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Effect of olive storage conditions on Chemlali olive oil quality and the effective role of fatty acids alkyl esters in checking olive oils authenticity

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ABSTRACT

The present paper accounts for the study of the storage of Chemlali olive fruits at two conditions of limited aerobiosis: in closed plastic bags and in open perforated plastic boxes for different periods before oil extraction. The ultimate objective is to investigate the effect of the container type of the postharvest fruit storage on the deterioration of the olive oil quality. The results have shown that the oil quality of Chemlali olives deteriorated more rapidly during fruit storage in closed plastic bags than in perforated plastic boxes. Therefore, the use of perforated plastic boxes is recommended for keeping the olives for longer periods of storage. The repeated measures analysis of variance of all parameters analyzed indicated that the olive oil quality is mainly affected by the olives storage conditions (containers type and storage periods). Finally, blends of extra-virgin olive oil and mildly deodorized low-quality olive oils can be detected by their alkyl esters concentrations.

1. Introduction

Virgin olive oil is a valuable vegetable oil extracted from fresh and healthy olive fruits (*Olea europaea* L.) by mechanical processes (pressing or centrifuging) and without heat, solvents or any preliminary refining (Ammar, Zribi, Ben Mansour, et al., 2014; Garcia & Yousefi, 2006). It is practically the only vegetable oil that can be consumed directly in its raw state as well as it contains important nutritional elements (fatty acids, vitamins, sterols, etc.). Extra-virgin olive oil (EVOO) is considered as the best olive oil for its superior organoleptic characteristics (aroma and taste). It has a potential health benefits, remarkable antioxidant properties and chemical composition (Ammar, Zribi, Gargouri, Flamini, & Bouaziz, 2014; Jafari, Kadivar, & Keramat, 2009; Méndez & Falqué, 2007).

It is well-known that the storage period has an influence on the quality of fruit and oil of black-ripe olives. The main factor behind the deterioration of olive oil is accredited to the poor handling of the olives during the time between harvesting and processing. Indeed, the storage of olive fruits that develop all kinds of degenerative processes in a short period of time is carried out by simple heaping in fruit piles, waiting for their processing (Rabiei, Ghorbani, & Hajnajari, 2011).

It is in this context that the present paper lies to study the effect of the storage period of olive fruit (*Olea europaea* cv. Chemlali) in closed plastic bags and in open perforated plastic boxes on the olive oil quality. In fact, oils were extracted and their quality immediately

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evaluated after fruit harvest and at the end of five predetermined temporal storage periods of 3, 6, 11, 17 and 25 days. The chemical quality parameters such as acidity, peroxide value, specific extinction coefficient at 232 and 270 nm, total polyphenols contents, pigment contents and fatty acid alkyl esters (FAAEs) together with sensorial quality determined for 25 days, were analyzed.

The fatty acid methyl esters and fatty acid ethyl esters contents are not only known to be closely related to the health conditions of olive fruits but also obviously higher if olives undergo hydrolytic and fermentative processes, thus increasing the amounts of both free fatty acids and alcohols. What is worthy to note is that oils obtained from fermented fruits are low-quality virgin olive oils, having unpleasant sensorial features that prevent them from being classified as extra-virgin olive oils, thus leading to the decrease in their commercial value.

Unfortunately, EVOO is also easy to falsify. In fact owing to its prominence, it has always been illegally mixed with cheaper and low-quality oils (Harwood & Aparicio, 2000; Jabeur et al., 2014), especially to obtain EVOO sold in supermarkets and discount stores at low cost (Bendini, Cerretani, Salvador, Fregpane, & Lercker, 2009). The so-called lampante low-quality olive oils cannot be used as raw foodstuff for direct human consumption, as they have an acidity level that is too high, and their volatile profile is characterized by 'soft' off-flavours, derived from low-quality olives or from inappropriate procedures during oil extraction or storage.

The newest and most common adulterations of extra-virgin olive oil are the dilution by mild deodorized low quality olive oil. The latter is obtained from lampante virgin oil with an unpleasant flavor, subjected to a mild thermal deodorization, developed under vacuum and at low temperature (100–120 °C) for removing undesired substances that negatively influence its flavor (mainly winey-vinegary, fusty and musty). After correction of the sensorial defects of these oils, is often used to be used for an illegal blending with extra-virgin olive oils (Pérez-Camino, Cert, Romero-Segura, Cert-Trujillo, & Moreda, 2008). In general, such a blending does not produce an easily detectable modification of the chemical composition because of the mild conditions used in the deodorization.

