

## Relationship between volatile compounds of olive oil and sensory attributes

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### Article history

Received: 8 September 2011

Received in revised form:

30 May 2012

Accepted: 6 June 2012

### Abstract

This study was carried out to study some quality indices (acid value, peroxide value and UV absorption  $K_{232\text{nm}}$ ,  $K_{270\text{nm}}$  and  $\Delta K$ ) of virgin olive oil of three varieties (Coratina, Koronakii and Picual) at two stages of ripening. Also, organoleptic tests, phenolic content,  $\alpha$ -tocopherol and oxidative stability measured by Rancimat method at 100°C were determined. Fatty acid composition and volatile compounds of virgin olive oil samples were analyzed by gas chromatography (GC) and gas chromatography mass spectrum (GC-MS) systems. Twenty-five compounds were isolated and characterized by GC-MS. The presences of some of these compounds in virgin olive oil were not previously reported. All results indicated that there were a wide variation in the chemical and aroma characteristics of the selected virgin olive oils.

### Keywords

Olive oil  
volatile compounds  
quality indices  
stability

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### **Introduction**

Virgin olive oil represents the main fats in the countries of the Mediterranean basin where production the olive oil is concentrated. Nowadays, there are some studies (Luna *et al.*, 2006) indicated the beneficial effects on the health of the so called "Mediterranean diet" found on the consumption of great amounts of vegetables, cereals, fish and olive oil. The cultivation of the olive trees is spreading in countries where the virgin olive oil consumption is very modest like Australia, Argentina and South Africa (Angerosa *et al.*, 1999). But another reason, owing to the increasing demand for olive oils of high quality, seems to be related to the increased popularity of this commodity, in addition to its health properties (Angerosa *et al.*, 2004), it is represented by its peculiar sensory characteristics that, because of use of virgin olive oil as a seasoning of cooked and especially raw foods have great repercussions on their acceptability (Baccouri *et al.*, 2007).

The distinctive aroma of virgin olive oil is attributed to a large number of chemical compounds of different chemical classes, i.e., aldehydes, alcohols, esters, hydrocarbons, ketones and probably, to other unidentified volatile compounds (Vichi *et al.*, 2003; Kalua *et al.*, 2007). The volatile composition of virgin olive oil depends on the levels and activities

of the enzymes involved in the various pathways (Angerosa, 2002) which are genetically determined (Campeol *et al.*, 2001). The pathway starts with the production of 9 and 13 hydroperoxides of linoleic and linolenic acids mediated by lipoxygenase. The subsequent cleavage of 13-hydroperoxides of catalyzed by very specific hydroperoxidelyases and leads to  $C_6$  aldehydes, whose unsaturated ones can isomerizes from cis-3 to the more stable trans-2 form. The mediation of alcohol dehydrogenase reduces  $C_6$  aldehydes to corresponding alcohols, which can produce esters because of the catalytic activity of alcohol acetyl transferase (Angerosa *et al.*, 2004). But an additional branch of the lipoxygenase pathway is active when the substrate is linolenic acid (Angerosa *et al.*, 2000). Lipoxygenase would catalyze besides the hydroperoxide formation, also its cleavage via an alkoxy radical giving rise to the formation of stabilized 1, 3-pentene radicals. The last compound can dimerize leading to  $C_{10}$  hydrocarbons (known as pentene dimmers) or couple with a hydroxy radical present in the medium producing  $C_5$  alcohols, which can be enzymatically oxidized to corresponding  $C_5$  carbonyl compounds (Angerosa *et al.*, 1999).

A recent investigation pointed out that olive seeds contain enzymatic activities metabolizing 13-hydroperoxides other than hydroperoxidelyase that are responsible for a decrease in the content of  $C_6$

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unsaturated aldehydes during the olive oil extraction (Angerosa *et al.*, 2002). The other accumulation products come from possible fermentations or conversion of some amino acids or from enzymatic activities of moulds or finally from oxidative processes but are generally related to the off-flavour of virgin olive oil (Angerosa *et al.*, 1998). Other factors that influence the volatile are ripening cycle of the fruits (Aparicio & Morales, 1998) and the processing equipment (Angerosa *et al.*, 2001). The effects of climate and soil type have also been studied (Cosio *et al.*, 2006). Many studies have been carried out in order to characterize the composition of the aroma of some olive oils (Haddada *et al.*, 2007). The aim of this work was to investigate the influence of ripening degree of olive fruits on aroma composition of olive oil by GC-MS and quality indices (acid value, peroxide value, UV absorption  $K_{232\text{nm}}$ ,  $K_{270\text{nm}}$  and  $\Delta K$ ). Also, determination of organoleptic testes, phenolic content,  $\alpha$ -tocopherol, oxidative stability measured by Rancimat method and fatty acid composition by gas chromatography.

