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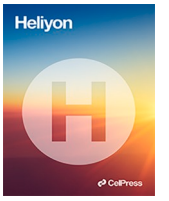

Research article

Stability of yerba mate extract, evaluation of its microencapsulation by ionic gelation and fluidized bed drying

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ABSTRACT

Studies show that yerba mate (*Ilex paraguariensis*) has high antioxidant capacity occasioned by its high contents of total phenolic compounds. Microencapsulation, specifically ionic gelation, since it does not use heating during process, is considered as an alternative for preserving and applying the extract. The purpose of this study was to evaluate general characteristics and stability of hydroalcoholic extract of yerba mate, conduct the extract microencapsulation by ionic gelation followed by microparticle fluidized bed drying. The extract was evaluated for color stability, total phenolic compounds, and antioxidant activity for nine weeks and at three temperatures (5, 15, and 25 °C). From the extract, a double emulsion (W/O/W), generation of microparticles (ionic gelation by dripping), and fluidized bed drying were conducted. The extract had 32912.55 mg GAE/100 g of phenolic compounds and 2379.49 μmol TE/g of antioxidant activity. The main compound observed was chlorogenic acid (5-CQA) with 0.35 ± 0.01 g/100 mL. In the stability study, the temperature was observed to influence in phenolic compounds reduction, as well as in total color difference of the extract. Double emulsion has shown to be stable and appropriate for use. The values of microparticles total phenolic compounds and antioxidant activity were 423.18 ± 8.60 mg GAE/100 g and 21.17 ± 0.24 μmol TE/g, respectively. After drying, the moisture of microparticles was reduced from 79.2% to 19%. The extract had high total phenolic compound content and high antioxidant activity. Storage at the lowest temperature (5 °C) assured better preservation of extract total phenolic compounds. The dried microparticles showed content of total phenolic compounds and antioxidant activity with potential for commercialization and future application in food matrices.

1. Introduction

The consumption of yerba mate (*Ilex paraguariensis*) traditionally occurs as a beverage, such as *chimarrão* (a yerba mate hot drink), *tereré* (a yerba mate cold drink), or mate tea. Due to its characteristics and versatile nature, yerba mate extract has currently been used by food and pharmaceutical industries, being found in food and dietary supplements. When added to food, the extract may contribute

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to an increase in quality in different aspects, such as sensorial and extension of the shelf-life [1].

Among the compounds standing out in yerba mate are xanthines and saponins, the first ones being associated to stimulant properties as those observed in caffeine and theobromine. However, phenolic compounds are the most prominent ones; the high antioxidant capacity found in yerba mate is attributed to these compounds. Another property verified in yerba mate is its antimicrobial potential, which enables it to be used in some products as preservative [2,3].

Many studies involving polyphenol encapsulation have been showing how beneficial this technique can be to preserve the less stable compounds when they are free. In these studies, the improvement in phenolic compounds bioavailability and stability is evident [4,5]. According to the research conducted by Akbarmehr et al. [3], microencapsulation of yerba mate bioactive compounds by spray-dryer can be considered a practical and economical solution to preserve functional food compounds. As reported by the authors, microencapsulation can mask undesirable flavors and bitter taste of encapsulated material.

A technique used to microencapsulate a compound of interest is ionic gelation, which is based on the formation of a dispersion or emulsion containing the bioactive compound, with its subsequent injection into the crosslinker solution leading the biopolymer to a gelling process [6]. The advantages in the formation, processing and versatility of the operation mode are avoiding the use of extreme temperatures that can affect the stability of the core or the wall material. In addition, pre-treatments are not required: the ionic gelation method is considered a competitive alternative to encapsulate and protect SO, compared to encapsulation techniques such as spray drying, freeze drying, fluidized bed drying, and coacervation [7]. Among the core materials often used in ionic gelation is sodium alginate which, with the addition of calcium salts, will be converted into sodium alginate, a water-insoluble compound. Another possibility is the use of low methoxyl pectin, more suitable for vegan products, as sodium alginate is most often used in dairy products (non-vegan foods). The use of low-methoxy pectin promotes microparticles with soft structure and has application in products of a wide pH range [8]. Since the high moisture content of the particles obtained by ionic gelation can cause limited stability and lead to an incompatibility with the food matrix, the strategy of subsequent drying of these particles can be a viable alternative for greater stability and better possibility of commercialization of these particles [9]. The use of a commercial hydroalcoholic extract was chosen in order to facilitate a future industrial application of the microparticles, since the extract would be easily available and commercially standardized.

The purpose of this study was to evaluate general characteristics and stability of hydroalcoholic extract of yerba mate, specifically the consequences of storage in color, phenolic compounds, and antioxidant activity, as well as to conduct the microencapsulation by ionic gelation and promoting microparticle fluidized bed drying with the aim of improving the stability of active compounds present in yerba mate and boosting their application in industrialized foods.

2. Material and methods

2.1. Material

Materials used: food grade yerba mate extract 64° GL, supplied by the *Heide Extratos Vegetais Company (*Pinhais/Brazil*)* - the extract has the code PRD00336. The composition is 50% yerba mate leaves; the extraction used water and 70% alcohol without heating - preservatives were not used due to the high alcohol content; analytical grade reagents – PA; canola oil of Liza® brand (*Cargill Agrícola S.A.*, Mairinque/Brazil); polyglycerol polyricinoleate – PGPR (*Concepta Ingredients*, São Paulo/Brazil); GENU® amidated low methoxyl pectin LM-104 AS-Z (*CP Kelco*, Limeira/SP); food grade calcium chloride (*Dinâmica*, Diadema/SP).

2.2. Characterization of hydroalcoholic extract of yerba mate (Ilex paraguariensis)

2.2.1. pH

IAL methodology [27] was used with a potentiometer (Digimed, model DM 20, Brazil).

2.2.2. Soluble solid content

It was measured in °Brix using a refractometer (Abbe Refractometer, model 10450, USA), according to IAL methodology [10].

2.2.3. Total titratable acidity

10 g of yerba mate extract were weighted, and 90 mL of distilled water were added. The sample was titrated with sodium hydroxide 0.1 N to pH 8.10. The methodology used was adapted from AOAC [3] and IAL [10].

2.2.4. Moisture

This step was conducted as per methodology adapted from AOAC [11] and IAL [10], by the vacuum drying oven principle and then removing the water present in food by evaporation. The sample has its mass recorded before being put in the oven and after drying and stabilization in desiccator.

2.2.5. Colorimetric evaluation: measurement of instrumental color and color change with pH variation

A portable colorimeter CR-400 (Konica Minolta Sensing Inc., Osaka, Japan) was used for evaluation by CieLab system, L*, a*, and b* parameters (Konica Minolta, 2007). Chroma (C*), hue angle (h) and total color change (ΔE) values were calculated according to the equations [1–3]:

$$C^* = \left(a^{*2} + b^{*2}\right)^{1/2} \quad (1)$$

$$h = arctan\left(\frac{b^*}{a^*}\right) \quad (2)$$

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

Color measurement was conducted in extract (1:5) diluted in alcohol 70%. Since it is considered a semi-transparent solution, the sample reading occurred in test tubes partially covered with white opaque paper, so a white background would be encountered by the incoming light beam. This was performed according to the pictures found in Konica Minolta (2007), as the result could be compromised by the color of test tube rack using during measurement.

