



Investigation of some quality-control parameters in Edremit yağlık olive oil

Fevzi KILIÇEL¹, Süleyman KILINÇ², Hacer Sibel KARAPINAR^{*3}

¹Karamanoğlu Mehmetbey University, Science Faculty, Department of Chemistry, Karaman, Türkiye

²Karamanoğlu Mehmetbey University, Science Faculty, Department of Chemistry, Karaman, Türkiye

³Karamanoğlu Mehmetbey University, Scientific and Technological Research & Application Center, Karaman, Türkiye

sibelkarapinar@kmu.edu.tr, ²incekumtr@gmail.com, ³sibelkarapinar@kmu.edu.tr

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Edremit yağlık zeytini yağında bazı kalite-kontrol parametrelerinin incelenmesi

Abstract: Throughout history, people have searched for the secret of long life and these researchers continue at full speed in parallel with the developments in science and technology in recent years. It is inevitable that the secret of a long and healthy life depends on nutrition and that nutrition should be with natural and high-quality nutrients. The Mediterranean diet, which is based on olive oil, has emerged as a popular diet in recent years. Due to the positive effects of olive oil on health, it is also economically valuable. For olive oil users to reach pure and high-quality olive oil, olive oils should be evaluated within the scope of some quality control parameters with the help of various analyzes. In this study, the quality parameters of free fatty acidity, peroxide value, and specific absorption (K_{232} , K_{270} , and ΔK) values in ultraviolet light were determined in olive oils obtained from Edremit Yağlık olives. The compliance of the values found with the Turkish Food Codex Communiqué on Olive Oil and Edible Pomace Oil (Communiqué No: 2017/26) has been evaluated. As a result of the analyzes made, the amount of free fatty acid was determined as 0.11% in oleic acid, the average peroxide number was 0.50 meq O_2/kg oil, the mean K_{232} values were 0.4501, the K_{270} values were on average 0.0446, and the mean ΔK values were -0.0248. It has been determined that all studied samples comply with the criteria for extra virgin olive oil specified in the Turkish Food Codex 2017/26 Communiqué.

Key words: Free fatty acidity, olive oil, peroxide number, specific absorption

Özet: İnsanlar tarih boyunca uzun yaşamın sırrını araştırmışlar ve bu araştırmalar son yıllarda bilim ve teknolojideki gelişmelerle paralel olarak tüm hızıyla devam etmektedir. Uzun ve sağlıklı yaşamın sırrının beslenmeye bağlı olduğu, beslenmenin de doğal ve kaliteli besin maddeleri ile olması gerektiği kaçınılmaz bir gerçektir. Temelinde zeytinyağı olan Akdeniz tipi beslenme, son yıllarda popüler bir beslenme tarzı olarak karşımıza çıkmaktadır. Zeytinyağının sağlık üzerine olumlu etkileri sebebiyle ekonomik olarak da değerlidir. Zeytinyağı kullanıcılarının saf ve kaliteli zeytinyağına ulaşabilmesi için, zeytinyağlarının çeşitli tahlilleri yardımıyla bazı kalite kontrol parametreleri kapsamında değerlendirilmesi gerekmektedir. Bu çalışmada, Edremit Yağlık türü zeytinlerden elde edilen zeytinyağlarında kalite parametrelerinden serbest yağ asitliği, peroksit değeri ve ultraviyole ışığında özgül soğurma (K_{232} , K_{270} , and ΔK) değerleri tespit edilmiştir. Bulunan değerlerin, Türk Gıda Kodeksi Zeytinyağı ve Yemeklik Pirina Yağı Tebliği'ne (Tebliğ No:2017/26) uygunluğu değerlendirilmiştir. Yapılan analizler sonucunda, serbest yağ asitliği miktarı %oleik asit cinsinden ortalama %0.11, peroksit sayısı ortalama 0.50 meq O_2/kg yağ, K_{232} değerleri ortalama 0.4501, K_{270} değerleri ortalama 0.0446 olarak, ΔK değerleri ortalama -0.0248 olarak belirlenmiştir. Çalışılan tüm örneklerin, Türk Gıda Kodeksi 2017/26 Tebliği'nde belirtilen naturel sızma zeytinyağı kriterlerine uygun olduğu belirlenmiştir.

