EFFECT OF DIFFERENT MALAXATION TIMES AND TEMPERATURES ON THE EFFICIENCY OF EXTRACTION AND QUALITY OF OLIVE OIL

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Abstract

This research was involved in studying the effect of different malaxation time 50, 60 and 70 min. and malaxation temperatures 25, 30 and 35 °C of pastes in a mixer on the quality and quantity of olive oil at industrial scale using a three-phase continuous extraction system. This was in order to reach a minimum oil loss in the pomace and a high content of polyphenols and tocopherols in oil. Also, to obtain the highest, percent of oil from different malaxed olive pastes. The results obtained showed that using malaxation time of 60 min gave the highest oil percent from olive paste and the least residual quantity in pomace, while malaxation time of 50 min gave better characteristics than all samples taken from different stages of processing line, followed by malaxation for 60 min.

Results showed that oil percent is higher when malaxed at 35 °C, than malaxed samples at 25 °C and 30 °C. Free fatty acid, peroxide value and ultraviolet absorption recorded a slight increase for all samples by increasing temperature. Malaxed sample at 25 °C caused a higher increase in natural antioxidant and stability compared with that malaxed at 30 °C and 35 °C.

The best quality of oil is obtained from horizontal centrifugal decanter compared with that from vertical separators (1) and (2) (oily and water phases).

INTRODUCTION

Virgin olive oils are known to be more resistant to oxidation than other edible oils because of their content of natural antioxidants and lower unsaturation levels. The higher number of double bonds in fatty acids, the shorter the induction period for oil autoxidation. The stability of virgin olive oils is due to their natural phenolic compounds (Cinquanta, *et al.*, 2001).

Olive oil can be considered a good source of natural antioxidants. Moreover, the polyphenol and tocopherol contents of virgin olive oils were also found useful for the classification of the different commercial monovarietal oils when analysing the data by chemometric methods (Garcia, *et al.*, 2003).

Di Giovacchino *et al.* (2002) mentioned that the efficiency of malaxation depends upon the rheological characteristics of olive paste and upon the technological parameters of operation, such as time and temperature of malaxation. Malaxation reduced phenol concentration in oil and in by- products (Servili *et al.*, 1999).

The concentration of the majority of the oil constituents is changed during the malaxation. The oil yield increased substantially up to 45 min of paste malaxation times. Beyond 60 min, the yield tended to decrease (Ranalli *et al.,* 2003). Total phenol content of olive oils changed significantly when the malaxation time of olive paste increased from 15 to 90 minutes. (Di Giovacchino and Serraiocco, 2002).

The malaxation stage reduced the concentration of orthdiphenols in oil 50 – 70 % while the concentration of the monorthiphenols remained constant (Aranzazu *et al.,* 2001).

It has been reported that at laboratory scale that the concentration of phenolic compound in oil diminishes with increasing malaxation time (Servili *et al.,* 1999). Esther *et al.* (2001) also reported that malaxation reduced the concentration of phenolic compounds in the paste, oil and vegetable water.

There is no widely accepted explanation for the decrease of phenolics in oil during malaxation of the olive paste although it has been suggested that certain enzymes such as polyphenols oxides and peroxidase may play an important role in this phenomenon. (Servili *et al.*, 1999).

Malaxation for 60 minutes gave the highest yield, meanwhile, malaxation for 15 minutes produced better characteristics for olive oil for all olive varieties (Azza, 2002).

Di-Giovacchino (1991) found that the highest oil yield was obtained after an optimal mixing time of 60 minutes, also mixing temperature was very important because higher temperatures produce significant increases in extraction yield and also gave a higher velocity constant.

The amount of phenols in olive oil depends on the type of extraction system and the temperature during malaxation (Servili *et al.*, 1994). The concentration of phenolic compounds present in olive oil is strongly affected by the extraction condition used during processing (Servili, *et al.,* 1994 and Morales, *et al.,* 1999). The extraction conditions were not found to influence tocopherol level of virgin olive oil, (Psomiadou *et al.,* 2000).

The extraction conditions of virgin olive oil have a great influence on its sensory quality. During the centrifugation process, temperature and time of malaxation can be altered to potentially affect quality (Morales and Aparicio, 1999).

