**GRASAS Y ACEITES** 74 (2)
April-June 2023, e504
ISSN-L: 0017-3495
https://doi.org/10.3989/gya.0666221

# Elucidation of key odorants and sensory properties of five different extra virgin olive oils from Turkey by GC-MS-Olfactometry

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*Submitted: 01 June 2022; Accepted: 14 September 2022; Published online: 13 June 2023*

**SUMMARY:** The present study investigates the aroma, key odorants and sensory profile of extra virgin olive oils from five well-known Turkish cultivars. The aromatic extract obtained by the purge and trap extraction system, according to a sensory analysis, resembled the odor of olive oil. A total of 22, 21, 18, 22 and 21 aroma-active compounds were detected in the extracts of Ayvalık, Memecik, Gemlik, Sarı Ulak and Beylik olive oils, respectively. The results show that Ayvalık has the highest flavor dilution (FD) value of 1024 with hexanal, *(E)*-2-hexenal and *α*-farnesene. Memecik has the highest FD value at 2048 with (E)-2-hexenal. Gemlik has the highest FD value of 1024 with *(Z)*-3-hexenyl acetate, (E)-2-hexen-1-ol and *α*-farnesene. Sarı Ulak has the highest FD value at 2048 with *(E)*-2-hexenal. Beylik has the highest FD value of 2048 with *(E)*-2-hexenal and hexanal. All cultivars represent the characteristic green, cut-grass, fruity odor notes.

***KEYWORDS:*** *AEDA; Aroma-Active Compounds; EVOO; GC-MS-O; Olive oils.*

**RESUMEN:** ***Interpretación de olores clave y propiedades sensoriales de cinco aceites de oliva virgen extra diferentes de Turquía utilizando GC-MS-Olfatometría.*** El presente estudio investiga el aroma, los olores clave y el perfil sensorial de los aceites de oliva virgen extra de cinco cultivares turcos bien conocidos. El extracto aromático obtenido por el sistema de extracción mediante purga y trampa, según el análisis sensorial, se asemejaba al olor del aceite de oliva. Se detectaron un total de 22, 21, 18, 22 y 21 compuestos aromáticos activos en los extractos de los aceites de oliva Ayvalık, Memecik, Gemlik, Sarı Ulak y Beylik, respectivamente. Los resultados muestran que Ayvalık tiene el valor de dilución de sabor (FD) más alto de 1024 con hexanal, (E)-2-hexenal y α-farnesene, Memecik tiene el valor de FD más alto para 2048 con (E)-2-hexenal, Gemlik tiene el valor de FD más alto de 1024 con acetato de (Z)-3-hexenilo, (E)-2-hexen-1-ol y α-farneseno, Sarı Ulak tiene el valor de FD más alto de 2048 con (E)-2-hexenal, Beylik tiene el valor de FD más alto de 2048 con (E)-2-hexenal y hexanal, todos ellos representando las características de notas de olor afrutado, hierba cortada y verde.

***PALABRAS CLAVE:*** *Aceites de oliva; AEDA; AOVE; Compuestos Aromáticos-Activos; GC-MS-O.*

**Citation/Cómo citar este artículo**: Sevim D, Köseoğlu O, Kadiroğlu P, Guclu G, Ulaş M, Selli S. 2023. Elucidation of key odorants and sensory properties of five different extra virgin olive oils from Turkey by GC-MS-Olfactometry. *Grasas Aceites* **74** (2), e504. https://doi.org/10.3989/gya.0666221

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## 1. INTRODUCTION

Virgin olive oil (VOO) is different from other vegetable oils because only mechanical and/or physical procedures are used to obtained VOO from the olive fruit of the olive tree, *Olea europaea* L. Since it is not subjected to additional refining, there is no change in the volatile and non-volatile components. Therefore, the sensory and nutritional properties of VOO are also protected (Perestrelo *et al.,* 2017). Its composition depends on several factors such as production area, degree of fruit ripening and quality of olives, cultivar, climate conditions of regions, and the process systems (Ozturk *et al.,* 2021; Perestrelo *et al.,* 2017). Fresh and good-quality Extra Virgin Olive Oils (EVOOs) are distinguished by consumers and differentiated by their flavor and aroma (Kesen *et al.,* 2014). The aroma properties of VOOs, and especially the positive features of green and fruity, depend on many volatile components in VOO which are produced by enzymatic processes. The lipoxygenase pathway, which involves several volatile components resulting from the breakdown of polyunsaturated fatty acids, is a well-known enzymatic method for creating attractive aromatic volatiles in VOOs (Amanpour *et al.,* 2016).

