International Journal of Scientific and Technological Research
ISSN 2422-8702 (Online)
Vol 4, No.3, 2018

Physicochemical Characteristics of Extra Virgin Olive Oils Obtained By Ultrasound Assisted Extraction from Different Olive Cultivars

Alev Yuksel Aydar
Department of Food Engineering, Faculty of Engineering,
Manisa Celal Bayar University, Muradiye, Manisa 45140 Turkey
E-mail: alevyuksel.aydar@cbu.edu.tr

Abstract

In this study, the influence of malaxation time combined by ultrasound on the extraction yield, oxidative and quality characteristics of Extra Virgin Olive Oils extracted from of Edremit, Gemlik and Uslu cultivars were studied. The extraction yield, free acidity, peroxide value, p-anisidin value, total oxidation value, K232 and K270, total phenol compound , the content of total chlorophyll and total carotenoid compounds were determined. Different sonication and malaxation time combinations did not induce difference (P>0.05) in the Edremit oil yield and extractability indexes However, they were significantly different in Gemlik and Uslu oils. Oils obtained from olive pastes treated with 8 minute ultrasound improved Uslu oil yield 1.74% and the oil extractability 10.32%. Moreover, the amounts of chlorophylls and carotenoids were highest in samples subjected to 8 minutes sonication treatment whereas they were lowest in olive oils subjected to 12 minute ultrasound. Oils extracted by 8 minute sonication showed higher oxidative stability as proven by their lower FFA, p-AV, PV and TOTOX values than those extracted by 4 and 12 minute sonication. The total phenol content of EVOOs decreased as ultrasound time increased.

Keywords: Extra Virgin Olive Oil, Malaxation, Physicochemical Properties, Olive Cultivars, Ultrasound.

1. Introduction

Olive oil is extracted from the olive fruits (*Olea europaea L.*) by using physical or mechanical processes that include crushing of olive fruits, malaxation of olive paste, and solid–liquid separation by pressure or centrifugation (Bejaoui, Beltrán, Sánchez-Ortiz, Sánchez, & Jiménez, 2015). The best commercial grades of olive oils (virgin and extra virgin) produced by mechanical procedures (Ben Brahim, Marrakchi, Gargouri, & Bouaziz, 2015), preserve the unique composition and delicate aroma of oil, and therefore can be consumed without further treatments (Boskou, 2007). Olive oil extraction begins with the crushing step which breaks up the cells of olive plant tissue so that oil droplets run out of mesocarp cells (Angerosa, Mostallino, Basti, & Vito, 2001; Jiménez, Beltrán, & Uceda, 2007). The malaxation step contributes the small oil droplets merging into large drops that can be easily separated by centrifugation (Clodoveo, Durante, & La Notte, 2013) and has several effects on the quality and sensorial characteristics of oils (Clodoveo, 2012; Inarejos-García, Gómez-Rico, Salvador, & Fregapane, 2009). Malaxation is a low (20-30 rpm) and continuous kneading of olive pastes and is significant to achieve high oil quality and optimum extraction yield (Puértolas & Martínez de Marañón, 2015). Therefore, during this step, malaxation time and temperature should be carefully monitored to improve oil quality and yield (Clodoveo, 2012; Puértolas & Martínez de Marañón, 2015).

Nowadays, the most used method to improve olive oil extraction is to increase malaxation time or/and temperature (Ben Brahim et al., 2015). In olive oil extraction, a longer malaxation time at optimum temperature with a preheating process give a greater amount of oil. However, long malaxation times and high temperatures can cause deterioration in oil quality and reduce the oxidative stability of olive oil (Di

Giovacchino, Costantini, Ferrante, & Serraiocco, 2002; Taticchi et al., 2013) due to the activity of the enzymes (polyphenol oxidase and peroxidase), naturally present in the olive paste, which may affect the percentage of free fatty acids content, peroxide value and phenolic content of oil (Stefanoudaki, Koutsaftakis, & Harwood, 2011). Therefore, finding innovative techniques to improve the yield and quality of extra virgin olive oil (EVOO) have become significant for many researchers (Clodoveo & Hachicha Hbaieb, 2013). Several researches have reported the positive effects of the malaxation time and temperature on the yield and quality of virgin olive oil (Angerosa et al., 2001; Di Giovacchino et al., 2002; GóMez-Rico, Inarejos-García, Salvador, & Fregapane, 2009; Inarejos-García et al., 2009; Stefanoudaki et al., 2011). Researchers also used the coadjuvants (Ben Brahim et al., 2015; Espínola, Moya, Fernández, & Castro, 2009) and methods like pulse electric field (Abenoza et al., 2013; Puértolas & Martínez de Marañón, 2015), microwave (Clodoveo & Hachicha Hbaieb, 2013), and ultrasound process (Bejaoui et al., 2015; Clodoveo et al., 2013; Jiménez et al., 2007) to improve malaxation step.
Ultrasound is an emerging technology applied in several industrial operations such as heat and mass transfer, drying, filtration, defoaming, emulsification, freezing, degas and extraction (Kadam, Tiwari, Álvarez, & O'Donnell, 2015; Kumcuoglu, Yilmaz, & Tavman, 2014). This method is based on the cavitation phenomena which break cell membranes and facilitate the release of cellular components (Achat et al., 2012). Ultrasound assisted extraction has been applied in enrichment of olive oil with phenolic compounds from olive leaves (Achat et al., 2012).
Recently, ultrasound treatment during olive oil extraction has been studied before the malaxation of olive paste using a laboratory-scale olive oil extraction system (Bejaoui et al., 2015; Clodoveo et al., 2013; Jiménez et al., 2007). Concurrently, consumer trends towards consumption of olive oil have increased, and with them the interests of the oil industry on increasing the oil yield to the maximum without affecting the quality of the oil. In order to better understand the effect of ultrasound in the extraction of olive oil, and develop ultrasound assisted malaxer that lead to increase oil yield, it is necessary to understand how oxidative, nutritional and quality paramaters of olive oil change in different combinations of ultrasound and malaxation time applied during this process. Therefore, the aims of this work were to determine the combine effect of ultrasound and malaxation on the oil yield and extractability indexes, to determine oxidative stabilities, nutritional and quality parameters of olive oil, and to evaluate the potential benefit of the application of ultrasound adapted in malaxation step on improving the actual extraction process of virgin olive oil without applying any pre-heating or heating stage.