A color change test of the extract with pH variation at a range of 2–8 was also conducted.

For pH change, standard solutions of NaOH 1 M and HCl 1 M were used. In order to determine the pH values, a Digimed potentiometer (model DM 20, Brazil) was used. At first, the initial pH of extract was measured and then, by using a burette, a solution of HCl 1 M was added until the extract reached a pH value of 2, which one aliquot was separated. Subsequently, NaOH 1 M was added until pH values of 3, 4, 5, 6, 7 and 8 were reached, always separating one aliquot from each. In the end, pictures of the samples were taken, and the color change was analyzed according to the pH value.

2.2.6. Total phenolic compounds

Phenolic compounds of the extract were determined by the Folin Ciocalteau spectrophotometric method, according to Erkan-Koç et al. [12].

The analyses were performed with extract diluted in alcohol 70%. The methodology required the construction of a 5-point standard curve of gallic acid (40, 80, 120, 160, and 200 μg/mL). A blank was also prepared, which had all used reagents except for the sample, which was replaced by distilled water. Samples and standard curve readings were made in a UV/Visible spectrophotometer (Agilent Technologies, Cary 60 MY13110012, United States of America) at a wavelength of 750 nm. The results were shown in mgEAG/100 g.

2.2.7. Antioxidant activity by DPPH and ABTS methods

Both analyses were performed according to Jiménez-Zamora et al. [13].

DPPH (2,2-diphenyl-1-picrylhydrazyl) is a free radical molecule which in solution has dark violet color. When combined with other compound having a hydrogen atom that can be donated, it is reduced and its color changes from violet to light yellow [14].

In order to conduct this assay, yerba mate extract diluted in alcohol 70% was used. A 6-point Trolox standard curve (50, 100, 200, 400, 600, and 800 μM) was made. Both samples and standard curve were read at a wavelength of 515 nm in a UV/Visible spectrophotometer (Agilent Technologies, Cary 60 MY13110012, United States of America). The results were shown in μmol TE/g (Trolox Equivalent).

In the other analysis, a solution containing the reagent 2,2- azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) and a sodium persulfate solution 2.6 mM was mixed in equal parts and incubated in dark for 16 h. A 5-point Trolox standard curve (100, 500, 1000, 1500, and 2000 μM) was made. The samples of yerba mate extract diluted in alcohol 70% and standard curve were read after 6 min of incubation in the absence of light at a wavelength of 734 nm. For absorbance reading, an UV/Visible spectrophotometer (Agilent Technologies, Cary 60 MY13110012, United States of America) was used. The results were shown in μmol TE/g (Trolox Equivalent).

2.2.8. Identification and quantification of individual components by HPLC

The identification and quantification of chlorogenic acid, theobromine and caffeine present in concentrated extract was performed by high efficiency liquid chromatography equipment (Shimadzu CBM20A) with UV-DAD detector (SPD-M20A), quaternary pump LC-20AT and column Zorbax Eclipse plus C18, with dimensions of 4.6 × 250 mm and particles 5 μm. The methodology was based on Tfouni et al. [15]. It should be emphasized that, for chlorogenic acid (CGA), only 5-CQA without isomers was identified.

For alkaloids theobromine and caffeine, standards Sigma-Aldrich (Saint Louis, MO, EUA) C0750 Caffeine – powder, ReagentPlus® and T4500 Theobromine ≥98.0%, respectively, were used. Sample preparation was made by homogenizing 5 mL of yerba mate extract, 1.0 g of magnesium oxide, and 50 mL of water Mili-Q. This solution was kept on boiling for 15 min and let it cool to be filtered through filter paper, completing 100 mL volume with Mili-Q water. A filter membrane syringe (0.45 μm and 15 mm diameter) was used to transfer the samples to the 1.5 mL vials. The mobile phase consisted of methanol:water solution (30:70).

Chlorogenic acid identification was made through standard Sigma-Aldrich (Saint Louis, MO, USA) C3878 chlorogenic acid ≥95% (titration). An amount of 5 mL of extract, 2 mL of Carrez I solution and 2 mL of Carrez II solution was used, completing the volume with 100 mL of Mili-Q water. The transfer filtering to the vials occurred as mentioned before, with mobile phase consisting of A: water (pH 2.7) + 1 g/L of NaH_2PO_4 and B: acetonitrile.

2.3. Color stability, total phenolic compounds, and antioxidant activity of yerba mate extract

The stability study of extract included the comparison between three storage temperatures (5, 15, and 25 °C) during nine periods. Weekly analyses of instrumental color, total phenolic compounds, and antioxidant activity (by DPPH) were conducted.

2.4. Production of simple emulsion (W/O)

W/O emulsions were prepared by adding canola oil of Liza® brand (Cargill Agrícola S.A., Mairinque/Brazil) and polyglycerol polyricinoleate – PGPR as a surfactant (Concepta Ingredients, São Paulo/Brazil).

The following ratios were used: 5%: 35 g of yerba mate extract +60 g of canola oil +5 g of PGPR.

At first, PGPR surfactant was solubilized in oil under magnetic stirring at 42 ± 2 °C for 5 min. The mixture was cooled until reaching the room temperature (25 °C). The oil/PGPR dispersion was put in jacketed reactors and connected to an ultra-thermostatic water bath to keep emulsion temperature controlled at 25 ± 2 °C. The aqueous phase (extract) was added to the mixture by dripping, using a burette coupled with a silicone hose. Homogenization was made with Turratec (Tecnal model TE102, Piracicaba, Brazil) at 14.000 rpm.

2.5. Characterization of simple emulsion (W/O)

2.5.1. Sedimentation index

Immediately after preparation of emulsion, it was transferred to a 100 mL graduated cylinder (diameter: 27 mm, height: 173 mm), which was sealed and kept on the bench at room temperature for 24 h. Stability evaluation was made by observing whether the separation of emulsion phase occurred or not 24 h after emulsification process. Stability was measured by average height of upper phase, with sedimentation index (SI) being described by equation [4].

$$SI(\%) = \frac{v_S}{v_T} x\ 100 \tag{4}$$

where: V_S = volume of sediment phase e V_T = total volume of the sample.

2.5.2. Mean diameter and size distribution (polydispersity index)

Determined by light scattering in Horiba LV950 equipment, using paste module (quartz plates) appropriate to pasty samples. Canola oil was used as sample dispersant through direct dispersion at a 1:6 dilution ratio ($g_{emulsion}$:$g_{canola\ oil}$). Measurements were made in duplicate at 25 °C after 24 h. The mean diameter was based on mean diameter of a sphere of the same area (surface mean diameter – D [2,3] or Sauter mean diameter) shown in equation [5]. The samples were analyzed in quintuplicate by wet route, according to the method adapted from Carvalho [16].

$$D_{[3,2]} = \frac{\sum n_i d_i^3}{\sum n_i d_i^2} \tag{5}$$

where: d_i stands for drop diameter and n_i stands for the number of drops.

