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STUDIES ON THE PRODUCTION AND QUALITY OF PURE OLIVE OIL

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Abstract

Since acidity is taken as a criterion for classifying olive oil into its known quality grades, the lampente olive oil is non edible oil and does not fit human consumption as it is. Because of it's high acidity this oil should undergo refining to decrease the acidity, and blending with virgin olive oil to produce pure olive oil having a maximum acidity in terms of oleic acid of 1.5 gm/100 gm.

Different concentrations of refined olive oil were mixed with extra virgin olive oil (5, 10, 15 and 20%) to produce pur olive oil.

Characteristics of the oils, Absorbency in ultra violet, colour, the fatty acid composition, the unsaponifiable matter components (hydrocarbons and sterols), natural antioxidants as polyphenols and tocopherols and oxidative stability were studied.

The results indicated that blending refined olive oil with extra virgin olive oil improved its characteristics and produced pure olive oil containing higher percentage of natural antioxidants than refined olive oil. Poly-phenols increaed from 116 to 118, 121 and 122 mg/kg and tocopherols increased from 100 to 105, 110, 115, and 121 mg/kg oil in the blends of 5,10,15 and 20% extra virgin oils 20% extra virgin oils, respectively.

The oxidative stability was improved and the induction period increased with the increase of the extra virgin oil levels in the blends. It increased from 6 days in refined oil to 6-7, 7-8 and 8-9 in the blends of 5, 10, 15 and 20%, respectively.

The results indicated of that 20% is the best ratio of extra virgin olive oil added to refined olive oil. At this percentage the characteristics and the organoleptic test are better for the consumer, but economically speaking 5% or 10% are acepted since it increases its nutritional value and matches with the recommended standard.

INTRODUCTION

Pressed olive oils obtained from the fruit 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). If the acidity due to free fatty acids is lower than 1% (expressed as oleic acid) the olive oil is labelled as "extra virgin". If the acidity is hgiher than 3% the oil has to be refined and the refined oil is usually blended with extra virgin olive oil. These mixtures are designated as "olive oil" or "Pure olive oil". Since virgin olive oils are not refined, the phenolic compounds are partly preserved and these compounds are reportedly responsible for their higher stability to autooxidation (Nergiz and Unal 1991).

Trade in olive oil is covered by an international standard that distinguishes between four different grades of virgin olive oil and the trade standard establishes the values for the quality parameters for each of these categories: free acidity (in oleic acid), peroxide value (in meq of oxygen/kg of oil), absorbency in ultraviolet and organoleptic characteristics.

The oils belonging to the first three categories may be used for direct food consumption whereas lampante virgain olive oil is first refined and after blending with a proportion of edible virgin olive oil, can be sold for consumption under the name "olive oil". Refined olive oil is the olive obtained from alteration in the initial glyceridic structure but olive oil is the oil consisting of a blend of refined olive oil and virgin olive oil fit for consumption as it is (looc 1996).

Kiritsakis *et al.* (1983) reported that olive oil is one of the very few if not the only plant which can be consumed in its natural state without being further treated or refined. The oil obtained from healthy mature olive fruits by mechanical means, without any chemical treatment, is called "virgin" and this oil is of the highest quality. Deterioration of oil seeds during storage prior to crushing for oil extraction has been a problem for oil seeds processing industry. If deterioration occurs, the free fatty acid level of the oil contained in the seed increase along with a concomitant decrease in quality and economic value. The quality of olive oil is highly dependent on its degree of acidity. Naturally occuring acidification of olive oil with aging was due to enzymatic hydrolysis more than to autocatalysis (Cantarelli, 1960).

Lipolytic enzymes are involved in many undesirable changes. The acidity of olive is increasing progressively even when the fruit remained on the tree due to the activation of the enzyme lipase. The acidity of the oil is also increased during the period the fruit remain on the collection nets. The reason for the development of acidity in the olives left long on the nets, is that both the endogenous lipases of the fruit and the lipases of the microorganisms that may grow on the fruit are likely to affect the lipolysis. (Kiritsakis and Markakis 1987).