Aroma components are among the most crucial agents which affect the quality of VOOs and play a vital role in consumer preference. The volatile composition of VOO is known to consist of hundreds of volatile compounds including unsaturated aldehydes, ketones, alcohols, esters, furans and terpene compounds present in low concentrations, from a few ppm or even less (Perestrelo *et al.,* 2017). Gas chromatography-olfactometry (GC-O) can be used to detect these compounds, which are typically classified as odor-active or non-odor-active, based on their current quantity (Ben Brahim *et al*., 2018). The aroma-active compounds (AAC) in Turkish VOOs have been the subject little research. Kesen *et al.* (2013) utilized the aroma extract dilution analysis (AEDA) for the first time in Turkish VOOs and found that guaiacol, 1-penten-3-ol, hexanal, octanal and (Z)-3-hexenyl acetate were the aroma actives with the highest FD factors in VOOs.

The most important feature which distinguishes EVOO from other oils is its smell and special taste. Its characteristic aroma shows green and fruity properties due to volatile components, some of which come directly from the fruit and some which are due to the degradation of polyunsaturated fatty acids as a result of lipoxygenase (LOX) enzyme activity (Guclu *et al.,* 2016). The sensory characteristics of olive oil primarily depend on the concentration and nature of the volatile compounds found in olives (Genovese *et al.,* 2021). Therefore, olive oils are evaluated in a sensory analysis, for positive (fruity, bitter, pungent) and negative (warming-muddy residue, moldy-moist, vinous-vinegar, metallic, stinking (old-stale), heated or burnt, straw-woody, coarse, machinery. oil, black water, salt water, whitish, earthy, wormy, cucumber, wet wood) properties (IOC, 2018).

In Turkey, Ayvalık, Memecik and Gemlik are the most dominant and important olive varieties with distribution of 20.66%, 19.11% and 48.71%, respectively. Also, Beylik and Sarı Ulak varieties are among the important varieties of West and East Mediterranean Regions, respectively. The main aims of this investigation were: i) to identify the volatile compounds in VOOs obtained from the economically important olives of Ayvalık, Memecik, Gemlik, Sarı Ulak and Beylik with the three-phase extraction system, ii) to detect the ACCs with the AEDA approach and GC-MS-Olfactometry and (iii) to determine the sensory profiles of the samples.

## 2. MATERIALS AND METHODS

### 2.1. Sampling

EVOOs were used in this work. The EVOOs from the Ayvalık (AY), Memecik (ME), Gemlik (GE), Sarı Ulak (SU) and Beylik (BE) cultivars were collected from the South Aegean, North Aegean, South Marmara, West and East Mediterranean Regions in Turkey, respectively. All olives were harvested during the yellowish-purplish period, which we call the ideal harvest time. All the EVOOs provided by the producers were obtained under a three-phase extraction system during the 2014/15 and 2015/16 crop seasons. The olives were crushed with a hammer crusher after leaf separation and washing. They were then subjected to malaxation at 30-35 °C for 30-45 minutes. Then, olive oil, pomace and olive oil mill wastewater were separated from the olive paste with the help of a decanter, and the olive oil obtained was purified from remaining impurities by passing it through a separator with 200 L of water per hour. 500 mL were taken from each sample and then stored in bottles at 4 °C. Quality parameters (QP), volatile

compounds and GC-MS-O, aroma extract dilution, and sensory analyses were performed for both crop seasons.

### 2.2. Quality parameters of samples

The QP detected were free fatty acidity (FFA) (represented as an oleic acid percentage) (IOC, 2017a), peroxide value (PV) (represented as meq$O_2$/kg of oil) (IOC, 2017b) and characteristics of ultraviolet absorption at 232 and 270 wavelengths ($K_{232}$ and $K_{270}$) (IOC, 2019). The samples were analyzed in triplicate.

### 2.3. Sensory assessment of samples

The sensorial evaluation of the EVOO was performed by an IOC (International Olive Council)-approved panel of fully qualified judges. The samples were sensory analyzed according to the parameters outlined in the IOC approved technique COI/T.20/Doc. no 15 (IOC, 2018). A 15-ml sample was placed in a blue tasting glass. The temperature of the sample was maintained at 28±2 °C. The sensory assessment of the sample was defined using the median of panelists' scores obtained via sensory analysis.