With respect to the second objective of this study, it pertains to the investigation of the effect of incorporating mild deodorized olive oils with variable amounts in extra-virgin olive oil by the determination of fatty acid methyl and ethyl esters contents. Actually, the effectiveness of these chromatographic determinations was examined for the minimum detectable limit of adulterated soft deodorized oil.

2. Materials and methods

2.1. Chemicals and reagents

Ethanol (⩾99.9%) and n-heptane (99.0%) were obtained from Riedel-de Haën (Steinhein, Germany). Cyclohexane (99.5%), n-hexane (99.0%) and diethyl ether (⩾99.7%) were purchased from Merck KGaA (Darmstadt, Germany). Potassium hydroxide (85.0%) was obtained from CDM (Karlsruhe, Germany) and potassium iodide (>99.0%) was purchased from Chem-Lab (Zedelgem, Belgium). Sodium hydroxide (>99%) was supplied by Scharlau (Chemie, S.A, Spain). Acetic acid (100.0%) and chloroform (>99.1%) were from Prolabo (AnalaR NORMAPUR, France). Folin–Ciocalteu reagent was obtained from Fluka (Buchs, Switzerland). Lauryl arachidate (>99%) and methyl heptadecanoate (>99%) standards were purchased from Sigma–Aldrich (St. Louis, MO, Germany).

2.2. Samples

2.2.1. Olive fruits

Black-ripe olives (*Olea europaea*. Chemlali) fruits of good quality were hand harvested from the Sfax region (southern Tunisia) on February 04, 2012 at ripening phase. The maturation index (M.I.) of the used fruits was 5.8. The olive maturity index (MI) was determined according to the method developed by Boskou (1996) on the basis of the evaluation of the olive skin and pulp colors. MI values range from 0 (100% intense green skin) to 7 (100% purple flesh and black skin). After leaves deletion, washed fruits were mixed to ensure a homogeneous sampling before storage. The olives were randomly divided into batch of 10 kg.

The control sample was processed immediately after harvesting, while the other lots were stored in a room at temperature of 12 ± 2 °C by night and 18 ± 2 °C by day with a relative humidity of 60% during different periods (3, 6, 11, 17 and 25 days) and at two conditions of limited aerobiosis: in closed plastic bags (polyethylene high-density (PE-HD) bags: length = 50 cm and width = 40 cm) and in open perforated plastic boxes (a small format (length = 50 cm, width = 30 cm and height = 40 cm) of perforated polyethylene high-density (PE-HD) boxes).

2.2.2. Oil extraction

Olive oil was extracted using a procedure that imitates the industrial process. Fruits were firstly crushed by a laboratory blender and the resulting paste was mixed for 30 min in the presence of warm (30 °C) distilled water (about 30% of the paste *w/w*) to facilitate oil separation. Olive oil was obtained after the centrifugation of the paste at 3000 rpm for 10 min and stored in dark glass bottles at −20 °C for future analyses.

2.2.3. Deodorized olive oil and blends preparation

Mild deodorized olive oil (DO) was provided by laboratory-scale plant (AGRO-ZITEX, Sfax, Tunisia). It was originally obtained by submitting low-quality oil to a mild refining process at 110 °C under steam stripping for up to 4 h. The small quantity of mild deodorized olive oils was due to the difficulties to find such oils. Blends were prepared in the laboratory, by mixing an extra-virgin olive oil with deodorized olive oil, all of which were produced in 2012, at different increasing amounts: 1, 5, 10, 20, 30, 40 and 50 g/100 g.

2.3. Analytical methods

2.3.1. Quality indices determinations

The titratable acidity (free fatty acids) was determined according to the method proposed by ISO660, (1996). Besides, while peroxides were determined according to the method proposed by ISO3960, (2001), UV spectrophtometric constants (K_{232} and K_{270}) were carried out according to the analytical methods described by COI (2010). As for the total polyphenols, they were determined according to the previously published protocol, making use of Folin–Ciocalteau methodology described by Zribi et al. (2013). Moreover, the gallic acid was applied as standard reference and the results were expressed as gallic acid equivalents (ppm). Next, carotenoids and chlorophylls (mg/kg of oil) were determined at 470 and 670 nm, respectively, in cyclohexane using the specific extinction values according to the method of Haddada et al. (2008).

2.3.2. Determination of sensory quality

The sensory quality evaluation was determined according to the International Olive Oil Council (COI, 2011a,b) by the Tunisian National Office of Oil panel. The panel, recognized according to IOOC, consists of selected and well-trained olive oil experts monitored in accordance to their skills in the distinction between similar samples by an experienced panel leader.

2.3.3. Determination of fatty acids alkyl esters (FAAEs) and waxes

Fatty acids alkyl esters and waxes were determined by gas chromatography (GC-FID) according to the method reported in