## Material and methods

### Source of olive fruits

Three varieties of olive fruits, i.e., Coratina, Koronakii and Picual were obtained from a private farm at El-Khatba, Giza Governorate, Egypt. All varieties were collected by hand at the mid. of October and November during the crop season 2010. Only healthy fruits, without any kind of infection or physical damage were processed.

### Reagents, solvents and standards

All solvents in this study were purified and distilled before use. Folin-Ciocalteu reagent was obtained from Gerbsaure Chemical Co. Ltd., Germany.  $\alpha$ -tocopherol and gallic acid standards were obtained from Koch Light Laboratories Ltd. England.

### Oil extraction

After harvest, fresh olives (1.5-2.0 kg) were washed and deleafed, crushed with mill and pressed using hydraulic laboratory (Carver) press. Oil produced from each extraction was 200-250 ml/kg, filtered then transferred into dark glass bottles and stored in the dark at 4°C until analysis.

### Quality parameters

Acidity, peroxide value and UV absorption characteristics,  $K_{232\text{nm}}$  (conjugated dienes) and  $K_{270\text{nm}}$  (conjugated trienes) and  $\Delta K$  [ $\Delta K = k_{270} - (k_{266-4} + (k_{274+4})/2)$ ] were carried out following the analytical methods described by A. O. A. C.

(2005).

### Oil stability

Oxidative stability was evaluated by the Rancimat method (Gutierrez, (1989). Stability was expressed as the oxidation induction time (h), measured with the Rancimat 679 apparatus (Metrohm Co., Herisou, Switzerland), using 5.00 g oil heated to  $100^\circ\text{C} \pm 2^\circ\text{C}$  with an air flow of 20 l/hr<sup>1</sup>.

### Total phenolic content

Total phenol content was calorimetrically quantified (Ranalli *et al.*, 1999). Phenolic compounds were isolated by triple extraction of a solution of oil (10 g) in hexane (20 ml) with 30 ml of a methanol-water mixture (60:40, v/v). The Folin-Ciocalteu reagent was added to a suitable aliquot of the combined extracts, and the absorption of the solution at 725nm was measured. Values are given as milligrams of gallic acid per kilogram of oil (Gutfinger, 1981).

### $\alpha$ -tocopherol content

$\alpha$ -tocopherol was estimated by HPLC with direct injection of an oil-in-hexane solution:  $1.5 \pm 0.01$  g of oil dissolved in hexane to 10 ml (Sales *et al.*, 2000). The volume of injection was 20  $\mu\text{l}$ . The mobile phase consisted of hexane / ethyl acetate (70:30, v/v) at a flow rate of 1 ml/min  $\alpha$ -tocopherol was quantified by the external standard method. Results are given as milligrams of  $\alpha$ -tocopherol per kilogram oil.

### Fatty acid composition

The fatty acid methyl esters were prepared as described in the International Olive Oil Council (IOOC, 2009). Methyl esters were prepared from olive oil, after saponification and analyzed by gas chromatography (Pye-Unicam model 104) equipped with flame ionization detector and glass coiled column (1.6 m X 4 mm) supported on chromosorb (W-AW 100-200 mesh), was used. The samples ( $\mu\text{l}$ ) were injected into the column using a Hamilton microsyringe. The gas chromatographic conditions for isothermal analysis were: temperatures: column 170°C detector 300°C and injector 250°C, flow rates: hydrogen 33 ml/min., nitrogen 30 ml/min and air 330 ml/min. Peak areas were measured using a spectra physics chronjet integrator according to the method of Farag *et al.*, (1984).

### Organoleptic test

The organoleptic test was determined for the extracted oil according to the International Olive Oil Council (IOOC, 2009). The oil samples (15 ml) were presented in covered blue glasses (diameter, 70 mm, capacity, 130 ml) at  $28^\circ\text{C} \pm 2^\circ\text{C}$ . The glass warmed

and after removing the cover, the samples were smelled then tested by the panelist to judge its flavour. The different attributes of the oils were assessed and their intensities were evaluated as a mean value of the panelists score.

#### Analysis of volatile compounds

About (100 g) of olive oil was placed in a distillation flask with a little amount of distilled water. Steam was allowed to pass for four hours. The distillate was collected in an ice-cooled receiver, saturated with sodium chloride and extracted for several times with pure ether. The extract was kept at  $-10^{\circ}\text{C}$  until analysis by GC-MS.