Anahtar Kelimeler: Serbest yağ asitliği, zeytinyağı, peroksit sayısı, özgül soğurma

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1. Introduction

The olive known since prehistoric times belongs to the *Oleaceae* (Olive Fruits) family. This family, which spreads in tropical and temperate regions, is represented by 25 genera and about 600 species in the world. The species belonging to the genus *Olea* are plants where a large part of them are trees and shrubs, and they are spread in areas with relatively difficult growing conditions. This genus includes 33 species in the world. The only species with edible fruit is *Olea europaea* L., in which the cultivated olive is included. In our country, *O. europaea* species is represented by two varieties. These are *O. europaea* var. *europaea* Zhukovsky and *O. europaea* var. *sylvestris* (Miller) Lehr. The olive, which has two varieties (Delice, male olive, and wild olive), was spread in Northern, Western, and Southern Anatolia (Tokuşoğlu, 2010).

According to the data for 2021 in our country, the number of fruiting olive trees is 157.850, and from these trees, 555.833 tons of table, 1.182.147 tons of oil, a total of 1.738.680 tons, and in 2022, 2.976.000 tons of olives were produced (TUIK, 2023).

Olive oil is a natural oil with a unique taste and smell, which can vary from clear green to yellow, obtained only from the fruits of the olive tree (*Olea europaea*) by applying mechanical or physical processes (Aşık Uğurlu and Özkan, 2011). The physical processes carried out while obtaining olive oil do not bring about any change in the quality of the olive oil. Olive oil is distinguished from other seed oils by its natural state, that is, by consuming it without refining (Boskou et al., 2006).

Approximately 98% of olive oil consists of the major components triglycerides, fatty acids, and phosphatides,

while the remaining part consists of the minor components sterols, phenolic substances, free fatty acids, hydrocarbons, aliphatic and triterpene alcohols, and volatile components (Aydn et al., 2020). Especially tocopherol and phenolic substances are among the components that are studied intensively. These components make a significant contribution to the time that fats are preserved from spoilage, especially because they have an antioxidant effect. In addition, these components have many important effects in terms of their positive contributions to human health (Covas et al., 2006; Owen et al., 2000). Apart from health, the unique aroma of olive oil is formed by minor components. It consists of aroma, taste, and smell. Taste is often associated with phenolic compounds (Visioli and Galli, 1998) and smell is associated with volatile components (Kalua et al., 2007).

The minor components of olive oil vary depending on the olive variety, climate, level of ripening, and processing conditions (Gimeno et al., 2002)

Although the consumption intensity of olive oil has been concentrated in the regions where it is produced more in the past years, it has started to be consumed in the regions where it is not produced because it is known to benefit human health today. Olive oil production is mostly concentrated in the Marmara, Aegean, Mediterranean, and Southeastern Anatolia regions (Unakitan et al., 2012).

People's growing desire to live healthy and long has also increased their interest in olive oil. The most important feature that distinguishes olive oil from many oils is that it is a type of oil that can be used by consumers without side effects. The fact that olive oil is not subjected to heat treatment and is obtained by passing it through cold pressing is an important feature that increases its benefit to human health (Karabulut, 2013; Küçükkömürler and Uluksar, 2018). Olive oil is an important source of healthy living. It has a unique smell, aroma, and antioxidant properties. It also contains many vitamins, flavonoids, and sterols. Today, olive oil is mostly used in kitchens for cooking, salads, health care, and body care (Ünsal, 2008; Özata and Cömert, 2016; Küçükkömürler and Uluksar, 2018).

The oleic acid, hydroxytyrosol and caffeic acid found in olive oil have shown great protection factors against cancer. Olive oil consumption has benefits for the large intestine and also shows active properties in the prevention of breast cancer (Waterman and Lockwood, 2007; Kiritsakis and Shahidi, 2017).

A diet rich in extra virgin olive oil reduces the risk of cardiovascular disease (Armutcu et al., 2013). Mediterranean-type nutrition improves major risk factors for cardiovascular diseases such as lipoprotein profile, blood pressure, glucose metabolism, and antithrombotic profile (Kiritsakis and Shahidi, 2017).

