UDC 664.8.03:665.327.3
ISSN 0352-9193

conference paper

Influence of Olive Storage and Processing on Some Characteristics of Olive Oil

Utjecaj uvjeta čuvanja i prerade maslina na neka svojstva maslinovog ulja

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Received: September 22, 1994
Accepted: June 19, 1995

Summary

Some properties and quality of olive oil obtained by pressing and by centrifugal extraction from fruits of blended varieties of olives (about 30–40 % of Oblica and about 60–70 % of Drobnica) and Lastovka variety were examined. Because of untoward circumstances the olive fruits were kept until processing in sea or tap water. The content of free fatty acids, peroxide value, unsaponifiable matter, chlorophyll, absorbances in the UV range and intensity of bitterness were determined in oils.

Keeping the fruits in the water (especially in sea water) from the time of harvest to the time of processing, has proved to be a fairly good method of olive storage, because in the oil samples kept in this way for several months no significant hydrolytical or oxidative changes occurred.

Olive oils obtained by the technological process of continuous centrifugal extraction have a higher content of chlorophyll and bitter substances than the olive oils obtained by pressing. The oil obtained from the olives of Lastovka variety had a particularly high intensity of bitterness.

Sažetak

Ispitivana su neka svojstva i kakvoća maslinovog ulja dobivenog prešanjem ili kontinuiranom centrifugalnom ekstrakcijom iz plodova miješanih sorti masline (oko 30–40 % Oblice i oko 60–70 % Drobnice) i masline Lastovke. Zbog nepovoljnih uvjeta plodovi su masline prije prerade čuvani duže vrijeme u morskoj ili slatkoj vodi. U tim je uljima određen udjel slobodnih masnih kiselina, peroksidni broj, neosapunjive tvari, klorofil, apsorbancija u UV-području i intenzitet gorčine.

Čuvanje maslina u vodi (posebno u morskoj), od berbe do prerade, pokazalo se kao dobar način skladištenja maslina jer (ni nakon nekoliko mjeseci) u uzorcima ulja nisu nastupile bitne hidrolitičke ili oksidacijske promjene.

Ulja dobivena centrifugalnom ekstrakcijom imala su veći udjel klorofila i tvari gorka okusa od ulja dobivenih prešanjem. Najjače izražen gorak okus imalo je ulje dobiveno iz masline Lastovke.

Introduction

The olive (*Olea europaea* L.) belongs among the oldest cultivated plants in the world. In the past it played a significant role in the life of the people: it gave wood for fuel and timber, the leaves for feed, the fruit for food, oil for nourishment, spice and medicine, and for light.

Olive oils is considered as an oil of a high biological and nutritious value. Even Hippocrates, the father of medicine, was aware of the therapeutical properties of olive oil, and it was recommended as food and medicine by Galen, an ancient physician *(1)*. Olive oil is generally used without refining, owing to which it maintains the specific aroma and all natural components it had in the fruit. Thanks to the high content (65–85 %) of mono-unsaturated oleic acid (18:1 ω9) and natural antioxidants, oxidation changes occur more slowly than in the oils which contain more polyunsaturated fatty acids. The ratio of tocopherol and polyunsaturated fatty acids in olive oil is optimal and higher than 1:8 (m (vitamin E)/mg) / (m (linoleic acid)/g), so that an increase in the daily consumption of essential fatty acids does not incur the loss of vitamin E in the body *(1)*. The properties of olive oil make its use as foodstuff, in pharmaceutical and cosmetic industry increasingly popular.

A series of factors affect the properties and quality of olive oil. These include above all the conditions of growing (climate and soil), and the cultivar of the olive,

Presented at the 2[nd] Croatian Congress of Food Technologists, Biotechnologists and Nutritionists, Zagreb, June 15–17, 1994 then the conditions and time of harvesting and storage of fruit prior to processing, as well as conditions of processing and storage of the oil. Inadequate conditions at any of these phases may cause hydrolytic and oxidative destruction of triacylglycerols and aromatic substances, resulting in products which will affect the quality and shelf life of this oil *(1)*.

