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Compositional differences between veiled and filtered virgin olive oils during a simulated shelf life

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ABSTRACT

The main chemical and physical parameters of veiled and filtered virgin olive oils (VOOs) that are linked to its health and sensory properties, such as phenolic and volatile compounds, were evaluated during a four-month simulated shelf life at room temperature during which the oils were exposed to diffused light for 12 h per day. The specific settings of the vertical centrifuge used to treat the four industrial VOO samples extracted in different Mediterranean areas determined the "veiling" stabilization and reduced the formation of deposits at the bottom of the oil bottles. Cryo-SEM of the veiled oils showed the presence of micro-dispersed water particles that did not contain apparent vegetable fragments. By the end of the storage period, the changes in the quality parameters showed no negative effects on the oxidative stability of the veiled oils compared to the filtered oils. A higher phenolic concentration of Tunisian, Spanish, Greek and Italian veiled VOOs (50.8, 110.1, 389.6 and 389.4 mg/kg, respectively) was detected at the end of storage period compared to filtered samples (20.1, 83.2, 196.1 and 209.6 mg/kg, respectively).

1. Introduction

The main operators in the olive oil sector are engaged in ensuring the production of virgin olive oil (VOO) with high quality characteristics. Indeed, the production of VOO requires a cooperative effort to maintain and improve the health and sensory features that are indicative of a top-quality product and the careful selection of the most appropriate agronomic and technological practices. This is possible thanks to a continuous stream of recent research publications and studies on the different variables involved in the VOO production process (Almeida, Valli, Bendini, & Gallina Toschi, 2017; Caruso et al., 2017; Jimenez, Sánchez-Ortiz, Lorenzo, & Rivas, 2015; Servili et al., 2015; Veneziani et al., 2017). Agronomic and technological innovations are increasingly being accompanied by studies related to the different techniques able for preserving the quality parameters of VOO after mechanical extraction and during its storage period (Fortini et al., 2016; Guerrini & Parenti, 2016; Jabeur, Zribi, & Bouaziz, 2017; Krichene, Salvador, & Fregapane, 2015; Sacchi, Caporaso, Paduano, & Genovese, 2015). Appropriate selection of packaging material and good practices in preservation and stabilization are directly connected to the market demand and consumer's requirements, and these parameters are equally important for producing a high-quality VOO. Currently, there is increasing interest in veiled VOOs that appear more natural and less processed, which makes consumer's perception of the quality of the product higher not only for its potential health properties but also for visual attributes and superior organoleptic characteristics (Gordillo, Ciaccheri, Mignani, Gonzalez-Miret, & Heredia, 2011; Koidis, Triantafillou, & Boskou, 2008). The structure and physicochemical composition of veiled oils are highly influenced by mechanical extraction processes. Indeed, the natural turbidity, due to microdroplets of vegetation water and small solid fragments of olive peel and pulp, is influenced by the use of a two- or three-phase extraction system, that modify moisture contents and cloudy levels, and the subsequent vertical centrifugation step (Guerrini & Parenti, 2016; Papadimitriou et al., 2013). The fresh olive oil is a water-oil emulsion in which the different substances present, including phenols, proteins (Koidis & Boskou, 2006; Lerker, Frega, Bocci, & Servidio, 1994), sugars (glucosides or protein glycation products as described by Papadimitriou et al., 2013) and emulsifying agents (phospholipids and mono- and diacylglycerols (Koidis et al., 2008)), aggregate at the interphase between the oil and vegetation water and participating in the formation of the colloidal system, making them responsible for the stability of the veiled oil (Guerrini & Parenti, 2016). The traditional and new filtration processes (Bakhouche, Lozano-Sanchez, Ballus, Martinez-Garcia, 2014; Guerrini,

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Masella, Migliorini, Cherubini, & Parenti, 2015; Lozano-Sanchez et al., 2012), usually used after the vertical centrifugation phase, are a controversial subject. Veiled VOOs are characterized by some positive characteristics; however, there are other negative aspects connected to the quality of VOOs that can be removed by filtration. The veiled VOO appears to be a product with greater health and sensory properties due to its higher phenolic content, which ensures increased stability and antioxidant activity (Esposto et al., 2015; Gómez-Caravaca et al., 2007; Papadimitriou et al., 2013; Taticchi et al., 2017) compared to the corresponding filtered oils (Brkić Bubola, Lukić, Mofardin, Butumović, & Koprivnjak, 2017; Lozano-Sánchez, Segura-Carretero, & Fernández-Gutiérrez, 2011; Papadimitriou et al., 2013; Tsimidou, Georgiou, Koidis, & Boskou, 2005). Some studies showed increases in the contents of oleuropein and ligstroside derivatives in the filtered oils as a consequence of the stabilization treatment (Bakhouche, Lozano-Sanchez, Ballus, Martinez-Garcia, 2014; Fortini et al., 2016; Gómez-Caravaca et al., 2007; Jabeur et al., 2017). This trend, which often contradicts the results of the oxidative stability, was explained in other reports by the fact that the reduction of the oil moisture content during the filtration phase determine a analytical artifact into the extraction method used to assess the VOO secoiridoids, and therefore the procedure was not able to effectively extract the right amount of phenolic compounds (Bakhouche, Lozano-Sanchez, Ballus, Bendini, 2014; Lozano-Sánchez et al., 2012). The unfiltered oils could also have negative organoleptic properties due to the different contents of residual vegetation water that could impact fermentation. The growth of indigenous microflora, mainly yeasts and molds (Cadez et al., 2012; Guerrini & Parenti, 2016; Koidis et al., 2008), can generate off-flavor substances, which reduces the quality of the veiled VOO during the storage period.