2. MATERIALS AND METHODS

2.1. Olive Fruit Samples

Olive fruits (*Olea europaea L.*) from Turkish Edremit, Gemlik and Uslu cultivars were harvested in Akhisar (Manisa- Turkey) area in the 2014/2015 crop season. Olives with maturity indexes 3.35, 3.15 and 3.12 for Edremit, Gemlik and Uslu cultivars respectively were picked at the early ripening stage in October. The olives were put into 25 kg boxes and brought to the pilot plant. Moisture content (% weight /weight) of olive fruit was determined at 120 °C until fruit reached the constant weight. Soxhlet extraction method was used to determine the total oil content (TOC) of olive samples and TOC was expressed as percentage on fresh matter basis.

2.2. Ultrasound Assisted Olive Oil Extraction

A laboratory mill, equipped with a metal crusher, a mixer and a basket centrifuge, were used to extract oil from olive fruits. After washing process, each batch was divided in twelve homogenous portions (4 kg each). The ultrasound system used in this study was ultrasonic bath (150kW and ultrasonic frequency 25 KHz; Tank volume 4.5 L). Flow chart of olive oil extraction is shown in Figure 1. Each olive paste sample was centrifuged for 1 minute and oil was collected in 500 ml dark color bottles. No co-adjuvant and pre-heating process was applied during extractions and all experiments were performed in triplicate.

2.3. Temperature Measurement

The temperature measurements were taken with a K type thermocouple by placing the 3 probes in different points of ultrasound tank for every 2 minutes during ultrasound treatment. A strong linear correlation was observed between ultrasound time and temperature (r=0.9720) (Figure 2). The ultrasound tank was cleaned before every measurement. The laboratory mill, equipped with a metal crusher, mixer and basket centrifuge were also subjected to the cleaning process before and after every measurement by

water to avoid contamination which can affect the accuracy of extraction yield and quality characteristic measurements. All parts of extraction unit were cleaned with water before the next test.

