Influence of storage of extra virgin olive oil on physicochemical and sensory parameters

S. Adrover[1], M.C. Garau[1], C. Rosselló[2], S. Simal[2], A. Femenia[2*].

1. Institute of Agricultural Research and Training (IRFAP). Government of the Balearic Islands, C/ d'Eusebi Estada, 154, 07009 Palma (Balearic Islands, Spain).
2. Department of Chemistry, University of the Balearic Islands, Ctra. Valldemossa km. 7.5, 07122 Palma (Balearic Islands, Spain).

*Corresponding Author: antoni.femenia@uib.es

Abstract

The acceptance of olive oil by consumers mainly depends of its organoleptic characteristics. In this work the evolution of physicochemical and sensory characteristics of olive oil has been determined (positive and negative attributes) by using different methods of storage: (1) at temperature of 20ºC, (2) at using a modified atmosphere, (3) at absence of light and (4) at low-temperature (6-8ºC). All oil samples were stored in glass bottles (250 ml). Olive oil used was obtained from olives of the Empeltre variety. It is important to highlight that the initial olive oil samples exhibited a relatively high acidity value (0.74% oleic acid). Sensory analysis was performed by the Official Tasting Panel of Extra Virgin Olive Oil from Catalonia. Sensory analysis was carried out together with the evaluation of the main physicochemical properties (acidity, K_{270} and K_{232}, peroxide index and also total polyphenols), in order to establish the overall quality of the different olive oils. Oil samples were monitored, every two months.

The results indicated that, after 16 months of storage, the oil could only be classified as extra virgin when storage was carried out at low-temperature. The samples kept using other conditions, that is at 20ºC, in an inert atmosphere on under the absence of light, exhibited different negative sensory attributes (in particular humidity and rancidity) after only 4 months of storage. Furthermore, all the latter samples presented a free acidity exceeding the limit of 0.8% oleic acid, after this four month period.

Therefore, it seems that temperature is the key factor to avoid or minimize the oxidation process in extra virgin olive oils when their initial degree of acidity is close to the limit established by the actual EU legislation (Regulation EEC 2568/91).

Keywords: Extra virgin olive oil, storage conditions, Empeltre, organoleptic properties

Introduction

Olive oil is widely used in the Mediterranean cuisine and is appreciated for its delicious taste and aroma, as well as for its nutritional benefits (Baiano et al, 2009), primarily related to its balanced fatty acid composition and the presence of considerable amounts of natural antioxidants (Cioffi et al, 2010), which rank this product as first among dietary fats. Moreover, specific characteristics and the presence of other important components in the unsaponifiable fraction, which accounts for the nutritive and health-giving properties, distinguish extra-virgin oil from others edible vegetable oil.

However, 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). It is known that these reactions are catalyzed by light, heat and atmospheric

oxygen, and partly slowed down by compounds belonging to the unsaponifiable fraction (phenolic compounds, carotenoids, tocopherols, etc.) (Bendini et al, 2009).

The antioxidant effects of extra virgin olive oil seem to be a result of the presence of phenolic compounds, hidroxytyrosol, tyrosol, etc. which depends on the cultivar, climatic and degree of ripeness of the fruit. Carotenoids and α-tocopherol are also important antioxidant since they can protect oil from auto- and photo-oxidation (Psomiadou and Tsimidou, 2002a).

It is a matter of great concern for the olive oil industry to preserve the positive attributes of oil during the time elapsing from production to bottling, and up to purchasing. 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 organoleptic quality of this product (Bendini et al, 2009).

All the efforts made in the olive grove and in the oil mill to produce virgin olive oil with good sensory characteristics can be undermined if improper storage conditions are used.

This research was conducted to determine the effects of different storage conditions on the physicochemical and sensorial characteristics of virgin olive oil.

Materials and Methods

a) Olive oil samples and storage conditions

Extra virgin olive oil was produced from the Empeltre cultivar (Coop. Sant Bartomeu, Sóller 2008) at a centrifugation system (tree-phases decanter). Once in the laboratory, the oil was poured into 250 ml amber glass bottles. The first batch (*control*) was stored in these bottles in a thermostated chamber at 20ºC and under diffuse light (11W, 595 lm and 12h/day), simulating the conditions of supermarket shelf, the second batch was stored after displacing the headspace air by argon (*atmosphere*) at 20ºC, the third series was stored in darkness (*Dark*) at 20ºC, and finally, the fourth batch was stored in a cool room at 4-6ºC (*temperature*).