The separation of oil from stoned and crushed olive fruits is performed mechanically in basically three ways: by pressing, centrifuging and by selective filtration. Oil may be obtained by a combination or modification of those technological processes, and by extraction from the olive residue (cake, pomace, husk) with organic solvents. Extraction of the husk yields technical olive oil which, when refined, may be used for food *(1,2)*. Depending on the quality, olive oil is classified into several categories according to official regulations *(1,3,4)*.

Experimental

The present study involved the examination of some properties and quality of olive oil obtained by pressing and by centrifugal extraction from fruits of blended cultivars of olives (about 30–40 % of Oblica and about 60–70 % of Drobnica) grown in the surroundings of Zadar and Lastovka in the surroundings of Dubrovnik.

Three samples of oil (marked 1P to 3P) obtained by pressing and three samples obtained by centrifugal extraction (marked 4C to 6C) were analyzed, as well as one sample of refined olive oil (7R). The samples came from the 1991/92 harvest, and because of extremely untoward circumstances (the war, electric power cuts) the harvest and processing were not carried out in the required period of time. The oil sample 1P was obtained from the olives collected from October 1991 until May 1992 and was processed in early June. The samples 2P and 3P were obtained from the fruits collected from December 1991 until March 1992, and were processed in February and March. From the harvesting time until processing the olive fruits were kept in sea or tap water. The samples 4C to 6C were obtained from olives collected from November 1991 to January 1992, and they were processed in the same period. Up to the processing time they had been kept in sea water. The obtained oils were kept in stone jars (samples 2–4), in plastic drums (samples 1–5) and in tins (sample 6) until the sampling time (September 1992).

The measurements involved the mass fraction of free fatty acids (FFA) and peroxide value *(3)*, unsaponifiable matter *(5)*, chlorophyll *(6)*, specific absorbances in the UV range (expressed as the absorbance of 1 % solution of the fat in the specified solvent, in a thickness of 1 cm) conventionally indicated by K from which ΔK *(3)* and R-values were estimated *(7)*. The assessment of the intensity of bitterness of olive oil was carried out according to the method by Gutierrez-Rosales *et al. (8)*, which is based on the measurement of absorbance at 225 nm of the extract eluted with a methanol/water mixture from the C18 column. The absorbances measured are calculated to 1 % solutions and as such used in the formula for the calculation of the intensity of bitterness.

Results and Discussion

The results of measurements are given in the following tables as mean values of two or more measurements.

According to regulations valid in Croatia *(4)*, virgin olive oil, depending on the FFA content, is classified into three categories: virgin olive oil extra – up to 1 % FFA, virgin olive oil fine – up to 2 % FFA and virgin olive oil up to 4 % FFA. According to the regulations by the European Community *(2)* the permissible FFA content for the third category amounts to 3.3 %.

The data given in Table 1 show that pressed oils had somewhat higher FFA content and a lower peroxide value than the oils obtained by centrifugal extraction. However, these quality properties are undoubtedly more influenced by the conditions of collection and storage of olive fruits, and also by the period and conditions of the storage of the oil than by processing method. The sample 1P had the highest acidity (3.3 %), which was to be expected, since, as mentioned earlier, it was obtained from the fruits collected and stored for an extended period. The lowest FFA content was found in the oil 4C (0.7 %), manufactured from the fruits collected in November and the first half of December, and which were kept in sea water for a short time prior to processing.

Hydrolytic changes in olive oil occur in the fruit itself and are caused by enzymes and microorganisms present. Inappropriate storage may prompt the enzyme activity and the growth of microorganisms, resulting in increased acidity. Keeping olive fruits in water, especially in sea water, has proved to be a fairly good way of storage, unless it is too long. Data in the literature recommend for extended keeping of olives an addition to the water of 3 % salt and 0.03 % of citric acid, or 2 % of metabisulphite, or 0.3 % of acetic acid *(1)*; the best storage is in a thin layer (10–15 cm) in cool and well aired premises *(1,2,10)* through a short period of time (8–10 days).