Olive oil is high in oleic acid and contains many phenolic components, it is very stable to autoxidation even in deep frying. Olive oil production in the future is strictly dependent on the decrease in cost of production and an increase in the value of the products; there are many more opportunities for improvments in these areas than in extraction or refining technology which is mostly up to date. Fedeli (1983). Many workers reported that there is a good correlation between the absorbance of olive oil at 232 nm and its degree of oxidation. The conjugated hydroperoxide absorb at 232 nm while at other lengths (270 nm) the secondary oxidation products (aldehydes and ketones) absorb. Conjugated diene and trienes, formed during refining or bleaaching of olive oil, have absorbance at 270 nm. (Montefredine and Luciano 1968), Ninnis and Ninni (1968) Bartolomeo, and Sergio (1969) and Jimenez and Gutierrez (1970).

The intensity of the color formed is used as an indication of the degree of oxidation of olive oil (quality indicator). It may vary among different olive oil extracted at different times. The pigments (Chlorophylls, pheophytins, xanthophylls, and carotenes) mostly present in olive fruit , at harvesting time, are responsible for the color of the olive oil. It is green at the beginning of the season when the olive fruit is still immature and the presence of chlorophyll is dominant. As maturity advances the oil turns golden yellow because of the carotenes present while overripe fruit gives olive oil of a green-light brown color mainly due to pheophytins. The method of the oil extraction also affects the oil color. The Rapanelli-decanter oil is greener than the Rapanelli Sindea oil because the latter contains less chlloropophyll (Kiritsakis, 1982), Koutsaftakis *et al.*, (1979), Carocci, (1963) and Petruccioli (1965).

Baggio *et al.*, (1988) reported that olive oil appeared to be a unique nutritional source of dietary lipids applicable to the treatment of mild hyper cholesterolemia. Its favorable effect on HDL and on apo A serum levels makes it as therapeutically useful as PU FAS (Poly unsat fatty acid) in lowering the cholesterol level. The unique

effect of olive oil (rich in oleic) on serum lipids the favorable ratio between PUFAS (poly unsaturrated fatty acid and antioxidant contents. Vitamin E and its high content of essential fatty acids gave it high biological and nuritional value and made it an excellent source of dietary lipids. Trevisan *et al.*, (1990) showed that consumption of olive oil is inversely correlated with cholesterol and glucose level and blood pressure. Olive oil also increases HDL (high density lipoproteins) cholesterol and has a moderate effect on LDL (low density lipoproteins) cholesterol, promoting in a balanced way a desirable blood lipids profile.

Therefore, this study aims to prepare a good mixture of pur olive oil by blending separately refined olive oil with different concentration of extra virgin olive oil because there are large quantities of lampante olive oil (low price olive oil) in ARE which is not suitable for food consumption.

Also, the physico chemical physico chemical properties, unsaponifiable matter components, fatty acid composition, total phenol and tocopherols and oxidative stability of the blends were determined.

MATERIALS AND METHODS

Materials :

1. Refined olive oil was obtained from Gianaclis Company.
2- Ripe olive fruits variety Coronakii were obtained from a private farm at El-Mansouria, Giza governorate in November 1996. olive oil was extracted by pressing in a new small traditional mill that the Ministry of Agriculture has spread in the areas of olive production and cultivation. Refined olive oil and extra virgin olive oils were mixed for preparing pure olive oil blends of 95:5, 90:10, 85:15, and 80:20 (w/w).
3- Folin-Ciocalteus reagent was obtained from Gerbsaure chemical Co.Ltd. Germany.
4. Tocopherol was obtained from Calbiochem San Diego, California, U.S.A.

Methods :

Analytical Methods:

Refractive index at 25oC, free fatty acids (as oleic acid percent), peroxide value (as millieequivalent/kg oil), Saponification values, Iodine value and unsaponifibale matter percent were determined according to methods described by the A.O.A.C (1980).

Absorbency in ultraviolet:

Specific Extraction E 1% 1cm of olive oil. The u.v. absorption of 1% solution of the oil in cyclohexane in 1-cm cell was measured according to FAO/WHO 1970, at 232 and 270 nm using a Shemadzu spectrophotometer (u.v. vis. 120-02).

Color:

The color of the various samples were determined according to AOCS method (1960) using the Lovibond Tintometer Limited E.

Preparation of the fatty acid methyl esters:

The methyl esters of olive oil was prepared using benzene: methanol concentrated sulfuric acid (10 : 86 : 4) and methylation was carried out for one hour at 80-90^{o}C acording to Stahl (1967).

Identification of the fatty acids methyl esters:

Gas liquid chromatography apparatus (A Pye-Unicum Model 4550) was used for the identification of the fatty acid methyl esters. The conditions used were inditical to those reported by Ismael, (1989). Peak areas were measured using spectrophysic integrator.

