

Original Research Article

Essential oil and antioxidant activity of green mate and mate tea (*Ilex paraguariensis*) infusions

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Abstract

The soluble solids content, total phenolic content (Folin–Ciocalteu method), antioxidant activity (ferric thiocyanate method) and essential oil composition of green mate and mate tea (roasted green mate) infusions were analysed. The essential oil was obtained by hydrodistillation, and the volatile compounds were separated by high resolution gas chromatography and identified by mass spectrum and Kovats index. Although the antioxidant activity of green mate and mate tea infusions were equivalent, the soluble solids content in green mate was higher (4.2%) than in mate tea infusions (3.2%). Important compounds that add flavor to plant infusions—such as linalool, present in high concentrations in the green mate essential oil—were oxidized into linalool oxides after roasting. Limonene decreased from 19.5% to 7.3%, and furfural and methylfurfural were formed.

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1. Introduction

Mate (*Ilex paraguariensis*) leaves contain many bioactive compounds, such as phenolic acids, which seem to be responsible for the antioxidant activity of green mate infusions, both in vivo and in vitro (Bracesco et al., 2003; Schinella et al., 2000; Filip et al., 2000; Gugliucci, 1996; Gugliucci and Stahl, 1995; Campos et al., 1996).

Present in minor concentrations, volatile compounds greatly influence the quality of infusion beverages made from plant parts, of which the one most studied is *Camellia sinensis*. This species is the source of black tea and green tea (Baptista et al., 1998; Kato and Shibamoto, 2001; Kumazawa and Masuda, 1999, 2001; Owuor, 1992; Shimoda et al., 1995). The only

study that reported the volatile compounds present in *Ilex paraguariensis* (Kawakami and Kobayashi, 1991) was conducted with one green mate sample from Argentina and one mate tea sample from Brazil, which prevented the observation of the changes that occurred during the roasting process. The antioxidant activity of volatile compounds from different plants contributes to flavor and has been the subject of published recently research (Lee and Shibamoto, 2001; Zin et al., 2002; Mau et al., 2003).

Green mate is obtained after the scorching, crushing and drying of leaves and stems, and can be stored for up to 1 year before commercialization, depending on the consumer's preference. Mate tea is obtained by the roasting of green mate (160 °C for approximately 12 min). Changes that might occur in the antioxidant activity of green mate as a consequence of the roasting process have not yet been described.

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The purpose of this study was to observe the effect of the roasting of dried green mate leaves on the antioxidant activity of mate infusions, their soluble solids content, total phenolic content and essential oil composition.

2. Material and methods

2.1. Material

Two kilograms of dried green mate (loose leaf) and two kilograms of mate tea (loose leaf) were obtained directly from the producer in Pinheiro Fernandes, Paraná, Brazil, during the harvesting period in August 2002. Both the green mate and the mate tea came from the same batch.

2.2. Mate infusions and soluble solids content

A quantity of 250 mL of hot distilled water (95 °C) was added to 3.0 g ± 0.1 g of green mate and mate tea leaves which had been previously ground to powder in a domestic coffee grinder (Protex Silex model E 160B). After five minutes, the supernatant was filtered under vacuum and cooled to room temperature (22 °C approximately). The soluble solids content was determined from 25 mL of green mate and mate tea infusions dried to constant weight at 105 °C in an oven. Three different preparations of green mate and mate tea were used for analysis.

2.3. Antioxidant activity (ferric thiocyanate method, FTC) and total phenolic content

The total phenolic content was determined by using the Folin–Ciocalteu reagent (Singleton and Rossi, 1965), and the results were expressed as Gallic acid equivalents. The FTC method was used to evaluate the ability of mate infusions to inhibit lipid oxidation by measuring the peroxide level during the initial stage of lipid oxidation. The reaction medium contained mate infusions at 0.02 g of soluble solids/100 mL in absolute ethanol, an emulsion of 2.51% linoleic acid in ethanol, 0.05 M phosphate buffer (pH 7.0) and distilled water. This medium was placed in a screw-capped tube, shaken and incubated in an oven at 40 °C in the dark. The same reaction medium, without the infusions or any other antioxidant was used as the control sample (Zin et al., 2002). Precisely, 3 min after the addition of 0.02 M ferrous chloride in 3.5% hydrochloride acid to the reaction medium, the absorbance was measured at 500 nm every 24 h, until 1 day after the absorbance of the control had reached its peak (a total of 96 h). A synthetic antioxidant (BHT) was used for comparison purposes, in the same concentration. The FTC method

