

Chemical and Sensory Analysis of Some Egyptian Virgin Olive Oils

Cinzia Benincasa¹, Anna Russo¹, Elvira Romano¹, Muhammad E. Elsorady², Enzo Perri¹ and Innocenzo Muzzalupo^{1*}

¹CRA - Olive growing and oil industry research centre, Rende (CS), Italy

²Department of Oils and Fats, Food Technology Research Institute, Agriculture Research Center, Giza, Egypt

Abstract

In the paper here presented, virgin olive oils produced in the year crop 2010/2011 in two different areas of Egypt, Siwa oasis and Giza, were characterised by their chemical-physical parameters. Also, the analysis of volatile compounds by SPME/GC/MS was carried out and the results compared with those provided by the panel test for the same samples. SPME/GC/MS analysis revealed that most of the volatile compounds determined in the virgin olive oil samples under investigation, contribute to characterise the sensory notes related to the rancid perceptions, oily and fatty persistency justifying and hence the negative attributes noticed by the sensory evaluation. Sensory and SPME/GC/MS analysis of olive oils samples here investigated reveal that most of the volatile compounds are characterised by sensory notes related to the rancid perceptions.

Keywords: Oil quality; Panel test; SPME/GC/MS; Volatile compounds

Introduction

The olive tree (*Olea europaea* L.) is one of the most important crops in the Mediterranean countries. The origins of the cultivation of the olive tree lie rooted in legend and tradition. It probably started about 5000-6000 years ago within a wide strip of land by the eastern Mediterranean sea and in the adjacent zones comprising Asia minor, part of India, Africa, and Europe [1,2]. Others believe that the olive tree originated from Africa (Ethiopia, Egypt). This is where olive trees were first cultivated systematically, and from where they spread to Cyprus, Morocco, Algeria, Tunisia and to western places by the Phoenicians [3].

Virgin olive oil (VOO), an excellent natural food, is obtained from olive fruit (*Olea europaea* L.) by mechanical or physical procedures. Its composition varies widely, depending on fruit variety, degree of fruit ripeness, environmental conditions, growing region, and techniques of processing and storage [4]. VOO has shown a high resistance to oxidative deterioration due to fatty acid composition and phenolic antioxidants.

As they are the least processed forms of olive oil, extra virgin or VOOs have more monounsaturated fatty acids than other olive oils. No other naturally produced oil has as a large amount of monounsaturated as olive oil, mainly oleic acid, and these higher proportions of monounsaturated fats in the diet are linked with a reduction in the risk of coronary heart disease. VOO also contain more phenols such as oleuropein derivatives and hydroxytyrosol, and antioxidants such as vitamin E and carotenoids, that may help prevent the oxidation of LDL particles, providing health benefits for the heart such as favorable effects on cholesterol regulation, antiinflammatory, antithrombotic, antihypertensive as well as vasodilatory effects both in animals and in humans [1, 5-7]. Another health benefit of olive oil seems to be its property to displace omega-6 fatty acids, while not having any impact on omega-3 fatty acids. This way, olive oil helps to build a more healthy balance between omega-6 and omega-3 fats [8].

The chemical composition of VOO consists of a major portion that includes triacylglycerols and that represents more than 98% of the total oil weight and a minor one, that is present in very low amounts (about 2% of oil weight), including more than 230 chemical compounds such

as aliphatic and triterpenic alcohols, sterols, hydrocarbons, volatile compounds and antioxidants [9].

The nutritional value of VOO arises from high levels of oleic acid and phenolic compounds [8,10], whereas the aroma is strongly influenced by volatile compounds [11]. Nutritional value and pleasant flavor have contributed to an increase in consumption of olive oil which has fostered cultivation of olives outside the traditional olive oil producing region of the Mediterranean and into newer areas where cultivars adaptability, different climatic conditions and different agronomic practices may alter olive quality [12]. Olive oil quality may be defined from commercial, nutritional or sensorial perspectives [13].

The International Olive Council [14] and the European Commission [15] have defined the quality of olive oil based on parameters that include free acidity, peroxide value (PV), UV specific extinction coefficients (K_{232} and K_{270}) and sensory score. In particular, the quantity of free acidity is an important factor for classifying olive oil into commercial grades [16,17]. The general classification of olive oils into the different commercial grades is based on free acidity and sensory characteristics (taste and aroma). The commercial grades separate oil obtained from the olive fruit solely by mechanical or physical means (virgin) from the other oils that contain refined oils [18].

The organoleptic quality of olive oils depends on several factors, one of which is the cultivar. The organoleptic quality of the oils was assessed in the light of the following parameters: aromas, total phenols and phenol composition [19].

In most cases quality parameters change by the time the oil reaches the consumer [20]. Olive oil is susceptible to both hydrolytic and

***Corresponding author:** Innocenzo Muzzalupo, PhD, CRA - Olive growing and oil industry research centre, Rende (CS), Italy, Tel: +39 0984 4052208; Fax: +39 0984 402099; E-mail: innocenzo.muzzalupo@entecra.it

Received November 08, 2011; **Accepted** December 15, 2011; **Published** December 17, 2011

Citation: Benincasa C, Russo A, Romano E, Elsorady ME, Perri E, et al. (2011) Chemical and Sensory Analysis of Some Egyptian Virgin Olive Oils. J Nutr Food Sci 1:118. doi:10.4172/2155-9600.1000118

Copyright: © 2011 Benincasa C, et al. This is an open-access article distributed under the terms of the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original author and source are credited.

oxidative reaction [13] that can adversely affect oil quality parameters. For instance, an increase in PV, K_{232} and K_{270} values and development or loss of certain volatile compounds is very common between extraction and consumption [16,20]. The presence or absence of particular volatile compounds may also be a good indicator of olive oil quality changes.

Several factors are known to affect the quantitative profile of olive fruits. Among these factors, the degree of ripeness, the geographic origin and the nature of the cultivar are certainly those that have a pronounced influence on the composition. Some studies were already published concerning the influence of these factors on some French [21], Spanish [22], Italian [6,23], Portuguese [24] and Tunisian [25] cultivars.

