapsulating agent is very important, as the efficiency and protection of the generated particles will be directly affected by this factor (Yamashita et al., 2017). Therefore, previous studies have demonstrated the use of maltodextrin (Fang & Bhandari, 2011) and gum arabic (Mahdavee Khazaei et al., 2014), or even a mixture of the two (Dag, Kilercioglu & Oztop, 2017; Ramírez, Giraldo & Orrego, 2015) as efficient encapsulators, as they guarantee the final product characteristics suitable for powder products, such as high water solubility, low viscosity and the ability to form stable solutions (Pereira Souza, Deyse Gurak & Damasceno Ferreira Marczak, 2017). Moreover, they enable good water retention for bioactive compounds when applied for this purpose, meaning they can improve the efficiency of the freeze-dried process (Yamashita et al., 2017).

Therefore, extracting and encapsulating polyphenols from cashew by-products were studied in this work as an alternative to use this material which is so rich and little used, with the objective of evaluating the use of maltodextrin and gum arabic associated with freeze-drying in the retention and protection of phenolic compounds extracted from the cashew by-product. Additionally, the effect of encapsulants on the simulated gastrointestinal digestion of these phenolic compounds and the antioxidant activity of the extracts was evaluated.

## 2. Materials and methods

### 2.1. Raw material and chemicals

Cashew apple bagasse (CAB) was donated by a local fruit pulp production industry (Fortaleza, Ceará, Brazil) as a by-product from cashew apple processing. All chemicals used were of analytical grade. Formic acid, acetonitrile, and methanol solvents were LC-MS grade (Merck, Darmstadt, Germany). Analytical standards were purchased from Sigma Aldrich (St Louis, USA). Maltodextrin (DE 20) and pure powdered gum arabic (CAS 9000-01-5 | 104228) was provided by Química Contemporânea Ltda (São Paulo, Brazil).

### 2.2. Preparation of CAB extract

Before the extraction process, the CAB was dried in a forced air circulation oven (Tecnal-TE-394/2) at 30 °C for 48 h until reaching 5.0 % moisture, and then crushed to reduce its size with an industrial blender. Next, a mixture of 1:10 (CAB sample: extracting solution) was prepared with hydroalcoholic solution (42.16 % ethanol) and taken to an ultrasonic bath (SolidSteel Ultrasonic Bath. SSBU), employing a method optimized by Silveira et al. (2021): frequency of 40 kHz and power of 100 W for 37 min at 30 ± 1 °C. Later, the mixture was centrifuged at 4500 rpm for 10 min at room temperature and filtered (Whatman filter paper no. 1). The supernatant was collected and concentrated in a rotary evaporator (Rotaevaporador RV 3 V Ika. 10003324) at a temperature of 40 °C during 2 h, yielding an extract with 9.0 % solids in the sample.

### 2.3. Preparation of freeze-dried CAB extract

Prior to the freeze-drying process, CAB extract was incorporated with 11 % (w/w) of encapsulants in each extract formulation so that its mixture with each encapsulant reached 20 % solids. Then, three formulations were prepared: CAB extract + maltodextrin = MD, CAB extract + gum arabic = GA, and CAB extract + maltodextrin with gum arabic (1:1, w/w) = MG. Afterwards, the formulations were frozen in an ultra-freezer (CL 90–40 V, Brand Terroni Scientific Equipment) at −38 °C for 24 h, and then dried in a freeze dryer (LS3000 Terroni, Scientific Equipment) at −50 ± 2 °C and 112 μmHg for 24 h. After drying, the granulometry of the samples was standardized in 30 mesh (600 μm) using sieves and were placed in a vacuum metallized package to protect them from light and oxygen until the analysis (Flowchart 1).

### 2.4. Moisture of the CAB phenolic extract

The moisture based on the gravimetric method to constant weight was determined according to method No. 943.06 (section 37.1.10B) of AOAC (2012).

### 2.5. Phenolic profile

The CAB extract was analyzed before and after being freeze-dried with the carrier agents. The UPLC-HRMS (Ultra-performance Liquid Chromatography coupled to High Resolution Mass Spectrometry) analyzes were accomplished according to the method previously described by Alves Filho et al. (2019). First, the chromatographic separations were performed on an Acquity/Xevo UPLC-ESI-qTOF system (Waters Co., Milford, MA, USA), equipped with an Acquity UPLC BEH C18 column (Waters, 150.0 × 2.1 mm × 1.7 μm) at 40 °C. The mobile phase composed of water and acetonitrile, both containing 0.1 % of formic acid, ranged from 2 to 95 % of acetonitrile in 15 min at a flow of 0.4 mL/min. The samples were filtered (Filter of PTFE 0.22 μm) and injected in aliquots of 5.0 μL. The mass accuracy and reproducibility were maintained by infusing 0.2 ng/μL leucine–enkephalin solution ($[M-H]^-$ ion at $m/z$ 556.2771) through lockspray at a flow rate of 20 μL/min. MS data were recorded for $m/z$ values in the range of 110–1180 Da in negative mode with a scan time of 0.1 over an analysis time of 19 min. The compounds were tentatively characterized through molecular formula provided by the MassLynx 4.1 software program from their accurate masses (error <5 ppm), isotopic patterns (i-fit) and MS fragmentation patterns, as well as literature survey on previous occurrence in Anacardiaceae family using the Scifinder Scholar database. In addition, compounds were assigned by comparison with reference standards when available. The citric acid, catechin, isorquercitrin (quercetin 3-O-glucoside), quercitrin (quercetin 3-O-rhamnoside) and quercetin standards were purchased from Merck.

### 2.6. Total phenolics (TP) and total antioxidant activity (TAA)

The cashew apple by-products were evaluated for Total Polyphenols (TP) and Total Antioxidant Activity (TAA) contents by FRAP in the CAB extract and the three freeze-dried extract formulations. In the case of powders, a dilution in distilled water was performed until reaching the initial total solids content of the formulations (20 °Brix). It was found that the encapsulants did not interfere in the readings measured in the wavelengths set up for the analyzes.

#### 2.6.1. TP analysis

The TP content was determined according to the methodology described by Obanda, Owuor and Taylor (1997) for CAB extract and freeze dried samples. First, 30 μL of each sample was diluted in 220 μL of distilled water for analysis. Then, 250 μL of the Folin-Ciocalteu reagent (Sigma-Aldrich, Germany) (1:3; v/v) plus 500 μL of distilled water were added and the mixture was kept at rest for 30 min in the absence of light at room temperature. Next, the absorbances were measured at 700 nm using a microplate analyzer (Synergyx Mx, Biotek, USA). Gallic acid (HPLC grade, Sigma-Aldrich) in the concentrations from 0 to 50 μg $mL^{-1}$ was used for external standard calibration curve and the results were expressed in mg of gallic acid equivalent (GAE)/100 g dry mass (DW).

