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Alternative to traditional olive pomace oil extraction systems: Microwave-assisted solvent extraction of oil from wet olive pomace

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ABSTRACT

The microwave assisted solvent extraction parameters of olive pomace oil was investigated. The oil extracted was compared with that obtained by the conventional industrial extraction method in terms of yield and some physical and chemical properties. This is the first attempt to extract oil from wet olive pomace directly using a closed vessel pressurized microwave system. The individual and interaction effects of independent parameters on extraction yield were analyzed by using response surface methodology. The microwave power (150–300 W), irradiation time (5–20 min), solvent-to-sample ratio (2–10) were identified as significant independent variables. Optimum extraction conditions were 287 W, 16 min and 10:1 (solvent-to-sample ratio). The maximum oil yield obtained at these optimum conditions was 6.85 g/100 g dry matter. Although the wet olive pomace was processed in microwave extraction the yield of oil was higher than that of oil extracted by the conventional industrial extraction method from dry olive pomace. In addition, total phenolic content (985 mg caffeic acid/kg oil) and amount of tocopherols (278.07 mg/kg oil) were higher while peroxide value (17.8 meq O_2/kg oil) and polycyclic aromatic hydrocarbons content (0.44 μg benzo[α]pyrene/kg oil) were lower in microwave assisted solvent extraction compared to conventional industrial extraction method.

1. Introduction

Olive pomace is a by-product of the olive oil industry. Around 35–40 g/100 g of processed olives are released as solid waste (pomace) which contains water, oil, olive skin, olive pulp, and kernels (Akay, Kazan, Celiktas, & Yeşil Celiktas, 2015). Although olive pomace can be utilized for animal feed supplement and energy production or disposed on the field, its economic interest is primarily due to residual oil (Meziane, 2013). The residual oil content of pomace is around 4–15 g/100 g dry matter according to cultivation region of olive and the extraction method (Akay, Kazan, Celiktas, & Yesil-Celiktas, 2015). The moisture content of released pomace depends heavily on whether the extraction system used is two-phase or three-phase. However, drying is essential in both cases because the crude olive pomace oil is generally recovered by solvent extraction in conventional industrial oil extraction (CE) method.

In CE, drying of pomace is one of the most energy intensive operations and includes high temperature (400–800 °C) exposures (Sánchez Moral & Ruız Mendez, 2006). The rotary dryer that heated up by a hot gas stream is the most commonly used drying system in which both the pomace to be dried and the hot drying gases are introduced into the drying chamber (Montero, Miranda, Arranz, & Rojas, 2011). This application causes alteration of the double bonds in the hydrocarbon chains of chlorophyll that result in oil with intense green colors. Drying stage also accounts for an increase in the content of oxidized compounds, significantly higher formation of conjugated dienes, oxidized triglycerides, formation of triglyceride dimers and polymers (Gomes & Caponio, 1997; Göğüş & Maskan, 2006). Polycyclic aromatic hydrocarbons (PAHs) can also be found in crude pomace oil because of the direct contact of combustion fumes with pomace and the polymerization of the sugars at drying temperatures above 400 °C (Guillén, Sopelana, & Palencia, 2004; León-Camacho, Viera-Alcaide, & Ruiz-Méndez, 2003; Skupinska, Misiewicz, & Guttman, 2004).

Microwave is an emerging technology that is used in many food processing applications to improve their efficiency (Chandrasekaran, Ramanathan, & Basak, 2013; Knorr et al., 2011; Tamborrino, Romaniello, Zagaria, & Leone, 2014). For instance, microwave-assisted solvent extraction (MASE) can be used in oil extraction processes as an alternative to traditional solvent

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extraction. The extraction of oil from the olive cake (Amarni & Kadi, 2010), the green coffee (Tsukui et al., 2014) and cottonseed (Taghvaei, Jafari, Assadpoor, Nowrouzieh, & Alishah, 2014) has been studied using MASE. The fast heating and destruction of the biological cell structure of plant tissues under microwave conditions provide very powerful extraction procedures in a shorter time (Azadmard-Damirchi, Habibi-Nodeh, Hesari, Nemati, & Fathi 2010; Clodoveo & Hbaieb, 2013). The microwave implementation in different oil extraction processes has been found as a promising technology by a number of recently published studies due to some benefits such as improved oil and phenolic compound extraction yield (Clodoveo & Hbaieb, 2013; Taghvaei et al., 2014), low oxidation (Tamborrino et al., 2014) and reduced solvent consumption (Tsukui et al., 2014). Amarni and Kadi (2010) studied the microwave assisted solvent extraction of oil from olive cake. However the microwave system used in this study was a modified domestic microwave oven, which is very basic and different than the closed vessel pressurized microwave. Moreover, in mentioned study dry pomace was used to extract oil. Most of the other studies present in the literature are based on the CE of pomace oil and drying characteristics of olive pomace.

