A Study on the Effect of Harvest Time on Quality of Egyptian Olive Oil

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ABSTRACT

Time of harvest may have a profound influence on the yield, quality and stability of olive oil. However, there are few clear guidelines that producers can use to determine the optimum harvesting time. Therefore, the aim of the present study was to investigate the effect of harvesting date on the compositional quality of extracted oils from Maraky and Wettagen cultivars grown in Siwa's Oasis. Some physical properties and oil quality were determined during ripening period of fruits from August up to January during the season of 2005/2006. Quality parameters included FFA, PV, UV absorption, fatty acid profile, pigments (chlorophylls and carotenoids), tocopherols, phenolic compounds, sterols and squalene.

The results indicated that, the amount of oil recovered from each cultivar in the early/ unripe (August and September) and overripe (December and January) stages of maturity were very low. The highest amount of oil extracted was achieved in November in both two cultivars. The FFA content was in the lowest level in the immature fruits and was the highest level in the overripe fruits. Oleic and palmitic acids were found to decrease while linoleic acid increased during ripening. The percentage of oleic acid in olive oil from the two cultivars was obviously lower than that reported in literature from other areas and higher for linoleic and palmitic acids. Chlorophyll and β-carotene, tocopherol, phenolic compounds, sterol and squalene contents were in the highest levels in September and were in the lowest levels in January. Data obtained may advance our knowledge on the physical and chemical changes that take place during fruit ripening and from this we can define monitoring parameters that will assist olive producers to determine optimum harvest time for optimal olive oil quality. These parameters include International Olive Oil Council standards as well as the minor components in olive oil as pigments, polyphenols and sterols which play an important role in olive oil quality.

Keywords: harvest period, physical properties, olive oil quality, fatty acids, pigments, tocopherols, phenolic compounds, sterols, squalene.

INTRODUCTION

Consumers increasingly understand the importance of olive oil quality and stability as well as the nutritional benefits of monounsaturated oil with naturally low levels of saturated fatty acids. Olive oil quality may be defined from compositional quality, nutritional or organoleptic perspectives (Duran, 1990). There are several ways of defining olive oil quality and perhaps there is no single universal definition that adequately satisfies all situations. In general, quality is defined as "The combination of attributes or characteristics of a product that have significance in determining the degree of acceptability of that product by the user'' (Gould, 1992). The International Olive Oil Council (IOOC, 2004) has defined the quality of olive oil, based on parameters that include free fatty acid (FFA), peroxide value (PV), UV absorption at 232 and 270 nm and sensory score. In particular, the quantity of FFA is an important factor for classifying olive oil into commercial grades (Rossell, 1986, Boskou, 1996). Moreover, IOOC (2004) indicated that extra virgin olive oil should be contain FFA less than 0.8% as oleic acid, peroxide value less than 20 meq.O2/kg, high percentage of oleic acid (more than 55% of total fatty acids), moderate amount of linoleic acid (less than 21% of total fatty acids) and very low percentage of polyunsaturated linolenic acid (≤ 1% of total fatty acids). This profile keeps shelf life of extra virgin olive oil up to two years.

The best time to harvest olive fruits is when the olives are ripe, oil formation is completed and the oil is at the top quality and quantity. Ripening begins when the first violet making appears on the fruit and ends when the whole skin and flesh have completely changed colour (Mailer *et al*., 2004).

Olive oil quality is affected by genetic, agronomic and environmental factors. Some studies have shown that the climate conditions, particularly rainfall and water irrigation during the growing and

the stage of repining of the olive fruit, influence the quality of the olive oil. Olives harvested relatively early, yield low amount of oil with a fruity flavour, lower acidity and greener colour than olives harvested late in the season (Garcia *et al*., 1996). During ripening, important chemical changes occur inside the drupes which are related to the synthesis of organic substances that may affect virgin olive oil quality (Montedoro *et al*., 1986).

The fatty acid profile is important in determining olive oil quality and stability. Oleic acid is considered beneficial and high levels are encouraged. Although oleic acid changed little over the maturity period, linoleic acid was found to increase while palmitic acid decreased. Higher levels of polyunsaturated fatty acids are nutritionally beneficial but reduce oil stability. Harvest time can be used to select for stability or/and nutritive value. Linolenic acid also decreased with maturity, this component is unstable and can increase the rate of oxidation. However, in Australia it has been found that linolenic acid levels often exceed international standards (more than 1% of total fatty acids), thus making it a possible problem in international trade (Mailer, 2004).

Chlorophylls and carotenoids are the pigments responsible for the colour of virgin olive oil (Cichelli & Pertesana, 2004). As the harvesting time of the olive fruits increases, pigment content decreases (Minguez-Mosquera *et al*., 1990 & 1992). They mentioned that chlorophylls and carotenoids can undergo oxidation under certain conditions, being degraded to uncoloured products. Roca & Minguez-Mosquera (2001) found that during ripening, the concentration of chlorophylls decreased continuously in all varieties, concomitant with the increase in anthocyanin pigmentation. In the different olive varieties, chlorophylls always disappeared more rapidly than carotenoids, in both skin and pulp in there stages of ripeness (Criado *et al*., 2007).

Tocopherols, known as provitamin E, contribute to the stability of olive oil due to their role as radical quenchers. Total tocopherol content is reported to be between 100 and 300 mg/kg oil for most commercial olive oils. Tocopherol concentration is significantly reduced towards the end of the harvest period (Boskou, 1996).

Virgin olive oil contains a considerable amount of polyphenols that have a great effect on the stability, the sensory and nutritional characteristics of the product (Montedoro *et al*., 1992, Tsimidou *et al*., 1992, Tovar *et al*., 2001). It has long been known that the levels of phenols in olive oils can be influenced by the cultivar (Brenes *et al*., 1999), the degree of maturation (Cinquanta *et al*., 1997), and the industrial processes employed for oil extraction, as well as environmental conditions (Motilva *et al*., 2000). The phenol profile can be followed from the fruit to the oil production and through storage, and may serve as a good indicator of olive oil quality. Indeed, there have been proposals to include phenols in the olive oil quality standard (Blekas *et al*., 2002, Psomiadou *et al*., 2003).