At pre-established intervals the samples were withdraw from storage and analysed. The sampling times were the following: initial (0 month) and 2, 4, 6, 8, 10, 12 and 16 months after bottling and storage in the conditions already described.

b) Analysis of Virgin Olive Oil

Quality and Oxidation Indices

Free acidity as percentage of oleic acid; peroxide value (PV) expressed as milliequivalents of active oxygen per kilogram of oil (meq O_2/kg) and K_{232} and K_{270} extinction coefficients, calculated from absorption at 232 and 270 nm, were measured following the analytical methods described in European Commission Regulation (EEC) 2568/91 and later amendment.

Total phenol content

Phenolic compounds were extracted following the procedure of Vazquez-Romero et al, (1974). The total phenol content was determined spectrophotometrically at 725 nm, and the concentration was expressed as milligrams of caffeic acid per kilogram of oil.

Sensory analysis

Sensory evaluation of the olive oils was performed by the "Pannell de Tast Oficial d'Olis Verges de Catalunya", according to the previous European Community Regulation (EC) No 640/2008. The panel was constituted at least by 9 tasters, trained and prepared physically and psychically to distinguish between similar samples. Tasters

scored the flavour descriptors of the samples on a normalized sheet (from 0 to 10). The average score was calculated for each sample. Sensory descriptors can be classified into "positive attributes" (green or ripe fruity and other pleasant attributes such as leaf, bitter, pungent, sweet, astringent, grass artichoke, tomato, almond, apple, others) and "negative attributes" (winey-vinegary, fusty, mouldy, muddy sediment, rancid, metallic, others).

c) Statistical analysis

Data were subjected to analysis of variance using the Excel 2003 software program. Where statistical differences were noted, differences among packages were determined, using the last significant difference (LSD) test. Significance was defined at $p<0.05$.

Results and Discussion

a) Physicochemical parameters

The results obtained in this study clearly show that when olive oil samples, exhibiting an initial acid value (0.74% oleic acid), are stored under different conditions, the use of an inert atmosphere or darkness is not sufficient to stop the auto-oxidation reactions which diminish the overall quality of the olive oil.