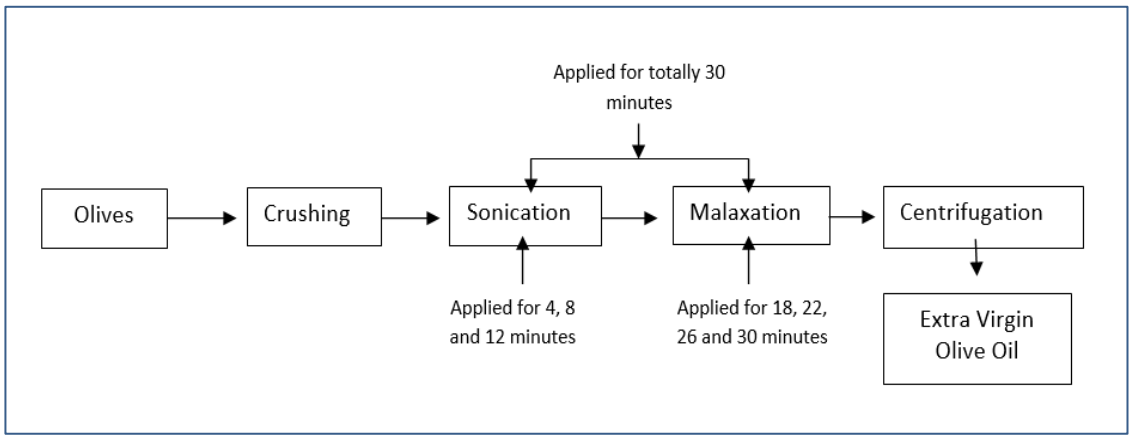

Figure 1. Flow chart of olive oil extraction

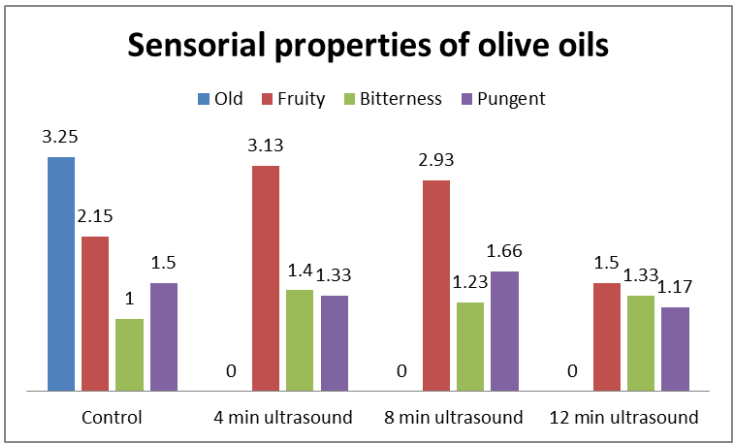

Figure 2.Sensorial properties of olive oils

2.4. Extraction Yield

Extractability index (EI) was defined as the percentage of the extracted olive oil from the total oil content of the fruit (TOC). The ‘‘extractability index’’ (EI) was calculated using the formula:

EI =(Yield/TOC) x100 [1]

Where TOC is total oil content of olive fruits determined by Soxhlet Method and yield is the percentage of extracted olive oil weight (g). Yield (%) was calculated as the percentage in extracted VOO weight (g) (W= volume measured by olive oil density at extraction temperature) from the olives weight (W oil) (on a fresh matter basis):

$$Yield=(W_{oil}/W_{olives})*100 \quad [2]$$

2.5. Physicochemical Properties of Olive Oils

The ultra-violet absorptions (K232 and K270), the free fatty acid content (FFA) and peroxide value (PV) were used for evaluating the primary oxidation products (European Union Commission Regulation, 1991), whereas the p-anisidine value was used to monitor the secondary oxidation products (AOCS,

2009). Total oxidation (TOTOX) values of oil samples were determined using the Eq. (3) (Samaram et al., 2015).

Total oxidation (TOTOX) value = 2PV + p-AV [3]

Chlorophyll and carotenoid contents were determined colorimetrically at 670 and 470 nm, respectively, and the results were expressed as mg/kg (Gandul-Rojas & Mínguez-Mosquera, 1996).

Chlorophyll= $(A_{670}x10^6)/(613x100xd)$ [4]

Carotenoid= $(A_{470}x10^6)/(2000x100xd)$ [5]

where A is the absorbance and d is the spectrophotometer cell thickness (1 cm).

Phenols were extracted from olive oil by liquid–liquid extraction using methanol as solvent and concentration was measured using Folin-Ciocalteau reagent at 725 nm. The total phenolic content was expressed as mg of gallic acid equivalents per kg of extra virgin olive oil (Montedoro & Servili, 1992).

2.6. Sensory Analysis

The sensory analysis was conducted by a panel test according to the method of Aydar et al. (2017). The sensory quality of the oil samples were evaluated by 10 trained panellists. The panelists evaluated the overall acceptability of each oil sample (taking into account any positive and negative taste), using a numerical scale 1–5 (1 = not acceptable, 5 = extremely good), as well as bitterness and fruity etc. (1 = no bitterness, 5 = extremely bitter). EVOO classification establishes a mean value of the defects of 0 and a mean value for fruity above 0 according to EU legislation (Achat et al., 2012;Aydar et al., 2017; Puértolas and Martínez de Marañón, 2015).

2.7. Statistical Analysis

All data were analyzed using SAS 9.2. (SAS Institute Inc., Cary, NC, 2009). One way ANOVA were run on all individual data groups to determine the significance differences. A Tukey's multiple comparison tests, at $p < 0.05$, was applied to determine differences among data groups. Correlation coefficients between instrumental results were also determined using SAS.

3. RESULTS and DISCUSSIONS

3.1. Effect of malaxation combined by ultrasound on extraction yield

Some of the oil can not be released in olive oil extraction because it is emulsified with olive mill waste water and remained with olive pomace . For this reason, oil (20-25%) remains in the mesocarp cell even after centrifugation (Aguilera, Beltran, Sanchez-Villasclaras, Uceda, & Jimenez, 2010). The effect of ultrasound on oil extraction can be explained by the development of cell disruption and hence mass transfer phenomenon (Achat et al., 2012). Ultrasound helps to evacuate oil from crushed mesocarp cells (Puértolas & Martínez de Marañón, 2015). Extraction efficiency is accepted as one of the main parameters determining the effectiveness of olive oil extraction. This parameter indirectly accounts for the oil content in the olive water and pomace (Angerosa et al., 2001; Aydar et al., 2017; Puértolas & Martínez de Marañón, 2015). The oil contents of the olives, extraction and oil yields are shown in Table 1. In the Edremit control samples olive pomaces subjected to 30 minute malaxation at 23.4 °C, the oil and extraction yield was 8.80 % and 49.26%, respectively. The application of a 4 minute and 8 minute ultrasound treatment (25 kHz) to the olive paste before malaxation increased the extraction yield by 0.67% and 0.73% and the oil extractability by 3.71% and 4.04%, respectively. 4 minute and 8 minute ultrasound treatments (25 kHz) to the Gemlik olive paste before malaxation increased the extraction yield by 1.63% and 1.53% and the oil extractability by 8.3% and 7.8%, respectively. The highest oil extractability was observed when 8minute ultrasound was aplied to Uslu cultivar olives.These results were similar to those observed in previous studies (Bejaoui et al., 2015; Clodoveo et al., 2013; Jiménez et al., 2007). However 18 minutes malaxation combined by 12 minute ultrasound caused a decrease in oil yield by 0.79%, 1.14% and 0.65% for Edremit, Gemlik and Uslu oils, respectively when compared to the control sample. This result can be explained by this lowest malaxation time (18 minute) among all treatments did not allow the oil droplets to liberate from lipo-vacuoles of mesocarp cells of olive fruits. Statistical analysis of all the oil data showed no effect ($P > 0.05$) of ultrasound treatment on oil yield and extractability index values.