The polydispersity index (PDI) was calculated according to Moura et al. [17], by using equation [6]:

$$\mathrm{PDI} = \frac{d_{90} - d_{10}}{d_{50}} \tag{6}$$

where: d_{10}, d_{50} and d_{90} are diameters at 10%, 50% and 90% of accumulated volume, respectively. That is, d_{90}-d_{10} is data range and d_{50} is the mean diameter.

2.6. Production of double emulsion (W/O/W) and microencapsulation

Microparticles were obtained from ionic gelation technique. Unlike the extract, which is considered to be a quite hydrophilic compound, the ionic gelation is applicable to hydrophobic or poorly soluble active ingredients only, thus a double emulsion W/O/W was required to be made.

After preparing a simple emulsion (W/O) and conducting all applicable tests, a double emulsion (W/O/W) was prepared by adding a pectin solution. The ratios were defined according to the literature [17–19].

A pectin solution 2% was prepared and added to the simple emulsion at the following ratio: 20% of simple emulsion to 80% of pectin solution 2%. These were homogenized by using a rotor stator disperser Turratec (Tecnal model TE102, Piracicaba, Brazil) at 14,000 rpm for 5 min, finally generating the double emulsion (W/O/W).

Prior to microencapsulation, a calcium chloride 3% solution acidified with citric acid 10% until pH around 3 was prepared. When in contact with double emulsion droplets, this solution causes the trapping, changing the droplets to a spherical shape.

The particles were produced through dripping by using an Encapsulator model B-390 (Büchi, Flawill, Switzerland). Process variables followed the conditions defined at Moura et al., 2018a, with minor changes such as vibration frequency 1100 Hz, electrode voltage 2000 V, and pressure between 300 and 400 mbar.

2.7. Characterization and drying of microparticles

2.7.1. Microstructure

The evaluation of particle microstructure was conducted by methodology adapted from Moura et al. [8], by using an optical

microscope (model BX41, Olympus brand, Japan) and an external light source for viewing surface details. A stereoscope with external light and magnification of 40× (MZS-250, Dimex) was also used to study the microstructure more completely.

2.7.2. Moisture

This was conducted according to methodology adapted from AOAC [3] and IAL [10].

2.7.3. Instrumental color

The particles were equally distributed in a glass capsule and 9 colorimetric determinations were made. A Chromameter CR-400 equipment (Konica-Minolta Sensing Inc., Osaka, Japan) programmed at CieLab system was used (Konica Minolta, 2007).

2.7.4. Mean diameter and size distribution (Polydispersion index)

The samples had their mean diameters and size distribution determined at Laser Light Scattering Analyzer LA-960 (Horiba Instruments, Inc., Japan) by light scattering, using the liquid dispersion mode (filtered water). The mean diameter determined was based on mean diameter of a sphere of the same volume (De Brouckere diameter – D [3,4]) and the results were generated directly by equipment software.

Polydispersion index (PDI) was calculated according to Moura et al. [17], using equation (6).

2.7.5. Active ingredient extraction to determine total phenolic compounds and antioxidant activity

In order to extract the active ingredients from wet particles, a methodology adapted from Moura et al. [17] was used, weighting approximately 15 g of sample, adding 12 mL of EDTA 0.2 M, and stirring for 30 min. Then, 20 mL of alcohol 70% was added and the sample was homogenized in Turratec (Tecnal model TE102, Piracicaba, Brazil) for 5 min, followed by filtration through filter paper. The extract was collected at an Erlenmeyer flask wrapped in aluminum foil, and the content retained on the filter paper was recovered. Then, 20 mL of acetone 70% was added and homogenization and filtration steps were repeated, proceeding to the third extraction with alcohol 70% once again. The last extraction was made with acetone 70%, for a total of four extractions. The extract was transferred to a 100 mL volumetric flask also wrapped in aluminum foil, which was completed with alcohol 70%. This extract was used for total phenolic compounds and antioxidant activity analyses by DPPH and ABTS methods.

2.7.6. Total phenolic compounds

These were determined according to the Folin Ciocalteau spectrophotometric method, used by Erkan-Koç et al. [12]. The method was conducted according to item 2.1.6, with the difference that the extract analyzed was that one obtained from wet particle extraction.

2.7.7. Antioxidant activity by DPPH and ABTS methods

This was measured according to Jiménez-Zamora et al. [13] as per item 2.1.7, with the sample comprising the extract obtained from wet particle extraction.

2.7.8. Encapsulation efficiency (EE)

The amount of total phenolic compounds effectively retained in microparticle structure after processing was measured. The encapsulation efficiency was calculated on wet basis by equation [7].

$$EE\ (\%) = \frac{\frac{mg\ \text{of active in microparticle}}{100\ g\ of\ \text{microparticle}}}{\frac{mg\ of\ \text{active added in the mixture}}{100\ g\ of\ \text{mixture}}}\ x\ 100 \tag{7}$$

2.7.9. Microparticle drying

Microparticles were dried in fluidized bed dryer, a process conducted by LabMaq (Ribeirão Preto/SP) using Fluidized Bed Dryer equipment FBD 1.0. Before the drying process, the particles were sieved and grounded (tamis of 1.4 mm and 710 μm), and a cotton piece of cloth was used to absorb the excess of moisture. After going through moisture analysis, the particles were taken to the equipment at specific conditions: inlet temperature 50 °C; outlet temperature 43 °C, and time of 40 min.

2.8. Statistical analysis

All the experiments were carried out in triplicate and submitted to analysis of variance for determining significant differences between the averages using one-way (ANOVA) analysis and Tukey test comparisons ($p < 0.05$) performed with SAS University software (version 94, 2017).

3. Results and discussion

3.1. Characterization of yerba mate hydroalcoholic extract

The pH found in yerba mate extract was 5.86 ± 0.01 [Table 1]. Chaicouski et al. [20] when characterizing soluble yerba mate

extract to study its composition and properties found a pH value of 5.89. Santos et al. [22] also observed a pH value of 5.2 for yerba mate aqueous extract. Both extracts were aqueous, however they resulted in values close to the one obtained in this study.

The soluble solid content shown by yerba mate hydroalcoholic extract was 28.7 ± 0.12 °Brix, which corresponds to 28.7 g of soluble solids in 100 g of extract, or 28.7% [Table 1]. Chaicouski et al. [20] in yerba mate hydroalcoholic extract achieved a soluble solid content of 24.25°Brix, a value close to the one found in this study. Riccio [21] obtained high soluble solid contents, except for aqueous extract (1.29°Brix). Alcoholic and hydroalcoholic extracts had values of 45.57°Brix and 41.12°Brix, respectively. These values may have differences due to the region where the raw material is collected, issues related to soil and climate, or how the processing was conducted for subsequent obtaining the extract. Leaf age may also influence the concentration of nutrients.

