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Cyclically pressurized extraction of solutes from ground coffee: Kinetic experiments and modeling

Ronald Wbeimar Pacheco Ortiz [a], Cristina Benincá [b], Everton Fernando Zanoelo [a,*]

[a] Federal University of Paraná, Graduate Program of Food Engineering, Department of Chemical Engineering, Curitiba, PR, Brazil
[b] Federal University of Rio Grande, School of Chemistry and Food, Santo Antônio da Patrulha, RS, Brazil

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ABSTRACT

The main aim of this study was to investigate the influence of pressure and pressure cycles on the kinetics of solute removal from ground coffee. Extraction experiments were performed with water at a low temperature (≈17 °C) in the pressure range from 91.4 kPa to 338.2 kPa for 25,200 s. A semi-empirical pure diffusive and a hybrid diffusive-convective model described correctly the transient experimental extraction curves at constant and cyclic pressurized conditions, respectively. With 80% of probability the modeling approach supports, and explains in terms of transport phenomena, the significant positive effect of the examined factors on the kinetics of extraction. According to it, the rise of pressure increases the diffusivity, while the pressure cycles cause a movement of fluid through the solid microchannels and consequent solute transfer by convection. Coffee lipids were also extracted under constant and cyclic pressurized conditions with a polar (water) and a nonpolar (chloroform) solvent. The negligible fraction of total lipids in the aqueous phase at equilibrium refutes an important alleged advantage of hydrostatic pressure cycling extraction, that is, it extracts insoluble compounds. Moreover, a non-significant influence of pressure and pressure cycles ($p > 0.02$) on total extracted lipids at equilibrium was observed when using chloroform.

1. Introduction

Coffee is nowadays the second most important commodity worldwide, only behind petroleum [1,2]. Brazil is the world-leading producer and exporter of green coffee beans [3,4], but the role it plays in the global market of soluble coffee, where it still ranks first in terms of export [3,4], deserves attention.

The main reason for such a particular concern is that consumption of soluble coffee has increased at rates higher than those for roasted ground coffee. In fact, approximately a half of the coffee available for market around the world is currently used as raw material for production of soluble coffee [1,5]. In spite of some domestic problems, such as a number of only 7 Brazilian soluble coffee manufacturers [6] and an insufficient production of *Robusta* coffee used in a high proportion when compared to *Arabica* to produce soluble coffee [7], the Brazilian exports of soluble coffee raised from 3,120 in 2009/10 to 3,486 thousand 60 kg bags in 2012/13 [3].

From a technological point-of-view, an approach to keep the Brazilian leadership in such a sector is to improve the quality and reduce the costs involved in the manufacture of soluble coffee. In other words, it means to replace the conventional method of solute extraction from coffee ground by a better procedure in terms of these responses. Leaching by percolation through beds of ground coffee is the primary technique currently applied in the manufacture of instant (soluble) coffee [8,9]. Because the temperature is always an important factor on extraction [10,11] percolation takes place in a temperature range from 100 to 200 °C [8,9,12]. So, to keep the water in a liquid phase, the battery of extraction columns are pressurized up to about 1550 kPa. At these circumstances the yield of solute extraction varies approximately between 30% and 46% [8,12] and the time of extraction is not higher than 1800 s [13]. However, the benefit of the high temperatures on the yield and time of solute removal has expected adverse effects on energy consumption, as well as on the aromatic properties of the end product [14]. Moreover, the high pressure for pressurized liquid extraction (PLE) has a detrimental influence on the capital cost [15]. It is important to remember that units to recovery volatiles compounds and avoid the production of poor flavor instant coffee are commonly found in large-size plants [8], but they have no negligible impact on costs.

In this framework, the kinetics of extraction of coffee solubles at low temperature by involving an innovative technique is the main

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[*] Corresponding author. Tel.: +55 41 33613583; fax: +55 41 33613674.
E-mail address: everton.zanoelo@ufpr.br (E.F. Zanoelo).

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Nomenclature

β	proportionality constant in Eq. (8) ($kg\ kg^{-1}$)
D	diffusion coefficient ($m^2\ s^{-1}$)
ε	mean absolute relative difference between experimental and calculated $\bar{X}_L$ (%)
γ	parameter of the equilibrium-dependent model in Eq. (7) (s^{-1})
m_L	mass of solvent fed in the extraction chamber (kg)
m_{S0}	dry mass of coffee fed in the extraction chamber (kg)
$\dot{m}_S$	mass flow rate of solute ($kg\ s^{-1}$)
ω	frequency of pressure cycles (s^{-1})
P	pressure of extraction (kPa)
P_{atm}	atmospheric pressure (kPa)
r	direction of solute transfer (m)
R	radius of the spherical particle of coffee (m)
R^2	coefficient of determination
ρ	density of aqueous solution ($kg\ m^{-3}$)
t	time of extraction (s)
$\bar{u}$	average velocity in the solid microchannels ($m\ s^{-1}$)
U	uncertainty in coffee yield determination (%)
$\bar{X}_L$	solute mass fraction in the liquid phase ($kg\ kg^{-1}$)
X_{LE}	equilibrium solute mass fraction in the liquid phase ($kg\ kg^{-1}$)
X_S	solute mass fraction in the solid ($kg\ kg^{-1}$)
$\bar{X}_S$	average solute mass fraction in the solid phase ($kg\ kg^{-1}$)
X_{S0}	initial solute mass fraction in the solid ($kg\ kg^{-1}$)
X_{SE}	equilibrium solute mass fraction in the solid phase ($kg\ kg^{-1}$)
Y	yield of total solutes in water defined in Eq. (8) (%)
Y_{oil}	yield of coffee oil (%)

