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Pressure cycling extraction as an alternative to percolation for production of instant coffee

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1. Introduction

ABSTRACT

Pressure cycling (300 s at 91.4 kPa + 300 s at 338.2 kPa per cycle) was performed to extract water-soluble compounds from ground coffee at \approx 17 °C. The efficiency of solute removal (*E*) at equilibrium, and the extraction rate constant by dispersing the solids in the solvent (k) were examined. The obtained aqueous solution was freeze-dried at \approx 2.25 Pa for 48 h and the produced instant coffee was analyzed by GC–MS to speculate about its quality. For comparison, an additional set of experiments by involving three different extraction procedures was also carried out (i: infusion at 91.4 kPa and \approx 17 °C; ii: infusion at 91.4 kPa and \approx 100 °C; iii: pressurized extraction at 338.2 kPa and \approx 17 °C). Pressure cycling extraction presented the optimal *E* (79 ± 6%), and a *k* (2.9 × 10⁻³ s⁻¹, or 1033 s to reach equilibrium) comparable to that found at 100 °C in percolator batteries of commercial extractors. The qualities of lyophilized coffee from pressure cycling extraction and from the best expected condition of extraction for the purpose of retention of coffee volatiles (i.e.; 91.4 kPa and \approx 17 °C) were analogous.

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Manufacturing plants of instant coffee are the destination of almost 50% of the global production of green coffee [1,2]. In spite of the growing importance of such a kind of industry, negligible innovations have been proposed to make it more competitive in terms of operating/capital costs and quality of produced soluble coffee. One of the few recent advances was in the operation of extraction [3] that precedes the stages of water evaporation and spray or freeze drying of concentrated coffee liquor [4,5].

It has been demonstrated that the yield of extraction of solutes from ground coffee assisted by cyclic pressurization at only 17 °C and \approx 338 kPa is around 26% [3], which is just \approx 4 percentage points lower than that obtained by industrial percolation at 100 °C and 101.3 kPa [4]. Yields up to 36–46% may be obtained in percolator batteries of commercial extractors, but this is only possible under much higher pressures and temperatures (200 °C and \approx 1500 kPa) [5]. It has been concluded the time of extraction by cyclic pressurization was much longer than that by percolation in real-size plants of soluble coffee production [3]. However, from a practical

point of view this is only an apparent drawback whose reason was the convective resistance to mass transfer due to the low permeable bed of solids formed in the sachets with ground coffee used in that investigation [3]. Such a problem is typically solved by dispersing the coffee grounds in water, a well-known approach whose importance has been already discussed in the literature [6,7].

Despite the mentioned advantages of pressure cycling extraction when compared to conventional leaching by percolation (with obvious positive effects on reduction of capital and operating costs), the influence of applying such an innovative extraction procedure on retention of volatile substances is an important matter not yet well-explored for the purpose of instant coffee production. Although coffee beans contain a complex mixture of approximately 700-1200 volatiles from many different chemical families [5,8–11], none these species have been quantified in extracts obtained under conditions of cyclic pressurization of the extraction chamber. The same is true for the approximately 40-100 key coffee odorants (e.g.; 2-ethyl-3,5-dimethylpyrazine, 2,3-diethyl-5-methylpyrazine, guaiacol, 4-ethylguaiacol, 4-vinylguaiacol) [5,10–13] that essentially determine the power flavoring of coffee.

Nowadays, it is only supposed that the low temperature and short time of dispersed-solid extraction under cyclic pressurization would contribute positively for the retention of volatiles







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Nomenclature

Aca	area under the peak of caffeine in the GC-MS chro-	J	total number of identified aromatic hydrocarbons
	matogram	k	extraction rate constant (s ⁻¹)
A_i	area under the peak of an <i>i</i> th specie in the GC–MS chro-	п	number of replicates
	matogram	ω	frequency of pressure cycles (s^{-1})
Ar _i	area under the peak of an <i>j</i> th aromatic hydrocarbon in	Р	pressure (kPa)
-	the GC–MS chromatogram	t	time of extraction (s)
β	ratio between the inlet mass of water and dry ground	Т	temperature of extraction (°C)
	coffee (kg kg $^{-1}$)	U_E	uncertainty in <i>E</i> for $\alpha = 0.05$
Ε	efficiency of total solute removal (%)	U_{Ye}	uncertainty in Y_e for $\alpha = 0.05$
E_{w}	efficiency of total solute removal represented by a white	<i>Y</i> Ae	equilibrium solute mass fraction in the aqueous phase
	bar in Fig. 1a (%)	-	$(kg kg^{-1})$
E_d	efficiency of total solute removal represented by a dark	Ye	vield of extraction (%)
-	bar in Fig. 1a (%)	Ymax	yield of total solute removal by exhaustive extraction
I	total number of compounds identified by GC-MS	mux	(32%)

Table 1

compounds in the coffee extracts. It is essentially inferred from the well-known role of these factors on retention of thermolabile compounds in extracts of coffee, and of plants in general [8,9,14,15]. The expensive aroma recovery units typically used to minimize the loss of coffee odorants in the coffee industry, where volatiles are condensed and later added to the dry product, confirms the need of avoiding extraction at high temperatures for long time to produce instant coffee [4,5,16].

