Vol.50, n. 4 : pp. 735-741 July 2007
ISSN 1516-8913 Printed in Brazil

BRAZILIAN ARCHIVES OF
BIOLOGY AND TECHNOLOGY
AN INTERNATIONAL JOURNAL

Physicochemical Characterization of Different Trademarks of Compound Yerba Maté and their Herbs

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ABSTRACT

The objectives of this study were to evaluate the physicochemical characteristics of the main herbs used in the mixture of yerba maté with other aromatic herbs and the characterization of the trademarks of compound yerba maté. Moisture, water extract, total ash, acid-insoluble ash and caffeine concentration were determined. Results showed higher values of moisture content, total and aci-insoluble ash and lower water extracts in the herbs. Determinations were carried out in nine trademarks of compound yerba maté. In most cases they complied with the standards of the country with the exception of one trademark from Argentina.

Key words: Yerba Maté, herbs, compound, regulations, MERCOSUR

INTRODUCTION

Yerba maté (Ilex *paraguarienses* Saint Hilaire) is a product consumed in South America in the MERCOSUR region (Argentina, Brazil, Paraguay and Uruguay. Maté is the traditional way of consuming it (approximately a 97% of the production). This infusion is generally prepared with hot water, although in summer cold water may be used in warm regions (De Bernardi and Prat Kricum, 2001). The elaborated product may present different organoleptic characteristics. People in Argentina, Paraguay and Uruguay prefer a seasoned and grossy-ground yerba maté; while in Brazil, they prefer a product finely ground with no seasoning.
Recently, a product derived from yerba maté, called compound yerba maté, increased its market share. It is obtained by mixing yerba maté with other aromatic herbs. These herbs confer a flavor different from the traditional one and it has a good acceptance among many yerba maté consumers.
Food Codes or equivalent standards of MERCOSUR countries differ in compound yerba maté characterization. Argentine standards admit the addition of herbs up to 40% (in weight) (CAA, 2000); Paraguayan standards admit it up to 15% (INTN 3500201, 2002) and Brazilian standards, recently approved, do not have maximum values (Resolution RDC Nº 277,2005). Chilean and Uruguayan standards do not define this kind of product (Decrete-law 287/02, 2002; Decrete-law 315/94, 1994).
Argentine standards (CAA, 2000) accept the use of recognized physiologycal inocuosness, mentioning seven of them with their popular name and also admit the use of others that the National Health Authority could approve. Disposition 1637/2001 (ANMAT, 2001) mentions 35 herbs and their parts that can be consumed. Brazilian standards admit the addition of 46 herbs and fruits

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mentioning the scientific and popular names and their parts that can be consumed (Resolution RDC Nº 267, 2005). Paraguayan standards admit the addition of herbs of "recognized physiological innocuousness" that have to be authorized by the Health Authority (INTN 3500221, 2000).

Paraguayan standards are the most complete in defining general requirements mentioning organoleptics, physicochemical and microbiological parameters (Table 1). Physicochemical parameters are similar to those of yerba mate, but microbiological parameters are more permissive (INTN 3500193, 1995). Argentine standards require maximum values for moisture content (9.5% in dry basis) and acid-insoluble ash (2% in dry basis). Required values for yerba maté are 9.5 and 1.5%, respectively and Brazilian standards do not have requirements.

Several papers on physicochemical characterization of yerba maté have been carried out (Schmalko et al.,1995; Ramallo et al.,1998), but only a few of them deal with the physicochemical characteristics of herbs used in compound yerba maté. In the Brazilian decree-law 519 (1998), characteristics of some herbs used in infusion preparation (as tea for example) can be found. It is very important to know the physicochemical characteristics of herbs used in compound yerba maté. From these, some standards can be modified, especially Argentine ones, which allow the addition of herbs up to a 40%.

Yerba maté moisture sorption isotherms were determined at its different processing steps (Kanzig et al.,1985; Kanzig et al.,1987). From these, maximum moisture content to avoid microbial growth can be determined, resulting in 9.5%, on dry basis. Scipioni et al. (2002, 2003), determined moisture adsorptions isotherms for five herbs. From these isotherms, the maximum moisture content to avoid microbial growth can be determined.