Changes in the unsaponifiable sterol fraction of virgin olive oil have been reported during fruit ripening (Malta & Benzo, 1972, Amelotti *et al*., 1973, Camera *et al*., 1975, Tiscornia et ai., 1978). In general, the sterol fraction did not vary substantially, except for a significant increase in Δ-5-avenasterol that coincided with the highest oil content in the fruit while β-sitosterol declined during the ripening period (Salvador *et al*., 2001).

Atta & Ahmed (2005) found that total polyphenols and α-tocopherols decreased as a result of the ripening stages. Unsaponifiable matter decreased through ripening showing the highest levels for harvested fruit by mid September (1.55%) and decreased to less than 1.37% by late October.

The effect of fruit ripeness on the antioxidant content of 'Hojiblanca' virgin olive oils was studied by Beltrán *et al*. (2007). In general, the antioxidants and the related parameters (phenolic content, tocopherols, carotenoid and chlorophyllic pigments) decreased as olive fruit ripened.

Some parameters that are not included in the IOOC (2004) standards, such as phenolic compounds, are known to have a significant effect on the quality, stability and nutritional value of olive oil. Therefore, the minor components as pigments, tocopherols, polyphenols, sterols and squalene which play an important role in olive oil quality were evaluated and discussed in this research work. The aim of the present study was to investigate the effect of harvest time on the compositional quality of extracted oils from Maraky and Wettagen cultivars of olive grown in Siwa's Oasis. Study focused on the physical and chemical changes that may take place during fruit ripening and may help us to define monitor parameters that can assist olive producers to determine the proper harvest time to insure optimal olive oil quality.

MATERIALS AND METHODS

Harvesting of olive fruits

The experiments were carried out using two olive varieties; Maraky and Wettagen, cultivated in the Oasis of Siwa, Egypt. Four selected olive trees of each olive variety were chosen. Sufficient amounts of olive fruit samples were hand-packed from all sides of olive tree once-a-month from August up to January during the season of 2005/2006. The collected samples were packed in polyethylene bags and kept at – 20°C until analyzed.

Maturity index

The maturity index was determined using 100 randomly selected olive fruits of each sample to obtain a numerical value for the olive sample appearance. Olives were cut in halves to expose the internal flesh and to permit grading. The olives were sorted into categories and the total number of olive fruits of each category was counted and recorded as described by Boskou (1996).

Weight of fruits

One hundred olive fruits from each sample were randomly selected and weighed, and the average weight of the fruit was calculated.

Moisture content

Approximately 1kg of fruits was crushed using a hammer mill. After mixing the sample thoroughly, 30 g of paste were transferred to a previously weighed Petri dish. The sample was dried in a fan-forced oven at 80°C for 24 hours (Boskou, 1996). The dry weight of the sample was recorded and the moisture content of the fruit was calculated as a percentage of the fruit weight.

Extraction of olive oil samples

Olive fruit samples were crushed using an experimental crusher mill, and packed in a cheese cloth then pressed by using hydraulic laboratory press (Carver). The resulting liquid phase was centrifuged (2000g) and the upper oil layer was collected and dried over anhydrous sodium sulphate then filtered through a Watmann filter paper No.1. The oil obtained was weighed and kept in a brown glass bottles at 5°C until analysis.

Bitter index of olive oil

The bitter index was evaluated by extraction the bitter components from 1.0±0.01 g oil by dissolving in 4 ml hexane and passed through an octadecyl (C18) column (Sep-Pack Cartridges; Waters, Milford, MA), activated previously with methanol (6 ml) and washed with hexane (6 ml). After elution, 10 ml of hexane was used to eliminate fat, and then the retained compounds (25 ml) were eluted with methanol/water (1:1, v/v) as described by Gutierrez *et al*. (1992). The absorbance of the extract was measured at 225 nm against methanol/water (1:1, v/v). On the other hand, bitterness was sensorialy evaluated by ten panelists as described by Abdalla & Zeitoun (1997). A scale from one to five was used. One indicates very low bitterness, 2 light, 3 moderate, 4 great and 5 extreme bitterness.

Quality parameters of olive oils

Free fatty acid, peroxide value and UV absorption

Free fatty acids, given as % of oleic acid were determined by the American Oil Chemists Society (AOCS, 1998). Peroxide value, expressed as meq. O2/kg oil was determined using the International Union of Pure and Applied Chemistry (IUPAC, 1992). K_{232} and K_{270} extinction coefficient were calculated from absorption at 232 and 270 nm, respectively, by Shimadzu UV-2101 spectrophotometer, using a 1% solution of oil in cyclohexane (AOCS, 1998).

Fatty acid composition

Fatty acid composition of different olive oil samples was determined as follows:

Fifty mg of lipid was transferred into screw-cap vial, then 2 ml benzene and 10 ml 1% H_2SO_4 in absolute methanol were added and the vial was covered under stream of nitrogen before heating, then saponification and methylation were carried out for 60 min. Fatty acid methyl esters (FAME) in each vial were extracted with 5 ml of petroleum ether. Analysis of fatty acid methyl esters was carried out using Gas Chromatography GC-4C Shimadzu CM (PFE) equipped with flame ionization detector (FID) and glass column (3m × 3mm i.d) packed with 5% DEGC on 80/100 chromosorb. Column temperature was 180°C isothermal and detector temperature was 270°C. Gas flow rates were 20 ml/min for N_2, 75 ml/min for H_2 and 0.5 ml/min for air. Standard mixture of FAME was analyzed under identical conditions prior to running the sample. The concentration of FAME was calculated by triangulation method (Abdalla, 1999).

Determination of pigments

Extraction of all pigments was carried out under green light, 15 g of olive oil were extracted as described by Minguez-Mosquera *et al*. (1990). Chlorophylls and β-carotene (mg/kg oil) were determined at 472 and 670 nm, respectively in cyclohexane by the method of Minguez-Mosquera *et al*. (1991).

