Monitoring the moisture reduction and status of bioactive compounds in extra-virgin olive oil over the industrial filtration process

Abdelhakim Bakhoucha a,b, Jesús Lozano-Sánchez a,b,c,*, Cristianó Augusto Ballus d, Melchórt Martínez-García e, Miguélá González Velascó e, Álvaro Olavarría Govantes e, Tullía Gállina-Toschi c, Alberto Fernández-Gutiérrez a,b, Antonio Segura-Carretero a,b

a Department of Analytical Chemistry, Faculty of Sciences, University of Granada, Fuentenueva s/n, E-18071 Granada, Spain
b Research and Development Functional Food Center, Health Science Technological Park, Avenida del Conocimiento s/n, E-18016 Granada, Spain
c Department of Agricultural and Food Sciences, Alma Mater Studiorum, University of Bologna, Piazza Gobbiánich, 60, I-47521 Cesena, FC, Italy
d Department of Food Science, Faculty of Food Engineering, University of Campinas (UNICAMP), Rua Monteiro Lobato, 20, 13083-862 Campinas, SP, Brazil
e Oleoestepa, S.C.A., P.I. Sierra Sur, c/El Olivo, s/n, Estepa, Seville, Spain

* Corresponding author. Department of Analytical Chemistry, Faculty of Sciences, University of Granada, Fuentenueva s/n, E-18071 Granada, Spain. Tel./fax: +34 958249510.
E-mail address: jesusls@ugr.es (J. Lozano-Sánchez).

A R T I C L E   I N F O
Article history:
Received 9 October 2013
Received in revised form 9 December 2013
Accepted 10 December 2013

Keywords:
Extra-virgin olive oil
Filtration
Moisture
Phenolic compounds
Quality

A B S T R A C T
The aim of this study was to evaluate extra-virgin olive oil (EVOO) moisture and phenolic compounds content during industrial filtration, which is widely applied in the most olive-oil industries of the main producing countries of the Mediterranean as a final step prior to selling the oil. For this purpose, conventional filtration process was performed in duplicate using two lots (lot 1 and lot 2), for a total amount of 45,000 kg of EVOO each. The EVOOs were from the main Spanish olive varieties (Hojiblanca, Manzanilla, Picual, and Arbequina). Cloudy EVOOs were filtered using Vitacel® L-90 and Filtracel® EFC-950 as filter aids together with filtration tank. The moisture content was determined in unfiltered and filtered EVOOs. In addition, the individual phenolic compounds were qualitatively and quantitatively characterized by HPLC–ESI-TOF/MS. The results clearly showed that filtration sharply decreased moisture. Nevertheless, the time course of phenolic compounds during filtration differed for each family. Whereas phenolic alcohols and flavones decreased during filtration, secoiridoids tended to increase, while lignans were the least affected group. Although filtration can make EVOO brilliant and can increase its shelf life by reducing its moisture content, filtration sacrifices certain phenolic compounds which could affect EVOO oxidative stability and its nutritional quality. Consequently, to maintain olive-oil quality, producers need to take into account both moisture loss as well as the antioxidant content during EVOO filtration.

© 2013 Elsevier Ltd. All rights reserved.

1. Introduction
Extra-virgin olive oil (EVOO) is a natural product obtained from pressing olive fruit (Olea europaea) (Gordillo, Ciacccheri, Mignani, González-Miret, & Heredia, 2011). The characteristic aroma, taste, and color of this oil distinguish it from other edible vegetable oils. The excellent organoleptic and nutritional properties of EVOO, together with the current tendency of consumers to select minimally processed foods, have prompted a re-assessment of its consumption in the daily diet (Fregapane, Lavelli, León, Kapuralin, & Desamparados Salvador, 2006). Furthermore, the increase of EVOO intake has also been related to the healthier properties of some minor constituents, such as phenolic compounds (Tripoli et al., 2005).

