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Filtered vs. naturally sedimented and decanted virgin olive oil during storage: Effect on quality and composition

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Delta-5-avenasterol (PubChem CID: 5281326)
Campesterol (PubChem CID: 173183)
Erythrodiol (PubChem CID: 101761)
Uvaol (PubChem CID: 92802)
Oleic acid (PubChem CID: 445639)
Linoleic acid (PubChem CID: 5280450)
Palmitic acid (PubChem CID: 985)

ABSTRACT

The aim of this study was to compare quality changes and compositional characteristics (fatty acids, total phenols, sensory profile) during 12 months storage of Buža and Istarska bjelica cv. virgin olive oils clarified by filtration at an industrial scale (filter press equipped with cellulose filters) and those clarified by natural sedimentation and decantation, as well as the influence of filtration of virgin olive oils on sterols and triterpene dialcohols composition. There were no significant differences in hydrolytic deterioration and sensory scores during the whole storage period between filtered and naturally sedimented oil samples. After 6 months, natural sedimentation was more favorable as regards delaying of oxidative deterioration, while filtration provided a more stable sensory profile. As regards total phenols the impact of the clarifying procedure was dependent on cultivar and fruit ripeness degree and it was more emphasized than the impact of storage. Filtration slightly affected the fatty acids composition, but natural sedimentation and decantation had no effect on it. Nevertheless, filtration did not compromise the confirmation of virgin olive oil authenticity according to the fatty acid composition, as well as according to sterols and triterpene dialcohols.

1. Introduction

Virgin olive oil (VOO) is highly appreciated for its unique flavour, as well as for its healthy and nutritional properties, mainly associated to high monounsaturated oleic acid content and a broad range of biological active minor components, such as phenols and sterols. Furthermore, higher concentration of monounsaturated fatty acids, antioxidants and some sterols contribute to VOO stability (Singh, 2013; Velasco & Dobarganes, 2002). VOO is obtained exclusively by mechanical and physical processes which include collecting, washing and crushing of olive fruits, malaxation of olive paste, centrifugation, decantation or filtration, storage and oil packaging. Immediately after extraction from olive fruits, VOO is turbid because of suspended solid particles of plant tissue and vegetable water emulsified in the oil, which can deteriorate its quality by facilitating hydrolysis or oxidation of lipid matrix (Brkić Bubola & Koprivnjak, 2015). For optimal conservation conditions of VOO, according to International Olive Council (IOC) trade standards (IOC., 2016), moisture should not exceed 0.2 g/100 g. In VOO in which the moisture content is high and particles of olive tissue are present, negative sensory characteristics could develop during storage, especially *muddy sediment* and *vegetable water* defects. The occurrence of these sensory defects in VOO causes its classification in lower classes of quality, which has a lower selling price. To avoid the risk of sensory degradation, olive oil should be clarified in some

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way. Usually, VOO is left to naturally settle down and then is separated from the sediment by decanting. Sometimes it takes several months before oil becomes clear and during this period it should be one or more times decanted. During decantation, oil is exposed to oxygen which can cause sensory defects associated with oxidation, such as a rancid flavour. Sometimes it is difficult to determine the right time for decanting, since the sedimentation of water and particles of olive fruit tissue is influenced by parameters such as: size of water and tissue particles, temperature during storage, storage tank height, etc. For all these reasons, in recent times, filtration is used for oil clarification to hasten finalization of the product.

However, during VOO filtering, depending on filtration system and filter aid used, quantitative and qualitative changes could take place, especially on minor components, affecting the VOO quality (Lozano-Sánchez, Cerretani, Bendini, Segura-Carretero, & Fernández-Gutiérrez, 2010), as well as major components i.e. unsaturated fatty acids (Sacchi, Caporaso, Paduano, & Genovese, 2015). Certain minor (sterols, triterpene dialcohols, etc.) and major components (fatty acids) are used as authenticity parameters of VOO (IOC., 2016; European Union Commission, 2016) to prevent adulteration of VOO with some other oils. Therefore, it is of high importance to collect information related to influence of each production step on authenticity parameters, filtration as well, since deviations from regulatory limits caused by application of different filtration systems cannot be excluded.

Filtration systems applicable on VOO can be classified in three groups: a) conventional systems based on the use of organic or inorganic filter aids in conjunction with filtration equipment (filter tanks and filter presses), b) membrane filtration and c) new filtration systems proposed as alternative processes (the polypropylene filter bag system and filtration based on the flow of an inert gas) (Lozano-Sánchez et al., 2010). At the industrial scale, the most widespread is the so-called precoat-bodyfeed conventional filtration system, which employs filtration aid and vertical leaf-filter or horizontal filter press as filtration apparatus (Masella, Parenti, Spugnoli, Baldi, & Mattei, 2011). Filter press using a cellulose filter plate as filter aid are mostly used by small companies which cannot afford to purchase other types of filters (Guerrini, Masella, Migliorini, Cherubini, & Parenti, 2015).