Determination and identification of phenolic compounds

A modification of the Gutfinger (1981) method was used to determine total polyphenol content. Oil (10 g) was dissolved in hexane (50ml) and extracted 3 times with 20 ml portions of 80% aqueous methanol. The mixture was shaken for 2min. for each extraction. The sample was made up to 100 ml with water and left to stand in a dark cupboard overnight. An aliquot (1ml) was transferred to a 10 ml volumetric flask to which 5 ml of water was added. Folin- Ciocalteau reagent (0.5 ml) was then added and the sample shaken and left for 3 minutes. Saturated Na_2CO_3 (1ml) was added and the sample was shaken again. The sample was made up to volume with water and allowed to stand for 1 hour. The absorption was read at 725nm. Standard solutions of caffeic acid were used to construct a standard calibration curve.

Phenolic compounds of olive oil samples were identified and determined using high performance liquid chromatography (HPLC) according to the method of Caponio *et al*. (2001). The HPLC (Hewlett Packard Serious HP 2100) consisting of a model P 4600 pump with a Waters R401 detector, a U6K injector, and a Waters Bondapak C-18 column (30 cm × 4 mm). The solvent mixture of hexane and isopropanol (99.5 : 0.5 v/v) was used as the mobile phase at a flow rate of 1 ml/min. Samples were dissolved in methanol, and 10 ul of this solution was injected into the column.

Determination of tocopherols, sterols and squalene

Unsaponifiable matter of olive oil samples was extracted as described by Abdalla (1999). Tocopherols and sterols were analyzed in olive oil samples using GC (Pye-Unicam, Dundee University, UK) equipped with a glass column (25m × 0.4mm) packed with 3% *OV*-1 as recommended by Ghosh & Bhattacharyya (1996). The temperature programme was 250 to 300°C for 5min. The injector and detector temperatures were maintained at 320 and 330°C, respectively. The carrier gas was nitrogen at a flow rate of 30 ml/min.

Squalene was determined in olive oil samples using GC equipped with capillary silica column (25 m × 0.5 mm) packed with *SE* 52 (Carlo Ebra) as described by Bondioli *et al*. (1993). The temperature programme was 130 to 250°C at 5°C /min. Detector temperature was 260°C. The carrier gas was hydrogen at a flow rate of 1ml/min.

RESULTS AND DISCUSSION

Characteristics of olive fruits

The most obvious physical change during maturity, and one that is often used to determine fruit ripeness, is the maturity index. Maturity index is used in Europe as a strong indicator of the optimum time to harvest (Lavee, 1996). Olive fruits colour changed rapidly at different harvesting period in Maraky variety than for Wettagen variety, the maturity of Maraky variety was faster than Wettagen variety (Table 1).

Data in Table (1) show that the weight of 100 fruits was higher in Maraky variety than in Wettagen variety and it increased in the two varieties from August to December, then slightly decreased during January. It was obvious that Maraky fruits were bigger in size than Wettagen fruits. The results in Table (1) indicate that the maturity index and the fruit weight varied clearly according to cultivars and ripening period. These results are in agreement with data obtained by Ali (1999), Nergiz & Engez (2000), El-Makhzangy *et al*. (2001).

Moisture content is a major factor for olive fruits as it generally contributes to more than 50% of the fruit weight. The moisture content of the fruit can be influenced by numerous factors including rainfall, evaporation, irrigation events, and tree health. Moisture has several effects on the fruit and oil quality. There is a tendency for moisture content to reduce as the fruit matures. Data presented in Table (1) show that moisture content was generally high, more than 60%, in the two cultivars. There was no big difference in moisture content in August and September. However, the moisture levels showed an overall downward trend across the period from September up to December, and then slightly increased by January. The Wettagen cultivar showed higher moisture content with a maximum of 64.9% in August which decreased

Table 1. Some characteristics of two Egyptian olive cultivars (Maraky and Wettagen) during ripening*

	Harvest date*					
	August	**September**	**October**	**November**	**December**	**January**
Maturity Index (MI)						
Maraky	1.5±0.2	1.8 ± 0.2	2.8 ± 0.2	4.7 ± 0.2	5.9 ± 0.3	6.1 ± 0.3
Wettagen	1.3±0.2	1.4 ± 0.2	2.1 ± 0.2	4.0 ± 0.3	5.4 ± 0.2	5.6 ± 0.3
Weight of 100 fruits (g)						
Maraky	481.5±14.5	488.2 ± 13.2	541.6 ± 13.6	553.6 ± 16.2	572.8 ± 18.5	556.5 ± 19.3
Wettagen	412.4±11.3	416.6 ± 12.3	455.3 ± 14.5	493.4 ± 12.4	524.2 ± 11.4	502.7 ± 14.2
Moisture (%)						
Maraky	61.5±1.4	61.2 ± 1.4	56.1 ± 1.4	53.2 ± 1.3	52.1 ± 1.6	55.1 ± 1.1
Wettagen	64.9 ± 1.6	64.8 ± 2.2	60.2 ± 1.7	57.6 ± 1.6	55.1 ± 1.4	57.8 ± 1.5
Oil % (fresh weight)						
Maraky	15.6 ± 0.8	18.1 ± 0.8	22.5 ± 1.1	26.8 ± 1.5	25.4 ± 1.4	20.8 ± 1.2
Wettagen	13.8 ± 0.5	16.2 ± 0.6	19.2 ± 0.7	24.1 ± 1.1	23.8 ± 1.1	17.8 ± 0.8

*Average value of three determinations ± SD

to 55.1 % in December, while the Maraky cultivar showed relatively low average moisture level with a maximum of 61.5 % in August decreased to 52.1 % in December. These results are in agreement with those of Ali (1999), who found that moisture content of olive fruits varied according to cultivars and seasons and it ranged from 60 to 70%. Moreover, He also found that Maraky fruits had the lowest moisture content when harvested in November (53.7 and 53.1% in the first and second seasons, respectively). Similar behaviour has been reported by Nergiz & Engez (2000).

Olive fruit yield and oil content are the major contributors to profitability for olive growers where currently no premium is paid for quality. Factors which are considered to contribute to fruit yield and oil content would include cultivar, environment and seasonal effects (Gutierrez *et al*., 1999).

