DOI: 10.29050/harranziraat. 1516474
Harran Tarım ve Gıda Bilimleri Derg. 2024, 28(3):471-479

Research Article/Araştırma Makalesi

Effect of early harvest on the aroma compounds and bioactive properties of natural olive oils

Erken hasadın naturel zeytinyağlarının aroma bileşiklerine ve biyoaktif özelliklerine etkisi

Tugba KILIC[1*] Gulcan KOYUNCU[2]

[1,2] Kilis 7 Aralik University, Technical Sciences Vocational School, Food Processing Department, Kilis, Türkiye

[1]https://orcid.org/0000-0002-2573-6220; [2]https://orcid.org/0000-0001-7406-5331

To cite this article:
Kılıç T. & Koyuncu G. (2024). Effect of early harvest on the aroma compounds and bioactive properties of natural olive oils. Harran Tarım ve Gıda Bilimleri Dergisi, 28(3):471-479

DOI: 10.29050/harranziraat. 1516474

***Address for Correspondence:**
Tuğba KILIÇ
e-mail:
tubacelik@kilis.edu.tr

Received Date:
16.07.2024
Accepted Date:
29.08.2024

ABSTRACT

In this study, the effect of harvest time on various quality characteristics of natural oils obtained from Kilis yağlık variety olives was determined. Free fatty acid (FFA), acid number, peroxide value, iodine number, refractive index, color, total phenol (Folin Ciocalteu) and antioxidant capacity (DPPH) values were determined in olive oils. Volatile compounds in olive oils were obtained by solid phase microextraction (SPME) and detected by gas chromatography-mass spectrometry (GC-MS). It was determined that %FFA and peroxide values increased as the harvest time progressed. Similar to the total phenol content determined as 516.61 mg GAE L^{-1} in early harvest and 77.70 mg GAE L^{-1} in normal harvest, antioxidant capacities also decreased with ripening. A total of 37 different volatiles were determined in olive oils, and a decrease in aldehyde ratio and an increase in alcohol and acid ratios were detected with ripening. While the rate of pleasant compounds (hexanal, α-farnesene, etc.) was high in early harvest olive oil, an increase in the rate of off-flavor compounds (acetic acid, nonanal, etc.) was observed in normal harvest olive oil. It has been determined that harvest time is an important factor in olive oil quality.

Key Words: Olive oil, early harvest, bioactive properties, aroma composition, quality

ÖZ

Bu çalışmada, Kilis yağlık çeşidi zeytinlerden elde edilen doğal yağların çeşitli kalite özellikleri üzerine hasat zamanının etkisi belirlenmiştir. Zeytinyağlarında serbest yağ asitliği (FFA), asit sayısı, peroksit sayısı, iyot sayısı, kırılma indisi, renk, toplam fenol miktarı (Folin Ciocalteu) ve antioksidan kapasitesi (DPPH) belirlenmiştir. Zeytinyağlarında bulunan aroma maddeleri katı faz mikro ekstraksiyon (SPME) ile elde edilmiş ve gaz kromatografisi-kütle spektrometrisi (GC-MS) aracılığıyla tespit edilmiştir. Hasat zamanı ilerledikçe %FFA ve peroksit sayısı değerlerinin artış gösterdiği tespit edilmiştir. Toplam fenol madde miktarı erken hasatta 516.61 mg GAE L^{-1}, normal hasatta 77.70 mg GAE L^{-1} olarak belirlenirken; antioksidan kapasiteleri de olgunlaşmayla düşüş götermiştir. Zeytinyağlarında toplam 37 farklı uçucu bileşik belirlenmiş olup, olgunlaşma ile aldehit oranında düşme, alkol ve asit oranlarında artış tespit edilmiştir. Erken hasat zeytinyağında hoş kokulu bileşiklerin (hekzanal, α-farnesen vb.) oranı daha fazla iken normal hasat zeytinyağında istenmeyen bileşiklerde (asetik asit, nonanal vb.) artış görülmüştür. Zeytinyağı kalitesinde hasat zamanının önemli bir faktör olduğu belirlenmiştir.