In this study, some Egyptian VOOs has been analysed in order to evaluate their levels of quality. For this purpose, the chemical composition, the volatile compounds and their sensorial analysis were determined.

Materials and Methods

Samples

The samples subjected to the study were produced in Egypt and analysed at CRA - olive growing and oil industry research centre laboratory (Italy). VOOs were obtained from the agricultural research center, Giza, Egypt. The VOOs were Maraqui and Wattagen cultivar, cultivated in Siwa oasis and Coratina, Koroneiki and Arbequina cultivar which were cultivated in Giza.

These olive oil samples were investigated because they come from two cities which have a very different climate and geographical position (Figure 1). In fact, Giza, the third largest city in Egypt, is located on the west bank of the Nile River. Giza experiences an arid climate, but often with high humidity due to the River Nile's Valley effects.

The Siwa Oasis is an oasis located between the Qattara Depression and the Egyptian Sand Sea in the Libyan Desert, nearly 50 km east of the Libyan border. Siwa is very rich in water and produces large quantities of high quality dates and it is popular for its palm and olive trees (Figure 1).



Figure 1: Egypt map highlighting the geographical position of Siwa and Giza town.

Olive oil extraction was carried out by means of a two phase continuous extraction system (Toscana Enologica Mori, Italy). Olives were crushed by using a hammer mill, operating at 3000 rpm, malaxation of pastes was done in a mixer at 14 rpm and 30 °C for 1 h. Separation of the paste into oily must and pomace was performed by a two phase centrifugal decanter working at 3500 rpm. Finally, a horizontal centrifuge at 40 °C operating at 6500 rpm and fed with 1 litre tap water per kg of oily must was used to remove the remaining solids from the must. All oil samples were filtered through anhydrous Na_2SO_4 and stored at -18 °C in dark glass bottles prior to analysis.

Percentage of free acidity, PV and ultra-violet absorption

Free acidity, PV and UV light absorption (K_{232} , K_{270} , ΔK) were determined following the official analytical methods described in EC Regulation 2568/91 [15]. The mean of the data was calculated from three biological repeats obtained from three independent experiments.

Fatty acids composition

The fatty acid composition was determined as methyl esters following the procedures described in the enclosures of the Commission Regulation EEC no. 2568/91. In general, 0.15 g of oil was dissolved in 1 ml of hexane and 0.1 ml of a methanolic solution of KOH (1 N). The resulting solution was shaken vigorously for 5 min. Subsequently, 0.25 ml of the supernatant was taken, deposited in a vial and dissolved in 1.5 ml of hexane. The mean of the data was calculated from three biological repeats obtained from three independent experiments.

GC-FID analysis

1 μl hexane solution was injected into a gas chromatograph (Agilent 6890N) equipped with a capillary column SP-2340 (60 m \times 0,25 mm i.d., 0,2 μm f.t., Supelco). The separation was carried out with a programmed temperature (110 °C held for 5 min, increase of 3 °C min^{-1} to 150 °C and held for 16.33 min, increase of 4 °C min^{-1} to 230 °C and held for 27 min) and an FID detector at 260 °C. The results are expressed in percentage of chromatographic areas [8]. The mean of the data was calculated from three biological repeats obtained from three independent experiments.

Tocopherols analysis

Olive oil (6 g) was dissolved with hexane and made up to volume (10 ml). This solution was filtered (PTFE filter 0.2 μm , 25 mm, Whatman) and 20 μl were injected into an HPLC system (Agilent 1100) equipped with a Zorbax NH_2 column (25 cm \times 4.6 mm i.d., 5 μm particle size, Agilent) using an isocratic mobile phase of hexane:ethyl acetate (80:20). The flow rate was 2 ml min^{-1} and the detector was a fluorescence spectrophotometer with a programmed wavelength (295 nm and 325 nm). The results are expressed in mg of α , β , γ and δ tocopherol per kg of oil [6]. The mean of the data was calculated from three biological repeats obtained from three independent experiments.

Volatile compounds

Olive oil (2 ml) was dissolved in a 10 ml vial where a fixed quantity of internal standard (2-methyl-4-pentanol) was added. The olive oil samples were directly analysed by SPME/GC/MS using a Varian 4000 GC/MS mass spectrometer [26]. Particularly, a DVB/CARB/PDMS 70 μm solid phase micro extraction fibre and a GC capillary column VF-5ms 60 m \times 0,25 mm i.d., 0,25 μm f.t. were used.

Instrumental parameters were as follows: split ratio 50:1; helium gas flow 1.2 ml min^{-1} . Injection volume 1 μl ; column oven: T = 50 °C

hold for ten minutes, then ramp to 180 °C at 25 °C min⁻¹; then ramp to 220 °C at 10 °C min⁻¹.

Injection at 250°C; transfer line at 270 °C; ion source at 200 °C. Pre-incubation time 20 min at 40 °C; adsorption time 5 min; adsorption time 3 min [26]. The mean of the data was calculated from three biological repeats obtained from three independent experiments.

Sensory analysis

A panel of eight trained assessors carried out an evaluation of sensory characteristics according to the International Olive Oil Council (IOOC) method for sensory analysis of olive oil [27]. The oil samples (15 ml each) were presented in covered blue glasses at 28 ± 2 °C. The cover was removed and the sample was smelled and tasted by each panelist. The sensory attributes were evaluated using a special profile sheet. The mean of the data was calculated from three biological repeats obtained from three independent experiments.

Results and Discussion

Percent of free acidity

VOO contains about 98 g per 100 g of neutral lipids, mainly triglycerides (96-97 g per 100 g) followed by a small quantity of diglycerides. The quality indices of the samples analysed are listed in Table 1. The data revealed that the free acidity values of all the oils were below 1.0, except for the sample of Maraqui cultivar which was 1.20 ± 0.10 g of oleic acid per 100 g of oil and fell within the accepted values for extra-VOOs and VOOs as the standard free acidity limit for extra-VOO and VOO are 0.8 and 2.0 g per 100 g maximum, respectively [14,15].