Extraction technology and kneading temperature are known to effect oil yields and the properties of the oils and oil by-products (Hermosos Fernandez *et al.*, 1994).

Yield, pomace moisture content and pomace oil content from comparative extraction trials at 30 and 45 °C have been reported under industrial conditions (Koutsatfakis and Stefanoudaki, 1997).

The objective of this work is to study the effect of different malaxation times and temperatures on the efficiency of extraction and quality of olive oil.

MATERIALS AND METHODS

Materials:

Olive fruits variety Coratina was purchased from a private farm in Ismalia and the processing line of Salhia was used in this study during 2002 season.

Samples from four different points of processing line were obtained. These points are illustrated in figure (1)

Sample (1): Represented the horizontal centrifugal (decanter) which contained the oil and traces of water.

Sample (2): Represented the vertical separator (1) which contained the oil without water (oily phase).

Sample (3): Represented the vertical separator (2) which contained the oil as trace with the vegetable water after being separated by centrifuging (water phase).

Sample (4): Represented the olive pomace obtained. This sample would determine the residual oil in pomace. Hence, it may determine the efficiency of the extraction process.

Methods:

1. Malaxation time:

Olive processing at industrial scale was carried out using a three-phase continuous extraction system. Olives (1500 kg) were crushed by using an Inox hammer mill, operating at 3000 rpm. and equipped with a sieve of -5-mm holes. Malaxation of pastes was made in a mixer at 14 rpm, and 30 °C for 50, 60 and 70 min. Solid-liquid separation of the paste into oil, pomace and vegetable water was performed by a three phase centrifugal decanter working at 3000 rpm. Finally, a vertical centrifuge [Separators (1) and (2)] operating at 6200 rpm and fed with 0.25 lit water/kg oily must, was used to remove the remaining solids from the must. Oils from the decanter and separators (1) and (2) (oily and water phase) were filtered through sodium sulfate before analysis.

2. Malaxation temperature :

Malaxation of pastes was made in a mixer (14 rpm) at 25 °C, 30 °C and 35 °C for 50 minutes. Solid-liquid separation of the paste into oil, pomace and vegetable water was performed by a three phase centrifugal decanter working at 3000 rpm. Finally, a vertical centrifuge [Separators (1) and (2)] at 40 °C operating at 6200 rpm and fed with 0.25 lit tap water/Kg st, was used to remove the remaining solids from the must. Oils from the decanter and separators (1) and (2) (oily and water phase) were filtered through sodium sulfate before analysis.

Physical and chemical properties:

Refractive index at 25 °C, free fatty acid (as oleic percent) and peroxide value were determined according to methods described by the A. O. A. C. (1990).

Absorbance in ultraviolet: Ultraviolet and visible spectra were conducted using Pye Unicum Double Beam Recording Spectrophotometer model SP 1600 as described by Kates (1972). The sample was dissolved in freshly distilled cyclohexane and the absorption was taken at 232 and 270 nm.

Stability: The stability of samples was determined by the Rancimat method at 100 °C with an air flow rate of 20 l/hr according to the method described by Mendez *et al.* (1997).

Total polyphenols: The polyphenols were extracted from the olive oil by aqueous methanol (60v/v), and then concentrated. Total polyphenols in the methanol extract were determined with a folin-ciolateaus reagent according to the method described by Gutfinger (1981).

Total tocopherols: The total tocopherols content of the oil samples were determined in the virgin olive oils according to the method of Wong *et al.* (1988).

Total polyphenols in vegetable water: The total polyphenols were extracted from 14 ml vegetable water by 4 x 20 ethyl acetate and then 4 x 19m n-butanol, the combined extracts were filtered through Na_2SO_4 and evaporated under vaccum, leaving a brown residue, which was redisolved in MeoH. (1 ml). (Capasso. *et al.,* 1992) and then estimated according to the method described by Gutfinger (1981).

Pomace samples: Pomace samples which were obtained from the three-phase centrifugal decanter were dried in oven at 60 °C till constant weight. Then, the oil content was determined according to the method described by A. O. A. C. (1990). Another amount for pomace in each case was used for oil extraction, and the yielded crude oil was stored in the dark at low temperature (4 - 5 °C).