Anahtar Kelimeler: Zeytinyağı, erken hasat, biyoaktif özellik, aroma kompozisyonu, kalite

Introduction

Olive (*Olea europea L.*) is one of the plants frequently grown in Mediterranean weather. 90% of the olives produced in the world are processed for oil (Gündeşli and Küden, 2020). Olive oil is the oil obtained from olive tree fruits by physical methods. Natural olive oils are oils that can be consumed without chemical processing (unrefined) (García-Vico et al., 2017; Perestrelo et al., 2017). The term early harvest olive oil is used for olive oils obtained by using olives of various ripeness, from green to pink, at the beginning of the olive harvest season (September-October) (Dıraman and Dibeklioğlu, 2009). The mono and polyunsaturated fatty acid ratios of early harvest olive oils are optimal, and the amounts of important compounds such as phenol, tocopherol and chlorophyll are high (Dag et al., 2011).

Considering its nutritional and health benefits, olive oil has an important place among animal and vegetable oils (Kılıç, 2020). Olive oil, one of the products of the Mediterranean diet, is frequently preferred by consumers due to its bioactive properties and unique aroma (Armutçu et al., 2013; Kiralan et al., 2021; Zarrouk et al., 2008). Olive oil gains its bioactive properties from substances such as phenolic compounds it contains and the antioxidant effects of these substances (Armutçu et al., 2013; Çakmak Arslan, 2022; Zarrouk et al., 2008). Phenolic compounds are also responsible for the sensory properties of olive oil, such as bitterness, astringency, and pungency (Büyükgök and Saygın Gümüşkesen, 2017). The main phenolic compounds found in olive oil are tyrosol, hydroxytyrosol, oleuropein, shikimic acid, coumaric acid, caffeic acid, and p-ferulic acid (Boskou, 2006). The fact that olive oil is rich in unsaturated fatty acids such as oleic acid also contributes to its bioactive properties (Perestrelo et al., 2017).

Aroma is one of the important parameters affecting the sensory properties of olive oil. The aroma of olive oil is composed of approximately 200 compounds, including groups such as aldehydes, esters, terpenes, ketones, alcohols, and hydrocarbons (Kesen et al., 2013; Kiritsakis, 1998). 50-75% of the compounds in olive oil aroma are from the aldehydes group (Kiritsakis, 1998). C-5 and C-6 compounds in olive oil are formed through the lipoxygenase chain reaction, and these compounds are responsible for the characteristic aroma of olive oils (Zarrouk et al., 2008). The main aroma substances found in olive oils include hexanal, (E)-2-hexenal, (Z)-3-hexen-1-ol, and 1-hexanol (Kiritsakis, 1998). The compounds responsible for the aroma of olive oil are affected by factors such as harvest, storage, variety, maturity, growing conditions, and processing technique (Kılıç, 2020).

The olive varieties with high production potential in Kilis province are Kilis yağlık and Gemlik. No study has been found in the literature regarding the aroma of early harvest Kilis yağlık olive oil. The aim of this study is to determine some properties of early and normally harvested Kilis yağlık natural olive oils. It is aimed to determine the physical, chemical, and bioactive compounds and properties of natural olive oils as well as aroma composition. Within the scope of the study, the changes caused by the harvest time in olive oils were examined.

Materials and Methods

Materials

Early harvest (October) and normal harvest (November) olive oils obtained from the 2022 crop were supplied from an olive oil factory in Kocabeyli village of Kilis province. The samples were kept in a dark, cool place and packaged until analysis. Analyzes were performed in triplicate and the results are given as mean±standard deviation.

Quality analysis

The peroxide value of the oils was determined titrimetrically according to the AOCS (Cd 8-53), iodine number AOCS (Cd 1-25), FFA, and acid number AOCS (Cd 3d-63) methods. The refractive index was read according to the AOCS (Cc 7-25) method with an Abbe refractometer (Soif WYA-

2S, China). L*, a*, and b* values were determined using the Konica Minolta Chroma Meter (CR-400, Japan) color measurement device with the AOCS (Cc 13e-92) method (AOCS, 1997). Hue (Hue˚) and chroma (ΔC*) values were calculated using the formulas below (Artes et al., 2002).

Hue˚= arctg(b*/a*) (1)

ΔC*= (a*2 +b*2)1/2 (2)

Total phenol and antioxidant capacity

For the analysis of total phenol content and antioxidant capacity, the extraction process was performed according to Sousa et al. (2014). After adding 2.5 mL hexane and 2.5 mL methanol-water (80:20 v/v) to the olive oils (4 mL), the mixture was centrifuged for 10 min. The extraction process was repeated twice by removing the upper phase. The total phenol content of the oil extracts obtained was determined spectrophotometrically at 760 nm (Shimadzu UV-1700, Japan) using the Folin Ciocalteu colorimetric method, and the results are given as mg GAE L^{-1} (Singleton et al., 1999). The antioxidant capacity of the oils was determined spectrophotometrically according to the DPPH*(2,2-diphenyl-1-picrylhydrazyl) radical scavenging capacity method. The extract (150 µL) and DPPH solution (2850 µL) were kept in the dark for 24 h, and absorbance was measured at 515 nm. Results were calculated as trolox equivalent (µmolTE mL^{-1}) (Thaipong et al., 2006).