In addition to being rich in monounsaturated fatty acids, olive oil contains small ingredients with antioxidant properties (Fitó et al., 2007). This is because olive trees in the Mediterranean basin have developed several antioxidant defense mechanisms to protect themselves from environmental stress (Visioli and Galli, 1998; Aparicio and García-González, 2013).

Oxidative stress is defined as an imbalance between the body's oxidant and antioxidant systems in favor of oxidants. The oxidative stress produced by free radicals has been linked to the development of various diseases, such as cancer and neurodegenerative diseases (Fitó et al., 2007). Protocatechuic and syringic acid have also been found to have antioxidant activity. Tyrosol, p-hydroxyphenylacetic acid, o-coumaric acid, p-coumaric acid, p-hydroxybenzoic acid, and vanillic acid were found to have little or no effect. Their contribution to the stability of the oil is minimal (Aparicio and García-González, 2013; Papadopoulos and Boskou, 1991).

In a study conducted to evaluate the possible differences between the antihypertensive effects of monounsaturated (MUFA) (extra virgin olive oil) and polyunsaturated fatty acids (PUFA) (sunflower oil), it was found that 8% of those who received the MUFA diet did not need drug treatment. A slight reduction in saturated fat intake combined with the use of extra virgin olive oil significantly reduces the need for a daily dose of antihypertensive drugs (Ferrara et al., 2000).

Freshly squeezed extra virgin olive oil contains oleocanthal, a compound whose pungency evokes a strong stinging sensation in the throat. This substance is one of the solutions of the anti-inflammatory drug ibuprofen. Here, the fact that oleocanthal acts as a natural anti-inflammatory compound with a profile strikingly similar to that of ibuprofen is indicative of pharmacological activity (Beauchamp et al., 2005). In this regard, it is considered anti-inflammatory and inhibitory (Kiritsakis and Shahidi, 2017).

Aging and related atherosclerosis, Morbus Parkinson's, Alzheimer's disease, and cognitive decline are major health problems in developed societies. Monounsaturated fatty acids are recognized to play a protective role against age-related cognitive decline and Alzheimer's disease (Kiritsakis and Dugan, 1985; Berr et al., 2009; Aparicio and García-González, 2013).

In this study, some quality parameters (free fatty acidity, peroxide value, and specific absorption in ultraviolet light) were analyzed in the samples of olive oils obtained from Edremit Yağlık type olives and their compliance with the Turkish Food Codex (TFC) Communiqué on Olive Oil and Edible Pomace Oil (Communiqué No: 2017/26) was evaluated.

2. Materials and Method

2.1. Material

The oils obtained from the Edremit Yağlık type olives used in the analyzes were obtained from local olive oil producers engaged in agricultural activities in the borders of Çanakkale Province-Bayramiç and Yenice District, Balıkesir Province-Edremit District during the 2020 harvest period. During the study, olive oil samples were stored in brown glass bottles and in an unlit environment.

2.2. Method

2.2.1. Free fatty acidity determination

The free fatty acid (%FFA) content of the extra virgin olive oil sample was determined from %oleic acid using the standard AOCS Ca-5a-40 method. The determination of FFA % in oils was made by titration under non-water

conditions. 1 g of extra virgin olive oil sample was carefully weighed and shaken with the addition of 15 ml of ethylalcohol. At the end of the addition of 2-3 drops of phenolphthalein indicator, titration was performed with 0.01N NaOH solution until the onset of pink color. The consumed NaOH has been identified. Equation 1 determined the FFA results.

$$\% \text{FFA} = (V \times N \times M) / (G \times 10) \quad (1)$$

V: Amount of NaOH spent in titration

N: NaOH normality spent in titration (0.01N)

M: Fatty acids molecular mass (282g)

G: Sample mass

2.2.2. Determination of peroxide number

The peroxide number (PV) of a sample of extra virgin olive oil is a measure of the amount of meq O₂ in kg of oil or an expression of the hydroperoxide content in the oil. Analyses were performed using the standard AOCS Cd-8b-90 method. 1 g of the olive oil sample was taken and mixed by adding 1 ml of chloroform and 1.5 ml of acetic acid. 0.1 ml of KI was dripped onto it and kept in the dark for 180 seconds. Then 25 ml of pure water and 3 drops of 1% starch solution were added to it. It is the detection of turbidity and gray color peroxide as a result of the control. Subsequently, titration was performed with 0.002 N regulated Na₂S₂O₃ until the color of the solution was clear. The value of Na₂S₂O₃ spent has been determined. According to Equation 2, the peroxide number is calculated and expressed as milli equivalent gram oxygen/kg fat.

