

Influence of the fruit ripening degree on the virgin olive oil quality and conservation of *Empeltre* olives cultivated in a disadvantaged area

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Abstract

Physicochemical and organoleptical olive oil properties might be greatly influenced by conditions of the growing area such as climate and orography. Thus, the influence of the particularities of the Tramuntana region (in the Balearic Island of Mallorca) may affect the quality preservation of the *Olea europea* var. *Empeltre* olive oil. The main objective of this work was to determine the optimal harvesting period of the *Empeltre* olives in order to obtain high quality olive oil and appropriate oxidative stability during the storage period. For this purpose, olive samples were harvested at different ripening degrees. Thus, acidity, peroxide index and the extinction coefficients (K_{232} and K_{270}) were evaluated every three months during a storage period of nine months. In addition, the oxidative stability was measured by the Rancimat method at the end of the storage period. Olive oil obtained at early stages of ripening presented lower acidity values, lower peroxide index, and, also, high oxidative stability than samples harvested at later stages of ripening. Evaluation of the olive oil main characteristics during the storage period, indicated that no significant changes were found between olive oil samples prepared from different olives types. Similar results were observed for acidity and for specific extinction coefficients (K_{232} and K_{270}) values. However, peroxide index values were significantly lower and oxidative oil stability was higher when the olives used for the oil elaboration were collected at the beginning of the harvesting period. Moreover, although the organoleptical analysis showed that all olive oil sample types could be considered as extra virgin oils, important differences on the fruity attribute were detected between different olive oil types.

The possibility to improve the olive oil quality and, in addition, the preservation of the *Empeltre* olives cultivated in this disadvantaged region and under these conditions seemed to go forward the harvesting period.

Keywords: Virgin olive oil, ripening degree, oil quality, *Olea europea* var. *Empeltre*.

Introduction

Olea europea var. *Empeltre* is an olive tree commonly cultivated in the Mediterranean area and used for olive oil production. Among the different categories of olive oil, the 'extra virgin olive oil' is outstanding in gastronomic, nutritional, therapeutic and economic importance (Méndez & Falqué, 2007). There is enough scientific evidence supporting that the increase on oil into the Mediterranean diet is associated with a reduction of overall, cardiovascular and cancer mortality and neurodegenerative diseases (Salvador et al., 2001).

In Majorca Island, the *Empeltre* variety is one of the most cultivated olive-tree and the oil from this variety is commercialized together with other varieties under the Protected Designation

of Origin (PDO) “Oli de Mallorca”. The oils obtained from this variety are highly appreciated in the origin region. In 2009, around 1449 ha of olives were inscribed under this PDO, and a 70% of those olive trees correspond to the Empeltre variety.

This cultivar is located mainly in the Tramuntana mountain chain. According to the Directive 75/268/CEE, this location is considered in the European Community list, as a disadvantaged area for agriculture with particular crop conditions. Oil quality from an organoleptical and physicochemical point of view might be affected by external factors linked to those region conditions. The particular climate and orography of olive crop in this area determines the optimal period of olive crop, which differs from other regions. The olive ripening changes according to the growing area, olive variety, temperature and cultural practices.

During ripening, important chemical changes occur inside the drupes, which are related to the synthesis of chemical compounds that may affect virgin olive oil quality (Salvador et al., 2001). Unless the mainly effects of the maturation degree over yield, color, fruit deterioration, etc., ripening degree should be considered a priority criterion in order to define the physicochemical and organoleptical quality of oil. This is because of the chemical composition of the oils elaborated depending on the collection time; often more important than the effects due to the variety factor (Sánchez-Casas et al., 2006).

Moreover, since oil is produced in a limited period of time, but consumed throughout the year, it must be stored, and the initial conditions are going to determine the commercial life of the olive oil. Thus, the quality of virgin olive oils decreases over the course of time as a consequence of oxidative and hydrolytic degradations which might also promote the partial loss of other minor constituents with health-promoting effects (Caponio et al, 2005). The storage conditions of bottled virgin olive oil, as well as all the agronomical and technological variables of the processing stages, are particularly relevant for preserving the highly valued quality of this product (Bendini et al, 2009).

The main objective of this research was to determine the optimal harvesting period of Empeltre olives cultivated in a disadvantaged area in view to obtain extra high quality olive oil and evaluating its preservation during a storage period of 9 months.