#### 2.6.2. FRAP test

The iron-reducing antioxidant power test (FRAP) was conducted according to the method reported by Pantelidis, Vasilakakis, Manganaris, and Diamantidis (2007) in the CAB extract before and after freeze-drying. For the CAB extract and freeze-dried samples, 90 μL of the extracts (in different dilutions) solubilized in 270 μL of water reacted with 2.7 mL of the FRAP reagent (0.3 M acetate buffer, 10 mM TPTZ solution (2,4,6-Tris (2-pyridyl)-s-triazine) and 20 mM ferric chloride solution) in a dark environment during 30 min in at a temperature of 37 °C. The absorbance was measured at 593 nm using a Jenway 6705 UV/Vis spectrophotometer, and the antioxidant activity was calculated from a standard calibration curve prepared with ferrous sulfate solutions (500–1500 μM). The results were expressed in μM ferrous sulfate/g (DW).

### 2.7. Powder characterization

#### 2.7.1. Physical analysis

The moisture content was determined as described in item 2.4. Water activity (aw) was measured using a water activity analyzer (AQUALAB, 3TE Decagon series, Pullman, USA) at 25 °C.

Next, we employed the methodology described by Cano-Chauca et al. (2005) with a few modifications for the solubility analysis. To do so, 25 mg of sample plus 25 mL of distilled water were added in a beaker and homogenized until complete dissolution. Then, the solution was centrifuged at 3000 rpm for 10 min and a 20 mL aliquot of the supernatant was transferred to a pre-weighed Petri dish, which was subsequently dried in an air circulation oven at 105 °C until constant weight. Solubility was calculated and expressed as a percentage between the final weight after drying and the sample weight.

The hygroscopicity analysis was performed according to Cai and Corke (2000), with some modifications. A previously weighed Petri dish containing the sample (1 g) was placed in an airtight glass container containing saturated NaCl solution (75 % RH). The samples were kept in this condition for 90 min at 25 °C, after which they were weighed. Hygroscopicity was expressed in g of adsorbed moisture/100 g of dry matter.

#### 2.7.2. Microstructure

The morphology of the lyophilized extracts was evaluated by Scanning Electron Microscopy (SEM) with the images obtained by an Inspect S50-FEI Scanning Electron Microscope. The samples were initially covered with a gold and platinum film (Au-Pd, 35 nm) and subjected to an acceleration voltage of 10 kV.

In addition to SEM, the crystalline phases of the powders and encapsulants were evaluated by X-ray diffractometry (XPert Pro MPD model - Panalytical) with the analysis parameters of 35 kV voltage and current of 25 mA, dispersion angle varying 10–100° 2θ, step size 0.04 and 1 s of exposure in each step.

### 2.8. In vitro simulated gastro-intestinal digestion

The *in vitro* analysis simulating the physiological digestion process was performed according to de Lima et al. (2014), consisting of two sequential phases: gastric and enteric digestion. In the gastric phase, the samples (2 g for powder and 5 g for liquid extract) were diluted in 100 mL of 0.01 M HCl solution, pH 2.0 (adjusted with 6.0 M HCl), in which a pepsin solution was added (300 U/mL). The mixture was homogenized, transferred to an Erlenmeyer and incubated at 37 °C in a shaking water bath for 2 h at 100 rpm.

Next, titration with 0.5 mol/L NaOH was performed until pH 7.5 to simulate the pH found in the intestine of an individual. Dialysis was performed for two hours in dialysis membranes (33 × 21 mm, molecular weight: 12.000–16.000, porosity: 25 Angstrons – INLAB, Brazil) containing 0.1 mol/L $NaHCO_3$ equivalent to titratable acidity. After the pH adjustment, the dialysis membranes were added and stirred in a water bath at 37 °C/30 min at 100 rpm, then 5.0 mL of pancreatin solution and bile salts were added and stirred in a bath at 37 °C/2h. This step simulates the digestion of food in the intestine. The contents of the membrane (dialysate) were then removed at the end of this step. Immediately after the enteric phase, 2 mL of each sample was extracted and analyzed for TP and TAA concentrations as described above. The results were expressed in mg GAE/100 g (DW) and in μM $Fe_2SO_4$/g (DW), respectively, and in percentage of bioaccessible material (%) for TP and TAA through the difference of its final content in the digested material and the initial content found in the extracts before and after encapsulation.

### 2.9. Statistical analysis

All experiments were carried out in quintuplicate (n = 5). The results were evaluated by analysis of variance (ANOVA) and subjected to the Tukey's test to determine significant differences ($p < 0.05$) between the samples using the Statistica 10.0 statistical software program (StatSoft, 2010). The graphs were made using the Origin Pro version 8 software program (OriginLab Corporation, Northampton, MA, USA).

### 2.10. Chemometric analysis

The previous physical and chemical parameters (total phenolics, total antioxidant activity, bioaccessibility of total phenolics and total antioxidant activity, moisture, water activity, solubility, and hygroscopicity) determined for the phenolic extract of cashew apple by-product before and after freeze-drying with different encapsulating agents (maltodextrin; gum arabic; and a mixture of 50 % of both materials) were used to create a numerical matrix to perform the multivariate analysis. The matrix was imported by the PLS Toolbox™ program (version 8.6.2, Eigenvector Research Incorporated, Manson, WA, USA) for supervised analysis by Partial Least Square Discriminant Analysis (PLS-DA) with sample clustering based on type of encapsulation material: maltodextrin; gum arabic; and a mixture of both materials (50 % each). The data was autoscaled (mean centered with subsequent variance scaling) for the chemometric approaches and the Simplified PLS (SIMPLS) algorithm was applied to decompose the matrix for modeling. Important correlations were obtained using the first 2 Latent Variables (2 LV) at a 95 % confidence level (Alves Filho et al., 2019).

For the chromatographic data, UPLC-HRMS chromatograms (range 5.15–12.3 min) in BPI format were pre-processed using Masslynx version 4.1 as ASCII text files (*.txt) (33 × 3666), imported for Matlab version 2020a, and Icoshift applied for alignment. The matrix was exported to the Unscrambler X™ v.10.4 program (CAMO software, Woodbridge, NJ, USA) for unsupervised principal component analysis (PCA) to provide an overview about the chemical variability. The Singular Value Decomposition (SVD) algorithm was used for PCA after smoothing, baseline correction, normalization (area), and mean centered processing applied over the variables.