Samples of olives at each harvest in the present study were analyzed for oil content using cold press system. It can be seen from the results given in Table (1) that the amount of oil recovered for each cultivar in the early stages of maturity was low. However, as the fruit matured, the differences in the amount of oil extracted from each cultivar became greater. The highest amount of oil extracted was from Maraky variety and the maximum oil recovery of 26.8% of fresh weight was achieved in November. The amount of oil increased in the two varieties during the entire sampling period from August to November, and then decreased. In accordance, Basuny & Mostafa (2004) indicated that the amount of extracted oil from olive fruits harvested in October (unripe fruits) was lower as compared to the oil yield from olive fruits harvested in December (over ripe fruits).

Quality parameters of olive oil

Bitterness, free fatty acids, peroxide value and UV absorbance

In particular, bitterness decreased with maturity index (MI). The oils obtained from green olives were excessively bitter according to the panelist's comments. This does not imply rejection of the oil, but if the level of bitterness is too high it could cause some problems for consumer acceptance (Gutierrez *et al*., 1992, Mailer *et al*., 2005). In the present study, a high level of bitterness was recorded in unripe green olives (Table 2). Treatment of green olives with alkali is a common practice to reduce bitterness (Abdalla & Zeitoun, 1997). The intensity of the bittereness of olive oil has been related to the presence of phenolic compounds derived from the hydrolysis of oleuropein (Garcia *et al*., 1996). Data are in agreements with our results of both bitterness (Table 2) and phenolic compounds (Table 5).

The quantity of free fatty acids (FFA) is an important factor for classifying olive oil into commercial grades (Rossell, 1986, Boskou, 1996, Kalua *et al*., 2007). Extra virgin olive oil should not contain free fatty acid (FFA) more than 0.8 as oleic acid % and peroxide value should be less than 20 meq.O_2/kg oil (IOOC, 2004). The FFA content of Maraky olive oil was in lower levels than of Wettagen olive oil.

Table 2. Bitterness, FFA, PV and UV absorption of Egyptian olive oils during ripening*

	Harvest date*					
	August	**September**	**October**	**November**	**December**	**January**
Bittereness "Absorbance at 225 nm"						
Maraky	0.61 ± 0.1	0.64 ± 0.2	0.46 ± 0.1	0.23 ± 0.1	0.17 ± 0.0	0.15 ± 0.0
Wettagen	0.60 ± 0.1	0.62 ± 0.1	0.43 ± 0.1	0.21 ± 0.1	0.18 ± 0.0	0.14 ± 0.0
Organoleptic score						
Maraky	4.7 ± 0.4	4.9 ± 0.2	3.9 ± 0.2	2.8 ± 0.2	2.1 ± 0.2	1.6 ± 0.2
Wettagen	4.6 ± 0.3	4.8 ± 0.2	3.7 ± 0.3	2.5 ± 0.2	1.7 ± 0.2	1.3 ± 0.2
Free fatty acids (% oleic acid)						
Maraky	0.2 ± 0.0	0.2 ± 0.0	0.4 ± 0.1	0.5 ± 0.1	0.8 ± 0.1	1.8 ± 0.4
Wettagen	0.5 ± 0.0	0.5 ± 0.1	0.6 ± 0.1	0.7 ± 0.1	1.2 ± 0.2	2.2 ± 0.4
Peroxide value (meq.O_2/kg oil)						
Maraky	3.1 ± 0.3	3.1 ± 0.4	3.5 ± 0.4	3.9 ± 0.4	5.3 ± 0.3	7.9 ± 0.8
Wettagen	2.7 ± 0.3	2.8 ± 0.3	3.3 ± 0.3	3.8 ± 0.4	5.9 ± 0.4	8.9 ± 0.8
UV absorption (at 232 nm)						
Maraky	0.4 ± 0.1	0.4 ± 0.1	0.6 ± 0.1	0.8 ± 0.2	0.9 ± 0.2	1.2 ± 0.2
Wettagen	0.3 ± 0.1	0.4 ± 0.1	0.4 ± 0.1	0.7 ± 0.1	0.8 ± 0.2	1.3 ± 0.2
UV absorption (at 270 nm)						
Maraky	0.05 ± 0.01	0.06 ± 0.01	0.06 ± 0.01	0.07 ± 0.02	0.08 ± 0.02	0.10 ± 0.02
Wettagen	0.04 ± 0.01	0.04 ± 0.01	0.05 ± 0.01	0.06 ± 0.01	0.06 ± 0.01	0.07 ± 0.02

*Average value of three determinations ± SD

The FFAs were generally low in immature fruits. An increase was observed in FFA as ripening progressed. During over-ripening (December and January), FFA in Wettagen oil had exceeded the IOOC standard of 0.8% as oleic acid, while the fruits were still on the trees. Maraky variety had also increased in FFA towards late maturity but not to the same extent as Wettagen variety. The maximum levels of FFAs were 2.2 and 1.8 as % oleic acid in Wettagen oil and in Maraky oil, respectively in January. It is known that the increase of free acidity is mainly due to enzyme activity caused by olive tissue damage (Boskou, 1996, Salvador *et al*., 2001).

Peroxide values, expressed as meq.O_2 per kg oil, were less than 20 in extracted oil of the two varieties during ripening. There was no big difference in peroxide values in extracted oils of the two varieties. Different research works indicated that peroxide value was influenced by year more than by time of harvest (Mailer *et al*., 2005).

Spectrophotometric absorption at 232 and 270 nm were measured in olive oil samples during ripening. The UV characteristics confirmed the results of peroxide values. Again, there was no big difference in UV absorption values in extracted oils of the two varieties. These results of FFA, PV and UV absorption as olive oil quality parameters are in agreement with Salvador *et al*. (2001) and Pardo *et al*.(2007).

Fatty acid profile

The fatty acid profile is an important factor in consideration of oil quality. Olive oil is considered to be highly nutritional oil due in part to the high level of monounsaturated oleic acid (Duran, 1990, Abdalla, 2007).