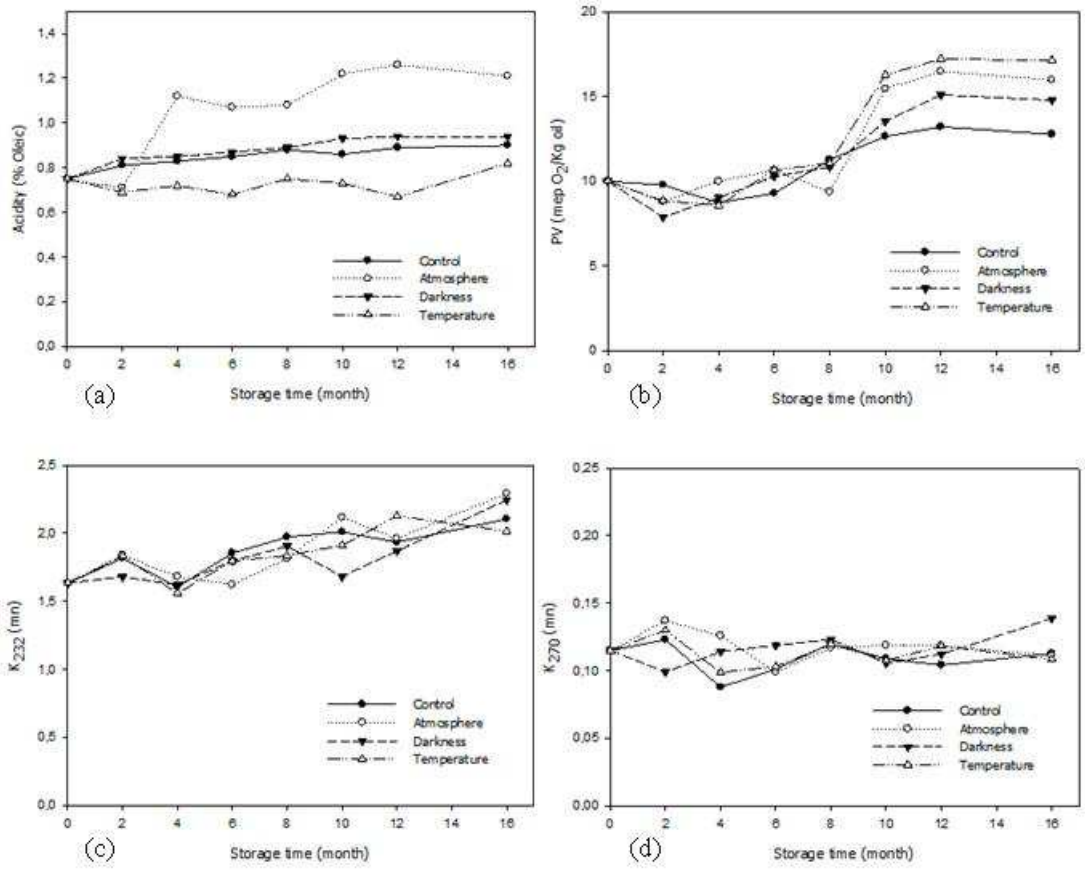

Figure 1. Evolution of quality parameters, acidity (a), peroxide value (b), K_{272} (c) and K_{232} (d) during olive oil storage.

In fact, only the use of low temperature was effective in controlling the oxidation of lipids. This can be clearly inferred from the evolution of acidity (Fig 1a). Thus, samples stored at 20 ºC (control, inert atmosphere and darkness) from the fourth month of storage failed to meet the requirements of extra virgin olive oil (0.8 % oleic acid). On

the contrary, samples stored at low temperature kept the status of extra virgin up to 16 months.

Other quality parameters (Fig 1b, 1c and 1d), showed an increase with time for the period analysed, however the limits corresponding to a quality extra virgin olive oil were not exceeded during storage (PV < 20 meq O_2/kg oil; K_{232} < 2.5 and K_{270} < 0.22).

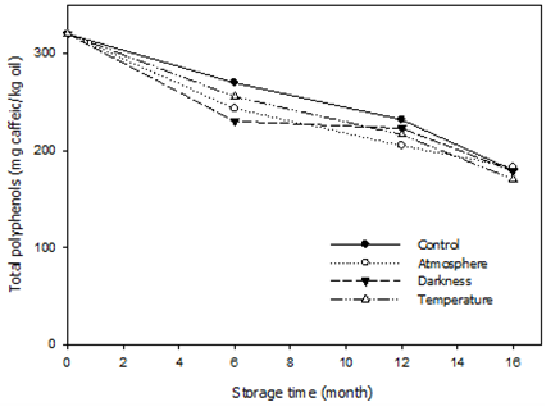

Figure 2. Evolution of total polyphenols during olive oil storage period.

The phenolic composition is associated with the oxidative stability, the biological properties and the sensory characteristics of the olive oil (Inajeros-Garcia et al, 2010). Total polyphenols were evaluated at 0, 6, 12 and 16 months of storage (Fig.2). As it can be observed, total polyphenols decreased during storage and no significant differences were observed during storage. This trend has been observed by other authors for studies related to the olive oil storage, under the presence of light (Caponio et al, 2005), using conditions of inert atmosphere (Cinquanta et al, 1997) and studying the effect of temperature (Di Giovacchino et al, 2002).

b) Sensory parameters

The positive attributes of the different olive oil samples are presented in Table 1. As it can be seen, initially, the olive oil of Empeltre variety was characterized by a high intensity of fruity olive, which also included the attributes of sweetness, pungent, bitter green leaf and, also, the feature of not being too astringent.

Samples stored at 20ºC (control, atmosphere and darkness) exhibited an overall decrease of these attributes during the storage period. This decrease could be partially justified by the observed decrease of the total polyphenol content (Inajeros-Garcia et al, 2010).