A GC Varian 240-MS equipped with a 1078 split/splitless injector coupled with a mass spectrometer Varian Saturn 3 was used. A fused-silica capillary column VF-5MS, 30 m X 0.25 mm i.d.,  $1\mu\text{m}$  film thickness was employed. Helium was used as a mobile phase at a pressure of 15 psi with a flow rate of 2.2 ml/min and a linear velocity of 30.7 cm/s at  $35^{\circ}\text{C}$ . The GC oven heating was started at  $35^{\circ}\text{C}$ . This temperature was maintained for 8 min. then increased to  $45^{\circ}\text{C}$  at a rate of  $1.5^{\circ}\text{C}/\text{min}$ , increased to  $150^{\circ}\text{C}$  at a rate of  $3^{\circ}\text{C}/\text{min}$ . then increased to  $180^{\circ}\text{C}$  at a rate of  $4^{\circ}\text{C}/\text{min}$ , and finally increased to  $210^{\circ}\text{C}$  at a rate of  $3.6^{\circ}\text{C}/\text{min}$ . and maintained at this temperature for  $14.5^{\circ}\text{C}/\text{min}$ : the total time of analysis was 80 min. The injector temperature was maintained at  $250^{\circ}\text{C}$ . The temperature of the transfer line was fixed at  $220^{\circ}\text{C}$ . The mass spectrometer was operated in the electron ionization mode at an ionization voltage of 70 eV in the mass range of 10-350 amu at a scan rate of 1s/scan and a manifold temperature of  $180^{\circ}\text{C}$ . The GC-MS was operated through the software Saturn GC-MS version 5.2 (Varian). The volatile compounds were identified by comparison of their mass spectra and retention times with those of authentic reference compounds. When standards were not available, identification of the volatile compounds was obtained by comparing their mass spectral data with those of the NIST-92 library. Integration of all of the chromatographic peaks was performed choosing the three masses (Servili *et al.*, 2003).

#### Statistical analysis

The results are reported as the mean values. Data were compared on the basis of standard deviation of the mean values. In addition, Duncan's multiple range tests were used to determine significant differences among data. Statistical analysis was performed using the Statistical 5.00 Package (Stat Soft 97 edition).

## Results and Discussion

### Chemical composition of olive fruits

Table 1 show the chemical composition of Coratina, Koronakii and Picual olive fruits during two stages of ripening. All varieties contained more than 50% moisture and 12% ash. Crude proteins and carbohydrates were present in equal proportions. The crude fiber level was about twice that of proteins and carbohydrates. The oil content of olive fruits varieties during mid. October was lower than that mid. of November. The chemical compositions of both cultivars were in accordance with the chemical composition reported by Raina *et al.* (1986) and Basuny and Mostafa (2004).

### Quality indices

Some physico-chemical characteristics of olive oil samples of the three varieties (Coratina, Koronakii and Picual) during two stages of ripening (mid. October and November) were analysed. Figures 1, 2, 3, 4 and 5 show very low values for the classical physico-chemical parameters (acidity  $\leq 0.80$ ; peroxide value  $\leq 20.00$  meq.O<sub>2</sub>/kg; K<sub>232</sub>  $\leq 2.50$ ; K<sub>270</sub>  $\leq 0.22$  and  $\Delta\text{K} \leq 0.01$ ) and the values were falling within the "extra virgin" category, as stated by IOOC (2009).

### Fatty acid composition

The distribution of fatty acids, from all olive oil samples extracted from Coratina, Koronakii and

Table 1. Chemical composition (% on dry basis) of three investigated olive fruits harvest at two repining stages

Constituent	Coratina		Koronakii		Picual	
	Harvest months					
	Mid. October	Mid. November	Mid. October	Mid. November	Mid. October	Mid. November
Moisture	60.32±5.00	54.60±3.90	57.93±4.01	55.40±4.15	62.53±6.11	57.60±4.22
Crude oil	41.03±3.25	47.71±4.72	38.86±3.02	43.92±4.10	36.43±2.95	40.28±3.15
Crude proteins	10.90±1.10	9.32±1.00	11.33±2.15	10.62±1.55	11.31±1.90	10.61±1.33
Crude fibers	18.15±2.50	15.22±2.01	21.75±3.00	19.82±2.90	20.10±3.02	18.40±2.80
Ash	12.84±1.60	12.45±1.22	13.54±1.78	13.30±1.53	13.80±2.10	13.67±3.02
Total hydrolysable carbohydrate	17.08±2.00	15.30±1.90	19.52±2.33	12.31±2.15	18.36±2.80	17.04±2.50

Results are the means of three replicates ± SD

Table 2. Fatty acid composition of virgin olive oil extracted from Coratina, Koronakii and Picual varieties.