Peroxide value

The PV is a measure of primary oxidation. The data (Table 1) revealed that in all cultivars the PVs were not higher than 2.76 ± 0.15 meq O₂ kg⁻¹. None of the oil samples analysed exceeded the maximum peroxide value for extra-VOO (20 meq O₂ kg⁻¹) [14]. These results concur with those obtained for Coratina cultivar by Clodoveo et al. [27].

Specific extinction coefficient at 232 nm, 270 nm and ΔK

K₂₃₂ parameter is mainly indicative of the conjugated dienes. Data in Table 1 showed that the minimum and maximum values for the absorbance at 232 nm were recorded respectively for Maraqui (1.43 ± 0.06) and Arbequina (2.26 ± 0.09) oil. The absorbance at 270 nm, mainly indicative of the conjugation of trienes and of the presence of carboxylic compounds, gives the minimum value for Maraqui oil (0.07 ± 0.02) and the maximum value for Koroneiki oil (0.18 ± 0.01). The values recorded at 232 and 270 nm for all samples analysed complied with IOOC limits for extra VOO. Also, all the values for ΔK lie inside the limits specified for extra VOO in the standard [14].

Fatty acid composition

Fatty acid composition is an essential aspect of the qualitative assessment of olive oil. Unsaturated Fatty Acids (UFA) are of great importance because of their nutritional implications and effect on the oxidative stability of oils [28].

The fatty acid composition of the oils analysed are shown in Table 2. The data revealed that the identified fatty acids in all the samples were typical of olive oil and consisted of myristic acid (C_{14:0}), palmitic acid (C_{16:0}), palmitoleic acid (C_{16:1}), stearic acid (C_{18:0}), oleic acid (C_{18:1}), linoleic acid (C_{18:2}), linolenic acid (C_{18:3}), arachidic acid (C_{20:0}), eicosenic acid (C_{20:1}), behenic acid (C_{22:0}) and lignoceric acid (C_{24:0}). These fatty acids play an important part in the sensory characterization of olive oil [29]. Table 2 shows that in all the samples, the oleic acid was always the most abundant fatty acid (monounsaturated), representing 67 g per 100 g of the total fatty acid composition at least, except for Arbequina sample where the percentage value was only 44.00 ± 1.65 g per 100 g, although other authors have found different values that differ significantly from what we have found [30]. Palmitic acid was also the most dominant saturated fatty acid (SFA) in all samples investigated. The content varied between 11.69 ± 0.52 for Maraqui oil and 21.36 ± 0.98 g per 100 g for Arbequina oil. Linoleic acid is a di-unsaturated fatty acid; when present in notable quantities, it can contribute to the oxidation of olive oil during storage [29]. The analytical results showed that the content of this acid varied between 8.87 ± 0.69 and 28.02 ± 1.38 g per 100 g. These results agree with those obtained by other authors

	Maraqi	Wattagen	Arbequina	Koroneiki	Coratina
free acidity (as g oleic acid per 100g)	1.20±0.10	0.45±0.05	0.76±0.05	0.30±0.05	0.35±0.05
peroxide value (meq O ₂ kg ⁻¹ oil)	2.39±0.10	2.76±0.15	2.45±0.10	2.69±0.15	2.16±0.010
K₂₃₂ (Abs)	1.428±0.06	2.189±0.08	2.264±0.09	1.968±0.07	1.782±0.07
K₂₇₀ (Abs)	0.067±0.01	0.097±0.01	0.133±0.01	0.182±0.01	0.117±0.01
Δk	0.000±0.00	0.000±0.00	0.000±0.00	0.000±0.00	0.000±0.00
Sensory analysis					
Defects	Yes	Yes	Yes	Yes	Yes
Fruity	5.5±0.4 floral, fruity olive mature, melon, almond flavors	5.0±0.3 fruity olive mature almond	6.2±0.4 floral, fruity olive ripe pear, melon, banana flavors	6.0±0.2 aromatic herbs and fruity olive ripe fruity green olive, almond flavors	6.5±0.3 aromatic herbs and fruity olive ripe fruity green olive, almond flavors
Bitterness	3.3±0.5	3.5±0.3	4.2±0.5	4.3±0.4	5.4±0.3
Pungency	2.5±0.2	2.2±0.4	3.3±0.3	3.5±0.2	5.0±0.4

Table 1: Quality parameters of some Egyptian virgin olive oils. Values represent the mean ± standard deviation of three independent biological replicates.

[31]. The fatty acid composition of the studied olive oils complies with the requirements of the IOC trade standard [14], except for Arbequina olive oil.

The samples of Maraqui and Wattagen showed the lowest total SFA (14.83 ± 0.20 and 14.81 ± 0.20 , respectively) and the highest total UFA (85.08 ± 1.13 and 85.12 ± 1.14 , respectively), while samples of Arbequina showed the highest total SFA (23.28 ± 0.36 g per 100 g) and the lowest total UFA (76.58 ± 0.74 g per 100 g) as compared to the other VOOs

The ratios between the total SFA to total UFA again confirmed the above results, that the oils from Maraqui and Wattagen cultivars had the lowest ratios (0.17 ± 0.10). On the other hand, Arbequina oil had the highest ratio (0.30 ± 0.20).

In general, quality olive oils should show a percentage content of oleic acid higher than 73%, a percentage content of linoleic acid lower than 10% and a ratio of these two acids greater than 7. From the data obtained, these values are fully respected by the oils from Siwa oasis.

Tocopherols

Tocopherols are particularly important functional components in foods. They have vitamin E properties and display antioxidant activity, which protect the body tissues against the damaging effects caused by the free radicals that result from many normal metabolic functions. Among all tocopherol homologues, α -tocopherol presents the highest biological potency [32,33]. It is the predominant representative of Vitamin E in VOO. The concentration of α -tocopherol, reported in the literature, for good-quality VOO's, is usually in the range 100-800 mg kg⁻¹. β - and γ -tocopherols are found in smaller amounts, and δ -tocopherol only in traces [6,7,34].