However, in the case of samples stored at low temperature, the decrease of the different attributes was less marked, showing clearly that this method of storage better preserved the organoleptic properties of the extra virgin olive oil.

In table 2, the intensity values corresponding to the sensory defects are presented. As it can be seen, samples stored at 20 ºC (control, atmosphere and darkness), from the fourth month of storage started to show sensory defects, in particular humidity and stale. From this point, these attributes increased in intensity over time. However, the samples stored at low temperature, did not present any negative attribute for the period analyzed.

Table 1. Intensity evolution of positive attributes during olive oil storage (mean ± standard deviation)

	Storage (month)						
Positive atributes	**0**	**4**	**6**	**8**	**10**	**12**	**16**
Control							
Fruity	5,0±0,4	2,8±0,4	2,3±0,4	2,4±0,5	2,4±0,5	2,5±1,0	2,0±0,5
Green leaf	3,7±0,4	1,6±0,2	0,7±0,6	0,6±0,7	0,5±0,7	0,4±0,4	0,0±0,0
Bitter	4,4±0,5	2,8±0,3	2,3±0,3	3,1±0,7	2,0±0,5	1,8±0,2	2,2±0,4
Pungent	4,7±0,4	3,6±0,3	3,3±0,4	3,2±0,4	2,2±0,4	2,2±0,6	2,2±0,5
Sweet	5,0±0,2	4,0±0,2	4,0±0,3	4,0±0,1	4,2±0,3	4,1±0,3	4,2±0,5
Astringent	2,0±0,2	1,5±0,5	0,0±0,5	0,5±0,9	0,0±0,0	0,0±0,0	0,0±0,0
Atmosphere							
Fruity	5,0±0,4	3,2±0,5	2,7±0,6	2,5±0,8	2,3±0,4	1,7±1,2	1,0±0,3
Green leaf	3,7±0,4	1,6±0,0	1,6±0,4	0,0±0,5	0,0±0,0	0,0±0,6	0,0±0,0
Bitter	4,4±0,5	3,0±0,3	3,0±0,3	2,2±0,3	1,9±0,5	1,9±0,3	1,7±0,8
Pungent	4,7±0,4	3,7±0,5	3,6±0,2	3,7±0,3	2,8±0,2	2,6±0,6	2,6±0,5
Sweet	5,0±0,2	4,6±0,1	4,4±0,3	4,2±0,2	4,5±0,4	4,5±0,2	4,5±0,6
Astringent	2,0±0,2	0,0±0,0	0,0±0,1	0,0±0,5	0,0±0,0	0,0±0,0	0,0±0,0
Darkness							
Fruity	5,0±0,4	2,8±0,3	1,7±0,7	1,7±0,7	1,7±0,0	1,7±0,7	0,0±0,7
Green leaf	3,7±0,4	0,6±0,4	0,6±0,6	0,0±0,6	0,0±0,0	0,0±0,0	0,0±0,0
Bitter	4,4±0,5	3,0±0,3	2,6±0,4	3,0±0,6	1,7±0,3	1,6±0,5	1,5±0,5
Pungent	4,7±0,4	3,3±0,4	3,1±0,3	2,8±0,4	2,9±0,4	2,9±0,3	2,9±0,3
Sweet	5,0±0,2	4,6±0,3	4,5±0,7	4,1±0,1	4,2±0,3	4,2±0,4	4,2±0,4
Astringent	2,0±0,2	0,0±0,0	0,0±0,1	0,0±0,6	0,0±0,0	0,0±0,0	0,0±0,0
Temperature							
Fruity	5,0±0,4	3,8±0,4	3,9±0,5	3,9±0,4	3,9±0,5	3,9±0,4	3,1±0,4
Green leaf	3,7±0,4	2,5±0,3	2,0±0,6	2,0±0,6	1,8±0,0	1,8±0,4	1,9±0,2
Bitter	4,4±0,5	4,0±0,5	4,0±0,4	4,0±0,3	3,7±0,5	3,7±0,3	3,5±0,3
Pungent	4,7±0,4	4,4±0,7	4,2±0,5	4,4±0,3	4,3±0,3	4,4±0,3	4,4±0,1
Sweet	5,0±0,2	4,1±0,1	4,2±0,6	4,3±0,2	4,4±0,3	5,0±0,3	4,5±0,1
Astringent	2,0±0,2	1,5±0,5	1,5±0,6	1,5±0,4	1,5±0,6	1,3±0,5	1,6±0,4