### 2.4. Analysis of volatile compounds and GC-MS-Olfactometry conditions

The aroma substances in the EVOO samples were analyzed using the purge and trap extraction technique. Representative tests were performed on the aromatic extract to determine the extraction method's reliability. The aroma compounds of the EVOOs were extracted according to the method of Amanpour *et al.* (2016). The extract was passed through sodium sulfate and concentrated to 0.5 mL at 40 °C in a "Vigreux" distillation column. The concentrated extract was directly injected into GC-FID (Flame Ionization Detector), GC-MS and GC-MS-O systems and the AACs were determined. The extractions were performed in three replicates (Kesen *et al.*, 2013). The GC with a flame ionization and a mass selective detector (Agilent 5973, USA), and a sniffing port (Gerstel ODP-2, USA) were used in the GC system at 250 ºC. A capillary column (DB-WAX 0.25 mm x 0.4 m x 60 m) was used for the separation of the aroma components. Chemical standards, retention index, and a mass spectral database were used to identify aroma compounds (NIST 98, Wiley 11). The injector and temperature were set to the same parameters as the GC. The substances were quantified in scan mode using a mass range of 29-350 amu and mass spectra were acquired in electron impact mode (energy voltage: 70 eV). Peaks were identified using standard solutions. The internal standard ((4-nonanol) method was used to calculate the volatile concentrations.

### 2.5. Aroma active compounds of samples

Three qualified sniffers used GC–MS–O to evaluate the AACs. The extract sniffing process was completed in three stages (25 min each). Based on previous research, the AEDA technique was used to determine the FD factors of the AAC (Ozkara *et al.,* 2019). $CH_2Cl_2$ was used to dilute the EVOOs: 1:1, 1:2, 1:4, … 1:4096. Sniffing continued until no odorant was detected.

### 2.6. Statistical analysis

Analysis of variance (ANOVA) and Principal component analysis (PCA) were carried out on the Minitab® 17 program (Minitab Inc., State College, PA, USA) to reveal the discrimination pattern of the EVOO samples.

## 3. RESULTS AND DISCUSSION

### 3.1. Quality parameters of samples

The general QP of the samples are shown in Table 1. As seen, the FFA, PV, $K_{232}$ and $K_{270}$ values of the VOOs did not exceed the limit defined for EVOO by IOC (IOC, 2021). The percentage of FFA in the oils ranged from 0.30 to 0.59 for the 2014 harvest year and from 0.22 and 0.77 for the 2015 harvest year. FFA is known to be the main criterion for classifying VOO. All the samples were determined to contain less than the maximum legal limit of 0.8 (oleic acid %) for EVOO (IOC, 2021). The PV of the oils ranged from 5.07 to 9.73 meq$O_2$/kg oil and from 4.59 to 12.33 meq$O_2$/kg oil, for 2014 and 2015 harvest years, respectively. The results showed that the PV of the samples were below the limit of 20 meq$O_2$/kg oil as established by the IOC (IOC, 2021) for classifying EVOO. According to the IOC limit, $K_{232}$ and $K_{270}$ values must be less than 2.50 and 0.22 for EVOO, respectively. The $K_{232}$ and $K_{270}$ values of the oils were determined to be below this legal limit.

**Table 1.** Quality parameters of samples

| Year | Sample | FFA (oleic acid %) | PV (meq $O_2$/kg oil) | $K_{232}$ | $K_{270}$ |
|---|---|---|---|---|---|
| | **ME** | 0.30 ± 0.01 | 6.88 ± 0.06 | 1.74 ± 0.02 | 0.15 ± 0.05 |
| | **AY** | 0.59 ± 0.00 | 8.68 ± 0.08 | 1.71 ± 0.05 | 0.11 ± 0.04 |
| 2014 | **GE** | 0.52 ± 0.03 | 5.75 ± 0.05 | 1.68 ± 0.04 | 0.12 ± 0.02 |
| | **SU** | 0.48 ± 0.01 | 9.73 ± 0.04 | 1.62 ± 0.06 | 0.12 ± 0.03 |
| | **BE** | 0.32 ± 0.03 | 5.07 ± 0.03 | 2.12 ± 0.05 | 0.18 ± 0.06 |
| | **ME** | 0.22 ± 0.00 | 8.24 ± 0.04 | 1.97 ± 0.05 | 0.17 ± 0.04 |
| | **AY** | 0.66 ± 0.02 | 9.83 ± 0.07 | 1.84 ± 0.03 | 0.16 ± 0.05 |
| 2015 | **GE** | 0.52 ± 0.01 | 4.59 ± 0.04 | 1.79 ± 0.04 | 0.14 ± 0.03 |
| | **SU** | 0.36 ± 0.01 | 7.61 ± 0.06 | 1.67 ± 0.06 | 0.13 ± 0.02 |
| | **BE** | 0.77 ± 0.03 | 12.33 ± 0.05 | 2.37 ± 0.05 | 0.20 ± 0.05 |