Separation and Identification of the unsaponifiable matter components :

The unsaponifiable matter were extracted after saponification of oil at room temperature according to the method outlined by Mordert (1968).

Hydrocarbon and sterol compounds were identified using a Hewlett Packard gas chromatograph (model 5890) as reported by Ismael (1989).

Determination of total polyphenols:

The polyphenols were extracted from the oil by aqueous methanol (60%). then the concentration of total polyphenols in the methanolic extract was estimated with Folin-Ciolcateau reagent. according to the method described by Gutfinger, (1981).

Determination of total tocopherols (as Vitamin E):

Total tocopherols were determined according to The Association of Vitamin Chemist's (1951).

Measurment of stability:

Oven test:

The oven test method suggested by Thompson (1960), was adopted for checking the stability of live oil samples. An oil sample (50 gm) was placed in a (250 ml) beaker covered with a watch glass and incubated at 63±1oC until rancidity took place. Rancidity was periodically assessed every 48 hours through determining the peroxide value.

RESULTS AND DISCUSSION

1- Physical and chemichal properties of extra virgin, refined olive oil and their blends :

Data in Table 1 represent the major physical and chemical properties of extra virgin olive oil, refined olive oil and their blends to produce pure olive oil. The refractive index of extra virgin, refined and pure olive oil with 5, 10, 15 and 20% were 1.4687, 1.4666, 1.4668, 1.4676 and 1.4678 respectively. From the data it could be stated that the refractive index slightly increased in the pure olive oil with the increase of extra virgin olive oil percentage in the blends.

From the results in Table 1, it is clear that the colour of refined olive oil was characterized by a higher degree of yellow units, and the colour of extra virgin olive oil was characterized by a higher degree of red units, blue units and yellow units indicate the presence of a large amount of pigments but the colour of refined olive oil was reduced to a lesser value because the pigments were removed. This decrease in red and blue color may be attributed to slight oxidation during refining. This decrease in red and blue color may be attributed to slight oxidation during refining. This agree with the looc (1996) and Ibrahim (1986).

The iodine value showed a similar trend. It showed a gradual increase with increasing the amount of extra virgin oil added to refined olive oil. The changes in refractive index and iodime value are due to the degree of unsaturation of the fatty acids of added oil (El-Kalyoubi and Mostafa, 1995).

The saponification values were 194.37, 191.86 and (191.99, 192.10, 192.43 and 192.75) for extra virgin, refined olive oil and their blends (pure olive oil with 5,10, 15 and 20%). The peroxide value and unsaponifiable matter % of the blends showed no remarkable changes. The peroxide value of the refined olive oil was 4.58

Table 1. Physico-Chemichal Properties of extra virgin, refined olive oil and their blends.

Phsico Chemical Properties	Extra Virgin olive oil	Refined	blends (Refined / Extra virgin olive oil)			
			95:5 (5%)	90:10 (10%)	85:15 (15%)	80:20 (20%)
Refractive index at 25°C	1.4687	1.4666	1.4668	1.4672	1.4676	1.4678
Colour	red yellow blue	red yellow blue	red yellow blue	red yellow blue	red yellow blue	red yellow blue
	7 70.0 6.3	5.4 79.9 4.8	5.5 79.5 4.9	5.8 79 5.3	6.1 78.4 5.3	6.4 76 5.7
Iondin Value	84.5	81.24	81.40	81.60	81.75	81.90
Saponification Value	194.37	191.86	191.99	192.10	192.43	192.75
Peroxide value mg/kg oil	5.41	4.58	4.65	4.68	4.70	4.73
Unsaponifiable matter %	1.14	1.08	1.082	1.087	1.09	1.093
Free fatty acids	0.74 %	1.32%	1.28%	1.26%	1.22%	1.20%
K 232	1.51	2.32	2.29	2.21	2.17	2.20
K 270	0.17	0.93	0.88	0.84	0.82	0.82

and 4.65, 4.68, 4.70 and 4.73 for the blends of extra virgin and refined olive oil at 5, 20, 15 and 20% levels. As known, the quality of olive oil greatly depends on its degree of acidity that should be below 3.3% to be used as food. There fore, it should be stated here that the acid value should be considered as one of the constants characterising edible oils specially olive oil. These results reveal that extra virgin olive oil has acidity less than 1% (Teresa *et al*, 1995) and the acidity of the refined oil was 1.32% which is higher than the refined oil blended with extra virgin olive oil (5, 10, 15 and 20%) to produce "pure olive oil" since the acidity was 1.28, 1.26, 1.22 and 1.20 respectively; these blends with good quality virgin oil can be consumed as separate products.