Fatty acid	Variety					
	Coratina		Koronakii		Picual	
	Mid. October	Mid. November	Mid. October	Mid. November	Mid. October	Mid. November
C16:0	15.01±1.50	16.40±1.80	14.73±1.22	16.78±1.91	18.57±2.00	19.67±2.13
C16:1	0.82±0.01	1.57±0.15	1.38±0.10	1.85±0.20	1.82±0.20	2.49±0.25
C17:0	0.03±0.001	0.08±0.001	0.05±0.001	0.08±0.001	0.09±0.001	0.00±0.00
C17:1	0.06±0.01	0.11±0.01	0.08±0.01	0.12±0.01	0.12±0.01	0.13±0.01
C18:0	1.84±0.20	2.42±0.35	2.56±0.40	2.21±0.25	3.61±0.50	2.85±0.56
C18:1	66.65±5.50	64.40±5.20	67.66±5.77	64.12±5.13	62.05±4.88	59.17±3.90
C18:2	12.89±2.10	12.56±2.00	11.68±1.95	12.89±2.11	12.31±1.89	13.95±2.33
C18:3	0.87±0.10	0.95±0.10	0.70±0.10	0.94±0.10	0.81±0.10	0.91±0.10
C20:0	0.44±0.01	0.42±0.01	0.42±0.01	0.41±0.01	0.45±0.01	0.33±0.01
C20:1	0.48±0.01	0.37±0.01	0.27±0.01	0.00±0.00	0.23±0.01	0.17±0.01
C22:0	0.11±0.001	0.12±0.001	0.10±0.001	0.11±0.001	0.00±0.00	0.00±0.00
C24:0	0.59±0.01	0.60±0.01	0.37±0.01	0.49±0.01	0.00±0.00	0.00±0.00
Monounsaturated	68.01±6.50	66.45±5.90	69.39±6.71	66.09±5.81	64.20±5.50	61.96±4.91
Poly unsaturated	13.76±2.50	13.51±2.30	12.38±2.00	13.83±2.66	13.12±2.15	14.86±3.00
Total saturated	18.23±3.10	20.04±3.22	18.23±3.00	20.08±3.30	22.72±3.50	23.18±3.60
Total unsaturated	81.77±8.00	79.96±7.90	81.77±8.00	79.92±7.80	77.32±7.22	76.82±6.90

Results are the means of three replicates ± SD

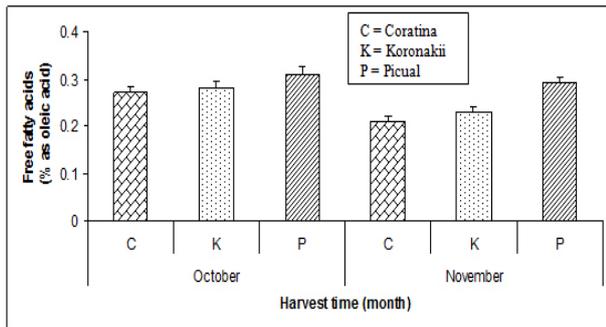


Figure 1. Effect of ripening stages on the free fatty acids (% as oleic acid) of virgin olive oil extracted from Coratina, Koronakii and Pical varieties. Results are the means of three replicates  $\pm$  SD

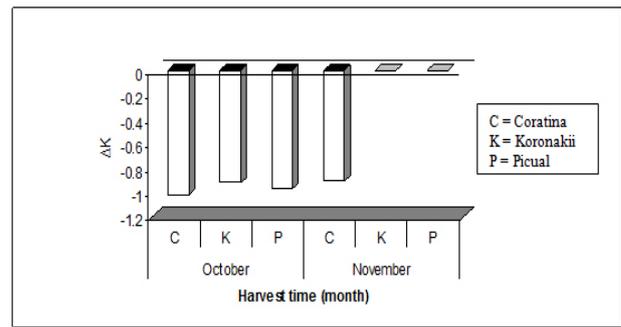


Figure 5. Effect of ripening stages on the  $\Delta K$  refers of virgin olive oil extracted from Coratina, Koronakii and Pical varieties. Results are the means of three replicates  $\pm$  SD.

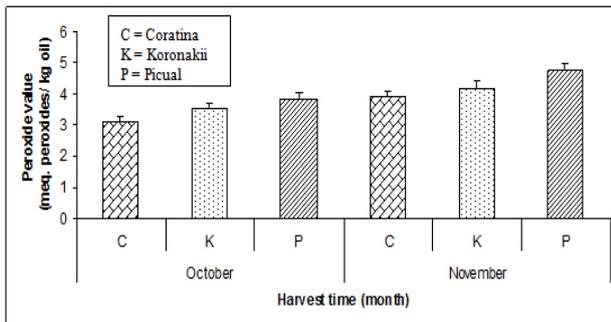


Figure 2. Effect of ripening stages on the peroxide value of virgin olive oil extracted from Coratina, Koronakii and Pical varieties. Results are the means of three replicates  $\pm$  SD

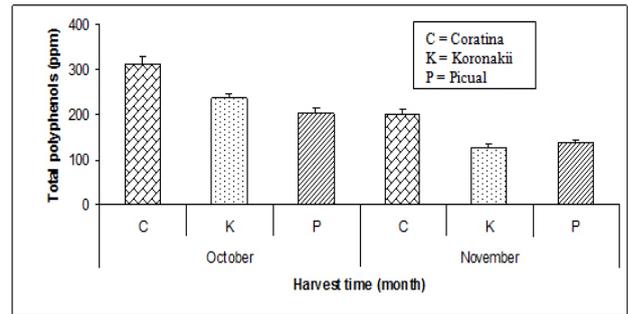


Figure 6. Effect of ripening stages on the total polyphenol content (ppm) of virgin olive oil extracted from Coratina, Koronakii and Pical varieties. Results are the means of three replicates  $\pm$  SD.