The moisture observed in hydroalcoholic extract was 91.60 ± 0.20% (Table 1). Due to its nature, the extract had a high moisture, a value that can affect the manner how it will be stored and packed as well as its processing [23].

In the literature related to the use of yerba mate extract in products, most authors choose to use the dry extract. Zanchett et al. [24] made this choice when developing a white chocolate with yerba mate extract. The moisture found was 5.63%, which consequently was lower to the hydroalcoholic extract moisture observed in this study. With the purpose of evaluating yerba mate extracts from Santa Catarina State, Da Croce [25] had results ranging from 25.21 to 41.16% for aqueous extracts. Nevertheless, the extracts had lower moisture than hydroalcoholic yerba mate extract of this study. As observed, extract moisture will depend on some factors such as the used solvent (ethanol or water) and processes the samples may go through, such as drying, reductions, among others.

Total titratable acidity found in yerba mate extract was 0.39 ± 0.00 g/100 g, considering citric acid as standard to achieve the result [Table 1]. This acidity was considered as low. According to Cecchi [23] the range of 0.2–0.3% is characteristic of fruits or vegetables with low acidity; the value observed is remarkably close to broccoli, which is approximately of 0.4%. In fruits with high acidity this value is considerably increased, such as lemon with a titratable acidity of around 6%. Riccio [21] characterized yerba mate extracts with different solvents: water, ethanol, and water:ethanol (1:1, v:v), achieving the following values: 0.14; 0.04 and 0.15 g citric acid/100 g, respectively. These values had differences according to the type of solvent used, but all remained below the acidity observed in extract analyzed in this study.

According to the color space L* a* b*, also called CIE LAB, L* represents luminosity ranging from lighter (100) to darker (0). a* and b* are chromatic coordinates ranging from +60 to −60. The first one varies from red (+) to green (−), and the second one from yellow (+) to blue (−). The more these values are away from the center, which is achromatic, the higher the color saturation [equation (1)], that is, in the center of chromaticity diagram where the values tend to zero, the colors are more diffuse, and as they move away, they become brighter and more intense (Konica Minolta, 2007).

The analyzed sample had L* value tending to darker [Table 1]. On the other hand, a* and b*, even being positive which would indicate red and yellow, respectively, since they are close to the center point of chromaticity diagram, had values indicating low purity and a more diffuse color. Frizon et al. [26] analyzed aqueous yerba mate extracts from different origins. The sample from young trees (12 years old) had the following values: L* 18.36; a*-2.37, and b* 28.17. Extract elaborated from older trees (80 years old) resulted in the following values: L* 34.38; a* −0.47, b* 45.14. Both had a higher trend to green and yellow, with the second one considered lighter than the first one. The extract used in the first study has shown to be darker than older tree extracts, but lighter than extract produced from young trees. The extract employed by Battiston et al. [27] to be used in white chocolate had values as follows: L* 38.30; a* 1.78, and b* 10.79. The hydroalcoholic extract of yerba mate in this study was also darker; however, with respect to yellow color trend, they have shown to be similar, with close values. These differences in color observed in the extracts may be related to several factors, not only the type of extraction reagent but also the region where the raw material was collected as well as the tree age.

Aiming to extend the use of particles prepared from the extract, we analyzed how the change in pH would affect the sample color. According to the results we found [Fig. 1], extract luminosity diminished as the pH was increased, that is, the higher the pH the darker the extract, and the lower the pH the lighter the extract. Luminosity had no significant differences in the range from 3 to 6, which comprises a great deal of food. Therefore, its use in products with this pH range would not lead to changes in extract color.

Table 1
Characterization of yerba mate hydroalcoholic extract (*Ilex paraguariensis*).

Analyses	Result
pH	5.86 ± 0.01
Soluble solid content	28.7 ± 0.12 °Brix
Total titratable acidity	0.39 ± 0.00 g/100 g
Moisture	91.60 ± 0.20%
L*	28.03 ± 0.40
a*	4.35 ± 0.10
b*	11.71 ± 0.54
C*	12.49 ± 0.54
h (hue)	0.39 ± 0.01
Total phenolic compound	32912.55 mg GAE/100 g
Antioxidant activity (DPPH)	1939.18 μmol TE/g
Antioxidant activity (ABTS)	2379.49 μmol TE/g
Chlorogenic acid (5-CQA)	0.35 ± 0.01 g/100 mL
Caffeine	0.29 ± 0.00 g/100 mL
Theobromine	0.03 ± 0.00 g/100 mL

GAE: Gallic acid equivalent. TE: Trolox equivalent.

Chromatic coordinates a* and b* also varied according to pH change. a* values were within 5–7 in samples, except for samples with pH 8. Regarding the b* value, as pH increased there was a trend towards a decrease in values representing the yellow color. Both a* and b* coordinates the pH 2 was significantly different from all the others. The range from 4 to 6 had no statistical difference at the 5% significance level. pH 7 was considered significantly different from all other values, as it occurred with pH 8. For this pH value the yerba mate extract shows a noticeably change in color [Fig. 1].

Friedman & Jurgens [28] demonstrated that some natural polyphenolic compounds present in coffee, tea, fruits and vegetables are damaged when exposed to a high pH (7–11). The chemical structure of phenolic compounds appears to have a profound effect on their susceptibility to such destruction as measured by their absorption spectra. In foods, pH values generally do not reach ranges above 7, not significantly compromising the phenolic compounds. The use of yerba mate extract in food products would therefore be feasible considering its stability to pH changes in the range from 2 to 7.

Hydroalcoholic extract of yerba mate has a high amount of total phenolic compounds [Table 1]. Before conducting Argentine yerba mate aqueous extract encapsulation, Deladino et al. [29] determined the total phenolic compound content. The value found was 62.11 mg GAE/g of yerba mate, which is considered below the value found in yerba mate extract analyzed in this study (329.12 mg g^{-1}).

Bioactive compounds present in yerba mate can have different concentrations, which depend on some variables such as climate and temperature of extraction, the type of solvent used, or even the drying conditions which the yerba mate leaves were subjected to, among others. All this reflects on the number of compounds that can be found.

Chaicouski et al. [30] obtained 1.47 g $kg^{-1}$of phenolic compounds in aqueous extract; for hydroalcoholic extract the value was 3.20 g kg^{-1}. Based on this data, hydroalcoholic extraction was considered to be more efficient, since it could extract polar and low polar substances. These results have shown to be considerably below the value found for hydroalcoholic extract in this study (329.12 g kg^{-1}). Yerba mate hydroalcoholic extract showed a high potential to be used in products for its high phenolic compound content, above the value found in several studies in literature.

In addition, the extract had high antioxidant activity [Table 1]. Both methodologies used have resulted in close values, which is expected as the values are shown in μmol/g Trolox equivalent.

When analyzing antioxidant activity by ABTS of *in natura* aqueous yerba mate extract, Gris [31] achieved 13.96 mmol Trolox.g^{-1} of sample, which result is superior to the one observed in this study. The same has not occurred for Riccio [21], who quantified the antioxidant activity of yerba mate extract obtained from dry leaves of *Ilex paraguariensis* in water and ethanol (1:1 v/v). When ABTS method was used, the author observed a value of 270 μmol Trolox.g^{-1} of yerba mate.