## 3. Results and discussion

### 3.1. Characterization of TP and TAA

According to Table 1, the encapsulating agents were able to preserve the phenolic content (TP) in the extracts after freeze-drying, with a significant decrease for all three tests compared to the pure extract (Table 1). However, it was observed that freeze-drying extracts with maltodextrin retained higher TP content and antioxidant activity ($p < 0.05$) when compared to three encapsulants used, followed by MG and GA. These results suggest a greater protection effect of bioactive compounds by maltodextrin.

As we can see, all encapsulants protected relevant phenolic compound content present in CAB extract (Table 1), with a significant reduction for all encapsulated samples. Despite this, there was a reduction of only 26.77 % for the sample with maltodextrin (MD), followed by its mixture (MG – 31.91 %) and gum arabic (GA- 34.31 %). When evaluated among themselves, it was noticed that maltodextrin differed statistically among the encapsulants, managing to preserve a higher amount of phenolic content. The loss of compound content can be related to several factors such as sample moisture, dry material complexity or hygroscopicity. Rezende, Nogueira, and Narain (2018) reported higher results of TP and TAA in acerola by-product powder using freeze-drying compared to spray-drying, probably because the latter technique uses high temperatures, which can accelerate oxidation and cause a greater decrease of these compounds. Maltodextrin has been widely used in the encapsulation of flavoring compounds due to its protection against oxidation (Paim, Costa, Walter, & Tonon, 2016; Rezende, Nogueira, & Narain, 2018).

Antioxidant activity differed statistically ($p < 0.05$) between the CAB extract and the other encapsulations, with an evident reduction, as was the case with the total phenolics. However, the samples with encapsulants showed a significant difference between them when evaluated separately. It was evidenced that maltodextrin showed greater TAA preservation, followed by the MG and GA samples, respectively (Table 1). Extracts with maltodextrin (MD and MG) showed higher values ($p < 0.05$) than those with only gum arabic (GA), implying greater maltodextrin efficiency. According to Queiroz, Lopes, Fialho, and Valente-Mesquita (2011), phenolic compounds are the main components that contribute to the antioxidant activity of foods. This antioxidant effect in edible products and fruits is also not only related to the presence of polyphenols, but also of vitamins such as vitamin C which are also found in cashew apples according to Reina et al. (2022), which may explain the presented results (Table 1). Ballesteros et al. (2017) also found a higher TP content and consequently TAA for samples which contained only maltodextrin as an encapsulating agent.

**Table 1**
Content of phenolic compounds and total antioxidant activity of freeze-dried CAB extracts using different encapsulants.

| Samples | Total phenolic compounds (mg GAE*/100 g DW) | Total antioxidant activityFRAP (μM $Fe_2SO_4$/g DW) |
|---|---|---|
| CAB | $3950.78 \pm 213.2^{A}$ | $1222.01 \pm 34.34^{A}$ |
| MD | $2893.34 \pm 20.18^{aB}$ | $815.85 \pm 112.35^{bB}$ |
| GA | $2595.16 \pm 80.77^{bB}$ | $417.51 \pm 34.10^{aB}$ |
| MG | $2689.96 \pm 103.52^{bB}$ | $698.17 \pm 84.93^{bB}$ |

Results expressed as the mean ± standard deviation. Freeze-dried extracts and CAB extract. The columns that have the same lowercase letter do not present a statistical difference between the samples for ($p < 0.05$). The columns that have the same uppercase letter between the freeze-dried samples and the control sample (CAB extract) for ($p < 0.05$) do not present a statistical difference. CAB: CAB extract; MD: CAB extract + Maltodextrin; GA: CAB extract + gum arabic; MG: CAB extract + Mixture of Maltodextrin and gum arabic (1:1 w/w); DW: Dry Weight of solids present in the original CAB extract.
* GAE – Equal Gallic Acid.

### 3.2. Screening phenolics by UPLC-QToF-MS$^E$

A total of 25 chromatographic peaks were tentatively identified in the CAB extract samples incorporated or not with maltodextrin and gum arabic, as shown in the UPLC-HRMS chromatogram (Fig. 1) (Table 2 - Supplementary Material).

Peak 3 was identified as catechin through its $[M-H]^-$ ions at *m/z* 289 ($C_{15}H_{13}O_6$) and comparison with authentic standards (Alves Filho et al., 2019; Silveira et al., 2021). Peak 4 showed a $[M-H]^-$ ion at *m/z* 293 ($C_{12}H_{21}O_8$), which was compatible with hydroxybutanoic acid ethyl ester, previously identified in cashew apple (Alves Filho et al., 2019).

Furthermore, we were able to characterize several constituents based on the diagnostic fragment ions belonging to flavonoid aglycones: $[M-H]^-$ ions at *m/z* 317 ($C_{15}H_9O_8$) and 316 were attributed to myricetin moiety; *m/z* 301 ($C_{15}H_9O_7$) and 300 were characteristic for quercetin, as well as *m/z* 315 ($C_{16}H_{11}O_8$) and 314 being consistent with isorhamnetin. Additionally, hexose, rhamnose and pentose units were characterized through neutral losses of 162, 146 and 132 Da, respectively. Galloyl derivative produced a sequence of neutral fragments of 152 Da due to the loss of galloyl moiety. In this regard, peaks 5 and 9 showed $[M-H]^-$ ions at *m/z* 787 ($C_{34}H_{27}O_{22}$) and 939 ($C_{41}H_{31}O_{26}$) were proposed to be the gallotannins tetragalloyl and pentagalloylhexoside, respectively (Dos Santos da Rocha et al., 2019).

Peaks 6, 7, 17–18 with $[M-H]^-$ ions at *m/z* 479 ($C_{21}H_{19}O_{13}$), 463 ($C_{21}H_{19}O_{12}$) and 609 ($C_{30}H_{25}O_{14}$) were identified as myricetin-O-hexoside, myricetin-O-rhamnoside and myricetin derivatives, respectively (Cunha, Brito, Moura, Ribeiro, & Miranda, 2017; Silveira et al., 2021). Peak 15 exhibited a $[M-H]^-$ion at *m/z* 317 that was identified as myricetin after comparison with an authentic standard. Peaks 8, 10–12, 14, 19–20 showed $[M-H]^-$ ions at *m/z* 463 ($C_{21}H_{19}O_{12}$), 433 ($C_{20}H_{17}O_{11}$), 447 ($C_{21}H_{19}O_{11}$), 593 ($C_{30}H_{25}O_{13}$), suggesting to be quercetin-*O*-hexoside, quercetin-*O*-pentoside, quercetin-*O*-rhamnoside and quercetin derivatives (Dos Santos da Rocha et al., 2019; Silveira et al., 2021). Peak 16 presented a $[M-H]^-$ ion at *m/z* 301, which was identified as quercetin by comparison with an authentic standard (Braga et al., 2021).

