

## **RESULTS AND DISCUSSION**

Virgin olive oil has a characteristic aroma, taste and colour that distinguishes it from other vegetable oils. Its excellent organoleptic and nutritive qualities (**Viola, 1969**), together with the current tendency of consumers to select the least-processed foods, have caused a re-evaluation of its consumption. It is now often favoured over other fats that have more complex processing steps. Such as decolouring, deodorizing and refining (**Bagordo, 1988**). However, it is a matter of concern for the oil industry to conserve the oil without loss of its positive attributes or deterioration of its quality. Various factors, such as air, heat and light, act as synergistic in the autooxidation of oil by producing hydroperoxides that can seriously and rapidly diminish the original characteristics (**Min and Smouse, 1985**). Hydroperoxides are formed by the action of oxygen on unsaturated fatty acids through free-radical reactions that continue if there are no antioxidants capable of stopping them (**Carlsson *et al.*, 1976**).

Pressed olive oils obtained from the fruits of *olea europaea* L. are known to be more resistant to oxidation than other edible oils because of their lower unsaturation and their unsaponifiable components, including tocopherols and phenolic compounds (**Perrin, 1992**). Seed oils contain more tocopherols than olive oils, but great amounts of phenolics are lost during oil processing (**Forcadell *et al.*, 1987**).

Since virgin olive oils are not refined, the phenolic compounds are partly preserved, and these compounds are reportedly responsible for their higher stability to autoxidation (**Nergiz and Unal, 1991**).

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The extent of oxidation in oils has been frequently evaluated by measuring peroxide value (PV). This index is related to the hydroperoxides, the primary oxidation products, which are unstable and readily decompose to form mainly mixtures of volatile aldehydic compounds. Because these compounds are directly responsible for rancid flavours (**Frankel, 1982**), they are considered important markers of oxidative rancidity.

### **1-Physical and chemical properties of Shemllali, Weteken, and Maraki. olive oils**

The color, refractive index, viscosity, acide value, Free fatty acids (% as oleic acid), peroxide value, iodine value, UV absorbance at 232 and 270 nm, TBA value, oxidative stability in hours, saponification value, unsaponifiable matter percent were determined for the investigated virgen olive oils, in addition the sensory properties also were measured- the obtained results are shown in Table (14, 15, 16) and graphically in Figs. (1,2,3).

**Table (12): Physical and chemical properties of fresh Shemllali olive oil.**

Parameter	Value
Color “5.25”: yellow	35
Red	2.6
Blue	0.8
Refractive index (at 25° C)	1.4679
Viscosity (cp) at 20° C	82.3
Acid Value	0.28
Free Fatty acids (% as oleic acid)	0.14
Peroxide value (meq/kg oil)	2.31
Iodine value (Hanus)	83.38
UV absorbance ( $E_{1\text{cm}}^{1\%}$ ) at 232nm.	1.92
UV absorbance ( $E_{1\text{cm}}^{1\%}$ ) at 270 nm.	0.07
T.BA value (as absorbance at 535 nm)	0.023
Stability in hours	25.2
Saponification value.	196.57
Unsaponifiable matters	0.73
* Sensory evaluation score	5
Total phenols content	204 ppm

**Table (13): Physical and chemical properties of fresh Weteken olive oil.**

Parameter	Value
Color “5.25”: yellow	35
Red	2.5
Blue	1.2
Refractive index (at 25° C)	1.4681
Viscosity (cp) at 20 °C	82.6
Acid Value	0.23
Free Fatty acids (% as oleic acid)	0.11
Peroxide value (meq/kg oil)	1.93
Iodine value (Hanus)	80.71
UV absorbance (E1cm <sup>1%</sup> ) at 232nm.	1.78
UV absorbance (E1cm <sup>1%</sup> ) at 270 nm.	0.03
T.BA value (as absorbance at 535 nm)	Zero
Stability in hours	28.6
Saponification value.	193.40
Unsaponifiable matter	0.95
* Sensory evaluation score	5
Total phenols content	258 ppm

**Table (14): Physical and chemical properties of fresh Maraki olive oil.**

Parameter	Value
Color “5.25”: yellow	35
Red	2.5
Blue	0.6
Refractive index (at 25° C)	1.4677
Viscosity (cp) at 20° C	80.2
Acid Value	0.22
Free Fatty acids (% as oleic acid)	0.11
Peroxide value (meq/kg oil)	1.15
Iodine value (Hanus)	85.08
UV absorbance (E1cm <sup>1%</sup> ) at 232nm.	1.81
UV absorbance (E1cm <sup>1%</sup> ) at 270 nm.	0.02
T.BA value (as absorbance at 535 nm)	Zero
Stability in hours	30.7
Saponification value.	191.93
Unsaponifiable matter	1.37
* Sensory evaluation score	5
Total phenols content	308ppm

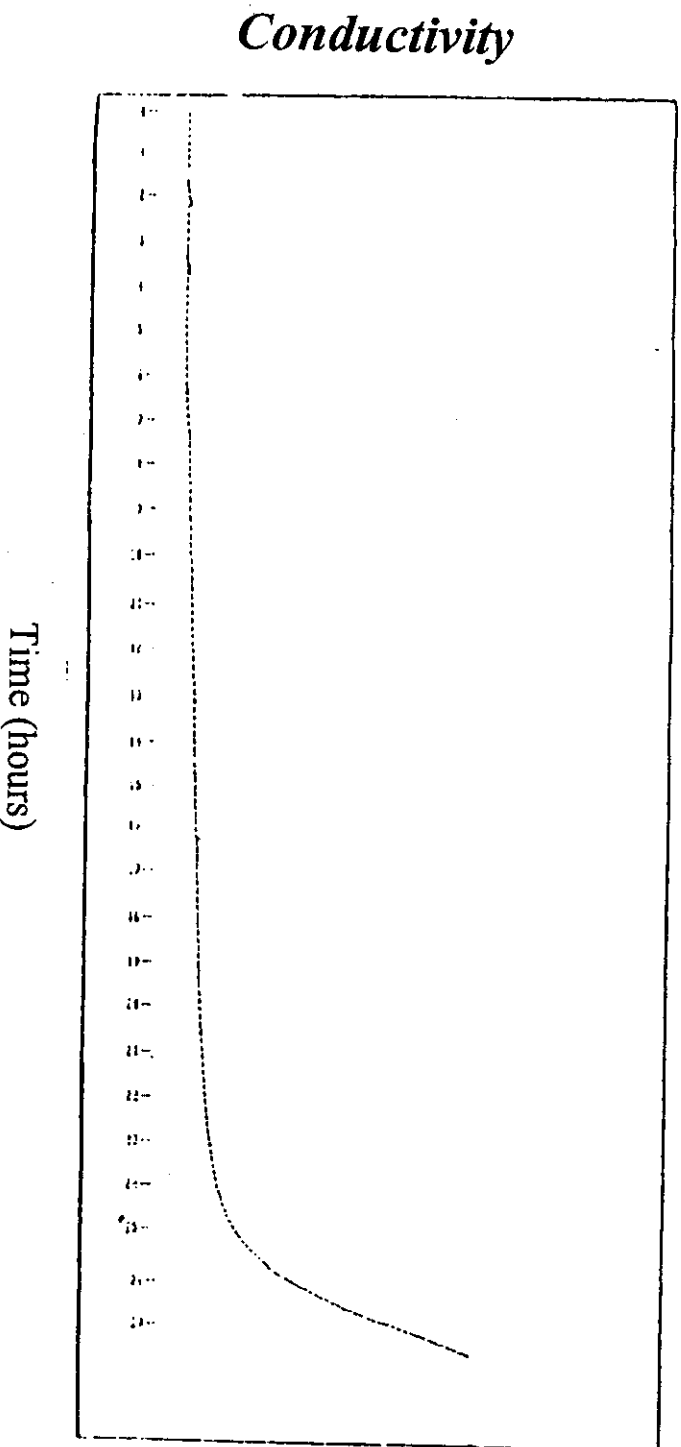


Fig (1): Oxidative stability of fresh Shemlali olive oil by rancenate.

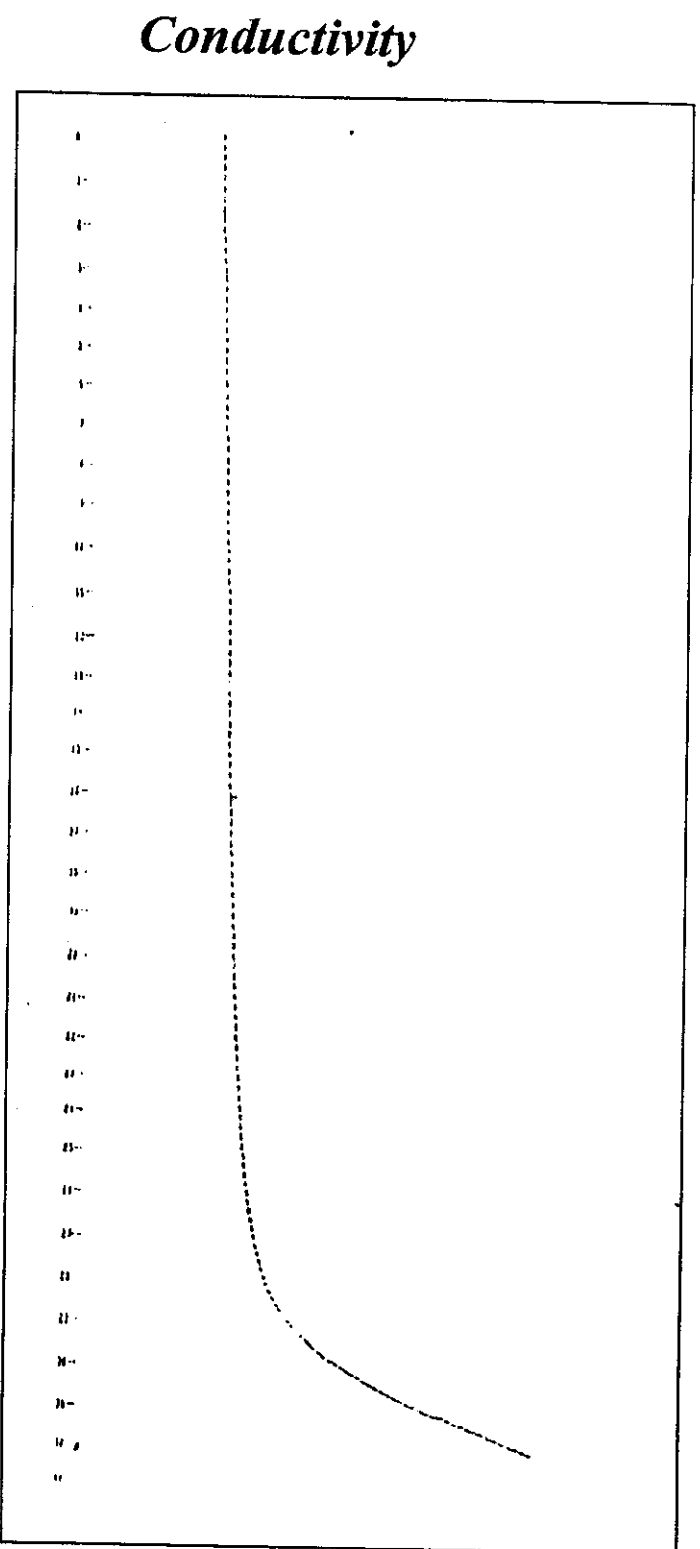
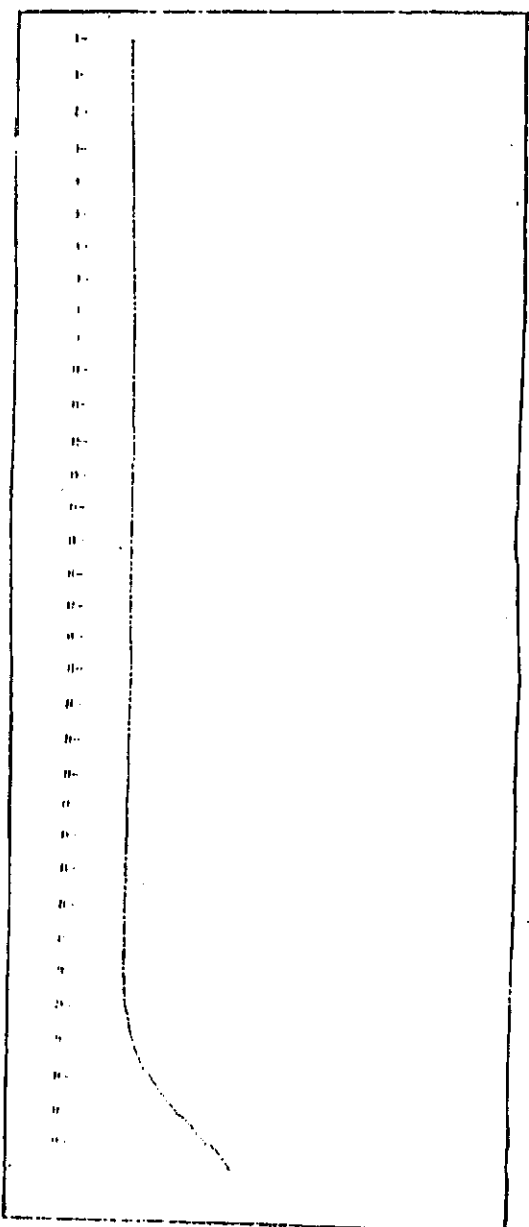


Fig (2): Oxidative stability of fresh Weteken olive oil by rancenate method

## Conductivity



Time (hours)

Fig (3): Oxidative stability of fresh Maraki olive oil by rancemate method



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The physical examination of oils includes several analyses of which the most important ones are the determination of color test. The color of olive oil (on the Lovibond scale) is considered of commercial importance for edible purpose. The color of the olive oil extracted from Shemlali, Weteken and Maraki was yellow (35), red (2.6) and blue (0.8) of Shemlali, yellow (35), red (2.5) and blue (1.2) of Weteken, yellow (35), red (2.5) and blue (0.6) of Maraki respectively.