Wolff et al. [32] analyzed the antioxidant activity of yerba mate extracts by DPPH method. Two analyzed extracts were

pH of the sample	L*	a*	b*
2.00	31.09 ± 0.77^{a}	5.12 ± 0.32^{c}	16.86 ± 0.72^{a}
3.00	29.00 ± 0.69^{b}	6.38 ± 0.33^{b}	13.47 ± 0.66^{b}
4.06	28.85 ± 0.59^{b}	6.34 ± 0.33^{b}	13.40 ± 0.48^{b}
5.00	28.68 ± 0.67^{b}	6.39 ± 0.28^{b}	12.80 ± 0.68^{b}
6.05	28.70 ± 0.81^{b}	6.65 ± 0.23^{b}	12.98 ± 0.61^{b}
7.07	27.30 ± 0.83^{c}	7.08 ± 0.22^{a}	10.84 ± 0.75^{c}
8.08	22.14 ± 0.53^{d}	1.70 ± 0.23^{d}	1.82 ± 0.42^{d}

n = 9 (number of repetitions used). Hydroalcoholic extract of yerba mate diluted in ethanol 70°GL at the ratio of 1:5. Mean values followed by different letters are significantly different from each other at p<0.05 by Tukey's test.

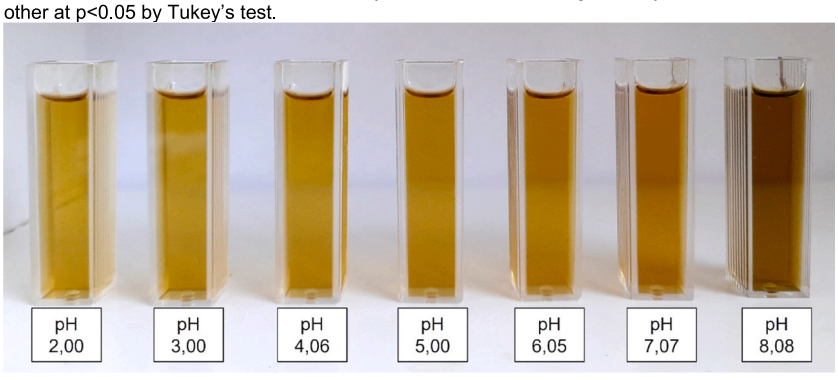

Fig. 1. Instrumental color results of hydroalcoholic extract of yerba mate (*Ilex paraguariensis*) with pH changes. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

hydroalcoholic ones with 50% (ethanol:water), and the results were 12,175.10 and 9888.89 μmol TE $L^{-1}$of extract. The difference between them was the time of extraction, 120 min, and 60 min, respectively. The longer time resulted in higher time of extraction. The results achieved by the authors was inferior to this study since it consists of antioxidant activity in 1 L of extract. Several factors can influence the antioxidant capacity of yerba mate extract, starting with the location of yerba plant, as well as the soil, solar exposure, and the time of year when the leaves were collected. The procedures used, including the time of extraction and the solvents, can result in an aqueous, alcoholic, or even hydroalcoholic extract, as in this study. In other words, the differences observed in values found in literature may comprise the mentioned aspects and be determinant to extract antioxidant activity in the methodologies adopted.

Analysis by HPLC of identifying and quantifying the components found in yerba mate extract has shown that the predominant

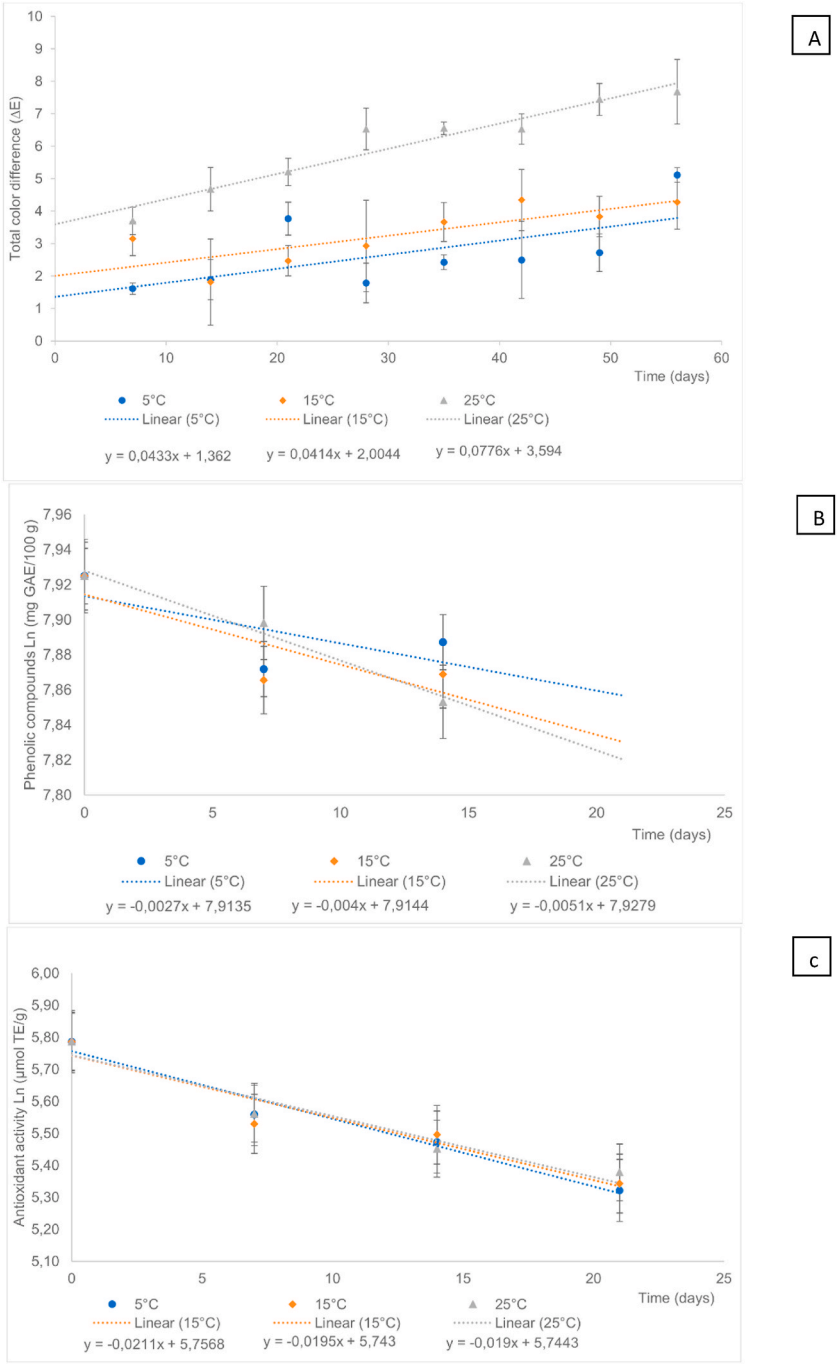

Fig. 2. Charts showing total color difference (ΔE) (A), total phenolic compounds (B), and antioxidant activity (C) of yerba mate over time. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

compound is chlorogenic acid. Due to the methodology employed, only 5-CQA was identified, without its isomers. The caffeine concentration was the second highest value found, with a content close to chlorogenic acid one. The component with the lowest concentration identified in extract was theobromine. This result agrees with Colpo [33], who analyzed the phytochemical profile and antioxidant capacity of yerba mate from Brazil, Argentina, and Uruguay. The analyzed extracts simulated the form of yerba mate consumption in order to know the compounds ingested by population normally consuming the product. A chromatographic analysis showed that chlorogenic acid was the compound with higher content in extract. On the other hand, theobromine was the substance with the lowest amount.

