Food Chemistry 213 (2016) 425–430

Contents lists available at ScienceDirect

Food Chemistry

journal homepage: www.elsevier.com/locate/foodchem

Pressurized liquid extraction and chemical characterization of safflower oil: A comparison between methods

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ARTICLE INFO

Article history:
Received 27 January 2016
Received in revised form 10 May 2016
Accepted 30 June 2016
Available online 1 July 2016

Keywords:
Pressurized liquid extraction
Safflower oil
Chemical characterization

ABSTRACT

This work investigates the extraction process of safflower oil using pressurized ethanol, and compares the chemical composition obtained (in terms of fatty acids) with other extraction techniques. Soxhlet and Ultrasound showed maximum global yield of 36.53% and 30.41%, respectively (70 °C and 240 min). PLE presented maximum global yields of 25.62% (3 mL min^{-1}), 19.94% (2 mL min^{-1}) and 12.37% (1 mL min^{-1}) at 40 °C, 100 bar and 60 min. Palmitic acid showed the lower concentration in all experimental conditions (from 5.70% to 7.17%); Stearic and Linoleic acid presented intermediate concentrations (from 2.93% to 25.09% and 14.09% to 19.06%, respectively); Oleic acid showed higher composition (from 55.12% to 83.26%). Differences between percentages of fatty acids, depending on method were observed. Results may be applied to maximize global yields and select fatty acids, reducing the energetic costs and process time.

1. Introduction

Safflower (*Carthamus tinctorius* L.), a plant of the *Compositae* family, has been widely used in the food industry in cooking oil, salad dressing, margarine production and colorant. In China, Egypt, United States, Mexico and Brazil, safflower is cultivated as oilseed and its poly-unsaturation becomes an important chemical characteristic. Safflower oil has saturated (palmitic, C16:0 and stearic C18:0) and unsaturated (oleic acid – C18:1, linoleic – C18:2 and linolenic – C18:3) fatty acids present in its the composition (Camas & Esendal, 2006; Yeilaghi et al., 2012). It is known that Oleic acid has desirable characteristics as frying stability and mild flavor, while the linoleic acid reduces the cholesterol level in the blood (Smith, 1993; Wilson, Nicolosi, Saati, Kotyla, & Kritchevsky, 2006). Additionally, Fan et al. (2009) cited the effects of safflower extracts as anticoagulant, antitumor, antihypertensive, antioxidant, neuroprotective, liver protectant, and inhibitor of melanin production. Thus, the safflower became a focus of study in scientific literature.

The content of oleic and linoleic acid in safflower oil may reach values ranging from 8 to 85%, depending on the extraction method employed (Han, Cheng, Zhang, & Bi, 2009). The oily fraction may be obtained from traditional approach (methods recommended by medicinal plant pharmacopeia, e.g. steam and water distillation, Soxhlet extraction, maceration, percolation, expression, cold fat extraction). However, these methods have several shortcomings, including long extraction time, excessive solvent consumption, water for cooling and electric energy (Dawidowicz & Wianowska, 2005). Hexane, a commonly employed organic solvent extracting, leads not only to toxicity problems in the residual matrix and extract, but also requires a further purification step. Furthermore, hexane has been considered as an hazardous air pollutant by the United State Environmental Protection Agency (US EPA) and more than 20 million kg of hexane are released to the atmosphere each year due to the extraction of vegetable oils (DeSimone, 2002). Therefore, several approaches are continuously investigated in order to improve these conditions, allowing high yields, fast and clean. In this respect, we highlighted the use of the microwave, supercritical fluid, ultrasound and pressurized liquids in the extraction of various compounds.

Ultrasound-assisted extraction (UAE) may be used to several goals and the range of published extraction applications include

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http://dx.doi.org/10.1016/j.foodchem.2016.06.111
0308-8146/

herbal, oil, protein and bioactive from plant materials (e.g. flavones, polyphenols) (Vilkhu, Mawson, Simons, & Bates, 2008). This action is due to cavitation, which generates high forces and microbubbles that enhances surface erosion, fragmentation and mass transfer resulting in high yield and fast rate of extraction. Moreover, it is possible to adopt mild processing conditions of temperature and pressure using GRAS ("Generally Recognized As Safe") solvents, causing minimum effect on extractable materials when compared to the conventional routes. The collapse from cavitation bubbles near the cell walls produces cell disruption and high penetration of the solvent into the cells (Awad, Moharram, Shaltout, Asker, & Youssef, 2012; Da Porto, Porretto, & Decorti, 2013; Toma, Vinatoru, Paniwnyk, & Mason, 2001; Vilkhu et al., 2008).

On the other hand, pressurized liquid extraction (PLE), also known as accelerated solvent extraction, appeared as alternative for extraction, since it allows for faster extraction and reduces solvent consumption through a "green" technology (Machado, Pasquel-Reátegui, Barbero, & Martínez, 2015). PLE may be conceptualized as a process that combines temperature and pressure with liquid solvents to achieve rapid and efficient extraction of analytes from several matrices (Sanagi, See, Ibrahim, & Naim, 2005).

Hence, the main objective of this work is to study the extraction of safflower oil (*Carthamus tinctorius* L.) using pressurized ethanol, and comparing the chemical composition obtained (in terms of fatty acids) with other extraction techniques (Soxhlet and UAE).

