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Composition, volatile profiles and functional properties of virgin olive oils produced by two-phase vs three-phase centrifugal decanters

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ABSTRACT

The present study was designed to compare the impact of a two- vs a three-phase decanter on the quality parameters, sterols, fatty acids, terpenes, phenolic compounds and volatile compounds, as well as antiradical activity of the obtained oils. Moreover, serum lipid antioxidant capacity and anti-inflammatory potential in human mononuclear cells were tested as more biologically relevant assays than the chemical radical scavenging assays. Results show that the cold pressed olive oils obtained from both technologies were of the extra virgin quality. Peroxide values were significantly lower in two-phase decanter samples, while ω6 polyunsaturated fatty acids were higher in two- vs three-phase samples. The significantly higher phenolics content in oil produced by two- vs three-phase centrifuge was attributed to higher hydroxytyrosol, as well as to higher tyrosol, vanillin and homovanillic alcohol levels. Evaluation of the effect of centrifuge system on oils' functional properties showed a clear superiority of two-phase decanter samples as to health maintenance, as these exerted higher ferric reducing capacity, elongation of serum lipid lag time and decrease in cytokine response of stimulated human mononuclear cells, compared to those from three-phase decanter. The two-phase oil samples contained more volatile aroma compounds and more C5–C6 alcohols and aldehydes.

1. Introduction

The low incidence of coronary heart disease, cancer and several chronic diseases in Mediterranean countries is linked to certain nutritional habits, but mainly to olive oil consumption (Boskou, 2007). Health promoting properties of olive oil are attributed to high contents of oleic acid and phytochemicals like sterols, polyphenols, tocopherols and terpenic acids (Boskou, 2007). In Greece, where 60% of cultivated land is devoted to olive growing, olive oil represents an important agronomic product; olive oil production reaches 350,000 tons per year approximately, of which 82% is extra virgin (Wikipedia, 2012).

It is used on salads, added at the table to soups and stews and for dipping. Development of extraction techniques with increased mechanization has facilitated increased olive oil production, with simultaneous decrease in manpower and with limited costs. During the last decades, centrifugation systems have become the most widespread techniques for olive oil production. Compared to the "traditional pressure technique", high production capacity of centrifugation decanters have markedly shortened the storage time of olives before processing, and help to avoid or reduce the risk of organoleptic contamination (Di Giovacchino, Sestili, & Di Vincenzo, 2002). Today, two different centrifugation systems are mainly used for olive oil production; the three-phase and two-phase centrifugation techniques, depending on the products produced at the end of processing. The three-phase system is a continuous process dating at 1970–1980, which has three exits for oil, water, and solids. The main drawbacks of this technology are the use of large quantities of warm water (10–30 L of added water per 100 kg of olive pastes) that results in the reduction of phenol content in oil (Salvador, Aranda, Gómez-Alonso, & Fregapane, 2003) and the production of significant volumes of olive mill waste waters that constitute an important environmental pollution problem (Mechri et al., 2007), although nowadays the centrifugal three-phase decanters were improved to be able to separate oil employing only a small quantity of warm water (0–20 L/100 kg of olives) to dilute the olive paste. These decanters are called "ARA" (Italian acronym meaning water saving decanter) (Amirante, Clodoveo,

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Leone, Tamborrino, & Patel, 2010). Contrary, two-phase decanters can work without water addition. In this process, the final products are olive oil and pomace. The important advantages of this decanter are the reduction in the amount of waste waters and the greater recovery of phenolic compounds (Di Giovacchino, Costantini, Serraiocco, Surricchio, & Basti, 2001; Garcia et al., 2001; Gimeno, Castellote, Lamuela-Raventós, De la Torre, & López-Sabater, 2002; Ranalli & Angerosa, 1996; Stefanoudaki, Koutsaftakis, Kotsifaki, Angerosa, & Di Girolamo, 1999). Apart from phenol content, olive oil chemistry and organoleptic characteristics that classify quality and uniqueness are influenced by extraction and processing techniques (Angerosa, Mostallino, Basti, Vito, & Serraiocco, 2000; Angerosa et al., 2004; Di Giovacchino et al., 2001, 2002). The better recovery of phenols in the two-phase systems is mainly due to their better solubility in water than in oily phases, which makes the amount of added water a crucial determinant for the concentration of phenols in the final product (Clodoveo & Hachicha Hbaieb, 2013; Di Giovacchino et al., 2001). However, although the two-phase decanters appear to be more suitable from the quality and environmental points of view, its use is not so widespread, mainly due to the high moisture content of the resultant pomace, which hinders the quantitative recovery of pomace oil by solvent extraction. Today, emerging technologies aiming to eliminate residues of oil in the pomace thus improving the working capacity of the industrial plants and creating more sustainable engineering solutions, are introduced and evaluated. To this end, pulsed electric fields, ultrasound and microwave treatment of olive paste during olive oil extraction, modification of heating scheme during malaxation, as well as modulation of the activity of endogenous fruit enzymes are under investigation (Clodoveo & Hachicha Hbaieb, 2013; Clodoveo, Hachicha Hbaieb, Kotti, Scarascia Mugnozza, & Gargouri, 2014; Esposto et al., 2013). One major benefit of these new approaches is expected to be the increase of oil yield and the subsequent decline of the poor quality pomace oil, which damages the image of the VOO and exerts a detrimental effect on VOO prices and on producer incomes (Clodoveo et al., 2014).