Table 2. Intensity evolution of negative attributes during olive oil storage (mean ± standard deviation)

	Storage (month)						
Defects	**0**	**4**	**6**	**8**	**10**	**12**	**16**
Control							
Muddy	0,0±0,0	0,0±0,7	0,0±1,0	0,0±0,7	0,0±0,8	0,0±0,0	3,3±1,2
Musty	0,0±0,3	2,4±0,4	4,1±1,0	4,1±1,4	4,1±0,7	4,0±0,6	4,7±1,5
Winey-Vinegary	0,0±0,0	0,0±0,5	0,0±0,9	0,0±0,6	0,0±0,7	0,0±0,6	0,0±0,8
Metallic	0,0±0,0	0,0±0,0	0,0±0,0	0,0±0,0	0,0±0,0	0,0±0,0	0,0±0,0
Rancid	0,0±0,0	0,0±0,7	0,0±0,5	0,0±1,2	0,0±0,7	0,0±0,8	4,0±1,1
Atmosphere							
Muddy	0,0±0,0	0,0±0,8	0,0±0,3	0,0±1,4	0,0±0,7	0,0±0,0	0,0±1,0
Musty	0,0±0,3	2,4±0,5	2,7±0,5	3,1±0,8	3,4±0,3	3,6±0,6	6,0±1,0
Winey-Vinegary	0,0±0,0	0,0±0,5	0,0±0,6	0,0±0,9	0,0±0,3	0,0±0,6	2,3±0,9
Metallic	0,0±0,0	0,0±0,0	0,0±0,0	0,0±0,0	0,0±0,0	0,0±0,0	0,0±0,0
Rancid	0,0±0,0	0,0±1,2	2,6±0,6	2,6±0,6	2,6±0,8	3,7±0,8	5,4±0,8
Darkness							
Muddy	0,0±0,0	0,0±0,5	0,0±0,5	0,0±1,3	0,0±1,4	0,0±0,6	0,0±1,1
Musty	0,0±0,3	3,9±0,9	3,9±0,6	3,9±1,0	3,7±0,9	4,2±0,7	7,8±0,1
Winey-Vinegary	0,0±0,0	0,0±0,5	0,0±0,9	0,0±0,3	0,0±0,8	0,0±0,3	0,0±1,1
Metallic	0,0±0,0	0,0±0,0	0,0±0,0	0,0±0,0	0,0±0,0	0,0±0,0	0,0±0,0
Rancid	0,0±0,0	2,0±0,6	2,0±0,6	2,2±0,6	2,2±0,4	2,2±0,6	3,7±1,6
Temperature							
Muddy	0,0±0,0	0,0±0,4	0,0±1,0	0,0±0,8	0,0±1,0	0,0±0,3	0,0±0,4
Musty	0,0±0,3	0,0±0,4	0,0±1,1	0,0±1,0	0,0±0,9	0,0±0,4	0,0±0,8
Winey-Vinegary	0,0±0,0	0,0±0,0	0,0±0,9	0,0±0,0	0,0±0,8	0,0±0,0	0,0±0,0
Metallic	0,0±0,0	0,0±0,0	0,0±0,0	0,0±0,0	0,0±0,0	0,0±0,0	0,0±0,0
Rancid	0,0±0,0	0,0±0,0	0,0±0,7	0,0±0,0	0,0±0,6	0,0±0,0	0,0±1,0

Conclusions

Extra virgin olive oil samples, exhibiting an initial free acidity value of 0.7% oleic acid, stored at 20 ºC failed to maintain the maximum quality beyond the fourth month of storage, showing an increase in free acidity and, also, important negative sensory attributes. On the contrary, low temperature storage was able to preserve the maximum sensory qualities within the category of extra virgin olive oil up to 16 months. The occurrence of these defects could probably be a consequence of the reactions of lipid oxidation. Moreover, the use of an inert atmosphere and/or the presence of light might have been acting as catalysts.

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