



Hydrostatic pressure cycling extraction of soluble matter from mate leaves

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ABSTRACT

The main aim of this study was to investigate the kinetics of solid–liquid extraction of soluble matter from leaves of *Ilex paraguariensis* assisted by pulsed hydrostatic pressure. A large set of experiments was carried out involving a mixture of distilled water and comminute leaves of mate fed in a batch extractor kept at approximately 16.7 °C. The influence of pressure on equilibrium solute concentrations and rate of extraction was examined in the pressure range from 91.4 to 338.2 kPa by applying or not hydrostatic pressure cycles. Whatever the case a significant increase of such responses with direct positive impact on extraction yield and time of extraction was experimentally observed by changing the investigated factor (e.g.; the extraction yield was increased from ≈13% at 91.4 kPa to approximately 34% and the time to have 90% of the highest efficiency was reduced from ≈17,000 s at 91.4 kPa to ≈6000 s by applying hydrostatic pressure pulses at only 338.2 kPa). An hybrid diffusive–convective model was suggested to represent the transient extraction of soluble compounds from the discoid particles. The classical Fick's law described the two-dimensional diffusion for the static long periods of mass transfer at constant pressure, while a model dependent on the gamma function computed the fraction of solute periodically extracted by convection from the internal solid microchannels during the rapid pulses of hydrostatic decompression. For all the investigated conditions the proposed analytical model well reproduced the kinetic experimental results of solute mass fraction in the solid and liquid phase.

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1. Introduction

An amazing average annual growth of 7.7% was registered for the Brazilian per capita intake of non-alcoholic ready-to-drink beverages prepared by infusion of shoots of *Camellia sinensis* and leaves of *Ilex paraguariensis* between 2005 and 2010 (ABIR, 2011). In this segment of beverages the aqueous extracts of mate have represented approximately 18% of the overall consumption with 13.9 million liters in 2004 and around 17.0 million liters in 2008 (ABIR, 2008). Based on this scenario and on the importance of mate crop and manufacturing in Brazil (e.g.; Zanoelo and Benincá, 2009; Rodrigues et al., 2010; Jensen et al., 2011), which is the second world-leading of mate production (Rodrigues et al., 2010), improvements in the conventional industrial process of solid–liquid extraction involving leaves of mate are strongly expected.

The main reasons for such process optimization are the high temperatures and long operation time required to have high extraction yields. Such conditions have not only drastic impacts on costs, but they present not negligible detrimental effects on thermolabile compounds and sensorial properties of the marketed beverage. For instance, the maximum yield by infusion at atmospheric pressure estimated by using a reliable equilibrium model

(Jensen and Zanoelo, in press) (≈0.37 kg solute per kg dry solid) demanded extraction time longer than 36,000 s at temperatures close to the boiling point of water. Because the extraction time exponentially reduces when the yield of extraction decreases, a maximum extraction yield close to 80% of the aforementioned value was experimentally reported by infusion of mate leaves for only 1800 s (Sambiassi et al., 2002), but a high temperature equal to 348 K was again necessary to have it. An additional analogous investigation available in the literature (Linares et al., 2010) also confirms that conventional infusion of mate leaves is a time-consuming operation ($t > 3600$ s for equilibrium) whose maximum extraction yield (≈0.35 kg solute per kg dry solid) requires at least moderate temperatures (in this case T was approximately 343 K).

Among the large number of innovative solid–liquid extraction processes reported in the literature, such as microwave-assisted and pulse electric field-assisted extraction (e.g.; Naviglio and Ferrara, 2008; Zhang et al., 2011; Lebovka et al., 2012), only supercritical fluid extraction (SFE) (Esmelindro et al., 2005; Cardozo et al., 2007; Jacques et al., 2007; Cassel et al., 2008), pressurized liquid extraction (PLE) (Jacques et al., 2006, 2008; Grujic et al., 2012) and ultrasound-assisted extraction (Jacques et al., 2006) have been tested in a laboratory scale to obtain soluble compounds from leaves of mate. Anyway, almost all of them were rather focused on the chemical characterization of the mate extracts (typically rich in saponins, caffeine, theobromine and chlorogenic acids)

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Nomenclature

α_n	positive roots of $J_0(r\alpha_n)$	r	radial direction of diffusion (m)
β	proportionality constant in Eq. (19) (kg kg^{-1})	R	radius of the discoid particle of mate (m)
D	diffusion coefficient ($\text{m}^2 \text{s}^{-1}$)	Γ	gamma function
δ	thickness of the discoid particles of mate (m)	T	temperature of extraction ($^\circ\text{C}$)
E	efficiency of extraction (%)	t	time of extraction (s)
ε	mass of solution entering the solid phase at a k th pressure pulse (kg)	\bar{X}_l	average solute mass fraction in the liquid phase (kg kg^{-1})
γ	parameter of the equilibrium-dependent model (s^{-1})	\bar{X}_s	average solute mass fraction in the solid phase (kg kg^{-1})
J_0	Bessel function of order zero	X_s	solute mass fraction in the solid (kg kg^{-1})
J_1	Bessel function of first order	X_{so}	initial solute mass fraction in the solid (kg kg^{-1})
k	k th pressure pulse	X_{se}	equilibrium solute mass fraction in the solid phase (kg kg^{-1})
K	total number of applied pressure pulses	X_l	solute mass fraction in the liquid phase (kg kg^{-1})
m_{so}	dry mass of solid fed in the extraction chamber (kg)	X_{le}	equilibrium solute mass fraction in the liquid phase (kg kg^{-1})
m_l	mass of solvent fed in the extraction chamber (kg)	Y	extraction yield (%)
m_{sol}	mass of solution leaving the solid phase at a k th pressure pulse (kg)	Y_∞	yield when all the solute is extracted from the leaves (%)
M_0	moisture content of mate leaves (d.b.)	z	axial direction of diffusion (m)
ω	pulse rate (s^{-1})		
P	pressure of extraction (kPa)		