The aim of this work was to study the compound yerba maté in order to determine its physicochemical characteristics for its standardization. To reach this general objective, the following specific ones must be achieved:

a) The determination of herbs physicochemical characteristics
b) Chromatographic method (HPLC) for caffeine determination must be validated in compound yerba maté
c) The determination of physicochemical parameters in trademarks of compound yerba maté.

MATERIALS AND METHODS

Materials

Herbs

Herbs were selected according to the composition mentioned on the package labels of trademarks of compound yerba maté. Only the most commonly used herbs were analyzed. Samples were obtained from a company which elaborated compound yerba maté. They were: *Aloysa triphylla* L., *Lippia integrifolia* (G.) H., *Lippia turbinata* G., *Matricaria chamomilla*, *Mentha piperita* L., *Mentha pulegium* L., *Minthostachys mollis* H.B.K:, *Peumus boldea* M., *Tilia cordata* M**.**/*T. platyphyllos* S., and the yerba maté (*Ilex paraguariensi*s) used in this factory.

Compound yerba maté

Nine samples of trademarks were analyzed. They were purchased from the markets in Posadas (Argentina), Encarnación (Paraguay) and Camboriú (Brazil) in March 2005. Declared composition on labels is shown in table 2 with scientific names. Table 3 shows scientific and popular names of these herbs.

Methods

Moisture content

Moisture content was determined by measuring the loss in mass at 103 ± 2ºC for 6 h (IRAM 20503, 1995).

Total and acid-insoluble ash

Two grams of sample were calcined in a porcelain capsule and put into a furnace for 5 h. Then, the material was cooled and weighed in order to determine total ash (IRAM 20505, 1996). This material was dissolved in a solution of HCl (10% v/v) and heated until boiling point and maintained for 1 h. The mixture was filtered using a ash free paper; the residue was washed with water, put into a porcelain capsule and heated in a furnace for 5 h. Then, the material was cooled and weighed in order to determine acid-insoluble ash (IRAM 20507, 2003).

Table 1 - Requirements for compound yerba maté in Paraguay

Organoleptic parameters		Observations			
Odor		Characteristic			
Color		Characteristic			
Flavor		Characteristic			
Physicochemical parameters		**Percentual values (%)**			
		Minimum		**Maximum**	
Moisture at 105ºC±0.5ºC		-		10.3	
Ash at 550 ºC		-		9.00	
Insoluble ash in HCl at 10 % at 1000ºC		-		1.50	
Water extract		25		-	
Caffeine		0.6		-	
Type	Dried and powdered yerba maté leaves	65.0		-	
	Branches, twigs, herbs and other herbs	-		35.0	
Impurity		-		1	
Yerba maté seeds		-		1	
Microbiological parameters					
Microorganisms		**Referent values**			
		n	**c**	**m**	**M**
Molds and yeast CFU/g		5	2	3.500	5000
Total coliforms MPN/g		5	2	90	200
Coliforms MPN/g at 44 ± 1ºC		5	2	7	15
Escherichia coli/g		5	0	0	0
Salmonella sp25/g		5	0	0	0

n= number of analyzed samples; c= unity number that presents values between m-M; m= minimum acceptable value; M= maximum value; CFU= colony former unities; MPN= most probable number

