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Improvements in the malaxation process to enhance the aroma quality of extra virgin olive oils

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ABSTRACT

The influence of olive paste preparation conditions on the standard quality parameters, as well as volatile profiles of extra virgin olive oils (EVOOs) from Morisca and Manzanilla de Sevilla cultivars produced in an emerging olive growing area in north-western Spain and processed in an oil mill plant were investigated. For this purpose, two malaxation temperatures (20/30 °C), and two malaxation times (30/90 min) selected in accordance with the customs of the area producers were tested. The volatile profile of the oils underwent a substantial change in terms of odorant series when different malaxation parameters were applied.

1. Introduction

Virgin olive oil (VOO) is the principal source of fat above all in the Mediterranean diet (Aparicio & Harwood, 2003). Spain is a traditionally olive-growing country of the Mediterranean which produces and exports high quality VOO from a wide variety of cultivars. The Spanish olive grove is present in 34 of the 50 Spanish provinces and occupies an area of 2,509,677 ha. The areas of olive production in Spain – in descending order – are Andalusia (60.4%), Castilla-La Mancha (15.8%), Extremadura (10.2%), Catalonia (4.6%), Valencia (3.7%), Aragon (2.3%) and others (3.1%) including Galicia (AAO-Agencia para el aceite de oliva, 2013). Galicia (Northwestern Spain) was centuries ago, in the Middle Ages, a great producer of oil. Nowadays, there is a resurgence of this culture, from a family and artisan production to half-scale production. The oils produced in this area are thought to possess a characteristic aroma profile, but – to our knowledge – there are scarce data on their composition (Reboredo-Rodríguez, González-Barreiro, Cancho-Grande, & Simal-Gándara, 2012; Reboredo-Rodríguez, González-Barreiro, Cancho-Grande, & Simal-Gándara, 2013a, 2013b).

VOO oil is highly appreciated by consumers for its healthy and sensory properties. The olfactory attributes of VOO arise mainly from the occurrence of C5 and C6 saturated and unsaturated aldehydes, alcohols and esters responsible for some typical sensory notes (such as 'cut grass', 'fruity' and 'floral').

The processing and characteristics of different quality olive oils are controlled and defined by the European Commission Implementing Regulation No 29/2012 (European Union Commission, 2012). The production of a high quality VOO is not only strongly dependent on the nature of the cultivar and the use of healthy olive fruit with an appropriate degree of maturity, but also it is influenced by other several factors like edaphoclimatic conditions, agricultural practices, extraction methods, processing techniques and/or storage conditions (Boselli, Di Lecce, Strabbioli, Pieralisi, & Frega, 2009; Inarejos-García, Gómez-Rico, Salvador, & Fregapane, 2009).

The extraction process of VOO, consisting only of physical methods, includes olive crushing, malaxation of the pastes and separation of the oil phase. Malaxation of the olive paste, obtained previously from the olive crushing, is a crucial step of the process where the olive paste is subjected to a slow continuous kneading, aimed at breaking off the emulsions formed during the crushing process and facilitating adequate coalescence (Angerosa, Mostallino, Basti, & Vito, 2001; Stefanoudaki, Koutsaftakis, & Harwood, 2011).

According to Clodoveo (2012) malaxation of olive paste must be considered much more than a simple physical separation, because a complex bioprocess takes place that is very relevant to the quality and composition of the final product. During malaxation considerable changes in the oil's chemical composition occur because of the partition phenomena between oil and water and vice versa and the catalytic activity of fruit enzymes. Different enzymatic reactions of oxidoreductases naturally present in olive

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pulp (such as polyphenoloxidases-PPO, lipoxygenases-LOX and peroxidases-POD) involving in the transformation of volatile and phenolic compounds take place (Boselli et al., 2009; Taticchi et al., 2013). The rate and the extent of these reactions are greatly affected by malaxation time and temperature, two technological parameters that can markedly modify not only the oil yield but also the composition and quality of the final VOO produced (Inarejos-García et al., 2009).

In the present work, we have examined the effect of different malaxation operating conditions commonly used by Galician producers on the standard quality parameters, as well as the volatile profile of EVOOs obtained from two olive fruits, Manzanilla de Sevilla and Morisca. The aim of this work was to find the right combination of malaxation time and temperature in order to ensure the best quality of the resulting oils with a characteristic aroma.

2. Material and methods

2.1. Oil samples

For this study, olives from two olive orchards with a different variety each were collected in November 2011 in the Southeast of Galicia (NW Spain). Each variety presented one maturation index (MI) according to the method proposed by the International Olive Oil Council (IOOC-International Olive Oil Council, 1984), based on the evaluation of the olive skin and pulp colours of the fruit. Two different oils were done: a mixture of Morisca and Verdial de Badajoz cultivars (90:10%) (MI = 3.4) and a mixture of Manzanilla de Sevilla and 'unknown' cultivars (95:5%) (MI = 2.1), with one in a higher proportion than the other. It should be noted the huge difficulty to obtain monovarietal oils in the olive oil mills of this area; a little percentage of a different olive variety is usually found in the olive batches processed.