Table 3 lists the results relative to the tocopherol homologues found in the Egyptian olive oils. As expected, the data show the predominance of α -tocopherol in all olive oil samples analysed, followed by β -, γ -, and δ -tocopherol, respectively. These findings appear to agree with the results obtained by other authors [6,35,36] who showed that good-quality oils generally have α -tocopherols concentration of more than 100 mg kg⁻¹, with α -tocopherol accounting for approximately 95 g per 100 g of the total. In general, olive oils of Coratina, Arbequina and Koroneiki coming from Giza showed a higher content of total tocopherols.

Volatile compounds

The current olive oil official regulations [15] classify the most frequent off flavors into four groups: fusty, mustiness-humidity, winey-vinegary, and rancid. Fusty is the characteristic flavor of oils obtained from olives in an advanced stage of fermentation. Mustiness-humidity is the characteristic flavor of oils obtained from olives stored under humid conditions for several days with the consequence of the development of various kinds of fungi. Winey-vinegary is a sensory note due to the high concentration of acetic acid, ethyl acetate and ethanol. Rancid is a common sensory characteristic of all oils and fats that have undergone a process of auto-oxidation caused by a prolonged contact with air. The first three defects are due to inadequate fruit preservation before olive oil processing while the last is produced during olive oil storage. All these defects are currently considered in the Organoleptic Assessment of Virgin Olive Oil [27], whose purpose is to determine the criteria needed to assess the flavor characteristics of VOO. The method is applicable to the classification of the VOOs as a function of the intensity of the defects, according to the judgement of a group of selected and trained assessors working as a panel [27]. The acceptability of the oils depends on the defect intensities.

	Maraqi	Wattagen	Arbequina	Koroneiki	Coratina
C _{14:0}	0.02±0.00	0.02±0.00	0.01±0.00	0.04±0.00	0.01±0.00
C _{16:0}	11.69±0.52	11.98±0.55	21.36±0.98	14.93±0.65	14.36±0.68
C _{16:1}	0.35±0.06	0.56±0.08	3.32±0.25	1.02±0.11	0.47±0.07
C _{17:0}	0.05±0.00	0.06±0.00	0.06±0.00	0.03±0.00	0.05±0.00
C _{17:1}	0.06±0.00	0.07±0.00	0.15±0.02	0.04±0.00	0.05±0.00
C _{18:0}	2.69±0.20	2.34±0.19	1.43±0.13	2.12±0.18	1.88±0.15
C _{18:1}	74.80±2.88	73.26±2.91	44.00±1.65	71.86±2.59	67.62±2.05
C _{18:2}	8.87±0.69	9.21±0.75	28.02±1.38	8.08±0.75	13.62±0.93
C _{20:0}	0.27±0.05	0.31±0.06	0.27±0.05	0.38±0.06	0.37±0.06
C _{18:3}	0.69±0.09	0.79±0.10	0.79±0.09	0.85±0.10	0.93±0.11
C _{20:1}	0.31±0.06	0.23±0.05	0.30±0.06	0.37±0.06	0.40±0.07
C _{22:0}	0.08±0.00	0.06±0.00	0.10±0.01	0.14±0.01	0.12±0.01
C _{24:0}	0.03±0.00	0.04±0.00	0.05±0.00	0.05±0.00	0.06±0.00
Σ SFA*	14.83±0.20	14.81±0.20	23.28±0.36	17.69±0.24	16.85±0.25
Σ USFA**	85.08±1.13	85.12±1.14	76.58±0.74	82.22±1.01	83.09±0.82
SFA/USFA	0.17±0.10	0.17±0.10	0.30±0.20	0.21±0.12	0.20±0.15
18:1/18:2	8.43±1.15	7.17±1.01	1.57±0.75	8.89±1.15	4.69±0.99
18:2/18:3	12.85±0.75	12.92±0.78	35.46±1.00	9.51±.59	14.64±0.85
18:1/USFA	0.87±0.75	0.86±0.75	0.57±0.44	0.87±0.75	0.81±0.72

*SFA = saturated fatty acid **USFA = unsaturated fatty acid

Table 2: Fatty acid composition of Egyptian olive oils. The values represent the mean percentage ± standard deviation of three independent biological replicates.

The stimulation of olfactory receptors from volatile compounds present in VOOs, transported by the airstreams during inhalation and expiration actions, gives rise to perception of their aroma. It is generally assumed that the intensity of stimuli elicited by volatile substances is related to their amount; therefore the simpler approach of searching for possible relationships between volatile compounds and sensory characteristics of VOOs is to relate the concentrations of volatiles to some sensory notes, especially defective, of VOOs

In olive fruits stored in piles, under high humidity conditions [11], the presence of several species of genus *Aspergillus*, together with *Ascomycetes*, *Penicillium notatum*, have been reported as being among the most abundant *Deuteromycetes*. These microorganisms have the ability to oxidise free fatty acids, producing volatile compounds such as methyl ketones (2-heptanone, 2-nonanone). Other fungi (*Alternaria*, *Fusarium*, *Rhizopus*) have also been detected although they are less abundant. Yeasts, on the other hand, are able to reduce carbonyls and partially esterify alkyl moieties, and some species of the genera *Candida*, *Pichia* and *Saccharomyces* have been detected under the described conditions. The result is an olive oil characterised by the sensory note mustiness-humidity. Odour qualities of some volatiles compounds here identified by matching with NIST mass spectra library are discussed below.

Molecules like 2-decenal, heptanal, trans-2-nonenal, cis-2-nonenal and cis-2-decenale are responsible of fatty odour; 2,4 decadienal is correlated to deep fried taste; hexanoic acid is correlated to sweaty and pungent odour; trans-2-nonenal is correlated to paper like, sharp and cut grass; cis-2-nonenal is correlated to green taste [11].

