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Optimization of Olive Oil Extraction Process by Response Surface Methodology

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ABSTRACT

Extraction yield and product quality of olive oil depend on various factors, particularly fruit's maturity and processing methods. In this present study, kneading process was optimized by response surface methodology in terms of oil yield and quality parameters. As a sample, olive fruits were used at two different maturity stages (purple versus black stages). Prediction of response was proposed with a full quadratic second order regression equation, and regression parameters were checked for each model in order to evaluate model's adequacy. Proposed models for yield and some quality parameters of olive oil were good enough for a successful prediction of experimental results. Oil yield was maximized as free acidity and peroxide value were controlled under the specified corresponding values for "extra virgin olive oil". Optimal kneading conditions for oil production were at 30°C for 45 min for purple fruits and 28°C for 55 min for black counterparts.

Keywords: Olive oil, Physicochemical properties, Oil yield, Statistics, Modelling

Zeytinyağı Ekstraksiyon İşleminin Yanıt Yüzey Metodu ile Optimizasyonu

ÖZ

Zeytinyağının ekstraksiyon verimi ve kalitesi başlıca meyve olgunluğu ve işleme metotları gibi farklı faktörlere bağlıdır. Bu çalışmada yoğurma işleminin yağ verimi ve kalite parametreleri bakımından yanıt yüzey yöntemi ile optimizasyonu amaçlanmıştır. Örnek olarak iki farklı olgunlukta (mor ve siyah dönemler) zeytin meyvesi kullanılmıştır. Yanıtların tahmininde ikinci dereceden regresyon eşitliği kullanılmış ve her bir model için model yeterliliğini değerlendirmek için regresyon parametrelerine bakılmıştır. Yağ verimi ve bazı kalite parametrelerine yönelik önerilen modellerin deneysel sonuçları tahmin etmede yeterli düzeyde oldukları bulunmuştur. Serbest asitlik ve peroksit değeri natürel sızma zeytinyağı için belirlenmiş olan değerlerin altında tutulurken, yağ verimi maksimize edilmiştir. Optimum yoğurma şartları ise mor dönem zeytinyağları için 30°C, 45 dakika, siyah dönem zeytinyağları için 28°C, 55 dakika olarak bulunmuştur.

Anahtar Kelimeler: Zeytinyağı, Fizikokimyasal özellikler, Yağ verimi, İstatistik, Modelleme

INTRODUCTION

Virgin olive oil is a product extracted from the fruit of olive tree solely by mechanical or other physical methods, which include washing, crushing, kneading of resulting pastes and separation of oily phase, or filtration according to the Codex Alimentarius, IOOC, and EC regulations. The critical factors of the extraction process for oil yield in terms of product quality are kneading time, temperature as well as fruits' maturity.

Olives are harvested at the spotted-purple or purple-black stage to produce oil. The most specific indicator to determine maturity is the oil accumulation in olives. At this point it should be noted that the percentage of oil in fresh olive fruit continuously increases as fruit ripens. This favourable effect of the maturity on olive oil yield is well documented and it is attributed to the decrease in moisture content with increasing maturity [1-11]. Additionally free acidity increases and the peroxide value decreases depending on an increase in maturity index [3, 4, 6, 7, 10, 12-14].

Kneading conditions (also mentioned as malaxation) are other critical factors to produce the highest quality and quantity of oil from fruits. Kneading process at different temperature and time pairs makes the olive paste uniform and breaks up the oil/water emulsion. Changes in quality parameters such as free acid and peroxide value as a function of temperature, time, and maturity stage have been studied [14, 15] Kneading conditions related changes in quality parameters are associated with an increase in lipase activity (responsible for free acidity increase) and an intensification of the primary oxidation processes (responsible for the increase of the peroxide value) [16-19].
As mentioned above, oil extraction in terms of yield performance and product’s quality is under effects of many factors, but being in different extents. Additionally, there may be interactions in between these factors. Thus, statistical analysis is often used to evaluate factors’ effects and their possible interactions, as well as to optimize processes conditions. Response surface methodology (RSM) enables the evaluation of the effects of independent process variables and their interactions on dependent variables for developing, improving, and optimizing processes [20].

