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# Chemical Characterization of Candy Made of Erva-Mate (*llex paraguariensis* A. St. Hil.) Residue

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The aim of this work was to evaluate the chemical properties of the residues from erva-mate processing and also to determine the candy-making performance with addition of residues from erva-mate on consumers' acceptance and purchase intent of this new product. The candies containing different amounts of mate powder were evaluated through overall acceptability test and purchase intent. Mate powder showed high contents of dietary fiber, total ash, and total polyphenols. The total dietary fiber content of the mate candies ranged from 5.7 to 6.29% on a dry matter basis. Supplementation with mate powder caused significant increases in polyphenol and mineral contents of mate candies. The incorporation of mate powder increased the hardness of the candies and produced desirable results in their nutritional characteristics. The sensory tests indicated that mate candies were acceptable and approved in relation to purchase intent.

KEYWORDS: Erva-mate; residue; chemical composition; mate candies; acceptability

## INTRODUCTION

Economically, *Ilex paraguariensis* A. Saint Hilaire is a very important plant in South America. It is grown naturally and cultivated in southern Brazil, northeastern Argentina, and eastern Paraguay (1). The commercial product made with it, called "mate", "yerba-mate", or "erva-mate" is used in the preparation of several types of beverages, such as "*chimarrão*", "*tererê*", soft drinks, and teas (2). Its popularity is increasing in the United States, Canada, and Europe (1).

Erva-mate shows central nervous system stimulant properties attributed to its content of methylxanthine alkaloids such as caffeine and is also known for containing compounds with antioxidant proprieties, such as phenolic acids (3-6). Other effects of erva-mate have also been reported, and these effects explain its popular use as a hepatoprotective, choleretic, diuretic, hypocholesterolemic, antirheumatic, antithrombotic, anti-inflammatory, antiobesity, and antiaging agent (7, 8).

The processing of erva-mate consists of three different phases: (a) a rapid drying process called "*sapeco*", with the aim of inhibiting enzymatic activity and reducing moisture level; (b) a partial drying stage, which is usually performed in rotating cylinders heated by burning wood; and (c) a further drying and subsequent grinding stage (9). During the grinding stage of erva-mate processing, the larger twigs are not added to the final product, so these twigs and a little of the ground leaves are ground again to make the twigs smaller. In this second grinding stage, residue is generated from twigs and leaves of very small granulometry (referred to as mate powder), which is not added to the final product and is eventually discarded. However, there is no information available in the literature about this material or its application as a food ingredient. Therefore, because of the known benefits of ervamate to human health, the residues from erva-mate processing can have several applications, which could increase the consumption of this raw material and, consequently, enhance its market.

Erva-mate residues, therefore, emerge as an alternative to develop new products and to add value to food industry residues that are usually discarded. Thus, the aim of this work was to evaluate the chemical properties of the residues from erva-mate processing and also to determine the candy-making performance with addition of residues from erva-mate on consumers' acceptance and purchase intent of this product.

# MATERIALS AND METHODS

**Materials.** Residues from the grinding stage of *I. paraguariensis* processing (mate powder), which consist of twigs and a few leaves, were supplied by three erva-mate processing industries in Catanduvas, Santa Catarina state, Brazil.

Folin-Ciocalteu, sodium carbonate, gallic acid, 2,2'-azinobis[3ethylbenzthiazoline sulfonate] (ABTS<sup>++</sup>), 6-hydroxy-2,5,7,8-tetra-

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methylchroman-2-carboxylic acid (Trolox), theobromine, and caffeine were obtained from Sigma Chemical Co. (St. Louis, MO). The reagents used were of analytical grade.

**Preparation of Mate Powder.** The residues from *I. paraguariensis* processing were mixed and screened to pass through a 60 mesh sieve (Britsh Standard Screen). The residue, referred to as "mate powder", was packaged in an airtight plastic bag and stored at  $-18 \pm 2$  °C until required.

**Mate Candy Formulation.** The mate candy formulations were prepared by blending the following ingredients: sugar (55.00 g/100 g), water (40.8 mL/100 g), pectin (4.00 g/100 g), ascorbic acid (0.20 g/100 g), and mate powder in different proportions (0.4, 0.8, and 1.2 g/100 g). The ingredients were cooked on a conventional stove at  $100 \pm 2$  °C for 22 min. The dough was manually rolled on a pattern board into sheets of uniform thickness and divided into pieces of about 1.5 cm<sup>3</sup>. Ten mate candies from each batch of the three cooking replications were analyzed for chemical and physical measurements.

**Chemical Analysis of Mate Powder and Mate Candies.** Moisture, total ash, lipid, and crude protein (N  $\times$  6.25) were determined through AOAC methods (*11*). Soluble and insoluble dietary fiber contents were determined by enzymatic–gravimetric method (*11*). Total carbohydrate was calculated by the difference. Energy values (kilocalories) were obtained by applying factors 4, 9, and 4 for each gram of protein, lipid, and carbohydrate, respectively (*12*).

The concentrations of calcium (Ca), magnesium (Mg), iron (Fe), zinc (Zn), manganese (Mn), and aluminum (Al) were determined through atomic absorption spectrophotometry using a Perkin-Elmer Analyst 300 spectrophotometer. Potassium (K) and sodium (Na) were determined by flame photometry (B262 Micronal) with a scale reading from 0 to 150 mg  $L^{-1}$ . Phosphorus (P) was determined by using a UV–vis spectrophotometer (Hitachi model U-1800). Mineral contents were determined according to the method described by the AOAC (11).

