Scientia Chromatographica 2019; 11(1):28-33
Instituto Internacional de Cromatografia
http://dx.doi.org/10.5935/sc.2019.006
ISSN 1984-4433

SAMPLE PREPARATION

A novel coffee oil extraction procedure employing pressurized solvents

Jair Sebastião da Silva Pinto [1], Suzana Lucy Nixdorf [2], Marcela Jordan-Sinisterra [3], Fernando Mauro Lanças [3*]

[1] Escola Superior de Agricultura Luiz de Queiroz-ESALQ/USP, 13418-900 Piracicaba-SP, Brazil.

[2] Departamento de Engenharia de Alimentos, Universidade Federal de Sergipe, Av. Marechal Rondon, s/n, 49100-000, São Cristóvão, SE, Brazil.

[3] Institute of Chemistry of São Carlos, University of São Paulo. Brazil. Corresponding author: F.M. Lanças, flancas@iqsc.usp.br

Resumo

No presente trabalho, o óleo de café torrado foi obtido usando dois métodos de extração, sendo um sistema de extração por solvente pressurizado (PSE), proposto no presente trabalho, e o outro um método endossado pela AOAC (Association of Official Analytical Chemists) INTERNATIONAL usando um sistema Soxhlet. Os rendimentos (% p/p) obtidos em cada um deles foram semelhantes, o que permitiu comparar as condições de cada método. Foi demonstrado que no sistema PSE o consumo de solvente (20 mL) e o tempo de extração (20 min) são menores, do que o volume usado no sistema Soxhlet (200 mL) e o tempo de análise usado (480 min). Isso faz do PSE um método de extração promissor para uso na indústria.
Palavras chaves: Óleo de café, café arábica, extração com solvente pressurizado (PSE), extração acelerada por solvente (ASE), química analítica verde (GAC).

Abstract

In the present work, roasted coffee bean oil was obtained using two extraction methods, a pressurized solvent extraction system (PSE) and a method endorsed by the AOAC (Association of Official Analytical Chemists) INTERNATIONAL, using a Soxhlet extraction system. The yields (% w/w) obtained in each of them were similar, which allowed the conditions of each method to be compared, demonstrating that in the PSE the solvent consumption (20 mL) and the extraction time (20 min) is less at the volume used in the Soxhlet system (200mL) and the analysis time used (480 min). This makes PSE a promising extraction method for use in industry.
Keywords: Coffee oil, arabica coffee, pressurized solvent extraction (PSE), accelerated solvent extraction (ASE), green analytical chemistry (GAC).

1. Introduction

The Coffee is one of the agricultural products that over the years have established itself on the list of the most consumed in the world (1). Therefore, the demand for processed coffee products in the international market increases every day, highlighting the aroma and flavor extracts of roasted coffee, sensory attributes that are due to a mixture of a wide variety of volatile compounds obtained during the roasting process (2), qualities widely used in so-called sophisticated foods (3).

Studies have shown that ketones, pyrazines, furans, phenols and pyrrole are the main compounds responsible for the aroma of these roasted coffee beans (4). However, it has also been shown that these volatile compounds vary according to geographical location, the botanical variety of coffee beans (robusta, arabica, among others) and mainly, according to the roasting process carried out (time, temperatures, between others) (5). Observing that coffee oil is one of the main carriers of the aroma and flavor of roasted coffee (1), which is obtained between 10-15% by weight (depending on the variety of the bean) of the organic residues of the brewed coffee, the so-called spent coffee grounds (SCG) (6).

Because of this, coffee oil is widely used as a flavoring in food industries (ice cream, beverages, instant coffee) (7) or as a natural ingredient in cosmetic and grooming products (8,9). As a "superior line" product with high quality, it must meet some specifications to be consumed and exported, one of those requirements being the determination of acidity, which also varies according to the soil of the crop, the climatic conditions, the management of the harvest, the species, etc. So, the raw material need to be first analyzed, to see if it is able to produce a final product with low acidity. The analysis of acidity and free fatty acids are extremely important parameters that allow us to know the state of deterioration of the product, during the storage of the raw material and, also the state of the oil after it has been extracted (10).

One of the most used techniques for the extraction of coffee oil is the one that uses organic solvents; the drawback is that this technique leaves permanent residues in the products that are considered toxic for human consumption (11). Therefore, modern and environmentally friendly technologies, such as the extraction of supercritical fluids (SFE), helps to give added value to the by-products of processes where different types of food participate (fruits, grains, coffee), being therefore widely used in the food industry (12).