Determination of aroma composition

The extraction of aroma compounds in natural olive oils was determined using the SPME technique with modifications to the method of Szkudlarz et al. (2003). Oil samples were placed in 20 mL vials, and adsorption of volatile compounds was achieved at 45°C for 50 min using divinylbenzene carboxen polydimethylsiloxane fiber (50/30 µm, 2 cm, DVB/CAR/PDMS, Supelco Inc., USA). Separation of aroma substances was carried out using a DB-HeavyWax column (60 m x 0.25 mm x 0.25 µm) on a Shimadzu GC-MS-QP2020 (Kyoto-Japan) mass spectrophotometer connected to a Shimadzu GC-2010 Plus (Kyoto-Japan) gas chromatograph. Injection temperature is 250°C. Oven temperature: It was brought from 40°C to 80°C by increasing 3°C per minute and held for 1 minute, and then it was brought to 240°C by increasing 5°C per minute and held for 6 minutes. The flow rate of helium used as carrier gas is 1.05 mL min^{-1}. Peaks; were identified by comparing with mass spectra in Wiley 7.0, NIST-98, and Flavor 2L libraries, and aroma compounds of olive oil samples were given as % peak area. Analyses were performed in triplicate.

Results and Discussions

Physical and chemical properties of natural olive oils

The physical and chemical properties of olive oils are among the factors that determine the quality of olive oil and are affected by the maturity level of the olive. The %FFA, acid, and peroxide values of early harvest natural olive oil were found to be significantly lower than natural olive oil (Table 1). This is an expected situation, as it is known that the oil content of early harvested olives is low, but the oil is of superior quality and is not subject to spoilage reactions. Free fatty acidity is an important factor in the classification of olive oils. According to the Turkish Food Codex Communique on Olive Oil and Pomace Oil, early harvest olive oil meets the 0.8% free fatty acidity criterion of natural extra virgin olive oil, while normal harvest olive oil exceeds this limit and is classified as natural first olive oil (Anonymous, 2017). According to the communique, the upper limit value of the peroxide number, which provides information about the degree of oxidation, is 20 meq O^2 kg^{-1}. This value was achieved in olive oils from both harvest periods. The number of free fatty acids and peroxides affected by enzymatic activities increases depending on harvest time and fruit maturity (Mele et al., 2018). FFA values of Kilis yağlık olive oils obtained from different years and locations varied between 0.33-0.86% and peroxide values between 2.33-6.85 meq O^2 kg^{-1} oil (Arslan and Özcan, 2014). Kıralan et al. (2009) reported the

FFA value of Kilis yağlık olive oil as 0.41% and the peroxide number as 6.24 meq O^2 kg^{-1} oil. Piscopo et al. (2018) found that the FFA value and peroxide number of olive oils obtained from the Grossa di Gerace variety were 0.39% and 5.77 meq O^2 kg^{-1} in the early harvest period; they reported it as 1.01% and 7.81 meq O^2 kg^{-1} during the normal harvest period. %FFA and peroxide values in the literature were found to be compatible with the study. Erdoğan (2020) supported the conclusion that ripening causes an increase in acidity by stating the FFA values of Kilis yağlık olive oil in 6 different maturity periods as 0.40, 0.55, 0.66, 0.58, 0.93, and 0.98%. FFA is produced by the oxidation of aldehydes contained in aroma substances. While these aldehydes decrease with ripening, free fatty acidity increases (Sadeghi et al., 2019). The results of the current study support this situation.

The iodine amount in olive oils was determined to be higher in early harvest. The iodine number in oils provides information about the saturation and unsaturation values of the oil. The decrease in iodine number is attributed to the destruction of double bonds as a result of oxidation and polymerization (Alireza et al., 2010). It is possible to say that early harvest olive oil has not been oxidized and thus has a higher iodine binding capacity. The refractive index of normal harvest olive oil was determined to be higher. The refractive index of oils is affected by the degree of saturation and the presence of conjugated double-bonded fatty acids (Arya et al., 1969).