Fatty acid composition of extracted olive oils from the two varieties under study during harvesting times of September, November and January are shown in Table (3). Fatty acid analysis at various stages of maturity indicated that individual fatty acids vary considerably in proportion during fruit development. This is of significant importance to olive oil producers in selecting oil with good stability and a superior nutritional fatty acid profile. Numerous fatty acids were present in the examined olive oils, palmitic, stearic, oleic and linoleic were measured as major fatty acids. Myristic, palmitoleic, linolenic and arachidic were also present in small amounts in all intervals. Palmitic acid ($C_{16:0}$) decreased in proportion to the other fatty acids over

Table 3: Fatty acid composition of Egyptian olive oils during ripening*

Fatty acids (%)	Olive variety / Harvest date					
	Maraky			Wettagen		
	September	November	January	September	November	January
Myristic $C_{14:0}$	0.3 ± 0.1	0.3 ± 0.1	0.2 ± 0.1	0.6 ± 0.1	0.5 ± 0.1	0.3 ± 0.1
Palmitic $C_{16:0}$	20.2 ± 1.8	15.5 ± 1.1	13.3 ± 0.6	17.1 ± 1.2	14.8 ± 1.1	12.7 ± 0.6
Palmitoleic $C_{16:1}$	1.6 ± 0.2	1.1 ± 0.1	0.7 ± 0.1	1.8 ± 0.2	1.5 ± 0.1	0.6 ± 0.1
Strearic $C_{18:0}$	4.5 ± 0.3	6.9 ± 0.3	8.3 ± 0.2	6.6 ± 0.2	6.7 ± 0.4	8.6 ± 0.3
Oleic $C_{18:1}$	51.6 ± 3.5	46.4 ± 2.3	47.3 ± 3.5	44.9 ± 3.4	38.8 ± 2.1	40.3 ± 3.1
Linoleic $C_{18:2}$	19.6 ± 1.1	27.5 ± 2.1	28.8 ± 2.2	25.2 ± 2.1	34.3 ± 2.1	36.1 ± 2.2
Linolenic $C_{18:3}$	1.3 ± 0.2	1.5 ± 0.2	0.9 ± 0.1	2.9 ± 0.2	2.6 ± 0.2	0.9 ± 0.1
Arachidic $C_{20:0}$	0.9 ± 0.1	0.8 ± 0.1	0.5 ± 0.1	0.9 ± 0.1	0.8 ± 0.1	0.5 ± 0.1
Saturated	25.9 ± 2.4	23.5 ± 2.1	22.3 ± 2.4	25.2 ± 2.4	22.8 ± 2.1	22.1 ± 1.4
Mono-unsaturated	53.2 ± 4.5	47.5 ± 2.7	48.0 ± 2.9	46.7 ± 3.5	40.3 ± 2.7	40.9 ± 2.9
Poly-unsaturated	20.9 ± 1.2	29.0 ± 1.3	29.7 ± 1.1	28.2 ± 1.2	36.9 ± 1.3	37.0 ± 1.1
Unsaturated/Saturated	2.9 ± 0.2	3.2 ± 0.3	3.5 ± 0.3	3.0 ± 0.2	3.4 ± 0.2	3.6 ± 0.4
Mono-unsaturated/ Poly-unsaturated	2.55 ± 0.4	1.64 ± 0.3	1.62 ± 0.3	1.66 ± 0.4	1.10 ± 0.3	1.11 ± 0.4

*Average value of three determinations ± SD

the time from around 20 to about 13 % in Maraky and from 17% to about 13% in Wettagen variety during ripening period from September up to January. Even at these levels, olive oil contains relatively high levels of saturated oil as compared to seed oils. This is important parameter to consider in future research and possible selection of cultivars of olives with a desirable level of saturated oil. Maraky olive oil consistently had a slightly higher level of palmitic acid than Wettagen olive oil. Moreover, stearic acid ($C_{18:0}$) was similar in the two cultivars and relatively increased during ripening from about 5 to 9% of total fatty acids. Oleic acid ($C_{18:1}$) was found be slightly change during ripening. Maraky olive oil contains high level of oleic being 51.6% in September then decreased to 46.4% by November and slightly increased to 47.3% by January while Wettagen olive oil contains low level of oleic being 44.9% in September then decreased to 38.8% by November and increased to 40.3% by January.

Australian oils as well as Siwa's Oasis olive oils vary considerably in their fatty acid profile and sometimes do not meet the expectations of high oleic oil. This can be attributed to that olives growing in Siwa is considered as a moderately salt tolerant plant. Chartzoulakis (2003) found that fatty acid composition of olive oil was affected by salinity which resulted in increase of palmitic and total saturated fatty acids on the contrary to oleic acid that decreased by increasing the salt concentration in irrigation water.

Linoleic acid ($C_{18:2}$) increased gradually during ripening and the oil becoming more poly-unsaturated with time. It reached the maximum level of 28.8% and 36.1% in January in Maraky and Wettagen, respectively. Moreover, the results indicated that unsaturated/saturated ratio increased while mono-unsaturated to poly-unsaturated ratio decreased during ripening. Shelf life and oil stability are closely related to the degree of saturation and polyunsaturation of oils.

Olive oil has to conform to international fatty acid standards and Egyptian olive oil sometimes is outside of those requirements. In particular, linolenic acid ($C_{18:3}$) sometimes exceeds the maximum standard of 1.0%. This can occur more often in immature oil as the linolenic acid level decreases as the fruit matures. Linolenic acid was found to be very high early in the season but decreased to acceptable levels (IOOC Standard <1.0%) by November and January.

Rana & Ahmed (1981) indicated that Libyan olive oil contained oleic acid (43-46%) lower than that reported in literature from other areas and had higher levels for linoleic (29-32%) and palmitic (17-18%). These results are in agreement with Egyptian olive oil fatty acids of the two varie-

ties grown in Siwa where both Libyan and Egyptian olive varieties have similar high salinity and environmental conditions. Chartzoulakis (2005) found that salinity reduced the fruit weight and oil content while increased the moisture content of olive fruits. He found that the ratio of unsaturated to saturated fatty acids decreased, due to salinity. Moreover, Shahat & El-Sayed (2003) found that the percentage of oleic acid in oil of Wettagen variety was lower than that reported from other areas and higher in linoleic and palmitic acids. These results are in agreement with the results of Wettagen variety in the present study. Rotondi *et al*. (2004) found that the fatty acid composition of olive oil is important parameter in the length of shelf life that is quantitatively affected by two main factors; the olive variety used in the production of the oil and the ripening stage at which the olives are harvested. Changes observed from first harvest to last harvest in the oleic/linoleic acid ratio showed decreasing trend during ripening.