Refractive index is one of the important physical methods of analysis of oils, it is used basically for the estimation of the degree of unsaturation, as well as its correlation with the iodine value. The refractive index values at 25° C of the olive oil extracted from Shemlali, Weteken and Maraki were (1.4679, 1.4681 and 1.4677) respectively.

The viscosity of oils under investigation were found to be (82.3, 82.6 and 80.2 CP at 20° C) respectively. The values of these physical properties are within the limits of *Egyptian Standard for Olive Oil (1993)*, *IOOC (1996)* and *Mousa et al., (1996)*.

The acid value is a measure of the amount of free fatty acids present in the oil due to hydrolysis of its triglycerides. The data shown in tables (14, 15 and 16) indicate. That the acid values of Shemlali, Weteken and Maraki olive oils were (0.28, 0.23 and 0.22) respectively. In the other hand the free fatty acids (% as oleic acid) in oils under investigation were found to be (0.14, 0.11 and 0.11%) respectively. The low free fatty acids content indicates no hydrolysis in the oils had taken place. *According to IOOC (1996)* these oils could be classified as extra virgin olive oil.

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Results from the same Tables (12, 13 and 14) indicate that the peroxide values for fresh Shemllali, Weteken and Maraki olive oils were (2.31, 1.93 and 1.15 meq/kg oil) respectively. While *Egyptian Standard* for olive oil (1993) and *IOOC* (1996) allowed the peroxide value to be up to (20 meq/kg oil). The peroxide test gives a whole picture on the course of oil oxidation. In general, the peroxide values for fresh olive oils of Shemllali, Weteken and Maraki were within the permissible limits for human consumption.

The iodine value, which reflects the degree of unsaturation of an oil, was determined for Shemllali, Weteken and Maraki olive oils and the results are shown in Tables (12, 13, 14). The iodine values of the three olive oil cultivars (Shemllali, Weteken and Maraki) were (83.38, 80.71 and 85.08) respectively, which indicates that the oils belonged to the non drying oils. Category. This result is in accordance with *Egyptian standard* (1993), *IOOC* (1996) and *Ranalli et al* (1996).

Also, from the same tables, it could be noticed that the UV absorbences at 232, 270 nm were (1.92 and 0.07) of Shemllali, (1.78 and 0.03) of Weteken and (1.81 and 0.02) of Maraki respectively. Which indicate that oxidative rancidity was too low. It was stated in *IOOC*, (1996) that  $K_{270}$  (extra) has to be less than 0.25.

The thiobarbituric acid value is supposed to be a more sensitive test for detection of the oxidative rancidity of oils. Obtained results showed that the absorbance at 535 nm for virgin olive oils were (0.023, Zero and Zero) for Shemllali, Weteken and Maraki respectively which indicates the

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freshness of virgin olive oils under investigation .

Results from Tables (12,13 and 14) and Figs (1, 2 and 3) indicate that oxidative stability of Shemllali, Wetecken and Maraki, at 100° C using rancimate method, were (25.2), (28.6) and (30.7) hours respectively. These results are in agreement with that reported by **Hasenhettl and Wan (1992)** and **Larcker et al., (1994)**.

The saponification value is an index of the mean molecular weight of the fatty acid composition of the oil. The saponification values presented in Tables (12,13 and 14) indicated that no significant changes were taken place for the saponification values of Shemllali, Wetecken and Maraki olive oils.

The unsaponifiable matter contents of olive oils extracted from fresh Shemllali, Wetecken and Maraki fruits were (0.73, 0.95 and 1.37%), respectively. In other words Maraki fruits contained nearly 1.8 times unsaponifiables as that as Shemllali fruits and also it contained nearly 1.3 times unsaponifiables as that as Wetecken fruits. It is of interest to note that unsaponifiables are among the factors that can suppress the oil oxidative rancidity and the phenols content were (204ppm, 258ppm and 308ppm) respectively. This result is in good agreement with that of **Fedeli (1977)**, **Egyptian Standard (1993)** and **IOOC (1996)**.

From results in the same Tables (12,13 and 14) the sensory evaluation of fresh olive oils graded (5) and that classified the oils as high quality oils according to **IOOC (1979)**.

Finally, from the above results in Tables (12,13 and 14) and Figs. (1, 2 and 3), it could be concluded that the virgin olive oils under investigation were of high quality according to **Egyptian Standard (1993)** and **IOOC (1996)**.

## **Result & Discussion**

### **1- Fatty acids composition of olive oils extracted from fresh Shemllali, Weteken and Maraki fruits :**

The fatty acids are the integral constituents of every fat or oil. The degree of complexity of the glycerides basically depends upon the number and amount of various fatty acids in it. Also, the physical and chemical characteristics of lipids are largely depend upon their fatty acid composition. Hence, gas – liquid chromatography was used in this investigation for the qualitative and quantitative determination of individual fatty acid methyl esters ( $C_8$ -  $C_{20}$ ).

The results in Table (15) and figs (4, 5, 6 and 7) show the fatty acids composition of the olive oils samples under investigation. For simplicity, the fatty acids constituents of olive oils were divided in to three main groups, i.e., trace ( $<1\%$ ), minor ( $<10\%$ - $>1\%$ ) and major ( $>10\%$ ) components. Fresh olive oil of Shemllali cultivare contained 8: 0, 10:0, 12:0, 14:0, and 20:0 acids as trace amounts. The fatty acids 16:0, 18:0, 18:2, and 18:3 occurred as minor components where 16:0, 18:1 were present as major constituents. These fatty acids pattern were similar to that obtained with olive oil extracted from Weteken cultivare except for 18:3 where it present as trace substance. While fresh olive oil of Maraki cultivare contained oleic acid (18:1) present as a major unsaturated fatty acid since, it amounted to 81.86% meanwhile, palmitic acid (16:0) was present as a major saturated fatty acid as it reached 12.58%. On the other hand, linoleic acid (18:2) was found in a low content (3.95%). Also this fatty acids of Maraki cultivare were similar to that obtained with olive oil extracted from Shemllali cultivare.

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**Table (15): The fatty acids composition of olive oil samples obtained from Shemllali, Weteken and Maraki varieties.**

Fatty acids	(%) of Shemllali olive oil	(%) of Weteken olive oil	(%) of Maraki olive oil
8:0	0.09 <sup>a</sup>	0.11 <sup>a</sup>	0.06 <sup>a</sup>
10:0	0.19 <sup>a</sup>	0.5 <sup>a</sup>	0.32 <sup>a</sup>
12:0	0.06 <sup>a</sup>	0.11 <sup>a</sup>	0.08 <sup>a</sup>
14:0	0.57 <sup>a</sup>	0.39 <sup>a</sup>	0.73 <sup>a</sup>
16:0	19.49 <sup>a</sup>	21.74 <sup>a</sup>	12.58 <sup>a</sup>
16:1	1.50 <sup>a</sup>	2.6 <sup>a</sup>	2.73 <sup>a</sup>
18:0	2.75 <sup>a</sup>	2.95 <sup>a</sup>	0.74 <sup>a</sup>
18:1	70.86 <sup>a</sup>	67.93 <sup>a</sup>	81.86 <sup>a</sup>
18:2	2.31 <sup>a</sup>	2.88 <sup>a</sup>	3.95 <sup>a</sup>
18:3	1.32 <sup>a</sup>	0.66 <sup>a</sup>	1.93 <sup>a</sup>
20:0	0.86 <sup>a</sup>	0.58 <sup>a</sup>	0.89 <sup>a</sup>
T.S	24.01	25.93	15.4
T.U	75.99	74.07	90.47
TS/TU	0.32	0.35	0.17

Where 8.0: (Caprolic acid)

10.0: (Capric acid)

12.0: (Lauric acid)

14.0: (Myristic acid)

16.0: (Palmitic acid)

16.1: (palmitoleic acid)

18.0:( Stearic acid)

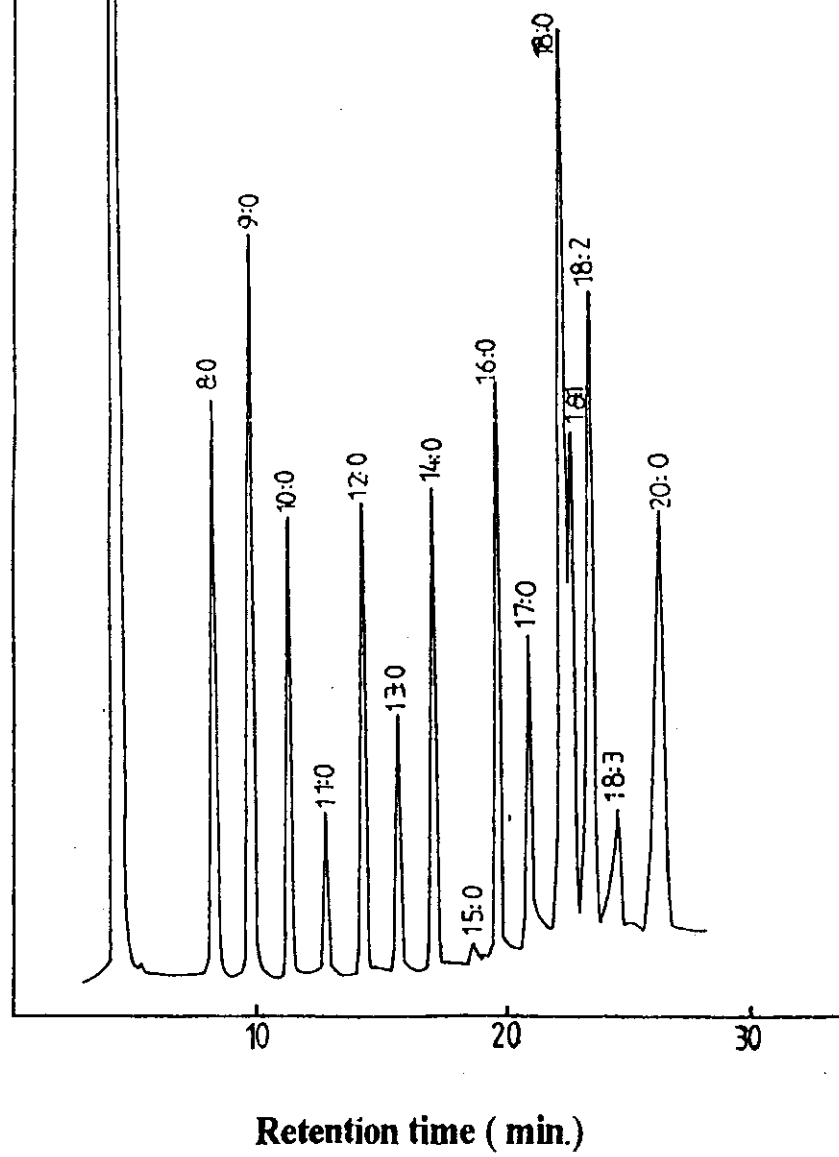
18.1: (Oleic acid)

18.2: (Linoleic acid)

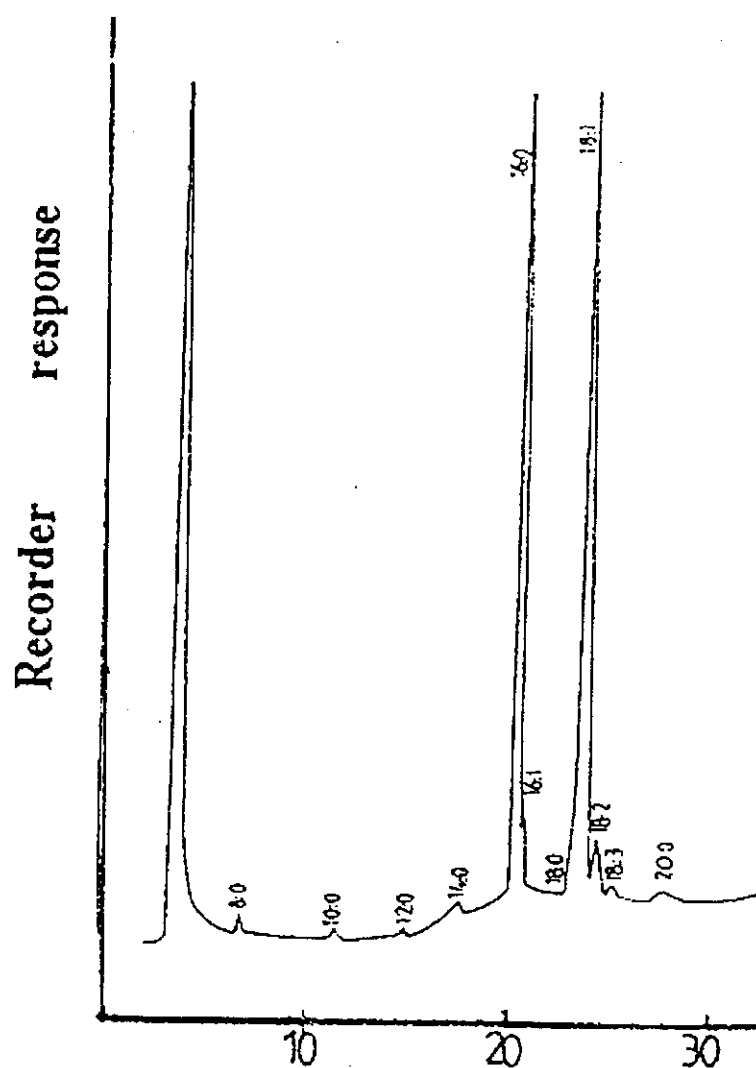
18.3: (Lenolenic acid)