response time is related to the period necessary for the absorbance to reach a maximum value. Several studies report a 96 h period, which includes one day after maximum absorbance value is achieved (e.g., Zin et al., 2002; Sánchez-Moreno et al., 1999). The low absorbance value of the reaction media after addition of FTC reagents indicates the efficacy of the test samples/synthetic antioxidant to inhibit lipid oxidation.

2.4. Extraction of essential oil from green mate and mate tea and identification of the volatile compounds

The mate samples (50 g) were placed in a 2 L round-bottom flask with distilled water in a Clevenger apparatus. The steam distillates were extracted with dichloromethane, and analysed using a gas chromatograph coupled to a mass spectrometer (Shimadzu, QP 5000), operating at an MS ionization voltage of 70 eV. The chromatograph was equipped with a capillary column DB-5 (30 m × 0.25 mm × 0.25 μm), and helium was used as the carrier gas. The following chromatographic conditions were used: injector at 220 °C, detector at 230 °C; gas flow 1.0 mL/min; split 1/20; initial column temperature of 60 °C with heating to 220 °C at 3 °C/min.

Studies that have evaluated the volatile compounds present in tea extracts have usually reported one single extraction for each sample (Kawakami and Kobayashi, 1991; Togari et al., 1995; Kumazawa and Masuda, 2001). Kovats retention indexes were obtained through the co-injection of the sample with a homologous mixture of *n*-alkanes (C₉H₂₀–C₂₅H₅₂), and calculated using the Van den Dool equation (Van den Dool and Kratz, 1963). The identification of the volatile compounds was performed through the comparison of the mass spectra of the substances and the data bank of CG-EM (NIST 62 lib), the related literature and the Kovats retention index (Adams, 1995; Jennings and Shibamoto, 1980).

3. Results

The phenolic compounds present in the infusions correspond to 26.6% and 22.2% of the total solids in green mate and mate tea infusions, respectively (Table 1). Both green mate and mate tea presented the same efficacy when compared with the synthetic antioxidant (BHT) with regard to the prevention of linoleic acid oxidation. The maximum absorbance reached by the control sample occurred after 72 h; therefore, total reaction time was 96 h (Fig. 1).

The profile of volatile compounds of green mate and mate tea are shown in Fig. 2 (a, b). A total of 30 compounds were detected, and 23 were identified in green mate essential oil. Forty-six compounds were

detected in mate tea essential oil, and 20 were identified by comparing the mass spectrum and Kovats index (Table 2).

Table 1

Soluble solids and total phenolics present in green mate and mate tea infusions^a

	Green mate	Mate tea
Soluble solids (mg/mL)	4.2 ± 0.00	3.2 ± 0.00
Total phenolics (mg/mL) ^b	1.13 ± 0.06	0.71 ± 0.01

^aNumber of samples (*n*) = 3.

^bGallic acid standard curve $y = 0.026 + 11.14x$, $r = 0.99$.

4. Discussion

Both green mate and mate tea infusions showed comparable antioxidant activities, which indicates that the roasting stage, although it modifies both the profile of volatile compounds and the phenolic content of the infusion, does not affect the antioxidant property, as measured by the ferric thiocyanate method. Oxidation is a very complex process that involves several steps, and all methods used to measure antioxidant activity present specific limitations. The method used in this study compares the *in vitro* antioxidant activity of mate infusions with a synthetic antioxidant and proves the efficacy of green mate and mate tea infusions as antioxidants.