Octanol is correlated to fatty and sharp taste; 2-nonanol is correlated to floral, fruity and blue cheese; E-2-nonenal is associated to fatty and waxy; heptanal is associated to fatty, greasy and ham-like; hexane is correlated to spicy; octane is correlated to sweet and alkane [37]. Octan-2-ol is associated with earthy and fatty; hexanoic acid with sharp and rancid; octanoic acid with rancid and fatty; heptanal with oily, fatty and woody; octanol with fatty and sharp [38]. The sensory characterisation of many of the volatile compounds here described with mouldy, woody, earthy and nutty sensory notes can explain the mustiness humidity sensory attribute. Noticeable is the high concentration of esters which are characteristic of olive oils obtained from over-ripe olives as well as due to the enzymatic activity of the cited microorganisms.

In light of all the previously mentioned characteristics, oil from Maraqui cultivar showed a presence of 3-nonen-1-ol (Z) as the most abundant volatile compound ($2676 \pm 30.25 \text{ mg kg}^{-1}$) (Table 4). Results revealed that hexane-2,4-dimethyl was the second most dominant

compound in the oil from Maraqui cultivar ($396.32 \pm 3.56 \text{ mg kg}^{-1}$). Both octanal and 2-decenal (Z) were also found at lower concentrations (ie. 213.34 ± 2.00 and $192.94 \pm 2.21 \text{ mg kg}^{-1}$, respectively), both 10-undecenal were detected at $176.65 \pm 2.25 \text{ mg kg}^{-1}$. The concentrations of the other detected compounds were less than 100 mg kg^{-1} .

Data in Table 4 shows that Wattagen oil contained 1-undecanol and 1,10-decanediol as the most prevalent volatile compounds (7150.12 ± 78.65 and $7150.12 \pm 85.24 \text{ mg kg}^{-1}$, respectively), followed by propanoic acid 2-hydroxy-2-methyl-ethyl ester as the third most dominant volatile compounds ($2165.04 \pm 20.25 \text{ mg kg}^{-1}$). Data also revealed that this oil also contained octane, nonanoic acid, octanal and 7-tetradecenal (Z) at concentrations of 1690.01 ± 15.21 , 667.16 ± 6.25 , 569.18 ± 5.21 and $545.70 \pm 5.68 \text{ mg kg}^{-1}$, respectively, and octanoic acid, 2-nonenal (Z), heptanal and 4-nonenal (E) at concentrations of 335.82 ± 1.98 , 335.22 ± 2.54 , 253.81 ± 2.25 and $245.08 \pm 2.11 \text{ mg kg}^{-1}$, respectively. Both of hexane-2,4-dimethyl and 2,4-decadienal (E,E) were also found at concentrations of 244.18 ± 2.58 and $141.50 \pm 2.01 \text{ mg kg}^{-1}$ respectively. Both 1-octanol and 2-nonanone were detected at 122.12 ± 1.98 and $111.69 \pm 1.52 \text{ mg kg}^{-1}$, respectively. The concentrations of 4,4,6-trimethyl-cyclohex-2-en-1-ol, 2-decanone and E-3-pentadecen-2-ol were around 102.81 ± 1.08 and $101.57 \pm 1.58 \text{ mg kg}^{-1}$, respectively. The concentrations of the other detected compounds were less than 100 mg kg^{-1} .

Moreover, the most abundant volatile compound (Table 4) in Arbequina sample was 3-nonen-1-ol (Z) ($2192.07 \pm 30.65 \text{ mg kg}^{-1}$) similar to the value obtained for Maraqui sample. Data showed that the concentrations of 10-undecenal and 2-decenal-(E) were 830.33 ± 19.52 and $686.04 \pm 14.56 \text{ mg kg}^{-1}$, respectively. Arbequina oil also contained smaller concentrations of octanal ($206.85 \pm 1.59 \text{ mg kg}^{-1}$), pentane-3-methyl ($170.16 \pm 2.15 \text{ mg kg}^{-1}$) and hexane- 3- methyl ($102.11 \pm 1.24 \text{ mg kg}^{-1}$). The concentrations of the other detected compounds were less than 100 mg kg^{-1} .

Results in Table 4 revealed that Koroneiki oil contained 2-nonen-1-ol (z) as the most abundant volatile compound ($2799.95 \pm 33.25 \text{ mg kg}^{-1}$), although detected only in this oil, followed by 10-undecenal and 2-decenal-(Z), 264.63 ± 4.52 and $243.77 \pm 2.65 \text{ mg kg}^{-1}$, respectively. Data also showed that the concentrations of hexane 2,4 dimethyl and nonanoic acid were 238.17 ± 2.68 and $197.35 \pm 1.57 \text{ mg kg}^{-1}$, respectively. The concentrations of the other detected compounds were less than 100 mg kg^{-1} .

Sample of Coratina showed the lowest number of detectable volatile compounds (only 5 compounds), 1,9-nonanediol being the

	Maraqi	Wattagen	Arbequina	Koroneiki	Coratina
α-tocopherol	691.75±23.52	541.34±19.52	634.34±22.35	678.75±22.95	766.00±24.53
β-tocopherol	6.39±2.20	6.53±2.23	5.94±2.12	3.02±1.05	3.57±1.12
γ-tocopherol	11.81±4.12	6.67±2.32	16.62±4.98	14.82±3.98	27.17±5.66
δ-tocopherol	1.30±0.21	4.54±1.12	0.79±0.19	0.42±0.12	1.92±0.32
total tocopherols	711.24±21.55	559.09±20.54	657.69±22.12	697.00±22.56	798.65±24.82

Table 3: Tocopherols content (mg kg^{-1}) in Egyptian olive oils. The values represent the mean \pm standard deviation of three independent biological replicates.