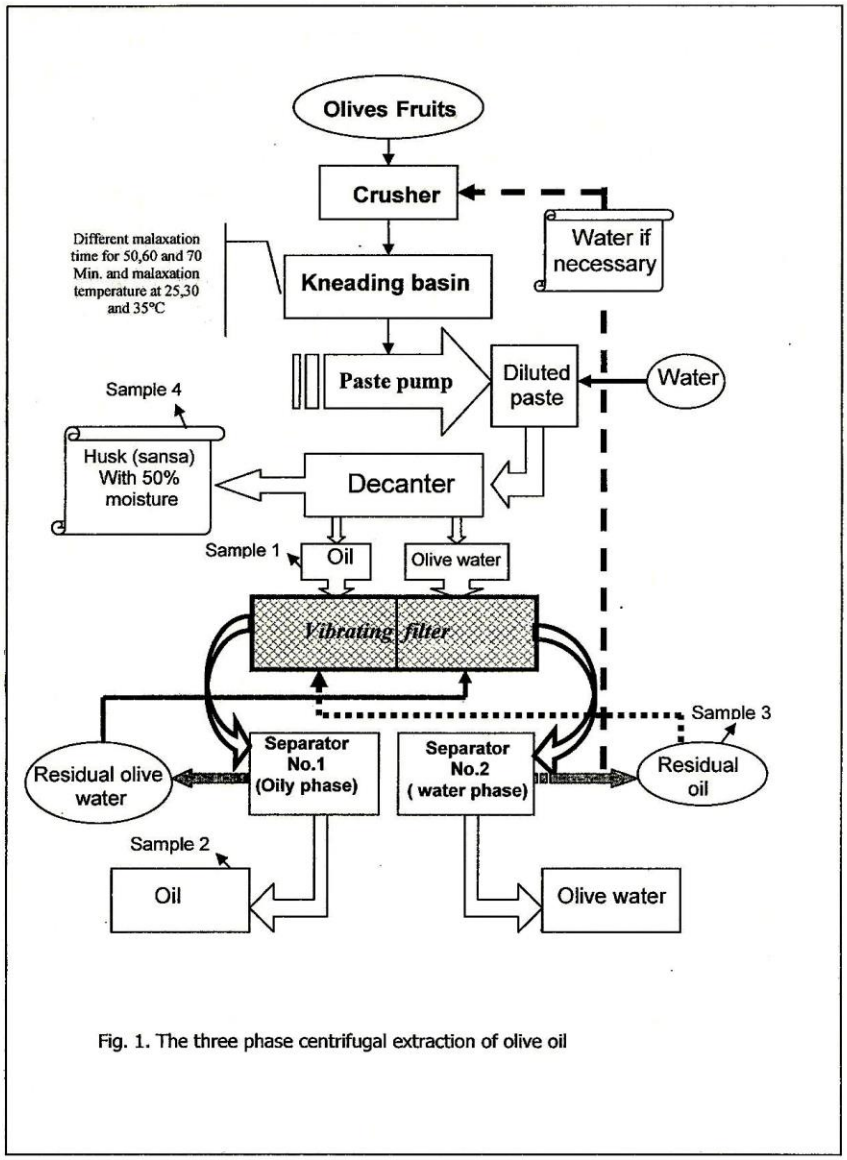

Fig. 1. The three phase centrifugal extraction of olive oil

RESULTS AND DISCUSSION

Physical and chemical properties of Coratina olive oil.

Results in Table 1 show the physical and chemical charateristics of Coratina olive oil, which was obtained by crushing in the laboratory. From the data in this table, it could be observed that the refractive index, FFA, peroxide value, UV absorbance at 232 and 270 nm, total polyphenols, total tocopherols and stability of Coratina olive oil were, 1.4683, 0.68 %, 2.11 meq/kg oil, 0.290 and 0.050, 381 ppm, 284 mg/kg and 40.0 hr., respectively. Also fatty acids composition of Corationa olive oil are presented in Table 6. Separation and determination of fatty acid methyl esters were carried out by GLC to identify their types and amount. From the results, it could be noted that 5 fatty acids were identified in Coratina olive oil. Palmitic acid was the dominating saturated fatty acid and represented 15.75 %, followed by stearic acid which was 1.12 %. On the other hand, oleic acid represented the major unsaturated fatty acid (69.42 %), followed by linoleic acid, (12.90 %), and linolenic acid which was the lowest unsaturated fatty acid (0.75 %).