Table 1. Physical and chemical properties of natural olive oils

	Early harvest natural olive oil	**Normal harvest natural olive oil**
FFA (oleic acid%)	0.26±0.00	1.54±0.00
Acid number	0.51±0.00	3.06±0.00
Peroxide value (meq O_2 kg^{-1})	5.43±0.31	11.29±1.01
Iodine number	85.80±4.02	79.74±4.54
Refractive index	1.4683±0.00	1.4696±0.00
L*	14.64±0.27	19.08±0.42
a*	1.96±0.10	2.42±0.15
b*	1.29±0.16	1.73±0.11
ΔC*	2.35±0.16	2.98±0.12
Hue°	33.34±2.54	35.65±2.73

L*, a*, b* values and ΔC* and Hue° results calculated from these values were determined to be higher in normal harvest natural olive oil. The increase in L* value indicates that normal harvest natural olive oil is lighter in color. A low a* value in early harvest olive oil indicates a greener oil color. A high b* value indicates that normal harvest natural olive oil is more yellow. These changes are attributed to the decrease in carotenoid and chlorophyll content with ripening in olives (Arslan and Özcan, 2014). Both color tone and color saturation were determined to be higher in normal harvest natural olive oil.

Piscopo et al. (2018), L*, a*, and b* values in olive oils were 7.67, 0.63, and 2.67, respectively, in the early harvest Carolea variety; 7.65, 0.52, and 2.45 in the normal harvest Carolea variety; 7.36, 0.83, and 3.19 in the early harvest Grossa di Gerace variety. They stated that the values were 7.81, 0.78, and 2.90 in the normal harvest Grossa di Gerace variety.

Bioactive Properties of Natural Olive Oils

Phenolic compounds, which eliminate the negative effects of free radicals, contribute to oxidative stability. Phenolic compounds, which are an important quality criterion in this respect, also affect color, flavor, and sensory properties. The total phenol content of early harvest natural olive oil was determined to be approximately six times higher than that of normal harvest natural olive oil (Table 2). Similar to the total phenol, the DPPH antioxidant capacity of early harvest natural olive oil was found to be much higher. It has been

stated in the literature that there is a parallelism between the total amount of phenolic substance and antioxidant capacity (Piscopo et al., 2018). It has been determined that early harvest has a high effect on the bioactive properties of natural olive oil. This is an expected situation, as there are differences in the composition of olives with early harvest.

Table 2. Bioactive properties of natural olive oils

	Early harvest natural olive oil	**Normal harvest natural olive oil**
Total phenol (mg GAE L^{-1})	516.61±3.74	77.70±2.27
DPPH (µmol TE mL^{-1})	787.80±4.93	280.27±1.92

Rotondi et al. (2004) reported that the phenol content of olive oils decreased with ripening, decreasing from 441 mg GAE kg-1 to 209 mg GAE kg-1. Skevin et al. (2003) determined the total phenol amount as 193-387 in early harvest olive oils and 91-173 mg caffeic acid kg-1 oil in normal harvest. Piscopo et al. (2018) reported that the amount of phenolic substance and antioxidant capacity in oils obtained from two different types of olives changed depending on the harvest time. In early harvest olive oils, the total phenol amount is 337 and 453 mg GAE kg-1 and DPPH capacity is 24.47 and 33.66%; in normal harvest olive oils, the total phenol amount is 269 and 353 mg GAE kg-1 and DPPH capacity is 16.68 and 23.13%. It is also supported by these studies in the literature that the total phenol amount decreases with fruit ripening. Total phenol amount of olive oilsvaries depending on the variety, harvest year, harvest time, processing, and growing conditions (Büyükgök and Saygın Gümüşkesen, 2017). It is thought that the differences in the amount of phenol substances in the literature are due to these factors.

Aroma compounds of natural olive oils

Volatile and semi-volatile compounds determine sensory characteristics, and aroma is a very important quality criterion for olive oil. A total of 29 different aroma compounds, including 9 aldehydes, 7 terpenes, 5 alcohols, 4 acids, 2 esters, 1 hydrocarbon, and 1 ketone were detected in early harvest natural olive oil, and 35 different aroma compounds, including 9 aldehydes, 8 terpenes, 6 alcohols, 5 acids, 4 esters, 2 ketones, and 1 hydrocarbon, were detected in normal harvest natural olive oil (Table 3).