In this study, kneading conditions and fruit’s ripeness dependent changes in olive oil yield as well as quality parameters were investigated to figure out the relations between factors and proposed responses. Additionally, extraction process was optimized to achieve the highest oil yield with the best quality.

MATERIAL and METHODS

Olive fruits (Memecik cultivar) were collected from an orchard located in Muğla, Turkey. Two maturity stages (purple and black stages) were determined by monitoring suitable season. Before oil extraction, some physical properties of olives were determined for each maturity level. All measurements were carried out triplicate and results were presented in Table 1.

Table 1. Physical properties of olive fruits at purple and black stages

Fruit Properties	Purple Stage**	Black Stage
Maturity Index	4.76±0.43b	6.63±0.49a
Width of Olive (mm)	16.33±1.38a	14.11±1.06c
Length of Olive (mm)	23.61±1.73a	21.90±1.75b
Weight of Olive (g)	3.49±0.76a	3.11±0.55b
Moisture of Olive (%)	52.36±0.82a	47.52±0.26b
Dry Matter Content (%)	47.64±0.82b	52.48±0.26a
Oil Content (%)*	48.42±0.51a	49.18±0.92a

*: Determined by the Soxhlet method and calculated on dry weight basis
**: Different letters within a row indicate means that are statistically different at α=0.05 level

Extraction of Olive Oil

The extraction of olive oil was carried out using the Abencor system (Hakki Usta Machinery, Aydin, Turkey). Kneading of olive paste was carried at different conditions according to the experimental design for each maturity stage given in Table 2. Separated oil was filtered through anhydrous sodium sulphate on cotton layer to remove remaining aqueous phase and stored in amber glass bottles at 4ºC for analysis. Oil yield for each trial was given as g oil 100 g^{-1} olive paste.

Quality parameters of olive oil

Free acidity of oil sample was determined by the American Oil Chemists’ Society (AOCS) method Ca 5a-40 [21] and results were given as an equivalent of oleic acid (%). The AOCS method Cd 8-53 [22] was used for determination of peroxide value (meq O_2 kg^{-1} oil) of oil sample. All measurements were carried out in triplicate for each sample.

Experimental Design

A central composite design was selected for optimization of kneading conditions (temperature and time), each at five levels with 13 runs including five central points. Optimization of independent variables of temperature (X_1) and time (X_2) were carried out to achieve the best performance of olive oil yield (Z_1), and the lowest levels of free acidity (Z_2) and peroxide (Z_3). Experimentally determined responses for each corresponding trial were given in Table 2 for both olive groups (purple and black stages). Response surface methodology (RSM) was used for optimization using Minitab Software (Minitab 16.1.1) [23]. Full quadratic second order regression model including the linear, quadratic and two factor interaction effects was proposed for the prediction of process (Eq. 1).

Table 2. Central composite design and experimental results

Run Order[a]	Temperature (°C)	Time (Min)	Purple Yield[b]	Purple Free Acidity[c]	Purple Peroxide Value[d]	Black Yield[b]	Black Free Acidity[c]	Black Peroxide Value[d]
1	45	30	7.82	0.13	11.19	9.97	0.86	7.43
2	45	60	4.67	0.14	11.81	3.29	0.87	7.94
3	35	45	11.99	0.21	9.42	12.99	0.71	8.24
4	20.85	45	13.18	0.12	9.09	13.36	0.79	6.70
5	35	45	13.45	0.13	9.25	13.27	0.71	8.24
6	35	45	12.81	0.15	9.23	13.18	0.71	8.23
7	35	45	12.63	0.13	9.24	13.18	0.71	8.24
8	49.14	45	2.29	0.17	11.73	2.01	0.83	7.58
9	25	60	14.09	0.13	9.20	14.46	0.73	7.72
10	35	66.21	11.35	0.17	10.30	13.18	0.79	8.27
11	25	30	14.18	0.12	7.54	11.90	0.75	7.37
12	35	45	13.54	0.14	9.26	13.08	0.71	8.22
13	35	23.79	12.72	0.13	9.23	12.72	0.80	8.05

