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Influence of fruit ripening on agronomic parameters, quality indices, sensory attributes and phenolic compounds of Picudo olive oils

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ABSTRACT

This study assesses variations in agronomic and physicochemical parameters, sensory properties and phenolic content of oils obtained from Picudo olives at nine different ripening stages. The agronomic data obtained in this study suggest that the optimal harvesting period for the Picudo cultivar are from samples IV onwards, although harvest is hampered by the fruit resistance to detachment. Data from sensory analysis show that the maximum organoleptic values and the lowest sensory profile changes were found between harvest samples III and V. Finally, principal component analysis and cluster analysis applied to the data analyzed showed a correlation between the ripening stage and the different variables studied. Oils obtained from unripe fruits were found to have the attribute green leaf and a high content of the phenolic compound vanillin. On the other hand, when the fruit was harvested during veraison, the oil obtained had the attributes grass and olive fruity. Finally, when the fruit was picked at an advanced ripening stage, the oil obtained was sweeter. The results obtained in this study show that the optimal harvest periods for Picudo cultivar were IV and V, with ripening indices of 1.85 and 2.0, respectively.

1. Introduction

Olive ripeness is one of the most important factors associated with the quality of virgin olive oil (VOO) (Youssef et al., 2010). From an agronomic standpoint, ripeness is paramount to olive tree growing, since it depends on the fruit variety, resistance to detachment, weight and the fat/humidity ratio, among other factors. All these factors must be carefully controlled to maximize the profitability of Picudo olive groves. Likewise, the phenolic composition and sensory properties of olive oil vary significantly depending on the olive ripening stage (Rivas, Sanchez-Ortiz, García-Moyano, & Lorenzo, 2013). These variations are caused by a series of chemical reactions and enzymatic activity, such as that of glycosidases, phenol oxidases or phenol polymerases (Ryan, Antolovich, Prenzler, Robards, & Lavee, 2002; Yousfi, Cert, & Garcia, 2006) that induce variations on the chemical structure and concentration of some compounds.

The dramatic increase in the demand for VOOs cannot only be explained by their health properties, but also by their organoleptic properties (Rotondi, Alfei, Magli, & Pannelli, 2010). The ripening stage of the olive fruit is one of the most important factors determining the sensory quality of VOOs (Jimenez Herrera et al., 2012). Consequently, it is a very relevant criterion for determining the organoleptic quality of olive oil, since the impact of the ripening stage of the fruit on the chemical composition of the oil is greater than that of the fruit variety itself (Sánchez Casas et al., 2006). Finally, it is worthy of note that in the European Union the quality of VOO is determined according to its sensory properties and physicochemical indices.

Olive fruits undergo a series of changes during ripening. Accordingly, it is necessary to develop a methodology for determining the best harvest time for each variety, in order to optimize the productivity of groves and obtain high-quality olive oil. Several authors have reported the influence of harvest time on VOO quality (Beltran, Aguilera, Del Rio, Sanchez, & Martinez, 2005; Youssef et al., 2010). This illustrates the need to determine the best ripening stage for the picking and processing of each olive cultivar (Gómez-González, Ruiz-Jiménez, & Luque de Castro, 2011).

Concerning native cultivars such as the Picudo variety, for an optimal exploitation of the grove it is essential to assess the impact of the ripening stage on the agronomic parameters and the quality of VOO. Currently, due to the spreading of mechanized harvesting, cultivars with low resistance to detachment are replacing native cultivars in Andalusia, resulting in a loss of diversity in olive oil varieties in this region. The Picudo variety – whose name ("prominent peak") comes from the shape of the drupe with a pointed and curved end – is a highly productive and widespread cultivar in Andalusia, where it is the fourth cultivar in terms of cultivated area. This cultivar currently covers more

Abbreviations: VOO, virgin olive oil; RI, ripening index; PCA, principal component analysis; UPLC-TOF-MS, ultrapressure liquid chromatography–time of flight mass spectrometry; IOOC, International Olive Oil Council.

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than 60,000 ha, which means that it is the tenth major olive cultivar in Spain. It is a specific variety of the Priego de Córdoba and Baena Protected Designations of Origin. It is very vigorous and is well-adapted to cold regions with limestone soils. This olive variety is mainly used for oil production. Despite the economic relevance of Picudo olive oil, there is little literature available on this highly productive cultivar (Gómez-Rico, Fregapane, & Salvador, 2008).

Considering all these aspects, the objective of this study was to further investigate the impact of ripening stage on the quality, agronomic parameters, sensory properties and phenolic content of Picudo olive oil. A multivariate descriptive statistical analysis allowed us to establish a relationship between the high number of experimental variables and the olive oils analyzed. In addition, we identified the variables with the greatest impact on the global quality of VOO throughout the ripening process. Therefore, the ultimate goal of this study is to determine the optimal harvesting time for Picudo olives in order to obtain olive oils with the best agronomic, physicochemical and sensorial properties.