Lastly, peaks 21 and 23 gave $[M-H]^-$ ions at *m/z* 327 ($C_{18}H_{31}O_5$) and 329 ($C_{18}H_{33}O_5$) and were annotated as trihydroxyoctadecadienoic acid and trihydroxyoctadecaenoic acid, respectively, due to their fragmentation patterns being similar to literature data (Galvão et al., 2018).

When comparing the chromatograms, the CAB extracts dried with the different encapsulant agents were able to preserve most of the peaks corresponding to the compounds present in the initial extract (before being freeze dried).

### 3.3. Powder characterization

#### 3.3.1. Morphology

The images obtained by Scanning Electron Microscope (SEM) for the powder extracts (MD, GA, and MG) are presented in Fig. 2A (300× approximation) and B (150× approximation), in which it can be observed that the freeze-drying process resulted in a powder with irregular sizes in the form of a plate, resembling broken glass and very porous, indicating a change in the original structure of the encapsulants used, which are round and smooth (Ballesteros et al., 2017). Ballesteros et al. (2017) obtained similar images for their freeze-dried samples when encapsulating a polyphenol extract from coffee grounds dried by freeze-drying and spray-drying. Also, Rezende, Nogueira, and Narain (2018) reported images from freeze-dried pulp and acerola residue extracts (Malpighia emarginata DC).

Previous studies have shown that freeze-drying cause changes in particle morphology, as it removes water without the use of high temperature creating structures with different sizes, which is typical for this type of drying (Mahdavee Khazaei, Jafari, Ghorbani, & Hemmati Kakhki, 2014; Saikia, Mahnot, & Mahanta, 2015).

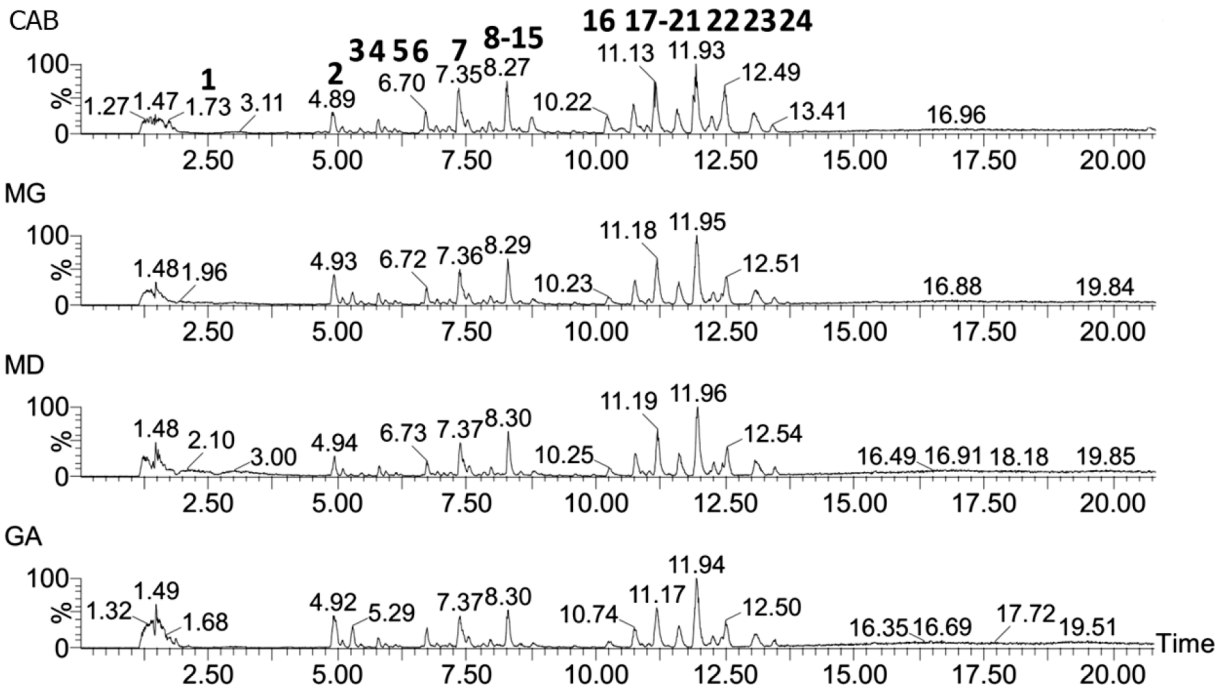



**Fig. 1.** UPLC-HRMS chromatograms in negative ionization mode of extract and powders derived from cashew apple phenolic extract. CAB: extract without encapsulants; MD: CAB extract + Maltodextrin; GA: CAB extract + gum arabic; MG: CAB extract + Mixture of Maltodextrin and gum arabic (1:1 w/w).

### 3.3.2. Structural characteristics

Fig. 3A shows XRD standards for maltodextrin and gum arabic, and B for dry samples (MD, GA, and MG). The results observed in the XRD analysis of the freeze-dried CAB extracts revealed a low crystallinity degree of the samples, regardless of the wall material used (Fig. 3.B - Supplementary Material). A large widening of the peaks is perceived in the diffractograms, with an amplitude around $2\theta = 15°$, presenting an amorphous background up to about $2\theta = 40°$ (Fig. 3.B - Supplementary Material), indicating that the obtained materials are predominantly amorphous. The amorphous state could be due the fact that the material did not crystallize during the drying process because maltodextrin has high molecular weight and high viscosity which increased the glass transition temperature. The amorphous surface is not in thermodynamic equilibrium, and provides the material with sensitive temperature, humidity, and pressure characteristics (Gurak, Cabral & Rocha-Leão, 2013).

Ballesteros et al. (2017) observed that small crystalline regions give rise to wide peaks, while larger crystalline regions appear in finer and better-defined peaks. No crystalline regions are observed in either the carrier agents or in the freeze-dried CAB extracts (Fig. 3. - Supplementary Material). However, the behavior presented by the freeze-dried extracts did not differ from those presented by the isolated encapsulants (Fig. 3.A - Supplementary Material), indicating that there are no crystalline regions in the samples, which is related to the encapsulant used for protecting phenolic compounds. In addition, da Fonseca Machado et al. (2018) reported that the drying method did not influence the structure of anthocyanin-rich blackberry extracts. This indicates that the drying technique used in this study does not affect the solid state of the polymer, but rather that the carrier agent provides structural differences in powders.