Total Polyphenol and Antioxidant Activity of Mate Powder and Mate Candies. Extracts were prepared with 2 g of sample mixed with methanol 80% (v/v) and shaken for 1 h. The extracts were filtered and transferred into an amber bottle and purged with a stream of nitrogen.

The total polyphenol content (TPC) was determined according to a modified Folin-Ciocalteu procedure (13). The appropriate dilutions of extracts were oxidized with Folin-Ciocalteu reagent, and the reaction was neutralized with sodium carbonate. The absorbance of the resulting blue color was measured at 725 nm after 60 min in a UV-vis spectrophotometer (Hitachi model U-1800). The TPC was expressed as gallic acid equivalents in grams per 100 g of dry material.

To determine the antioxidant activity in vitro, ABTS was used to assess the free radical scavenging capacity. The free radical cation ABTS was performed through the reaction of 7 mM ABTS with 2.45 mM potassium persulfate for 12–16 h at room temperature in the dark. The decrease of ABTS absorbance at 754 nm (initial absorbance =  $0.7 \pm 0.02$ ) in the presence of an antioxidant was monitored for 7 min. Trolox (0–600  $\mu$ M) was used for calibration, and the results were expressed on a micromolar Trolox per gram of dry matter basis (*14*).

Methylxanthines of Mate Powder and Mate Candies. Samples (15 g each) were boiled for 10 min in an aqueous solution of sulfuric acid 20% (v/v) (150 mL) and then filtered. After being neutralized with a 25% (v/v) ammonium hydroxide aqueous solution, the filtrate was extracted using a chloroform/isopropanol mixture (3:1, v/v). The dried organic phase was concentrated, yielding the methylxanthines extract. This residue was submitted to high-performance liquid chromatography (HPLC) analysis through dilution in the mobile phase (15).

Aliquots (10  $\mu$ L/sample) were injected into a liquid chromatograph (Shimadzu LC-10) equipped with a reverse-phase column (Shim-pack C<sub>18</sub>, 4.6 mm i.d. × 250 mm long) thermostated at 30 °C and a UV–visible detector (Shimadzu SPD 10A,  $\lambda = 280$  nm). An isocratic mobile phase of an acetonitrile/0.1% formic acid (15:85, v/v) mixture (*16*) was used with a flow rate of 1.0 mL min<sup>-1</sup>. Prior to injection, all of the samples were centrifuged (5000 rpm/10 min). For quantitative analysis, a standard calibration curve was obtained by plotting the area of peaks against different concentrations (1.0–100.0  $\mu$ g mL<sup>-1</sup>;  $r^2$  =

0.99) of caffeine. Similarly, for each sample the final concentration of the compounds was determined by average content after three consecutive injections.

**Physical Characteristics of Mate Candies.** The yield of the mate candies was estimated by difference between the weight results of the mate candies before and after cooking.

The hardness of the mate candies containing 0.4-1.2% of mate powder was measured with a Brookfield Engineering texturometer, model LFRA 1000 (Brookfield Engineering, London, U.K.). The individual samples of mate candies were placed on the platform, the probe was attached to the crosshead of the instrument, and the level of penetration was 50% of their original height. Hardness of the mate candies was measured with a cylindrical probe of 39 mm diameter, and the conditions for texture profile analysis (TPA) were kept at a pretest and post-test speed of 1 mm/s. The absolute peak force was considered as the hardness of the mate candies (*17*).

Color measurements of mate candies were carried out using a colorimeter Minolta Chroma Meter CR-400 (Konica Minolta) with D<sub>65</sub> illuminant and 10° angle of vision. The mean values for  $L^*$  (lightness),  $a^*$  (redness to greenness),  $b^*$  (yellowness to blueness), chroma  $[(a^{*2} + b^{*2})^{1/2}]$  and hue angle  $[\tan^{-1} (b^*/a^*)]$  were determined according to the CIE Laboratory system. The mean values of the top and bottom surface color of each candy were evaluated. Color parameters were determined in triplicate, and 10 measurements were carried out for each sample.

Consumer Acceptance and Purchase Intent of Mate Candies. A total of 100 volunteers, habitual candy consumers (men and women, between 17 and 62 years old), were recruited. Consumer acceptance test was performed according to the methods described by Meilgaard et al. (18) to evaluate the overall acceptability of mate candy samples that had been stored for 5 days in high-density polyethylene bags at room temperature ( $25 \pm 2$  °C). A five-point hedonic scale ranking from "dislike extremely" to "like extremely", corresponding to the lowest and highest scores of "1" and "5", respectively, was used.

The purchase intent was also evaluated on a five-point scale from "definitely would not buy" to "definitely would buy" corresponding to the lower and higher scores of "1" and "5", respectively (*18*).

**Statistical Analysis.** All analytical determinations were carried out in triplicate. Mean  $\pm$  SD values were calculated, and the data were subjected to analysis of variance (ANOVA). Tukey's test for multiple comparisons of means was performed to determine differences ( $p \leq 0.05$ ) between treatments. Correlations between antioxidant activity and TPC were also investigated.