(e.g.; Cardozo et al., 2007). A reliable evidence that extraction towards process optimization was not considered is the high pressure range (10,000–25,000 kPa) always adopted in these investigations (at least for SFE and PLE) (Esmelindro et al., 2005; Cardozo et al., 2007; Jacques et al., 2006, 2007, 2008; Cassel et al., 2008; Grujic et al., 2012) whose influence on the capital cost is important in a real industrial extraction unit. Moreover, water was never applied as solvent in these investigations involving SFE, PLE and ultrasound-assisted extraction of soluble matter from mate leaves, which is a mandatory elementary aspect for producing ready-to-drink tea-like beverages.

In this framework, the current study mainly aims to enhance the rate of removal of soluble matter from leaves of mate by applying hydrostatic pressure cycling extraction (HPCE). The increase of the equilibrium solute concentration in the liquid phase is an additional important objective with direct positive consequences on the maximum yield of extraction. With these purposes the kinetics of solute extraction was experimentally investigated in the pressure range from 91.4 to 338.2 kPa at 1:600 pulses per second, or without pressure pulses, for 25,200 s. All the experiments were carried out in a cylindrical pressurized vessel where comminute leaves of mate and distilled water were placed at ambient temperature ($\approx 17^\circ\text{C}$). An hybrid diffusive-convective model whose solution was analytically obtained was suggested to reproduce the kinetics of solid-liquid extraction at all the investigated operating conditions.

2. Materials and methods

2.1. Experiments

Leaves of mate dried on commercial scale and cut into smaller pieces by industrial equipments were used in the experiments of extraction. In order to have a relatively homogeneous mass of particles in terms of size and shape, which are factors that have significant effect on mass transfer during extraction (e.g.; Bucic-Kojić et al., 2007), the material fed in the extractor was preliminarily taken to a size analysis. It was made with a set of only two screens of the Tyler standard series (12 and 20 mesh) (Perry and Chilton, 1973) arranged serially in a stack shaken manually. Only the material retained on the 20 mesh screen was removed and applied for the investigation of the kinetics of extraction. The particles were

shaped like discs 1.13×10^{-3} m in average diameter and 0.23×10^{-3} m thick. The diameter represents the arithmetic mean between the opening of the screen through which the fraction passed and the other on which it was retained, while the thickness is the mean of several measurements early made with a digital micrometer (Jensen and Zanoelo, in press).

It is well-known that insufficient water removal during the stage of drying of solids negatively affects solid-liquid extraction from grains or vegetable matrices (e.g.; Hofmann et al., 2012). However, in the absence of a Brazilian legislation regarding the commercial limits for the moisture content of packed mate leaves (ANVISA, 2005) mate manufacturers usually produce a final product with high values for this parameter (Zanoelo et al., 2008). For instance, it typically well exceed the upper limit stated for commercial tea shoots ($\approx 3.6\%$ d.b.) (Temple and van Boxel, 2000). Based on these aspects, the moisture content of the comminute leaves of mate used in all the extraction experiments ($M_0 = 8.0 \pm 0.1\%$ d.b.) was determined in triplicate by oven drying at 105°C for 24 h (AOAC, 1990).

Aqueous exhaustive extraction of soluble matter was performed to experimentally determine the total content of soluble constituents in the examined solid particles. The experiment was performed with a sachet made of filter paper (0.08 kg m^2) containing 7×10^{-3} kg of solid sample. In order to have the sachet fully immersed in the solvent it was put within a heavy stationary stainless steel screen basket. The entire structure was placed in an extractor open to the atmosphere and filled with 0.4 kg of distilled water kept at the boiling temperature at ≈ 91.4 kPa by using a hot plate with a manual control of temperature (752A, Fisatom Equipamentos Científicos Ltda, São Paulo, Brazil). The extraction vessel was periodically refuelled with hot fresh water to replace the quantity evaporated during extraction for 18,000 s. The difference between the initial and final mass of leaves again determined by oven drying at 105°C for 24 h (AOAC, 1990) represents the total content of solute initially found in the solid phase. Under the same identical conditions the procedure was repeated three times. It is valuable to be aware that besides the importance for commercial purposes of having revealed the large content of solute typically found in the leaves of mate, the current experiment makes possible to calculate the equilibrium solute concentration in the solid phase based on the monitored experimental results of solute mass fraction in the solvent. The initial and the equilibrium solute concentrations in

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