EVOO quality has been related to its composition, and the moisture content is considered one of the main parameters to evaluate this quality (Gordillo et al., 2011; Hatzakis & Dais, 2008; Ruiz-Dominguez, Raigón, & Prohens, 2013). Freshly produced EVOO is naturally turbid, containing micro-droplets of vegetation water and solid particles from olive fruits (Brkić Bubola, Koprivnjak, & Sladonja, 2012). Although, it could be considered by some consumers less processed, the higher water content in water-in-oil emulsion maintains the stability of suspended solids for several weeks or even months until complete deposition (Gordillo et al., 2011). Indeed, the high polar phase content (water) may augment the alteration of EVOO during storage in milling companies and throughout the market period, by increasing the hydrolytic rate of the triacylglycerols. This process increases free acidity, exposing
EVOO to oxidation in the presence of oxygen, light or high temperature (Yun & Surh, 2012). It is well known that oxidation leads to the formation of volatile products, which not only change the initial flavor of EVOO but also decrease the nutritional quality and may even lead to the formation of toxic products (Bendini et al., 2013; Stefanoudaki, Williams, & Harwood, 2010). Additionally, the acidity of cloudy EVOO affects the time course of phenolic compounds over storage by increasing the degradation of the secoiridoid group (Brenes, García, García, & Garrido, 2001). On the other hand, the cloudy aspect makes sales difficult in some new markets, where consumers tend to prefer brilliant EVOO.

In recent years, most companies prior to bottling and sales apply a filtration step to remove suspended solids and reduce EVOO moisture content. The objective is to maintain EVOO quality and increase its shelf life before consumption. Besides protecting EVOO from chemical degradation by reducing its water content, the filtration step makes it more brilliant for consumer acceptance. In this sense, different filtration systems have been applied in the olive-oil industry: conventional filtration systems (filter tanks and filter presses), cross-flow filtration (tangential-flow filtration), inert-gas-flow filtration systems, and filter bags (Frankel, Bakhouche, Lozano-Sánchez, Segura-Carretero, & Fernández-Gutiérrez, 2013; Lozano-Sánchez et al., 2012).

Nevertheless, reducing the moisture content could affect the polar fraction of EVOO responsible of its oxidative stability. Phenolic compounds are the main components of this fraction with a strong antioxidant effect (Alacón Flores, Romero-González, Garrido Frenich, & Martínez Vidal, 2012; Anastasopoulos et al., 2011; Zaneti et al., 2013). Therefore, the time course of these two parameters during filtration needs to be studied. The aim of this work was to monitor moisture and individual phenolic compounds during EVOO filtration step at industrial scale. This is the first study available in which the effect of the industrial filtration process on phenolic compounds has been evaluated step by step using a high-performance liquid chromatography (HPLC) coupled to electrospray time-of-flight mass spectrometry (TOF-MS). The industrial filtration system evaluated is widely applied in most EVOO industries of the main producing countries of the Mediterranean.

2. Materials and methods

2.1. Samples

The EVOOs used in this study were obtained in November 2012 from industrial mills equipped with a hammer crusher, a horizontal malaxator, and a two-phase decanter (Oleostepa S.L., Seville, Spain). For this work, the industrial filtration was performed in duplicate using two lots (lot 1 and lot 2), with a total amount of 45,000 kg of EVOOs each. The first consisted of EVOOs from the olive varieties Hojiblanca (52%), and Manzanilla (48%) and the second one from Hojiblanca (40%), Picual (40%), and Arbequina (20%) olive varieties. Both mixtures of cloudy EVOOs were filtered at room temperature using the following organic filter aids: Vitacel® L-90 (30 kg, composed of 100% cellulose) and Filtrace® EFC-950 (60 kg, composed of 70% cellulose and 30% lignin). The cake layer was performed in conjunction with filter tank. For the filtration, each lot underwent a preliminary phase of filtering through specially prepared combinations of filter aids and EVOO. In this preliminary step, the filtration equipment was covered with organic filter aids and the cake layer was formed. Afterward, filtration was conducted under a constant flow and increasing differential pressure. A total of 48 filtered and unfiltered samples were collected from both lots 1 and 2 following the procedure depicted in Fig. 1. A same analysis was performed to represent the results and eliminating confounding factors which could affect EVOO composition, the moisture content measured and the phenolic fraction was isolated from samples without storage.

2.2. Chemicals and reagents

All chemicals were of analytical reagent grade. Methanol, n-hexane, sodium hydroxide and isopropanol were purchased from...