Adehydes constitute the main aroma component group of olive oils (Kesen et al., 2013). The main aldehyde compounds in all oil samples were (E)-2-hexenal, hexanal, (E)-2-heptanal, and nonanal. A decrease is observed in many of the aldehydes produced with ripening in olives (da Silva et al., 2012). This decrease in aldehydes was also determined within the scope of the study. Since aldehydes are found in higher amounts in early harvest olive oils, green, grass, fruit, and raw odour are felt to be more dominant in these oils. Karagoz et al. (2017) support the study by stating that hexanal content decreases from 39.8% to 16.6% with ripening in olive oil. The ratio of hexanal/nonanal aldehydes gives information about the oxidation state. If this ratio falls below 2, it indicates that the oil is oxidized (Kesen et al., 2013). Both early harvest and normal harvest olive oils remained above the value of 2, showing that they were of good quality.

Volatile alcohols produced by the action of the alcohol dehydrogenase enzyme contribute significantly to the aroma of olive oil (Kesen et al., 2013). Alcohols found in high amounts in olive oils are (Z)-3-hexen-1-ol, 1-hexanol, and 1-penten-3-ol. The amount of volatile alcohol in olive oils increased with ripening. In a study conducted in Spain, the study was supported by reporting that the amount of 1-hexanol increased as the harvest time progressed (Gomez-Rico et al., 2009). Since 1-hexanol is formed by the transformation of hexanal, it is considered normal that aldehydes decrease and alcohols increase. Arapoglou (2010) reported that an increase in alcohol was observed

with ripening in oils obtained from Throumbolia and Koroneiki olive varieties.

Table 3. Aroma compounds of natural olive oils (%)

RT*	Aroma group	Aroma description	Aroma compounds	Early harvest natural olive oil	Normal harvest natural olive oil
11.441	Aldehyde	Grass, fruit	Hexanal	16.25±2.20	9.97±0.54
17.430	Alcohol	Green, vegetable	1-Penten-3-ol	1.81±0.15	2.25±0.03
18.886	Aldehyde	Green, leaf	(*E*)-2-Hexenal	19.30±4.26	16.82±1.10
20.453	Hydrocarbon	Fruit, grass	(*E*)-5-Octadecene	n.d.	1.82±0.45
20.868	Ketone	Plant, fruit, mushroom	3-Octanone	n.d.	1.98±0.26
21.933	Ester	Fruit, dessert, pear	Hexyl acetate	0.55±0.03	0.91±0.15
22.603	Aldehyde	Waxy, orange, herbaceous	Octanal	1.87±0.22	0.68±0.05
24.171	Aldehyde	Green, oily	(*E*)-2-Heptenal	9.80±2.10	6.69±0.50
24.807	Ketone	Grass, fresh, plant	Methyl heptenone	n.d.	1.77±0.11
25.390	Hydrocarbon	-	(*Z*)-1-Ethyl-2-methylcyclopentane	2.36±0.20	n.d.
26.350	Alcohol	Fruit, alcohol	1-Hexanol	5.03±0.75	5.39±0.06
26.572	Ester	Oily, green	1-Octen-3-ol acetate	n.d.	0.78±0.25
27.437	Alcohol	Green, fruit	(*Z*)-2-Hexen-1-ol	1.20±0.27	1.80±0.11
27.620	Alcohol	Grass, banana	(*Z*)-3-Hexen-1-ol	8.90±1.05	9.40±1.12
28.135	Aldehyde	Waxy, orange peel	Nonanal	1.97±0.96	2.88±0.51
27.970	Ester	Fruit, waxy	Hexyl butanoate	2.85±0.10	1.71±0.26
28.430	Aldehyde	Fruit, soap, oily	(*E*)-2-Octenal	0.30±0.05	0.15±0.10
29.026	Alcohol	Earthy, mushroom	1-Octen-3-ol	n.d.	0.73±0.41
29.678	Acid	Sour, vinegar	Acetic acid	3.47±0.56	4.98±0.54
30.645	Aldehyde	Fatty, vegetable	(*E,E*)-2,4-Heptadienal	2.40±0.10	0.40±0.08
31.665	Terpene	Woody, spice, honey	α-Copaene	3.23±0.58	3.05±0.13
32.037	Aldehyde	Oily, grass	(*E*)-2-Nonenal	0.10±0.00	0.09±0.08
32.209	Terpene	Citrus, flower	Linalool	2.03±0.73	1.77±3.56
32.674	Ester	Sweet, bergamot	Linalyl acetate	n.d.	0.94±5.47
33.275	Ketone	-	4-Methyl-3-octanone	2.72±0.06	n.d.
34.130	Terpene	Flower	Lavandulol	2.51±0.64	2.22±2.25
35.240	Aldehyde	Oily, earthy	(*E*)-2-Decenal	1.63±0.26	1.04±0.20
35.712	Terpene	Citrus, sweet	(*Z*)-β-Farnesene	0.30±0.17	0.29±0.10
35.775	Acid	Butter, sharp	Butanoic acid	1.94±0.40	2.50±0.72
36.588	Terpene	Flower	α-Terpineol	0.10±0.02	0.90±0.50
37.895	Terpene	Orange, lavender, green	α-Farnesene	1.10±0.11	0.67±0.20
38.178	Terpene	Flower, citrus	Citronellol	n.d.	1.74±0.45
40.185	Terpene	Flower, sweet	Geraniol	0.21±0.00	1.57±0.25
41.355	Acid	Sweet, sharp	Hexanoic acid	n.d.	1.98±0.14
41.955	Alcohol	Flower, rose, orchid	Phenylethyl alcohol	0.50±0.04	1.82±0.02
46.180	Acid	Oily, rancid	Octanoic acid	1.82±0.86	2.94±0.93
48.430	Acid	Cheese	Nonanoic acid	3.78±1.10	5.38±0.03