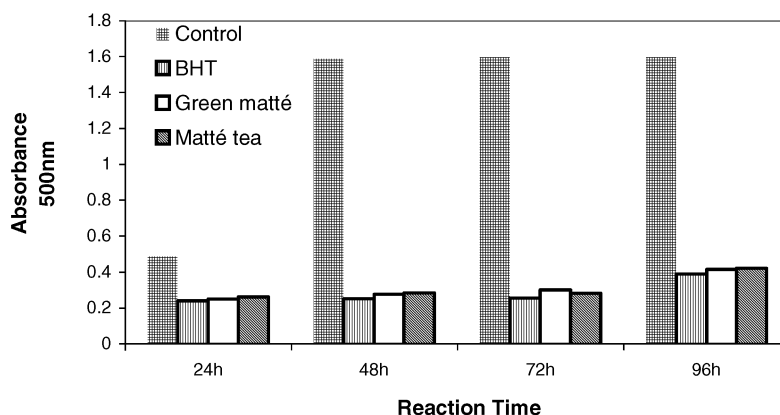


Fig. 1. Antioxidant activity of green mate and mate tea related to BHT and a Control without any antioxidant substance. Results are expressed as Absorbance, a parameter of lipid oxidation by the FTC method, as described in Section 2.

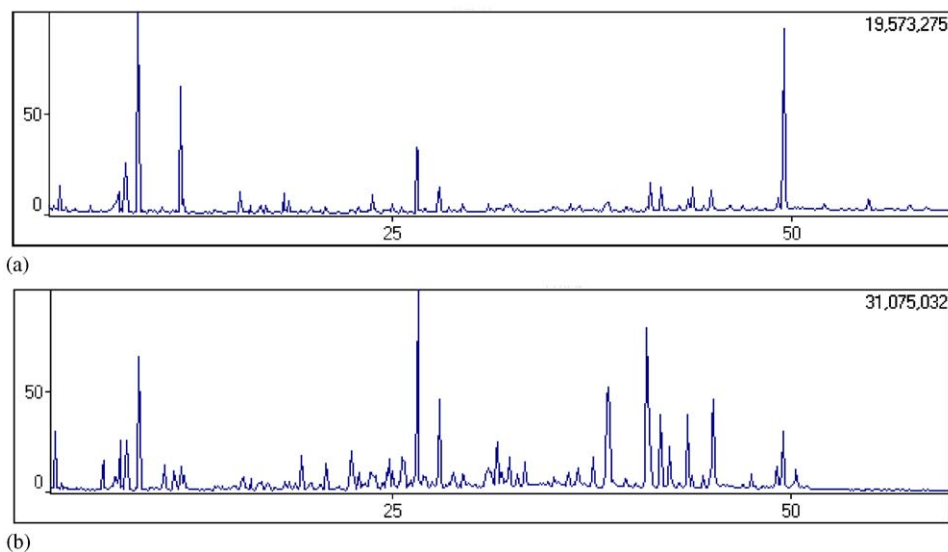


Fig. 2. Volatile compounds profile of mate tea essential oil.

Table 2
Volatiles compounds identified in green mate and mate tea based on Kovats Index and mass spectra

Kovats Index: experimental	Kovats Index: literature ^a	Compound	Green mate: relative %	Mate- tea: relative %
—	830	Furfural	n.f.	1.69
—	854	Hexenal (E)-2	1.50	n.f.
932	939	α -Pinene	0.51	n.f.
955	961	Benzaldehyde	n.f.	0.34
957	962	Methyl-5-furfural	n.f.	1.17
982	985	Hepten-2-one < 6 methyl-5 >	0.66	0.41
990	991	Myrcene	1.10	n.f.
995	998	Furfuryl methyl sulfide	1.88	1.80
1001	1001	<i>n</i> -Octanal	0.56	n.f.
1008	—	Furfuryl methyl sulfide isomer	4.32	1.92
1028	1031	Limonene	18.22	5.40
1070	1074	Linalool oxide < <i>cis</i> >	n.f.	0.98
1087	1088	Linalool oxide < <i>trans</i> >	n.f.	0.78
1093	1098	Linalool	12.16	n.f.
1104	1098	<i>n</i> -Non-anal	1.06	0.45
1189	1189	α -Terpineol	2.17	n.f.
1191	1190	Methyl salicylate	n.f.	0.46
1204	1204	<i>n</i> -Decanal	0.64	0.44
1253	1255	Geraniol	1.91	n.f.
1259	1261	Decenal (E)-2	1.10	n.f.
1313	1314	(E,E)-2,4-decadienal	n.f.	1.34
1383	1383	β -(Z)-damascone	1.74	0.67
1412	1409	β -(E)-damascona	0.95	0.70
1426	1426	α -(E)-ionone	n.f.	1.51
1452	1453	Geranyl acetone	7.05	11.35
1485	1485	β -(E)-ionone	2.81	4.83
1559	1559	Longicamphenylone	n.f.	0.56
1563	1564	(E)-nerolidol	n.f.	0.67
1700	1700	Heptadecane	0.68	n.f.
1927	1927	Methyl hexadecanoate	2.65	n.f.
2106	2100	<i>n</i> -Heneicosane	1.19	n.f.
2302	2300	<i>n</i> -Tricosane	1.15	n.f.