These results are in agreement with those reported by Cinquanta *et al.,* (2001).

Effect of different malaxation time and temperature on the total lipids of the paste and the pomace of Coratina olive:

The extraction condition of virgin olive oil have a great influence on its quality, so, the effect of different malaxation time 50, 60 and 70 mintues and malaxation temperatures at 25, 30 and 35°C. were determined and the obtained results are shown in Table 2.

Data in Table 2 indicated that the malaxation for 60 mintues gave the highest yield of oil (44.78 %), followed by malaxation for 70 min 44.15 %, then malaxation for 50 min (43.78 %). The results also showed that, the least residual quantity of oil in the pomace (9.65 %) was obtained when malaxation was carried out for 60 min which gave the highest extraction rate. This increase in oil content of Coratina olive by increasing malaxation time to 60 and 70 min compared to the malaxation for 50 min may be due to the formation of a large quantity of minute oil droplets coalesced if droplet diameter is greater than 30 mm which can then be easily extracted as reported by Azza (2002) who gave similar results.

On the other hand, malaxation temperature 35 °C at gave the highest oil percent of olive paste, and the least residual quantity of pomace oil. Similar observation was reported by Di-Giovacchino (1991) who found that a higher temperature produces significant increase in extraction yield.

Effect of different malaxation time and temperature on the physical and chemical properties of virgin olive oil;

Tables 3 - 6 show the changes in the physical and chemical properties of olive oils taken from different stages of production line, horizontal centrifugal (decanter) and vertical separators (oily and water phases) resulting from different malaxation times of 50, 60 and 70 minutes and malaxation temperatures at 25, 30 and 35 °C.

From the data in Tables 3 - 6 it could be noticed that, the acid value, peroxide value, UV absorbance at 232 and 270 mm, increased by increasing malaxation time and malaxation temperature for all samples taken from the decanter and separators (1) and (2) This increase in the previous parameters related to the oxidative degradation and hydrolysis of oils by increasing malaxation time and temperature.

These results are quite in agreement with those obtained by Morales and Aparicio (1999). Also these results are in agreement with those reported by Azza (2002), who stated that the increase of malaxation during olive oil extraction caused an increase in its peroxide value

The sample taken from the decanter recorded a better quality as compared to the other samples taken from the separators, oily and water phases (Tables 3, 4 and 5).

Effect of different malaxation time on the physical and chemical characteristics of pomace oil:

Data in Table 7 show the effect of different malaxation time (50, 60 and 70 min) on the physical and chemical characteristics of oil extracted from pomace.

According to the results given in the table, it could be observed that the refractive index, free fatty acids, peroxide value and UV absorbance at 232 and 270 nm increased by increasing the malaxation time. This increase in all values may be due to the extraction method of the oil from pomace. On the other hand, the stability at 100 °C decreased by increasing malaxation time. This decrease may be due to the decrease of its natural antioxidant (polyphenols and tocopherols) as shown in Table 7.

Effect of different malaxation times and temperatures on the natural antioxidants of olive oil:

The phenolic compounds constitute an important group of naturally occurring compounds in the plant. The amount of phenolic compounds in virgin olive oil is an important factor when evaluating the oil quality because natural phenols improve its resistance to oxidation.

From the results in Table 8, it could be noted that the total polyphenols, total tocopherols and stability of samples taken from decanter, and separators (1) and (2), were decreased by increasing the malaxation time. This decrease in total polyphenols may be due to certain enzymes such as polyphenoloxidase and peroxidase which may play an important role in this phenomenon (Servili *et al.,* 1999). These results are in agreement with those reported by (Servili *et al.,* 1999 and Esther *et al.,* 2001).

Also the natural antioxidant content of virgin olive oil is significantly affected by the temperature used during extraction through a three-phase decanter. Results in the same table show that total polyphenols and α-tocopherols of olive oil were decreased as a results of increasing extraction temperature. Similar results have been reported in some studies (Morales *et al.*, 1999 and Servili *et al.*, 1994). Also induction period (stability) was decreased in olive oil by increasing the extraction temperature.