The oils were elaborated under identical conditions at a semi-industrial scale. Thus, all oil samples were processed in an oil mill plant (Almazara Profy, Industrias Céspedes e Hijos, S.L.) with a production capacity of 200 kg/h equipped with an olive washing machine, a hammer crusher, a horizontal kneader with a non-hermetic closure and a two-phase horizontal decanter. Leaves and dirt were removed by washing under cold running water before extraction. The olive paste corresponding to a mixture of Morisca and Verdial de Badajoz (90:10%) cultivars was kneaded at 20 ± 3 °C during 30 and 90 min, as well as 30 ± 2 °C during 30 and 90 min. On the other hand, the olive paste corresponding to a mixture of Manzanilla de Sevilla and 'unknown' (95:5%) cultivars were kneaded only at 30 ± 2 °C during 30 and 90 min. The temperature at three different points (left, centre and right of the malaxer) was checked by using an infrared thermometer at 10 min intervals. The conditions used in terms of temperature and time of malaxation were selected according to producers customs.

Four oil samples were obtained for each set of conditions and were stored in dark-brown glass bottles without headspace at 10 °C in the dark. The samples were allowed to settle and racked for about 4 months. This is the procedure typically used by local producers before marketing their oil (Reboredo-Rodríguez et al., 2013b).

Genetic and morphological determinations of representative olive samples were performed by the Pomology Group of the Department of Agronomy at the University of Cordoba (Spain), using fingerprinting based on Simple Sequence Repeat (SSR) markers. The results were used to characterise the studied cultivars. Accurately identified varieties included in the database of the World Olive Germplasm Bank of Cordoba (WOGB), which is the main repository of olive genotypes in Spain, were used as reference samples.

2.2. Analytical methods

2.2.1. Quality indices, fatty acids, sterols and erythrodiol+uvaol assays

Standard quality indices (viz., free acidity; peroxide value; UV absorption characteristic, K_{270}, K_{232}; waxes, trilinolein and ΔECN), fatty acids, sterols and triterpenic dialcohols composition were carried out, following the analytical methods described by European Commission's Regulation EEC/2568/91 and subsequent amendments (European Union Commission, 1991, 2003, 2007). The values of these parameters in different olive oils can be limited by regulations established by the European Union.

Fatty acid assays were determined according to EEC/2568/91 and subsequent amendments (European Union Commission, 1991, 2003).

Sterols and erythrodiol+uvaol were determined by following the procedures set out in Annexes V and VI of Regulation EEC/2568/91 and subsequent amendments (European Union Commission, 1991, 2003).

2.2.2. Extraction and identification of volatiles

Volatile compounds were extracted from the EVOO samples by Dynamic Headspace (DHS) with an automatic sampler device, the Master DHS (DANI Instruments S.p.A., Cologno Monzese, Milan, Italy), following our previous work (Reboredo-Rodríguez et al., 2012). In short, the samples (volume: 9.0 mL of EVOO, fast shaking) were directly placed into the DHS sampler in standard 20 mL vials that can be heated at a chosen temperature (40 °C). An inert gas flow (He, flow: 150 mL/min) was used to purge the sample (time: 90 min) in order to carry out the volatile compounds; then the purged gas passed through a cooled trap (0 °C) where the compounds were concentrated. The trap was quickly heated in backflush to a high preset temperature (250 °C) transferring the compounds to the chromatographic column in a narrow band and a reduced volume of gas. The Master DHS has a device especially designed to remove humidity from the desorbed gas before entering into the GC–MS system. It was kept at low temperature during the injection phase and was heated during the baking step to eliminate retained water.

Afterwards, volatile compounds were separated and identified on a Trace GC gas chromatograph with a PolarisQ ion trap mass selective detector (ITMS) interfaced to a PC computer running the software Xcalibur 1.4, from Thermo Finnigan (Rodano, Italy). Chromatographic separations were done with a ZB-WAX fused-silica capillary column (60 m × 0.32 mm ID, 0.50 μm film thickness, Phenomenex, Torrance, CA, USA). The carrier gas, helium, was circulated at 1 ml/min in the constant flow mode. A split/splitless injector in the split mode was used (split ratio, 1:10). The injector temperature was 200 °C. The oven temperature programme was as follows: 40 °C for 5 min; 2 °C/min ramp to 125 °C; 10 °C/min ramp to 250 °C and holding for 5 min. The transfer line temperature was 250 °C, and the ion trap manifold temperature 200 °C. The ion energy for electron impact (EI) was set constantly at 70 eV. Identification of the volatile compounds was achieved by comparing the GC retention times and mass spectra over the mass range 35–300 amu for the samples with those for pure standards analysed under the same conditions. Mass detection was performed in the selected ion recording (SIR) mode for quantification. The concentration of volatile compounds in EVOO samples was calculated taking into account the method recoveries (Reboredo-Rodríguez et al., 2012).

2.3. Calculation of the odorant series values

An odorant series is defined as a group of volatile compounds with similar aroma descriptors (Peinado, Mauricio, & Moreno, 2006). The total intensities for every odorant series were calculated as sum of the odour activity value (OAV) (defined as concentration