aspect to be currently investigated. Among the alternative extraction techniques available in the literature, such as pulsed electric field assisted [16], ultrasound assisted [17], and supercritical fluid extraction [18], one that allows for high yields at short time, moderate pressures and ambient temperature is the cyclically pressurized extraction (pulsed hydrostatic pressure extraction or hydrostatic pressure cycling extraction) [19,20]. Based on these believed advantages, which should contribute to improve the quality of the extract and reduce the costs of production and investment, cyclically pressurized extraction with water was applied to remove total solutes from ground coffee. The influence of pressure and cycles of pressure on the yield and time of extraction was considered, and the optimal results in terms of these responses were compared with those obtained by the conventional method of manufacture of instant coffee. Experiments for extracting coffee lipids were also performed under cyclic pressurized conditions with water and chloroform, mainly to check whether it is able or not to extract insoluble compounds, an important claimed advantage of this innovative method of extraction [21].

2. Materials and methods

2.1. Experiments

A fraction of a package of commercial ground coffee in the size range between 4.17×10^{-4} and 4.95×10^{-4} m was used in all the extraction experiments. It represents the material retained on a 35 mesh screen in a size analysis made with a set of only two screens of the Tyler standard series (32 and 35 mesh) [22]. The moisture content of the solid determined in triplicate by oven drying at 105 °C for 24 h was 5.4 ± 0.1% in dry basis [23]. Such a preliminary characterization of the starting solid material in terms of size and moisture was necessary because both these factors have in general a significant effect on solid–liquid extraction (e.g., [11,24]).

Aqueous exhaustive extraction was initially performed with the purpose of determining total solubles in the examined solid sample. The experiment was conducted by immersing a sachet with 7×10^{-3} kg of coffee in 0.4 kg of distilled water at its normal boiling point for 18,000 s. Analogous exhaustive extraction of total lipids with a mixture of chloroform (Vetec, Duque de Caxias, Brazil) and methanol (Neon, São Paulo, Brazil) of analytical grades was applied as recommended by Bligh and Dyer [25]. In both the cases, a gravimetric method was used to determine the total soluble matter in water [23] and total lipids soluble in the mixture of organic solvents [25]. Under the same identical conditions both the procedures were repeated three times.

The extraction experiments at constant and cyclic pressurized conditions always involved a mixture of 3×10^{-2} kg of solvent (distilled water or chloroform) and 1.5×10^{-3} kg of ground coffee placed in a sachet. The extraction chamber partially filled with the solid–liquid mixture was a polycarbonate cylinder internally equipped with a piston. To keep the system at the desired pressure, it was moved with an hydraulic press (15 tons, Schulz, Joinville, Brazil) up to a fixed position defined by the Boyle's law. The extraction chamber was immersed in a refrigerated bath (SL 152/10, Solab, Piracicaba, Brazil) to have a constant low temperature of extraction. Based on more than 140 measurements with a calibrated K type thermocouple connected to a datalogger (TH-060, Instrutherm, Sao Paulo, Brazil) an average temperature of extraction close to 17.2 ± 0.1 °C was found.

The solute mass fractions in the aqueous phase were periodically determined for 25,200 s by a gravimetric method [23]. Sampling was made at a time step of 1200 and 3600 s under cyclic and constant conditions of pressurization, respectively. In the experiments for coffee oil extraction, the mass of total lipids removed from the coffee sample was only determined at equilibrium, based on the method of Bligh and Dyer [25].

All the used sachet were made of paper filter with pore size equal to 1.4×10^{-5} m ($80\ g\ m^2$). A digital balance with a precision of $\pm 10^{-7}$ kg (AS 220/C/2, Radwag Wagi Elektroniczne, Radom, Poland) and an oven (400/ND, Ethik Technology, Vargem Grande Paulista, Brazil) were used to weight and dry the samples when necessary, respectively.

On the whole, a set of 8 kinetic experiments (+1 at 91.4 kPa) was performed to determine total solute (in water) by varying the pressure and pressure cycles at 4 and 2 levels ($4^1 \times 2^1 = 8$), respectively. In particular, P was close to 135.3, 202.9, 270.5, 338.2 kPa, while ω was zero and 1:600 cycle per second (i.e.; 300 s pressurization +300 s depressurization per cycle). A frequency equal to zero or only 1:600 cycles per second ($\omega = 1/600\ s^{-1}$) was adopted because in a previous analogous study the reduction of frequency to a value close to $1/1200\ s^{-1}$ had a negligible influence on the kinetics of cyclically pressurized extraction [19]. The effect of increasing the frequency to a value higher than $1/600\ s^{-1}$ (i.e.; 300 s pressurization +300 s depressurization per cycle) was not possible because the pressurization currently involves a manual procedure with a hydraulic press that takes at least 30 s. Although the influence of temperature and size of particle on leaching is certainly significant, these factors were not currently considered because many available results confirm this finding (e.g.: [11,24]). Anyway, it is also important to mention that would be difficult to keep the pressure in the extraction chamber at a desired pressure if a temperature