Pigments and tocopherols

The pigments that colour olive oil are chlorophylls and carotenoids (Minguez-Mosquero *et al*., 1990 & 1991). While measurement of chlorophyll concentration in olive oil is not required by IOOC, it is important for a number of seasons. Chlorophyll is implicated in autoxidation and photooxidation mechanisms, therefore affecting the oxidation stability of the oil. Colour is also an important attribute to consumers, who associate the green hues from the chlorophyll in the oil with freshness of product (Ryan *et al*., 1998).

In the present study, total chlorophylls and β-carotene were measured in olive oils during harvesting in September, November and January (Table 4). Chlorophyll content, as expected, was high in the immature olives and rapidly decreased with time as the colour changed from green to black. The two cultivars showed a decrease in chlorophyll during ripening period from September up to January.

The chlorophyll content of olive oil has been shown to decrease markedly during ripening (Gutierrez *et al*., 1999, Roca & Minguez-Mosquera, 2001). Some researchers found that the concentration of chlorophylls was 80 mg/kg oil in very early ripening period, falling to about 2 mg/kg oil when the fruit is very ripe (Salvador *et al*., 2001). Moreover, Morello *et al*.(2004), Basuny & Mostafa (2004) found that chlorophylls decreased rapidly during ripening and carotenoid content followed a similar trend to that of chlorophylls but the percent loss was lower.

Maraky olive oil contained higher amount of total tocopherols than Wettagen olive oil (Table 4). α- tocopherol was the most abundant component. Both cultivars showed a trend of reduction in tocopherol content as the fruit matured. Maraky olive oil reached the highest level of total tocopherols being 218.2 mg/kg oil in September and decreased to 151.4 mg/kg oil by January, while Wattegan olive oil had low level of 168.3 mg/kg oil then decreased to 118.1 mg/kg oil in January. The tocopherol content is highly variety-dependent with concentration ranging from 5 to 300 ppm. Usual values reported for good quality oils vary between 100 and 300 ppm (Angerosa & Di Giovacchino, 1996, Salvador

Table 4: Pigments and tocopherols of Egyptian olive oils during ripening*

	Olive variety / Harvest date					
	Maraky			Wettagen		
	September	November	January	September	November	January
Pigments(mg/kgoil)						
Total chlorophylls	11.5±1.5	6.5±0.5	2.8±0.2	11.1±0.8	5.7±0.4	1.8±0.3
β- Carotene	4.9±0.4	3.8±0.4	2.6±0.2	3.6±0.3	2.6±0.2	1.4±0.2
Tocopherols (mg/kg oil)						
α- Tocopherol	181.2±11.6	173.5±8.6	133.2±6.8	142.5±6.3	134.0±4.9	105.4±6.8
β- Tocopherol	2.4±0.2	2.1±0.2	1.8±0.3	3.1±0.2	2.6±0.2	1.8±0.2
δ- Tocopherol	1.5±0.2	1.3±0.2	1.1±0.2	1.1±0.2	0.8±0.2	0.6±0.2
γ- Tocopherol	33.1±5.2	19.2±1.1	15.3±1.1	21.6±0.8	15.7±1.1	10.3±0.5
Total Tocopherols	218.2±13.6	196.1±7.8	151.4±8.8	168.3±5.8	153.1±7.4	118.1±5.6

*Average value of three determinations ± SD

et al., 2001). Basuny & Mostafa (2004) found that the range of α-tocopherol contents in virgin olive oil of the three varieties (Picual, Arbequine and Koronakii) varied between 153 to 289 ppm. There were differences in the α-tocopherol content during the harvest periods. Oils from December harvest contained lower α-tocopherol than oils extracted during October and November of the three varieties under study.

Polyphenols and phenolic compounds

Polyphenols are perhaps the most important of the minor components in olive oil due to its powerful antioxidant effect and the resulting contribution to shelf life stability. Young/unripe olives are typically high in polyphenol content and this component has been shown to relate closely to oil stability (Mailer & May, 2002). The polyphenols also have been shown to be closely associated to the organoleptic characteristics of the oil, being largely responsible for pungency and bitterness attributes. Sensory analysis has shown that oils extracted from immature fruits are generally more pungent and bitter than oil from mature fruits and again this is related to the polyphenol content (Table 2).

In the present study, total polyphenols were determined in the two cultivars during different ripening period and phenolic compounds were identified using HPLC. The results in Table (5) indicate that Maraky cultivar had considerably higher levels of polyphenols than Wettagen cultivar. The fruits of the two cultivars had very high levels at early maturity which decreased as the fruits matured. Maraky cultivar had high level of polyphenols in September being 535.1 mg/kg oil which decreased to 338.2 mg/kg oil in January. Wettagen cultivar had low level of polyphenols being 422.2 mg/kg oil in September which decreased to 226.2 mg/kg oil in January.

It is often suggested that polyphenol levels above 200 mg/kg oil are necessary to produce good quality of virgin olive oil. Levels above 400 mg/kg oil may be too high for the average consumer, producing pungent oil (Tovar *et al*., 2001). From the data in Table (5), it can be seen that polyphenol levels in Maraky olive oil are above 400 mg/kg oil until early December. The two cultivars maintained reasonable levels of polyphenol on average, even into late maturity. This is not always the case with some Australian olive oil producing very low polyphenol levels, below 100 mg/kg oil (Mailer, 2004).