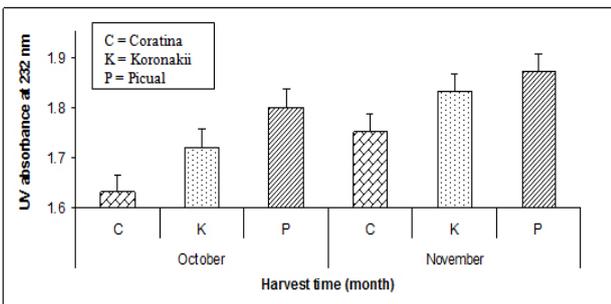


Figure 3. Effect of ripening stages on the conjugated diene formation of virgin olive oil extracted from Coratina, Koronakii and Pical varieties. Results are the means of three replicates  $\pm$  SD

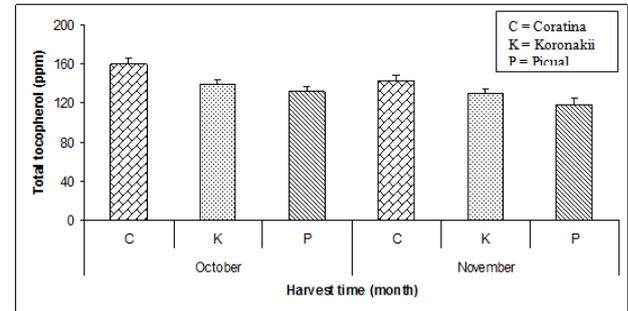


Figure 7. Effect of ripening stages on the total tocopherol content (ppm) of virgin olive oil extracted from Coratina, Koronakii and Pical. Results are the means of three replicates  $\pm$  SD

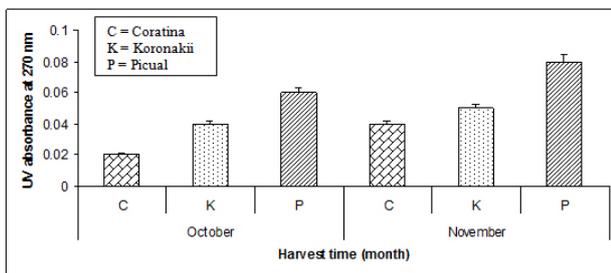


Figure 4. Effect of ripening stages on the conjugated triene formation of virgin olive oil extracted from Coratina, Koronakii and Pical varieties. Results are the means of three replicates  $\pm$  SD

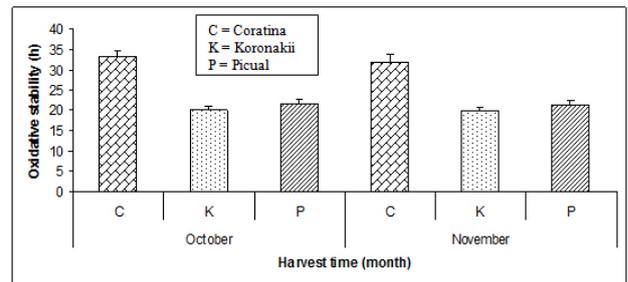


Figure 8. Effect of ripening stages on the oxidative stability (h) of virgin olive oil extracted from Coratina, Koronakii and Pical varieties. Results are the means of three replicates  $\pm$  SD

Pical fruits during two ripening stages covered the normal composition range expected for olive oil (Table 2). Oleic acid is the main monounsaturated fatty acid, representing high concentrations (59.17 – 67.66%) according to varieties. Palmitic acid, the major saturated fatty acid, ranged between 14.83%

and 19.67%, whereas linoleic acid was dominant polyunsaturated fatty acid ranging from 11.68 5 and 13.95% (Table 2). Variations in fatty acid contents observed in olive oil samples obtained from all varieties are probably related to cultivar-environmental interaction during the development

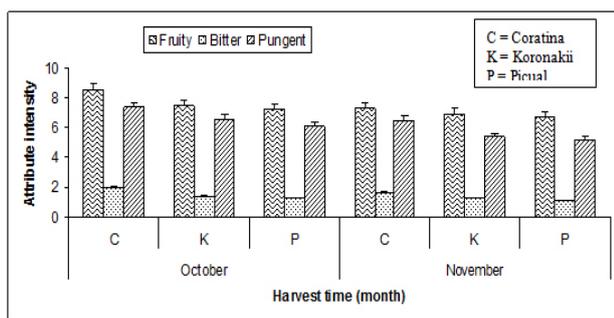


Figure 9. Effect of ripening stages on the Sensory characteristics of virgin olive oil extracted from Coratina, Koronakii and Picual varieties. Results are the means of three replicates  $\pm$  SD

and the maturity of the fruits (Lavee and Wodner, 1995). The results of the present study are in agreement with the finding of other authors (Bruni *et al.*, 1994; Schiratti, 1999). It was observed that olive oil products during mid. October was rich in total USFA, essentially due to its higher content in oleic acid, while, mid. November was rich in total SFA due to its high content in palmitic acid. These values are similar to those reported by some authors for other olive oil varieties (Ben Temime *et al.*, 2006; Krichene *et al.*, 2007).