*RT: Retention time, n.d.: Not detected. Aroma descriptions taken from https://www.thegoodscentscompany.com/.

With maturation, the aroma composition of olive oil changes and undesirable volatile compounds increase. Butanoic, hexanoic, and acetic acid rates increase with autooxaidation and fermentation of olive oils (Gonzalez and Aporico, 2013). An increase in the rates of acids determined in the study was also observed. Volatile composition of olive oils is affected by

many factors such as variety, region, maturity level, harvesting and processing method, storage time and conditions, and aroma extraction method (da Silva et al., 2012). The differences between literatures arise from these factors.

Conclusions

This study was conducted to determine the physical, chemical, bioactive properties, and volatile compounds of olive oils obtained from the Kilis yağlık variety at different harvest times. Free fatty acidity and peroxide value were determined to be lower, and physical properties were superior in early harvest olive oil. It was observed that the total phenol and therefore the antioxidant capacity decreased with ripening. Aroma composition is an important quality criterion because it affects the sensory properties of olive oil. It has been determined that the rate of pleasant smelling compounds such as green, grass, fruits, and vegetables is higher in early harvest olive oil. It has been determined that undesirable aroma compounds occur or increase in olive oil with ripening. Olives need to be processed at the optimum harvest time, where they provide both high oil yield and high quality (high bioactive properties, desired aroma) oil that can be obtained from olives. It is thought that the harvest time of the study will be a reference to evaluate the quality criteria of olive oil.

Conflict of interest:

The authors declare that they have no competing interests.

Author contributions:

The authors have an equal contribution. All authors have read and agreed to the published version of the manuscript.

References

Alireza, S., Tan, C. P., Hamed, M., & Man, Y. B. (2010). Effect of frying process on fatty acid composition and iodine value of selected vegetable oils and their blends. International Food Research Journal, 17, 295-302.

Anonymous (2017). Zeytinyağı ve pirina yağı tebliği. Türk Gıda Kodeksi, Tebliğ No: 2017/26, Ankara.

AOCS (1997). Official methods and recomended practices of analysis of association of official chemist. AOCS, 5th Edition, Washington DC, ABD.

Arapoglou, D. (2010). Characterization and seasonal variation of the quality of virgin olive oil of the Throumbolia and Koroneiki varieties from Southern Greece. Grasas y Aceites, 61, 221-231. DOI: https://doi.org/10.3989/gya.108709.

Armutçu, F., Namuslu, M., Yüksel, R., & Kaya, M. (2013). Zeytinyağı ve sağlık: Biyoaktif bileşenleri, antioksidan özellikleri ve klinik etkileri. Konuralp Medical Journal, 5(1), 60-68.

Arslan, D., & Özcan, M. M. (2014). Changes in chemical composition and olive oil quality of Turkish variety 'Kilis Yağlık' with regard to origin of plantation. Global Journal of Agricultural Innovation, Research & Development, 1, 51-56. DOI: https://doi.org/10.15377/2409-9813.2014.01.02.3

Artes, F., Minguez, M. I., & Hornero, D. (2002). Analysing changes in fruit pigments. In D. MacDougall (Ed.), Colour in Food: Improving Quality (pp. 248-282). Cambridge: CRC Press/Woodhead Publishing.