UV absorbance measurements of different sample of extra virgin olive oil, refined olive oil and pure olive oil at 5, 10, 15 and 20% levels were measured at a wave length of 232 nm and 270 nm (as ultraviolet spectrophotometric analysis) and the results are tabulated in Table 1.

From these results, it could be noticed that E 1% 1cm of the samples at 232 nm were 1.51, 2.32, (2.29, 2.21 and 2.20). While at 270 nm were 0.17, 0.93, 0.88, 0.84, 0.82 and 0.82) for extra virgin olive oil, refined olive oil, (pure olive oil at 5,10,15 and 20%), respectively. It is clear that the absorbances of olive oil sample decrease by increasing the wave length from 232 nm (specific for conjugated diene compounds) to 270 nm (specific for triene compounds). Thus high absorbance in refining olive oil is related to olive oil oxidation or to refining process or to both of them and low absorbance at 232 nm, 270 nm correspond to good olive oil quality.

2- Fatty acid composition of olive oil:

Gas liquid chromatography analysis of fatty acids of extra virgin olive oil, refined olive oil and their blends are presented in Table 2. From these data it can be stated that the saturated fatty acid contents were 15.69% and 17.89 for refined and extra virgin olive oil but it were 15.82%, 16.00%, 16.13% and 16.30% for 5,10, 15 and 20% of pure olive oil blends. While the unsaturated fatty acid were 84.31, and 82.11 for refined and extra virgin olive oil and 84.18, 84.87% for the pure olive oil blends respectively. It can be noticed that the saturated fatty acid content in refined olive oil decrease as the result of refining. This might be due to the removal of some saturated triglycerides during refining process (El-Agaimy *et al*1990). The major fatty acids in all samples were oleic acid, palmitic acid and lino-

Table 2. Fatty acids composition, %, of extra virgin, Refined olive oil, and their blends.

Phsico Chemical Properties	Extra Virgin olive oil	Refined	blends (Refined / Extra virgin olive oil)			
			95:5(5%)	90:10 (10%)	85:15 (15%)	80:20 (20%)
Palmitic (16:0)	16.50	13.25	13.43	13.61	13.84	14.07
Palmito oleic (16:1)	1.93	2.40	2.37	2.27	2.25	2.20
Stearic (18:0)	1.39	2.44	2.4	2.39	2.29	2.23
Oleic (18:1)	73.43	74.83	74.78	74.71	74.65	74.58
Linoleic (18:2)	6.36	6.60	6.58	6.58	6.55	6.51
Linolenic (18:3)	0.39	0.48	0.48	0.44	0.42	0.41
Total Saturated	17.89	15.69	15.82	16.00	16.13	16.30
Total Unsaturated	82.11	84.31	84.00	84.00	83.87	83.70

leic acid. Increasing oleic acid in the oil and increasing its level in the diet is recommended nutritionally and is of unquestionable interest in preventive medicine (Jacoto, 1994) and (Ibrahim *et al* 1995). and it is suggested that dietary oils with a greater proportion of mono unsaturated fatty acids may provide the best balance for lowering cholesterol levels and reducing the suseptibility for lipid peroxidation damage (Jacob, 1994) and Ibrahim *et al* (1995).

Unsaponifiable matter components of extra virgin and refind olive and their blends :

From the results presented in Table 3, It can be observed that squalene is the major hydrocarbon in all samples of olive oil. It was 66.29% and 62.9% for the extra virgin and refined olive oil respectively and 63.13, 63.42 and 63.57% for the 5,10, 15 and 20% bicads of pure olive oil respectively. This is in agreement with the findings of Kiritsakis (1991); Lanzon *et al* (1994) and Ibrahim *et al* (1995). As for the sterols, B-sitosterol is the main sterol in extra virgin and refined olive oil and all blends; They were 15.51, 16.20, 16.15. 16.06% respectively. Similar results were mentioned by Awatif (1994); Dawood (1993).

Table 3. Unsaponifiable matter components of Extra Virgin, Refined Olive oil and their blends.