[a]Randomized, [b] g oil 100 g^{-1} olive paste, [c] %, [d] meq O_2 kg^{-1}oil

$$Z = \beta_0 + \sum_{i=1}^{2} \beta_i X_i + \sum_{i=1}^{2} \beta_{ii} X_i^2 + \sum_{i=1}^{1} \sum_{j=i+1}^{2} \beta_{ij} X_i X_j \quad \text{Eq. (1)}$$

where Z was the dependent variable, the X was the independent variables, β_0 was the constant coefficient, β_i was the linear coefficient (main effect), β_{ii} was the quadratic coefficient, and β_{ij} was the two factors interaction coefficient. Response surfaces of the predicted values obtained by proposed models were plotted in the studied variable ranges by Sigma Plot Software (SPSS Inc., Chicago, IL). Model adequacy was evaluated by considering parameters of R^2 value, adjusted-R^2 value of regression.

RESULTS and DISCUSSION

According to EU-regulations, olive oils are classified with respect to free acidity and some physicochemical properties including peroxide. As shown in Table 2, free acidity (% oleic acid) and peroxide values (meq O_2 kg^{-1} oil) were in the range of 0.12-0.21 and 7.54-11.81 for purple stage olive oil and 0.71-0.87 and 6.70-8.24 for black stage olive oil, respectively. Oil samples obtained from olives at purple stage were found to be in the highest quality category “extra virgin olive oil”. Results of free acidity indicated that some of the oil samples obtained from fruits at black stage was out of standards (free acidity ≤ 0.8%, peroxide value ≤ 20 meq O_2 kg^{-1} oil) established for the highest quality category “extra virgin olive oil” at some extraction processes (trial 1, 2 and 8) being higher than 0.8%. An increase in free acidity could be attributed to fruits’ maturity and extraction conditions. In literature it was reported that free acidity increased with ripening of olive fruits [4, 6, 24]. In some studies kneading conditions was reported to be effective on quality parameters of olive oil and especially an increase in temperature caused higher free acidity [11, 17-19, 25]. Extraction performance evaluated for olive fruits at two different maturity indexes was considered and oil yields for each trial were given in Table 2. Oil yield measured for purple and black stage olive fruit was seen to vary from 2.29 to 14.18 g oil 100 g^{-1} olive paste and from 2.01 to 14.46 g oil 100 g^{-1} olive paste depending on studied parameters, respectively (Table 2). Oil yield observed in the present study agrees with that reported for Memecik cultivar by Nergiz ve Ergönül [26]. Besides of kneading conditions, maturity of olive fruit has been reported to be effective on oil yield [1-11]. Models produced by response surface methodology were given in Table 3. Adequacies of models were evaluated by checking regression coefficient for each model. Models were found to estimate with high success for olive oil yields for all stages of interest. Regression coefficients were higher than 0.90 for models belonging to oil yields. Free acidity and peroxide values of oil samples were also found to be predicted by related models with high performances.

Olive fruits harvested at two maturity stages were processed for oil production and results are given in Table 2 depending on raw material maturity and they are shown in Figures 1 and 2 for purple and black stages, respectively. Oil yield (g oil 100 g^{-1} olive paste) from olives at purple and black stage was found to be varied under effects of kneading conditions (Figures 1 and 2). The highest extraction yield was obtained in a kneading process relatively at low temperature and for long time irrespective of fruit maturity (Figures 1-2). Time related change in oil yield was linear for processed olive fruits at two maturity stage, whereas curve effect was observed with changing temperature (Figure 1 and 2). Table 3 also displays that time has significant parameter in the model just as first order level, whereas first and second order terms are significant for temperature. It is valid for both models obtained for studied maturities. Espínola et al. [11] reported the coincident results related to kneading temperature and time effects on olive yield.