	Maraq	Wattagen	Arbequina	Koroneiki	Coratina
1-propyl-cyclopentanol	16.75±0.52	n.d.	n.d.	n.d.	n.d.
1-octanol	17.75±0.54	122.12±1.98	34.60±1.32	n.d.	n.d.
1,9-nonanediol	n.d.	n.d.	n.d.	33.20±1.05	632.05±15.01
1,10 decanediol	38.09±1.12	7150.12±85.24	n.d.	n.d.	n.d.
1-undecanol	n.d.	7150.12±78.95	n.d.	n.d.	n.d.
2-isopropyl-5-methyl-1-heptanol	14.65±0.20	n.d.	n.d.	n.d.	n.d.
2-nonen-1-ol (Z)	n.d.	n.d.	n.d.	2799.95±33.25	n.d.
4,4,6-trimethyl-cyclohex-2en-1-ol	n.d.	102.81±1.08	n.d.	n.d.	n.d.
5-isopropyl-6,6-dimethylhept-3-ene-2,5-diol	n.d.	14.38±0.25	n.d.	n.d.	n.d.
3-nonen-1-ol (Z)	2675.59±30.25	n.d.	2192.07±30.65	n.d.	n.d.
E-3-pentadecen-2-ol	n.d.	101.57±1.58	n.d.	9.67±0.15	n.d.
Z-11-pentadecenol	n.d.	n.d.	n.d.	34.88±0.56	n.d.
Z-9-pentadecenol	n.d.	40.88±0.55	n.d.	n.d.	n.d.
10-heneicosene	n.d.	3.67±0.01	n.d.	n.d.	n.d.
heptanal	75.49±1.02	253.81±2.25	72.96±0.99	23.86±0.51	n.d.
octanal	213.34±2.00	569.18±5.21	206.85±1.59	48.45±0.63	n.d.
2-nonenal (E)	n.d.	38.29±0.88	n.d.	n.d.	n.d.
2-nonenal (Z)	n.d.	335.22±2.54	n.d.	n.d.	n.d.
4-nonenal (E)	38.34±0.55	245.08±2.11	n.d.	53.85±0.54	n.d.
6-nonenal (Z)	23.89±0.52	n.d.	n.d.	n.d.	n.d.
2-decenal-(E)	n.d.	n.d.	686.04±14.56	65.58±1.00	n.d.
2-decenal-(Z)	192.94±2.21	n.d.	n.d.	243.77±2.65	60.16±0.98
2-undecenal	8.94±0.21	25.60±0.52	n.d.	n.d.	n.d.
10-undecenal	176.65±2.25	n.d.	830.33±19.52	264.63±4.52	n.d.
10-octadecenal	9.62±0.08	0.42±0.01	n.d.	n.d.	n.d.
13-octadecenal (Z)	n.d.	n.d.	n.d.	n.d.	85.04±1.85
2,4 decadienal (E,E)	21.07±0.58	141.50±2.01	n.d.	n.d.	n.d.
7-tetradecenal (Z)	n.d.	545.70±5.68	n.d.	n.d.	n.d.
2,4 dodecadienal	n.d.	n.d.	80.87±1.95	n.d.	32.65±0.98
2-4-pentadien-1-ol-3-pentyl (Z,Z)	16.09±0.56	n.d.	n.d.	n.d.	n.d.
2-decanone	22.74±0.65	101.57±1.52	n.d.	n.d.	n.d.
2-nonanone	18.70±0.32	111.69±1.52	n.d.	n.d.	n.d.
cyclotridecanone	n.d.	41.32±0.68	n.d.	n.d.	n.d.
furanone	n.d.	n.d.	4.03±0.03	n.d.	n.d.
δ nonalactone	4.54±0.08	7.48±0.08	n.d.	n.d.	n.d.
2-hexanone-4-methyl	n.d.	15.43±0.35	n.d.	n.d.	n.d.
cyclohexanone 3,3,5,5 tetramethyl	n.d.	17.19±0.31	n.d.	n.d.	n.d.
cyclopentanone 3-butyl	10.67±0.19	14.05±0.32	n.d.	n.d.	n.d.
4hydroxy4methylhex5enoicacidtertbutyl ester	n.d.	56.39±0.98	n.d.	n.d.	n.d.
9-hexadecenoic acid methyl ester (Z)	30.85±0.54	n.d.	n.d.	n.d.	n.d.
hexanoic acid propyl ester	n.d.	57.17±0.65	n.d.	n.d.	n.d.
propanoic acid 2hydroxy2methylethyl ester	n.d.	2165.04±20.25	n.d.	n.d.	n.d.
tetranoic acid ethyl ester	n.d.	n.d.	n.d.	16.01±0.35	n.d.
valeric acid-4-tridecylester	10.71±0.11	n.d.	n.d.	n.d.	n.d.
2H-pyran-2-one-tetrahydro-6-nonyl	4.33±0.02	n.d.	n.d.	n.d.	n.d.
2-n-octylfuran	7.34±0.15	n.d.	n.d.	n.d.	n.d.

2-octen-(Z)	n.d.	33.03±0.78	n.d.	n.d.	n.d.
phenol 3,5-bis (1,1 dimethylethyl)	n.d.	n.d.	98.87±1.03	n.d.	n.d.
pentane-3-methyl	n.d.	n.d.	170.16±2.15	n.d.	n.d.
octane	n.d.	1690.01±15.21	n.d.	n.d.	n.d.
vinylcaprylate	16.68±0.33	n.d.	n.d.	n.d.	n.d.
butane-2-methyl	27.50±0.45	n.d.	n.d.	n.d.	n.d.
E-2-methyl-tetradecen-1-olacetate	n.d.	n.d.	n.d.	3.49±0.01	n.d.
furan-2-pentyl	16.33±0.32	46.59±0.65	28.30±0.52	8.73±0.25	n.d.
hexane 2,4 dimethyl	396.32±3.56	244.18±2.58	1.36±0.01	238.17±2.68	n.d.
hexane 3-methyl	47.69±0.59	76.93±0.99	102.11±1.24	13.95±0.26	n.d.
hexanoic acid	n.d.	92.38±1.02	n.d.	n.d.	n.d.
nonanoic acid	26.11±0.42	677.16±6.25	41.34±0.61	197.35±1.57	n.d.
Z-8-methyl-9-tetradecenoic acid	n.d.	23.41±0.44	n.d.	n.d.	n.d.
octanoic acid	n.d.	335.82±1.98	n.d.	n.d.	n.d.