Hence, this study evaluates the compositional differences between veiled olive oils, which are obtained from specific settings of vertical centrifuge, and filtered olive oils produced in different Mediterranean areas (Tunisia, Spain, Greece and Italy). The main goal of this study is the determination of the evolution of the phenolic and volatile fractions of VOO that have a significant influence on the health and sensory properties of high-quality olive oils. Oils characterized by different chemical composition were packaged in clear glass bottles and monitored during a storage period of 4 months at room temperature, and during this period they were exposed to diffused light for 12 h per day.

2. Materials and methods

2.1. Veiled and filtered VOO

This study was carried out using four industrial oils extracted from olives harvested in different Mediterranean area (area of Sfax, Tunisia; Andalusia region, Spain; Creta island, Greece; Apulia region, Italy) representing different cultivar: Chemlali and Chetoui in Tunisian oil, Arbequina and Picual in Spanish oil, Koroneiki in Greek oil, Coratina and Ogliarola Garganica in Italian oil. The harvested period was: the first weeks of November in Greece and Italy, the first weeks of December in Tunisia and the end of November in Spain. The olives were processed by industrial extraction plants using two-phase systems in Spain, Greece and Italy and using a three-phase system in Tunisia (Kalogeropoulos, Kaliora, Artemiou, & Giogios, 2014). All the VOOs, obtained from the different extraction plants were then centrifuged using an Alfa Laval UVPX507AGT-14 (Alfa Laval SpA, Tavarnelle Val di Pesa (FI), Italy) with a speed of 6300 rpm and a gravity disc of 98 mm. The temperature was maintained at 25 °C using an Alfa Laval Thermal Conditioning Module prior to beginning the centrifugation step. Approximately 1000 kg of each VOO was centrifuged to prepare the veiled samples, and 500 kg of each veiled oil was stabilized by filtration using a Wolhfarth "Farminox" (BRUNO WOLHFARTH srl, Sordio (LO), Italy) system with IF450 filter papers to obtain the filtered samples. About 30 L of each VOO obtained was packaged in a clear, 0.5 L glass bottle, stored at 25 °C and exposed to diffused light 12 h per day using automatically controlled 58 W and 300 lux neon bulbs. The bottles were moved every week to standardize the impact of photooxidation on the products. Each month, three veiled and filtered samples of the Tunisian, Spanish, Greek and Italian oils were analyzed to determine their turbidity, legal quality parameters, and their contents of phenolic and volatile compounds.

2.2. VOO analyses

2.2.1. Moisture content

The moisture contents in the veiled and filtered VOOs were evaluated following evaluated following the ISO662 method (ISO662, 1998).

2.2.2. Turbidity grade

The level of turbidity in the VOO was determined in nephelometric turbidity units (NTU) About 25 g of VOO was put in a glass vessel and inserted in a closed vessel chamber of a Ratio Turbidimeter Hach Model 18900 (Hach Company Loveland, Colorado, US) to detect the turbidity grade.

2.2.3. Quality indices

The free acidity, peroxide value and UV absorption characteristics (K_{232}, K_{270} and ΔK) were determined according to the analysis methods described by Regulation 1989/03 (OJEC, 2003).

2.2.4. Phenolic compounds

The extraction and HPLC analysis of the phenolic compounds were carried out as reported by Selvaggini et al. (2014) using a Spherisorb ODS1 column (5 µm, 4,6 mm × 250 mm, Waters, Milford, MA, USA). The HPLC instrument, an Agilent Technologies system Model 1100 (Agilent Technologies, Santa Clara, CA, USA), was equipped with a vacuum degasser, a quaternary pump, an autosampler, a thermostated column compartment, a diode array detector (DAD), and a fluorescence detector (FLD). ChemStation software (Agilent Technologies, Palo Alto, CA, USA) was used for instrument control and data processing.

2.2.5. Volatile compounds

The main flavor and off-flavor volatile compounds of the veiled and filtered VOOs were assessed and quantified by headspace solid-phase microextraction followed by gas chromatography − mass spectrometry (HS-SPME/GC-MS). GC-MS analysis was performed using a Varian 4000 GC-MS controlled by Varian MS Workstation Software, Version 6.6. The operating conditions for the analysis and the identification of the volatile compounds were as described by Veneziani et al. (2015).

2.3. Cryo-SEM and energy-dispersive X-ray microanalysis of veiled and filtered VOOs

Portions of the veiled and filtered VOOs were quickly frozen in liquid nitrogen, and they were stored frozen until analysis. A frozen hydrated (FH) sample was mounted under liquid nitrogen gas on an aluminum stub with Tissue-Tek, transferred to a dedicated cryo-preparation chamber (SEM cryo-unit, SCU 020, Bal-Tech, Balzers, Liechtenstein), freeze-fractured by a motor-driven fracturing microtome at −120 °C, surface-etched for 3 min at −80 °C under high vacuum (P < 2 × 10-4 Pa), and sputter-coated with 10 nm of gold in an argon atmosphere (P < 2.2 × 10-2 Pa) to produce an electrically conductive surface. The FH specimens were then transferred to a cryostage (−180 °C) inside a scanning electron microscope (Philips SEM 515, Eindhoven, The Netherlands) equipped with the SEM cryo-unit, SCU 020.

EDXMA of the FH specimens was performed with a SEM using an acceleration voltage of 17 kV, a takeoff angle of 16.5°, and a working distance (sample to the final lens of the SEM instrument) of 12.0 mm. Spectra from 0 to 20 keV were collected at increments of 10 eV per