2. Experimental

2.1. Materials

Ethanol (99.5% of purity) and fatty acid methyl ester mix (code 18913-1AMP, Supelco®) were purchased from Dinamica and Sigma-Aldrich (St. Louis, USA), respectively. Safflower oil (commercial) and safflower seeds (lot number 2200-1) were obtained from Pazze Alimentos (Panambi, RS, Brazil). Commercial safflower oil was analyzed as received. Safflower seeds were dried in an air-circulation oven at 30 °C ± 1 °C/48 h and ground in a cyclone mill (Marconi, MA-020). The material not retained on a 10-mesh sieve were stored in glass containers and placed in a domestic refrigerator until further use.

2.2. Soxhlet extraction

This approach was performed according to a methodology described by Instituto Adolf Lutz (2008), with some modifications. Briefly, 5 g of safflower seeds were placed inside a cellulose-filter paper cartridge and extracted with 180 mL of ethanol at 80 °C. Reflux was kept for 60 min and 240 min. After extraction, the solution was evaporated in an air-circulation oven and placed in a desiccator overnight. After reaching constant weight, the extracted mass was measure with an analytical balance. The global yield (G_0) was calculated by the ratio between the extracted mass (Han, Cheng, Zhang, & Bi) in dry basis and the initial mass fed into the extraction cell (I_m), according to Eq. (1). Assays were performed in triplicate.

$$G_o = \frac{E_m}{I_m} \times 100 \quad (1)$$

2.3. Ultrasound-assisted extraction (UAE)

Extraction process using ultrasound was carried out according to Filippi, Bilibio, Bender, Carniel, and Priamo (2015). This process used an ultrasonic bath (model SB-5200DTDN, Scientz), frequency of 40 kHz, power output of 250 W and ultrasonic transducer fitted at the lower extremities in the inner tank (L × W × H: 300 × 240 × 150 mm). The extraction procedure was developed as follows: ethanol was added in an Erlenmeyer flask (protected at the top with plastic wrap to prevent solvent evaporation) and the bath temperature was adjusted to 70 °C (below its boiling point). After reaching the desirable temperature, an amount of safflower seeds (previously weighed) was placed in a permeable filter and added to the system. Safflower oil was extracted during 60 min and 240 min, following the same previous procedure to quantify the extracted mass. Assays were performed in triplicate.

2.4. Pressurized liquid extraction (PLE)

The schematic diagram of the PLE unit (continuous mode) is presented in Fig. 1 and similar to Pitipanapong et al. (2007). The system is composed by: (S) solution reservoir; B: thermostatic bath (521-3D, Nova Etica); LC: digital HPLC pump (Shimadzu, LC-20AT) that operates at a flow rate of 0.01–10.0 mL min^{-1} and used is for organic solution delivery; E: stainless steel extraction cell (with an internal volume of 7.5 mL, heating jacket to keep the water circulation in order to keep the temperature extraction), projected to operate at pressure and temperature up to 400 bar and 250 °C, respectively; VA: needle valve (MS-01164, Swagelok); C: borosilicate collection vessel. All pipes and connections were of stainless steel (1/8″). The procedure is similar to the one presented by Machado et al. (2015) with some modifications: approximately 5 g of safflower seeds was placed into the extraction cell containing glass spheres. In the cell outlets, two stainless steel filters were used to avoid the carrying over of particles. The experimental assay began with: (*i*) the connections in the cell outlets, heating jacket in the thermostatic bath, and adjustment of temperature; (*ii*) after the system reaches the desirable temperature (40 or 30 °C), the needle valve was opened and the solvent was pumped into the extraction cell at a specific flow rate (1, 2 or 3 mL.min^{-1}); (*iii*) when the solvent was visualized in the pipe output, the needle valve was closed, allowing the system reach the extraction pressure (100 bar or 200 bar). After being kept in equilibrium for 1 h to stabilize the system, VA was opened and the extraction began; (*v*) extraction flow rate was monitored directly in the LC and the pressure was controlled by VA manipulation and also monitored by the LC display. At scheduled time intervals (up to 60 min) aliquots were collected in a vessel, and then evaporated in an air-circulation oven and placed in a desiccator overnight. At the end of the experiment, the system was slowly depressurized to atmospheric pressure. The oil mass extracted was determined by measuring it an analytical balance after reaching a constant weight, according to Eq. (1).

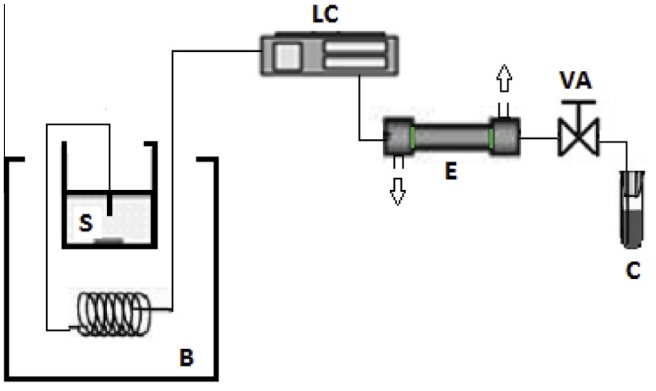

Fig. 1. Schematic diagram of the PLE technique. S: Solvent reservoir; B: Thermostatic bath; LC: Liquid pump; E: Extraction cell with heating jacket, VA: Needle valve; C: collection vessel.