Table 3. Regression coefficients of proposed models for the investigated responses of virgin olive oil extracted from fruits at two maturity stages

Variables	Purple			Black		
	Yield	Free Acidity	Peroxide Value	Yield	Free Acidity	Peroxide Value
β_0	-13.3137*	0.896437***	9.03670*	-33.5193***	1.69232***	-0.148459^{ns}
β_1	1.5890***	-0.028051***	-0.15857^{ns}	2.3093***	-0.03398**	0.418569***
β_2	0.2749*	-0.002559^{ns}	0.00471^{ns}	0.5898**	-0.02015**	0.020753^{ns}
β_{11}	-0.0250***	0.000348***	0.00506*	-0.0283***	0.00050***	-0.005832***
β_{22}	-0.0015^{ns}	0.000014^{ns}	0.00092^{ns}	-0.0009^{ns}	0.00020**	-0.000163^{ns}
β_{12}	-0.0051*	0.000039^{ns}	-0.00158^{ns}	-0.0154***	0.00007*	0.000172^{ns}
Model	***	**	***	***	**	**
Linear	***	**	ns	***	**	**
Quadratic	***	**	*	***	***	***
Cross-product	*	ns	ns	***	*	ns
R^2	98.81	85.10	92.71	97.71	89.66	87.56
Adj-R^2	97.96	74.45	87.51	96.08	82.28	78.68

^{ns}, not significant ($p > 0.05$); *, significant at $p \le 0.05$; **, significant at $p \le 0.01$; ***, significant at $p \le 0.001$

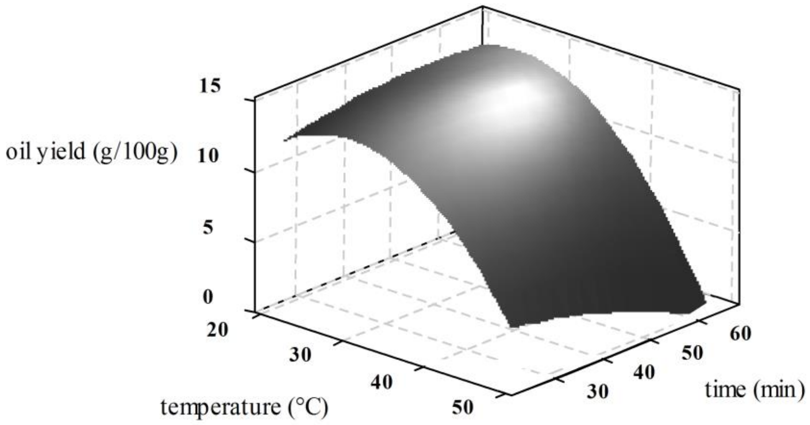

Figure 1. Influences of kneading conditions on yield of olive oil extracted from purple fruits

Free acidity of oil extracted from black olive fruits was higher than that measured for oil produced from fruits at purple stage, as shown in Figure 3 and 4. Being agree with our results, Finotti et al. [4], Salvador et al. [6], Rotondi vd. [12], Baccouri et al. [7], Baccouri et al. [13], Youssef et al. [14] and Lazzez et al. [10] reported that free acidity of oil depends on maturity of olive fruits. Temperature and time dependence of free acidity are shown in Figures 3 and 4 for oils produced from purple and black fruits, respectively. Strong temperature dependency of free acidity was seen for oil sample produced from purple olives, whereas significant change in this value was not seen with time (Figure 3). Table 3 also reveals similar results, where time related model parameters of free acidity (belonging to oil from purple olives) were found not to be significant (p>0.05). Contrary to trend observed for purple fruits, significant time dependent change in free acidity was seen for oil sample extracted from black fruits (Table 3, Figure 4). Temperature was another factor and its increase resulted in an increase in free acidity which was superior especially temperature level higher than 35°C (Figure 4). Regression analysis also revealed that time temperature interaction also significantly affected free acidity of oil sample (from black stage fruits) (p≤0.05) (Table 3). Di Giovacchino et al. [19] and Olias et al. [17] revealed similar results and pointed out the little increase in oil acidity as a result of hydrolytic action of the fruit enzymes being temperature induced. Espínola et al. [11] and Kalua et al. [18] also reported a presence of little influence on oil acidity with kneading temperature. Literature survey also showed that oils exhibiting higher acidity are obtained as a result of long kneading time at high temperature [16-19].