#### **RESULTS AND DISCUSSION**

Chemical Composition of Mate Powder and Mate Candies. The chemical composition of mate powder and mate candies is shown in Table 1. Lipid and protein contents of mate powder were lower than those of erva-mate as reported by Esmelindro et al. (9). The results of the chemical analyses revealed that mate powder is an important source of dietary fiber, mainly insoluble fiber. Mate powder showed higher contents of total dietary fiber (59.14% dm) than commercial erva-mate (21.89% dm) as reported by Esmelindro et al. (9) and higher than the levels reported for other high-fiber foods, such as wheat bran, rice bran, and barley bran (19). The consumption of about 10 g of mate powder would provide about 24% of the requirement for dietary fiber (25 g day<sup>-1</sup>), as recommended by the Food and Agriculture Organization/World Health Organization for adult individuals (20). Thus, mate powder appears to be an interesting alternative in the manufacture of high-fiber foods.

Agronomic byproducts have traditionally been undervalued; however, there is now a trend to search for new sources of dietary fiber (21). High-fiber ingredients have many properties that influence the functional properties of foods (22). In addition, the consumption of these high-fiber foods has been extensively studied with regard to health benefits (23).

Table 1. Chemical Composition (Grams per 100 g) of Mate Powder<sup>a</sup> and Candies Containing Mate Powder (Dry Basis)

		type of candy			
component	mate powder	0.4 <sup>b</sup>	0.8 <sup>b</sup>	1.2 <sup>b</sup>	
moisture	$8.01\pm0.09\mathrm{b}$	$21.29 \pm 0.43$ a	$21.19 \pm 0.19$ a	$21.10 \pm 0.37$ a	
total ash	$5.67 \pm 0.02 \ { m a}$	$0.03\pm0.01~{ m d}$	$0.06\pm0.01~{ m c}$	$0.10\pm0.02\mathrm{b}$	
lipid	$3.76 \pm 0.09 \ { m a}$	$0.03\pm0.01~{ m d}$	$0.05\pm0.02~{ m c}$	$0.07\pm0.02$ b	
crude protein	$9.77 \pm 0.06 \ { m a}$	$0.06\pm0.01~{ m d}$	$0.13\pm0.01~{ m c}$	$0.16\pm0.01$ b	
dietary fiber	$59.14 \pm 0.19$ a	$5.70\pm0.08$ d	$6.08\pm0.10~\mathrm{c}$	$7.29\pm0.10$ b	
soluble dietary fiber	$5.79 \pm 0.09$ a	$5.43\pm0.04$ b	$5.46\pm0.06$ b	$5.48\pm0.05$ b	
insoluble dietary fiber	$53.35 \pm 0.30~{ m a}$	$0.27\pm0.04~{ m d}$	$0.62\pm0.23\mathrm{c}$	$1.00\pm0.15$ b	
carbohydrate <sup>c</sup>	$13.66\pm0.12\text{d}$	$\textbf{72.92}\pm\textbf{0.36}~\textbf{a}$	$72.51\pm0.32~\text{b}$	$71.31\pm0.22\mathrm{c}$	
energy (kcal/100 g)	127.56 $\pm$ 1.21 d	$292.81 \pm 1.38$ a	$290.93\pm1.50$ b	$286.45 \pm 1.01~{ m c}$	

<sup>a</sup> Residue from erva-mate (*I. paraguariensis*) processing. <sup>b</sup> Mate powder amount in mate candies (%). Mean values  $\pm$  SD of triplicate determinations. Mean values in the same line followed by different letters are significantly different ( $p \le 0.05$ ). <sup>c</sup> By difference.

 Table 2. Mineral Content (Milligrams per 100 g) of Mate Powder<sup>a</sup> and Candies Containing Mate Powder (Dry Basis)

		type of candy		
component	mate powder	0.4 <sup>b</sup>	0.8 <sup>b</sup>	1.2 <sup>b</sup>
aluminum calcium iron phosphorus magnesium manganese potassium sodium zinc	$\begin{array}{c} 32\pm1a\\ 899\pm11a\\ 31\pm1a\\ 118\pm6a\\ 314\pm4a\\ 91\pm3a\\ 919\pm11a\\ 10.8\pm0.5a\\ 7\pm0.1a \end{array}$	$\begin{array}{c} 0.14 \pm 0.00 \text{ d} \\ 3.96 \pm 0.03 \text{ d} \\ 0.14 \pm 0.00 \text{ d} \\ 0.52 \pm 0.01 \text{ d} \\ 1.38 \pm 0.01 \text{ d} \\ 0.40 \pm 0.00 \text{ d} \\ 4.04 \pm 0.03 \text{ d} \\ 0.09 \pm 0.00 \text{ d} \\ 0.07 \pm 0.00 \text{ d} \end{array}$	$\begin{array}{c} 0.30 \pm 0.03 \ c\\ 8.43 \pm 0.05 \ c\\ 0.30 \pm 0.00 \ c\\ 1.15 \pm 0.03 \ c\\ 3.03 \pm 0.02 \ c\\ 0.87 \pm 0.04 \ c\\ 8.88 \pm 0.20 \ c\\ 0.15 \pm 0.03 \ c\\ 0.13 \pm 0.02 \ c \end{array}$	$\begin{array}{c} 0.53 \pm 0.02 \text{ b} \\ 13.02 \pm 0.40 \text{ b} \\ 0.49 \pm 0.02 \text{ b} \\ 1.80 \pm 0.03 \text{ b} \\ 4.50 \pm 0.04 \text{ b} \\ 1.40 \pm 0.03 \text{ b} \\ 13.55 \pm 0.20 \text{ b} \\ 0.19 \pm 0.04 \text{ b} \\ 0.18 \pm 0.03 \text{ b} \end{array}$

<sup>*a*</sup> Residue from erva-mate (*l. paraguariensis*) processing. <sup>*b*</sup> Mate powder amount in mate candies (%). Mean values  $\pm$  SD of triplicate determinations. Mean values in the same line followed by different letters are significantly different ( $p \le 0.05$ ).