Arya, S. S., Ramanujam, S., & Vijayaraghavan, P. K. (1969). Refractive index as an objective method for evaluation of rancidity in edible oils and fats. Journal of the American Oil Chemists Society, 46, 28-30. DOI: https://doi.org/10.1007/BF02632705

Boskou, D. (2006). Olive Oil: Chemistry and Technology. Washington: AOCS Publishing.

Büyükgök, E. B., & Saygın Gümüşkesen, A. (2017). Influence of olive ripeness degree and harvest year on chemical and sensory properties of Kilis Yağlik and Memecik olive oil. Gıda, 42(6), 799-806. DOI: https://doi.org/10.15237/gida.GD17077

Çakmak Arslan, G. (2022). Natürel sızma zeytinyağının kısa ve uzun süreli ısıl işlem sırasındaki stabilitesinin riviera zeytinyağı ile karşılaştırmalı olarak değerlendirilmesi. Düzce Üniversitesi Bilim ve Teknoloji Dergisi, 10(3), 1615-1630. DOI: https://doi.org/10.29130/dubited.1110616

da Silva, M., Freitas, A. M. C., Cabrita, M., & Garcia, R. (2012). Olive oil composition: Volatile compounds. In D. Boskou (Ed.), Olive Oil-Constituents, Quality, Health Properties and Bioconversions (pp. 17-46). London: IntechOpen.

Dag, A., Kerem, Z., Yogev, N., Zipori, I., Lavee, S., & Ben-David, E. (2011). Influence of time of harvest and maturity index on olive oil yield and quality. Scientia Horticulturae, 127(3), 358-366. DOI: https://doi.org/10.1016/j.scienta.2010.11.008

Dıraman, H., & Dibeklioğlu, H. (2009). Characterization of Turkish virgin olive oils produced from early harvest olives. Journal of the American Oil Chemists Society, 86(7), 663-674. DOI:

https://doi.org/10.1007/s11746-009-1392-5

Erdoğan, İ. (2020). Kilis'te yetiştirilen Kilis yağlık zeytin çeşidinin fenolojik ve pomolojik özellikleri ile zeytinyağının yağ asitleri kompozisyonu (Yayımlanmamış yüksek lisans tezi). Kilis 7 Aralık Üniversitesi Lisansüstü Eğitim Enstitüsü, Kilis.

García-Vico, L., García-Rodríguez, R., Sanz, C., & Pérez, A. G. (2017). Biochemical aspects of olive freezing-damage: Impact on the phenolic and volatile profiles of virgin olive oil. LWT- Food Science and Technology, 86, 240-246. DOI: https://doi.org/10.1016/j.lwt.2017.07.052

Gomez-Rico, A., Inarejos-Garcia, A. M., Salvador, 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, 3587-3595. DOI: https://doi.org/10.1021/jf803505w

Gonzalez, D. L. G., & Aporico, R. (2013). Instrumental approaches to understand the sensory quality of virgin olive oil. In: Workshop on Olive Oil Authentication, (pp. 70-86), 10-11 June, Madrid, Spain.

Gündeşli, K., & Küden, A. (2020). Bazı yerli ve yabancı zeytin çeşitlerinin meyve kalite özelliklerinin ve soğuklama gereksinimlerinin saptanması. Anadolu Journal of Agricultural Sciences, 35, 1308-8769. DOI: https://doi.org/10.7161/omuanajas.655591

Karagoz, S. G., Yilmazer, M., Ozkan, G., Carbonell-Barrachina, A. A., Kiralan, M., & Ramadan, M. F. (2017). Effect of cultivar and harvest time on C6 and C5 volatile compounds of Turkish olive oils. European Food Research and Technology, 243(7), 1193-1200. DOI: https://doi.org/10.1007/s00217-016-2833-7

Kesen, S., Kelebek, H., Sen, K., Ulas, M., & Selli, S. (2013). GC–MS–olfactometric characterization of the key aroma compounds in Turkish olive oils by application of the aroma extract dilution analysis. Food Research International, 54, 1987-1994. DOI: https://doi.org/10.1016/j.foodres.2013.09.005

Kılıç, S. (2020). Kazdağları bölgesine ait zeytin, zeytin yaprağı ve zeytinyağlarının element tayini ve metot validasyonu. Eurasian Journal of Biological and Chemical Sciences, 3(1), 48-54. DOI: https://doi.org/10.46239/ejbcs.739834