The improvement obtained in our study (0.67% and 0.73%, for 4 min. and 8 min. ultrasound to Edremit cultivar) is comparable to the results obtained by Clodoveo et al., (2013). These authors reported a significant increase of a 0.70% and 1.00% in 'Coratina' oil yield, for 4 min. or 8 min. ultrasound treatment (35 kHz; 150 W) of olive pastes, respectively, with a subsequent pre-heating (19 minutes and 8 minutes) and a malaxation step of 30 min at 30 °C. Jimenez et al. (2007) found that there was no significant increase in the yield of 'Picual' olive oils obtained by ultrasound application (150 W and 25 kHz) compared to oils extracted without any ultrasonication. Our results suggest that olive variety and extraction conditions (time, temperature, pre-heating and/or coadjuvant utilization) have crucial effects on ultrasound extraction efficiency.

In the olive oil industry, an extra malaxation step is required to reach the optimal paste temperature, usually around 15–20 min for 30 °C (Jiménez et al., 2007). Moreover, in our study no pre-heating process was applied to olive pastes which could contribute to increase the quantity of extracted EVOO per day in industrial scale.

Table 1. Effect Of Ultrasound And Malaxation Time On Extractability Index

Treatment	Temperature (°C)	Oil content (%)	Oil Yield (%)	Δ OY (%)	Extraction yield (%)	Δ EY (%)
Edremit						
Control	23.40	17.87	8.80^{a}±1.36	-	49.26^{a}±7.58	-
4	24.70	17.87	9.47^{a}±0.82	+0.67	52.97^{a}±4.60	+3.71
8	31.83	17.87	9.53^{a}±0.87	+0.73	53.30^{a}±4.87	+4.04
12	36.30	17.87	8.01^{a}±1.16	-0.79	44.84^{a}±6.50	-4.42
Gemlik						
Control	20.20	19.62	11.93^{ab}±0.19	-	60.8^{ab}±0.95	-
4	22.40	19.62	13.56^{a}±0.14	+1.63	69.1^{a}±0.73	+8.3
8	30.00	19.62	13.46^{ab}±0.17	+1.53	68.6^{ab}±0.89	+7.8
12	33.50	19.62	9.97^{b}±0.04	-1.14	55.0^{b}±0.19	-5.8
Uslu						
Control	21.60	16.86	6.86^{ab}±0.65	-	40.70^{ab}±3.85	-
4	23.80	16.86	7.76^{ab}±0.49	+0.90	46.00^{ab}±2.93	+5.30
8	33.50	16.86	8.60^{a}±0.52	+1.74	51.02^{a}±3.06	+10.32
12	35.80	16.86	6.21^{b}±0.97	-0.65	36.87^{b}±5.77	-3.83

Data are given as mean values (mv) ± standard deviations, n=3.
Mean within a column sharing the same letter is not different (p>0.05).

3.2. Effect of malaxation combined by ultrasound on physicochemical parameters of olive oil

The results for free acidity, peroxide number and UV absorptions are shown in Table 2. The FFA values of oil samples were between 0.44 and 0.69 % oleic acid . The FFA test has traditionally been used as a basic commercial criterion for grading olive oil. According to the FFA, PV and UV results, all oil samples were classified as extra virgin olive oil. No difference ($P > 0.05$) was observed between FFA values of the 4 minute and 8 minute sonication treatments in Edremit oils, while 4 minute and 8 minute sonication treatments differed from the control and 12 minutes sonication treatments.