Materials and methods

Experimental field and plant material

The experiment was carried out in Sóller in a traditional farm, located in the Tramuntana chain mountains at 195 m of altitude and close to the sea. The climate conditions were, in 2009: average temperature, 17.39°C; relative humidity, 71.72% and total rainfall 1082 mm. Olive-trees (*Olea europaea* var. Empeltre) were spaced at 8 x 8 m and, in each sampling, 10 kg of healthy olives were picked by hand from the selected trees. Olives were carried the same day to the pilot plant at the Institute of Agriculture Research and Training and processed before 24 h. Two different stages of maturity were harvested: (i) when most of the fruits were comprised between yellowish to green with red spots colour and (ii) when most of olives skin colour varied from purple in more than half fruit to black.

Ripening index

The ripening description followed the method described by the International Olive Oil Council in a randomly sample of 100 olives (Salvador et al., 2001). Therefore, olives were classified in the categories: 0- intense green; 1- yellowish green; 2- green with red spots; 3- red or purple in more than half of the fruit; 4- black with white pulp; 5- black with half purple pulp; 6- black with purple pulp; 7-black with completely purple pulp (Uceda & Frías, 1975). The

ripening index (RI) was calculated according to the equation 1, where N_i indicates the number of fruits from each i categories, from 0 to 7.

$$RI = (\sum N_i \cdot i) / 100 \quad [1]$$

Oil extraction and storage conditions

Two virgin olive oils from each stage of ripening were extracted separately using a laboratory oil mill Abencor (Comercial Abengoa, Seville, Spain). The olive paste was kneaded for 45 min at 28°C. The oil obtained was left and separated by decanting. The total amount was measured in a test tube, filtered and bottled. The oil content was calculated as a percentage of fresh olive paste weight and expressed as g of oil/100 g olives.

Oil samples were stored in 250 ml amber glass in a controlled chamber at 20°C at darkness. At established intervals, samples were withdrawn from storage and the physicochemical analyses were carried out. The sampling times were the following: initial (0 month), 3, 6 and 9 months after bottling.

Physicochemical analyses

The physicochemical quality parameters were determined at the beginning of the oil extraction and during a storage period of nine months. Oil samples were monitored every three months. Analyses were carried out following the analytical methods described by Regulation (EC) No 2568/91 of the European Union Commission. Free acidity was given as percentage of oleic acid and determined by titration with 0.1N KOH of an oil solution in a previously neutralized solvent (ethanol: ethyl ether, 1:1) and using phenolphthalein as indicator. Peroxide value was expressed as milliequivalents of active oxygen per kg of oil (meq O_2 /kg) and determined by a mixture of oil, chloroform and acetic acid left to react with potassium iodide in darkness. The free iodine was then titrated with a 0.01 N sodium thiosulfate solution. The specific extinction coefficients, K_{270} and K_{232} , were measured from absorption in cyclohexane solution at 232 and 270 nm, respectively, with 1 cm path length in an UV/VIS DU 700 series spectrophotometer (Beckman Coulter, Inc., USA).

Oxidative stability

The oil oxidative stability was measured at the end of the storage period using a Rancimat Model 679 apparatus (Metrhom Co., Basel, Switzerland); 5 g of oil were weighed and heated at 100°C and air was bubbled through the oil at a flow rate of 10 l/h. Results were expressed as induction time in hours (Gutiérrez, 1989).

Sensory analysis

The sensory evaluation was carried out by a trained and certified tasting panel of the Agrifood Laboratory of Atarfe (Granada, Spain). The oil was graded with the median of the defects and the median for the “fruity” attribute, according to the method described in the Regulation (EC) No 640/2008 of the European Union Commission.

Results and discussion

Samples ripening and oil efficiency

Olive oil quality can be affected by the ripening degree of the olives at harvesting. The two different samples considered in this study were, the first one (RI 1), when olive skin was between yellowish to green with red spots, and the second (RI 2), one month later, when olives colour was from purple in more than half fruit to black. Table 1 presents oil samples harvesting date, fruit ripening index (RI), olive weight and oil efficiency.

Table 1: Sampling characteristics of Empeltre olives harvested at early and late stages of maturity.