Component	Extra Virgin olive oil	Refined olive oil	blends Refined /Extra virgin olive oil			
			5%	10%	15%	20%
Hydrocarbons						
C18	0.03	0.22	0.20	0.20	0.19	0.16
C20	0.50	0.73	0.72	0.71	0.69	0.65
C22	2.57	1.33	1.39	1.44	1.46	1.5
C24	0.54	0.86	0.84	0.83	0.81	0.80
C26	0.32	0.49	0.47	0.46	0.45	0.44
C28	6.36	5.50	5.56	5.59	5.64	5.68
Squalene	66.29	62.9	63.13	63.29	63.42	63.57
C30	3.19	4.52	4.44	4.40	4.37	4.33
Sterols						
Campsterol	2.00	3.63	3.55	3.45	3.41	3.36
Stigmasterol	2.69	3.62	3.55	3.50	3.47	3.44
B sitosterol	15.51	16.20	16.15	16.13	16.09	16.06
Total Hydrocarbons	79.8	76.55	76.75	76.92	77.03	77.13
Total Sterols	20.2	23.45	23.25	23.08	22.97	22.86

Natural antioxidants of extra virgin, refined olive oil, and their blends:

The total polyphenols contents of olive oil samples are given in Table 4. It can be noticed that the polyphenols in extra virgin olive oil were 150 mg/kg oil. It was 116 mg/kg oil in the refined olive oil. Thus, virgin olive oils contain polyphenols which are usally removed from other edible oils in the various fefinings stages, Roncero *et al.*, (1973). The blends contain 118, 119, 121, 122 mg/kg oil for the 5, 10, 15 and 20% of pure olive oil. High polyphenol content was associated with a high resistance to oxidation of the oils. A linear relationship was found between polyphenol content and the oxidative stability of the virgin oils during storage. After removel of the polyphenols the oxidative stability of the oils decreased considerably and seemed to depend on polyunsaturated fatty acid concentration (Gutfinger 1981). Also from data in Table 4 it can be seen that the content of tocophenols in extra virgin olive oil is remarkably higher (198 mg/kg oil) than in refined olive oil (100 gm/kg oil). From these results it is evident that the tocopherols content in pure olive oil blease were 105, 110, 115 and 121 mg/kg oil. It is evident that refined olive oil is characterized by the lowest content of tocopherol. as reported by Fedeli (1988). Also, andrikopoulos *et al.* (1989) found that virgin olive oil, olive pomace oil, and refined olive oil contained an average of 113, 81, 156 and 37 mg/kg of tocopherol. Ninnis *et al.* (1969) reported that the tocopherol content of olive oil can be used for detecting adulteration of the oil with seed oils. The oxidative stability of olive oil is related to the presence of tocopherols.

Table 4. Natural antioxidants of extra virgin, refined olive oil, and their blends.

Component	Extra Virgin olive oil	Refined olive oil	blends Refined /Extra virgin olive oil			
			5%	10%	15%	20%
Total Polyphenols mg/kg	150	116	118	119	121	122
Total tocopherols mg/kg	198	100	105	110	115	121

Oxidative stability:

Oxidative stability evaluations were done by storage of the oil at 63oC and the

determination of peroxide value. From the results summarized in Table 5 and fig 1, it can be seen that the oxidative stability of extra virgin olive oil was markedly higher than the refined olive oil since they were 11 days and 6 days, respectively. The high resistance to oxidation of the extra virgin olive oil may be attributed to its high polphenols and tocopherol contents which are considered natural antioxidants. These results are in agreement with those reported by Gutfinger (1981). Addition of extra virgin olive oil to refined olive oil at four different concentrations of 5, 10, 15 and 20% improved the oil stability. The degree of effectiveness was dependent on the concentration used to produce pure olive oil since the tability increased from 6 from days to 6-7, 7, 8 and 8-9 days. This finding indicated that refining steps caused a pronounced decrease in the induction period of extra virgin oil since the natural antioxidants are removed or destroyed during refining steps. These results are in agreement with that reported by Woerfel (1981) and Ibrahim (1986). The peroxide value serves as an indication of oil quality. Generally it can be stated that the peroxide value is an indication o fprimary level of oil oxidation (Augustin and Berry, 1983). It could be recommended that addition of extra virgin olive oil to refined olive oil may be a step towards the production of better quality pure olive oil, more stable, higher nutritional value that suits the edible consumption.

Table 5. Induction period of extra virgin, refined olive oil and their blends.