Table 2. Effect of ultrasound on physicochemical characteristics of olive oil

Treatment	K 232	K 270	FFA (%)	PV ($mEqO_2$ / kg	p-AV	TOTOX	Total chlorophyll (mg/kg)	Total carotenoid (mg/kg)	Total phenolics (mg/kg)
Edremit									
Control	1.361^{c} ±0.011	0.073^{b}±0.004	0.56^{c} ±0.02	4.57^{b} ±0.28	0.77^{a} ±0.10	9.90^{c} ±0.57	3.76^{b}±0.01	2.43^{b}±0.05	72.55^{a}±1.26
4 min	1.989^{a} ±0.116	0.158^{a} ±0.002	0.59^{b} ±0.02	5.03^{a} ±0.16	0.82^{a} ±0.06	10.89^{b} ±0.33	6.18^{a}±0.03	3.40^{a}±0.02	62.00^{b}±0.60
8 min	1.025^{d} ±0.006	0.070^{c} ±0.004	0.60^{b} ±0.01	4.58^{b} ±0.17	0.79^{a} ±0.04	9.96^{c} ±0.36	6.86^{a}±0.05	3.62^{a}±0.02	54.03^{c}±1.60
12 min	1.510^{b} ±0.015	0.084^{b}±0.006	0.69^{a} ±0.28	5.25^{a} ±0.14	0.84^{a} ±0.05	11.34^{a} ±0.27	4.14^{b}±0.17	2.15^{b}±0.01	36.17^{d}±0.64
Gemlik									
Control	1.318^{a}±0.048	0.088^{a}±0.011	0.44^{a}±0.05	4.71^{b}±0.17	0.47^{b}±0.05	9.89^{b}±0.34	2.41^{c}±0.19	0.54^{b}±0.30	63.47^{a}±4.93
4 min	1.322^{a}±0.038	0.100^{a}±0.006	0.47^{a}±0.07	4.73^{b}±0.36	0.55^{a}±0.03	10.01^{b}±0.71	5.74^{b}±0.42	1.28^{a}±0.10	56.33^{b}±4.29
8 min	1.320^{a}±0.078	0.088^{a}±0.012	0.47^{a}±0.05	4.64^{b}±0.10	0.53^{ab}±0.05	9.80^{b}±0.25	6.59^{a}±0.15	1.42^{a}±0.37	44.00^{c}±4.00
12 min	1.405^{a}±0.071	0.087^{a}±0.008	0.47^{a}±0.11	5.77^{a}±0.11	0.58^{a}±0.05	12.12^{a}±0.23	5.45^{b}±0.10	0.65^{b}±0.06	37.87^{c}±0.88
Uslu									
Control	1.375^{b}±0.010	0.089^{b}±0.010	0.54^{b} ±0.08	5.23^{b} ±0.45	0.50^{a} ±0.13	10.97^{a}±0.84	2.71^{c}±0.21	2.44^{c}±0.03	52.79^{a}±1.47
4 min	1.443^{a} ±0.036	0.105^{a} ±0.010	0.56^{b} ±0.06	5.27^{a} ±0.39	0.45^{a} ±0.16	10.99^{a}±0.74	3.16^{b}±0.00	3.27^{b}±0.08	44.85^{b}±2.08
8 min	1.393^{b} ±0.033	0.075^{b} ±0.005	0.61^{ab} ±0.01	5.09^{b} ±0.44	0.61^{a} ±0.14	10.78^{a} ±0.90	4.54^{a}±0.05	3.71^{a}±0.44	38.83^{c}±1.46
12 min	1.408^{ab} ±0.018	0.087^{b}±0.008	0.67^{a} ±0.03	5.28^{a} ± 0.15	0.54^{a} ±0.01	11.11^{a} ±0.28	2.60^{c}±0.17	2.62^{c}±0.06	28.54^{d}±1.22

Data are given as mean values (mv) ± standard deviations. Mean within a column sharing the same letter is not different (P>0.05)

The peroxide values (PV) ranged from 4.57 to 5.77 mEq O_2/kg oil where all samples had values below 20 mEq O_2/kg oil, the acceptable limit of the IOOC (International Olive Oil Council, 2003) and the Turkish Food Codex (Turkish Food Codex, 2010). When olive oils were stored for 6 months in bottles at 20–25 °C after the oil extraction, the FFA and PV values did not exceed the recommended limits. Peroxide values of oils subjected to 4 and 12 minute sonication treatment were slightly higher than untreated oils. K 232 is related to the primary oxidation of oil and the conjugation of polyunsaturated fatty acids and K 270 is related to secondary oxidation products of oils and it is an indication of aldehydes, ketones and carbonyl compounds (Boskou, 2007). The ultra violet (UV) absorption values 232 and 270 nm, ranged from 1.025 to 1.989 and 0.070 to 0.158, respectively for Edremit oils. The UV absorption extinction coefficients, both K232 and K270, were all below the limits established for extra virgin olive oils by IOOC (International Olive Oil Council, 2003) and the Turkish Food Codex (Turkish Food Codex, 2010). The results are similar to those presented by previous researchers (Bejaoui et al., 2015; Clodoveo et al., 2013; Jiménez et al., 2007), as they also observed that all oils obtained by ultrasound were classified as extra virgin olive oil.

The differences in UV absorptions, K232 and K270 coefficients, can be affected by cultivar, fruit quality, climatic and ecological conditions, harvest time, altitude, crop season, growing location and storage conditions. Low FFA, PV, K232 and K270 values, in the virgin olive oils depend on high quality fruit and the small scale extraction systems (Nahed, Ahmed, & Girgis, 2010). Significant diff erences were found between treatments for both UV absorbance at 270 nm and 232 nm ($P<0.05$). This may be a consequence of temperature differences between the olive pastes due to ultrasound treatment. In general, for quality parameters, olive oils obtained by 8 minute ultrasound and 22 minute malaxation showed the lowest values in K232 and K270.