Mate powder is also a source of total ash, which reflects on the mineral content shown in **Table 2**. The total ash content of the mate powder sample is similar to those found in previous papers for *I. paraguariensis*, which are 5.07-6.60% dm (9, 24).

Supplementation of candies with mate powder at different levels significantly ( $p \le 0.05$ ) increased the total ash, lipid, total fiber, and protein contents, whereas carbohydrate and energy contents decreased, as can be seen in **Table 1**.

The total dietary fiber value of mate candies ranged from 5.7 to 7.29% dm. These levels were within the range reported for high-fiber foods (25-27). In contrast to the consumption of traditional candies (28), the consumption of mate candies would contribute to the daily intake of dietary fiber.

The relationship to inorganic elements was verified through the presence of nine elements: Na, K, Ca, Mg, Fe, Al, P, Mn, and Zn, which are considered to be essential for the metabolism of living organisms (30) (**Table 2**). Mineral elements are important for growth, development, and preservation of body tissues as well as for performing specific functions in the human body; the importance of their inclusion in diet has been discussed in nutrition reviews (29).

The minerals found in highest quantity in mate powder were Ca and K. Actually, these elements have been found in large amounts in mate (31, 32). These minerals are essential for human nutrition; they play important roles in the formation of bones, teeth, and tissues in the human body (35).

The values for Zn and Mg found in mate powder, 7.0 and 314 mg 100 g<sup>-1</sup> dm, respectively, serve as essential activators in a series of metabolic reactions catalyzed by enzymes (34) and are, therefore, very important elements for reproduction and growth (35). The Zn content observed in mate powder appears to be much higher than the Zn levels observed in other teas

 Table 3. Total Polyphenol Content and Antioxidant Capacity of Mate

 Powder<sup>a</sup> and Candies Containing Mate Powder (Dry Basis)

		type of candy			
	mate powder	0.4 <sup>b</sup>	0.8 <sup>b</sup>	1.2 <sup>b</sup>	
TPC (g/100 g)	$11.51 \pm 1.02 \text{ a}$	$0.12\pm0.00~\text{d}$	$0.18\pm0.00~\text{c}$	$0.27\pm0.01~\text{b}$	
ABTS <sup>c</sup>	$371.58 \pm 4.18 \text{ a}$	$4.05\pm0.87~\text{d}$	$6.31\pm0.64~\text{c}$	$7.58\pm0.96~\text{b}$	

<sup>*a*</sup> Residue from erva-mate (*l. paraguariensis*) processing. <sup>*b*</sup> Mate powder amount in mate candies (%). Mean values  $\pm$  SD of triplicate determinations. Mean values in the same line followed by different letters are significantly different ( $p \le 0.05$ ). <sup>*c*</sup> Scavenging capacity at 7 min ( $\mu$ M Trolox/g).

such as green, black, and oolong teas (36) and similar to the levels observed in mate leaves (32).

The Fe found in mate powder, 31 mg 100 g<sup>-1</sup> dm, is an important micronutrient for human nutrition. Fe has been largely used in food fortification programs in Brazil due to the high rates of anemia (37).

The value of 10.8 mg 100 g<sup>-1</sup> dm found for Na is higher than the values found in commercial mate (31). This mineral regularizes muscular system function and heartbeat when found in association with potassium (33).

The levels of fortification with the mate powder assayed caused a significant ( $p \le 0.05$ ) increase in mineral content of the mate candies, mainly K (**Table 2**). Therefore, mate powder appears to be an interesting source of minerals and trace elements.

Despite the high content of minerals in the mate powder, this does not mean that all of the minerals present are available to the consumer, because the existence of antinutritional factors and poor digestibility can affect their availability.

**Total Polyphenol Content (TPC) and Antioxidant Activity.** The TPC of mate powder and mate candies and their free radical scavenging capacities are shown in **Table 3**. Mate powder showed higher contents of total polyphenol (11.51% dm) than those reported for commercial erva-mate analyzed by other authors, 10.71% dm (*38*) and 6.21% dm (*6*).

A large quantity of the solids found in mate infusions is made up of polyphenolic compounds, as determined by the Folin—Cicalteu method. This fraction is expected to be composed mainly of phenolic acids, such as chlorogenic and caffeic acids (39), which are effective antioxidants (5). Antioxidant properties are found in commonly used beverages (mate, green tea, black teas; red, rosé, and white wines; and orange juice) and are positively correlated with the TPC found in dried mate leaves. Studies have suggested the role of phenolic compounds as the major source of natural antioxidants in foods of plant origin (40).