But works using an extraction method based on Soxhlet principles are also reported, such as the work by Phimsen et al., where they obtain a 13% yield when extracting from ground coffee an oil that is used as a biohydrotreated fuel, using hexane as a solvent (13). Topala et al., compared the composition of the coffee oil extracted by Soxhlet and by SFE, using n-hexane and ethyl acetate as solvents, obtaining similar results in both processes. But in turn, better performance can be observed in less time when extracting with supercritical CO_2 and n-hexane, but when using ethyl acetate as a solvent, it is possible to identify phenolic acids (14).

The methodology used in this work is described by AOAC (15), using Soxhlet extraction with 200 mL of petroleum ether as solvent. PSE is an extraction procedure, which uses organic solvents at high pressure (70 - 140 bar) and temperature above the boiling point (50 - 200 ºC) (16). Aiming to carry out the extraction of solid sample coffee oil quickly and with a lower solvent consumption, compared to traditional methods (17). In fact, this occur because the maximization in mass transfer rates and solubility can be reached, causing the breaking in solute-matrix interactions allowed by superficial equilibrium breaking, and promoting viscosity and solvent superficial tension reduction.

In this work a "home-made" PSE system (18), simple and of low cost, was applied for coffee oil extraction of the raw beans. The data obtained with PSE were compared to those obtained using the official AOAC (Soxhlet) extraction method.

2. Materials and methods

2.1. Reagents

All reagents and solvents were analytical grade. Petroleum ether was from Merck (Rio de Janeiro, Brazil). Nitrogen was from AGA (São Paulo, Brazil).

2.2. PSE System

The PSE system (Figure 1), previously described in detail (18), consist of one pneumatic pump, wherein the solvent is placed and pressurized directly with nitrogen, different sizes stainless steel extraction cells with filters; valves; oven and connections. The pressurized solvent percolates the sample disposed in the extraction cell, removing the analytes from the sample.

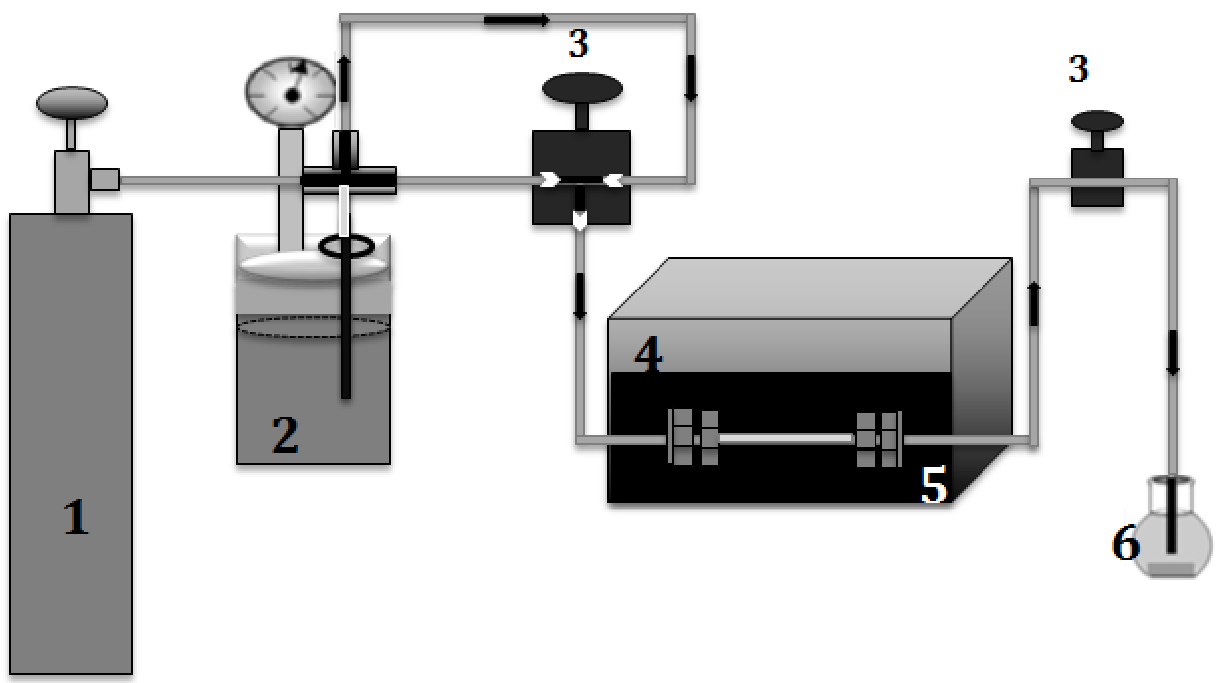

Figure 1. Representative drawing of the main components of the Pressurized Solvent Extraction (PSE) system. 1. Gas tank; 2. Solvent vessel; 3. Flow control valves; 4. Cell heater; 5. Extraction cell; 6. Collector