$$\text{PV} = (N \times V \times 100) / G \quad (2)$$

G: Sample weight

V: Amount of Na₂S₂O₃ consumed in titration (ml)

N: Normal of Na₂S₂O₃ used in titration

2.2.3. Determination of specific absorption

The method recommended by the standard AOCS Ch 5-91 was used to determine the specific absorption values. The samples were thoroughly homogenized and filtered through filter paper before being analyzed. A 0.25 g sample was weighed in a 25 ml balloon. The balloon containing the sample was completed with hexane to 25 ml and homogenized by mixing thoroughly. The absorbance values of the samples at different wavelengths (232, 266, 270, and 274 nm) were read out in the spectrophotometer. K₂₃₂, K₂₆₆, K₂₇₀, and K₂₇₄ are calculated according to Equation 3.

$$K\lambda = A\lambda / C \times L \quad (3)$$

$$K_{232}: A_{232} / C \times L$$

$$K_{266}: A_{266} / C \times L$$

$$K_{270}: A_{270} / C \times L$$

$$K_{274}: A_{274} / C \times L$$

The value of ΔK is calculated by Equation 4.

$$\Delta K = K_{270} - [(K_{266} + K_{274}) / 2] \quad (4)$$

K₂₃₂: Specific absorption value at 232 nm

K₂₆₆: Specific absorption value at 266 nm

K₂₇₀: Specific absorption value at 270 nm

K₂₇₄: Specific absorption value at 274 nm

A₂₃₂: Absorbance value read at 232 nm

A₂₆₆: Absorbance value read at 266 nm

A₂₇₀: Absorbance value read at 270 nm

A₂₇₄: Absorbance value read at 274 nm

C: Concentration of solution (g/100 ml)

L: Bathtub thickness (cm)

3. Results

3.1. Free fatty acidity determination results

As a result of the analysis, the amount of free fatty acidity in Edremit Yağlık type olive oil varies between 0.05% and 0.25% in terms of % oleic acid and the average 0.11% ± 0.05. The results meet the requirement of ≤0.8, which is the criterion of extra virgin olive oil determined according to the Turkish Food Codex Communiqué on Olive Oil and Pomace Oil (Communiqué No: 2017/26). It is seen that the determined results are in line with the literature. The determined analysis results are shown in Figure 1. As can be seen from Figure 1, in only two of the largest samples, our value is 0.25%, which is well below the limit value of 0.8%. According to these results, it was determined that the olive oils analyzed were of high quality in terms of free fatty acidity.

In a study conducted to determine the chemical, physical and sensory properties of olive oils produced in Çanakkale, it was reported that while the free fatty acid ratios were below the limit value specified in the TFC olive oil communiqué in all other samples except two samples, the peroxide values were generally by the standards. At the same time, it was determined that there were significant differences in terms of sensory parameters between olive oils (Öğütçü et al., 2008).

In another study conducted to reveal some characteristics of olive oils obtained from Chemlali and Chetoui variety olives taken from the northern and southern regions of Tunisia, it was determined that the free fatty acid values of Chemlali and Chetoui olive oils obtained from Southern Tunisia were between 1% and 0.9%, the free fatty acid values of olive oils obtained from Northern Tunisia were 0.1% (Chemlali) and 0.3% (Chetoui), and peroxide values were between 3.2-8.3 meq O₂/kg (Issaoui et al., 2010).

Various quality parameters have been examined in Queslari variety olive oils obtained from seven different regions of Tunisia. Accordingly, free fatty acid, peroxide values, UV specific absorbance values were found to be by the extra virgin olive oil standard, while palmitic acid (12.4-15.2%), oleic acid (68.1-74.7%), stearic acid (1.6-2.1%) and linoleic acid (6.7-14.9%) were the major fatty acids determined (Youssef et al., 2011).