20.0: (Arachedic acid)

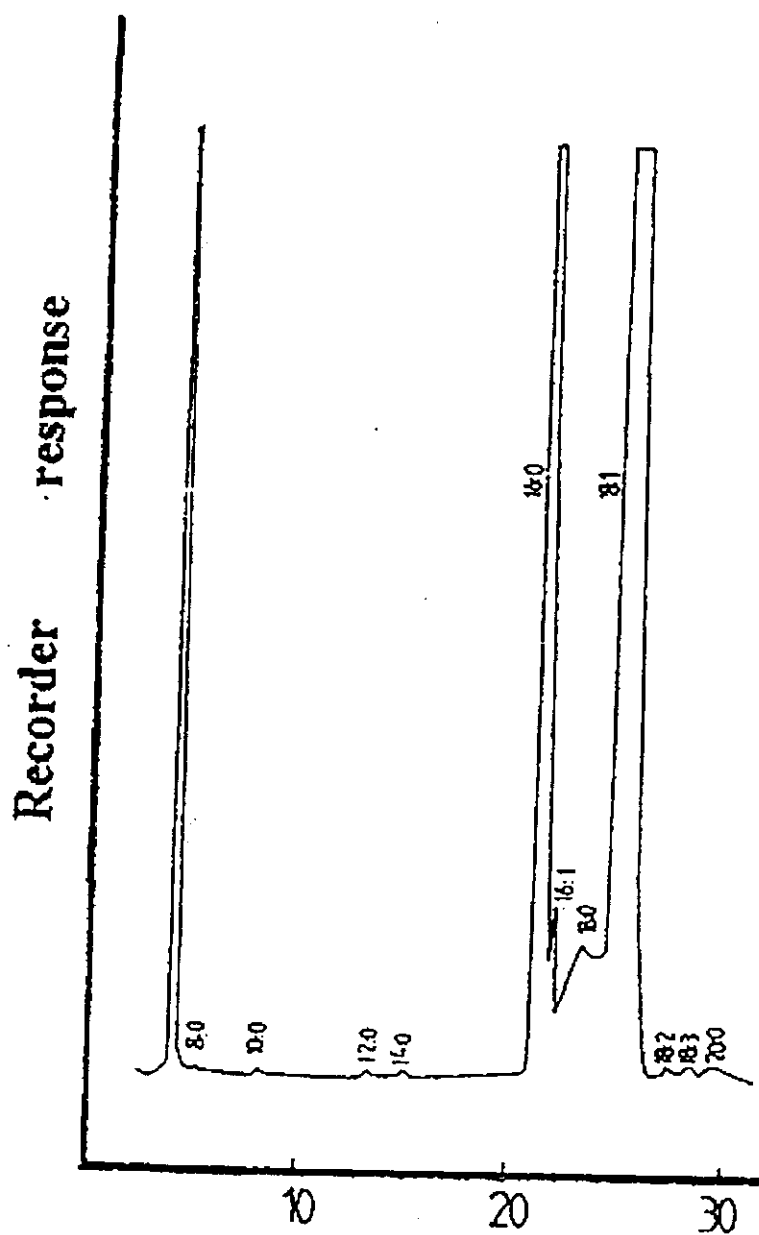
Recorder response



**Fig (4) : GLC chromatogram of standard fatty acid methyl esters.**

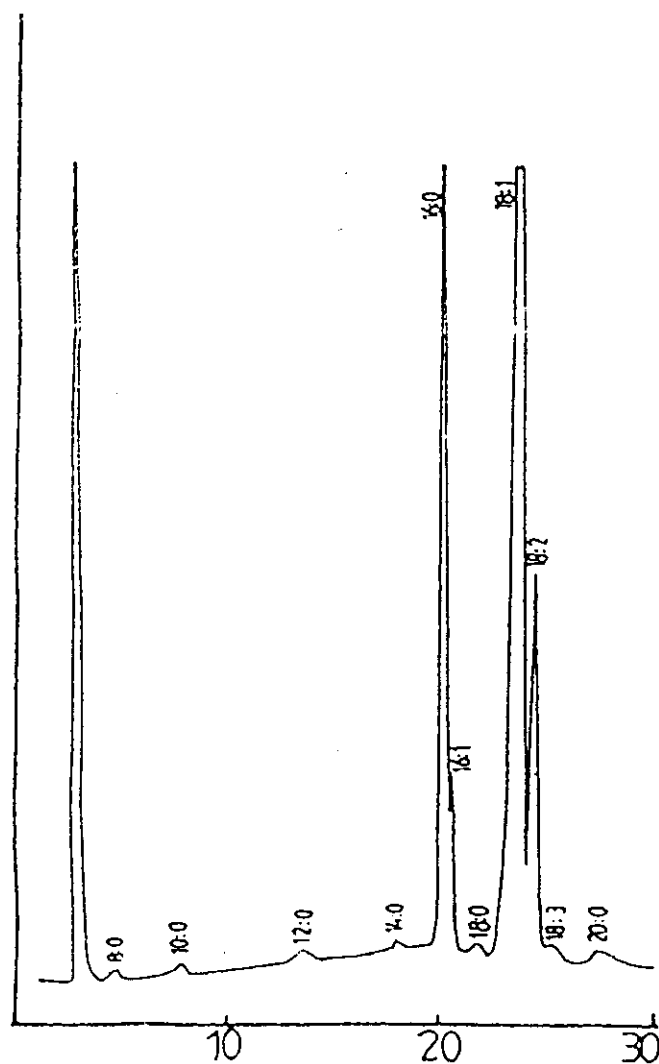


**Fig (5): GLC chromatograms of fatty acid methyl esters extracted from Shemlalli fruits.**



**Fig (6) : GLC chromatograms of fatty acid methyl esters extracted from Weteken fruits.**





*Retention time (min)*

**Fig (7) : GLC chromatograms of fatty acid methyl esters extracted from Maraki fruits.**

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The fatty acids composition of olive oil samples obtained from both Shemllali, Weteken and Maraki cultivars were in accordance with the fatty acids patterns reported by *Seragnol and Morcove (1985)*; *Fujii et al., (1986)*; *Yassa et al., (1990)* and *Zeitoun et al., (1991)*. The data of these researchers demonstrated that oleic and palmitic acids were present as major. Unsaturated and saturated acids, respectively.

### **2- Unsaponifiable matter composition of fresh Shemllali, Weteken and Maraki varieties.**

Analysis of components of the unsaponifiable matter fraction found in edible oils are of extreme importance in establishing not only their origin but also the extraction technique, the final treatment and the possible adulteration.

The unsaponifiable matters content of virgin olive oils under investigation were (0.73, 0.95 and 1.37) respectively. And the unsaponifiable matter. Composition of fresh olive oils were determined by using GLC and the data are shown in Tables (16, 17 and 18). Results from these Tables indicate that the unsaponifiable matter components of olive oils composed of (62.33%, 71.84% and 80.12%) hydrocarbons and (15.63, 18.06 and 19.88) sterols compounds respectively. The hydrocarbons contained mainly squalene, which amounted to (39.37, 51.07 and 66.72) from the unsaponifiable matter content in other words squalene represented (63.16%, 71.08% and 83.27%) respectively of total hydrocarbons. As for sterol compounds  $\beta$  – sitosterol represented the major sterol and it amounted to (12.08, 13.78 and 16.13%) from the total unsaponifiable matters of Shemllali, Weteken, and Maraki olive oils respectively.

Followed by stigmasterol (1.51, 1.82, and 2.642 %), meanwhile

**Result & Discussion****Table (18): Unsaponifiable fraction of fresh Shemllali, Weteken and Maraki varieties olive oil**

<b>Compound</b>	<b>Shemllali variety (0.73%)</b>	<b>Weteken variety (0.95%)</b>	<b>Maraki variety (0.37%)</b>
C <sub>12</sub>	0.014	0.027	0.088
C <sub>14</sub>	0.243	0.252	0.278
C <sub>16</sub>	0.023	0.041	0.089
C <sub>18</sub>	0.397	0.477	0.765
C <sub>21</sub>	0.039	0.043	0.084
C <sub>22</sub>	0.076	0.088	0.149
C <sub>23</sub>	0.422	0.509	0.539
C <sub>24</sub>	4.02	5.93	6.78
C <sub>26</sub>	0.184	0.235	0.291
C <sub>27</sub>	0.127	0.189	0.281
C <sub>28</sub>	0.503	0.519	0.523
Unknown	0.314	0.459	0.484
Squalene	39.37	51.07	66.72
Unknown	2.04	3.11	3.25
Cholestrol	0.110	0.114	0.097
Ergosterol	0.073	0.093	0.151
Campesterol	0.336	0.471	0.863
Stigmasterol	1.51	1.82	2.642
β- Sitosterol	12.08	13.78	16.13
Total hydrocarbons	62.33	71.84	80.12
Total sterols	15.63	18.06	19.88

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campasterol, ergosterol and cholesterol were also detected in minor percentages as shown in Table (18).

These results are in agreement with those reported by *Khalil et al (1983)*, *Egyptian standard (1993)*, *Lanzon et al (1994)*, *Ranalli and Angersa (1996)* and *IOOC (1996)*.

### **4- Effect of packing materials on physical, chemical and sensory properties during storage of olive oils:**

The type of package has a dramatic effect on shelf life of the finished oil. An oil which has been carefully processed to maximize palatability may be damaged by improper selection of the container. In our study olive oils were packaged in four sorts of containers namely, polyethylene terephthalate bottles (PET), high density polyethylene bottles (HDPE), Amber glass bottles (AGB), and clear glass bottles (CGB). After the introduction of oil into the containers, they were tightly capped and stored under different conditions for 12 months i.e, dark, light at room temperatures. Samples were withdrawn at different intervals to measure the changes in color, refractive index, viscosity, UV absorbance, free fatty acids content, iodine value, peroxide value, T.B.A value, and sensory properties of stored oils.

#### **4-1 Effect of packing materials on the color of fresh olive oil cultivar (under investigation) stored for 12 months at room temperature (different conditions).**

The organoleptic characteristics that describe an oil (aroma, color, taste, etc...) provide necessary qualitative subjective information, but instrumental methods are needed to be objective for oil quality control. Color is an important quality factor, and many instrumental methods are used for its determination. The color of olive oil is a quality indicator since, its intensity

is used as an indication of the degree of oxidation of stored olive oil. The obtained results of oils under investigation are shown in Table (19).

From the results exit in Table (19), Shemllali variety color had slightly decreased from 2.6 red to 2.1 red during the second six months of storage, in different conditions (Light and Dark) in both the PET and HDPE containers. In the other hand decrease of color from 2.6 red to 2.4 in light conditions and from 2.6 red to 2.5 in dark conditions in Amber glass bottles. In the clear glass bottles change from 2.6 red to 2.2 in light conditions and from 2.6 red to 2.5 in dark conditions. In the same Table Weteken and Maraki varieties, it could be noticed that the color had the same decreased in red color only. And from the data 2.5 red to 2.0 in PET and HDPE, from 2.5 red to 2.4 in AGb and from 2.5 red to 2.2 in light conditions and from 2.5 to 2.4 in dark conditions in CGb.

This decrement in color could be due to oxidation of carotene and chlorophyll pigments during storage. Light and air facilitate oxidations process as reported by Unal (1978) , Kiritsakis and Dugan (1984) and Mastrobattista (1990) .

Results from the same Table (19) indicate that no further decrease in color of olive oils were noticed till 12 month of storage. This means that the color of olive oils samples stored for 12 month using different containers and in different conditions were within the limits of IOOC, (1996) for the extra virgin olive oils.

#### **4-2 Effect of packing materials on the refractive index of fresh olive oils cultevarus stored for 12 month at room temperature (different conditions) .**

The refractive indexes of the investigated oils were measured at 25°C and the obtained results are shown in table (20).