### 3.3.3. Physical characterization of freeze-dried CAB extracts

The results of the physical analyses carried out on the freeze-dried samples are shown in Fig. 4. The moisture content of the extract decreased from 95 % (extract before drying) to about 6 % (extract after drying). It is known that low humidity is a fundamental property for powders, and even more so for amorphous powders, in which low humidity is an ally for maintaining the stability of these powders. Also, the moisture content is related to drying efficiency, powder fluidity and crystallization (Akhavan Mahdavi, Jafari, Assadpoor, & Dehnad, 2016). These authors also pointed out that the composition of wall material affects the moisture of the powders.

The MD and GA samples did not differ from each other, presenting the lowest values for water content and water activity (Fig. 4A and B), which are consistent with the results found by Suravanichnirachorn et al. (2018). The MG sample showed statistically higher values for these analyses.

Freeze-drying together with the encapsulants used produced powders with high solubility (74–78 %). These values (Fig. 4C) are similar (61–64 %) to those found by Saikia, Mahnot, and Mahanta (2015) for microencapsulated powders by freeze-drying using maltodextrin as an encapsulant (60–64 %) which were considered excellent values for solubility (Saikia, Mahnot, & Mahanta, 2015); this is associated with its ability to rehydrate if used as a food ingredient (Syamaladevi, Insan, Dhawan, Andrews, & Sablani, 2012). However, these values were lower than those found by Caparino et al. (2012) (about 89 %), who attributed this value to the increase in maltodextrin in the studied sample.

Hygroscopicity (Fig. 4D) resulted in powders with values below 3 %. Yamashita et al. (2017) observed that freeze-drying produces larger particles and with a smaller surface area than spray-dried powders, resulting in a lower water absorption. Therefore, lower hygroscopicity makes the compounds less susceptible to oxidation by moisture (Paim, Costa, Walter, & Tonon, 2016).

## 3.4. In vitro simulated digestion

TP and TAA of the extract powders with and without encapsulants were found to be significantly lower after the *in vitro* simulated digestion ($p < 0.05$) (Table 3 - Supplementary Material). A study suggested that the reduction of phenolics after gastric digestion is related to their sensitivity to slightly alkaline conditions (small intestine), which can promote a structural transformation of these compounds and influence their chemical properties, as well as their biological activity (Bermúdez-Soto, Tomás-Barberán, & García-Conesa, 2007). Thus, a reduction of

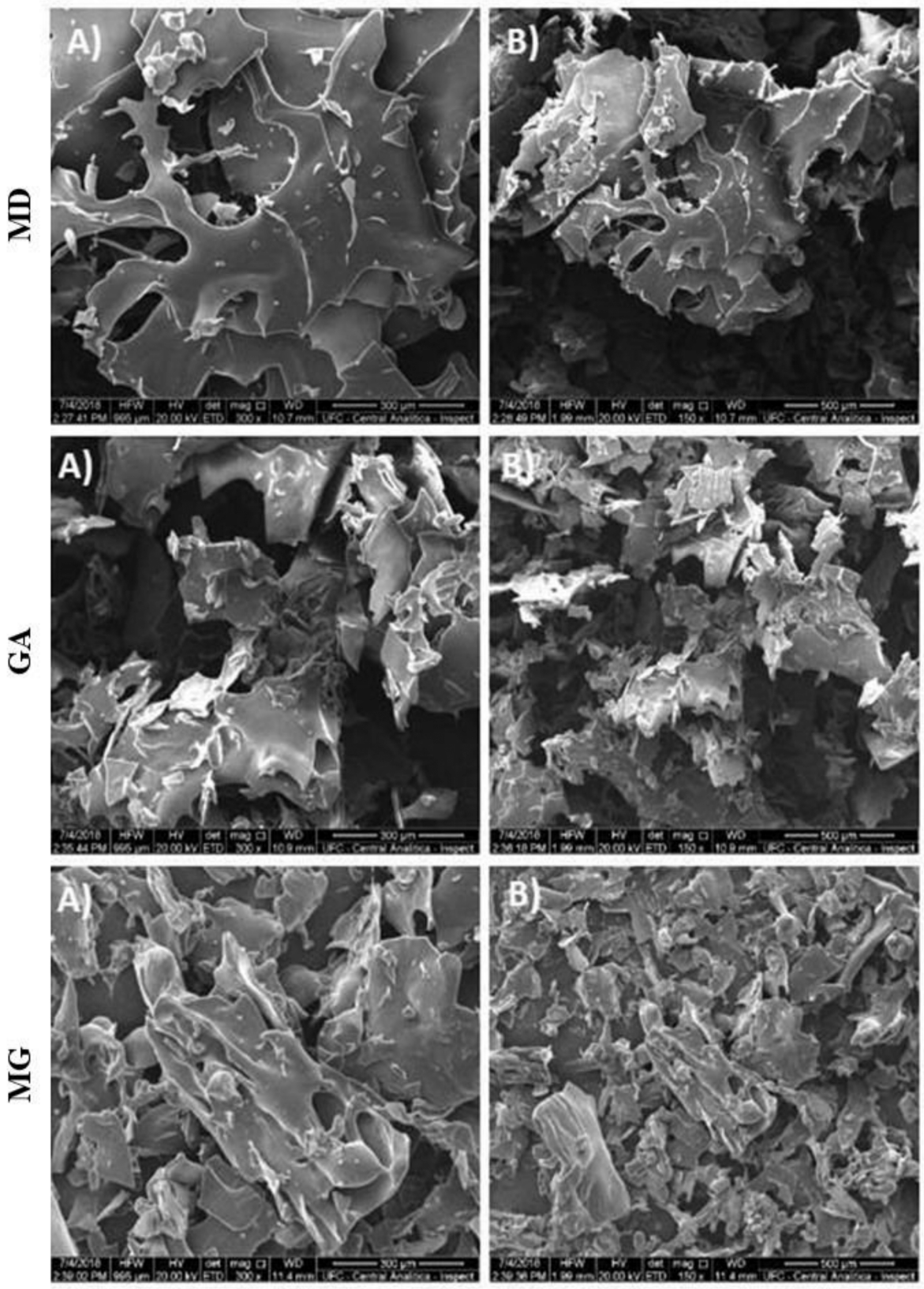



**Fig. 2.** Scanning electron microscopy for freeze-dried CAB extracts using different encapsulants with magnification of 300x (A) and 150x (B). MD: CAB extract + maltodextrin; GA: CAB extract + gum arabic; MG: CAB extract + maltodextrin and gum arabic (1:1 w/w).