2. Materials and methods

2.1. Chemicals

The standards of apigenin, luteolin, 6-methoxyluteolin, hydroxytyrosol, tyrosol, vanillin, 4-hydroxybenzoic, caffeic, *p*-coumaric, ferulic, sinapic and vanillic acids were purchased from Sigma-Aldrich (St. Louis, MO); and oleuropein was acquired from Extrasynthese (Genay, France). Stock solutions of phenolic compounds were prepared in methanol and stored at −20 °C. All the solvents used were of analytical or HPLC grade (Sigma-Aldrich). Water was of Milli-Q quality (Millipore Corp, Bedford, MA, USA).

2.2. Olives

Olive fruits were sampled from Picudo olive trees grown in the Mina Experimental Farm (Cabra, Andalusia, Spain), 37º30′N4º31′ W. The olive grove (27 ha) is located on a south-facing slope with only some light changes in the lateral curvature. The parent materials of the soil are white, miocene calcareous sandy marls. Elevations range from 500 to 550 m above mean sea level. The slope gradient varies from 0 to 20%, with an average gradient of 10%. The climate of the area is Mediterranean subtropical (papadakis). The climatic conditions during 2010 were as follows: annual accumulated precipitation = 1058.2 mm; annual average temperature = 17 °C; minimum temperature reached = −3.5 °C; maximum temperature reached = 40 °C.

Ten adult 16 year-old randomly chosen olive trees of the Picudo variety, spaced 12 × 12 m^2 were used in this study. Olive samples were randomly hand-picked weekly, starting on 25/10/2010 and ending 20/12/2010 (when there was no fruit left on the trees as a result of natural detachment). Every sampling date, all olive samples were homogenized and 100 fruits were randomly chosen for identification of their ripening stage. The olive ripening index (RI) was determined according to olive skin and pulp colour according to standards set by the International Olive Oil Council (IOOC, 1984). Only healthy fruits without any kind of infection or physical damage were processed.

2.3. Agronomical indices

2.3.1. Resistance to detachment (RD)

This parameter stands for the fruit resistance to detachment. It was evaluated using a dynamometer (Correx, Switzerland) measuring the force (in grammes) necessary to break the fruit peduncle and detach it from the branch.

2.3.2. 100-Drupe weight

Fruit weight was determined as the weight of 100 drupes randomly picked from aliquots of samples previously homogenized.

2.3.3. Oil content and humidity

Sample humidity was determined by drying olive fruits in an olive stove at 105° C according to the UNE Standard Spanish Method (UNE 55-020-73). A 40 g-sample is placed into a porcelain capsule and dried in a stove at 105 °C for six hours. Subsequently, the sample was cooled in a desiccator, weighted and reintroduced into the oven. This operation was repeated until variations in humidity weight loss and volatile loss were < 0.02 g. Subsequently, we used the Soxhlet extraction method to determine the total oil content using hexane (UNE 55030). Then, the dry matter used for the determination of humidity was introduced into the Soxhlet extractor, and oil extraction was performed with hexane for four hours. Afterwards, solvent traces were eliminated in the oven at 105° C. Then, using the oil extracted, oil content was determined with respect to the dry matter weight (% dry weight), and with respect to the wet matter weight (% wet weight).

2.4. Oil samples

Virgin olive oil samples were obtained using an Abencor analyzer (Abengoa S.A., Sevilla, Spain). This system reproduces the industrial process at a laboratory scale and consists of three basic elements: hammer mill, thermobeater, and pulp centrifuge (Martínez, Muñoz, Alba, & Lanzon, 1975). The milling of olive fruits was performed using a stainless steel hammer mill operating at 3000 rpm equipped with a 5 mm sieve. The resulting olive paste was immediately kneaded in a mixer at 50 rpm for 30 min at 30 °C. Centrifugation of the kneaded olive paste was performed in a basket centrifuge at 3500 rpm for 1 min. After centrifugation, the oil obtained was decanted and stored in amber glass bottles at 4 °C in darkness without headspace until analysis.

2.5. Analytical indices

Free acidity, peroxide value, and UV spectrophotometric indices (K_{232}, K_{270}) were evaluated according to the official methods described in Regulation EEC 2568/91 of the Commission of the European Union. All parameters were determined in triplicate for each sample.

2.6. Sensory analysis

An organoleptic analysis was performed by the panel of the "Priego de Cordoba" Designation of Origin laboratory during a series of oil-tasting sessions, in accordance with the European Community (EC) Regulation no. 640/2008. The tasters smelled and tasted each olive oil sample to evaluate its positive attributes according to the list of descriptors included in the method proposed by the IOOC (2005) for the *Organoleptic Assessment of Extra virgin Olive Oil labelled with a Designation of Origin*. The sensory profile of each VOO sample is expressed as the median value for each descriptor. The tasters evaluated direct or retronasal aromatic olfactory sensations (fruity, green leaf/fresh-cut grass, apple, almond, artichoke), and other positive attributes; gustatory sensations (bitterness and sweetness); and tactile/kinesthetic sensations (pungency). The tasters had to rate the intensity of the different descriptors on a continuous 0–10 scale. The total organoleptic score given to each oil sample was evaluated by the 12-member panel, according to the method described in Annex XII of the European Union Regulation (EC/2568/91). Each oil sample was rated according to a nine-point scale, 1 being the value for the poorest possible quality and 9 for the best possible quality.