From the same data it could be noticed that the total polyphenols of samples taken from separator (2) (water phase) recorded a higher decrease as compared with samples taken from horizontal centrifugal (decanter) and separator (1) (oily phase). The decrease in total polyphenols of the water phase may be explained by their water- solubility since higher water/paste ratio are used in triple - phase centrifugation, and therefore large amount of phenols are eliminated with water wastes (Salvador *et al.*, 2003 and Di-Giovacchino *et al.,* 1994).

Effect of different malaxation time and temperature on the total polyphenols of vegetable water:

The data in Table 9 indicated that the increase of malaxation time from 50 to 70 min, caused a slight decrease in total polyphenols of waste water. Similar results were found by Esther *et al.*, (2001).

From the data in this table, it could be observed that, total polyphenols of vegetable water was decreased by increasing the temperature during extraction. This decrease may be due to the increase of polyphenols solubility in oily phase with the increase of mixing temperature (Di- Giovacchino, 1999).

Table 1. Physical and chemical properties of Coratina olive oil.

Physical and chemical properties	Coratina olive oil
Refractive index at 25 °C	1.4683
Free fatty acid (%)	0.68
Peroxide value (meq/kg)	2.11
Diene at 332 nm	0.29
Triene at 270 nm	0.050
Total polyphenols (ppm)	381
Total tocopherol (mg/kg)	284
Stability at 100 °C (hr)	40
Fatty acidcomposition (%):-	
Palmitic acid	15.75
Stearic acid	1.12
Oleic acid	69.42
Linoleic acid	12.96
Linolenic acid	0.75

Table 2. Effect of different malaxation times and temperatures on the oil percent from the olive paste and pomace.

Malaxation time (min).	Oil percent (dry weight)	
	Olive pomace	Olive paste
50	10.95	43.78
60	9.65	44.78
70	9.84	44.15
Temperature		
25 °C	11.24	45.06
30 °C	10.86	45.89
35 °C	9.69	46.31

Table 3. Effect of different malaxation time and temperature on the refractive index of olive oil.

Sample	Refractive index at 25°C					
	Malaxation time (min)			Malaxation temperature (°C)		
	50	60	70	25	30	35
Oil from decanter	1.4683	1.4683	1.4682	1.4683	1.4684	1.4687
Oil from separator (1)	1.4683	1.4683	1.4683	1.4683	1.4683	1.4684
Oil from separator (2)	1.4683	1.4683	1.4683	1.4683	1.4689	1.4685

Table 4. Effect of different malaxation times and temperature on the free fatty acid of olive oil.

Sample	Free fatty acids (%)					
	Malaxation time (min)			Malaxation temperature (°C)		
	50	60	70	25	30	35
Oil from decanter	0.38	0.48	0.58	0.68	0.70	0.76
Oil from separator (1)	0.47	0.53	0.59	0.68	0.72	0.76
Oil from separator (2)	0.58	0.60	0.64	0.68	0.76	0.93

Table 5. Effect of different malaxation time and temperature on the peroxide value of olive oil.

Sample	Peroxide value (meq/kg oil)					
	Malaxation time (min)			Malaxation temperature (°C)		
	50	60	70	25	30	35
Oil from decanter	2.00	2.13	3.11	3.11	3.20	3.40
Oil from separator (1)	2.78	2.90	3.41	3.45	3.53	3.61
Oil from separator (2)	3.33	3.90	4.17	3.42	3.74	3.90

Table 6. Effect of different malaxation time and temperature on the Diene at 332 nm and Triene at 270 nm of olive oil.

Sample	Malaxation time					
	50 min		60 min		70 min	
	Diene at 332 nm	Triene at 270 nm	Diene at 332 nm	Triene at 270 nm	Diene at 332 nm	Triene at 270 nm
Oil from decanter	0.090	0.048	0.290	0.050	0.330	0.052
Oil from separator (1)	0.330	0.050	0.350	0.051	0.370	0.055
Oil from separator (2)	0.360	0.054	0.370	0.056	0.390	0.060
Sample	Malaxation temperature					
	25 °C		30 °C		35 °C	
	Diene at 332 nm	Triene at 270 nm	Diene at 332 nm	Triene at 270 nm	Diene at 332 nm	Triene at 270 nm
Oil from decanter	0.216	0.060	0.264	0.062	0.281	0.170
Oil from separator (1)	0.200	0.065	0.315	0.070	0.385	0.072
Oil from separator (2)	0.280	0.068	0.355	0.075	0.065	0.080

Table 7. Effect of different malaxation time on the physical and chemical properties of crude pomace olive oil.