2.3. Sample preparation

Fifty grams of raw *arabica coffee* beans were dried in stove at 100 ºC for 5 hours. After cool, mill grinding and sieved in mesh 0.42 mm.

2.4. Soxhlet extraction

The extraction of coffee oil is carried out from 8 grams of coffee previously prepared using the Soxhlet method (in triplicate), using 200 mL of petroleum ether for 8 hours at the heat plate (30 - 60 ºC). Then solvent has been evaporated in a stove (105 ºC) until residue has been dried, for approximately 1 hour. The coffee oil content was determined, after cooling, by gravimetric method.

2.5. PSE extraction

The pressurization vessel is completed with petroleum ether and pressurized to 69 bars. Eight grams of coffee samples, in triplicate, previously described, were placed in cell extraction (30 cm x 0.8 cm. I.D.) and transferred to the heat system maintained at 80 ºC. Initially the cell was filled out with petroleum ether and five minutes were necessary for thermal equilibrium. Static extraction was done for 10 minutes and dynamic extraction for more 10 min in a flow rate of 2 mL min^{-1} approximately. After finishing the extraction, the system was purged with nitrogen to eliminate all solvent remaining in cell. The solvent has been evaporated in rotary evaporator, being the remainder in the oil-extracted residue eliminated with nitrogen. The amount of oil was determined gravimetrically.

3. Results and discussion

Table 1 describes the main data obtained for both investigated systems. It clearly shows the workability of PSE applied as an alternative for traditional methodology (Soxhlet) within 95% agreement. Comparisons also show a reduction in solvent consumption from 200 mL to 20 mL (a reduction of 10 times), as well as, in time analysis - from 480 to just 20 minutes (a reduction of over 20 times), to carry out the extraction. This reduction on time allows optimizing analytical parameters, making easier the laboratory methodology validation and the processes monitoring. Besides, the method variability as measured by the relative standard deviations of three consecutive extractions, shows a 3 times lower RSD for the PSE procedure. These reductions allow improving a coffee oil plant production by better controlling the process variables.

Table 1. Coffee oil extraction

Method	Solvent	Solvent Volume (mL)	Extraction Time (min)	Yield (% w/w)	RSD (n=3)
Soxhlet	Petroleum ether	200	480	16.23	0.40
PSE	Petroleum ether	20	20	15.97	0.15

4. Concluding remarks

The results presented in this work evidenced that the extraction conditions carried out in both methods had a significant effect on the yield of coffee oil. It was also found viable to work with the PSE system when compared to the Soxhlet system, since with the first one similar result are achieved in less time and using a lesser amount of solvent in a more inert atmosphere. These avoid the decomposition of thermally labile compounds during the process, characteristics that all together allow us to consider that PSE can be considered in this case as a green analytical chemistry method (GAC) for the coffee oil production.

Acknowledgements

The authors are grateful to São Paulo Research Foundation, FAPESP (Grants 2017/02147-0, 2015/15462-5, and 2014/07347-9) and Conselho Nacional de Desenvolvimento Cientifico e Tecnológico, CNPq (Grant 307293/2014-9) for the financial support provided. This research project was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior-Brasil (CAPES)-Finance Code 001.

Conflict of interest statement

The authors have declared no conflict of interest.

References

[1] Hurtado-Benavides A, Dorado DA, Sánchez-Camargo ADP. Study of the fatty acid profile and the aroma composition of oil obtained from roasted Colombian coffee beans by supercritical fluid extraction. J Supercrit Fluids. 2016 Jul 1;113:44–52.