Kıralan, M., Bayrak, A., & Özkaya, M. T. (2009). Oxidation stability of virgin olive oils from some important cultivars in East Mediterranean Area in Turkey. Journal of the American Oil Chemists' Society, 86, 247-252. DOI: https://doi.org/10.1007/s11746-008-1337-4

Kiralan, S. S., Karagoz, S. G., Ozkan, G., Kiralan, M., & Ketenoğlu, O. (2021). Changes in volatile compounds of virgin olive oil flavored with essential oils during thermal and photo-oxidation. Food Analytical Methods, 14, 883-896. DOI: https://doi.org/10.1007/s12161-020-01926-w

Kiritsakis, A. K. (1998). Flavor components of olive oil—A review. Journal of the American Oil Chemists' Society, 75, 673-681. DOI: https://doi.org/10.1007/s11746-998-0205-6

Mele, M. A., Islam, M. Z., Kang, H. M., & Giuffre, A. M. (2018). Pre-and post-harvest factors and their impact on oil composition and quality of olive fruit. Emirates Journal of Food and Agriculture, 30(7), 592-603. DOI: https://doi.org/10.9755/ejfa.2018.v30.i7.1742

Perestrelo, R., Silva, C., Silva, P., & Câmara, J. S. (2017). Global volatile profile of virgin olive oils flavoured by aromatic/medicinal plants. Food Chemistry, 227, 111-121. DOI: https://doi.org/10.1016/j.foodchem.2017.01.090

Piscopo, A., Zappia, A., De Bruno, A., & Poiana, M. (2018). Effect of the harvesting time on the quality of olive oils produced in Calabria. European Journal of Lipid Science and Technology, 120(7), 1700304. DOI: https://doi.org/10.1002/ejlt.201700304

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, 3649-3654. DOI: https://doi.org/10.1021/jf049845a

Sadeghi, E., Mahtabani, A., & Karami, F. (2019). Considerıng the oxidative stability of cold-pressed sesame, sunflower, and olive oils under different storage conditions. The Annals of the University of Dunarea De Jos of Galati Fascicle VI- Food Technology, 43(2), 70-83. DOI: https://doi.org/10.35219/foodtechnology.2019.2.05

Singleton, V. L., Orthofer, R., & Lamuela-Raventos, R. M. (1999). Analysis of total phenols and other oxidation substrates and antioxidants by means of Folin-Ciocalteu reagent. Methods in Enzymology, 299, 152-178. DOI: https://doi.org/10.1016/S0076-6879(99)99017-1

Skevin, D., Rade, D., Strucelj, D., Mokrovsak, Z., Nederal, S., & Bencic, D. (2003). The influence of variety and harvest time on the bitterness and phenolic compounds of olive oil. European Journal of Lipid Science and Technology, 105, 536-541. DOI: https://doi.org/10.1002/ejlt.200300782

Sousa, C., Gouvinhas, I., Barreira, D., Carvalho, M., Vilela, A., Lopes, J., Martins-Lopes, P., & Barros, A. N. (2014). Cobrançosa' olive oil and drupe: chemical composition at two ripening stages. Journal of the American Oil Chemists' Society, 91(4), 599-611. DOI: https://doi.org/10.1007/s11746-013-2406-x

Szkudlarz, S., Jelen, H., Wojtasiak, R., & Wasowicz, E. (2003). Application of headspace-solid phase micro-extraction and multivariate analysis for plant oils differentiation. Food Chemistry, 83, 515-522. DOI:

https://doi.org/10.1016/S0308-8146(03)00147-X

Thaipong, K., Boonprakob, U., Crosby, K., Cisneros-Zevallos, L., & Byrnec, L. (2006). Comparison of ABTS, DPPH, FRAP, and ORAC assays for estimating antioxidant activity from guava fruit extracts. Journal of Food Composition and Analysis, 19, 669-675. DOI: https://doi.org/10.1016/j.jfca.2006.01.003

Zarrouk, W., Haddada, F. M., Baccouri, B., Oueslati, I., Taamalli, X. F., Lizzani-Cuvelier, L., Daoud, D., & Zarrouk, M. (2008). Characterization of virgin olive oil from Southern Tunisia. European Journal of Lipid Science and Technology, 110, 81-88. DOI: https://doi.org/10.1002/eilt.200700085