Values expressed as mean ± standard deviation AY: Ayvalık, ME: Memecik, GE: Gemlik, SU: Sarı Ulak and BE: Beylik. Experiments were conducted 3 times.

The results agree with previous studies carried out with cv. AY, MY and GE (Kesen *et al.,* 2013), AY and ME (Karagoz *et al*., 2017), ME and GE (Köseoğlu *et al.,* 2016). ANOVA analysis was performed to determine the significance of difference according to the varieties of olive oil samples. The difference among the means of the quality parameter results was not found significant at the 95% confidence level.

### 3.2. Sensory assessment of samples

Sensory analysis is a quality criterion in VOO standards, and evaluating the sensory quality of VOOs comprises assessing positive and negative properties. The results of the sensory assessment of the samples are shown in Figure 1. According to the IOC standard (IOC, 2021), samples are classified as EVOOs if the median of defects is "0" and the median of fruity is greater than "0". Sensory assessment showed that the studied oils had no defects and therefore considered as EVOOs. The highest fruity medians were found at 5.3 and 4.95 for the SU EVOO, from 2014 and 2015, respectively. With regards to the bitterness value, the highest medians were detected in the BE EVOO at 4 and 4.4 from 2014 and 2015, respectively. The pungent values were determined at the highest values in the AY (4.0), ME (4.0) and BE (4.0) EVOO from 2014 and in the BE (4.6) EVOO from 2015. According to the IOC standard (IOC, 2018) the term, "robust" can be used when the median of the positive attribute is more than "6.0", "medium" can be used when the median of the attribute is between "3.0" and "6.0" and "delicate" can be used when the median of attribute is less than "3.0". In this research, all samples were characterized as medium for fruity properties from both harvest years. Regarding the the bitterness value, the BE EVOO was classified as medium from 2014 and the AY, ME and BE EVOOs were classified as medium from 2015. The other samples were characterized as delicate. When we look at the results of pungent values, we can characterize the samples as medium for both years, except for the SU EVOO. SU EVOO was classified as delicate.

### 3.3. Volatile compounds of samples

Table 2 shows the identified and classified (µg/kg) volatile components in the samples. As shown in Table 2, a total of 52, 57, 51, 57 and 54 compounds, including aldehydes, alcohols, terpenes, acids, volatile phenols, ketones, esters, lactones, hydrocarbons and furans, were qualitatively and quantitatively identified in AY, ME, GE, SU and BE EVOO, respectively. GC–MS chromatograms of EVOOs are shown in Figure 2. As seen from these chromatograms, the most volatile components were found in BE EVOO. The highest amount of total volatile compounds (45364 and 31990 µg/kg) was determined in the BE EVOO, for 2014 and 2015, respectively. It was followed by the AY EVOO (34890.1 µg/kg) from 2014 and the SU EVOO (15282 µg/kg) from 2015. The lowest volatile compounds were found in the GE