Tonet et al. [2] quantified the bioactive compounds present in yerba mate dry extract by *spray dryer*, for subsequent use in fish hamburger. The extract had 49.6 mg g^{-1} of chlorogenic acid and 34.5 mg $g^{-1}$of caffeine. The compound concentration determined by the authors have shown to be higher than the one observed in this study.

Conversely, Bojić et al. [35] found 3 mg $g^{-1}$of chlorogenic acid, 5.4 mg $g^{-1}$of caffeine and 2.7 mg g^{-1} of the obromine in aqueous extract. The chlorogenic acid concentration was close to the value found in this study. Active compound concentrations may vary depending on the solution used for extraction, as well as the type of raw material used (*in natura* or processed leaf) and the extraction methodology adopted.

3.2. Color stability, total phenolic compounds, and antioxidant activity of yerba mate extract

Color stability of yerba mate extract was studied based on parameter ΔE (total color difference) [equation (3), Figure (2A)]. The ΔE result was adjusted for Arrhenius and adapted to zero order model, at $Q_{10} = 1.86$ and activation energy (E_a) = 11.18 kJ/mol. The Q_{10} value shows that each 10 °C increase will increase the total color difference 1.86 time. The activation energy obtained was high, showing that the temperature had a great influence on the total color difference during storage.

Dionisio et al. [34] used the peduncle fiber of cashew (*Anacardium occidentale* L.) to elaborate an extract (concentrated carotenoid extract), which would function as a natural food dye. Its stability was studied for 180 days stored at 5 °C. The authors measured the ΔE from initial and final storage time, which was 1.4, highlighting the extract has not substantially changed with time and the changes were considered unnoticeable. In this study, yerba mate extract color had more pronounced changes at a shorter period of time in comparison to the previously mentioned study, showing it can be more sensitive to storage conditions.

ΔE had a gradual increase through the days and as the storage temperature increased. In other words, the longer the time and the higher the storage temperature, the higher the changes in extract instrumental color, evidencing it changes more quickly when the extract is exposed to higher temperatures over time. Measuring this parameter was important to establish how the color of this extract, when applied to some product, could noticeably change after a certain storage time and at different temperatures.

In the stability evaluation of phenolic compounds present in yerba mate extract, an adjustment that best represented the changes occurred during storage was made, which was in a first-order model in this case.

When comparing the three curves related to phenolic compounds [Figure (2B)], we observe the curve corresponding to storage at 25 °C had a higher slope, indicating that, when stored at such temperature, the extract shows a significant change in its phenolic compounds. This parameter had the following values: $Q_{10} = 1.27$ and $E_a = 14.81$ kJ/mol. Such activation energy shows the temperature had a greater influence on phenolic compound degradation. Along with this information, Q_{10} indicates that each increase of 10 °C makes the phenolic compounds degrade 1.27 times as much.

Studies related to phenolic compound stability in vegetables show the storage temperature directly influences their preservation or degradation. It was verified that lower temperatures could preserve such compounds for a longer time [35]. This conclusion supports the results obtained with yerba mate extract.

Oliveira et al. [37] studied the effects of light in stability of phenolic compounds present in mate tea stored for 8 weeks at room temperature. It was observed that the sample stored protected from light did not show any significant difference in total phenolic compound content during the study period. However, mate tea stored under light exposure had a significant decrease in phenolic compounds. In 8 weeks of storage the decrease was 4.3%, evidencing the light can be a relevant factor in degradation of these compounds. It should be emphasized that not only the light exposure or experiment duration had effects on phenolic compound degradation. Keeping the sample at room temperature could also have contributed to the result achieved, with total deterioration. As observed in this study, the extract stored at 25 °C, which is considered as room temperature, had the highest reduction in phenolic compound concentration.

[Figure (2C)] shows that antioxidant activity of extracts stored at 5 °C, 15 °C, and 25 °C was reduced with time.

Grisi et al. [38] analyzed the antioxidant activity by DPPH of aqueous and hydroalcoholic extract of *jucá* fruit (*Libidibia ferrea*) stored at 5 °C for 90 days. The results observed were as follows: aqueous extract: 2.27 mol Trolox/g (day zero) to 1.63 mol Trolox (day 90); hydroalcoholic extract: 2.91 mol Trolox/g (day zero) to 1.92 mol Trolox (day 90). In this case, the fruits that had their antioxidant compounds extracted with a solution of ethanol/water 50% (v/v) had a better result in antioxidant activity quantification. There was a gradual loss, reaching the last day of the experiment with a considerable antioxidant activity. This study was conducted by using hydroalcoholic extract, and antioxidant activity was not affected by the storage temperature. It was dropping little by little, getting lost over the days.

Antioxidant activity determined by DPPH was adjusted to the first-order model, with $E_a = 3.45$ kJ/mol and $Q_{10} = 0.92$. The lower values obtained for activation energy and Q_{10}show that temperature had a lower influence on degradation of extract antioxidants over time.

3.3. Characterization of simple emulsion (W/O)

3.3.1. Microstructure

Emulsifiers are widely used in food industry. They are additives capable of diminish the surface tension between oil and water or between water and air, promoting emulsification and increasing the emulsion stability. They also improve food texture and uniformity, in addition to extend its shelf-life [39].

According to McClements [40], during prolonged homogenization there is a reduction in droplets and an increase in interfacial area, which at first can make the amount of emulsifier insufficient. Without the emulsifier to cover these areas, the droplets tend to agglutinate, compromising the stability. For the analyzed emulsion, it is possible to affirm the use of 5% of surfactant PGPR was sufficient to assure the coverage of interfacial areas, which maintained the stability of this emulsion.

3.3.2. Sedimentation index

The sedimentation index (SI%), equation [4], is related to the emulsion stability, defined by its capacity to maintain the properties over time. Emulsion instability can happen due to several physical or chemical factors. One of the physical changes is sedimentation caused by instability, where the droplets make a downward motion occurring due to their higher density in comparison to the liquid surrounding them [39]. The sedimentation index found for emulsion was 1.46%.