Changes in peroxide values of olive oils depending on maturity index of fruit are indicated in Figures 5 and 6 for oils obtained from purple and black stage fruits, respectively. Peroxide value of oil extracted from olive fruits at purple stage was higher than that measured for black fruits (Figures 5 and 6). Peroxide value change with fruit maturity agrees with that reported by Gutiérrez et al. [3], Salvador et al. [6], Rotondi et al. [12], Baccouri et al. [7], Baccouri et al. [13], Youssef et al. [14] and Lazzez et al. [10].

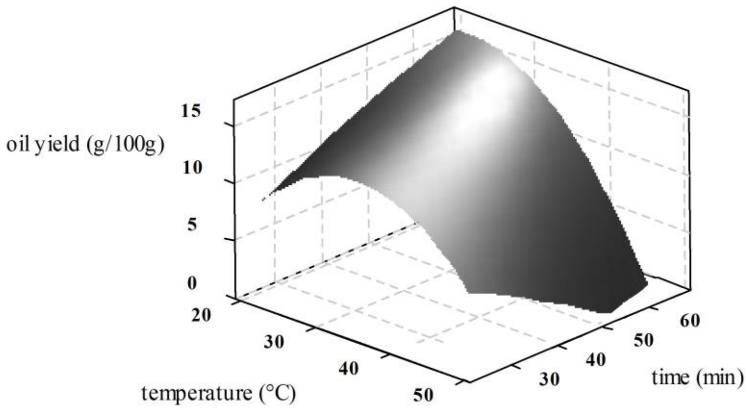

Figure 2. Effect of kneading conditions on yield of olive oil extracted from black fruits

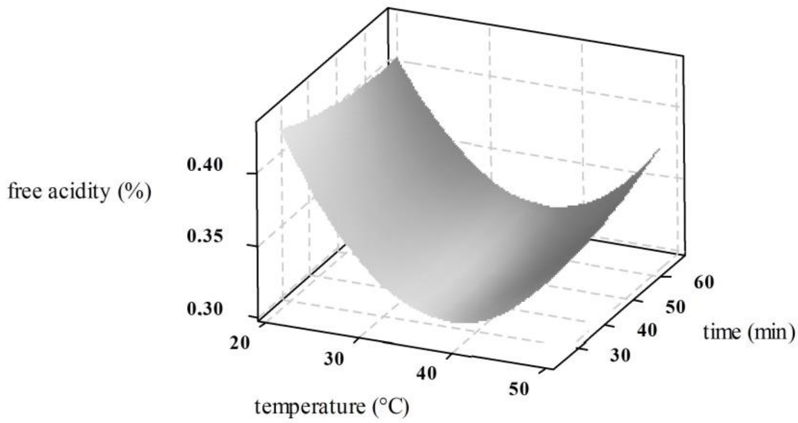

Figure 3. Effect of kneading conditions on free acidity of olive oil extracted from purple fruits