### **Result & Discussion**

In PET, HDPE, AG b and CGb containers under different conditions for 12 months. The initial RI values were (1.4679, 1.4681, and 1.4677) respectively for three varieties (Shemllali, Weteken and Maraki).. Then increased gradually until its reached to (1.4684,1.4686 and 1.4683) respectively for oils packed in HDPE bottles, stored in light at room temperature. In the other hand the value reached to (1.4682,1.4684 and 1.4682) respectively for samples stored in dark at room temperature. But the increase reached to {(1.4683 and 1.4682), (1.4685and 1.4684) and (1.4681and 1.4680)} respectively for oils packed in PET bottles, stored in light and dark conditions at room temperature respectively. In addition to the increase of samples packed in AGb which reached to {(1.4682 and 1.4681),(1.4683 and 1.482) and (1.4679 and 1.4679 )} respectively , stored in light and dark conditions at room temperature respectively . The increase in RI for samples of fresh olive oils packed in CGb reached to {(1.4683 and 1.4682), (1.4684 and 1.4683) and (1.4680 and 1.4679)} respectively, for samples stored in light and dark conditions at room temperature respectively.

This increase in the RI of stored olive oils due to the formation of high molecular weight compounds during oxidation period as reported by Arya *et al* (1969). He further added that, the refractive index of oil increased during oxidation period, change in the RI of an oil indicate the end of the induction period more easily than the change in its PV. Besides determination of RI is usually faster and easier than determination of PV. From the same Table (20), it could be observed that the changes in refractive index of oil samples under investigation packaged in AG b and CGb had almost the same rate of increase during storage period.

From the obtained results it could be noticed that the olive oils packed in AGb gave the lowest value of RI , where as olive oils packed in

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HDPE gave the highest value of RI at 25°C after storage for 12 month . On the other hand, no changes were observed in refractive indexes of oil being stored in AGb or CGb for 2 months. After 4 months storage of oils that packed in AGb And CGb the refractive indexes were increased to {(1.4682 and 1.4681), (1.4683 and 1.4682) and (1.4679 and 1.4679)} and {(1.4683 and 1.4682), (1.4684 and 1.4683) and (1.4680 and 1.4679)} respectively. For the three varieties no significant differences in RI values of olive oils packed in PET, AGb and CGb after storage for 12 month in dark conditions at room temperature. These results agree with those reported by **Khalaf (1992)**.

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**Table (20) : Changes in refractive index (25C) of Fresh olive oils packed in different containers during storage at different conditions .**

Storage time in months	PET		HDPE		AGb		CGb	
	light	Dark	light	Dark	light	Dark	light	Dark
<b>Samples of Shemllali</b>								
Zero time	1.4679	1.4679	1.4679	1.4679	1.4679	1.4679	1.4679	1.4679
2	1.4680	1.4679	1.4681	1.4680	1.4679	1.4679	1.4679	1.4679
4	1.4681	1.4680	1.4682	1.4681	1.4680	1.4679	1.4681	1.4680
6	1.4681	1.4681	1.4683	1.4682	1.4680	1.4680	1.4681	1.4681
8	1.4682	1.4682	1.4684	1.4682	1.4681	1.4681	1.4682	1.4682
10	1.4683	1.4682	1.4684	1.4682	1.4682	1.4681	1.4683	1.4682
12	1.4683	1.4682	1.4684	1.4682	1.4682	1.4681	1.4683	1.4682
<b>Sambles of weteken:-</b>								
Zero time	1.4681	1.4681	1.4681	1.4681	1.4681	1.4681	1.4681	1.4681
2	1.4682	1.4681	1.4682	1.4682	1.4681	1.4681	1.4681	1.4681
4	1.4683	1.4682	1.4683	1.4682	1.4682	1.4682	1.4684	1.4682
6	1.4684	1.4682	1.4684	1.4683	1.4682	1.4682	1.4684	1.4683
8	1.4683	1.4683	1.4685	1.4683	1.4682	1.4682	1.4684	1.4683
10	1.4684	1.4684	1.4686	1.4684	1.4683	1.4682	1.4684	1.4683
12	1.4685	1.4684	1.4686	1.4684	1.4683	1.4682	1.4684	1.4683
<b>Samples of Maraki :-</b>								
Zero time	1.4677	1.4677	1.4677	1.4677	1.4677	1.4677	1.4677	1.4677
2	1.4678	1.4677	1.4679	1.4678	1.4677	1.4677	1.4677	1.4677
4	1.4679	1.4678	1.4680	1.4679	1.4678	1.4677	1.4677	1.4677
6	1.4680	1.4679	1.4681	1.4680	1.4679	1.4678	1.4679	1.4678
8	1.4681	1.4680	1.4682	1.4681	1.4679	1.4678	1.4680	1.4679
10	1.4681	1.4680	1.4682	1.4682	1.4679	1.4679	1.4680	1.4679
12	1.4681	1.4680	1.4683	1.4682	1.4679	1.4679	1.4680	1.4679

Where:- PET(Polyethylene terephthalte ). HDPE (High density polyethylene bottles ).

AGb (Ambar glass bottles). CGb ( clear class bottles ).



The results clearly showed that there were no changes in the viscosity during storage of virgin olive oils packed in the tested types of containers . the viscosity at 20° C were (82.3,82.6 and 80.2 cp ) respectively . at zero time and after 12 month of storage it had the same values for all stored oils that packed in different types of containers at different conditions .

#### **4-4 Effect of packing materials on the free fatty acids (%) of virgin olive oils stored for 12 month at different conditions.**

Free fatty acids content is the most basic criterion of grading olive oils. Total free fatty acids value is essentially a measure of the free fatty acids in the oils. Oil is categorized in to edible ( $\leq 3.3$ ) and industrial acidity more than (3.3 %) according to **IOOC (1996)**. The virgin olive oils were packed in PET, HDPE, AGb and CGb and stored at room temperature 25 °C (Dark and Light) for one year. The samples were analyzed for free fatty acids content till the end of storage period and obtained results are shown in Table (22).

From the results mentioned in Table (22), it could be noticed that the free fatty acids of fresh olive oils at zero time were (0.14,0.11 and 0.11 %) for oils packaged in PET, HDPE, AGb and CGb and its increased gradually in all containers. After one year of storage the free fatty acids content reached to (0.22, 0.18, and 0.20 %) for oils packaged in PET, AGb and CGb. While the free fatty acids reached to (0.25 %) for oils packaged in HDPE bottles after storage for 12 month in light conditions at room temperature. However the FFA were reached to (0.20 ,0.17 and 0.20 %) in dark at room temperature . (0.22 %) for oils package in HDPE bottles for Shemllali variety. In the other hand the free fatty acids content reached to {(0.23 and 0.21), (0.24 and 0.22) , (0.20 and 0.18) and

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(0.21 and 0.20)} for Weteken variety oils packaged in PET, HDPE, AGb and CGb respectively in light and dark conditions. while the FFA content reached to {(0.22 and 0.19),

**Table (22): Changes in free fatty acids (% as oleic acid) of virgin olive oils packed in different containers during storage at room temperature**

Storage time in month	PET		HDPE		AGb		CGb	
	Light	Dark	Light	Dark	Light	Dark	Light	Dark
<b>Samples of Shemllali variety :</b>								
Zero time	0.14	0.14	0.14	0.14	0.14	0.14	0.14	0.14
2	0.17	0.15	0.18	0.16	0.15	0.15	0.15	0.15
4	0.18	0.16	0.19	0.17	0.16	0.16	0.17	0.16
6	0.18	0.16	0.21	0.17	0.16	0.16	0.17	0.17
8	0.20	0.17	0.22	0.18	0.17	0.16	0.18	0.17
10	0.20	0.19	0.24	0.20	0.17	0.16	0.19	0.18
12	0.22	0.20	0.25	0.22	0.18	0.17	0.20	0.20
<b>Samples of Weteken variety :</b>								
Zero time	0.11	0.11	0.11	0.11	0.11	0.11	0.11	0.11
2	0.13	0.12	0.15	0.14	0.12	0.12	0.12	0.12
4	0.15	0.14	0.17	0.15	0.14	0.12	0.15	0.13
6	0.17	0.15	0.18	0.16	0.15	0.13	0.16	0.15
8	0.18	0.17	0.19	0.16	0.17	0.14	0.18	0.15
10	0.20	0.18	0.20	0.18	0.18	0.17	0.18	0.18
12	0.23	0.21	0.24	0.22	0.20	0.18	0.21	0.20
<b>Samples of Maraki variety :</b>								
Zero time	0.11	0.11	0.11	0.11	0.11	0.11	0.11	0.11
2	0.13	0.12	0.15	0.13	0.12	0.12	0.13	0.12
4	0.15	0.14	0.17	0.15	0.13	0.14	0.15	0.14
6	0.16	0.14	0.20	0.17	0.15	0.15	0.17	0.16
8	0.18	0.17	0.23	0.19	0.17	0.15	0.18	0.17
10	0.20	0.18	0.25	0.20	0.20	0.18	0.19	0.18
12	0.22	0.19	0.26	0.23	0.21	0.18	0.22	0.20

(0.26 and 0.23), (0.21 and 0.18) and (0.22 and 0.20)} for Maraki variety oils packaged in PET, HDPE, AGb and CGb respectively in light and dark conditions. These results agree with those of **Unal (1987), Nkpa et al., (1990) , Khalaf (1992) and Hallabo et al., (1993) .**

Also, from the obtained results, it could be observed that packing fresh olive oils in different containers at room temperature (different conditions) had a marked significant effect on the free fatty acids. These results are within the limits of the **Egyptian Standard (1993), and IOOC (1996)**

**\*NPKa et al., (1990)** reported that, the FFA of the oils increased through out the storage period. At the end of the storage period the free fatty acids increased to be between (33.2 – 36.7 %) from the initial value of (3.0 %) for crude palm oil storage for 98 days in dark at 27° C and packaged in LMC, AGb, CGb and CPb. Plastic packaging materials could allow varying amounts of moisture ingress on the rate of hydrolysis which is negligible as can be seen when comparing the results obtained for the glass bottles and those obtained for the plastic bottles.

**Hallabo et al., (1993)** reported that, the FFA increased during storage CSO packaged in glass, HDPE and LDPE containers stored in dark at 20° C, the FFA values were (0.14 %, 0.15 and 0.17 %) for CSO packaged in glass, HDPE and LDPE after storage for ( 9,4 and 2 months ) respectively. However, the FFA of CSO package in glass containers, HDPE and LDPE were (0.18 %, 0.2 % and 0.17 %) after storage for ( 9 ,4 and one months) in dark at ( 40 °C ) respectively .

So, one can conclude that the acceleration of free fatty acids formation of fresh olive oils packed in plastic could be due to permeability of container.

**4 -5 Effect of packed materials on the peroxide value of fresh olive oils stored for 12 month at different conditions at room temperature**

Peroxide value is the most commonly used method for measuring the total hydroperoxides formed as the primary oxidation products in the oil. The peroxide value of oils under investigation was determined and the results are shown in Table (23).

The value at zero time were (2.31 , 1.93 and 1.15) meq O<sub>2</sub> /kg oil and increased gradually in all containers during storage . After 12 month of storage the peroxide value reached to (21.41, 40.4, 11.1 and 12.31 meq/kg) respectively for Shemllali variety in light conditions, while in Dark conditions the peroxide value had been (17.13, 32.3, 8.21 and 9.25 meq / kg) for oil stored in PET bottles, HDPE bottles, AGb. and CGb, respectively for the same variety . In other hand the peroxide value of Wetecken variety reached to [(18.74 and 13.53), (33.8 and 26.4), (10.41 and 8.12) and (11.11 and 8.88)] in light and dark conditions respectively for oil stored in PET, HDPE, AGb and CGb. While the peroxide value of Maraki variety reached to [(17.2 and 11.2), (27.6 and 18.8), (9.1 and 7.6) and (10.4 and 8.0)] in light and Dark conditions respectively for oil stored in PET , HDPE , AGb and CGb. Also ,it could be observed that there were clear differences between the peroxide value of virgin olive oils samples which had been stored in PET and either glass bottles or HDPE bottles, since the samples in HDPE bottles developed higher peroxide value than those which had been stored in glass bottles and PET. These results are in agreement with those of **Kiritsakis (1982)**, **Kiritsakis *et al.*, (1993)**, **Kiritsakis and Dugan (1984)**,

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**Table (23): changes in peroxide value (mq/kg oil) of fresh olive oils packed in different containers and different conditions during storage at room temperature: -**

Storage time in months	PET		HDPE		AGb		CGb	
	Light	Dark	Light	Dark	Light	Dark	Light	Dark
<b>Samples of Shemllali variety</b>								
Zero time	2.31	2.31	2.31	2.31	2.31	2.31	2.31	2.31
2	5.7	3.6	6.8	4.4	3.01	2.63	4.28	3.24
4	9.2	4.2	10.2	6.1	4.8	3.24	6.18	4.17
6	12.1	7.13	16.7	9.5	7.6	4.7	8.15	6.73
8	14.41	11.16	25.2	14.9	9.7	6.31	10.71	7.15
10	16.81	13.07	31.8	19.1	10.2	7.84	11.22	8.43
12	21.41	17.13	40.4	32.3	11.1	8.21	12.31	9.25
<b>* Samples of Wetenken variety:-</b>								
Zero time	1.93	1.93	1.93	1.93	1.93	1.93	1.93	1.93
2	4.6	3.3	6.3	4.1	2.87	2.19	3.77	2.95
4	7.4	3.8	8.7	5.9	4.39	2.93	5.2	4.12
6	10.2	6.4	13.5	7.3	6.17	4.02	7.34	5.62
8	11.5	9.21	18.6	10.2	7.69	5.71	11.4	6.81
10	14.23	10.98	28.7	16.9	8.53	7.28	12.67	8.25
12	18.74	13.53	33.8	26.4	10.41	8.12	11.11	8.88
<b>* Samples of Maraki variety:-</b>								
Zero time	1.15	1.15	1.15	1.15	1.15	1.15	1.15	1.15
2	4.1	2.8	5.8	3.9	2.44	1.8	3.14	2.6
4	6.3	3.2	8.4	5.2	3.9	2.7	4.7	3.5
6	8.7	5.2	12.2	6.8	4.7	3.5	6.1	4.1
8	10.3	7.4	16.5	9.9	6.5	5.01	8.2	5.3
10	12.8	9.1	23.2	14.6	7.48	6.3	9.3	7.2
12	17.2	11.2	27.6	18.8	9.1	7.6	10.4	8.0

- P.V.: exceeded the limits of Egyptian standard, (1993) and IOOC, (1996).