Sample code	Harvesting date	RI	Olive weight (g)	Oil content (g/100 g olives)
RI 1	29/10/2009	1.41	2.63±0.32	16.12±1.00
RI 2	25/11/2009	3.72	3.74±0.41	19.39±1.36

As expected, samples harvested in November presented more than 2 ripening index degrees than the oil obtained in October. The oil performance of the samples attained higher olive weigh and oil efficiency as the ripening index increased. Thus, the olive weigh and oil content obtained at late stages of ripening (RI 2) allowed increases of both parameters of 43% and 20%, respectively, than oil extracted at early periods (RI 1).

Physicochemical analysis

Physicochemical analysis of both oil samples, RI 1 and RI 2, was monitored every three months during the complete storage period.

Results of the free acidity are shown in figure 1a) as mean with standard errors. Note that lower values for this parameter will translate into a higher quality of oil. As can be observed, an increase in free acidity was observed as ripening progressed. Thus, acidity values ranged from 0.19 % in RI 1 to 0.34 % at RI 2 samples at the beginning of the storage period and from 0.17 % in RI 1 to 0.38% at RI 2 samples after 9 months of conservation. The same behavior has been observed by different authors for Cornicabra variety (Salvador et al., 2001) and for Blanqueta and Arbequina varieties (García et al., 1996). Olives at later stages of ripening give oils with higher levels of free acidity since they undergo an increase on enzymatic activity, especially by lipolytic enzymes, and more sensitive to pathogenic infections and mechanical damage (Salvador et al., 2001).

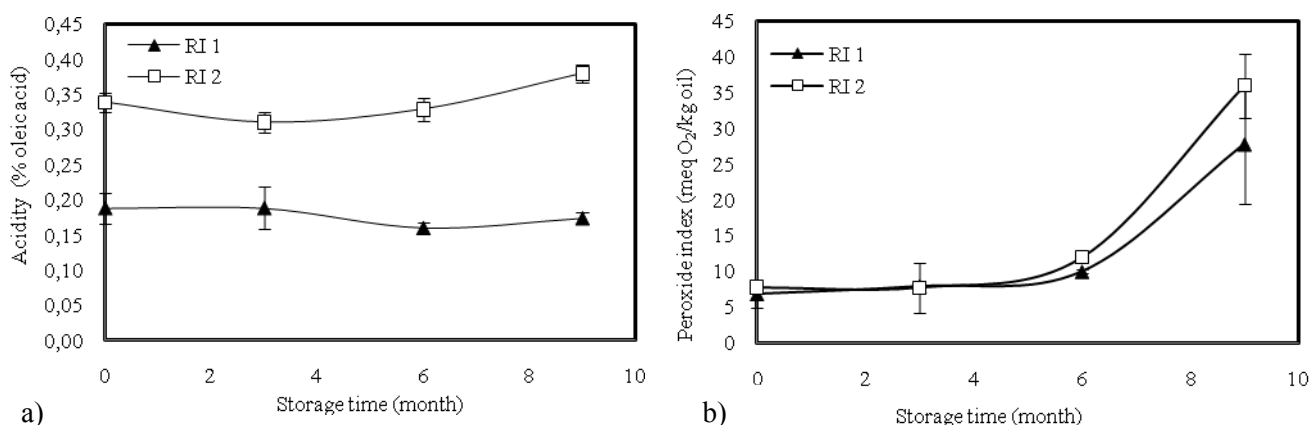


Fig. 1: Evolution of quality parameters of Empeltre olive oil harvested at different stages of maturity; acidity (a) and peroxide value (b) during olive oil storage.

With regard to the free acidity evolution during the storage period, after 9 months of oil preservation, a slightly increase in acidity was observed on RI 2 samples, although no significant changes were detected. Therefore, free acidity did not change during the storage time for each sample in the analyzed conditions. These results are in agreement with the study performed by Méndez & Flaqué (2007) during 3 months, which confirmed the increase of acidity in olive oil over time. In all ripening degrees, free acidity values throughout the storage conditions were significantly lower than the upper limit of 0.8 % required by the PDO 'Oli de Mallorca' Regulation APA/172/2003 for the extra virgin oil.