Table 2 - Composition declared in the package labels of compound yerba maté

Trademark	Composition
1 (Argentina)	Yerba maté: 50%, *Peumus b.*: 15%, *Mentha piperita*: 13% y *Cruces p.*: 7%
2 (Argentina)	Yerba maté, *Lippia t.*, *Rosa c.*, *Foeniculum v.*, *Chlorella v.*, *Mentha piperita*, *Aloysa t.*, *Peumus b.*, apple flavour
3 (Argentina)	Yerba maté: 95%, *Minthostachys m.*: 1%, *Lippia t.*: 1%, *Lippia i.*: 1%, *Peumus b.*: 1%, *Mentha piperita*: 0.375%, *Aloysa t.*: 0.375%, *Tilia c.*: 0.25%
4 (Argentina)	Yerba maté: 95%, *Mentha piperita .*: 1.5 %, *Lippia t.*: 1.5%, *Minthostachys m.*: 1%, *Melissa o.*: 1%
5 (Argentina)	Yerba maté: 81%, *Camelia s.* : 4%, *Chlorella v.*: 3%, *Aloysa t.*: 3%, *Equisetum a.*: 3%; *Hyssopus o.*: 3%; *Minthostachys m.*: 3%.
6 (Brazil)	Yerba maté: 90%, other plant species: 10% (*Citrus a.*, *Aloysa t.*, *Matricaria r.*, *Mentha piperita.*).
7 (Brazil)	Yerba maté: 90%, other plant species: 10% (2.33% *Matricaria r.*: 2.33%, *Melissa o.*: 2.33%, *Foemiculum v.*: 2.33%, sucrose: 3%.
8 (Paraguay)	Yerba maté, *Mentha piperita*, *Aloysa p*, *Aloysa t.*
9 (Paraguay)	Yerba maté: 91.7%, *Peumus b.*: 8%, essential oil of *Mentha piperita*: 0.3%.

Table 3 - Scientific and popular names of herbs used in compound yerba maté

Aloysa triphylla **L.: cedrón**	***Foeniculum vulgare*****: hinojo**	***Minthostachys mollis*** **H.B.K:: peperina**
Aloysa polystachya: burrito	*Hyssopus officinalis* L.: hisopo	*Pimpinella anisum* L: erva duce
Baccharis articulata: carqueja	*Ilex paraguarienses*: yerba maté o erva mate	*Peumus boldea* M..: boldo
Camelia sinensis: té verde	*Lippia turbinata* G.: poleo	*Rosa caninal* L.: rosa mosqueta
Citrus aurantium: naranja, laranja	*Matricaria recutita*: manzanilla o camomila	*Stevia rebaudiana Bertoni*: kaa-heé
Cruces peaedictus L.: cardo santo	*Melissa officitrals* L: melisa o cidreira	*Tilia cordata* M**.**/*T. platyphyllos* S.: tilo
Chlorella vulgaris: algas marinas, fucus	*Mentha piperita* L.: menta, hortella	*Lippia integrifolia* (G.) H.: incayuyo
Equisetum arvense L.: cola de caballo	*Mentha pulegium* L.: poleo fuerte	

Caffeine

In order to determine caffeine content a HPLC method, reported in IRAM 20512 (2003) was used. One gram of sample was mixed with 300 mL of water and 4.5g of MgO and they were put into a water-bath at 90ºC with agitation for 1 h. Then, the mixture was filtered and the liquid was injected in a liquid chromatograph KNK (Konic instrument, Spain) with a column Hewlett-Packard Lichospher 100, RP-18 (5 μm). Operation conditions were the following: mobile phase: acetonitrile/water (30:70 in volume); flux: 1 mL/min; detector: UV-visible at 272 nm.

In all cases, experiments were carried out in triplicate and the average and standard deviation were reported.

RESULTS AND DISCUSSION

Herbs

The different values of moisture, water extract, total ash and acid-insoluble ash content are shown in Table 4. As seen, a wide range of values were found for the different herbs and yerba maté. Water extracts were low in *Minthostachys m.* and *Lippia i.*; while the values found in the other herbs were similar to those found in different trademarks of yerba maté (Schmalko et al., 1995). Nevertheless, they were lower than those of yerba maté analyzed in this study. In many herbs (*Peumus b.*, *Aloysa t.*, *Mentha piperita.*, *Lippia t.* y *Mentha p.*), total ash content was higher than the maximum content admitted in yerba maté. Similar results were obtained with acid-soluble ash in *Peumus b.*, *Lippia i.*, *Mentha piperita.*, *Lippia t.* y *Mentha p.*. In *Peumus b.* and *Limpia t.*, the values obtained were three times higher than admitted in yerba maté. Therefore, maximum or minimum admitted values of these parameters for compound yerba mate should be modified, mainly in Argentina where a high percentage of herbs is added to yerba maté and Brazil, where there is no limit.