 Table 4. Contents of Caffeine and Theobromine (Milligrams per Kilogram)

 in Mate Powder<sup>a</sup> and Candies Containing Mate Powder (Dry Basis)

		type of candy			
	mate powder	0.4 <sup>b</sup>	0.4 <sup>b</sup>	0.4 <sup>c</sup>	
caffeine theobromine	$10130 \pm 40  ext{ a} \\ 1000 \pm 21  ext{ a}$	$\begin{array}{c} 50\pm2.4\text{ d}\\ 4\pm0.5\text{ d} \end{array}$	$\begin{array}{c} 90\pm3.5~\text{c}\\ 7\pm0.2~\text{c} \end{array}$	$\begin{array}{c} 180\pm5\text{b}\\ 10\pm0.4\text{b} \end{array}$	

<sup>*a*</sup> Residue from erva-mate (*l. paraguariensis*) processing. <sup>*b*</sup> Mate powder amount in mate candies (%). Mean values in the same line followed by different letters are significantly different ( $p \le 0.05$ ).

Antioxidant properties of mate powder and mate candies were evaluated on the basis of measuring the scavenging activity for ABTS radical cations by methanol extracts. All samples showed significant scavenging activities against ABTS radical.

The ABTS has a relatively stable blue-green color, which is measured at 600-700 nm. In the presence of an antioxidant such as Trolox or potential antioxidants in material extracts, the color production will be suppressed to a certain extent, in proportion to the concentration of antioxidants (41).

Addition of mate powder caused a significant increase ( $p \le 0.05$ ) in polyphenol content and also in the antioxidant capacity of mate candies (**Table 3**).

The higher polyphenolic content of the extracts from mate candies resulted in an increased antioxidant capacity. The mate candies containing 0.4% of the mate powder showed the lowest polyphenolic content and antioxidant capacity among the three formulations due to the lower concentration of mate powder in the candies.

Significant correlations were observed between TPC and ABTS scavenging activity (r = 0.926), indicating the role of polyphenolic compounds in inhibiting free radicals and radical cations under these systems. The results suggest that the polyphenolic compounds in these samples may be able to scavenge free radicals formed in biological systems.

**Methylxanthines.** Quantitative data for methylxanthines are shown in **Table 4**. Theophylline was not detected in the sample, whereas caffeine and theobromine were detected, which is in accordance with results on erva-mate reported in the literature (4, 15, 42). The methylxanthine values determined for mate powder were 1.01% dm for caffeine and 0.10% dm for theobromine. These levels were within the range reported for leaves of the *I. paraguariensis* varieties (43). The total methylxanthine contents (1.11% dm) are similar to those found in previous papers (1.10–1.85% dm) for *I. paraguariensis* leaves (44).

In this study, the caffeine content of the mate powder was higher than that determined by Jackes et al. (45) in mate tea leaves and was lower than that found by Monteiro and Trugo (46) in coffee. It is well-known that the parameters involved in the extraction procedure, such as size of sample and brewing conditions and interference in the solubility of bioactive compounds, as well as growing conditions and genetic characteristics, highly influence the content of such substances in plants (45, 47).

The addition of mate powder caused significant increases in the methylxanthine contents of the mate candies ( $p \le 0.05$ ) (**Table 4**), which means there is the opportunity of consuming functional compounds with these candies.

**Physical Characteristics of the Mate Candies. Table 5** shows that hardness values were dependent on candy solids contents. The amount of mate powder added into candies increased significantly ( $p \le 0.05$ ) yield and hardness values. It was presumed that the higher amount of solid content (mate powder) increased the friction force inside candies due to a more rigid microstructure of the matrix.

Table 5. Physical and Textural Characteristics of Candies Containing Mate ${\rm Powder}^a$ 

		type of candy				
parameter	0.4 <sup>b</sup>	0.4 <sup>b</sup>	0.4 <sup>b</sup>			
yield (%) hardness (g)	$\begin{array}{c} 70 \pm 0.7 \text{ c} \\ 263.55 \pm 24.20 \text{ c} \end{array}$	$72 \pm 0.8 \text{ b} \\ 359.54 \pm 23.47 \text{ b}$	$74 \pm 0.9  ext{ a}$ 420.11 $\pm$ 25.90 $ ext{ a}$			

<sup>*a*</sup> Residue from erva-mate (*l. paraguariensis*) processing. <sup>*b*</sup> Mate powder amount in mate candies (%). Mean values  $\pm$  SD of triplicate determinations. Mean values in the same line followed by different letters are significantly different ( $p \le 0.05$ ).

Table 6. Color Characteristics of Candies Containing Mate Powder<sup>a</sup>

	type of candy				
parameter	0.4 <sup>b</sup>	0.4 <sup>b</sup>	0.4 <sup>b</sup>		
L*	$28.57 \pm 0.54$ a	$29.17 \pm 0.57$ a	$28.52 \pm 0.64$ a		
a*	$-0.07 \pm 0.05$ a	$-0.13\pm0.03$ b	$-0.24\pm00~{ m c}$		
b*	$7.4\pm0.39~\mathrm{a}$	$7.47\pm0.23$ a	$7.36\pm0.29\mathrm{a}$		
chroma (C*)	$7.32\pm0.41~\mathrm{a}$	$7.13\pm0.38~\mathrm{a}$	$7.44\pm0.28$ a		
hue angle (h)	$91.44 \pm 2.44$ a	$91.70 \pm 2.15$ a	91.21 $\pm$ 1.33 a		

<sup>*a*</sup> Residue from erva-mate (*l. paraguariensis*) processing. <sup>*b*</sup> Mate powder amount in mate candies (%). Mean values  $\pm$  SD of triplicate determinations. Mean values in the same line followed by different letters are significantly different ( $p \le 0.05$ ).