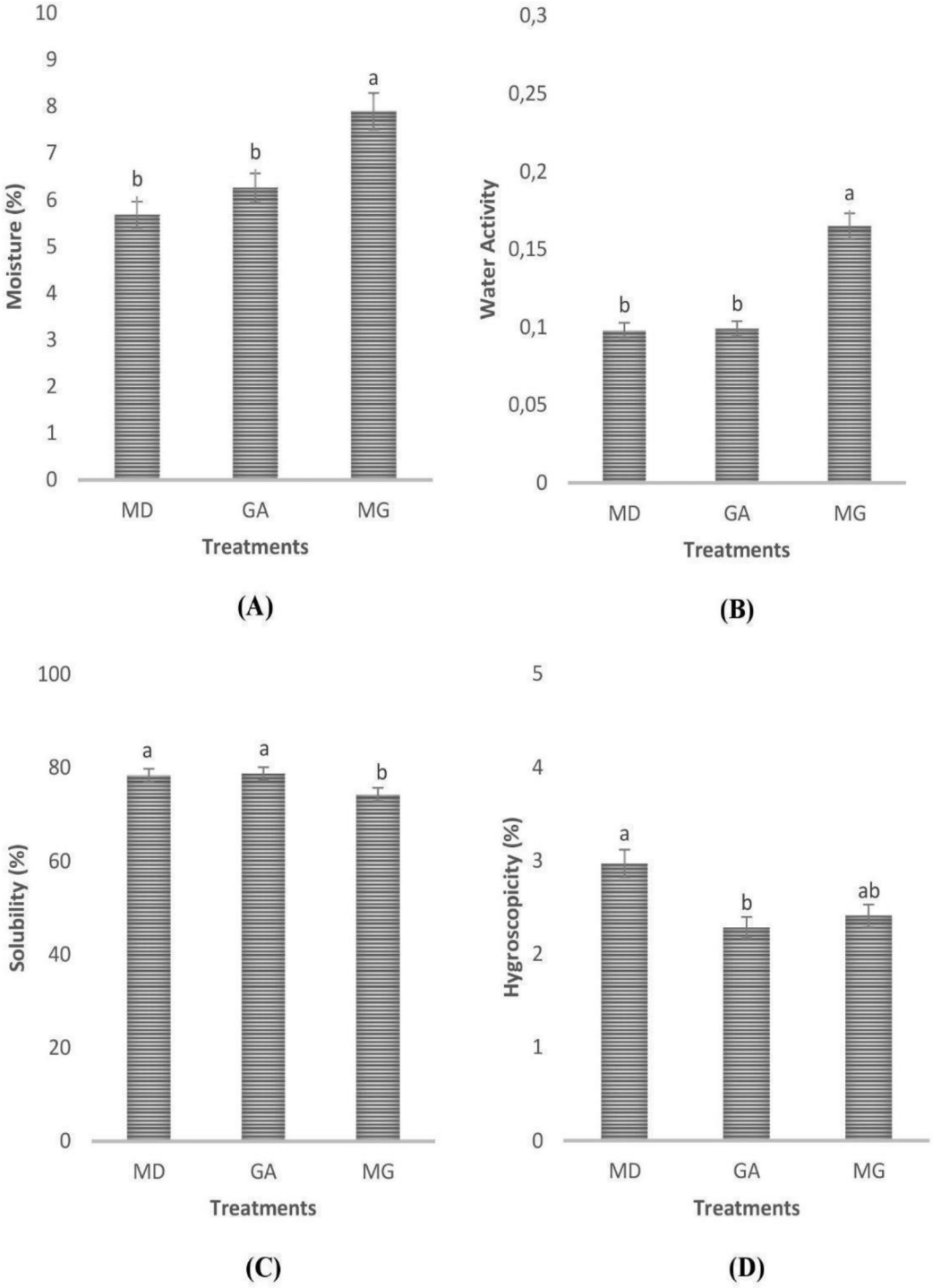



**Fig. 4.** Physical properties of the encapsulated powder with different encapsulants. MD: maltodextrin; GA: gum arabic; MG: maltodextrin + gum arabic (1:1 w/w). Results expressed as mean ± standard deviation. The results followed by the same letter in the columns do not differ statistically with ($p < 0.05$).

certain phenolic compounds after the enteric phase is common (Lucas-González, Viuda-Martos, Pérez Álvarez, & Fernández-López, 2018). Fonteles et al. (2016) reported a large decrease in cashew apple bagasse vitamin C after the enteric phase of bioaccessibility, with a reduction of up to 58 % for the samples studied using ultrasound.

In this study, CAB extract without encapsulant did not show any antioxidant activity after digestion, demonstrating the effectiveness of encapsulants in protecting the bioactive compounds present in the extract. Shah, Zhang, Li, and Li (2016) found similar results when evaluating the bioaccessibility and antioxidant activity of curcumin after encapsulation. Also, Gawlik-Dziki et al. (2012) evaluated the bioaccessibility of antioxidant compounds in broccoli extract, finding that TAA was reduced despite the good bioaccessibility of broccoli phenolics, in turn reporting that there may be a food synergy during the digestion of the studied TPs, and that it is necessary to address the correlation between the release of biologically active compounds under physiological conditions, their transepithelial passage and their chemical modifications.

### 3.5. Chemometric analysis

Physical and chemical parameters such as total phenolics (TP), bioaccessibility of total phenolics (TP*), total antioxidant activity (TAA), bioaccessibility of total antioxidant activity (TAA*), moisture, solubility, hygroscopicity and water activity (WA) were measured in cashew apple by-product extracts before and after freeze-drying with 3 different wall materials: maltodextrin; gum arabic; and a mixture of both materials (1:1 w/w). Furthermore, the compounds identified in the CAB extract before and after freeze-drying and after their bioaccessibility, (Fig. 1) (Table 2 – Supplementary material) enabled detailing the protective capacity of these encapsulant agents on the polyphenols of the studied extract. Therefore, a supervised chemometric model by PLS-DA was developed to classify and correlate cashew apple by-product extracts according to their physical and chemical parameters, as well as chromatographic analysis.

Fig. 5a presents the scores plotted in two dimensions (LV1 × LV2), and Fig. 5b illustrates the respective loadings, representing 78.61 % of the total variance. The absence of outliers negatively influencing the classification modeling was detected in the Hotelling $T^2$ × Q residual plot (Fig. 5c), where the extracts with higher model error (Q residual values near to 1) presented no strength for modeling by their low Hotelling $T^2$ values. Fig. 5d shows the VIP (Variable importance in Projection) plot, in which the most important variables (physical and chemical parameters) for separating the freeze-dried extract are presented according to LV1 scores (values higher than 1).