Oleuropein aglycon, ligstroside aglycone, tyrosol, hydroxytyrosol and pinoresinol were the most abundant phenolic compounds in the two varieties under study (Table 5). Maraky variety contained the highest amounts of all these phenolic compounds. All phenolic compounds except tyrosol and coumaric decreased during ripening, oleuropein aglycon and ligstroside aglycon were

Table 5: Total polyphenols and phenolic compounds of Egyptian olive oils during ripening*

	Olive variety / Harvest date					
	Maraky			Wettagen		
	September	November	January	September	November	January
Total polyphenols (mg/kg oil)	535.1 ± 16.5	455.2 ± 12.3	338.2 ± 11.6	422.2 ± 12.5	328.5 ± 12.3	226.2 ± 11.4
Phenolic compounds (mg/kg oil)						
Hydroxytyrosol	41.2 ± 5.5	35.3 ± 2.6	26.3 ± 2.2	29.3 ± 1.6	21.3 ± 0.9	10.1 ± 0.2
Tyrosol	47.3 ± 3.5	57.5 ± 3.6	55.6 ± 2.8	51.4 ± 2.2	47.6 ± 1.5	47.4 ± 2.1
Vanillic acid	14.1 ± 1.2	8.2 ± 0.5	ND	11.5 ± 0.4	5.1 ± 0.2	ND
p-Coumaric acid	17.2 ± 1.3	26.1 ± 1.4	25.2 ± 1.6	ND	ND	ND
Caffeic acid	22.3 ± 1.5	17.6 ± 0.8	8.5 ± 0.3	13.6 ± 0.6	7.3 ± 0.2	3.1 ± 0.2
Ferulic acid	27.5 ± 1.4	29.4 ± 1.2	21.5 ± 0.6	ND	ND	ND
Pinoresinol	43.5 ± 1.8	27.5 ± 1.2	21.4 ± 0.7	45.5 ± 2.1	42.4 ± 1.8	28.6 ± 0.8
Oleuropein aglycon	149.4 ± 8.8	96.6 ± 4.8	63.7 ± 2.5	133.2 ± 5.5	85.6 ± 4.8	43.4 ± 1.1
Ligstroside aglycon	102.7 ± 6.6	65.3 ± 4.2	41.2 ± 1.6	88.7 ± 3.8	61.4 ± 4.2	39.5 ± 0.8
Elenoic acid	28.5 ± 1.5	33.1 ± 1.1	24.1 ± 0.8	24.3 ± 1.1	18.2 ± 0.6	19.3 ± 0.6

*Average value of three determinations ± SD ND is not detected.

dramatically decreased during ripening in the two cultivars. P-Coumaric and ferulic acids were found in Maraky olive oil and were not detected in Wettagen olive oil. These two phenolic compounds were found in levels of 17 and 27 mg/kg oil, respectively, during harvesting in September and increased during harvesting in November to be 26 and 29 mg/kg oil, respectively.

Ryan *et al*. (1999) found that oleuropein was the principal phenolic compound in olive and its concentration decreased during fruit development. Badawy & Bassiuny (2001) concluded that phenolic compounds have been affected by stage of ripening and most of compounds decreased during ripening of olive fruits. Caponio *et al*. (2001) concluded that oleuropein and its aglycon form were especially responsible, among the phenolic compounds, for the bitterness of virgin olive oils; both decreased as ripening of the olives progressed. Basuny & Mostafa (2004) found that the amount of phenolic compounds in virgin olive oil was important factor when evaluating its quality. The total polyphenols in the virgin olive oil of three varieties (Picual, Arbequine and Koronakii) during olive ripening showed higher values in the first period and their level usually decreased with maturation. Understanding the levels of polyphenols and active phenolic compounds and their powerfull antioxidative effects allow growers to exert control over the polyphenol levels in their olive oil.

Sterols and squalene

Unsaponifiable matter decreased in Maraky olive oil from 2014 to 1509 mg/kg oil and from 2168 to 1527 mg/kg oil in Wettagen olive oil during harvesting from September up to January. Unsaponifiable matter in the two olive oils was in higher amount than other reported olive oils (Paganuzzi & Leoni, 1979, Rana & Ahmed, 1981, Atta & Ahmed, 2005).

Sterols are important constituents of olive oils because they relate to the quality of the oil and are widely used to check authenticity. The amount of total sterols and identification of sterol composition in the two olive varieties are shown in Table (6). Sterol contents were dramatically decreased during ripening in all samples. β-sitosterol was the predominant sterol followed by Δ^5-avenasterol in both Maraky and Wettagen varieties.

The amounts of sterols found in two Iranian virgin olive oils were 1040 and 1110 mg/kg oil and the predominating sterol was β-sitosterol (Paganuzzi & Leoni, 1979). The effect of sterols on the oxidation of a triglyceride mixture, similar in composition to olive oil, had been studied at 180 °C (Gordon & Magos, 1983). The Δ^5-Avenasterol and fucosterol were effective as antioxidants, whilst other sterols, including cholesterol and stigmasterol, were ineffective. The antioxidant effect of Δ5-avenasterol increased with concentration in range 0.01 to 0.1%. Moreover, Gordon & Magos (1984) and Salvador *et al*. (2001) published that Δ5-avenasterol occurs together with other sterols in many edible oils including olive oil. It is a particularly important unsaponifiable component because, unlike most other sterols, it acts as an antioxidant at elevated temperatures.

The content of β-sitosterol generally decreases during ripening, while Δ^5-avenasterol increases (Salvador *et al*., 2001). These results are in agreement with the results in the present study. The composition and antioxidant activity of total sterols in extra virgin olive oils with different extraction technologies from olives harvested at two ripening stages were studied by Cercaci *et al*. (2007). No significant differences were observed in the percent composition of sterols of extra virgin olive oils produced with different technologies during the same harvesting period. The latter, however, had a significant effect on the percent of β-sitosterol and Δ5-avenasterol in extra virgin olive oils produced with the same technology.