 Table 7. Mean Sensory Rating for Consumer Acceptance and Purchase

 Intent for Candies Containing Mate Powder<sup>a</sup> (Dry Basis)

		purchase intent scale <sup>b</sup> (%)				
type of candy	overall acceptability <sup>c</sup>	1	2	3	4	5
0.4 <sup>d</sup>	4.31 a	0.00	2.61	23.48	46.09	27.83
0.8 <sup>d</sup>	4.13 a	2.61	5.28	26.09	38.26	27.83
1.2 <sup>d</sup>	4.28 a	0.00	4.35	30.43	35.65	29.57

<sup>*a*</sup> Residue from erva-mate (*I. paraguariensis*) processing. <sup>*b*</sup> Purchase intent scores: 5 = definitely would buy, 4 = probably would buy, 3 = maybe would buy/maybe would not buy; 2 = probably would not buy; 1 = definitely would not buy. Mean values in the same column followed by different letters are significantly different ( $p \le 0.05$ ). Mean of 100 judgments. <sup>*c*</sup> A five-point hedonic scale with 1 = dislike extremely, 3 = neither like nor dislike, and 5 = like extremely was used. <sup>*d*</sup> Mate powder amount in mate candies (%).

The hardness values of the mate candies were higher than those determined by Izzo et al. (48), who obtained values of 200 g in candies formulated with cellulose gellan, and were lower than those found by Fadini et al. (49) and by Garcia and Penteado (50), who obtained values ranging from 1000 to 20000 g for candies prepared with and without gellan gum and from 1400 to 1800 g for gelatin candies fortified with vitamins, respectively.

The Hunter parameters  $L^*$ ,  $a^*$ ,  $b^*$ ,  $C^*$ , and h of mate candies are shown in **Table 6**. The color of the candies is one of the characteristics first perceived by the consumer and affects the acceptability of the product. None of the mate candies showed differences in  $L^*$ ,  $C^*$ , h, and  $b^*$  parameters. The mate candies showed significant difference ( $p \le 0.05$ ) in relation to the color green ( $a^*$ ), showing higher green coloration according to incorporation of mate powder. All of the formulations produced mate candies with hue angles around 90, indicating that their color was more green (h = 88-111) than brown (h = 40-75) or yellow (h = 64).

**Consumer Acceptance and Purchase Intent of Mate Candies.** The results of overall acceptability and purchase intent are shown in **Table 7**. The three formulations of mate candies showed acceptability scores higher than the minimum acceptable score, that is, 3 (neither like nor dislike), and all of the mate candies were rated with "definitely would buy" and "probably would buy" scores, confirming the overall acceptability results.

### Characteristics of a New Erva-Mate Candy

In conclusion, this study shows that mate powder contains high levels of minerals, total polyphenol, and total dietary fiber, with a predominance of insoluble dietary fiber content. Candies supplemented with mate power at 0.4-1.2% levels produced acceptable mate candies in relation to the overall acceptability and purchase intent, with improved nutritional values. Therefore, residues from erva-mate could be used as a supplement in human diet and as an ingredient with functional components in formulated foods such as mate candies, adding value to residues generated in the erva-mate processing. Further studies could investigate strategies to improve the sensory quality of candies containing high levels of mate powder.