**Khalil and El-Agaimy (1991), Khalaf (1992), and Hallabo *et al.*, (1993).**

These increments in peroxide value during storage were due to the presence of dissolved oxygen in oil, oxygen permeability through the Packaging materials and migration of packaging materials into the oil.

From the results in Table (23), it could be noticed that.

**Figg (1973), Figge and Koch (1973)** reported that, during storage of edible oil in plastic bottles the migration of packaging peroxide materials into oils could be took place, and the amount of the migrated materials increased by increasing temperature and time of storage.

**NKPA *et al.*, (1990)** reported that, the peroxide values increased during storage period. Higher peroxide values were recorded when the packaged oil samples were stored in direct sunlight. Hence the presence of light accelerates oxidative deterioration of the stored oil.

**NKPA *et al.*, (1992)** found that, during storage period the peroxide values for the oils stored in direct sunlight increased substantially the highest PV was recorded for the oil packaged in POLET. This may be due to the combined effects of the relatively higher permeability of POLET to oxygen and of the transmittance of sunlight. Containers of LMC gave the greatest protection to the oils against oxidative deterioration. The order of the packaging materials with respect to their abilities to offer protection to the oil against primary oxidation, is LMC>AGB, GGB, CPB, CGB>POLET.

POLETbottles is unsatisfactory as packaging material for RBD palm oil because it offers comparatively poor protection to the oil against the deleterious effects of primary oxidation, regardless of whether the oil was

stored in direct sunlight or in the dark.

**Kiritsakis and Dugan, (1994)** found that, the peroxide values of olive oil packaged in plastic bottles were 65 and 75 meq / kg for samples stored in diffused and direct sunlight respectively after storage for 5 months, and 15-20 meq/kg for oil covered with foil and 70-80 meq/kg for the oil sample without a foil cover. The same authors reported that, olive oil packaged in polyethylene bottles gave higher peroxide value than those in glass bottles stored in diffused light. When samples were caved with aluminum foil, lower peroxide values were recorded. Oil in covered glass bottles had lower peroxide values than that in covered plastic bottles after five months of storage.

From these results it could be noticed that, the initial peroxide value of the oils were relatively low, in a period of eight months the values were 25.2 18.6 and 16.5 in polyethylene bottles, these values were higher than what established by **Egyptian Standard (1993)** and **International Olive Oil Council (IOOC), (1996)**. Mean while, the peroxide value reached (9.7,7.69 and 6.5 meq/kg) after 8 months of storage for AGb and(10.71, 11.4 and 8.2 meq/kg), for CGb, and in PET(14.14,11.5and 10.3 meq/ kg ) in light conditions .

Thus, olive oil should be stored in colored glass bottles in order to minimized oxidative deterioration during storage.

#### **4-6-Effects of packing materials on the iodine value of fresh olive oils stored for 12 month at room temperature:**

The iodine value, which reflects the degree of unsaturation of an oil was determined for virgin olive oils stored in PET,HDPE, AGb and CGb and the results are shown in Table(24) .

**Table (24) : changes in iodine value of virgin olive oils packed in different containers during storage at room temperature .**

Storage time in months	PET		HDPE		AGb		CGb	
	Light	Dark	Light	Dark	Light	Dark	Light	Dark
<b>Sample of Shemllali variety:-</b>								
Zero time	83.38	83.38	83.38	83.38	83.38	83.38	83.38	83.38
2	83.34	83.36	83.31	83.35	83.36	83.37	83.36	83.37
4	83.31	83.34	83.27	83.31	83.34	83.37	83.33	83.35
6	83.25	83.32	83.22	83.26	83.29	83.33	83.26	83.34
8	83.23	83.29	81.98	83.17	83.24	83.31	83.25	83.30
10	83.20	83.26	81.86	83.03	83.22	83.30	83.23	83.28
12	83.12	83.21	81.61	82.84	83.18	83.28	83.16	83.25
<b>Samples of Wetenken variety :</b>								
Zero time	80.71	80.71	80.71	80.71	80.71	80.71	80.71	80.71
2	80.66	80.68	80.64	80.67	80.69	80.69	80.68	80.70
4	80.63	80.67	80.62	80.63	80.67	80.68	80.63	80.67
6	80.61	80.64	80.54	80.59	80.63	80.65	80.59	80.65
8	80.54	80.62	80.40	80.53	80.58	80.64	80.53	80.61
10	80.51	80.59	80.32	80.44	80.52	80.61	80.52	80.60
12	80.42	80.57	80.04	80.27	80.51	80.59	80.49	80.59
<b>* Samples of Maraki variety:-</b>								
Zero time	85.08	85.08	85.08	85.08	85.08	85.08	85.08	85.08
2	84.94	84.95	84.87	84.88	84.98	84.97	84.96	84.99
4	84.92	84.93	84.83	84.86	84.95	84.96	84.95	84.97
6	84.88	84.90	84.74	84.80	84.90	84.94	84.91	84.94
8	84.86	84.87	84.12	84.54	84.87	84.90	84.98	84.91
10	84.80	84.85	84.93	84.23	84.83	84.87	84.84	84.88
12	84.76	84.81	84.62	84.06	84.82	84.85	84.83	84.87

- Iodine value was determined by (Hanus) method.

It could be noticed that, the iodine value was (83.38,80.71 and85.08) respectively for three varieties at zero time and reached (83.12,81.61,83.18 and 83.16) in oil packed in PET, HDPE, AGb and CGb respectively in light conditions but in Dark conditions iodine value



reached (83.21,82.84,83.28 and 83.25) respectively for Shemllali variety. In other hand, the iodine value was [(80.42 and80.57), (80.04 and80.27),(80.51 and 80.59)and (80.49 and80.59)] for Weteke variety stored in PET, HDPE, AGb and CGb at light and dark conditions respectively .mean while the iodine value was [(84.76 and 84.81),(83.62 and 84.06),(84.82 and 84.85) and (84.83 and 84.87)]for Maraki variety stored in PET, HDPE, AGb and CGb at light and dark conditions respectively after 12 month of storage at different conditions (light and dark).

From the mentioned results, it could be observed that no clear changes were occurred in iodine value during storage period except for olive oils packed in plastic container which had low iodine value at the end of storage period. This decrease in iodine value may be due to conjugation of unsaturated fatty acids that took place in samples of olive oil packed in plastic bottles compared with that packed in PET and glass bottles.

#### **4-7 Effect of packing materials on the U.V. absorption of fresh olive oils stored for 12 month at room temperature (Light and Dark)**

The development of absorbance at 232 and 270 nm of fresh olive oils samples, which stored in PET, HDPE, AGb and CGb on the shelf for one year at room temperature, are illustrated in Table (25).

Table (25): Changes in U.V. Absorption during storage of fresh olive oils packed in different containers at different conditions

Storage time in months	PET				HDPE				AGb				Cgb			
	Light		Dark		Light		Dark		Light		Dark		Light		Dark	
	K <sub>232</sub>	k <sub>232</sub>	K <sub>232</sub>	k <sub>232</sub>	K <sub>232</sub>	k <sub>232</sub>	K <sub>232</sub>	k <sub>232</sub>	K <sub>232</sub>	k <sub>232</sub>	K <sub>232</sub>	k <sub>232</sub>	K <sub>232</sub>	k <sub>232</sub>	K <sub>232</sub>	k <sub>232</sub>
Sample of Shemilali variety:																
Zero time	1.92	0.07	1.92	0.07	1.92	0.07	1.92	0.07	1.92	0.07	1.92	0.07	1.42	0.07	1.92	0.07
3	2.01	0.09	1.99	0.08	2.04	0.14	2.02	0.11	1.96	0.07	1.93	0.07	2.00	0.08	1.94	0.07
6	2.13	0.12	2.00	0.11	2.22	0.19	2.14	0.18	1.99	0.08	1.97	0.07	2.10	0.15	1.98	0.10
9	2.2	0.15	2.11	0.14	2.31	0.28	2.27	0.25	2.08	0.11	1.99	0.09	2.16	0.16	2.01	0.13
12	2.4	0.28	2.13	0.20	2.63	0.33	2.34	0.28	2.09	0.13	2.00	0.12	2.19	0.19	2.05	0.18
* Samples of Wecken variety :-																
Zero time	1.78	0.03	1.78	0.03	1.78	0.03	1.78	0.03	1.78	0.03	1.78	0.03	1.78	0.03	1.78	0.03
3	1.88	0.05	1.85	0.03	1.93	0.09	1.92	0.06	1.82	0.03	1.79	0.03	1.86	0.04	1.81	0.03
6	2.01	0.09	1.89	0.07	2.12	0.18	2.04	0.11	1.84	0.05	1.81	0.03	1.91	0.07	1.87	0.05
9	2.11	0.13	1.97	0.11	2.24	0.23	2.13	0.17	1.87	0.09	1.83	0.06	1.97	0.12	1.92	0.08
12	2.14	0.19	1.99	0.15	2.43	0.29	2.27	0.13	1.90	0.12	1.84	0.10	2.00	0.16	1.94	0.11
* samples of Maraki variety :-																
Zero time	1.81	0.02	1.81	0.02	1.81	0.02	1.81	0.02	1.81	0.02	1.81	0.02	1.81	0.02	1.81	0.02
3	1.89	0.06	1.86	0.04	1.91	0.08	1.98	0.05	1.84	0.03	1.83	0.02	1.85	0.05	1.83	0.02
6	1.97	0.08	1.89	0.08	2.09	0.16	1.97	0.14	1.86	0.04	1.85	0.03	1.91	0.10	1.87	0.04
9	2.05	0.19	1.94	0.13	2.25	0.21	2.06	0.17	1.92	0.11	1.87	0.04	2.01	0.15	1.93	0.10
12	2.12	0.24	1.95	0.20	2.31	0.27	2.18	0.23	1.93	0.16	1.88	0.10	2.08	0.19	1.94	0.14

## **Result & Discussion**

The conjugated hydroperoxides absorb at 232 nm while at 270 nm the secondary oxidation products (aldehydes and ketones) absorb. Conjugated diene and trienes, formed during refining or bleaching of olive oil, also absorb uv at 270 nm, thus high absorbance at 270 nm is related to olive oil oxidation or to refining process or to both of them. Low absorbance values  $k_{232}$  and  $k_{270}$  correspond to good olive oil quality (Kiritsakis, 1990).

From the tabulated data in table (25) it could be observed that  $E_{232}$  and to  $E_{270}$  of fresh olive oils samples increased continuously with the increase of storage period and this observation is in agreement with the finding of Gutfinger *et al.*, (1975), and Kiritsakis and Dugan (1984).

Also, from the tabulated data in Table (25), it could be noticed that the  $E_{232}$ ,  $E_{270}$  of olive oils under investigation at zero time were ( 1.92 and 0.07) for Shemllali variety , (1.78 and 0.03) for Weteken , (1.81 and 0.02) for Maraki variety respectively. After 12 month of storage of fresh olive oils samples packed in PET, HDPE, AGb and CGb. The  $E_{232}$  reached [ (2.24 and 2.13), (2.63 and 2.34),(2.09 and 2.00)and (2.19 and 2.05)] respectively for Shemllali variety at light and dark conditions.

While the  $E_{232}$  reached [(2.11 and 1.99), (2.43 and 2.27), (1.90 and 1.85) and (2.00 and 1.94) ] for Weteken variety at light and Dark conditions. In other hand the  $E_{232}$  reached [(2.12 and 1.97),(2.31 and 2.18), (1.95 and 1.88) and (2.08 and 1.95)] for Maraki variety respectively .

From the obtained results, it could be noticed that the increase in  $E_{232}$  of fresh olive oils samples which had been stored in AGb were less than those stored in CGb, PET and HDPE throughout the storage period.

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In other words colored glass bottles improved the oxidative stability of different light. These results are in good agreement with the findings of **Warner and Mounts (1984)** and **Kiritsakis et al (1993)** and **Augstin and Berry(1993)**, and **Khalil and El-Agaimy (1991)**.