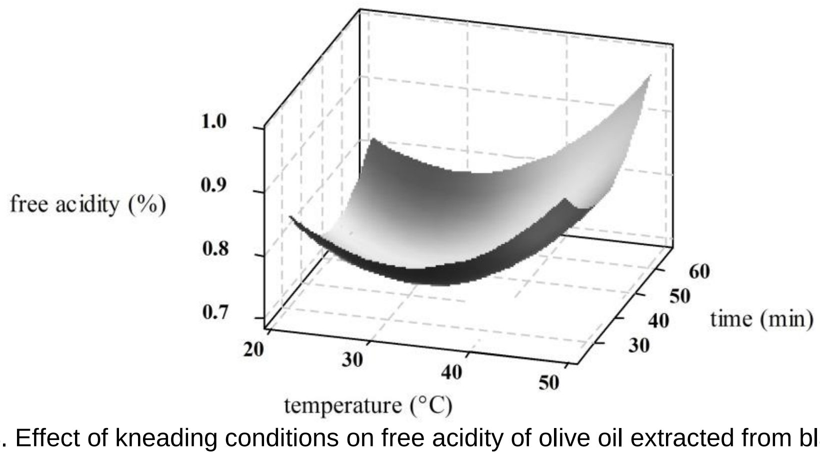

Figure 4. Effect of kneading conditions on free acidity of olive oil extracted from black fruits

Kneading conditions (temperature and time) were also examined in the present study to investigate their influences on peroxide values of produced olive oils. Significant temperature effect was found on peroxide value of oil samples extracted from fruits at both maturity stages, whereas time did not significantly change this quality parameter of olive oil in the studied conditions (Table 3). Figures 5 and 6 show the variation of peroxide values with temperature and time. Peroxide value increased with higher temperature levels, except for oil samples produced from black stage, for which temperature increase resulted in an increase in peroxide value up to certain point and further increments in temperature caused decreased in this quality parameter (Figures 5 and 6). Similarly, Vekiari and Koutsaftakis [15] was stated that oils exhibiting higher oxidative deterioration were obtained only at long mixing time and high temperatures. In addition, Ranalli et al. [16] explained that this was due to an intensification of the primary oxidation processes (responsible for the increase in peroxide index values).

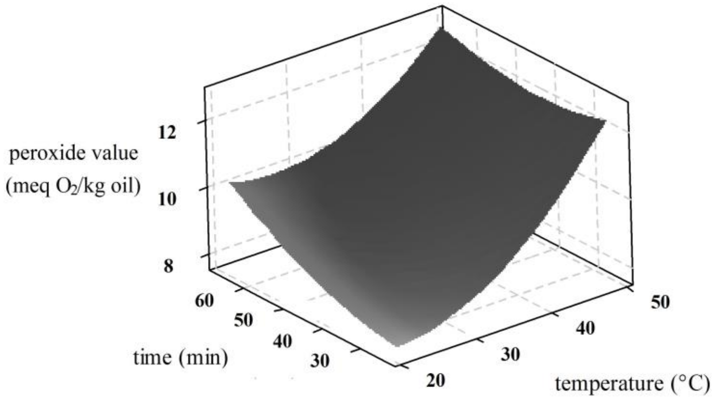

Figure 5. Effect of kneading conditions on peroxide value of olive oil extracted from purple fruits

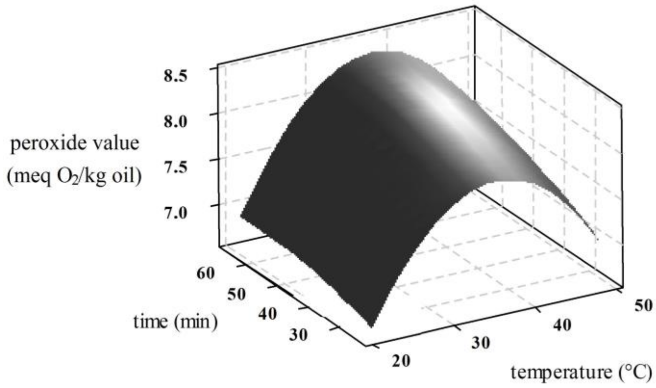

Figure 6. Effect of kneading conditions on peroxide value of olive oil extracted from black fruits