Squalene is the most abundant hydrocarbon in virgin olive oil and it is present in variable quantities ranging from 100 to 1200 mg/100 g oil (DeLeonardis *et al*., 1998), and varied considerably in relation to cultivar and olives ripeness. In the present study, Maraky and Wettagen olive oils contained relatively similar amount of squalene (Table 6). Squalene is regarded as partially responsible for the beneficial effects of olive oil against certain cancers (Smith *et al*., 1998, Abdalla, 2007). Very little is known for the contribution of squalene to the oxidative stability of olive oil or other edible oils and fats. The effect of squalene on the heat stability of rapeseed oil and model lipids was studied by Malecka (1991). She concluded that the addition of squalene in the amount of 0.4 % improved the stability of rapeseed oil and model lipids during heating at 170 C. On the other hand, the role of squelene in olive oil stability was studied by Psomiadou & Tsimidou (1999). They concluded that the weak

Table 6. Sterols and squalene of Egyptian olive oils during ripening*

	Olive variety /Harvest date					
	Maraky			Wettagen		
	September	November	January	September	November	January
Sterols (mg/kgoil)						
Cholesterol	2.6±0.5	ND	ND	5.6±0.6	ND	ND
Campesterol	51.2±5.5	52.8±3.3	62.3±6.2	91.6±10.6	81.1±7.9	69.5±5.2
Stigmasterol	9.7±1.2	5.1±0.2	ND	6.9±0.2	ND	ND
β- Sitosterol	932.9±26.2	796.7±26.5	780.4±24.9	1047.9±30.4	866.0±22.2	742.5±20.9
Sitosterol	18.3±1.3	17.3±1.3	12.7±1.6	16.7±0.9	10.32±0.9	6.6±0.9
Δ^5-Avenasterol	185.2±11.7	143.1±8.5	133.5±9.3	215.1±12.6	167.6±14.2	123.4±11.2
Δ^7-Avenasterol	18.1±1.2	ND	ND	4.2±0.9	ND	ND
Total Sterols	1218±31.5	1015±41.1	989±30.5	1388±32.8	1125±31.2	942±21.6
Squalene (mg/ kg oil)	538±18.5	511±24.1	339±12.9	572±25.6	518±24.5	417±21.4

*Average value of three determinations ± SD ND is not detected.

antioxidant activity of squalene in olive oil may be explained by competitive oxidation of the different lipids present which leads to a reduction of the oxidation rate. Squalene plays a rather confined role in olive oil stability even at low temperatures.

CONCLUSSION

The effect of harvest time on the compositional quality of extracted oils from Maraky and Wettagen cultivars of olive grown in Siwa's Oasis was investigated. Therefore, the minor components as pigments, tocopherols, polyphenols, sterols and squalene which play an important role in olive oil quality were evaluated during ripening of olive fruits.

The results indicated that the amount of oil increased in the two cultivars of olive during ripening until November and then decreased. Bitterness, pigments, tocopherols, total polyphenols and phenolic compounds, sterols and squalene were in the highest levels in olive oils extracted from unripe fruits harvested in September and were in the lowest levels in overripe fruits harvested in January. Moderate levels of most quality parameters were found in olive fruits harvested in November. It could be recommended that November month is the most proper time to harvest both Maraky and Wettagen cultivars of olive grown in Siwa's Oasis.

Although more research works are needed, the quality parameters investigated in this research work will assist olive growers to determine optimum harvest time for optimal olive oil quality.

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دراسة تأثير وقت الحصاد على جودة زيت الزيتون المصرى

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قد يكون لوقت الحصاد تأثير كبير على إنتاج وجودة وثبات زيت الزيتون. وهناك القليل من الإرشادات والتعليمات للمنتجين لتقدير أفضل وأمثل موعد للحصاد. لذا فإن هذا البحث يهدف الى دراسة تأثير موعد الحصاد على الجودة التركيبية للزيت المستخلص بالعصر الهيدروليكى من صنفى مراقى ووطجن المنزرعين فى واحة سيوة. وقد تم تقدير بعض الخواص الطبيعية للثمار ونسبة الرطوبة ومحتوى الزيت كما تم تقدير عدة عوامل لجودة الزيت وذلك خلال مراحل نضج الثمار من أغسطس الى يناير خلال موسم ٢٠٠٥/٢٠٠٦ وقد شملت عوامل جودة الزيت كلاً من الأحماض الدهنية الحرة ورقم البيروكسيد والامتصاص على أطوال موجات ٢٣٢ ، ٢٧٠ نانوميتر و تركيب الأحماض الدهنية و الصبغات (الكلوروفيل و الكاروتين) والتوكوفيرولات و المركبات الفينولية والإستيرولات والإسكوالين.

وأوضحت النتائج أن كمية الزيت المتحصل عليها من كل صنف كانت منخفضة جداً فى الموعد المبكر من الحصاد (أغسطس وسبتمبر) وهو مايسمى بمرحلة عدم تمام النضج وكذا الموعد المتأخر (ديسمبر ويناير) وهو مايسمى بمرحلة فوق النضج. وكانت أعلى نسبة زيت متحصل عليها خلال شهر نوفمبر لكلا الصنفين.

كان مستوى الأحماض الدهنية الحرة منخفضاً فى زيت الزيتون الذى تم حصاده مبكراً وكان عند أعلى مستوى وأعلى من الموصى به من قبل المركز الدولى لزيت الزيتون وذلك خلال مرحلة فوق النضج. وكان رقم البيروكسيد فى الحدود الموصى بها عالمياً.

وأوضحت نتائج الأحماض الدهنية إنخفاض محتوى كل من الأوليك والبالمتيك وإرتفاع محتوى حمض اللينولييك فى الزيت خلال مراحل نضج الثمار. وكانت نسبة حمض الأوليك فى زيت الزيتون لكلا الصنفين أقل فى حين كانت نسب اللينولييك والبالمتيك أعلى عن التى تم نشرها فى البحوث الأخرى من مناطق اخرى مختلفة.

كما أوضحت النتائج أن الصبغات والتوكوفيرولات والمركبات الفينولية والإستيرولات والإسكوالين كانت بأعلى نسب فى مرحلة عدم تمام النضج للثمار وبأقل نسب فى مرحلة فوق النضج.

توضح هذة الدراسة مايتم من تغيرات طبيعية وكيماوية لثمار وزيت الزيتون خلال مراحل النضج المختلفة. ومن نتائج هذة الدراسة يمكن الأخذ فى الإعتبار عدة محددات وعوامل هامة تساعد منتجى الزيتون على تقدير أمثل موعد للحصاد للحصول على أقصى وأفضل جودة لزيت الزيتون. وبجانب العوامل القياسية التى وضعها المركز الدولى لزيت الزيتون فإن هذة المحددات تشمل المركبات المتواجدة بنسبة منخفضة مثل الصبغات والتوكوفيرولات والفينولات والإستيرولات والتى تلعب دوراً كبيراً فى جودة زيت الزيتون.