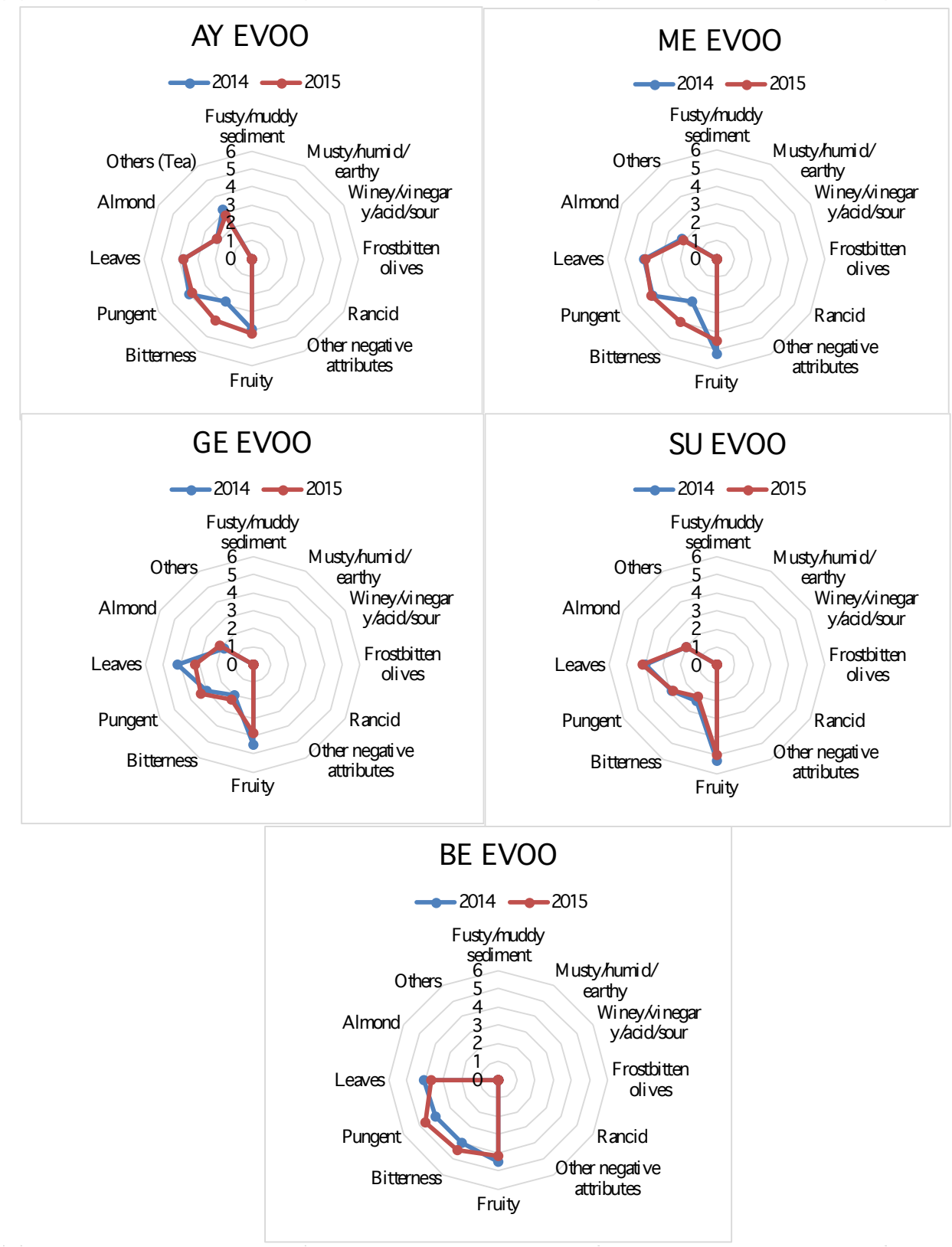


**Figure 1.** Sensory spider plot of Ayvalık, Memecik, Gemlik, Sarı Ulak and Beylik Extra Virgin Olive Oils. AY: Ayvalık, ME: Memecik, GE: Gemlik, SU: Sarı Ulak and BE: Beylik