	Extra Virgin olive oil	Refined olive oil	blends Refined /Extra virgin olive oil			
			5%	10%	15%	20%
Induction Period (days)	11	6	6-7	7	8	8-9

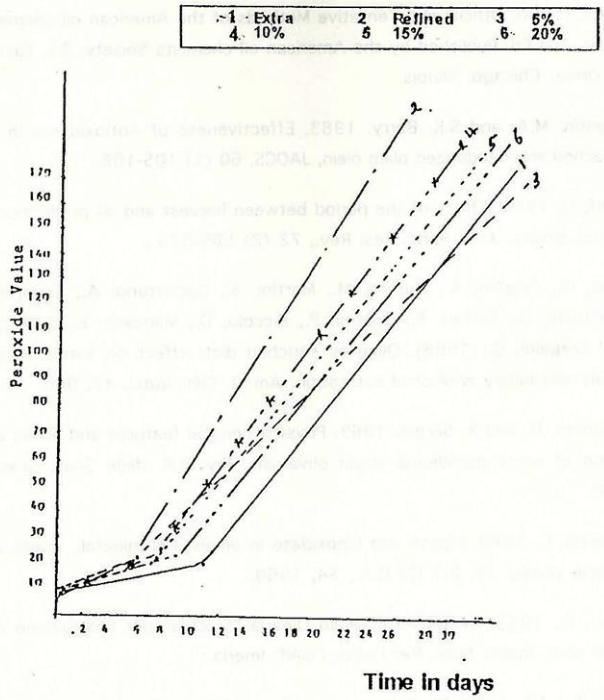

Fig. 1. Stability of extra virgin olive oil, refined olive oil and their blends (pure olive oil with 5%, 10%, 15% and 20%)

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دراسات عن إنتاج وجودة زيت الزيتون النقى

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تم خلط نسب من زيت الزيتون المكرر مع زيت الزيتون البكر الممتاز بنسبة ٥، ١٠، ١٥، ٢٠٪ لإنتاج زيت زيتون نقى Pure Olive Oil وذلك نتيجة لوجود الزيت اللمبانتى فى ج.م.ع بكمية كبيرة لإرتفاع درجة حموضته بسبب عوامل كثيرة. وحيث أن درجة زيت الزيتون تصنف على حسب درجة الحموضة فى الزيت لذلك فهو زيت غير صالح للغذاء فتجرى عليه عمليات تكرير لخفض نسبة الحموضة. ثم إستعمال الزيت المكرر فى إنتاج زيت زيتون يصلح للأكل.

ولقد تم دراسة صفات زيت الزيتون النقى الطبيعية والكيمائية ودراسة الإمتصاص بالـ U.V وكذلك اللون وتركيب الأحماض الدهنية والمواد الغير قابلة للتصبن والمواد الطبيعية المضادة للأكسدة كالفينولات والتوكوفيرولات وكذلك تم تقييم الثبات عن طريق تقدير رقم البيروكسيد .

وأوضحت النتائج أن إضافة زيت الزيتون البكر الى زيت الزيتون المكرر يعمل على تحسين صفاته وإنتاج زيت زيتون نقى تزداد به نسبة المواد الطبيعية المضادة للأكسدة كالفينولات حيث إرتفعت من ١١٦ إلى ١١٨، ١١٩، ١٢١، ١٢٢ ملجم /كجم. كما إرتفعت نسبة التوكوفيرولات من ١٠٠ الى ١٠٥، ١١٠ ، ١١٥ ، ١٢١ ملجم / كجم فى الزيت المكرر والمخلوط بنسبة ٥ ، ١٠ ، ١٥ ، ٢٠ ٪ على التوالى وأيضا إرتفعت درجة ثبات الزيت كلما إرتفعت نسبة الخلط من ٥ إلى ٢٠٪ حيث زادت من ٦ يوم فى الزيت المكرر الى ٦ - ٧ ، ٧ - ٨ ، ٨ - ٨ ، ٨ - ٩ يوم للتركيزات من ٥ ، ١٠ ، ١٥ ، ٢٠٪ على التوالى.

كما أنه لوحظ أنه أفضل نسبة للخلط هى ٢٠٪ عند إضافة زيت الزيت البكر الممتاز الى زيت الزيتون المكرر وهذه النسبة تعمل على تحسين صفاته والخواص الحسية للمستهلك.

ولكن وجد أنه إقتصادياً يمكن الخلط بنسبة ٥ أو ١٠٪ حيث أنها تخفض فى السعر وتعمل أيضاً على رفع قيمته الغذائية والصحية كما أنها تتفق مع الصفات القياسية المقررة من المجلس الدولى لزيت الزيتون.