n.f., not found.

^aAdams (1995).

About one-fourth of the solids present in the infusions are composed of phenolic compounds, as determined by the Folin–Ciocalteu method. It is expected that this fraction would be composed mainly of phenolic acids, such as chlorogenic and caffeic acids (Mazzafera, 1997; Clifford and Ramirez-Martinez, 1990; Carini et al., 1998; Bastos et al. 2005), which are effective antioxidants (Olthof et al., 2001). Chandra and Mejia (2004) found that the antioxidant capacity of plant infusions (*Ardisia compressa*, *I. paraquariensis* and *C. sinensis*) correlated positively with total polyphenol content of dried leaves.

The essential oil present in infusions may also contribute to their antioxidant activity. This proved true for the essential oil of eucalyptus (*Eucalyptus polyanthemus*) and clove (*Syzygium aromaticum*) (Lee and Shibamoto, 2001), for the essential oil of the roots, fruit and leaves of a plant native to Asia, *Morinda citrifolia* L (Zin et al., 2002), and also for extracts of *Terminalia catappa* leaves (Mau et al., 2003).

Some important changes in the essential oil composition occurred during the roasting of green mate. The compounds formed in this process, such as furans, furanones and terpene oxides might be responsible for the flavor (usually described as sweet and smoky) and colour (brownish) of mate tea beverages. Compounds responsible for the floral aroma, such as limonene, decreased markedly (from 18.2% to 5.4%) after the roasting process, and linalool was practically degraded and oxidized into linalool oxides. The major compounds identified in the green mate essential oil were limonene, linalool and geranyl acetone. The major compounds identified in the mate tea essential oil were trans-geranyl acetone, limonene and beta-E-ionone.

Compounds with basic structure of isoprene exhibit antioxidant activity (Mau et al., 2003). Thus, it is expected that mate essential oil should play an important role in the antioxidant activity of mate infusions. Further investigations are necessary to evaluate the antioxidant activity of the essential oil of mate infusions. Kawakami and Kobayashi (1991), when

studying the essential oil of one green mate sample of the Argentine “chimarrão”, identified the following major compounds: 1-penten-3-ol; 2-butoxyethanol; linalool, hexanoic acid, octanoic acid and nonanoic acid. Hexanoic acid, furfural, acetic acid, eugenol and linalool *cis*-oxide were the major compounds identified in one sample of mate tea produced in Brazil.

5. Conclusions

Both green mate and mate tea infusions have the same antioxidant efficacy as BHT, a well-known phenolic antioxidant, under the conditions used in this study. The roasting process leads to significant alterations in the essential oil of mate. Compounds that contribute to the green and floral aroma of green mate infusions were either reduced or destroyed after the roasting process. Other compounds, such as methylfurfural and furfural, which might contribute to the smoked flavor and aroma of mate tea infusions, were formed after the roasting process. The observation of the changes that occur in the antioxidant activity and in the compounds responsible for sensorial characteristics of plant infusions may contribute to the production of beverages with higher aggregated value and stimulate the consumption of mate products. These are, by far, healthier than other well-accepted non-nutritional beverages, such as sodas. Further researches on the composition of volatile compounds that affect the sensorial analysis of mate infusions, as well as researches on the antioxidant potential of mate essential oil, should be conducted.

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