[2] Chaichi M, Ghasemzadeh-Mohammadi V, Hashemi M, Mohammadi A. Furanic compounds and furfural in different coffee products by headspace liquid-phase micro-extraction followed by gas chromatography–mass spectrometry: survey and effect of brewing procedures. Food Addit Contam Part B [Internet]. 2015 Jan 2 [cited 2020 Jun 8];8(1):73–80. Available from: http://www.tandfonline.com/doi/abs/10.1080/19393210.2014.981601

[3] Lee LW, Cheong MW, Curran P, Yu B, Liu SQ. Modulation of coffee aroma via the fermentation of green coffee beans with Rhizopus oligosporus: I. Green coffee. Food Chem. 2016 Nov 15;211:916–24.

[4] Caporaso N, Whitworth MB, Cui C, Fisk ID. Variability of single bean coffee volatile compounds of Arabica and robusta roasted coffees analysed by SPME-GC-MS. Food Res Int. 2018 Jun 1;108:628–40.

[5] Díaz FO, Ormaza AM, Rojano B n. A. Effect of coffee roasting (Coffea Arabica l. var. Castillo) on cup profile, antioxidant compound content and antioxidant activity. Inf Tecnol. 2018 Aug 1;29(4):31–42.

[6] Jenkins RW, Stageman NE, Fortune CM, Chuck CJ. Effect of the type of bean, processing, and geographical location on the biodiesel produced from waste coffee grounds. Energy and Fuels. 2014 Feb 20;28(2):1166–74.

[7] Raba DN, Poiana MA, Borozan AB, Stef M, Radu F, Popa MV. Investigation on crude and high-temperature heated coffee oil by ATR-FTIR spectroscopy along with antioxidant and antimicrobial properties. PLoS One. 2015 Sep 14;10(9).

[8] Kobelnilk M, Fontanari GG, Cassimiro DL, Ribeiro CA, Crespi MS. Thermal behavior of coffee oil (Robusta and Arabica species). J Therm Anal Calorim. 2014 Mar 16;115(3):2045–52.

[9] Nosari ABFL, Lima JF, Serra OA, Freitas LAP. Improved green coffee oil antioxidant activity for cosmetical purpose by spray drying microencapsulation. Brazilian J Pharmacogn. 2015 May 1;25(3):307–11.

[10] Sajjadi B, Chen WY, Raman AAA, Ibrahim S. Microalgae lipid and biomass for biofuel production: A comprehensive review on lipid enhancement strategies and their effects on fatty acid composition. Vol. 97, Renewable and Sustainable Energy Reviews. Elsevier Ltd; 2018. p. 200–32.

[11] Khaw K-Y, Parat M-O, Shaw PN, Falconer JR. Solvent Supercritical Fluid Technologies to Extract Bioactive Compounds from Natural Sources: A Review. Molecules [Internet]. 2017 Jul 14 [cited 2020 Jun 8];22(7):1186. Available from: http://www.mdpi.com/1420-3049/22/7/1186

[12] Campos-Vega R, Loarca-Piña G, Vergara-Castañeda HA, Dave Oomah B. Spent coffee grounds: A review on current research and future prospects. Vol. 45, Trends in Food Science and Technology. Elsevier Ltd; 2015. p. 24–36.

[13] Phimsen S, Kiatkittipong W, Yamada H, Tagawa T, Kiatkittipong K, Laosiripojana N, et al. Oil extracted from spent coffee grounds for bio-hydrotreated diesel production. Energy Convers Manag. 2016 Oct 15;126:1028–36.

[14] Topala CM, Tataru LD. Infrared Spectra of Green Arabica Coffee Extraction using Supercritical Carbon Dioxide and Soxhlet Technique [Internet]. [cited 2020 Jun 8]. Available from: http://www.revistadechimie.ro

[15] Torres-Moreno M, Torrescasana E, Salas-Salvadó J, Blanch C. Nutritional composition and fatty acids profile in cocoa beans and chocolates with different geographical origin and processing conditions. Food Chem. 2015 Jan 1;166:125–32.

[16] Efthymiopoulos I, Hellier P, Ladommatos N, Russo-Profili A, Eveleigh A, Aliev A, et al. Influence of solvent selection and extraction temperature on yield and composition of lipids extracted from spent coffee grounds. Ind Crops Prod. 2018 Sep 1;119:49–56.

[17] Armenta S, Garrigues S, de la Guardia M. The role of green extraction techniques in Green Analytical Chemistry. Vol. 71, TrAC - Trends in Analytical Chemistry. Elsevier B.V.; 2015. p. 2–8.

[18] Pinto JSS, Lanças FM. Design, construction and evaluation of a simple pressurized solvent extraction system. J Braz Chem Soc. 2009;20(5):913–7.