*n.d.= not detected

Table 4. Volatile compounds (mg kg⁻¹) in Egyptian olive oils. Values represent the mean ± standard deviation of three independent biological replicates.

most abundant (632.05 ± 15.01 mg kg⁻¹). The oil also contained smaller concentrations of 13-octadecenal, 2-decenal (z) and 2,4-dodecadial (85.04 ± 1.85, 60.16 ± 0.98 and 32.65 ± 0.98 mg kg⁻¹, respectively).

Sensory analysis

VOO flavor is usually characterised by pleasant sensory notes that are much appreciated by consumers [39]. These sensory characteristics, together with nutritional aspects, are the main reasons for the increment of VOO consumption in recent years (IOOC, 2003). Olive oil is prized for its sensory attributes [40].

The oils analysed showed defects associated mainly with the perception of rancid (Table 1). Olive oils of Coratina, Arbequina and Koroneiki coming from Giza showed an higher perception of bitterness, spicy and fruity than the oils of Maraqui and Wattagen coming from Siwa. This could be explained by considering that Giza city has a climate more arid than Siwa oasis. In particular, Coratina, Arbequina and Koroneiki olive oils showed a perception of fruity whose average value was 6.3 ± 0.3, while Maraqui and Wattagen olive oils was 5.3 ± 0.3. Spicy and bitter attributes gave average values of 4 ± 0.4 and 4.6 ± 0.3 for Coratina, Arbequina and Koroneiki olive oils respectively, while average values of 2.3 ± 0.4 and 3.4 ± 0.3 were obtained for olive oils of Maraqui and Wattagen cultivars.

Conclusions

The geographic area of origin appears to play a significant role for the qualitative characteristics and the sensory attributes of the olive oils analysed. The results obtained showed that olive oils coming from Giza, located on the west bank of the Nile River with an arid climate but also with high humidity due to the River Nile's Valley effects, demonstrated excellent nutritional characteristics in terms of antioxidant compounds. In fact, the olive oils showed a higher content of total tocopherols and were characterised by a higher perception of bitterness, pungency and fruity.

As well as in quality olive oils, samples coming from Siwa Oasis, located between the Qattara Depression and the Egyptian Sand Sea in

the Libyan Desert and very rich in water, showed an higher percentage content of oleic acid. Moreover, the percentage content of linoleic acid was lower than 10% and the ratio of oleic and linoleic acids was greater than 7.

Finally, SPME/GC/MS analysis revealed that most of the volatile compounds are characterised by sensory notes related to the perceptions of rancid, oily and fatty. The high concentrations of aldehydes, mostly produced by oxidation of the unsaturated fatty acids, and acids, mostly produced by the oxidation of the aldehydes previously formed, found in the samples analysed clearly highly influence the final aroma of the olive oils justifying the negative attributes noticed by the sensory evaluation.

Acknowledgments

This research was supported by the RGV-FAO and RIOM II Projects. The authors would like to thank M. Pellegrino, A. Parise O., Godino G., O. Miranda and B. Rizzuti who helped with analytical techniques and with panel test.

References

1. Fernandez Diez MJ (1971) The olive, in the biochemistry of fruits and their products. Academic press, London, 2: 255-279.
2. Muzzalupo I, Perri E (2008) Genetic characterization of olive germplasms by molecular markers. Eur J Plant Sci Biotech 2: 60-68.
3. Loukas M, Krimbas CB (1983) History of olive cultivars based on their genetic distances. Hort Sci 58: 121-127.
4. Barranco D, Fernandez-Escolar R, Rallo L (1996) El cultivo del olivo, Junta de Andalucia, Consejería de Agricultura y pesca and ediciones mundiprensa, Madrid, Barcelona, Mexico.
5. Bouaziz M, Jemai H, Khabou W, Sayadi S (2010) Oil content, phenolic profiling and antioxidant potential of Tunisian olive drupes. J Sci Food Agric 90: 1750-1758.
6. Muzzalupo I, Stefanizzi F, Perri E, Chiappetta A (2011) Transcript levels of CHL P gene, antioxidants and chlorophylls contents in olive (*Olea europaea* L.) pericarps: a comparative study on eleven olive cultivars harvested in two ripening stages. Plant Food Hum Nutr 66: 1-10.
7. Psomiadou E, Tsimidou M, Boskou D (2000) α-tocopherol content of Greek virgin olive oils. J Agric Food Chem 48: 1770-1775.