In a study conducted to examine some properties of olive oils obtained from 11 varieties of olives belonging to the Marche region of Italy, it was revealed that 12 different free fatty acid values (0.1-0.2%) and peroxide values (3.9-8.2 meq O₂/kg) belonging to olive varieties were determined, at the same time eicosenoic, heptadanoic, eikosanoic, linoleic, linolenic, oleic, palmitic, stearic acid values were found and oleic acid values were highest in them (71.7-79.8%) (Cecchi and Alfei, 2013).

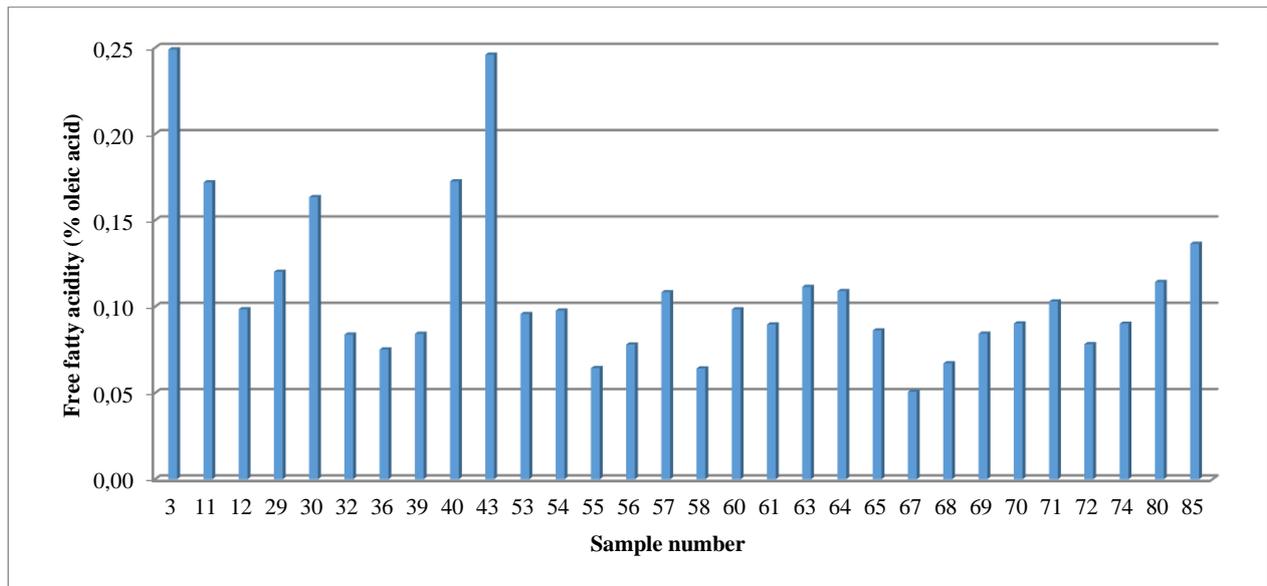


Figure 1. Free amounts of fatty acidity (% oleic acid)

The free fatty acidity in extra virgin olive oils of Sarulak type olives grown in Antalya, Mersin, and Karaman was determined between 0.50% and 0.83% and peroxide values were measured in the range of 3.99-4.33 meq O₂/kg (Arslan et al., 2013).

3.2. Peroxide count determination results

As a result of the analyzes, the number of peroxides in Edremit Yağlık type olive oil ranged between 0.22 meq O₂/kg oil and 1.23 meq O₂/kg oil and was measured as 0.50 ± 0.241 meq O₂/kg oil. All the determined values met the requirement of ≤20, which is the criterion of extra virgin olive oil according to the Turkish Food Codex Communiqué on Olive Oil and Pomace Oil (Communiqué No: 2017/26). All samples were determined to be consistent with the literature. The results of the analysis are shown in Figure 2. In Figure 2, it is seen that the peroxide values of our samples are very, very small than the limit value of twenty. Therefore, it was determined that the samples analyzed were quality olive oils.

In a study conducted by taking samples (n=30) from 10 different olive oil varieties offered for sale in and around Nizip, some of their physical and chemical properties were examined. In the evaluations, it was determined that the acidity and peroxide values of 40% of the samples were above the values determined in the Food Codex (Türkoğlu et al., 2012).