The oxidation tests showed considerable effects of extraction methods and conditions on the oxidative stability of olive oils (Table 2). In Uslu oils there were no significant differences among all treatment . In Edremit oils there were no differences between control and 8 minutes of sonication samples in terms of Totox values, while significant differences were observed by 4 minutes or 12 minutes of sonication. No difference was observed on p-AV of all oils ($P > 0.05$). In this study, p-AVs of oils ranged from 0.45 to 0.84 and Totox values ranged from 9.80 to 12.12 depending on the ultrasound treatment. In general, based on the Totox values of oil samples, our results depicted that control sample and olive paste subjected to 8 minute ultrasound showed better oxidative stability against oxidation than those subjected to 4 minutes and 12 minutes sonication. Previous studies also reported that virgin olive oils obtained by conventional methods exhibited low PV and Totox values. This could be due to the high levels of monounsaturated fatty acid (MUFA) present in fatty acid composition of virgin olive oil containing a double bond in their hydrocarbon chains, making them less easily oxidized than polyunsaturated fatty acids (PUFA) (Aydar, 2012; Franco et al., 2014).

Chlorophyll is the characteristic green pigment of olive oil determining the color intensity. The degree of green color of the olive oil is dependent on chlorophyll, carotenoids and oleuropein pigments. The pigment concentration of oils differs greatly depending on the variety and degree of ripeness of the fruit. When the oil is exposed to light, it is rapidly oxidized because chlorophyll acts as a pro-oxidant. Thus, early harvest oils must be stored in the dark, such as in a green bottle, in order to preserve their quality (Angerosa et al., 2001; Gandul-Rojas & Mínguez-Mosquera, 1996). The total chlorophyll contents in samples were between 2.41 mg/kg and 6.86 mg/kg and the total carotenoid content ranged from 0.54 mg/kg and 3.71 mg/kg. When ultrasound treatment was applied to olives pastes, the curve of carotenoid and chlorophyll content as a function of sonication time had a bell shape distribution. This can be explained by the fact that initially the total chlorophyll and carotenoid compounds increased up (or achieved) to a maximum value because of the cavitation phenomena, then these minor compounds decreased as the duration of sonication and temperature increased. Olive oils extracted by 8 minutes ultrasound and 22 minutes malaxation had highest concentration both for chlorophyll and carotenoid; whereas those subjected to 12 minutes ultrasound and 18 minutes malaxation had lowest concentration.

The phenolic compounds are important bioactive components in olive oil that protect it against oxidation (Montedoro & Servili, 1992). The sensory properties (flavor, bitterness, etc.) of olives and olive oils are influenced by phenolic compounds (Servili, Selvaggini, Taticchi, Esposto, & Montedoro, 2003; Taticchi et al., 2013). The untreated oils showed higher values of total phenols than oils subjected to the sonication treatment. The total phenol content of Edremit oils were 72.55 mg/kg, 62.00 mg/kg, 54.03 mg/kg and 36.17 mg/kg for untreated, 4 minute sonication, 8 minute sonication, and 12 minute sonication applied treatments, respectively and phenolic content of Uslu and Gemlik oils were similar to obtained in Edremit oils. The total phenol content decreased as the duration of ultrasound treatment on olive paste was

extended. This observation can be explained by the presence of oxygen, which acts as a cofactor in many enzymatic reactions and as a promoter of non-enzymatic oxidations. When the olive pastes were submerged in an ultrasound, both enzymes (polyphenol oxidase, peroxidase) and substrates (phenols) were released which could contribute to oxidation. The differences in the amount of phenolic content of olive oils compared to previous studies was probably due to a range of factors affecting total phenolic compounds such as maturity level, cultivar, climate and extraction method.
Figure 2 shows the intensity attributes evaluated by panelists. The panelists perceived no negative attributes to ultrasound assisted oils. The oils subjected to 4 minute ultrasound were evaluated to be fruitier and bitter than other oils; although the pungent property were highest in oils obtained by 8 minute ultrasound. The oils extracted by 30 min of malaxation were evaluated at the lowest fruity scale for oils. It can be concluded that ultrasound had no negative effect on the sensory properties of the oils. Jimenez et al. (2007) also did not find any defect associated with ultrasound on olive oil production.

4. CONCLUSIONS

Olive oil consumption has risen recently worldwide due to its beneficial effects on human health. To meet the growing needs of consumers and to produce high-quality olive oil, new extraction methods are needed. This work presented the potential of an ultrasound assisted malaxation of olive oil extraction. Concerning the free acidity, peroxide value, K232 and K270, all oils obtained with or without ultrasound could be classified as "Extra Virgin Olive Oil" according to European norms. The ultrasound treatment before malaxation caused a rapid temperature increase up to 36.3 °C when 12 minute ultrasoun was applied to Edremiy olives and improved the extractability index up to 10.32% Uslu olives subjected to 8 minute sonication . Our study showed that 12 minute ultrasound and 18 minute malaxation time combination was not a sufficient treatment to achieve optimum extractability, whereas oils subjected to 8 minute ultrasound and 22 minute malaxation increased the oil yield, chlorophyll and carotenoid contents. When olive pastes subjected to sonication up to 8 minutes, higher amounts of olive oils were extracted in an hour, without any preheating or coadjuvant requirement, which can be opposed to the several hours required in conventional extraction process. It can be concluded that sonication has a physical effect on the olive pastes causing minimal changes in their chemical properties.