Size distribution can be related to the type of homogenizer used in the emulsion elaboration process. Some types can produce narrow size distributions; however, those classified as high shear homogenizers tend to produce higher distributions. In general, if there is a wide difference between several size values, polydispersity tends to be higher [40].

According to McClements [40], the emulsion droplet size study contributes to the assessment of product shelf-life, as well as its texture and appearance. If the emulsion has any compound with a specific purpose, the drop diameter will also affect its characteristics of release in the organism. Also, according to the author, the higher the rotation speed and homogenization time, the smaller the droplet size. This occurs until a lower limit is achieved, to which the reference depends on the type of sample used. High shear dispersers as the one used in the experiment, usually produce droplets in diameter ranging from 1 to 10 μm. These values correspond to the one observed in the emulsion in this study, with droplets ranging from 1 to slightly above 10 μm. The mean diameter (D [2,3]), equation [5], observed was 4.166 μm ± 0.566 with polydispersity index (PDI), equation [6], of 0.935 ± 0.03.

3.4. Study of microparticles

3.4.1. Microstructure

Wet microparticles were observed in a stereoscope with magnification of 40× [Figure (3A)] and in an optical microscope with magnification of 100× [Figure (3B)]. The images show the emulsion droplets arrested in each particle, which shows the ionic gelation has accomplished its goal.

Encapsulation of active compounds of interest, which often degrade easily, is an efficient alternative to preserve them and enable their release on appropriate sites only. Ionic gelation can perform this role at a lower cost when compared to other techniques.

At a higher magnification it is possible to observe the particle surface in detail, which appears to be smooth and remains intact. In addition, it shows the small emulsion droplets partly composed of the extract are protected by the wall formed by the binding of pectin and calcium chloride.

Rocha [41], by using stereoscope micrographs, concluded the tomato juice microspheres elaborated by the author regardless the core material used (sodium alginate, maltodextrin, and sodium alginate + insulin), had a surface without fissures, assuring their preservation. This is a quality also observed in yerba mate microparticles.

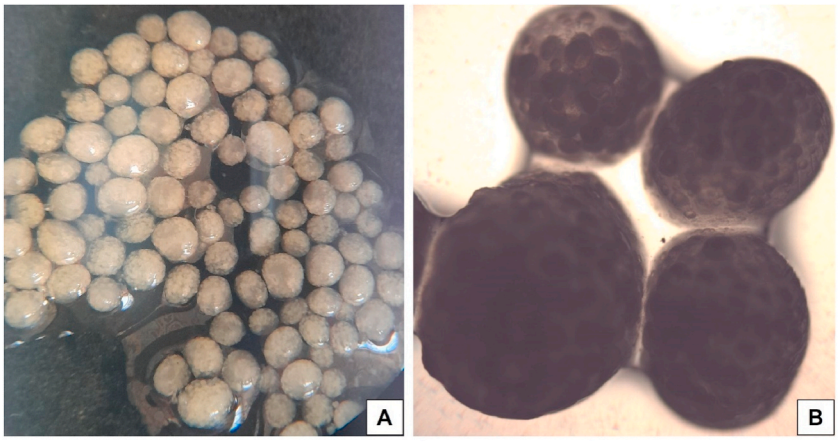

Fig. 3. Microstructure of yerba mate extract particles produced by ionic gelation, viewed at stereoscope with magnification of 40× (A), and at an optical microscope with magnification of 100× (B).

3.4.2. Characterization

The moisture content in particles was 79.24% ± 0.51 [Table 2]. Due to the microencapsulation technique of ionic gelation used, high moisture contents were expected.

Rocha [41] also used ionic gelation to produce tomato juice microspheres. The author evaluated three types of formulation, which were different with respect to the presence of sodium alginate, maltodextrin, or insulin). The particles had moisture contents of 93.4% (sodium alginate), 95.67% (maltodextrin), and 96.56% (inulin). Moisture was considered high for the three formulations. In this work, yerba mate extract microcapsules had high moisture, although lower than tomato juice microspheres probably due to the presence of oil in the double emulsion.

Ferreira [42] produced beet extract microcapsules by ionic gelation with the purpose of making use of their functional properties. Among characterization analyses, the author verified a moisture content equal to 92.7%, a value above the one observed for yerba mate microparticles.

The high moisture content found in microparticles can be associated with the use of pectin as core material, since it is a polysaccharide with strong affinity to water. Its use is widespread in food products due to versatility, which enables it to be used as an emulsifier, thickener, and stabilizer [43].

The high contents show the use of this technique is associated with high moisture particles and this information is crucial when choosing the applicability.

When microencapsulating the yerba mate freeze-dried extract by ionic gelation, Vargas et al. [44] had particle sizes equal to 180.43 μm (D50), with polydispersity index of 1.75. Gelation was conducted through a solution of sodium alginate and freeze-dried extract atomized in calcium chloride. The microparticles were smaller than those elaborated in this study. The same method was used to form the particles; however, they were prepared differently. The use of atomization rather than dripping interferes with the diameters. PDI was higher than in this study, indicating low homogeneity in particle sizes.

Therefore, it becomes evident that small changes in microparticle elaboration from their formulation and core material to how they are formed will cause differences in the diameters.

Wet particles had high luminosity (L*) of 68.15, which shows how bright the particles became [Table 2]. Even if a* was tending to green, the value is in the central area of the color wheel, which brings a diffuse hue to this chromatic coordinate. With respect to b*, the value has a trend to yellow. Due to the low chroma value is possible to notice the particles had a less saturated color.

When microencapsulating beet extract by ionic gelation, Ferreira [42] had L* and C* values of 26.80 e 10.53, respectively. Both values were lower than those observed in this study, which means the color of yerba mate particles was considered clearer and more saturated than beet microcapsules.

We could determine total phenolic compounds of wet microparticles: 87.86 ± 1.79 mg/100 g w.b [Table 2], which corresponds to 423.18 ± 8.60 mg/100 g d.b. However, the extract corresponds to 35% of the emulsion, and during microparticle elaboration, 20% of emulsion is used only. The quantification is expected to be lower, around 7% of the value observed in the extract. The encapsulation efficiency was 45%, equation [7]. In the encapsulation of pomegranate extract using ionic gelation by dripping and with pectin as wall material, EE values of phenolic compounds between 41.2 and 43.4% were found [9]. Since they are too sensitive, phenolic compounds tend to lose their properties of interest fast when compared to unprotected compounds as free extract. Although losses have occurred, the extract attributes are protected in the particles, enabling them to be preserved for a longer period. The Folin Ciocateau method was used to determine the total levels of polyphenols in the samples. This method, although widely used, is pointed out by some authors as controversial such as ascorbic acid, amino acids, citric acid and reducing sugars all have an interference effect [45]. In view of this, the antioxidant activity was also analyzed by the DPPH and ABTS methods.

If we consider the antioxidant activity by the DPPH and ABTS methods for particles before drying, the values found were 3.13 ± 0.06 μmol/g w.b. and 4.39 ± 0.05 μmol/g w.b., respectively [Table 2]. The microparticles showed EE = 27% by DPPH and 31% by ABTS, equation [7].