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# LITERATURE CITED

- Filip, R.; Lopez, P.; Giberti, G.; Coussio, J.; Ferraro, G. Phenolic compounds in seven South American *Ilex* species. *Fitoterapia* 2001, 72, 774–778.
- (2) Souza, V. C.; Lorenzi, H. Botânica Sistemática: Guia Ilustrado, Baseado em APG II; Instituto Plantarum: Nova Odessa, SP, 2005.
- (3) Saldaña, M. D. A.; Mazzafera, P.; Mohamed, R. S. Extraction of purine alkaloids from maté (*Ilex paraguariensis*) using supercritical CO2. *J. Agric. Food Chem.* **1999**, *47*, 3804–3808.
- (4) Saldaña, M. D. A.; Zetzl, C.; Mohamed, R. S.; Brunner, G. Extraction of methylxanthines from guaraná seeds, mate leaves, and cocoa beans using supercritical dioxide and ethanol. <u>J. Agric. Food Chem.</u> 2002, 50, 4820–4826.
- (5) Bravo, L.; Goya, L.; Lecumberri, E. LC/MS characterization of phenolic constituents of mate (*Ilex paraguariensis*, St. Hil.) and its antioxidant activity compared to commonly consumed beverages. *Food Res. Int.* 2007, 40, 393–405.
- (6) Deladino, L.; Anbinder, P. S.; Navarro, A. S.; Martino, M. N. Encapsulation of natural antioxidants extracted from *Ilex para-guariensis*. <u>*Carbohvdr. Polym.*</u> 2007, *71*, 126–134.
- (7) Schinel, A,G.; Fantinellib, J. C.; Mosca, S. M. Cardioprotective effects of *Ilex paraguariensis* extract: evidence for a nitric oxide-dependent mechanism. *Clin. Nutr.* 2005, *24*, 360–366.
  (8) Mendes, R. F.; Carlini, E. A. Brazilian plants as possible
- (8) Mendes, R. F.; Carlini, E. A. Brazilian plants as possible adaptogens: an ethnopharmacological survey of books edited in Brazil. *J. Ethnopharmacol.* 2007, 109, 493–500.
- (9) Esmelindro, M. C.; Toniazzo, G.; Waczuk, A.; Dariva, C.; Oliveira, D. Caracterização físico-química da erva-mate: influência das etapas do processamento industrial. <u>*Cienc. Tecnol. Aliment.*</u> (*Campinas, Braz.*) 2002, 22, 199–204.
- (10) Laufenberg, G.; Kunz, B.; Nystroem, M. Transformation of vegetable waste into value added products: (A) the upgrading concept; (B) practical implementation. *Bioresour. Technol.* 2003, 87, 167–198.
- (11) *Official Methods of Analysis*, 18th ed.; Association of Official Analytical Chemists: Gaithersburg, MD, 2005.
- (12) Watt, B.; Merrill, A. L., Composition of Foods: Raw, Processed, Prepared; USDA Nutrient Data Laboratory: Bethesda, MD, 1999.
- (13) Budini, R.; Tonelli, D.; Girotti, S. Analysis of total polyphenols using the Prussian blue method. J. Agric. Food Chem. 1980, 50, 3698–3703.
- (14) Re, R.; Pellegrini, N.; Proteggente, A.; Pannala, A.; Yang, M.; Rice-Evans, C. Antioxidant activity applying improved ABTS radical cation decolorization assay. *Free Radical Biol. Med.* 1999, 26, 1231–1237.
- (15) Reginatto, F. H.; Athayde, M. L.; Gosmann, G.; Schenkel, E. P. Methylxanthines accumulation in *Ilex* species—caffeine and theobromine in erva-mate (*Ilex paraguariensis*) and other *Ilex* species. *J. Braz. Chem. Soc.* **1999**, *10*, 443–446.
- (16) Robb, C. S.; Geldart, S. E.; Seelenbinder, J. A.; Brown, P. R. Analysis of green tea constituents by HPLC-FTIR. <u>J. Lia.</u> <u>Chromatogr. Relat. Technol.</u> 2002, 25, 787–801.

- (17) Bourne, M. Food Texture and Viscosity. Concept and Measurements; Academic Press: New York, 2002.
- (18) Meilgaard, M.; Civille, G. V.; Carr, B. T. Sensory Evaluation Techniques, 4th ed.;, CRC Press: Boca Raton, FL, 2007; pp 448.
- (19) Sudha, M. L.; Vetrimani, R.; Leelavathi. Influence of fiber from different cereals on the rheological characteristics of wheat flour dough and on biscuit quality. *Food Chem.* **2006**, *100*, 1365–1370.
- (20) Energy and Protein Requirements. Nutrition Meeting Report Series 51; Food and Agriculture Organization: Rome, Italy, 1973. *Technical Report Series* 522; World Health Organization: Geneva, Switzerland, 1973.
- (21) Rodríguez, R.; Jiménez, A.; Fernández-Bolaños, J.; Guillén, R.; Heredia, A. Dietary fiber from vegetable products as source of functional ingredients. <u>*Trends Food Sci. Technol.*</u> 2006, 17, 3– 15.
- (22) Nelson, A. L. Properties of high-fiber ingredients. <u>Cereal Foods</u> <u>World</u> 2001, 46, 93–97.
- (23) Mälkki, Y. Physical properties of dietary fiber as keys to physiological functions. <u>*Cereal Foods World*</u> 2001, 46, 196–199.
- (24) Gutkoski, L. C.; Schulz, J. G.; Sampaio, M. B.; Silva, D. da R. Avaliação de parâmetros físicos e químicos de marcas de ervamate processada em diferentes épocas. *B. Ceppa* **2001**, *19*, 95– 104.
- (25) Silva, M. R.; Silva, M. S.; Martins, K. A.; Borges, S. Utilização tecnológica dos frutos de jatobá-do-cerrado e de jatobá-da-mata na elaboração de biscoitos fontes de fibra alimentar e isentos de açúcares. *Cienc. Tecnol. Aliment. (Campinas, Braz.)* 2001, 21, 176–182.
- (26) Krüger, C. C. H.; Comassetto, M. C. G.; Candido, L. M. B.; Baldini, V. L. S.; Santtucci, M. C.; Sgarbieri, V. C. Biscoito tipo "cookie" e "snack" enriquecidos, respectivamente com caseína obtida por coagulação enzimática e caseinato de sódio. <u>Cienc.</u> <u>Tecnol. Aliment. (Campinas, Braz.)</u> 2003, 23, 81–86.
- (27) Vieira, M. A.; Tramonte, K. C.; Podestá, R.; Avancini, S. R. P.; Amboni, R. D. M. C.; Amante, E. R. Physicochemical and sensory characteristics of cookies containing residue from king palm (*Archontophoenix alexandrae*) processing. *Int. J. Food Sci. Technol.* 2008, doi 10.1111/j.1365-2621.2007.01568.x.
- (28) Gebhardt, S. E.; Thomas, R. G. *Nutritive Value of Foods*; Home and Garden 72; U.S. Department of Agriculture: Bethesda, MD, 2002; pp 70–76.
- (29) Velasquez-Melendez, G.; Salas Martins, I.; Cyerbato, A. M. Consumo alimentar de vitaminas e minerais em adultos residentes em área metropolitana de São Paulo, Brasil. <u>*Rev. Saude Publ.*</u> **1997**, *31* (2), 157–162.
- (30) De Angels, R. C. Fisiologia da nutrição: fundamentos para nutrição e para desnutrição. *EDASRT* **1977**, 2, 55–76.
- (31) Heinrichs, R.; Malavolta, E. Mineral composition of a commercial product from mate-herb (*Ilex paraguariensis* A. St. Hil.). <u>*Cienc.*</u> <u>*Rural*</u> 2001, 31, 781–785.
- (32) Giulian, R.; Santos, C. E. I.; Shubeita, S. de M.; Silva, de L. M.; Dias, J. F.; Yoneama, M. L. Elemental characterization of commercial mat tea leaves (*Ilex paraguariensis* A. St.-Hi.) before and after hot water infusion using ion beam techniques. <u>J. Agric. Food Chem.</u> 2007, 55, 741–746.
- (33) Franco, L. Tabela de Composição de Alimentos, 9th ed.; Atheneu: São Paulo, Brazil, 1998.
- (34) Belitz, H. D.; Grosch, W.; Schieberle, P. In *Food Chemistry*, 3rd ed.; Springer-Verlag: Berlin, Germany, 2004.
- (35) Cozzolino, S. M. F. *Biodisponibilidade de Nutrientes*; Barueri: Sao Paulo, Brazil, 2005; pp 878.
- (36) Cabrera, C.; Giménez, R.; López, M. C. Determination of tea components with antioxidant activity. *J. Agric. Food Chem.* 2003, 51, 4427–4435.
- (37) Torres, M. A. A.; Lobo, N. F.; Sato, K.; Queiroz, S. S. Fortificação do leite fluido na prevenção e tratamento da anemia carência ferropriva em crianças menores de 4 anos. <u>*Rev. Saude Publ.*</u> 1996, 30, 350–357.
- (38) Anesini, C.; Ferraro, G.; Filip, R. Peroxidase-like activity of *Ilex paraguariensis*. *Food Chem.* 2006, *97*, 459–464.