The aim of this study was to compare changes of VOO quality and compositional characteristics (fatty acids, total phenols, sensory profile) during storage of oils clarified by filtration at industrial scale (filter press equipped with cellulose filters) and those clarified by natural sedimentation and decantation, as well as the influence of filtration on sterols and triterpene dialcohols composition. Other studies which investigated the influence of filtration by filter press system on VOO quality and composition were mainly focused on differences between filtered and unfiltered olive oil, with or without the influence on shelf life (Fortini et al., 2016; Sacchi et al., 2015). To our knowledge, this is the first study on changes of sterol composition and triterpene dialcohols in VOO after filtration by filter press, as well as the first long period study about the differences between filtered and decanted olive oils. For this study two monovarietal VOO distinguishable in sensory and compositional point of view, cv. Buža and cv. Istarska bjelica (Istria region, Croatia), were chosen. Istarska bjelica cv. ripens late and traditionally the oil is obtained from green fruits and oil is rich in polyphenol compounds (Koprivnjak, Majetić, Brkić Bubola, & Kosić, 2012) resulting in oil with high intensities of bitterness and pungency, while its aroma reminds on green olive fruits and freshly cut green grass. Monovarietal oil from cv. Buža has a lower content of phenolic compounds than Istarska bjelica oil, and it is considered as milder oil with complex fruitiness which reminds on olive fruits, other fruits, aromatic herbs and almond (Brkić Bubola, Koprivnjak, Sladonja, & Lukić, 2012).

2. Materials and methods

2.1. Preparation of VOO samples

Monovarietal VOO samples were produced at the beginning of November 2013 from olives of two different cultivars, Buža and Istarska bjelica, grown in the same orchard in Istria region, Croatia. The ripening index (RI) of fruits was determined applying the method described by Garcia and Yousfi (2005) which is based on the evaluation of the olive skin and pulp colour. The RI of the cv. Buža fruits was 3 (reddish skin, green pulp) and cv. Istarska bjelica fruits was 1 (green skin and pulp).

Olive fruits of each cultivar were processed separately within 24 h after harvesting in the oil extraction plant Model SPI 222S, Pieralisi, Italy (knife crusher, malaxation at 26 ± 1 °C for 35 min, two phase centrifugal decanter). Three replications of VOOs filtration procedure were done immediately after fruit processing, using a 0.4 m × 0.4 m plate press-filter (Model EUR 20, Toscana Enologica Mory, Italy; capacity 350 L/h, filtration area 3.2 m^2) equipped with 19 cellulose filters (Type OV110, Omniafiltra, Italy; weight 1100 g/m^2, thickness 3.8 mm, density 0.29 g/mL) at a pressure of 1.5 kPa and a temperature ≥ 20 °C. Samples of filtered and unfiltered VOOs of each cultivar were packed into dark glass bottles (capacity 1000 mL). Three replications of each filtered and unfiltered olive oil per cultivar were analysed immediately after production (0 month), while remaining bottles of VOO samples were stored at 16–18 °C in absence of light. Three replications of unfiltered olive oils per storage period were left to clarify by natural sedimentation for 45 days, than were decanted and stored under the same conditions. Analyses of filtered and unfiltered-decanted VOO samples were done after 6 and 12 months of storage.

2.2. Analysis of moisture content and water activity

The moisture content in VOO samples was determined according to the ISO (1998). Ten g of homogenized sample were weighted in a vial, and placed in an oven at 103 ± 2 °C, after which the sample was weighed. After that, samples were returned in the oven and dried until constant weight.

Water activity of VOO samples was measured using a water activity meter HygroPalm 23, Rotronic (Bassersdorf, Switzerland). Samples (6.5 mL) were placed in 14 mm deep sample cups and after the equilibration was established, the water activity measurement ended automatically.

2.3. Quality parameters

Free fatty acids (FFA), peroxide value (PV) and spectrophotometric indices (K_{232}, K_{270} and ΔK) were determined according to the analytical methods described in the European Commission Regulation (EEC, 1991).

2.4. Sensory analysis

Quantitative descriptive analysis of VOO samples was performed by a panel of eight assessors trained for VOO sensory analysis according to the method proposed by IOC described in the European Commission Regulation (EEC, 1991) using evaluation sheets expanded with some pleasant odor and taste attributes (green grass, apple, tomato, almond, astringent and sweet). Different pleasant odor and taste attributes were quantified using a 10-cm long continuous rating scale from 0 (no perception, the lowest intensity) to 10 (the highest intensity). Furthermore,