In another study in which the peroxide values of different kinds of olive oils for the 2017 and 2018 harvest years were measured, the 2017 peroxide values of the oils of Ayvalık, Çöpaşı, Gemlik, and Yağlık varieties of olives were found between 2.00-5.00 meq O₂/kg, 1.99-7.43 meq O₂/kg, 1.49-6.47 meq O₂/kg and 1.99-9.90 meq O₂/kg, respectively, while the peroxide values of 2018 were quite low compared to the previous year and 0.88-1.63 meq O₂/kg respectively, It was detected between 1.00-4.00 meq O₂/kg, 1.00-2.75 meq O₂/kg and 1.00-2.50 meq O₂/kg (Özcan et al., 2019).

Free acid contents of the fat samples *Verticillium wilt* did not show a linear relationship with disease severity. The

peroxide value, which gives information about the primary oxidation products in vegetable oils, was found to vary between 8.07-14.20 meq O₂/kg oil in oil samples obtained from diseased trees. It has not exceeded the limit of 20 meq O₂/kg for natural olive oil in the Turkish Food Codex Communiqué on Olive Oil. Peroxide values showed a direct proportion to disease severity (Yorulmaz et al., 2017).

In a study in which the aroma substances of the olive oils obtained from Gemlik and Barnea olive varieties grown in Adana province were determined, the peroxide value in olive oils was determined as 10.37 meq O₂/kg oil in Gemlik variety oil and 8.60 meq O₂/kg oil in Barnea variety oil (Kesen et al., 2014).

The peroxide value of olive oil obtained from the Gemlik variety grown in the Erzin district of Hatay province was reported as 8.85 meq O₂/kg (Kelebek et al., 2012).

3.3. Specific absorption determination results

In Edremit Yağlık type olive oils, K₂₃₂ values were determined as 0.2401-0.5922 with an average of 0.4501±0.094, and K₂₇₀ values between 0.0305-0.0628 with an average of 0.0446±0.008. ΔK values were found in the range of -0.2640-0.0037, with an average of an average of -0.0248±0.0780. According to the Turkish Food Codex Communiqué on Olive Oil and Pomace Oil (Communiqué No: 2017/26), the K value at 232 nm in extra virgin olive oil is ≤2.5; the value of K at 270 nm is ≤0.22; ΔK value should be ≤0.01. As a result of all the analyzes, it was determined that the values determined were smaller than the TFC values and were compatible with the literature. The results of the determination of specific absorption are shown in Figure 3. It was found that the K₂₃₂ values of the analyzed samples were well below 2.5, the K₂₇₀ values were well below 0.22 and the ΔK values were well below 0.01. It was determined that the samples with the analyzed olive oil consisted of quality olive oils.

In a study conducted to determine the maturity index values of the fruits of the Ayvalık olive variety harvested at 3 different maturities in 9 different regions of Çanakkale