ACKNOWLEDGEMENTS

Manisa Celal Bayar University (Project no :BAP-CBU 2015:105), Aydar Olive Company (Akhisar-Manisa,Turkey) and İzmir Olive Research Institute (İzmir, Turkey) are also gratefully acknowledged.

REFERENCES

Abenoza, M., Benito, M., Saldaña, G., Álvarez, I., Raso, J., & Sánchez-Gimeno, a. C. (2013). Effects of Pulsed Electric Field on Yield Extraction and Quality of Olive Oil. *Food and Bioprocess Technology*, *6*(6), 1367–1373. http://doi.org/10.1007/s11947-012-0817-6

Achat, S., Tomao, V., Madani, K., Chibane, M., Elmaataoui, M., Dangles, O., & Chemat, F. (2012). Direct enrichment of olive oil in oleuropein by ultrasound-assisted maceration at laboratory and pilot plant scale. *Ultrasonics Sonochemistry*, *19*(4), 777–786. http://doi.org/10.1016/j.ultsonch.2011.12.006

Aguilera, M. P., Beltran, G., Sanchez-Villasclaras, S., Uceda, M., & Jimenez, A. (2010). Kneading olive paste from unripe “Picual” fruits: I. Effect on oil process yield. *Journal of Food Engineering*, *97*(4), 533–538. http://doi.org/10.1016/j.jfoodeng.2009.11.013

Angerosa, F., Mostallino, R., Basti, C., & Vito, R. (2001). Influence of malaxation temperature and time on the quality of virgin olive oils. *Food Chemistry*, *72*(1), 19–28. http://doi.org/10.1016/S0308-8146(00)00194-1

AOCS. (2009). *Official Methods and Recommended Practices of the American Oil Chemists’Society*. Champaign.

Aydar, A. Y. (2012). *Determination of Contact Angle of Olive oil and Canola Oil on a PTFE surface at*

Elevated Temperatures. NCSU. Retrieved from http://repository.lib.ncsu.edu/ir/bitstream/1840.16/8206/1/etd.pdf

Aydar, A. Y., Bağdatlıoğlu, N., & Köseoğlu, O. (2017). Effect of ultrasound on olive oil extraction and optimization of ultrasound-assisted extraction of extra virgin olive oil by response surface methodology (RSM). *Grasas Y Aceites. International Journal of Fats and Oils*, *68*(June).

Aydar 2017. (n.d.).

Bejaoui, M. A., Beltrán, G., Sánchez-Ortiz, A., Sánchez, S., & Jiménez, A. (2015). Continuous high power ultrasound treatment before malaxation, a laboratory scale approach: Effect on virgin olive oil quality criteria and yield. *European Journal of Lipid Science and Technology*. http://doi.org/10.1002/ejlt.201500020

Ben Brahim, S., Marrakchi, F., Gargouri, B., & Bouaziz, M. (2015). Optimization of malaxing conditions using CaCO3 as a coadjuvant: A method to increase yield and quality of extra virgin olive oil cv. Chemlali. *LWT - Food Science and Technology*, *63*(1), 243–252. http://doi.org/10.1016/j.lwt.2015.03.013

Boskou, D. (2007). *Olive oil, Chemistry and Technology*. *World review of nutrition and dietetics* (Vol. 97). http://doi.org/10.1159/000097916

Clodoveo, M. L. (2012). Malaxation: Influence on virgin olive oil quality. Past, present and future – An overview. *Trends in Food Science & Technology*, *25*(1), 13–23. http://doi.org/10.1016/j.tifs.2011.11.004

Clodoveo, M. L., Durante, V., & La Notte, D. (2013). Working towards the development of innovative ultrasound equipment for the extraction of virgin olive oil. *Ultrasonics Sonochemistry*, *20*(5), 1261–1270. http://doi.org/10.1016/j.ultsonch.2013.02.001

Clodoveo, M. L., & Hachicha Hbaieb, R. (2013). Beyond the traditional virgin olive oil extraction systems: Searching innovative and sustainable plant engineering solutions. *Food Research International*, *54*(2), 1926–1933. http://doi.org/10.1016/j.foodres.2013.06.014

Di Giovacchino, L., Costantini, N., Ferrante, M. L., & Serraiocco, a. (2002). Influence of malaxation time of olive paste on oil extraction yields and chemical and organoleptic characteristics of virgin olive oil obtained by a centrifugal decanter at water saving. *Grasas Y Aceites*, *53*(2), 179–186. http://doi.org/10.3989/gya.2002.v53.i2.302

Espínola, F., Moya, M., Fernández, D. G., & Castro, E. (2009). Improved extraction of virgin olive oil using calcium carbonate as coadjuvant extractant. *Journal of Food Engineering*, *92*(1), 112–118. http://doi.org/10.1016/j.jfoodeng.2008.10.038

European Union Commission Regulation. (1991). *(EEC) No 2568/91*. Brussels: Official European Commission Journal.