In Mendes et al. [46] study with *jaboticaba* extract particles elaborated by ionic gelation, the formulation composed of 1.5% of alginate/1 mL of extract were evaluated before and after passing through a thermal process (180 °C). The antioxidant activity was

Table 2
Characterization of wet and dried yerba mate (*Ilex paraguariensis*) microparticles.

Analyses	Wet microparticles	Dried microparticles
Moisture	79.24 ± 0.51%	18.64 ± 4.2%
L*	68.15 ± 1.57	39.19 ± 1.11
a*	−4.35 ± 0.11	4.19 ± 0.11
b*	23.97 ± 0.89	27.97 ± 0.70
C*	24.36 ± 0.87	28.28 ± 0.69
h (hue)	−0.18 ± 0.01	0.15 ± 0.01
Mean diameter	1321.76 ± 12.03 μm	1009.66 ± 28.06 μm
Polydispersity index	0.51 ± 0.01	1.10 ± 0.06
Total phenolic compounds	87.86 ± 1.79 mg GAE/100 g w.b.	379.86 ± 5.76 mg GAE/100 g w.b.
Antioxidant activity (DPPH)	3.13 ± 0.06 μmol TE/g w.b.	15.83 ± 0.17 μmol TE/g w.b.
Antioxidant activity (ABTS)	4.39 ± 0.05 μmol TE/g w.b.	19.20 ± 0.93 μmol TE/g w.b.

GAE: Gallic acid equivalent. TE: Trolox equivalent.

978.94 μM/g before and 773.50 μM/g after undergoing a high temperature (180 °C/20 min). This process caused 20.98% loss of antioxidant capacity of the particle, with values by ABTS analysis.

3.4.3. Drying

Partial drying of the particles had the purpose of increasing their scope of use and application, in addition to extend their shelf-life. Fluidized bed dryers are very efficient in terms of energy and maintaining the quality of dried products, without damaging heat sensitive materials [47].

Measuring the moisture content of the particles is essential to evaluate their compatibility with the product in which they will be employed. When using fluidized bed drying, the dry particles ended the process with 18.64 ± 4.2% of moisture, had a higher intensity of color (L*: 39.19 ± 1.11; a*: 4.19 ± 0.11, and b*: 27.97 ± 0.70) and lower mean diameter (1009.66 ± 28.06 μm) than wet particles [Table 2 and Fig. 4]. In addition, drying has not significantly affected the phenolic compound content (379.86 ± 5.76 mg/100 g w.b. or 468,97 ± 7,11 mg/100 g d.b.) and antioxidant activity (DPPH 15.83 ± 0.17 μmol/g w.b. ABTS 19.20 ± 0.93 μmol/g w.b.) [Table 2]. Similar values of phenolic compounds (376.40 ± 23.80 to 408.08 ± 12.54 mg/100 g d.b) were found by Silveira et al. [9] in the encapsulation of pomegranate extract using ionic gelation by dripping and subsequent drying.

> By reducing the moisture, the particles could be employed in drier products in which high moisture content would cause changes in their characteristics. Moreover, there is also the possibility of using a higher number of particles, bringing greater potential of bioactive compounds to food products.

After drying in a fluidized bed, the obtained value of phenolic compounds from the dry microparticles increased by 4.3 times. Likewise, for the dry microparticles there was also an increase of at least 4.3 times in the antioxidant activity. The results achieved in both methods were uniform, which was already expected and shows that, even using different methodologies, the particles had similar antioxidant activity in Trolox equivalent.

When microencapsulating hydroalcoholic extract of *jabuticaba* by ionic gelation, Mendes et al. [46] measured the phenolic compounds of microparticles prepared with several formulations, changing the alginate (core material) and *jabuticaba* extract contents. Samples having 1% of alginate/3 mL of extract and 1.5% of alginate/1 mL of extract had values of antioxidant activity of 16.02 mg GAE/g and 4.59 mg GAE/g, respectively. The value for the first formulation was close to the one found in yerba mate particles in dry basis [(Table 2)].

Ćorković et al. [48] elaborated hydrogel spheres with Aronia berry juice, aiming to exploit the already known antioxidant capacity of the fruit. The authors conducted assays to quantify by DPPH and ABTS the antioxidant activity of the spheres having alginate/pectin combination as core material, achieving values of 28.59 μmol/100 g for DPPH, and 35.24 μmol/100 g for ABTS. As in this study, the results achieved by ABTS have shown to be higher than those by DPPH. The antioxidant activity in yerba mate microparticles were considered to be higher.

4. Conclusion

Yerba mate extract showed high content of total phenolic compounds, mainly chlorogenic acid (5-CQA), and high antioxidant activity. During the stability study, the total color difference (ΔE) was observed to increase over time and as the temperature increased, which has also influenced the loss of phenolic compounds. Storage at a lower temperature (5 °C) assured better preservation of total phenolic compounds.

The ionic gelation technique has proven to be appropriate for microencapsulation of yerba mate extract. The microparticles had total phenolic compound content and antioxidant activity considered relevant for the use of these particles in future food products. The drying of microparticles may extend the shelf-life and increase the variety of foods for application.

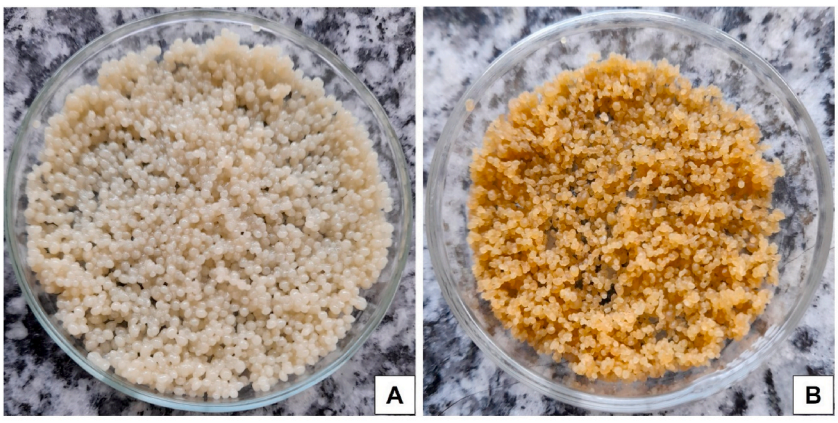

Fig. 4. Wet (A) yerba mate extract microparticles and after drying (B).

Author contribution statement

Ana Caroline Budin: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data.
Leonardo Vinícius Takano: Performed the experiments; Analyzed and interpreted the data.
Izabela D. Alvim: Performed the experiments; Contributed reagents, materials, analysis tools or data.
Sílvia C.S.R. de Moura: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Data availability statement

Data included in article/supp. material/referenced in article.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:
Silvia Cristina Sobottka Rolim de Moura reports financial support was provided by State of Sao Paulo Research Foundation - FAPESP, Brazil (Grant 2019/19647-0). Silvia Cristina Sobottka Rolim de Moura reports a relationship with Institute of Food Technology - ITAL, Brazil that includes: non-financial support.

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