A clear separation of the freeze-dried extracts was detected by modeling the classification based on the score plot (Fig. 5a), in which the GA sample scored negative for LV1 and positive for LV2, the MD sample scored null for LV1 and negative for LV2, and the MG sample scored positive for LV1 and LV2, observing a clear separation of the three samples correlated with the variables involved, demonstrating that the encapsulating agents influenced the studied parameters.

Thus, according to the correlation of Fig. 5a and 5b among the studied powders, MD resulted in a powder with higher total antioxidant activity (TAA) and total polyphenol (TP) values, as well as their respective bioaccessibility (TAA* and TP*) and also higher hygroscopicity. However, GA and MG mainly resulted in higher water activity (WA) and moisture values, constituting characteristics which can accelerate the oxidation of bioactive compounds, and which may have contributed to their lower concentration in the MG and GA samples. However, it is noted that all of the CAB extract powders maintained a high TP and TAA concentration (Table 1).

The classification performance of CAB extracts based on physical and chemical parameters and encapsulation material was evaluated by the statistical parameters described in Table 4 (Supplementary Material). The ability of the model for extract discrimination was measured under the "most likely rule" by sensitivity, specificity, and precision (Ballabio & Consonni, 2013; Jensen, Refsgaard, Bro, & Brockhoff, 2005). Considering that the sensitivity, specificity, and precision values are equal to +1, which represents perfect separation between the sample clusters, and that −1 indicates complete disagreement between the separation, the model exhibits high quality, which can be corroborated by bias values close to zero. Moreover, given that very low calibration and cross-validation errors (RMSEC and RMSECV) represent better modeling, and their closeness (similarity index by RMSEC/RMSECV) indicates a well-fitting model, this method showed high quality for distinguishing freeze-dried extracts (Freitas et al., 2018).

Regarding the difference in the chemical profile of the encapsulated extracts, unsupervised principal component analysis was applied in the

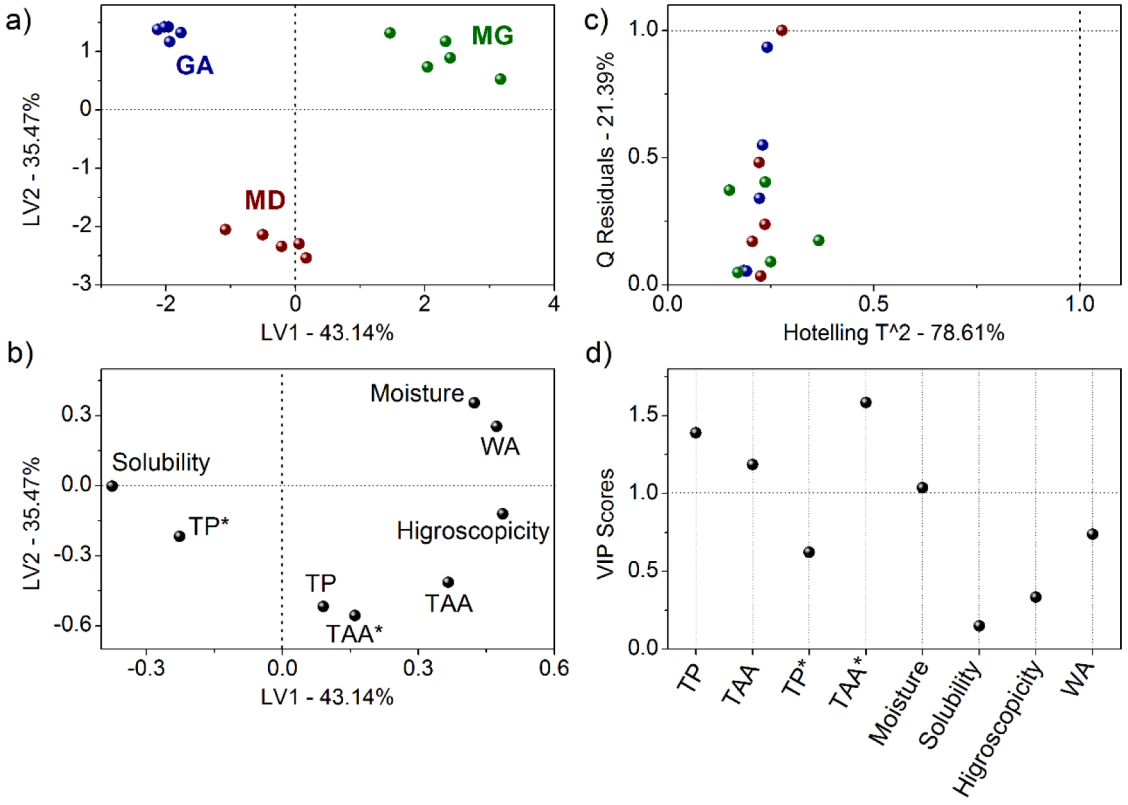



**Fig. 5.** Classification results by PLS-DA modeling from the cashew apple by-product extract after encapsulation using different materials: gum arabic (GA in blue color), maltodextrin (MD in red), and a mixture of both materials using 50 % each (MG in green). The LV1 × LV2 scores coordinate system (a); LV1 × LV2 loadings from the physical and chemical parameters (b); influence plot by Hotelling $T^2$ × Q residuals (c); VIP plot with the most important variables for samples separation represented by scores higher than 1. TP: total phenolics; TP*: total phenolics after bioaccessibility; TAA: total antioxidant activity; TAA*: total antioxidant activity bioaccessibility; WA: water activity.

chromatogram data. A scores plot (Fig. 6a) explained 55 % of the variance in PC1 and 32 % in PC2, corresponding to 87 % of overall variance. PC1 scores presented separation in two clusters: GA, MG and MD were located on the left side (negative), while Ex was on the right side (positive). In PC2, GA and Ex were on the lower side (negative), whereas MD was found in the upper section, indicating a difference among the encapsulated samples.

The PC loadings (Fig. 6b and c) showed the variables before and after the encapsulation. The first PC loading presented a strong association of catechin and a quercetin derivative (negative) with GA, MG and MD encapsulated samples. Ex showed correlations with myricetin samples. The second PC loading (Fig. 6c) showed a difference in phenolic composition among encapsulated samples with robust association of catechin to GA (negative). Furthermore, MD was correlated to the quercetin and myricetin derivatives (positive).

As observed in the chromatogram (Fig. 1), the encapsulant agent was able to preserve some of the compounds present in the extract, showing a similar chemical profile to the initial extract Ex. Furthermore, the addition of gum arabic maximized protection of catechin and maltodextrin for the quercetin and myricetin derivatives. Ballesteros et al. (2017) reported that maltodextrin retained more phenolic compounds and antioxidant activity than gum arabic in CAB extract. Moreover, it was more effective in preserving compounds, showing expressive antioxidant activity of *Eugenia stipitata* dry pulp microparticles during *in vitro* bioaccessibility (Iturri, Calado, & Prentice, 2021). These results are in agreement with our study, in which the addition of maltodextrin resulted in the highest antioxidant activity among samples dried with encapsulant agents, despite the decrease compared to the initial CAB extract (Table 1).