Caffeine

In order to test the application of the HPLC method for caffeine determination in compound yerba maté, caffeine concentration was determined in different mixtures of herbs-yerba maté. First, five determinations of caffeine content in yerba maté were carried out, resulting in an average value of 0.823 % (dry basis) and a standard deviation of 0.037 %. Then, caffeine concentration was determined in three mixtures of herbs-yerba maté: 10, 25 and 40%. These percentages include all mixtures shown in Table 2. The experimental values were fitted to a linear equation, resulting in:

$$Y = 0.844 - 0.0069 * X$$

where Y was the caffeine percentage (in dry basis) and X was the herb percentage. The fit was good, with a P value < 10^{-5}. As seen in Fig. 1, the intercept of the straight line had a value similar to yerba maté caffeine content (0.823%). According to the good fit obtained and the chromatograph observation, it was concluded that herbs did not produce interferences in the technique reported in IRAM standard 20512 (2003).

Table 4 - Moisture, water extract, total ash and acid-insoluble ash content (in %, dry basis) of the different herbs. Mean value ± Standard deviation of 3 measures.

Herbs	Moisture content	Water extract	Total ash	Acid-insoluble ash
Aloysa t.	12.60 ± 0.063	32.47 ± 0.942	10.48 ± 0.228	0.87 ± 0.140
Lippia t.	13.10 ± 0.079	27.66 ± 0.491	15.54 ± 0.935	5.12 ± 0.642
Matricaria r.	12.19 ± 0.174	26.59 ± 0.394	8.64 ± 0.202	0.94 ± 0.,087
Mentha piperita	13.82 ± 0.185	32.94 ± 1.004	13.73 ± 0.572	1.72 ± 0.520
Mentha p.	11.04 ± 0.055	28.29 ± 0.727	15.34 ± 0.297	1.98 ± 0.346
Minthostachys m.	11.78 ± 0.140	13.58 ± 0.919	5.08 ± 0.303	0.50 ± 0.130
Peumus b.	11.55 ± 0.137	35.38 ± 0.297	9.99 ± 0.043	5.53 ± 0.051
Tilia c.	14.64 ± 0.223	29.37 ± 0.380	8.53 ± 0.235	0.81 ± 0.231
Lippia i.	11.48 ± 0.015	21.13 ± 0.813	5.58 ± 1.022	1.63 ± 0.647
Yerba Maté	6.10 ± 0.100	38.02 ± 0.828	6.37 ± 0.360	0.42 ± 0.060

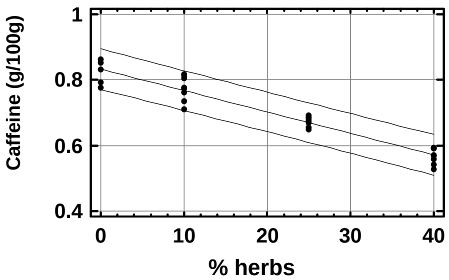

Figure 1 - Caffeine content (in g/100g of dry solid) as a function of % of herbs. Experimental values and fitted line

Compound yerba maté

Values of moisture, water extract, total ash, acid-insoluble ash and caffeine content for the different samples of compound yerba maté are shown in Table 5. As seen, trademarks 1, 2 and 5 had higher values of moisture content than the maximum admitted in the respective country standard. This was probably due to the fact that the herbs used had higher values of moisture content (Table 4) than yerba maté. It was observed in their adsorption isotherms that the moisture content corresponding to a water activity of 0.6, limit value for microbial growth (Rahman, 2005), was higher in many cases (*Minthostachys m.*, *Lippia i.*, *Matricaria r.*, *Lippia t.*, *Mentha piperita*) than the value of 9.5% admitted in the Argentine Food Code, and the value of 10.3% admitted in Paraguayan standards. In other two cases (*Baccharis a.*, *Stevia r.*) moisture content was lower than these two values (9.5% and 10.3%). In mixtures with herbs that had higher equilibrium moisture content than yerba maté, these two values could be used as the maximum admitted value. In mixtures with lower values of equilibrium moisture content than yerba maté, a lower maximum admitted value should be used, mainly in Argentina, where the percentage of herbs admitted in compound yerba maté was high.