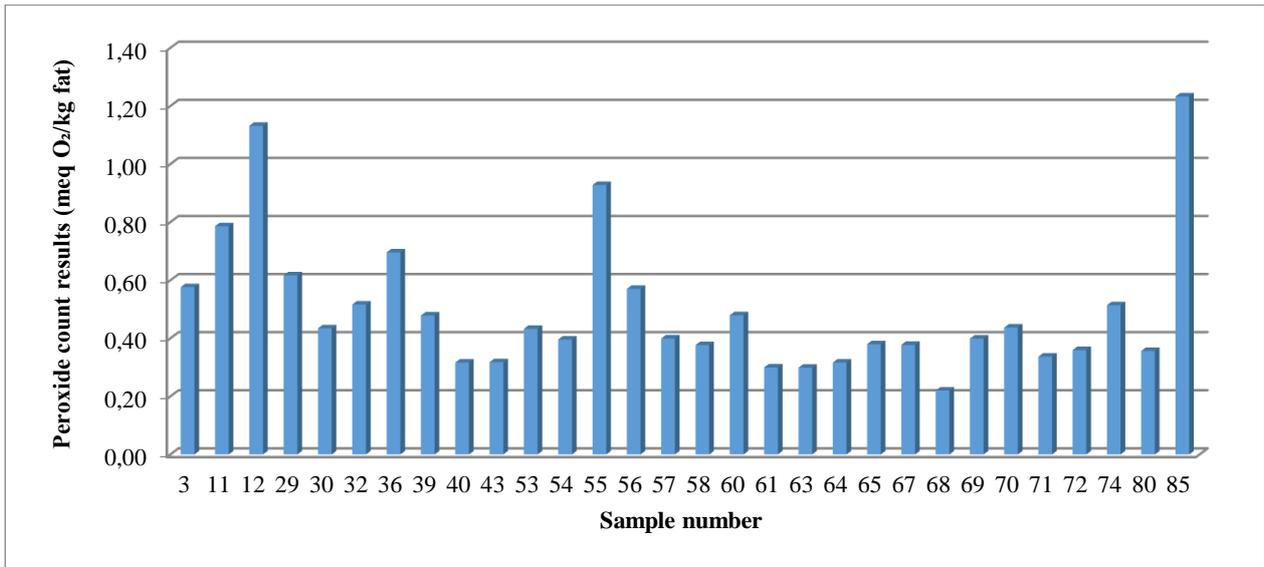


Figure 2. Peroxide count results (meq O₂/kg fat)

province Bayramiç and Edremit olive cultivation areas and the chemical properties of olive oils obtained, the K_{232} value for Edremit was 1.516 ± 0.181 ; K_{270} value is 0.091 ± 0.002 , K_{232} value for Bayramiç is 1.523 ± 0.041 ; K_{270} value was 0.092 ± 0.004 (Gündoğdu and Nergis, 2020).

In another study on the quality parameters of natural olive oils produced in the Hatay region, the K_{232} value was found to be the lowest at 1.67, the highest at 2.51, and the average at 2.02 ± 0.17 , the K_{270} value as the lowest 0.11, the highest 0.25 and the average as 0.16 ± 0.03 , the ΔK value as the lowest 0.00, the highest 0.01 and the average 0.004 ± 0.002 (Güler et al., 2006).

In a study conducted to determine some characteristics of olive oils obtained from G20/1 and G20/7 Clones and Gemlik variety olives, the specific absorption values in ultraviolet light were 2.57 ± 0.08 for the G20/1 clone, the K_{270} value was 0.20 ± 0.04 , the K_{232} value for the G20/7 clone was 2.32 ± 0.04 , the K_{270} value was 0.37 ± 0.09 and the K_{232} value for Gemlik type was 2.66 ± 0.09 , the K_{270} value was determined as 0.30 ± 0.03 (Özdemir et al., 2016).

In another study conducted to determine the effect of the harvest period on the quality parameters of Beylik olives and olive oil, the K_{232} value was determined as 0.67 ± 0.03 in October, the K_{270} value as 0.29 ± 0.001 , the K_{232} value as 0.83 ± 0.01 in November, the K_{270} value as 0.23 ± 0.01 and the K_{232} value as 1.11 ± 0.04 in December, the K_{270} value as 0.27 ± 0.01 (Özen and Keçeli, 2019).

In a study where the optimum harvest time of the Cornicabra olive variety was determined according to oil quality parameters at 4 different harvest times; It was found that UV-specific absorbance values and sensory quality decreased at 232 and 270 nm with peroxide as maturation increased, while free acidity amounts increased. It has been reported that the optimal maturity index for the Cornicabra olive variety should be higher than 3 and lower than 4-4.5 (Salvador et al., 2001).

In a study in which the effect of harvest time on some physical properties and pigment amounts of extra virgin olive oils obtained from Ayvalık, Domat, and Gemlik olive varieties was determined, 4 different harvest periods