Storage of olives in water prevents the contact of the fruits with the oxygen from the air, which decreases the

Table 1. Free fatty acids, peroxide value, unsaponifiable matter and chlorophyll in the olive oil sample examined

Tablica 1. Slobodne masne kiseline, peroksidni broj, neosapunjive tvari i klorofil u ispitivanim uzorcima maslinova ulja

Samples*	FFA w (% oleic)	Peroxide value (mmol O_2/kg)	Unsaponifiable w/%	Chlorophyll w/(mg/kg)
1P	3.3	3.52	1.3	0.6
2P	1.9	4.83	1.3	2.2
3P	1.6	5.35	1.4	1.4
4C	0.7	8.04	1.1	7.3
5C	1.9	7.70	1.2	7.8
6C	0.8	5.65	0.9	11.0
7R	0.3	1.51	0.8	0.01

*Samples 1–5 were obtained from blended cultivars, sample 6 from Lastovka, P-by pressing, C-by centrifugal extraction, R-refined olive oil

oxidative changes in the fruit itself; oxidative changes, however, occur during the storage of the oil as well, and thus the conditions and storage time of the oil are very important.

Data given in Table 1 show that the peroxide values are below the limit permitted by the regulations, and they range from 3.52 to 5.35 mmol O_2/kg for pressed oils and from 5.65 to 8.04 for centrifuged oils. According to regulations *(3,4)*, the maximal peroxide value for all categories is 10 mmol O_2/kg (20 meq O_2/kg), and for refined olive oil is 5 mmol O_2/kg.

Autooxidative reactions develop in olive oil relatively slowly, but, because of the presence of chlorophylls the olive oil is sensitive to photo-oxidation resulting in the formation of hydroperoxide radicals, and in the case of extended exposure to light, in oil decolouration *(1,11)*.

Oils obtained by centrifugal extraction had a considerably higher chlorophyll content than the pressed oils. Obviously, the use of greater quantities of water during the olive processing and oil manufacture causes a stronger extraction of chlorophyll pigment. Studies on olive oil have recorded observations that chlorophylls *a* and *b* and pheophytins *a* and *b* speed up the oxidation of oil in light, while in dark they act as antioxidants or as synergists with polyphenol compounds present in olive oil *(1,12)*.

Oxidative changes were followed also by measurements of absorbance in the UV range (K_{232} and K_{270}). The values of recorded absorbances were used to calculate the ΔK and R-values.

Results are presented in Table 2.

Data in Table 2 show that the values for absorbances at 232 and 270 nm, and the values for ΔK are below or at the limit specified in the regulations of the European Community *(3)* for virgin olive oil – extra quality.

Measurements of absorption maxima in the UV range are often employed to follow up oxidative changes in oils. Primary products of linoleic acid oxidation and other conjugated hydroperoxides and conjugated dienes show the absorption maximum at a wave length of 232 nm. Secondary products of oxidation – conjugated trienes have the absorption maximum at 270 nm. Conjugated dienes and trienes are formed during refining or bleaching of oil. Measurements of these values can reveal the oxidation of oil caused by inadequate storage, the refining of oil or addition of a refined oil into nonrefined (e.g. undeclared blending of olive oil by addition of some other refined oils). According to European regulations *(3)*, maximal absorbance at 270 nm (K_{270}) for virgin olive oil is 0.25, and for virgin olive oil – extra is 0.20. Olive oil with the specific absorbance at 270 nm > 0.20 is considered as virgin oil provided that after passage through a column with alumina the absorbance is < 0.11, and the absorbance > 0.11, suggests the presence of refined oil *(1,3)*. Assessment of the oil quality based on R-value may be applied with crude oils only. According to Wolff, the lower the R-value, the lower the quality of the oil, because it contains more secondary oxidation products. Thus, R-value (K_{232}/K_{270}) for high-quality olive oil is 12, and for oxidated 6 *(7)*. The R-values given in Table 2 show a high quality of oils analyzed, with the values for pressed oils being somewhat lower, which suggests a slightly greater presence of secondary oxidation products.