Results from Table (25) indicate that change in [absorbance at 270 nm were much higher than those for the absorbance at 232 nm under storing conditions. The absorbance at 270 at zero time were (0.07,0.03 and 0.02) for three cultivars respectively, but after 12 months of storage it reached [(0.23 and 0.20),(0.33 and 0.28),(0.13 and 0.12) and (0.19 and 0.18)] for Shemllali, it reached (0.19 and 0.15),(0.29 and 0.31),(0.12 and 0.10) and (0.16 and 0.11)] for Wetecken and it reached [(0.24 and 0.20),(0.27 and 0.23),(0.16 and 0.10) and (0.19 and 0.14)] for Maraki at light and Dark respectively in case of PET, HDPE, AGb and CGb.

U.V. absorption at 232 and 270 nm of the olive oils packed in AGb and stored for 12 month were much lower than those for the absorption at 232 and 270 nm of the olive oils packed in HDPE bottles and stored for 3 and 6 months of storage only. Therefore , it could be concluded that storing in AGb was more effective in retarding oxidation of virgin olive oils compared with other types of containers being investigated .

**Frankel, (1985), Rahmani,(1989) and Kiritsakis and Osman(1995)** reported that green glass bottles eliminate a part of UV light which promote formation of free radicals through the initiation stage and gave protection to oil and reduced a percentage of conjugated diene formed .

## Result & Discussion

### 4-8- Effect of packing materials on the TBA value of virgin olive oils stored for 12 month at different conditions :

Thiobarbituric acid (TBA) test is a condensation reaction between TBA and malonaldehyde, a product of fatty acid oxidation and this test measures the secondary products which formed from hydroperoxides .

The increasing in the amount of red pigment formed in the reaction between 2-thiobarbituric acid (TBA) and oxidised lipides as oxidative rancidity advances has been applied to a wide variety of fatty foods .

The TBA was determined in virgin olive oils (under investigation ) stored in AGb,CGb,PET and HDPE bottles .

**Table (26): Changes in TBA value during storage of fresh Shemllali olive oil packed in different containers at room temperature (light and dark).**

Storage period in(months)	PET		HDPE		AGB		CGb	
	Light	Dark	Light	Dark	Light	Dark	Light	Dark
Zero time	0.023	0.023	0.023	0.023	0.023	0.023	0.023	0.023
2	0.046	0.031	0.057	0.040	0.032	0.029	0.043	0.036
4	0.058	0.037	0.073	0.045	0.040	0.031	0.054	0.039
6	0.069	0.048	0.081	0.069	0.053	0.035	0.071	0.044
8	0.077	0.061	0.085	0.076	0.064	0.038	0.077	0.053
10	0.088	0.068	0.097	0.082	0.071	0.044	0.083	0.061
12	0.092	0.074	0.112	0.093	0.080	0.048	0.094	0.064
Total increasing after 12 month	0.453	0.342	0.528	0.428	0.363	0.248	0.445	0.320

**\*TBA (thiobarbituric acid) value refers to the absorbance of the color of oxidized products formed at 535 nm.**

### **Result & Discussion**

From the results shown in Table (26) it could be noticed that ,the TBA value was (0.023) at zero time and then increased gradually with the increase in the storage period till it reached (0.112 and 0.093) in HDPE bottles , (0.092 and 0.074) in PET bottles , (0.080 and 0.048) in AGb ,and (0.094 and 0.064) in CGb after 12 month of storage . both light and dark conditions respectively.

**Table (27): Changes in TBA value during storage of fresh Weteken olive oil packed in different containers at different conditions (light and Dark).**

Storage period in(months)	PET		HDPE		AGB		CGb	
	Light	Dark	Light	Dark	Light	Dark	Light	Dark
Zero time	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
2	0.029	0.017	0.035	0.027	0.017	0.008	0.023	0.014
4	0.056	0.031	0.064	0.043	0.031	0.019	0.047	0.026
6	0.068	0.042	0.083	0.054	0.038	0.024	0.059	0.037
8	0.073	0.047	0.094	0.059	0.044	0.032	0.068	0.045
10	0.081	0.056	0.102	0.066	0.046	0.037	0.081	0.047
12	0.086	0.059	0.114	0.071	0.061	0.039	0.085	0.054
Total increasing after 12 month	0.393	0.252	0.492	0.320	0.237	0.159	0.363	0.223

**\*TBA (thiobarbituric acid) value refers to the absorbance of the color of oxidized products formed at 535 nm.**

From the results shown in Table (27) it could be noticed that, the TBA value was (0.00 ) at zero time and then increased also gradually with the increase in the storage period till it reached (0.114 and 0.071) in HDPE

### **Result & Discussion**

bottles , (0.086 and 0.059 )in PET bottles , (0.061 and 0.039)in AGb, and (0.085 and 0.054) in CGb after 12 month of storage, both in light and dark conditions respectively.

**Table (28): Changes in TBA value during storage of fresh Maraki olive oil packed in different containers at different conditions (light and Dark).**

Storage period in(months)	PET		HDPE		AGB		CGb	
	Light	Dark	Light	Dark	Light	Dark	Light	Dark
Zero time	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
2	0.025	0.012	0.038	0.024	0.013	0.011	0.026	0.012
4	0.042	0.024	0.059	0.036	0.020	0.017	0.047	0.019
6	0.053	0.039	0.076	0.051	0.032	0.023	0.053	0.025
8	0.062	0.046	0.087	0.059	0.037	0.030	0.057	0.033
10	0.074	0.050	0.096	0.065	0.041	0.034	0.060	0.041
12	0.080	0.052	0.104	0.073	0.054	0.035	0.072	0.043
Total increasing after 12 month	0.336	0.223	0.460	0.308	0.197	0.150	0.315	0.173

**\*TBA (thiobarbituric acid) value refers to the absorbance of the color of oxidized products formed at 535 nm.**

Also, from the results shown in Table (28) it could be noticed that , the TBA value was (0.00 ) at zero time and then increased gradually with the increase in the storage period till it reached (0.080 and 0.050) in PET bottles , (0.104 and 0.073) and HDPE bottles , (0.054 and 0.035) in AGb and (0.072 and 0.043) in CGb after 12 month of storage at room temperature (light and dark conditions).

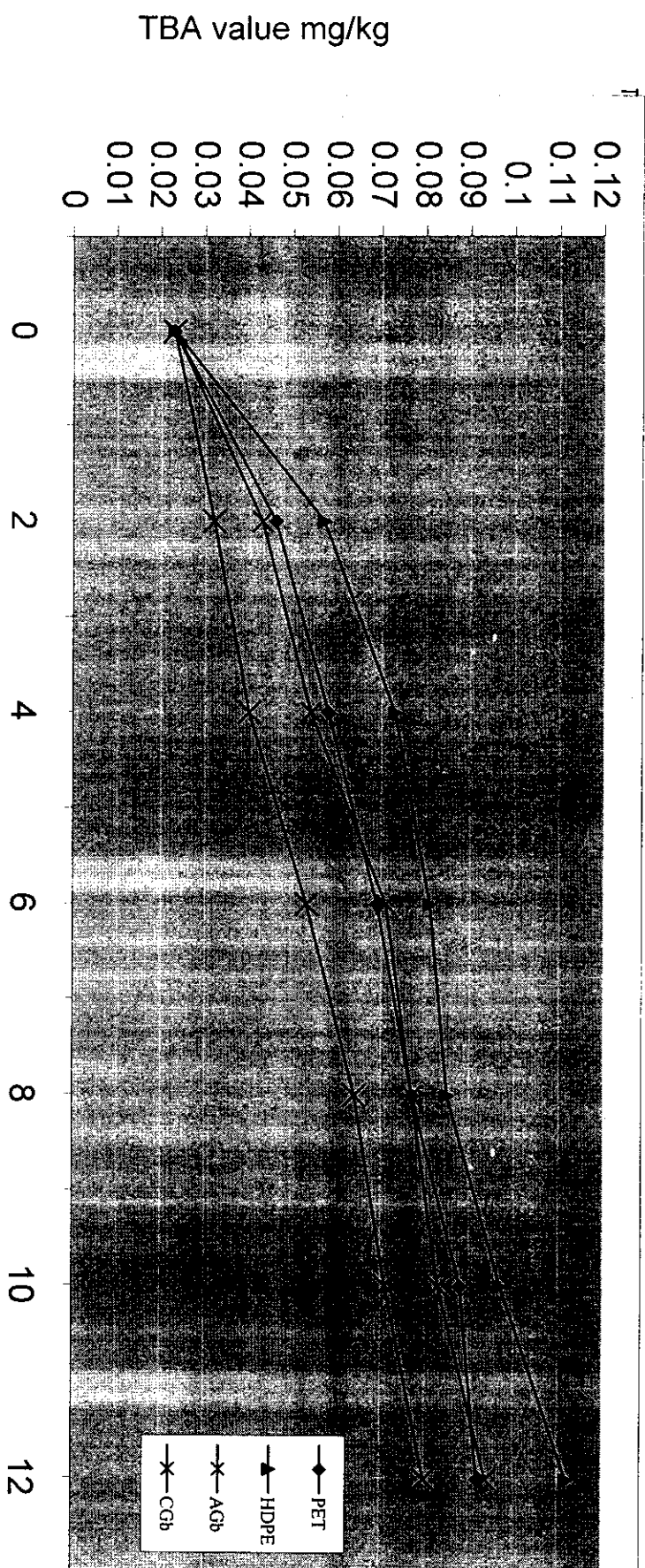
From the same Tables (26,27 and 28) and Figs. (8,9, 10, 11, 12,and 13)

### ***Result & Discussion***

illustrated the changeable rates of TBA in all tested oils in the course of storage the higher value of TBA was observed in HDPE bottles followed by PET, CGb, and AGb. These results are in agreement with **Hallabo *et al.*, (1993)** .

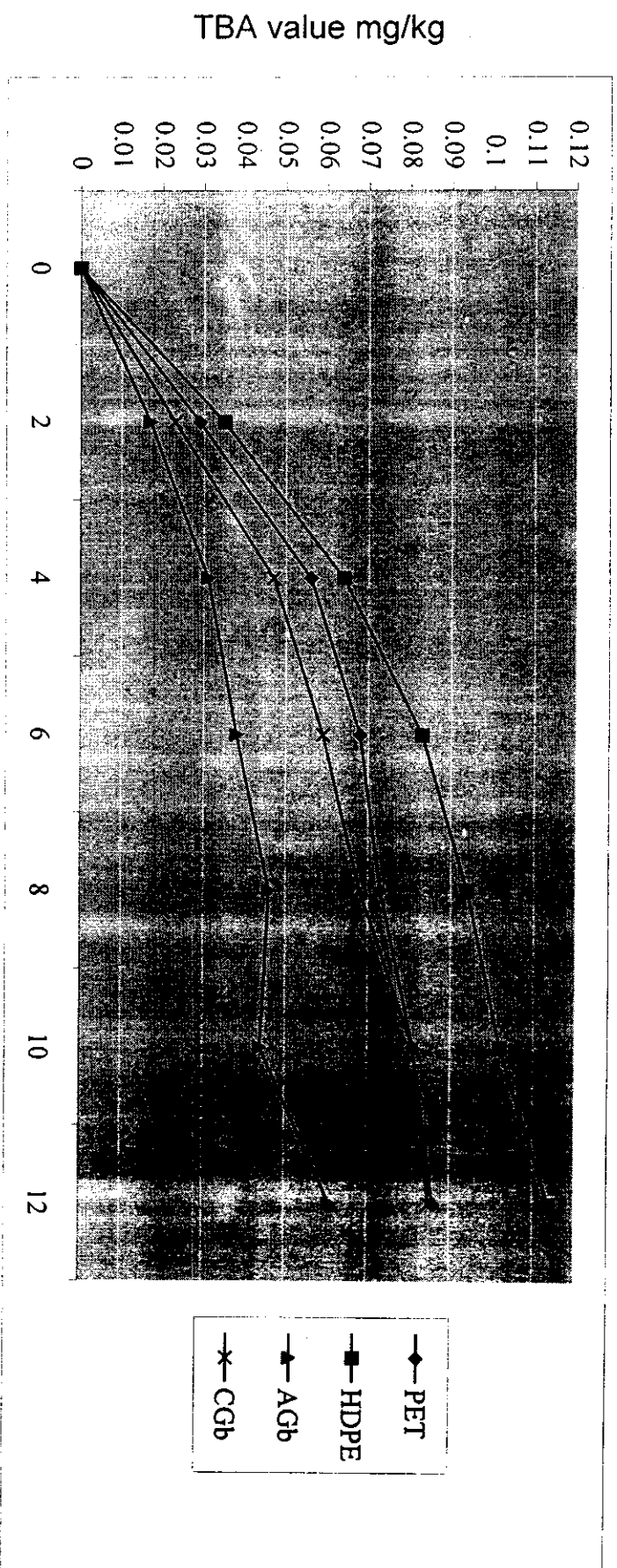
From the previous results it could be noticed that, the oils packaged in HDPE gave the highest TBA values, no significant difference in the TBA values developed in the oils packaged in AGb and CGb for olive oils stored in dark at room temperature. HDPE gave the highest value of TBA. While the lowest TBA value was in oil of AGb. **Hallabo *et al.*, (1993)** reported that glass containers gave good protection against the deleterious effect of autoxidation followed by HDPE containers and LDPE containers at 20°C. Also, it is clear that the glass bottles (especially green glass) were affective in protecting oil from oxidation. These results are in agreement with those of **Gutfinger *et al.*, (1975)** and **Uanl and Colakoglu (1983)**.