## 4. Conclusion

The polyphenol extract of cashew apple by-product presented high TP and TAA content, in addition to important bioactive compounds such as catechin, myricetin and quercetin, indicating functionality in an underutilized native Brazilian fruit by-product. The TP and TAA contents and their compounds were protected by the different encapsulant agents used for freeze-drying, resulting in powders of industrial importance, mainly in the sample in which maltodextrin was used.

All powders presented a brittle appearance and were predominantly amorphous, which is common for this type of drying, and may suffer variations when exposed to changes in temperature and humidity, thus requiring the use of packaging for proper conservation. They also showed high solubility, low humidity and low hygroscopicity, constituting suitable parameters for powder products. The correlations between samples regarding chemical and physical results showed reliable separation of the powders in chemometric tests, demonstrating that the use of encapsulating agent results in powders with higher TP and TAA retention and with better physical characteristics, such as MD, followed by MG and GA. Finally, the tests showed low calibration and cross-validation errors, thus indicating a well-fitting model with high sample separation quality.

Finally, numerous applications can be thought of for the encapsulated CAB extracts, mainly to enrich foods such as beverages which are poor in antioxidant activity to improve their quality. However, due to the lack of results regarding the thermal behavior of the lyophilized extracts, we suggest applying the samples studied in this research to foods which do not use heat treatment. Thus, we emphasize the need to carry out studies involving the behavior of extracts when subjected to thermal processing, as well as to better understand bioaccessibility in a food matrix.

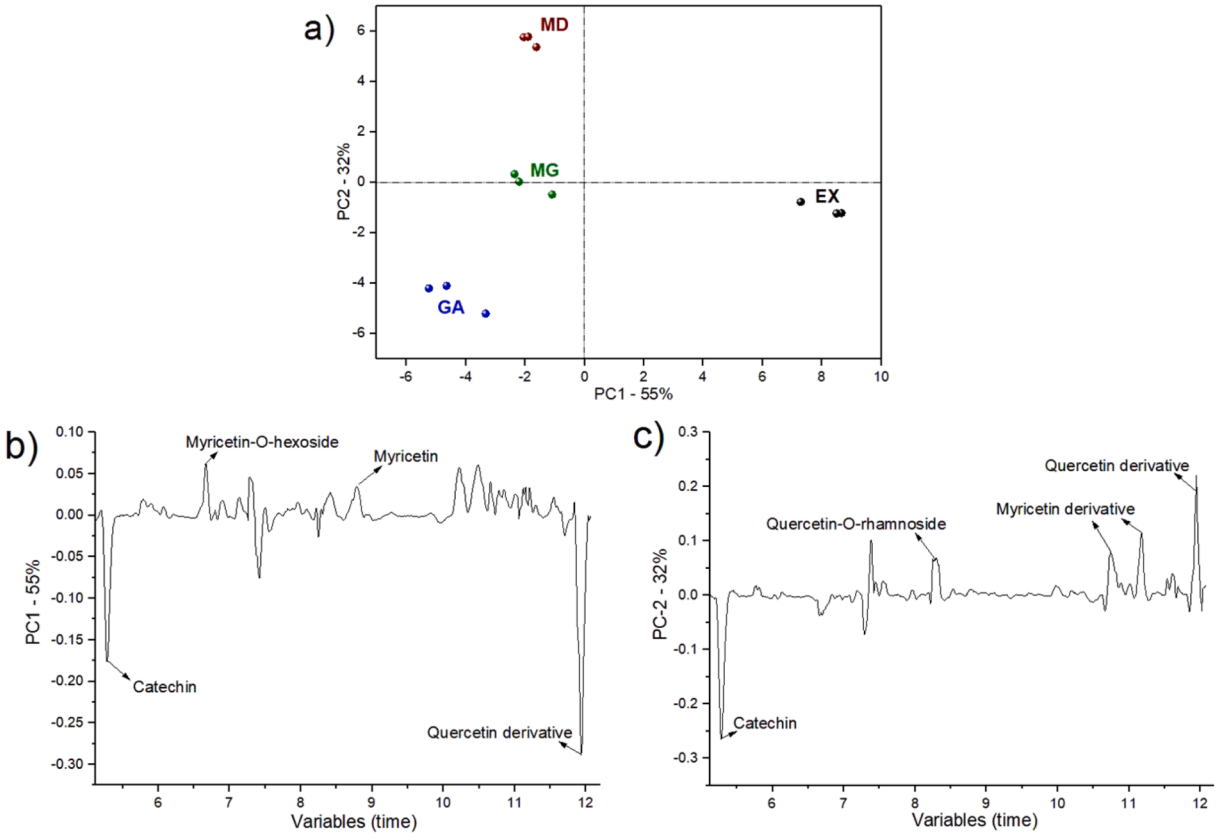
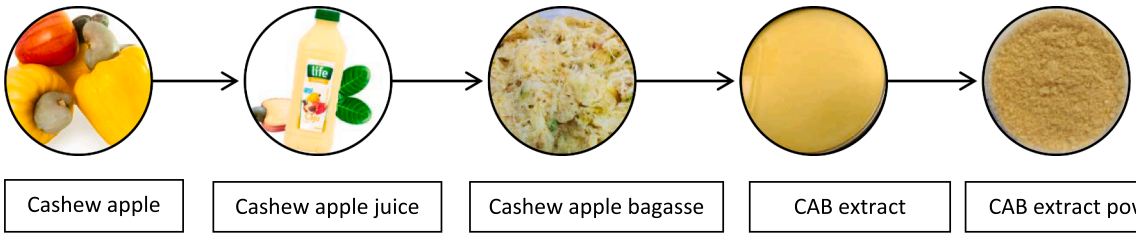



**Fig. 6.** A scatter plot (PC1 × PC2) and scores plot of CAB extract and their encapsulation. EX: CAB extract; MD: Extract CAB + Maltodextrin; GA: CAB extract + gum arabic; MG: CAB extract + Mixture of Maltodextrin and gum arabic (1: 1 w/w). Loadings in line from scores (b and c) for PC1 and PC2, respectively.




**Flowchart 1.** Process for obtaining the encapsulated CAB extract. CAB - Cashew Apple Bagasse.

## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## Data availability

Data will be made available on request.

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## Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fochms.2022.100149.

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