#### Total phenolic content

Olive oil is the only vegetable oil which contains remarkable amounts of phenolic compounds (which were represented basically by ortho-diphenols) acting as antioxidant substances and conferring to it a greater stability against oxidation during storage (Bendini *et al.*, 2007). The content of total phenols of olive

oils extracted from all varieties during mid. October showed the highest values in phenols with, 311.40, 235.00 and 202.30. mg /kg, respectively, whereas, mid. November recorded lower ones with 199.00, 127.30 and 135.60 mg/kg, respectively (Figure 6).

#### $\alpha$ -tocopherol content

Figure 7 shows the main components that are related to oil stability. The tocopherol content of virgin olive oil is important factor to protect lipids against autoxidation and, thereby, to increase its storage life. The range of  $\alpha$ -tocopherol contents in virgin olive oil of the olive varieties (Coratina, Koronakii and Picual) was between 118.90 to 160.60 mg/kg. There were differences in the- tocopherol content between the harvest periods. Oils from the mid. November contained lower  $\alpha$ -tocopherol than oils extracted during mid. October of the all varieties (Figure 7).

#### Oxidative stability

Stability to oxidation is an important property of olive oil which is improved by synergistic interactions between the various antioxidants present in the oil itself, and also depends on the lipid composition. Oxidative stability of the olive oils showed the same trend in relation to harvest period as the total phenol and  $\alpha$ -tocopherol contents of the oil differences were between the stability of virgin olive oils from the different harvesting time. The higher stability of olive oil in the first harvest period (mid. October) than the second harvest period (mid. November) this fact could be explained by a degradation of phenolic

Table 3. Volatile components (%) of virgin olive oil extracted from Coratina, Koronakii and Picual varieties

