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Effect of light exposure on the quality of extra virgin olive oils according to their chemical composition

Sonia Esposto *, Agnese Taticchi, Stefania Urbani, Roberto Selvaggini, Gianluca Veneziani, Ilona Di Maio, Beatrice Sordini, Maurizio Servili

Department of Agricultural, Food and Environmental Sciences, University of Perugia, Via San Costanzo s.n.c., 06126 Perugia, Italy

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ABSTRACT

The influence of light exposure on the quality of commercially available extra-virgin olive oils (EVOOs) of different chemical composition was studied as a function of storage (11 weeks) under conditions simulating market storage. By mildly stripping the polyphenols from oil 'A', with high levels of polyphenols and oleic acid, and oil 'B', exhibiting a medium level of polyphenols and a low level of oleic acid, 'C' and 'D' EVOOs were obtained. Ten EVOOs were produced as mixtures of these four oils. The initial concentrations of oleic acid and polyphenols in the 14 oils ranged from 64.5 to 77.7% and 18.1 to 1476.7 mg/kg, respectively. The extinction coefficient K_{270} well reflected the EVOO product quality. EVOOs richer in oleuropein derivatives showed superior oxidative stability, which resulted in lower off-flavour volatile compound production and α-tocopherol and polyphenols losses, and thus, higher EVOO sensory and health benefits.

1. Introduction

Extra-virgin olive oil (EVOO) is one of the most important components of the Mediterranean diet (Estruch et al., 2006; Konstantinidou et al., 2010). Compared to other vegetable oils, it is characteristically richer in monounsaturated fatty acids (MUFAs) and antioxidants, mainly secoiridoid derivatives and volatile substances, which confer the exclusive sensory and health properties to EVOOs (Angerosa et al., 2004; Bulotta et al., 2014; Servili et al., 2009; Terés et al., 2008).

Oxidation decreases the sensory and health-promoting qualities as well as the marketing value and consumer acceptability of an EVOO because it leads to the generation of low-molecular-weight off-flavour substances, loss of antioxidants, and accumulation of toxic compounds such as free radicals (Choe & Min, 2005, 2006, 2009). The predisposition of an EVOO to these negative phenomena depends on its exposure to pro-oxidant factors such as oxygen, temperature, light, and other activators (chlorophylls and transition metals) (Bendini, Cerretani, Salvador, Fregapane, & Lercker, 2010; Choe & Min, 2006; Khan, 1955), which can affect its oxidative stability from production to consumption. However, the response of an EVOO to oxidation depends on its chemical composition, particularly, on those substances that can directly or indirectly influence these phenomena, such as natural antioxidants and oleic acid, respectively (Bendini et al., 2010; Choe & Min, 2005, 2006, 2009). Because the levels of these two chemical groups are highly influenced by genetic, agronomic, and technological factors, EVOOs typically widely vary in oleic acid and antioxidant contents. A recent study on 740 commercial EVOOs differing in terms of olive cultivar used, geographic origin, and mechanical extraction method used revealed ranges of 50.5–80.5% for oleic acid, 91–665 mg/kg for α-tocopherol, and 50–900 mg/kg for secoiridoid derivatives and lignans (Servili et al., 2015).

Generally, EVOO stability ranges roughly from 9 to 18 months, based on the mentioned internal (chemical composition) and external factors (presence of pro-oxidants). Furthermore, EVOO shelf life appears quite unsteady and is often compromised in marketplaces, where long exposure to light can substantially reduce EVOO quality (Choe & Min, 2006; Bendini et al., 2010). Various studies have evaluated EVOO oxidative stability during shelf storage; however, in many cases, they involved laboratory tests, which do not consider several parameters that affect EVOO market storage (Hachicha Hbaieb, Kotti, Gargouri, Msallem, & Vichi, 2016; Luna, Morales, & Aparicio, 2006; Nishida, Yamashita, & Miki, 2007; Psomiadou & Tsimidou, 2002a, 2002b; Rahmani & Saari Csallany, 1998; Stefanoudaki, Williams, & Harwood, 2010). Fast assays, such as the AOM, Rancimat, DPPH, and OSI tests, only allow determining the capability of EVOOs to resist drastic oxidative stresses (Baldioli, Servili, Perretti, & Montedoro, 1996;

* Corresponding author.
E-mail address: sonia.esposto@unipg.it (S. Esposto).

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Carrasco-Pancorbo et al., 2005; Condelli et al., 2015; Mancebo-Campos, Desamparados Salvador, & Fregapane, 2014; Servili et al., 2009); hence, these studies cannot appropriately assess the stresses occurring in bottled EVOOs in the market. There are currently very few reports of in-depth studies on the evaluation of quality parameters and shelf life of commercial, bottled EVOOs, likely because such studies are time-consuming and involve multiple analyses, and numerous EVOOs with highly variable composition are available (Caponio, Bilancia, Pasqualone, Sikorska, & Gomes, 2005; Cinquanta, Esti, & Di Matteo, 2001; Coutelieris & Kanavouras, 2006; Del Nobile, Bove, La Notte, & Sacchi, 2003; Gutiérrez & Fernandez, 2002; Gõmez-Alonso, Mancebo-Campos, Desamparados Salvador, & Fregapane, 2007; Kalua, Bedgood, Bishop, & Prenzler, 2006; Mendez & Falque, 2007; Morello, Motilva, Tovar, & Paz Romero, 2004; Okogeri & Tasioula-Margari, 2002; Pagliarini, Zanoni, & Giovanelli, 2000; Pristouri, Badeka, & Kontominas, 2010).