The initial peroxide index of the olive oils analyzed (fig. 1b) did not show any difference between both maturity stages. Values of this parameter at the beginning of the storage period ranged from 6.83 to 7.67 meq O₂/kg in RI 1 and RI 2, respectively. These results were lower than the maximum values indicated by the PDO Regulation. Initial peroxide values were clearly higher to those presented by Mendez & Falqué (2007) in commercial oil of 18.3-19.7 meq O₂/kg, probably due to the use of only healthy fruits and the small scale system used for processing in this study. However, Pardo et al. (2007) presented slightly lower peroxide index in laboratory scale processing system, ranging from 3.30 to 5.70 meq O₂/kg for different varieties; Picual, Cornicabra, Manzanilla, Arbequina and a local variety.

When considering the oil evolution, 6 months after the extraction process, both samples increased significantly the peroxide index value and undergo the maximum value of 18 meq O₂/kg established for this regulated category. This was an indicator of the oxidation reactions in advanced phases due to the sensibility of extra virgin oil to photo-oxidation. According to results presented in this figure, oil samples at more advanced stage of maturity (RI 2) were degraded significantly faster from the ninth month than oils extracted earlier (RI 1).

Results of the ultraviolet absorbance at 232 and 270 nm are presented in table 2. Initial K₂₃₂ values were similar for oils obtained at early and late stages of maturity as reported Salvador et al. (2001) in a similar study, on which a clear trend on this parameter during ripening was not displayed. However, when the storage period was evaluated, an increase on K₂₃₂ was reflected after six months in both samples, especially in RI 2 samples at the end of the storage period.

The absorbance at 270 nm, which is an indicator of primary and secondary oxidations, did not show any difference between both samples during the complete storage period. All samples at any storage period accomplished the Regulated limits of 2.5 and 0.22, in K₂₃₂ and K₂₇₀, respectively.

Table 2: Evolution of spectrophotometric absorptions at 232 and 270 nm of Empeltre olive oil harvested at different stages of maturity during olive oil storage.

<i>Storage period (Months)</i>		<i>K₂₃₂</i>	<i>K₂₇₀</i>
RI 1	0	1.49 ± 0.06	0.14 ± 0.03
	3	1.52 ± 0.03	0.10 ± 0.00
	6	1.65 ± 0.10	0.10 ± 0.01
	9	1.81 ± 0.01	0.09 ± 0.00
RI 2	0	1.57 ± 0.12	0.12 ± 0.01
	3	1.48 ± 0.02	0.11 ± 0.01
	6	1.62 ± 0.02	0.11 ± 0.00
	9	1.91 ± 0.04	0.11 ± 0.01

Oxidation stability

The Rancimat method can provide an easy and fast test related to the resistance of oil to oxidation. The stability of the oils elaborated at different stages of maturity at the end of the storage period was 67.91 h for RI 1 samples, however, increasing the ripening degree of olives at harvesting, RI 2 samples, the oxidation stability decreased until 59.22 h. These results revealed the highest antioxidant potential of RI 1 samples, verifying the lesser degree of oxidation obtained by the peroxide value and K₂₃₂. Those values were lower to those reported by other authors studying different varieties grown in other areas (Pardo et al., 2011), probably because of the low stability of this variety.

Sensory analysis

All olive oils harvested at different degrees of maturity were classified as ‘extra virgin oil’ by the recognized oil taster panel, as shown in table 3. The highest fruity attribute was detected in oil samples RI 1, which increased in a 40% the oil samples extracted at later stages of maturity.

Table 3. Sensory evaluation of Empeltre olive oil at different stages of maturity.

Sample code	Median of fruity	Median of defects	Panel test classification
RI 1	7.00	0.00	Extra Virgin
RI 2	5.70	0.00	Extra Virgin

Conclusions

Extra virgin olive oil samples at early stages of maturity (RI 1=1.41) exhibited lower oil efficiency at the expense of an improvement on the initial free acidity value, peroxide value and oxidative stability than olive oil extracted at late stages of maturity (RI 2=3.72). Moreover, the oil degradation during storage was more accentuated in RI 2 samples, with higher free acidity, peroxide value and K_{232} . Sensory classification was ‘extra virgin oil’ in both samples, however, RI 1 samples achieved higher fruity attribute.

Acknowledgments

The authors thank the INIA pre-doctoral and FOGAIBA scholarship grants and the financial support provided by the Ministry of Education and Science of Spain (Project RTA2009-00119-C02).

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