Physical and chemical properties	Malaxation time (min)		
	50	60	70
Refractive index at 25 °C	1.4685	1.4686	1.4688
Free fatty acid (%)	0.900	0.9500	0.9500
Peroxide value (meq/kg)	14.69	15.500	15.9000
Diene at 332 nm	0.830	0.8500	0.9000
Triene at 270 nm	0.340	0.5000	0.5200
Stability at 100 °C (hr)	6.97	4.9000	4.5800
Total tocopherol (mg/kg)	71.00	60.0000	0.5700

Table 8. Effect of different malaxation times and temperatures on total polyphenols, total tocopherols and stability of olive oil.

Sample	Malaxation time (min)			Malaxation temperature (°C)		
	50	60	70	25	30	35
Total polyphenols (ppm)						
Oil from decanter	381.23	373.44	353.29	281.15	352.59	302.68
Oil from separator (1)	377.14	364.42	347.68	330.50	315.30	294.34
Oil from separator (2)	374.59	362.69	340.00	287.00	280.00	270.00
Total tocopherols (mg/kg)						
Oil from decanter	384.00	274.00	260.00	323.0	300.0	288.0
Oil from separator (1)	368.00	245.00	230.00	284.0	268.0	266.0
Oil form separator (2)	251.00	230.00	220.00	260.0	250.0	245.0
Stability at 100 °C (hr)						
Oil form decanter	41.23	39.20	38.36	39.80	26.50	34.32
Oil from separator (1)	39.92	38.20	36.54	32.22	31.00	30.00
Oil from separator (2)	38.75	36.95	35.47	28.60	27.40	25.60

Table 9. Effect of malaxation times and temperatures on the total polyphenols in vegetable water.

Malaxation time (min)	Total polyphenols (ppm)
50	374.68
60	373.180
70	372.125
Malaxation temperature (°C)	
25	478.61
30	477.32
35	474.07

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تأثير التقليب لفترات مختلفة وحرارة الخلط على كفاءة الاستخلاص وجودة زيت الزيتون

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يتضمن هذا البحث دراسة تأثير زمن التقليب لفترات مختلفة ودرجة وحرارة الخلط المختلفة على كمية وجودة زيت الزيتون فى المعاصر التى تعمل بنظام الإستخلاص المستمر بطريقة الـــ Three - phase لكى نصل إلى أقل فاقد من الزيت فى الكسبة وأعلى معدل من الفينولات الكليـــة والتوكوفيرولات فى الزيت المنتج وأيضاً على أعلى نسبة من الزيت في عجينة الزيتون .

توضح النتائج المتحصل عليها أن إستخدام التقليب لمدة ٦٠ دقيقة أعطى أعلى نسبة زيـــت مستخلصة من عجينة الزيتون وأقل كمية فاقد فى الكسبة وبينما التقليب لمدة ٥٠ دقيقة أعطى أحسن خواص لكل العينات التى تم أخذها من مراحل مختلفة من خط الإنتاج يليها التقليب لمدة ٦٠ ق .

أيضا توضح النتائج ارتفاع نسبة الزيت المستخلص من العينة التى تم خلطها على درجة ٣٥° م مقارنة بالعينات التى تم خلطها على درجة ٢٥° م ٣٠و° م. كما سجلت درجة الحموضـــة والبيروكسيد والقياس فى منطقة الـــ U.V. زيادة بسيطة بزيادة درجة حرارة الخلط .العينات التى تم خلطها على درجة حرارة ٢٥ ° م أدت إلى زيادة فى مضادات الأكسدة الطبيعية والثبات مقارنة بالعينات التى تم خلطها على درجة حرارة ٣٠°م و ٣٥°م

أيضا توضح النتائج جودة الزيت المأخوذ من الـــديكانتر مقارنـــة بـــالزيوت المـــأخوذة السيبارتور ١ ، ٢ (الطور الزيت والطور المائى) .