So far, there are no studies on extraction of oil from wet olive pomace by the pressurized microwave system in literature. The main aim of this study was to evaluate the effect of extraction parameters on the oil yield in MASE. It was also aimed to compare the yield and quality of oil obtained using MASE with those of CE technique. The extraction yield of olive pomace oil was optimized by using response surface methodology (RSM). The quality of the oil was evaluated in terms of the free fatty acids (FFA), peroxide value (PV), benzo[α]pyrene (BaP) content, wax content, total phenolic content, tocopherol content, color and fatty acid composition.

2. Materials and methods

2.1. Materials

Wet olive pomace (after two-phase decantation, Polat Makina, PMS 350/PX 30, Aydın, Turkey), freshly extracted pomace oil and pomace residue (oil free pomace) were obtained from the Güvenal Olive Oil Company located at Gaziantep, Turkey. The wet olive pomace had a moisture content of 52.7 g/100 g and an oil content of 7.05 g/100 g dry matter.

2.2. Chemicals

Standard samples of benzo[α]pyrene (≥96 g/100 g, HPLC), C32 wax ester, Supelco® 37 Fatty acid methyl ester (FAME) mixture component, α-, β-, γ- and δ-Tocopherols, Folin-Ciocalteau phenol reagent, caffeic acid, and silica gel (SG 60, 70–230 mesh) were purchased from Sigma–Aldrich (St. Louis, MO, USA). All solvents (acetonitrile, *n*-hexane, *n*-heptane, diethylether, 2-propanol) used for HPLC and GC analysis were of chromatographic grade. Other reagents and solvents were of analytical grade. Water was purified by an ultrapure water system of Millipore (Milli-Q system, Millipore, Bedford, MA, USA) for HPLC applications.

2.3. Total oil content of pomace

The olive pomace (10 g) was dried to the constant weight in an oven at 103 °C prior to oil extraction. Then, extraction was carried out by Soxhlet apparatus using *n*-hexane according to ISO (2006-a). The extraction was carried out in triplicate under the same conditions.

2.4. Conventional industrial extraction of olive pomace oil

The olive pomace obtained by two-phase extraction system was immediately dried in the countercurrent rotary dryer in the Güvenal Olive Oil Company. In this drying system, the residual pomace (oil free) was used as fuel. The hot flue gas obtained from combustion of this fuel was used as a drying medium for wet olive pomace. The dried pomace pulp was granulated and then oil was extracted by using semi-continuous solvent extraction (hexane). The crude olive pomace oil was obtained after the distillation of the miscella.

2.5. Microwave-assisted solvent extraction of oil

Closed vessel pressurized microwave system (Discover SP, CEM, Matthews, USA) operating at 0–300 W microwave output with 2450 MHz and 0–2.07 × 10^3 kPa pressure control was used. This microwave system consisted of an infrared sensor for temperature measurement coupled with an electromagnetic stirring with adjustable speeds and air cooling for simultaneous cooling. Experiments were carried out always under stirring (600 rpm) using closed 35 mL Pyrex vials. For extraction, the wet pomace samples were subjected to microwave by adding different volume of *n*-hexane at 150, 225 and 300 W. The mass of wet pomace (2.5 g) in the vial was kept constant for each experiment. After the irradiation treatment, the miscella was removed from the olive pomace residue by means of decanting followed by centrifugation (Hettich -EBA 20, Andreas Hettich GmbH & Co. KG,Germany) at 6000 rpm (3461 × *g*) for 10 min. The liquid extract layer was dried over anhydrous $MgSO_4$ and *n*-hexane was evaporated using a rotary vacuum evaporator (Heidolph, Hei-VAP Advantage, Heidolph Instruments GmbH & Co. KG, Germany) set to 40 °C and 200 rpm. The obtained oil was stored at −20 °C until the analysis.

2.6. Experimental design and optimization by response surface methodology

The central composite face-centered design with three factors and one response variable was developed as shown in Table 1. Face centered design was composed of 20 experiments including six replicates at the central point. All experiments were performed in randomized order according to the run number as arranged by the software. Microwave power (150–300 W), time (5–20 min) and solvent-to-sample ratio (2–10) were investigated as the independent variables. These selected upper and lower limits were based on the values obtained in preliminary experiments.

RSM (Stat-Ease, Design-Expert software, version 7) was used for ANOVA, regression analysis, optimization and model generation. The quadratic response surface model equation was fitted to the data obtained. The confidence level was kept at 99% for model while it was set at 95% for all other tests. The optimum extraction conditions were specified based on the maximum extraction efficiency while the other factors were kept in the studied range.

2.7. Physical and chemical properties of oils

2.7.1. Fatty acid content and peroxide value

The free fatty acid content and peroxide value of the pomace oils were determined according to analytical methods described in AOCS (1997-a) and AOCS (1997-b), respectively.

2.7.2. Moisture content

The moisture content of fresh pomace was determined by keeping 10 g of a sample in an oven at 103 °C until constant weight.