Period of storage (month)

Fig (8) change in TBA values of Fresh shemlali olive oil packed in PET, HDPE, AGb and CGb and stored at room temperature, Darkness.



Fig(10): Changes in TBA values of fresh Weteken olive oil packed in PET,HDPE,AGb and CGb and stored at room temperature, Lightness

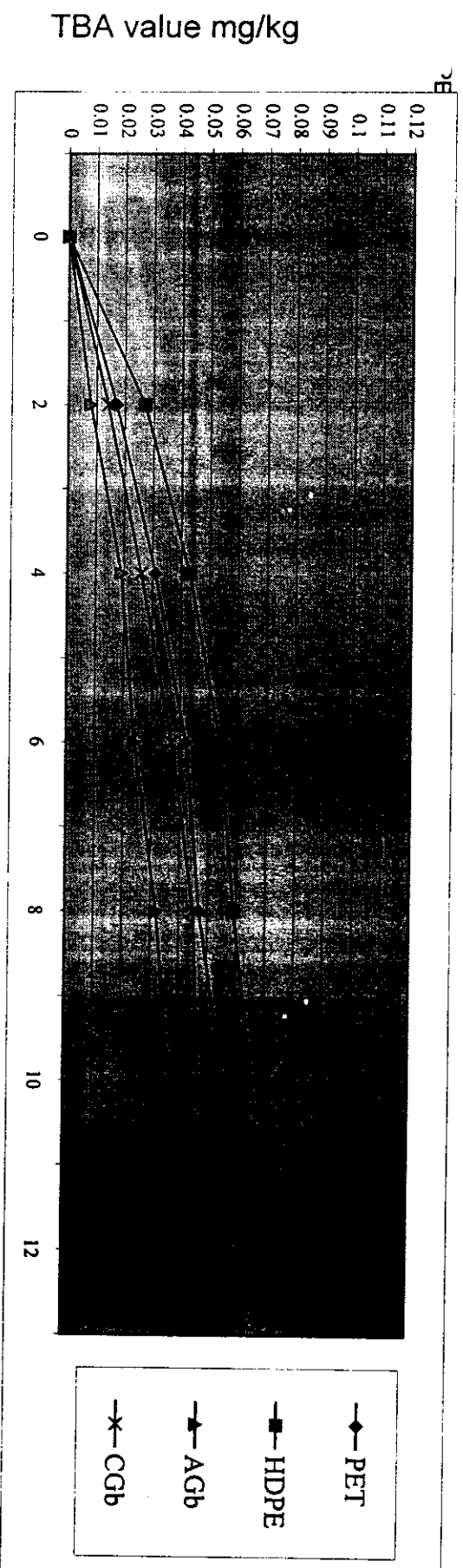


Fig (11) Change in TBA values of fresh Weteken olive oil packed in PET,HDPE ,AGb and CGb and stored at room temperature, Darkness

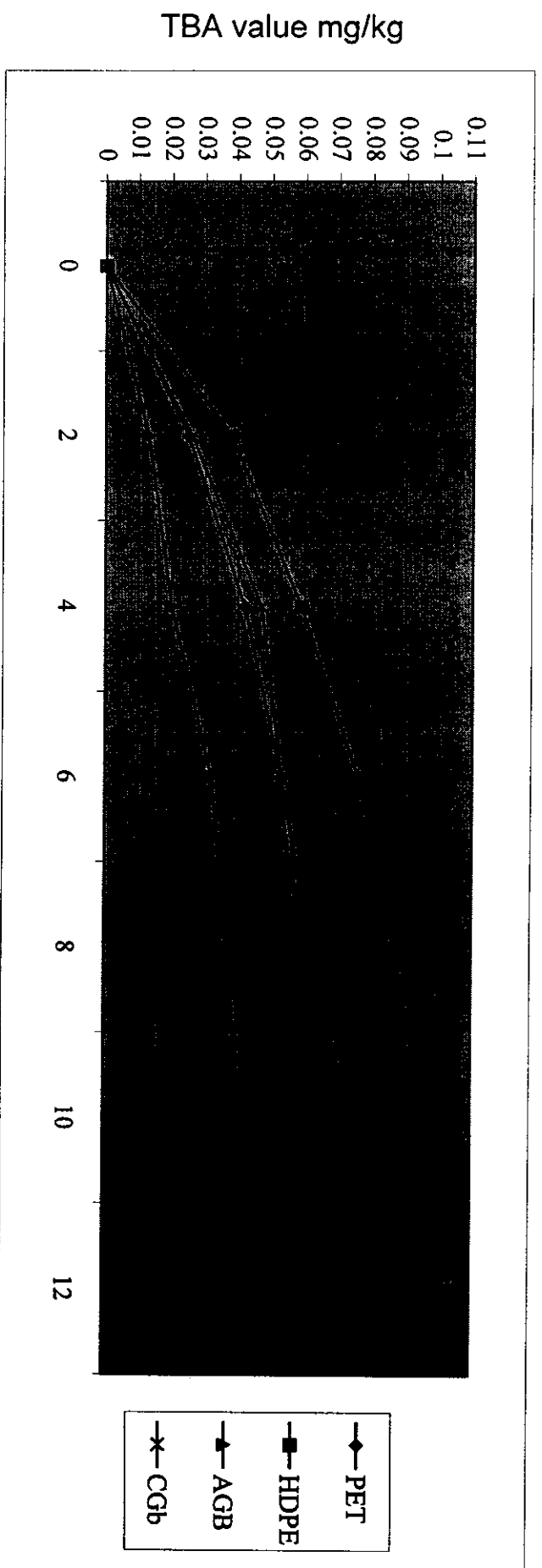
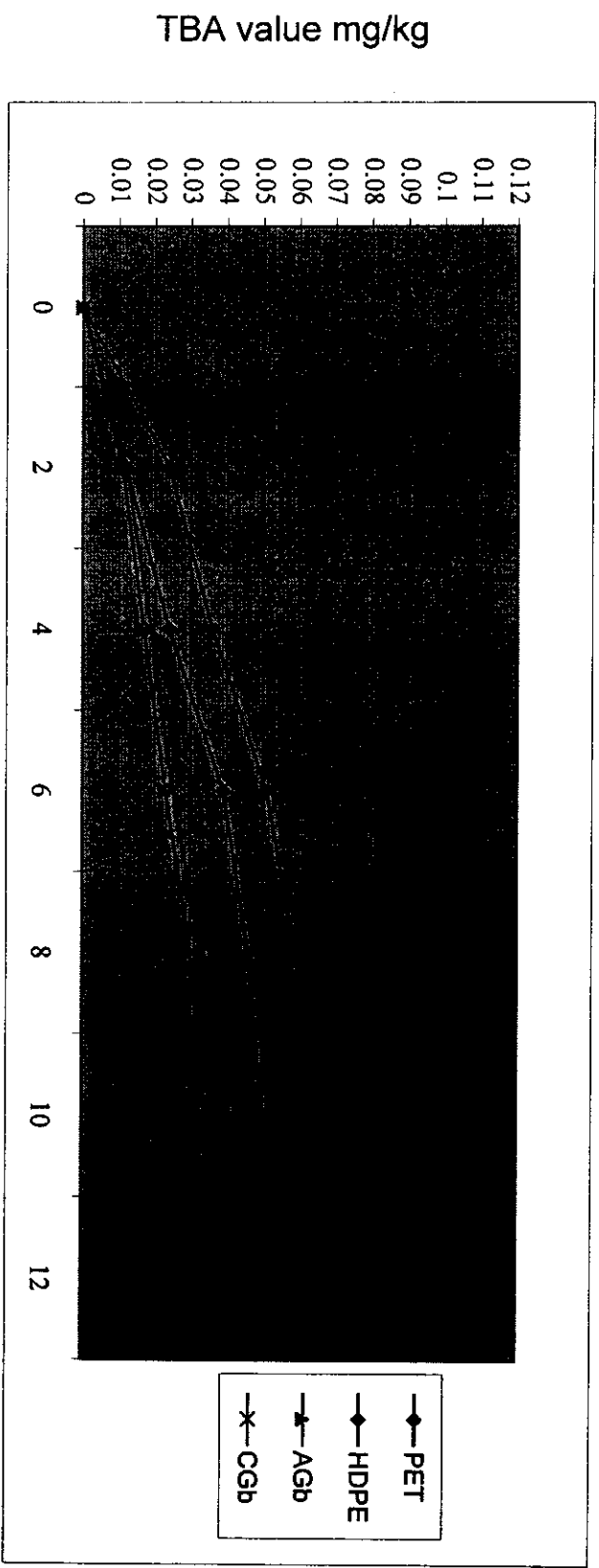


Figure (12) change in TBA values of Fresh Maraki olive oil packed in PET,HDPE ,AGb and CGB and stored at room temperature , Lightness.



Fig(13): Changes in TBA values of fresh Maraki olive oil packed in PET,HDPE,AGb and CGb and stored at room temperature, Darkness

**4.9- Effect of packing materials on the sensory properties of fresh olive oils under investigation stored for 12 moth at room temperature:**

Sensory properties of olive oil at zero time and during storage for 12 month (at 3 months intervals) in different types of containers were evaluated by 10 panelists. Virgin olive oils packed in plastic bottles and stored for 9 and 12 months were excluded from evaluation since, their peroxide value exceeded 20 meq/kg oil ( the maximum Egyptian standard limit ) after 6 months of Storage.

Also this study show the highest of evaluation since, (4.0) was recorded in oil packaged in AGb after storage for 12 month in light at room temperature followed by oils packaging in CGb after 12 month of storage . These oils were accepted to be used by consumer, while oils in PET and HDPE recorded  $<2.5$  after storage for 9,6 months respectively. Thus, The olive oils packaged in PET and HDPE were considered to be rejected by consumer after 9 and 6 months respectively. According to the regulation of Egyptian standards (EDS1986). Also the oils packaged in CGb recorded (3.0) after storage 12 month. And these oils were considered to be rejected by consumer after 12 month.

The results from this study indicated that virgin olive oils stored in AGb for 12 month kept significantly sensory properties as high that of the virgin olive oils at zero time.

Sensory properties decreased during storage period. Virgin olive oils packaged in AGb and CGb gave higher scores and accepted for panelists after storage for 12 moth in dark at room temperature

## **Result & Discussion**

meanwhile olive oils packaged in PET and HDPE bottles gave the lower scores < 3.0 and 2.5 after storage for 12 and 9 months respectively.

**Hallabo et al., (1993)** noticed that, flavor scores decreased during storage period. The flavor scores of oils packaged in glass, HDPE and LDPE containers were 6.0, < 5.0 and 5.0 after stored in Dark at 20° C for 9, 4 and 2 months respectively. On the other hand, Scores of oils in glass, HDPE and LDPE containers stored in dark at 40° C were 5.0, < 5.0 and < 5.0 after storage for 9,4 and one months respectively.

These results indicate that packing olive oil in AGb significantly eliminated changes of sensory properties of virgin olive oils compared with that packed in either CGb or REC or HDPE bottles. These results are in agreement with those of **Evans et al (1973)**, **Unal (1978)**, **Kiritsakis et al., (1993)**, **Kiritsakis and Dugan (1984)**, **Kiritsakis (1988)**, and **Abdul Magied (1994)**.

### **5- Effect of crude olive pomace oil on sunflower oil oxidative stability.**

Effect of blending crude olive pomace oil of (*Shemllali, Weteken and Maraki*) with sunflower oil (at 10, 20, 30 and 40 % levels) and addition these levels as follow:

M + W+S	( 1)	(2)	(3)
* Maraki.	50	75	85
* Weteken.	25	12.5	7.5
* Shemllali.	25	12.5	7.5

## **Result & Discussion**

In order to Improve the (oxidative stability of sun flower oil) the tests were carried out using oven test method at 63°C. The obtained results are shown in Table (29).

Completely refined sunflower oil, and extracted crude olive pomace oils were evaluated and their data are tabulated in Tables( 29,30,31,32).

### **5-1- Evaluation of sun flower oil:**

In order to study effect of crude olive pomace oil it on-refined sunflower oil properties, the acid value, free fatty acid content, peroxide value, iodine value, unsaponifiable matters, saponification number and oxidative stability were determined for the investigated refined sunflower oil. Such properties were needed to be sure from the freshness of oil and its quality characteristics, which are within the limits of the **Egyptian Standard (1993)**. The obtained results are shown in Table (29).

**Table (29) : Chemical properties of sunflower oil.**

Parameter	Value
1- Acid value.	0.083
2-Free fatty acids (% as oleic acid)	0.04%
3-Peroxide value (meq/kg oil)	1.6
4-Iodine value	134.17
5-Un saponifiable matter content	1.38%
6- Saponification Number	193.84

From the results shown in Table (29), it could be noticed that the acid value and free fatty acid content were (0.083 and 0.04), respectively, while the peroxide value was found to be only (1.6 meq / kg oil).



Iodine value (Hanus) as an identity characteristic parameter was (134.17), which indicate that oil was belonged to the semi drying oils category and these results were within the limits of the **Egyptian Standards for edible sunflower oil (1993)**.