To the best of our knowledge, research evaluating the quality of EVOOs in terms of oleic acid percentage and polyphenol contents during storage simulating supermarket conditions has not been performed to date. Here, we report a time-course study of the effect of storage at room temperature for 6 months under 11 h of light exposure per day on parameters related to product, health-promoting, and sensory qualities of EVOOs with different chemical compositions.

2. Materials and methods

2.1. Materials

Ethyl acetate, anhydrous sodium sulphate, methanol, ethanol, *n*-hexane, 2-propanol, and acetic acid were purchased from Sigma-Aldrich (Milan, Italy), and high-performance liquid chromatography (HPLC)-grade methanol and water for HPLC-MS were purchased from Fluka (Milan, Italy).

Two different EVOOs with high and medium polyphenol levels and high and low oleic acid percentages (named 'A' and 'B', respectively) were obtained from bulk suppliers. The oils were analysed upon receipt to determine the initial quality and to provide a quality baseline for the experiments. The initial polyphenol contents of A and B were 1476.7 and 682.5 mg/kg, respectively. These concentrations represented the sum of the following compounds: hydroxytyrosol (3,4-DHPEA), tyrosol (*p*-HPEA), secoiridoid derivatives, such as the dyaldeidic form of the elenolic acid linked to 3,4-DHPEA (3,4-DHPEA-EDA), the oleuropein aglycon (3,4 DHPEA-EA), and the aldeydic form of elenolic acid linked to *p*-HPEA (*p*-HPEA-EDA), and the lignans (+)-1-acetoxypinoresinol and (+)-pinoresinol. The initial oleic acid percentages for sample A and sample B were 64.8% and 77.7%, respectively. The α-tocopherol contents were 173.8 mg/kg for sample A and 220 mg/kg for sample B. The chlorophyll contents of EVOOs A and B were 20 and 19.8 mg/kg, respectively.

Polyphenol stripping was conducted by repeating the following procedure thrice: oil and water (1:1, v/v) were immediately mixed by vortexing for 3 min. The mixture was centrifuged in a basket centrifuge at 352 G-force for 8 min. The oily phase of the supernatant was recovered and filtered with sodium sulphate to remove any trace water. By this procedure, two different EVOOs, C and D, were obtained from A and B, respectively. The entire process was performed with as little air contact as possible.

A statistical central composite design (CCD) was built with samples A, B, C, and D, which were placed at the vertices of the square as starting points. By mixing of the samples in different percentage ratios, 10 EVOOs characterized by intermediate contents of oleic acid and hydrophilic phenols were obtained. The 10 oils obtained by applying the CCD were named: 'C + A', 'D + B', 'B + A', 'D + C', 'A + D', 'C + B', 'DC + DB', 'CA + BA', 'DB + BA', and 'DC + CA', where the two samples that composed each new EVOO were each present at 50% of the volume.

2.2. Experimental set-up for market storage simulation

Real marketplace storage conditions were simulated as follows. Eleven green-glass 750-mL bottles of each of the 14 EVOOs (oils A, B, C, D, C + A, D + B, B + A, D + C, A + D, C + B, DC + DB, CA + BA, DC + CA, and DB + BA) were placed in a climate chamber in 14 rows. The room temperature was set at 22 °C, and the bottles were exposed to 12 h of light (600 lx) per day by an automatic lighting system. The bottles were moved weekly from the first to the last position in the same row to ensure equal light exposure over the experimental period of 165 days. Every 15 days, the first bottle of each row was withdrawn and stored at 12 °C until analysis. Thus, the bottles were withdrawn from the climate chamber at times T0, T15, T30, T45, T60, T75, T90, T105, T120, T135, T150, and T165.

2.3. Analytical determinations

All determinations were done for each of the 14 EVOOs at all 12 time points mentioned above, unless mentioned otherwise.

2.3.1. Merchandise parameters

The free acidity content (g of oleic acid/100 g of oil), peroxide values (amount of hydroperoxides expressed as milli-equivalents of O_2/kg), K_{232} and K_{270} extinction coefficients, and fatty acid compositions of all the EVOO samples were determined according to the official methods of the European Commission (Commission Delegated Regulation (EU) 2015/1830).

2.3.2. Initial chlorophyll content determination

The chlorophyll contents were analysed as described by Pokorny, Kalinova, and Dysseler (1998).

2.3.3. α-Tocopherol and phenolic compound determination

The α-tocopherol contents were evaluated by HPLC with diode array and fluorescence detectors (HPLC-DAD-FLD; Agilent Technologies, Santa Clara, CA, USA) according to Esposto et al. (2015). Phenols were extracted according to the procedure reported by Esposto et al. (2013) and evaluated by HPLC-DAD (Agilent Technologies, Santa Clara, CA, USA) analysis.

2.3.4. Determination of the phenolic oxidation products

Phenolic oxidation products were analysed in all EVOOs at T0, T30, T60, T90, T120, and T150 by HPLC/electrospray ionization tandem mass spectrometry (HPLC-ESI-MS) and MS/MS (Agilent Technologies, Santa Clara, CA, USA) according to Di Maio et al. (2013).

2.3.5. Determination of the volatile compounds

The compositions of the volatile compounds of all EVOO head spaces were assessed using headspace solid-phase microextraction followed by gas chromatography/mass spectrometry (HS-SPME-GC/MS; Agilent Technologies, Santa Clara, CA, USA) as described by Esposto et al. (2013).

2.4. Statistical analysis

A CCD for two factors was created using the Modde 9.1 package, whereas principal component analysis (PCA) and partial least squares (PLS) regression were carried out using the SIMCA 13.0 chemometric package. Both packages were purchased from Umetrics AB (Umeå, Sweden). A priori one-way analysis of variance (ANOVA) followed by the Tukey test was conducted using the