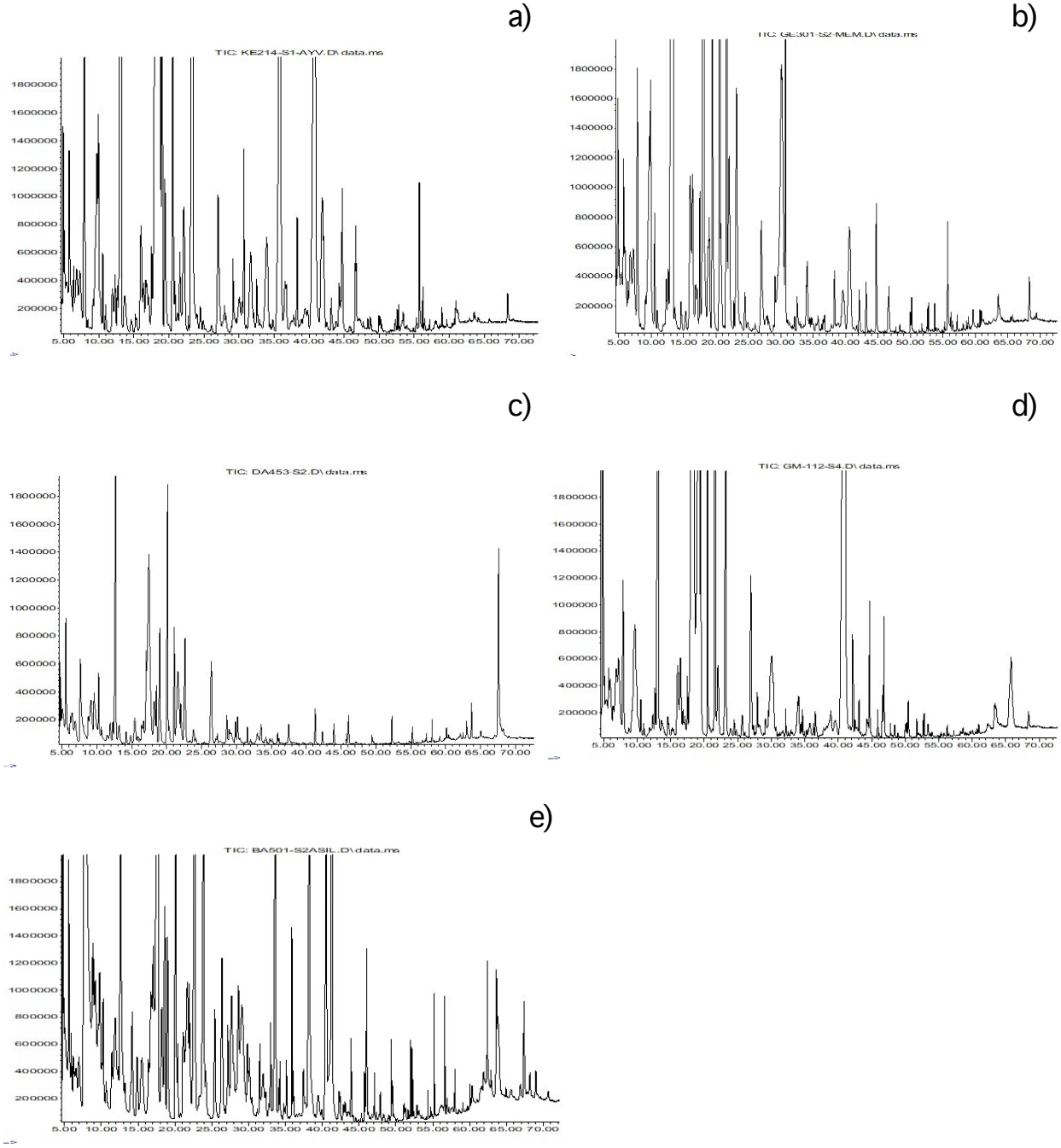


**Figure 2.** Volatile compound chromatogram of Ayvalık (a), Memecik (b), Sarı Ulak (c), Gemlik (d) and Beylik (e) Extra Virgin Olive Oils.

EVOO (20947.8 and 8677.8 µg/kg) from 2014 and 2015, respectively. The majority of the volatile components found in this research had been previously identified in the VOOs of the same and different olive cultivars (Aparicio and Morales, 1998; Vichi *et al*., 2007; Baccouri *et al*., 2008; Giuffrè *et al*., 2019; Žanetić *et al*., 2021). Among the volatile compounds detected in different EVOOs, aldehydes were identified and quantified as the major components with regard to the volatile part, followed by alcohols. The lipoxygenase pathway is activated during the olive oil extraction process, resulting in the release of en-

zymes. Aldehyde compounds are produced by the hydroperoxide lyase enzyme, which is then reduced into alcohols by the alcohol dehydrogenase enzyme in VOOs (Amanpour *et al.,* 2016). C5 and C6 aldehydes and alcohols are the four most common chemical groups of the 18 components which contribute positively to the aroma composition of VOOs from the positive sensory properties (Procida *et al*., 2016). As previously stated by Issaoui *et al.* (2015), Caporaso *et al.* (2016) and Žanetić *et al*. (2021) the volatile component profile of VOOs is affected by the region where it is grown, the geographical origin, the pedoclimatic conditions, the variety, the extraction systems and VOO storage conditions. It was also seen in the study that the volatile component profile changed according to year, variety and region.