The present research was designed to determine the effects of processing by two- and three-phase industrial decanters on olive oil quality parameters, composition and profile of volatile aroma compounds. Moreover, this study included the evaluation of some functional properties of the two- and three-phase centrifugation olive oils *in vitro*.

2. Materials and methods

2.1. Reagents and standards

Standards of β-sitosterol, stigmasterol, campesterol, squalene, 5-α-cholestane, and a mixture of fatty acid methyl esters (FAME) were purchased from Sigma (St Louis, MO, USA). Butylated hydroxytoluene (BHT), boron trifluoride in methanol solution (14 g/100 g BF_3/MeOH), bis-(trimethylsilyl)-trifluoroacetamide (BSTFA), stable radical 2,2-diphenyl-1-picrylhydrazyl (DPPH•), and (R)-(+)-limonene were provided by Sigma (Steinheim, Germany). Folin–Ciocalteu reagent, 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox®), homovanillic acid, 3-(4-hydroxyphenyl)-1-propanol, cinnamic acid, oleanolic acid, nonanal, 1-penten-3-ol, 2,4-heptadienal, and pentadecane were obtained from Aldrich (Steinheim, Germany). Tyrosol, protocatechuic acid, 3,4-dihydroxyphenylacetic acid and caffeic acid were purchased from Fluka (Steinheim, Germany); *p*-hydroxybenzoic acid, ursolic acid, vanillin, *p*-coumaric acid, syringic acid, ferulic acid, and ursolic acid were obtained from Sigma (Steinheim, Germany); vanillic acid was obtained from Serva (Heidelberg, Germany), hydroxytyrosol was from Extrasynthése (Genay-Cedex, France). 1-Heptanol and 4-heptanone were from Fluka (St Louis MO, USA). All the solvents used were of analytical grade and were obtained from Aldrich (Steimheim, Germany). Folin–Ciocalteu reagent and sodium carbonate were purchased from Merck (Darmstadt, Germany). All other reagents were of HPLC grade and were purchased from Merck or Aldrich. RPMI-1640 culture media was purchased from Gibco (Grand Island, NY, USA). Ficoll–Paque Plus was from GE Healthcare Biosciences (Piscataway, NJ, USA). Fetal bovine serum (FBS), penicillin/streptomycin and trypsin/EDTA were supplied by PAA Company (Somerset, UK). Quantikine sandwich ELISA kits for the measurement of interleukin-6 (IL-6), monocyte chemoattractant protein-1 (MCP-1) and tumor necrosis factor-alpha (TNF-α) were from R&D Systems (Oxford, UK). Phosphate buffer saline (PBS) tablets, lipopolysaccharide (LPS), copper sulfate benzene anhydrous and toluene anhydrous were from Sigma–Aldrich (St Louis MO, USA).

2.2. Olive samples and processing

This study included olive oil samples produced from olives (*Olea europaea* L.) of the Koroneiki cultivar, which is considered as the most prized Greek olive variety for oil production, originating from the area of Korone in Messinia, Peloponnese, Greece. This variety grows well on mountain slopes and produces very small fruit; the high ratio of skin to flesh giving the oil its coveted aromatic qualities. Olive fruits grown in an organic farm in Western Mani, Messinia, were picked by hand during January 2012. The maturity of olive fruits was confirmed by their color: 70% were black and 30% brown. Olives' average diameter was 11.2 mm and their average weight was 0.73 g. Overall, 400 kg of olive fruits were collected and were divided in two equal homogenous lots, which were transferred to two local oil mills equipped with two- and three-phase decanters, respectively. Olives were processed within 24 h, by the following procedure: (i) leaf removal from olive lots; (ii) washing of olives with water; (iii) milling of drupes by disk crusher; (iv) malaxation of the paste in a single-stage malaxation machine at 24–25 °C for either 60 min in the dual-phase system or for 30 min in the three-phase system, respectively; (v) centrifugation using an Amenduni (Italy) dual-phase decanter or fluidification by the addition of water and centrifugation using an Alfa-Laval (Sweden) three-phase decanter prior to separation of the oil by means of an automated vertical discharge centrifuge. In the dual-phase system no water was added in the decanter. Oil obtained from the two olive mills was put in 3 L air-tight containers, stored at 12 °C and transported to the laboratory.

2.3. Free acidity, peroxide value and UV spectrophotometric indices

Free acidity, expressed as percent of oleic acid (% w/w C18:1), peroxide value, given as milliequivalents of active oxygen per kilogram of oil (mequiv O_2 kg^{-1}) and UV absorption characteristics (K_{232} and K_{270}) were determined according to the European Communities official methods (EU, 2011).

2.4. Total phenolic content

Total phenolic content of oil samples was measured by the Folin–Ciocalteau assay. For the assay, phenolic compounds were isolated from a solution of oil in hexane by a triple extraction with a methanol/water mixture (60:40, v/v). The Folin–Ciocalteau reagent was added to a suitable aliquot of the combined extracts, and the absorption of the solution was measured at 725 nm using a Specord 20 (Analytik Jena, Jena, Germany) spectrophotometer. Gallic acid was used for the construction of a standard curve and the results were expressed as mg gallic acid equivalents (GAE)·100 g^{-1} oil.