Optimization of Process Parameters

Optimization of any process is significant to reach the desired targets. In the present study main target was the maximization of oil yield, but it was not limitless since changes in process conditions required to achieve higher yield could adversely affect product quality. In the light of these facts, oil yield was maximized, as quality parameters were controlled to be under the levels classified in EU-regulations (free acidity ≤ 0.8%, peroxide value ≤ 20 meq O_2 kg^{-1} oil). For olive fruits at purple stage, kneading conditions were found to be 30°C and 45 min to achieve the highest oil yield given as 14.2 g oil 100 g^{-1} olive paste. Under the same conditions, free acidity and peroxide values of olive oil were predicted as 0.33% and 8.8 meq O_2 kg^{-1}oil, respectively. Kneading at 28°C for 55 min was enough to reach the goals (15 g oil 100 g^{-1} olive paste, 0.72% free acidity, 7.9 meq O_2 kg^{-1} oil) for processing of black olive fruits.

CONCLUSION

In olive oil production, many factors are effective on process performance and product quality. In this regard, cultivar, maturity stage, process conditions are prime ones. Even for the same cultivar, oil extraction process could differ in terms of oil yield and final quality since maturity index and process conditions are still strong parameters to affect process. Current study and other related ones in literature confirm this fact. In conclusion, kneading process's conditions were optimized in terms of oil yield and final product quality (considering the parameters specified for extra virgin olive oil). This process was carried for processing of olive fruits at purple and black stages, separately. Developed models display high success to predict the experimental results and give reasonable optimal conditions, thus it could be said that response surface methodology is a good tool for optimization of kneading process for olive oil manufacturers.

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REFERENCES

[1] García, J.M., Gutiérrez, F., Castellano, J.M., Perdiguero, S., Morilla, A., Albi, M.A., 1996. Influence of storage temperature on fruit ripening and olive oil quality. *Journal of Agricultural and Food Chemistry* 44(1): 264-267.

[2] Boskou, D., 1996. Olive oil quality. In Olive oil: Chemistry and technology, Edited by D. Boskou, AOCS Press, Champaign, Illinois, 101–120p.

[3] Gutiérrez, F., Jimenez, B., Ruiz, A., Albi, M., 1999. Effect of olive ripeness on the oxidative stability of virgin olive oil extracted from the varieties Picual and Hojiblanca and on the different components

involved. *Journal of Agricultural and Food Chemistry* 47(1): 121-127.

[4] Finotti, E., Beye, C., Nardo, N., Quaglia, G.B., Milin, C., Giacometti, J., 2001. Physico-chemical characteristics of olives and olive oil from two mono-cultivars during various ripening phases. *Food / Nahrung* 45(5): 350-352.

[5] Ayton, J., Mailer, R.J., Robards, K., Orchard, B., Vonarx, M., 2001. Oil concentration and composition of olives during fruit maturation in south-western New South Wales. *Australian Journal of Experimental Agriculture* 41(6): 815-821.

[6] Salvador, M., Aranda, F., Fregapane, G., 2001. Influence of fruit ripening on 'Cornicabra' virgin olive oil quality A study of four successive crop seasons. *Food Chemistry* 73(1): 45-53.

[7] Baccouri, B., Zarrouk, W., Krichene, D., Nouairi, I., 2007. Influence of fruit ripening and crop yield on chemical properties of virgin olive oils from seven selected oleasters (*Olea europea* L.). *Journal of Agronomy* 6(3): 388-396.

[8] 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.

[9] Menz, G., Vriesekoop, F., 2010. Physical and chemical changes during the maturation of Gordal Sevillana olives (*Olea europaea* L., cv. Gordal Sevillana). *Journal of Agricultural and Food Chemistry* 58(8): 4934-4938.

[10] Lazzez, A., Vichi, S., Kammoun, N.G., Arous, M.N., Khlif, M., Romero, A., Cossentini, M., 2011. A four year study to determine the optimal harvesting period for Tunisian Chemlali olives. *European Journal of Lipid Science and Technology* 113(6): 796-807.