Table 2. Spectrophotometric analysis of olive oil in UV range
Tablica 2. Spektrofotometrijska analiza maslinova ulja u UV-području

Samples*	Specific absorbances		K_{270} with alumina**	ΔK	R-value K_{232}/K_{270}
	K_{232}	K_{270}			
1P	1.85	0.20	0.05	0.008	9.4
2P	2.02	0.18	0.06	0.007	11.0
3P	2.30	0.20	0.07	0.008	11.5
4C	2.11	0.14	0.04	0.002	15.0
5C	1.92	0.16	0.04	0.003	11.7
6C	2.01	0.15	0.04	0.001	12.8
7R	1.87	0.90	–	–	2.1

* for sample markings – see Table 1.
** for the purpose of establishing purity, where K_{270} exceeds the limit for the category concerned, it shall be determined again after passage over alumina column.

Oleuropein, the characteristic phenol glucoside in the olive fruit is responsible for the bitter taste of unripe fruits. Depending on the variety and on the manner of processing, olive oil can have a higher or lower oleuropein content. A low intensity of bitterness is desirable and characteristic of olive oil, while a high intensity of bitterness decreases the organoleptic properties of the oil *(1)*. Table 3 shows the results of measurements of oil bitterness intensity.

The results given in Table 3 demonstrate that the oils obtained by centrifugal extraction had a greater intensity of bitterness (0.32–1.88) than the pressed oils (0.02–0.26), and it was the greatest in the oil obtained from the olive cultivar Lastovka (1.88) which is specific for this variety *(13)*.

It is difficult to say here whether the technological process alone was the cause of the higher content of bitter substances, because the olives were collected at different times and kept for a shorter or longer time in water; since oleuropein is soluble in water, its content in the oil may be influenced by all this, as well as by the possibility of incomplete removal of leaves from the fruits.

Quality assessment of olive oil usually involves organoleptic (sensoric) examinations, which, when statistically processed, give objective and reliable results. Sensory evaluation of virgin olive oil has been proposed by

Table 3. Intensity of bitterness in samples of olive oil examined
Tablica 3. Intenzitet gorka okusa u ispitivanim uzorcima maslinova ulja

Samples*	Absorbance at 225 nm		Intensity of bitterness
	Read	Calculated	
1P	0.328	0.082	0.26
2P	0.242	0.061	0.02
3P	0.276	0.069	0.08
4C	0.395	0.099	0.48
5C	0.348	0.087	0.32
6C	0.816	0.204	1.88

* for sample markings – see Table 1.

regulations of the European Community *(3)* and must be caried out under controlled conditions by a group of specially selected and trained assessors (»panel test«). As we could not provide either suitable conditions or trained tasters, the preliminary organoleptic assesment was carried out by a group of the consumers familiar with the use of olive oil. According to these results intensity of bitterness had a greater influence on the organoleptic mark and acceptance of such oil by our consumers, than the increase of acidity. The most unacceptable was the sample 6C, which had very good chemical properties, but a high intensity of bitterness (1.88), although it was less than 2.5, at which point, according to observations by Gutierrez-Rosales *et al.* *(8)*, problems for the oil acceptance can arise.

Conclusion

Timely collection and timely processing of olive oil are extremely important (although not the most important) for the quality of olive oil, because owing to the presence of enzymes and water in the fruit various chemical and biochemical reactions take place, leading to changes in the quality of oil, especially with respect to the increase in free fatty acids.

Keeping the fruits in water from the time of harvest to the time of processing, especially in sea water, has proved to be a fairly good method of olive storage, because in the samples kept in this way for several months (because of extremely untoward circumstances caused by war) no significant hydrolytical or oxidative changes occurred.

Olive oils obtained by the technological process of continuous centrifugal extraction have a higher content of chlorophyll pigments and bitter substances than the olive oils obtained by pressing. The oil obtained from the olives of Lastovka cultivar had a particularly high intensity of bitterness.

Studies on olive oil should be performed systematically in order to examine the influence of individual factors on the quality of oil more thoroughly.

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