No.	Volatile component	R.T (min)	Variety					
			Coratina		Koronakii		Picual	
			Mid. October	Mid. November	Mid. October	Mid. November	Mid. October	Mid. November
1	3-Methyl-butanol	3.056	3.20 $\pm$ 1.00	7.50 $\pm$ 2.50	0.40 $\pm$ 0.01	0.50 $\pm$ 0.01	0.50 $\pm$ 0.01	1.00 $\pm$ 0.10
2	Pentan-3-one	3.644	5.30 $\pm$ 1.13	3.70 $\pm$ 1.00	5.70 $\pm$ 1.50	4.90 $\pm$ 1.30	1.50 $\pm$ 0.10	4.00 $\pm$ 1.15
3	2-Methyl butanal	4.233	3.00 $\pm$ 1.00	2.56 $\pm$ 0.90	0.37 $\pm$ 0.01	0.40 $\pm$ 0.01	0.53 $\pm$ 0.01	0.33 $\pm$ 0.01
4	1-Penten-3-one	4.345	14.50 $\pm$ 3.00	8.30 $\pm$ 2.20	2.50 $\pm$ 0.95	1.20 $\pm$ 0.10	5.60 $\pm$ 1.52	5.00 $\pm$ 1.50
5	Unknown	4.541	2.20 $\pm$ 0.85	3.20 $\pm$ 1.00	2.50 $\pm$ 0.90	3.00 $\pm$ 1.01	4.50 $\pm$ 1.15	2.70 $\pm$ 0.85
6	Unknown	4.849	2.00 $\pm$ 0.70	2.15 $\pm$ 0.72	8.50 $\pm$ 2.35	8.00 $\pm$ 2.00	3.10 $\pm$ 1.00	2.66 $\pm$ 0.95
7	Butyl acetate	7.035	5.50 $\pm$ 1.30	5.10 $\pm$ 1.15	7.25 $\pm$ 2.40	7.00 $\pm$ 2.00	5.90 $\pm$ 1.53	4.70 $\pm$ 1.00
8	Hexanal	10.790	1.20 $\pm$ 0.10	2.30 $\pm$ 0.70	0.95 $\pm$ 0.10	1.00 $\pm$ 0.10	3.10 $\pm$ 1.00	5.90 $\pm$ 1.55
9	E-2-Methyl-2-butenal	13.199	7.10 $\pm$ 2.10	6.10 $\pm$ 1.90	9.10 $\pm$ 2.50	4.50 $\pm$ 1.33	3.00 $\pm$ 1.00	6.00 $\pm$ 1.57
10	Ethylbenzene	14.404	14.50 $\pm$ 3.50	12.70 $\pm$ 2.90	12.50 $\pm$ 2.81	7.90 $\pm$ 2.00	4.90 $\pm$ 1.77	4.00 $\pm$ 1.183
11	2-Methyl-butyl acetate	22.698	1.40 $\pm$ 0.20	2.30 $\pm$ 0.80	1.80 $\pm$ 0.51	2.00 $\pm$ 0.50	5.80 $\pm$ 1.50	3.70 $\pm$ 1.00
12	Z-3-Hexenal	23.007	1.05 $\pm$ 0.10	1.41 $\pm$ 0.15	2.30 $\pm$ 0.66	3.00 $\pm$ 0.79	2.50 $\pm$ 0.95	3.00 $\pm$ 1.13
13	Z-2-Hexenal	31.049	0.43 $\pm$ 0.10	0.55 $\pm$ 0.10	0.75 $\pm$ 0.10	0.90 $\pm$ 0.15	2.50 $\pm$ 0.88	1.80 $\pm$ 0.55
14	E-2-Hexenal	37.830	8.40 $\pm$ 2.50	9.60 $\pm$ 2.80	9.50 $\pm$ 2.77	8.40 $\pm$ 2.40	14.50 $\pm$ 3.22	10.30 $\pm$ 2.90
15	Trans-2-Hexen-1-ol	44.527	6.10 $\pm$ 1.80	5.50 $\pm$ 1.50	4.50 $\pm$ 1.30	3.80 $\pm$ 1.15	3.00 $\pm$ 1.01	1.90 $\pm$ 0.80
16	Pentan-1-ol	49.515	3.40 $\pm$ 1.15	0.95 $\pm$ 0.15	1.10 $\pm$ 0.20	2.90 $\pm$ 0.71	2.50 $\pm$ 0.66	8.00 $\pm$ 2.30
17	Unknown	54.390	2.95 $\pm$ 0.80	1.50 $\pm$ 0.30	2.01 $\pm$ 0.71	2.50 $\pm$ 0.83	3.10 $\pm$ 1.00	1.00 $\pm$ 0.15
18	3-Methyl-2-butenylacetate	54.698	1.15 $\pm$ 0.20	5.60 $\pm$ 1.30	1.40 $\pm$ 0.35	3.90 $\pm$ 1.11	3.80 $\pm$ 1.00	4.10 $\pm$ 1.37
19	Hexyl acetate	57.809	1.10 $\pm$ 0.12	2.50 $\pm$ 0.83	1.90 $\pm$ 0.66	2.10 $\pm$ 0.70	2.30 $\pm$ 0.75	5.30 $\pm$ 1.60
20	E-2-Octenal	64.870	1.35 $\pm$ 0.20	1.60 $\pm$ 0.25	9.20 $\pm$ 2.50	8.90 $\pm$ 2.30	1.90 $\pm$ 0.50	1.80 $\pm$ 0.44
21	6-Methyl-5-hepten-2-one	67.252	1.70 $\pm$ 0.20	1.50 $\pm$ 0.15	1.00 $\pm$ 0.10	0.85 $\pm$ 0.01	7.80 $\pm$ 2.50	3.80 $\pm$ 1.31
22	Hexan-1-ol	68.204	2.15 $\pm$ 0.70	2.20 $\pm$ 0.63	4.30 $\pm$ 1.15	4.25 $\pm$ 1.11	2.30 $\pm$ 0.88	6.90 $\pm$ 2.15
23	Nonan-2-one	70.642	4.05 $\pm$ 1.00	1.60 $\pm$ 0.30	4.30 $\pm$ 1.20	2.60 $\pm$ 0.70	4.00 $\pm$ 1.01	3.00 $\pm$ 0.95
24	Unknown	71.345	2.46 $\pm$ 0.70	2.66 $\pm$ 0.85	3.45 $\pm$ 1.00	3.44 $\pm$ 1.00	2.33 $\pm$ 0.90	2.38 $\pm$ 0.95
25	Z-2-Hexen-1-ol	73.751	4.50 $\pm$ 1.18	8.60 $\pm$ 2.32	5.20 $\pm$ 1.50	9.00 $\pm$ 2.70	7.50 $\pm$ 2.11	4.50 $\pm$ 1.15