***Aldehydes.*** A total of 7, 8, 4, 11 and 12 aldehydes and 7, 8, 3, 10 and 12 aldehydes were identified in AY, ME, GE, SU and BE EVOO from 2014 and 2015, respectively. The primary aldehyde compounds in EVOO were (E)-2-hexenal and hexanal (Table 2). Total concentrations of aldehydes were found in the AY EVOO at 14055.7 and 5440.1 µg/kg, in the ME EVOO at 14722.2 and 6277.5 µg/kg, in the GE EVOO at 3465.8 and 993 µg/kg, in the SU EVOO at 20894.8 and 8194.4 µg/kg and in the BE EVOO at 27598.9 and 17346 µg/kg, for the 2014 and 2015 seasons, respectively. According to the results, the aldehyde compounds were higher in 2014 than in 2015. In the SU EVOO (E)-2-hexenal was determined to be the highest aldehyde compound with 14280 and 5265 µg/kg, and it was followed by the ME EVOO with 12363 and 5145 µg/kg, for 2014 and 2015, respectively. In the BE EVOO hexanal was found to be the highest aldehyde compound with 8753 and 6312 µg/kg, and it was followed by the AY EVOO with 4790 and 1091 µg/kg, and ME EVOO 886 and 468 µg/kg, for 2014 and 2015, respectively. It was reported by other authors (Kesen *et al*., 2014; Sacchi *et al.,* 2015; Ben Brahim *et al*., 2018; Giuffrè *et al*., 2019; Žanetić *et al.,* 2021) that (E)-2-hexenal and hexanal are common aldehydes in many VOOs, including AY, GE, ME, Halhalı, Nizip Yağlık, and Kilis Yağlık from Turkey, Mari from Iran, Jemri, Touffehi and Fakhari from Tunisian, Arbequina, Cornicabra, Morisca, Picolimon, Picudo and Picual from Spain, and Ravece from Italy. According to studies, the percentage of C6 aldehydes, particularly (E)-2-hexenal, increased during olive ripening, which was primarily detected when the olive fruit skin color changed from yellow-green to purple (Ben Brahim *et al.,* 2018). The amount of hexanal. which was the second major aldehyde in the samples, mostly decreased with maturation. In this study, the results are in agreement with these reports, that our samples were harvested during the yellowish-purplish period, which we consider the ideal harvest time. Among the aldehydes, (E)-2-hexenal and hexanal are responsible for the positive green sensory attributes in EVOO. The results showed that our samples' positive sensory attributes are in accordance with this criterion.

***Alcohols.*** Alcohols are associated with positive sensory properties such as green, bitter, fruity aromatic, and they present weaker sensory attributes than aldehydes (Žanetić *et al.,* 2021). Alcohols produced by the ADH enzyme, are found in plants and are responsible for the production of volatile alcohols which contribute to the aroma of VOO (Kesen *et al*., 2014). A total of 12, 16, 14, 16 and 15 alcohols and 16, 16, 11, 14 and 15 alcohols were determined in the AY, ME, GE, SU and BE EVOOs in the 2014 and 2015 seasons, respectively. In all EVOO samples (Table 2) alcohols were determined to be the second main group of volatile compounds, as confirmed in previous studies (Kesen *et al.,* 2014; Karagoz *et al*., 2017; Žanetić *et al*. 2021). The highest amount of alcohols was found in the first year, which was likely due to increased ADH enzyme activity, and determined in the ME EVOO at 6767.1 µg/kg, followed by the SU EVOO at 5820.9 µg/kg, and the GE EVOO at 5310.5 µg/kg, the BE EVOO at 5193 µg/kg and the AY EVOO at 5094.0 µg/kg. The second year, the highest total amount of alcohols was found in the BE EVOO at 4108 µg/kg, followed by SU EVOO at 3678 µg/kg, the ME EVOO at 3294.8 µg/kg, the GE EVOO at 3038 µg/kg and the AY EVOO at 2587.5 µg/kg. The results showed that (Z)-3-hexen-1-ol, (E)-2-hexen-1-ol and 1-hexenol were the dominant C6 alcohols in all the analyzed samples. These results are in agreement with other researchers (Baccouri *et al.,* 2008; Karagoz *et al.,* 2017). The contents of (Z)-3-hexen-1-ol main alcohols in the ME EVOO were 1625 µg/kg and 1115 µg/kg in the BE EVOO from 2014 and 2015, respectively. (Z)-3-hexen-1-ol is the most prominent green note, and in our study this chemical appears to have a green-leaf characteristic similar to freshly cut grass. (E)-2-hexen-1-ol were determined to be the predom-