- (39) Carini, M.; Facino, R. M.; Aldini, M.; Calloni, M.; Colombo, L. Characterization of phenolic antioxidants from mate (*Ilex paraguariensis*) by liquid chromatography/mass spectrometry and liquid chromatography/tanden mass spectrometry. <u>Rapid Commun. Mass Spectrom.</u> 1998, 12, 1813–1819.
- (40) Hagerman, A. E.; Riedl, K. M.; Jones, G. A.; Sovik, K. N.; Ritchard, N. T.; Hartzfeld, P. W.; Riechel, T. L. High molecular weight plant phenolics (tannins) as biological antioxidants. <u>J.</u> <u>Agric. Food Chem.</u> **1998**, *46*, 1887–1892.
- (41) Ragaee, S.; Abdel-Aal, E.-S. M.; Noaman, M. Antioxidant activity and nutrient composition of selected cereals for food use. *Food Chem.* 2006, 98, 32–38.
- (42) Gnoatto, S. C. B.; Bassani, V. L.; Coelho, G. C.; Schenkel, E. P. Influência do método de extração nos teores de metilxantinas em erva-mate (*Ilex paraguariensis* A. St.-Hil., Aquifoliaceae). <u>Ouim.</u> <u>Nova</u> 2007, 30, 304–307.
- (43) Mazzafera, P. Caffeine, theobromine and theophylline distribution in *Ilex paraguariensis*. <u>*Rev. Bras. Fis. Veg.*</u> 1994, 6, 149– 151.
- (44) Baltassat, F.; Darbour, N.; Ferry, S. Étude du contenu purique de drogues a caffeine: I. Le maté: *Ilex paraguariensis* Lamb. <u>*Plant*</u> <u>Med. Phytother</u>. **1984**, *18*, 195–203.
- (45) Jacques, R. A.; Krause, L. C.; Freitas, L. S.; Dariva, C.; Oliveira, J. V.; Caramão, E. B. Influence of drying methods and agronomic variable on the chemical composition of mate tea leaves (*Ilex paraguariensis* A. St.-Hil) obtained from high-

pressure CO<sub>2</sub> extraction. <u>J. Agric. Food Chem</u>. **2007**, 55, 10081–10085.

- (46) Montero, M. C.; Trugo, L. C. Determinação de compostos bioativos comerciais de café torrado. *Quim. Nova* 2005, 28, 637– 641.
- (47) Astill, C.; Birch, M. R.; Dacombe, C.; Humpheey, P. G.; Martin, P. T. Factors affecting the caffeine and polyphenol content of black and green tea infusions. *J. Agric. Food Chem.* 2001, 49, 5340–5347.
- (48) Izzo, M.; Stahl, C.; Tuazon, M. Using cellulose gel and carrageenan to lower fat and calories in confections. *Food Technol.* 1995, 49, 45–49.
- (49) Fadini, A. L.; Facchini, F.; Queiroz, M. B.; Anjos, V. D. de A.; Yotsuyanagi, K. Influência de diferentes ingredientes na textura de balas moles produzidas com e sem goma gelana. *B. Ceppa* 2003, 21, 131–140.
- (50) Garcia, T.; Penteado, M. V. C. Qualidade de balas de gelatina fortificadas com vitaminas A, C e E. <u>Cienc. Tecnol. Aliment.</u> (Campinas, Braz.) 2005, 25, 743–749.

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