Values of water extracts were higher in most of the samples than the value of 25% admitted in compound yerba maté in the Paraguayan standards or in yerba maté without mixtures. Trademark 1 was the exception. This had a high percentage of herbs, but it contained mainly *Peumus b.* and *Mentha piperita*, which had a water extract higher than 25% (Tables 2 and 4). Low values of water extract could be caused by a high percentage of twigs.

All samples had lower values of total ash than the values admitted in Paraguayan standards for compound yerba maté and Argentine and Brazilian standards for yerba maté, although the herbs, in most cases, had higher values (Table 4). Acid-soluble ash of trademark 1 (Table 5) was higher than the admitted value in the Argentine and Paraguayan standards. This was probably due to the fact that trademark 1 had included in its mixture *Mentha piperita* and *Peumus b.* (table 2), which had a high acid-insoluble content (table 4).

In all mixtures, yerba maté was the only compound having caffeine. Because of this, caffeine content should be related to the percentage used in the mixture. In Argentina and Paraguay the minimum value admitted for caffeine was 0.6%. If an addition of 40% was admitted in Argentina, the minimum value of caffeine in compound yerba maté should be 0.36%. Paraguayan standards admit a minimum value (0.6%) for yerba maté and compound yerba maté.

Considering this product was only consumed in the MERCOSUR region, it could be very favorable for the enterprises and consumers to have similar standards in these three countries.

Table 5 - Moisture, water extract, total ash, acid-insoluble ash and caffeine content (in %, dry basis) of the different trademarks for compound yerba maté. Mean value ± Standard deviation of 3 measures.

Trade mark	Moisture content	Water extract	Total ash	Acid-insoluble ash	Caffeine
1	10.82±0.053	23.89±0.954	7.33±0.382	2.15±0.201	0.494±0.0191
2	9.93±0.051	32.02±0.545	6.20±0.310	0.30±0.057	0.492±0.0222
3	6.73±0.079	33.02±0.597	7.16±0.214	0.77±0.060	0.447±0.0130
4	8.03±0.006	34.55±0.843	6.30±0.285	0.25±0.070	0.488±0.0195
5	10.20±0.125	32.86±0.358	6.33±0.180	0.62±0.090	0.494±0.0228
6	5.41±0.150	35.99±0.734	6.97±0.186	0.71±0.075	0.910±0.036
7	8.05±0.061	34.20±0.501	5.66±0.029	0.34±0.070	0.949±0.040
8	8.78±0.146	40.01±0.337	7.51±0.194	0.72±0.135	0.789±0.029
9	9.08±0.170	37.60±0.910	7.17±0.259	0.79±0.132	1.199±0.021

RESUMO

A erva-mate composta é um produto que se consome amplamente na região do MERCOSUL. Obtém-se misturando erva-mate com outras ervas aromáticas. O objetivo desta pesquisa foi o estudo das características físico-químicas das principais ervas usadas na mistura e a caracterização das marcas de erva-mate composta. Determinou-se a umidade, extração de água, cinzas totais, cinzas insolúveis ácidas e a concentração de cafeína. Encontraram-se nas ervas, valores padrões diferentes aos da erva-mate tais como valores maiores de conteúdo de umidade, cinzas totais, cinzas insolúveis ácidas e menores extratos de água. Fizeram-se determinações em nove marcas de erva-mate composta. Na maioria dos casos, cumpriam com as normas do país, exceto uma marca da Argentina

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Received: September 19, 2005;
Revised: May 31, 2006;
Accepted: March 14, 2007.

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