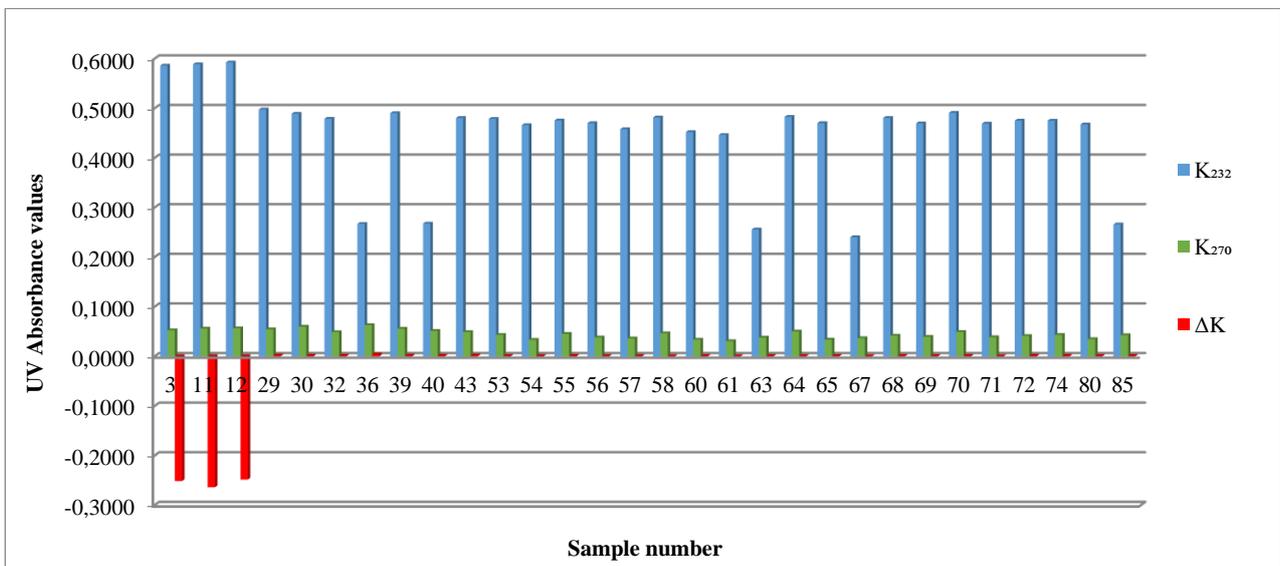


Figure 3. Specific absorption determination results (UV: Absorbance values K_{232} , K_{270} , ΔK)

including the first week of September, October, November, and December were selected. In extra virgin olive oils, specific absorbance (K_{232} , K_{270}) values were determined in UV light. Generally, as olive ripening increases in all varieties, a decrease in K_{232} and K_{270} values has been reported (Özkan et al., 2008).

Mean, standard deviations (SD), maximum and minimum values and range (i.e. maximum value – minimum value) obtained from five parameters (i.e. acidity, peroxide, K_{232} , K_{270} and ΔK) are shown in Table 1.

4. Discussions

In this study, it was investigated whether the olive oils obtained from Edremit Yağlık type olives harvested in 2020 complied with some quality parameters specified in the Turkish Food Codex Communiqué on Olive Oil and Pomace Oil, and the free fatty acidity, peroxide number, and specific absorption values were examined. As a result,

it was determined that all of the oil samples examined remained within the natural extra virgin olive oil standard values specified in the TFC communiqué.

Conflict of Interest

Authors have declared no conflict of interest.

Authors' Contributions

Fevzi Kılıçel: Project administration - review and editing - original draft - data curation. Süleyman Kılınç: Data curation - writing - editing - original draft. Hacer Sibel Karapınar: Writing - review and editing - original draft - data curation.

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Table 1. Data on the determination of free fatty acidity, peroxide number, and specific absorption number of Edremit Yağlık type olive oils (30 pieces)

Sample		Free fatty acidity (% oleic acid)	Peroxide value (meq O ₂ /kg)	K_{232} (UV)	K_{270} (UV)	ΔK
Edremit yağlık type olive oil	Mean	0.11	0.50	0.4501	0.0446	-0.0248
	St. Deviation	0.05	0.24	0.0942	0.0088	0.0780
	Minimum	0.05	0.22	0.2401	0.0305	-0.2640
	Maximum	0.25	1.23	0.5922	0.0628	0.0037
	Range	0.20	1.01	0.3521	0.0323	0.2677
Turkish Food Codex Communiqué on Olive Oil and Pomace Oil (Communiqué No: 2017/26)		≤0.8	≤20	≤2.5	≤0.22	≤0.01

UV: Absorbance values

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