Franco, M. N., Galeano-Díaz, T., López, Ó., Fernández-Bolaños, J. G., Sánchez, J., De Miguel, C., … Martín-Vertedor, D. (2014). Phenolic compounds and antioxidant capacity of virgin olive oil. *Food Chemistry*, *163*, 289–298. http://doi.org/10.1016/j.foodchem.2014.04.091

Gandul-Rojas, B., & Mínguez-Mosquera, M. I. (1996). Chlorophyll and Carotenoid Composition in Virgin Olive Oils from Various Spanish Olive Varieties. *Journal of the Science of Food and Agriculture*, *72*(1), 31–39. http://doi.org/10.1002/(SICI)1097-0010(199609)72:1<31::AID-JSFA619>3.0.CO;2-5

GóMez-Rico, A., Inarejos-García, A. M., Salvador, M. D., & Fregapane, G. (2009). Effect of malaxation conditions on phenol and volatile profiles in olive paste and the corresponding virgin olive oils (olea europaea L. Cv. cornicabra). *Journal of Agricultural and Food Chemistry*, *57*(9), 3587–3595.

http://doi.org/10.1021/jf803505w

Inarejos-García, A. M., Gómez-Rico, A., Salvador, M. D., & Fregapane, G. (2009). Influence of malaxation conditions on virgin olive oil yield, overall quality and composition. *European Food Research and Technology*, *228*(4), 671–677. http://doi.org/10.1007/s00217-008-0977-9

International Olive Oil Council. (2003). IOOC Trade Standard Applying to Olive Oils and Olive – Pomace Oils.COI/T.15.NC No 3/Rev. 1, 2003. Retrieved from http://www.internationaloliveoil.org

Jiménez, a., Beltrán, G., & Uceda, M. (2007). High-power ultrasound in olive paste pretreatment. Effect on process yield and virgin olive oil characteristics. *Ultrasonics Sonochemistry*, *14*(6), 725–731. http://doi.org/10.1016/j.ultsonch.2006.12.006

Kadam, S. U., Tiwari, B. K., Álvarez, C., & O'Donnell, C. P. (2015). Ultrasound applications for the extraction, identification and delivery of food proteins and bioactive peptides. *Trends in Food Science & Technology*, *46*(1), 60–67. http://doi.org/10.1016/j.tifs.2015.07.012

Kumcuoglu, S., Yilmaz, T., & Tavman, S. (2014). Ultrasound assisted extraction of lycopene from tomato processing wastes. *Journal of Food Science and Technology*, *51*(12), 4102–7. http://doi.org/10.1007/s13197-013-0926-x

Montedoro, G., & Servili, M. (1992). Simple and hydrolyzable phenolic compounds in virgin olive oil. 1. Their extraction, separation, and quantitative and semiquantitative evaluation by HPLC. *Journal of Agricultural and Food Chemistry*, *40*, 1571–1576. http://doi.org/10.1021/jf00021a019

Nahed, M. M., Ahmed, A. a a, & Girgis, a Y. (2010). Effect of the Cultivar Area and Variety on the Fatty Acid Composition and Overall Quality Index (Oqi) of Virgin Olive Oil, *88*(1), 273–284.

Puértolas, E., & Martínez de Marañón, I. (2015). Olive oil pilot-production assisted by pulsed electric field: Impact on extraction yield, chemical parameters and sensory properties. *Food Chemistry*, *167*, 497–502. http://doi.org/10.1016/j.foodchem.2014.07.029

Samaram, S., Mirhosseini, H., Tan, C. P., Ghazali, H. M., Bordbar, S., & Serjouie, A. (2015). Optimisation of ultrasound-assisted extraction of oil from papaya seed by response surface methodology: Oil recovery, radical scavenging antioxidant activity, and oxidation stability. *Food Chemistry*, *172*, 7–17. http://doi.org/10.1016/j.foodchem.2014.08.068

Servili, M., Selvaggini, R., Taticchi, A., Esposto, S., & Montedoro, G. (2003). Volatile Compounds and Phenolic Composition of Virgin Olive Oil: Optimization of Temperature and Time of Exposure of Olive Pastes to Air Contact during the Mechanical Extraction Process. *Journal of Agricultural and Food Chemistry*, *51*, 7980–7988. http://doi.org/10.1021/jf034804k

Stefanoudaki, E., Koutsaftakis, a., & Harwood, J. L. (2011). Influence of malaxation conditions on characteristic qualities of olive oil. *Food Chemistry*, *127*(4), 1481–1486. http://doi.org/10.1016/j.foodchem.2011.01.120

Taticchi, A., Esposto, S., Veneziani, G., Urbani, S., Selvaggini, R., & Servili, M. (2013). The influence of the malaxation temperature on the activity of polyphenoloxidase and peroxidase and on the phenolic composition of virgin olive oil. *Food Chemistry*, *136*(2), 975–983. http://doi.org/10.1016/j.foodchem.2012.08.071

Turkish Food Codex. (2010). *Turkish Food Codex on Olive Oil and Olive-Pomace Oil*. Retrieved from https://members.wto.org/crnattachments/2010/tbt/TUR/10_3315_00_e.pdf