Results are the means of three replicates  $\pm$  SD

Table 4. Relationship between volatile components and sensory characteristics

NO.	Volatile components	Sensory characteristics
1	3-Methyl-butanol	Fruity, sweet
2	Pentan-3-one	Fruity
3	2-Methyl butanal	Sweet
4	1-Penten-3-one	Pungent
5	Unknown	--
6	Unknown	--
7	Butyl acetate	Green, fruity, pungent
8	Hexanal	Green, grass
9	E-2-Methyl-2-butenal	Green fruit, aromatic
10	Ethylbenzene	Strong
11	2-Methyl-butyl acetate	Fruity, green,
12	Z-3-Hexenal	Green leaves, cut grass
13	Z-2-Hexenal	Fruity
14	E-2-Hexenal	Fruity, aromatic, cutgrass
15	Trans-2-Hexen-1-ol	Bitter almonds green-fruity
16	Pentan-1-ol	Pungent, strong
17	Unknown	--
18	3-Methyl-2-butenylacetate	Pungent
19	Hexyl acetate	Sweet, green, fruity
20	E-2-Octenal	Green, grassy
21	6-Methyl-5-hepten-2-one	Green, grassy, fruity, pungent
22	Hexan-1-ol	Fruity, aromatic
23	Nonan-2-one	Pungent, fruity
24	Unknown	--
25	Z-2-Hexen-1-ol	Almond, grass

compounds, mainly implicated in oil stability (Gutierrez *et al.*, 2001).

#### Organoleptic test

Organoleptic test of olive oil samples extracted from Coratina, Koronakii and Picual varieties during two ripening stages (mid. October and mid. November) were evaluated by 10 panelists (Figure 9). From a sensory point of view all the samples examined are belong to the extra virgin olive oil grade. The direct observation of the intensities of attributes detected by tasters showed that the oils studied were mainly characterized by high intensities of bitter, pungent, fruity.

#### Volatile compounds

Flavour is an important quality criterion for virgin olive oils. The identification of the compounds causing the flavour or off-flavour is therefore the key for quality control. Virgin olive oil has delicate and unique flavour (Angrosa *et al.*, 2000). All the identified volatiles are listed in Table 3. Twenty-five compounds were have been characterized by GC-MS analysis. The major constituents of the volatile fraction of Coratina oil in the first harvest (mid. October) were 1-penten-3-one (14.50%), ethylbenzene (13.50%), E-2-hexenal (8.40%), E-2-methyl-2-butenal (7.10%), trans-2-hexen-1-ol (6.10%), butylacetate (5.50%) and pentan-3-one (5.30%) (Table 3). The volatile fraction of Koronakii oil (mid. October) were ethylbenzene (12.50%), E-2-hexenal (9.50%), E-2-octenal (9.20%), E-2-methyl-2-butenal (9.10%), butylacetate (7.25%), pentan-3-one (5.70%) and Z-2-hexen-1-ol (5.20%)

(Table 3). The main constituents of that characterized volatile fraction of Picual oil (mid. October) were E-2-hexenal (14.50%), 6-methyl-5-hepten-3-one (7.80%), Z-2-hexen-1-ol (7.50%), butyl acetate (5.90%), 2-methyl-1-butylacetate (5.80%), 1-penten-3-one (5.60%) and ethylbenzene (4.90%) (Table 3).

These results indicate that there were quantitative differences between the volatile profiles of the varieties analyzed (Coratina, Koronakii and Picual). The present results are in agreement with the other studies such as Youssef *et al.*, (2011), Luna *et al.*, (2006) and Baccouri *et al.*, (2007). In general, results show that there were quantitative significant ( $P \geq 0.05$ ) difference for the volatile during the olive ripeness (mid. October and November) as well as between all varieties (Angrosa *et al.*, 2004).

#### The role of volatile compounds in the sensory quality of virgin olive oils

Table 4 shows the sensory characterization of the volatile compounds with the exception of a few that were mostly hydrocarbons. Although a consensus was held on the sensory descriptors, the assessors were suggested to limit the description of the sensory perceptions to the attributes defined by the International Olive Oil Council (IOOC, 1987).

Results presented in the Table (3), indicate that the compounds 1-penten-3-one and ethylbenzene) are characteristic of olive oil (Coratina variety). On the other side, it was found that the compounds e-2-methyl-2-butanol and ethylbenzene are characterized by the presence of olive oil (Koronakii variety). Also, olive oil (Picual variety) is distinguished by the presence of compound E-2-hexenal and the rate of the different ripening stages. From these findings we can deduce that each variety of olive oils, under study is characterized by the presence of volatile compounds differed from the other varieties.

The results presented in Table, 3 and 4 demonstrate that the Coratina variety gave the highest values in the organoleptic properties (Fruity, bitter and pungent). On the other hand, Coratina variety is characterized by the presence of compounds 1-penten-3-one and ethylbenzene in the two ripening stages. These compounds are directly related to an increase of concentration of the desired intensity of sensory attributes (fruity, bitter and pungent) Figure (9). Whereas, the rest olive varieties contained different volatile compounds at different concentration (Table 3). From the above findings one come conducted that there is a direct relationship between volatile compounds and sensory properties of varieties of olive oil.

## Conclusion

In conclusion, the results confirmed that there were great variations in all of the measured characters among the olive varieties. All these results indicated that olive oil aroma compounds accumulated differently according to the cultivar. In fact, accumulation of these metabolites had close dependence on the enzymatic store, which is genetically determined, according to results of other researches (Angrosa *et al.*, 1999).

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