8. Caravita MA, Benincasa C, De Rose F, Muzzalupo I, Parise A, et al. (2007) Omega-3 / omega-6 fatty acids ratio in olive oils from Italian olive varieties. *Agro Food Ind Hi Tec* 18: 17-18.
9. Servili M, Selvaggini R, Esposito S, Taticchi A, Montedoro GF, et al. (2004) Health and sensory properties of virgin olive oil hydrophilic phenols: agronomic and technological aspects of production that affect their occurrence in the oil. *J Chromatogr A* 1054: 113-127.
10. De Nino A, Mazzotti F, Perri E, Procopio A, Raffaelli A, et al. (2000) Virtual freezing of the hemiacetal-aldehyde equilibrium of the aglicones of oleuropein and ligstroside present in olive oils from Carolea and Coratina cultivars by ionspray ionization tandem mass spectrometry. *J Mass Spectrom* 35: 461-467.
11. Angerosa F (2002) Influence of volatile compounds on virgin olive oil quality evaluated by analytical approaches and sensory panels. *Eur J Lipid Sci Tech* 104: 639-660.
12. Patumi M, D'andria R, Marsillio V, Fontanazza G, Morelli G, et al. (2002) Olive and olive oil quality after intensive monocone olive growing (*Olea europaea* L., Cv. Kalamata) in different irrigation regimes. *Food Chem* 77: 27-34.
13. Duran RM (1990) Relationship between the composition and ripening of the olive and the quality of the oil. *Acta Horticulturae* 286: 441-452.
14. IOC (2006) Trade standard applying to olive oils and olive pomace oils in COI/T.15/NC 3/Rev. 2.
15. EEC (1991) Characteristics of olive and olive pomace oils and their analytical methods. Regulation EEC/2568/91 and latter modifications. Official Journal of the European Communities.
16. Boskou D (1996) Olive oil chemistry and technology. AOCS press, Champaign, IL, USA.
17. Rossell JB (1986) Classical analysis of oils and fats. (Eds), In Hamilton RJ and Rossell JB, Elsevier Applied Science Publishers, Ltd, England.
18. Kalua CM, Allen MS, Bedgood DR, Bishop AG, Prenzler PD, et al. (2007) Olive oil volatile compounds, flavor development and quality. A critical review *Food Chem* 100: 573-575.
19. Dhifi W, Khedher MB, Kchouk ME, Marzouk B (2006) Qualitative and quantitative of the aromas and polyphenols of some Tunisian olive oils. *Olivae* 105: 36-42.
20. Gutierrez F, Fernandez JL (2002) Determinant parameters and components in the storage of virgin olive oil. Prediction of storage time beyond which the oil is no longer of "extra" quality. *J Agric Food Chem* 50: 571-577.
21. Amiot MJ, Fleuriat A, Macheix JJ (1986) Importance and evolution of phenolic compounds in olive during growth and maturation. *J Agric Food Chem* 34: 823-826.
22. Botia JM, Ortuno A, Benavente-Gracia O, Báidez AG, Frias J, et al. (2001) Modulation of the biosynthesis of some phenolic compounds in *Olea europaea* L. fruits: Their influence on olive oil quality. *J Agric Food Chem* 49: 355-358.
23. Esti M, Cinquanta L, La Notte E (1998) Phenolic compounds in different olive varieties. *J Agric Food Chem* 46: 32-35.
24. Vinha AF, Ferreres F, Silva B, Valentão P, Gonçalves A, et al. (2005) Phenolic profiles of Portuguese olive fruits (*Olea europaea* L.): influences of cultivar and geographical origin. *Food Chem* 89: 561-568.
25. Lazze A, Perri E, Caravita MA, Khlif M, Cossentini M (2008) Influence of olive maturity stage and geographical origin on some minor components in virgin olive oil of the Chemlali variety. *J Agric Food Chem* 56: 982-988.
26. Cavaliere B, De Nino A, Hayet F, Lazze A, Macchione B, et al. (2007) A metabolomic approach to the evaluation of the origin of extra virgin olive oil: a convenient statistical treatment of mass spectrometric analytical data. *J Agric Food Chem* 55: 1454-1462.
27. Clodoveo ML, Delcuratolo D, Gomes T, Colelli G (2007) Effect of different temperatures and storage atmospheres on Coratina olive oil quality. *Food Chem* 102: 571-576.
28. IOC (2007) Sensory analysis of olive oil. Method for the organoleptic assessment of virgin olive oil. COI/T.20/Doc. 15/Rev. 2.
29. Martinez de Victoria E, Manas M (2001) El aceite de oliva en la dieta y salud humanas. In Barranco, D., Fernandez, E. P., and Rallo, L. (Eds). *El cultivo del olivo*. Madrid: Mundiprensa, pp. 663 – 684.
30. Ryan D, Robards K, Lavee S (1998) Assessment of quality in olive oil. *Olivae* 72: 23-31.
31. Dabbou S, Chehab H, Faten B, Dabbou S, Esposito S, et al. (2010) Effect of three irrigation regimes on Arbequina olive oil produced under Tunisian growing conditions. *Agr Water Manage* 97: 763-770.
32. Manai H, Haddada FM, Imen O, Trigui A, Daoud D, et al. (2006) Variability in the composition of olive oil produced from hybrids obtained by controlled crossbreeding. *Olivae* 106: 17-23.
33. Perri E, Mazzotti F, Raffaelli A, Sindona G (2000) High-throughput screening of tocopherols in natural extracts. *J Mass Spectrom* 35: 1360-1361.
34. Bruno L, Chiappetta A, Muzzalupo I, Gagliardi C, Iaria D, et al (2009) Role of geranylgeranyl reductase gene in organ development and stress response in olive (*Olea europaea*) plants. *Funct Plant Biol* 36: 370-381.
35. Boskou D, Blekas G, Tsimidou M (2006) Olive oil composition. In Boskou D (Ed.), *Olive oil, chemistry and technology* Champaign Illinois: AOCS Press, pp 41-72.
36. Speek AJ, Schrijver J, Schreus WHP (1985) Vitamin E composition of some seed oils as determined by high performance liquid chromatography with fluorimetric detection. *J Food Sci* 50: 121-127.
37. Perrin JL (1992) Les composés mineurs et les antioxygènes naturels de l'olive et de son huile. *Rev Fr des Corps Gras* 1/2: 25-32.
38. García-González DL, Tena N, Aparicio R (2007) Characterization of olive paste volatiles to predict the sensory quality of virgin olive oil. *Eur J Lipid Sci Tech* 109: 663-672.
39. Morales MT, Luna G, Aparicio R (2008) Comparative study of virgin olive oil sensory defects. *Food Chem* 91: 293-301.
40. Aparicio R, Morales MT, Alonso MV (1997) Authentication of European extra-virgin olive oils by their chemical compounds, sensory descriptors and consumer's attitudes. *J Agric Food Chem* 45: 1076-1083.
41. Visioli F, Bellosta S, Galli C (1998) Oleuropein, the bitter principals of olives, enhance nitric oxide production by mouse macrophages. *Life Sci* 62: 541-546.