[11] Espínola, F., Moya, M., Fernández, D.G., Castro, E., 2011. Modelling of virgin olive oil extraction using response surface methodology. *International Journal of Food Science & Technology* 46(12): 2576-2583.

[12] Rotondi, A., Bendini, A., Cerretani, L., Mari, M., Lercker, G., Toschi, T.G., 2004. Effect of olive ripening degree on the oxidative stability and organoleptic properties of cv. Nostrana di Brisighella extra virgin olive oil. *Journal of Agricultural and Food Chemistry* 52(11): 3649-3654.

[13] Baccouri, O., Guerfel, M., Baccouri, B., Cerretani, L., Bendini, A., Lercker, G., Zarrouk, M., Daoud Ben Miled, D., 2008. Chemical composition and oxidative stability of Tunisian monovarietal virgin olive oils with regard to fruit ripening. *Food Chemistry* 109(4): 743-754.

[14] Youssef, N.B., Zarrouk, W., Carrasco-Pancorbo, A., Ouni, Y., Segura-Carretero, A., Fernández-Gutiérrez, A., Daoud, D., Zarrouk, M., 2010. Effect of olive ripeness on chemical properties and phenolic composition of chétoui virgin olive oil. *Journal of the Science of Food and Agriculture* 90(2): 199-204.

[15] Vekiari, S.A., Koutsaftakis, A., 2002. The effect of different processing stages of olive fruit on the extracted olive oil polyphenol content. *Grasas y Aceites* 53(3): 5.

[16] Ranalli, A., Ferrante, M., De Mattia, G., Costantini, N., 1999. Analytical evaluation of virgin olive oil of first and second extraction. *Journal of Agricultural and Food Chemistry* 47(2): 417-424.

[17] Olias, J.M., Perez, A.G., Rios, J.J., Sanz, L.C., 1993. Aroma of virgin olive oil: biogenesis of the" green" odor notes. J*ournal of Agricultural and Food Chemistry* 41(12): 2368-2373.

[18] Kalua, C.M., Bedgood, D.R., Bishop, A.G., Prenzler, P.D., 2006. Changes in volatile and phenolic compounds with malaxation time and temperature during virgin olive oil production. *Journal of Agricultural and Food Chemistry* 54(20): 7641-7651.

[19] 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): 8.

[20] Myers, R.H., Montgomery, D.C., Anderson-Cook, C.M., 2009. Response surface methodology: process and product optimization using designed experiments. John Wiley & Sons, New Jersey, U.S.A.

[21] ASTM, 2007. D6304-04: standard test method for determination of water in petroleum products, lubricating oils, and additive by coulometric Karl Fischer titration in: Annual book of ASTM standards. ASTM International, West Conshohocken, PA, U.S.A.

[22] AOCS, 1989. Official Methods and Recommended Practices of the American Oil Chemists' Society. Champaign, Illinois, U.S.A.

[23] Tunç, İ., Çalışkan, F., Özkan, G., Karacabey, E., 2014. Mikrodalga destekli soxhlet cihazı ile fındık yağı ekstraksiyonunun yanıt yüzey yöntemi ile optimizasyonu. *Akademik Gıda* 12(1): 20-28.

[24] Dalgıç, L., Sermet, S.O., Büyükateş, K., Canlı, F., Özkan, G., 2013. Olgunlaşma sürecinin erken hasat memecik zeytinyağlarının raf ömrü ve bazı kalite kriterlerine etkileri. *Z&Z Akdeniz Kültürü Dergisi* 2911.

[25] Ranalli, A., Contento, S., Schiavone, C., Simone, N., 2001. Malaxing temperature affects volatile and phenol composition as well as other analytical features of virgin olive oil. *European Journal of Lipid Science and Technology* 103(4): 228-238.

[26] Nergiz, C., Ergönül, P.G., 2009. Organic acid content and composition of the olive fruits during ripening and its relationship with oil and sugar. *Scientia Horticulturae* 122(2): 216-220.