In this framework, the main aim of this study was to obtain and examine a set of responses of practical importance for production of instant coffee by applying four different procedures of solid-liquid extraction. The focus was on the efficiency of solute removal (E); kinetics of packed- and dispersed-leaching (k or t); total number of compounds (*I*), total area of peaks (ΣA_i), and area under peaks of aromatic hydrocarbons (ΣAr_i) in chromatograms of lyophilized coffee extracts from GC-MS analysis. The extraction was carried out under atmospheric pressure (91.4 kPa) at approximately 17 °C (run 1) and 100 °C (run 2), under constant pressurization of the extraction chamber at 338.2 kPa and 17 °C (run 3), and under cyclic pressurization (300 s at 91.4 kPa + 300 s at 338.2 kPa per cycle) at 17 °C (run 4) always for 7 h. In summary, based on these experiments it is expected to reveal the positive impact of pressure cycling extraction on the efficiency and kinetics of solute removal, as well as on the quality of instant coffee in terms of the examined GC-MS responses.

2. Materials and methods

2.1. Extraction experiments and freeze-drying of coffee extracts

Samples of commercial ground coffee from two different lots (*A* and *B*) preliminary taken to size determination by sieving through only the 32 and 35 mesh Tyler screens (Bertel, Caieiras, Brazil) were used as raw material for all the extraction experiments. The selected coffee particles with size limits between 4.25×10^{-4} m and 5×10^{-4} m had a moisture content close to 5.1% in wet basis. It was determined by a gravimetric method that involved the use of an oven operated at 105 °C for 24 h (400-2ND, Ethik Technology, Vargem Grande Paulista, Brazil) and a digital balance with a precision of $\pm 10^{-7}$ kg (AG-200, Gehaka, São Paulo, Brazil).

Except for water infusion at ≈ 100 °C taken place in an open flat bottom vessel heated with a hot plate (752A, Fisatom, São Paulo, Brazil), all the remaining extraction experiments (at ≈ 17 °C) were carried out in a cylindrical polycarbonate chamber internally equipped with a moving piston at one end, and sealed at the opposite side. In these later cases, the extraction vessel was immersed in a refrigerated circulating water bath with a PID control of temperature (SL 152/10, Solab, Piracicaba, Brazil). When the extraction was at 338.2 kPa or under cyclic pressurization ($\omega = 1:600 \text{ s}^{-1}$, that is, 300 s at 91.4 kPa + 300 s at 338.2 kPa per cycle), the piston was forced through the cylinder with a hydraulic press (15 tons, Schulz, Joinville, Brazil). In all the examined circumstances, a mass of approximately 1.5×10^{-3} kg of ground coffee was loaded in sachets made of paper filter (average pore size = 1.4×10^{-5} m) before taking them in contact with about 3.0×10^{-2} kg of distilled water for 7 h. The obtained extracts were stored at $-10 \,^{\circ}$ C for freeze-drying, but before doing so a single aliquot of $\approx 5 \times 10^{-3}$ kg was removed from each final water solution to determine the mass fraction of total solutes in that phase (by gravimetry at 105 °C for 24 h). Extraction was replicated two times with coffee *A* and *B* at each examined condition summarized in Table 1.

A set of eight crystal polystyrene glasses with approximately 80×10^{-2} kg of extract (i.e.; 4 glasses had extracts from lot *A* and 4 from lot *B*) was taken to a freeze dryer (Enterprise I, Terroni, São Carlos, Brazil). The drying operation was carried out at \approx 2.25 Pa for 48 h by keeping the condenser temperature close to -40 °C with R402B as refrigerant. The dry coffee powder was weighed (AG-200, Gehaka, São Paulo, Brazil) and stored at -10 °C for GC–MS analysis. The measured masses of instant coffee and solution per polystyrene vessel were used to compute the mass fraction of coffee solubles in the extract, a response that was already determined as early explained, but by exposing the solution at high temperature.

A single kinetic extraction experiment with dispersed solids was performed at the condition defined by run 4 in Table 1. Because the extraction was under cyclic pressurization in the already described cylindrical polycarbonate chamber, a magnetic stirrer operated at 2000 rpm was used (Q241 M, Quimis, Diadema, Brazil). The stir bar was a cylinder 5×10^{-4} m in diameter and 2×10^{-3} m in length. Four identical aliquots were periodically removed from the extractor and filtered through 1.4×10^{-5} m pore

Operating conditions for extraction of water soluble coffee compounds, yield and						
efficiency of extraction at 7 h. Uncertainty in Y_e and E are for $n = 6$ ($v = 5$) and $\alpha = 0.05$.						

Run	P (kPa)	T (°C)	ω (s ⁻¹)	$Y_e\pm U_{Ye}~(\%)$	$E \pm U_E (\%)$
1	91.4	17.0 ± 0.2	0	14 ± 2^{a}	44 ± 7^{a}
2	91.4	≈ 100	0	27 ± 1 ^b	85 ± 4^{b}
3	338.2	17.0 ± 0.2	0	24 ± 1 ^c	74 ± 4^{c}
4	338.2	17.0 ± 0.2	1:600	25 ± 2 ^{b,c}	79 ± 6 ^{b,c}

Yields or efficiencies followed by the same letters do not differ statistically by the *t*-test at p > 0.05

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