Also, the unsaponifiable matter of sunflower oil was 1.38 %, while its saponification number was (193.84 ).

The oxidative stability of sunflower oil by oven test at  $63^{\circ}\text{C} \pm 1$  was 6.0 days. These results indicate that refined sunflower oil was in a good quality and suitable as a media for studying antioxidant activities of crude olive pomace oil.

## **5.2- Evaluation of crude olive pomace oils:**

Olive pomace oils were extracted from olive pomace of (*Shemllali, Weteken and Maraki*) varieties, using hexane. The acid value, free fatty acid content, peroxide value, iodine value, unsaponifiable matters, saponification number, total phenols content of these oils are determined. Such properties could give an idea about the quality of oils under investigation. The obtained results are shown in tables (30,31 and 32).

**Result & Discussion****Table (30): Chemical properties of crude olive pomace oil of Shemllali variety.**

Parameter	Value
Acid value.	0.89
Free fatty acids (% as oleic acid)	0.44
Peroxide value (meq/kg oil)	17.03
Iodine value ( Hans)	81.31
Unsaponifiable matters content	1.96
Saponification Number	196.74
Total phenols content.	142ppm

**Table (31): Chemical properties of crude olive pomace oil of Weteken variety.**

Parameter	Value
Acid value.	0.82
Free fatty acids (% as oleic acid)	0.41
Peroxide value (meq/kg oil)	15.65
Iodine value ( Hans)	80.25
Unsaponifiable matter content	2.24
Saponification Number	195.38
Total phenols content.	168ppm

**Table (32): Chemical properties of crude olive pomace oil of Maraki variety.**

parameter	Value
Acid value.	0.78
Free fatty acids (% as oleic acid)	0.39
Peroxide value (meq/kg oil)	15.39
Iodine value (Hans)	81.94
Unsaponifiable matters content	2.55
Saponification Number	195.13
Total phenols content.	188ppm

From the obtained results shown in Tables (30.31 and 32) it could be noticed that acid value and free fatty acids content were (0.89, 0.82 and 0.78) and (0.044, 0.041 and 0.39%) respectively. These results indicated that hydrolysis of oil was at its early stages. Though, peroxide values of crude olive pomace oil were found to be (17.03, 15.65 and 15.00 meq/kg oil), for oils under investigation respectively, they were within the limits of IOOC, (1996).

Iodine values (Hans) were (81.31, 80.25 and 81.94), which indicate that oils belonged to the non drying oils category, while the unsaponifiable matter of crude olive pomace oils were (1.96, 2.24 and 2.55%), the saponification number were (196.74, 195.38 and 195.13), and total phenols content were (142, 168, and 188 ppm).

**Table (33): Induction period in days of sunflower oil blends during incubation at 63 °C**

<b>M W S (50+ 25+25)</b>	<b>Concentrations</b>	<b>Induction period (in days)</b>	<b>Relative stability</b>
Sunflower oil blend	0%	6.0	1.0
Containing crude	10%	7.3	1.21
Olive pomace oil at	20%	8.2	1.36
	30%	8.9	1.48
	40%	9.4	1.56
<b>(M W S) * (75 + 12.5+ 12.5)</b>			
Sunflower oil blend	0%	6.0	1.0
Containing crude	10%	7.8	1.30
Olive pomace oil at	20%	8.9	1.48
	30%	9.4	1.56
	40%	9.9	1.65
<b>(M W S) * (85+ 7.5+ 7.5)</b>			
Sunflower oil blend	0%	6.0	1.0
Containing crude	10%	8.1	1.35
Olive pomace oil at	20%	9.2	1.53
	30%	9.7	1.61
	40%	10.9	1.81

**5-3- Effect of blending crude olive pomace oils with sunflower oil on the oxidative stability of sunflower oil:**

This experiment was carried out to study the effect of blending crude olive pomace oils at 10, 20, 30 and 40 % levels after blended this oils on the oxidative stability of sunflower oil using oven test method at 63°C. The obtained results are illustrated in Table (33) and Figs. (14,15 and 16).

From the results shown in Figs. (14,15, and 16) and Table (33), it could be observed that addition of crude olive pomace oils to sunflower oil at levels 10, 20, 30, and 40% improved its oxidative stability since, the induction periods of these blends showed noticeable gradual increase with the increasing level of crude olive pomace oils. The induction period of control (sunflower oil) was (6) days and increased to (7.3, 7.8 and 8.1) days respectively when crude olive pomace oils were added at (10%) level, while, it increased to (8.2, 8.9 and 9.2) days, (8.9, 9.4 and 9.7) days and (9.4, 9.9 and 10.9) days with the addition of 20, 30 and 40% crude olive pomace oils, respectively.

Therefore, increasing % of crude olive pomace oils in the blend from 10 to 40 % was accompanied by an increase in the relative stability of sunflower oil to (1.21,1.36,1.48 and 1.56) , respectively for sample consists of ( $M_{50} + W_{25} + S_{25}$ ) . While it reached (1.30, 1.48, 1.56 and 1.65) for sample consists of ( $M_{75} + W_{12.5} + S_{12.5}$ ). and it reached to 1.35, 1.53, 1.61 and 1.81) for sample consists of ( $M_{85} + W_{7.5} + S_{7.5}$ ) .

These increments in the oxidative stability of the blends could be due to the lower polyunsaturated fatty acids (P) / saturated fatty acids (S)

### ***Result & Discussion***

ratio of crude olive pomace oils (0.3) instead of the higher P/sratio in sunflower (2.7) according to **Kiritsakis (1990)**.

In other words , by increasing level of crude olive pomace oils added to sunflower oil changed its P/S ratio towards the saturated side making the blend more saturated and more stable for oxidation comparing with sunflower oil (rich in polyunsaturated fatty acids) . On the other hand, crude olive pomace oils (under investigation) could be considered as an unsaponifiable rich fraction (1.96, 2.24 and 2.55 %) with high content of an antioxidant activity such as squalene,  $\beta$ -sitosterol and tocopherol.

## ***SUMMARY***

This study was carried out to investigate effect of different packing materials on keeping quality of virgin olive oils. During storage for one year, at room temperature (at different conditions). Also, the effect of addition of crude olive pomace oils (under investigation), to sunflower oil to declare its oxidative stability was studied. The obtained results could be summarized as follow:-

1-The physical and chemical properties of virgin olive oils were determined and the results indicate that color was 2.6 red and 0.8 blue and 35 yellow using Lovibond tintometer, for Shemllali variety and it was 2,5 red and 1.2 blue and 35 yellow for Weteken variety and it was 2.5 red and 0.6 blue and 35 yellow for Maraki variety respectively. The refractive index at 25°C was (1.4679, 1.4681 and 1.4677). Viscosity at 20°C was (82.3, 82.6 and 80.2 cp.) and acid values were (0.28, 0.23 and 0.22), free fatty acids (% as oleic acid) were (0.14, 0.11 and 0.11%), Peroxide values (as meq O<sub>2</sub>/ kg oil) were (2.31, 1.93 and 1.15), iodine value (Hanus) was (83.38, 80.71 and 85.08), absorbance in UV region at 232 nm was ( 1.92 , 1.78 and 1.81 ), at 270 nm was ( 0.07 , 0.03 and 0.02), TBA values ( as absorbance at 535 nm ) were (0.023 , zero , and zero), oxidative stability in hours was (25.2 , 28.6 and 30.7), hr at 100°C, by Rancimat method and unsaponifiable matter contents were ( 0.73, 0.95 and 1.37%), saponification numbers were ( 196. 57 , 193 . 40 and 191.93), while the total phenols content were (204 , 258 , 308) ppm.

## Summary

2-Fatty acids composition of virgin olive oils (Shemllali, Weteken and Maraki), by using GLC revealed that it contained (0.57, 0.39 and 0.73%) myrstic acid, (19.49, 21.74 and 12.58 %) palmitic acid, (1.50, 2.6 and 2.73%) palmitoleic acid, (2.75, 2.95 and 0.74%), stearic acid, (70.86, 67.93 and 81.86%) oleic acid, (2.31 , 2.88 and 3.95%) linoleic acid , (1.32 , 0.66 and 1.93%) linolenic acid and (0.86, 0.58 and 0.89 %), arachedic acid .

3-Using GLC the unsaponifiable matters of virgin olive oils contained total hydrocarbons (62.33, 71.84 and 80.12%) in which squalene represented (63.16, 71.08 and 83.27 %) respectively, and sterols (15.63, 18.06 and 19.88%), and  $\beta$  – sitosterol represented ( 77.28 , 76.30 and 81.11 %).

4-Physical and chemical changes which took place during storage of virgin olive oils that packed in 4 different containers at room temperature (at different conditions) were found to be:

4-1- The color of olive oil had slightly decreased during storage period (12 months) in all containers from 35 yellow, 2.6 red and 0.8 blue to 35 yellow, 2.1 red and 0.8 blue for Shemllali variety in (PET, HDPE) respectively in both light and dark conditions and to 35 yellow, 2.4 red and 0.8 blue. In AGb both light and dark conditions and to 2,2 red, 0.8 blue 35 yellow, 2.5 red 0.8 blue 35 yellow in CGb at both light and dark conditions respectively. The color changed from 2.5 red, 1.2 blue, 35 yellow, in all containers for Weteken variety to 2.0 red 1,2 blue and in both PET and HDPE in all conditions (light and dark) respectively. In



both AGb and CGb change had been ( 2.4 red , 1.2 blue and 35 yellow), (2.2 red , 1.2 blue and 35 yellow ) , ( 2.4 red , 1.2 blue and 35 yellow ) respectively in both light and dark Conditions respectively . While the results for Maraki variety change had been from (2.5 red , 0.6 blue and 35 yellow to (2.0 red , 0.6 blue and 35 yellow )in both HDPE and PET at light and dark conditions respectively. And change had been ( 2.4 red, 0.6 blue and 35 yellow) in AGb bottles at different conditions while change had been (2.2 red, 0.6 blue and 35 yellow) at light conditions and (2.4 red, 0.6 blue and 35 yellow ) at dark conditions

4-2 Refractive index of virgin olive oils was not markedly increased during storage in different containers for 12 months.

4-3- Also, Viscosity of virgin olive oils was found to be stable during storage in different containers for 12 month.

4-4-Free fatty acids (% as oleic acid) were increased with the progression of storage period. The minimum increment of free fatty acids content was found in case of oil packed in Amber glass bottles, whereas, the highest increment was found in the oil packed in plastic bottles (HDPE).

4-5-Peroxide value of virgin olive oils increased with increasing the storage period. The lowest increase in peroxide value was obtained with the oils packed in amber glass bottles, (8.21, 8.12 and 7.6 meq / kg oil after 12 months), respectively for three varieties. Whereas, the highest increase in peroxide value was found with the oil packed in

plastic containers (HDPE), since, it reached (40.4, 33.8 and 27.6) respectively after 12 month of storage (at light conditions)

4-6-Iodine value decreased gradually during storage in all different containers (at different conditions). The lowest decrease of all treatments was obtained with the oil packed in Amber glass bottles, whereas, the highest decrease was found with the oil packed in plastic bottles (HDPE). (Light conditions)

4-7-The oil absorption at 232 and 270 nm in the UV region increased gradually during storage in all different containers (at different conditions). The lowest increasing of all treatments was obtained with the oil packed in Amber glass bottles, whereas, the highest increase was found with the oil packed in plastic bottles (HDPE). (light conditions)

4-8-TBA value (as absorbance at 535) increased during storage period in all container types meanwhile the lowest increase was found in amber glass bottles (at dark conditions) whereas, the development of TBA value in plastic bottles was found to be highest than other containers.

4-9-Sensory evaluation of virgin olive oils stored in amber glass bottles for 12 months indicated that oils kept significantly its sensory properties as high as that of virgin olive oils at zero time.

5-The results indicated that, acid value, free fatty acid content and peroxide value of sunflower oil were (0.083, 0.04 % and 1.6 meq/ kg)

### Summary

oil, respectively. Also, iodine value (134.17), The unsaponifiable matter (1.38-%). While saponification number of sunflower oil (193.84)

6- The results indicated that, acid value, free fatty acid content and peroxide value of pomace oil of (Shemllali, Weteken, and Maraki) were (0.89, 0.82 and 0.78), ( 0.44, 0.41, and 0.39, ), ( 17.03, 15. 65. And 15. 39 meq / kg oil) respectively. Iodine values were (81. 31, 80. 25 and 81. 94). The unsaponifiable matters were (1. 96, 2. 24, and 2. 55, %) respectively. Saponification numbers were (196.74, 195.38, and 195.13,). The total phenolic content were (142, 168 and 188 ppm) respectively.

7-Blending crude olive pomace oil with sunflower oil at concentrations of 10, 20, 30, and 40 % increased the oxidative stability of sunflower oil from 6 days to (7.3, 8.2, 8.9 and 9.4days) of ( $M_{50} + W_{25} + S_{25}$ ) to ( 7.8 , 8.6 ,9.2 and 9.5 days) of ( $M_{75} + W_{12.5} + S_{12.5}$ ) and to ( 8.1, 9.0, 9.6 and 10.4 days) of ( $M_{85} + W_{7.5} + S_{7.5}$ ) respectively.