**Table 2.** Volatile compounds in samples (µg/kg)

| | LRI[a] | Compounds | Concentration (µg/kg)[b] | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | SU | | ME | | AY | | GE | | BE | |
| **Aldehydes** | | | **2014** | **2015** | **2014** | **2015** | **2014** | **2015** | **2014** | **2015** | **2014** | **2015** |
| 1 | 8.006 | Hexanal | 2379 | 1083 | 886 | 468 | 4790 | 1091 | 475 | 125 | 8753 | 6312 |
| 2 | 9.187 | *(E)*-2-Pentenal | 148 | 139 | 87.4 | 82.0 | 73.8 | 72.6 | ND | ND | 188 | 237 |
| 3 | 9.923 | *(Z)*-3-Hexenal | ND | ND | 623 | 131 | ND | ND | ND | ND | ND | ND |
| 4 | 9.982 | 3-Hexenal | 996 | 350 | ND | ND | ND | ND | ND | ND | 3243 | 1041 |
| 5 | 12.344 | Heptanal | 177 | 95.0 | ND | ND | ND | ND | ND | ND | 1188 | 1065 |
| 6 | 13.134 | *(E)*-2-Hexenal | 14280 | 5265 | 12363 | 5145 | 7752 | 3642 | 2780 | 769 | 10038 | 4023 |
| 7 | 17.015 | Octanal | 267 | 134 | 113 | 67.6 | 245 | 70.7 | ND | ND | 168 | 157 |
| 8 | 17.057 | *(Z)*-2-Heptanal | ND | ND | ND | ND | ND | ND | ND | ND | 2574 | 3168 |
| 9 | 20.939 | *(E,E)*-2.4-Hexadienal | 101 | 39.0 | ND | ND | 157 | 39.7 | ND | ND | 340 | 218 |
| 10 | 22.256 | Nonanal | 2261 | 907 | 477 | 256 | 893 | 454 | 179 | 98.5 | 562 | 595 |
| 11 | 24.636 | *(E,E)*-2.4-Heptadienal | 159 | 99.4 | 87.4 | 56.2 | 145 | 70.1 | 31.8 | ND | 173 | 124 |
| 12 | 33.528 | *(E)*-2-Decenal | 96.9 | 83.3 | 85.4 | 71.6 | ND | ND | ND | ND | 317 | 347 |
| 13 | 41.451 | *(E,E)*-2.4-Decadienal | 29.8 | ND | ND | ND | ND | ND | ND | ND | 55.1 | 58.9 |
| | | **Total** | **20894.8** | **8194.4** | **14722.2** | **6277.5** | **14055.7** | **5440.1** | **3465.8** | **993** | **27598.9** | **17346** |
| **Alcohols** | | | | | | | | | | | | |
| 1 | 6.451 | 2-Methyl-3-buten-2-ol | 518 | 222 | 242 | 175 | ND | ND | ND | ND | 229 | 202 |
| 2 | 10.599 | 1-Penten-3-ol | 662 | 455 | 304 | 145 | 416 | 121 | 191 | 95.0 | 596 | 576 |
| 3 | 11.039 | 3-Penten-2-ol | 242 | 121 | 183 | 84.1 | 486 | 84.1 | 151 | 74.0 | 178 | 164 |
| 4 | 12.724 | Isoamyl alcohol | 226 | 167 | 127 | 64.4 | 292 | 131 | 164 | 90.0 | 190 | 143 |
| 5 | 14.659 | 1-Pentanol | 62.7 | 52.7 | 62.1 | 48.7 | ND | 22.7 | 52.4 | 30.0 | 156 | 105 |
| 6 | 16.380 | 2-Hexanol | ND | 90.3 | 114 | 86.0 | 203 | 143 | ND | 95.3 | 246 | 169 |
| 7 | 17.205 | *(E)*-2-Penten-1-ol | 148 | 84.0 | 95.1 | 60.0 | ND | ND | ND | ND | ND | ND |
| 8 | 17.573 | *(Z)*-2-Penten-1-ol | 603 | 410 | 312 | 181.0 | 125 | 86.0 | 147 | 109 | 820 | 613 |
| 9 | 19.520 | 1-Hexanol | 695 | 443 | 1156 | 472 | 946 | 527 | 1618 | 1033 | 557 | 530 |
| 10 | 20.600 | *(Z)*-3-Hexen-1-ol | 1389 | 851 | 1625 | 602 | 1528 | 826 | 816 | 545 | 1447 | 1115 |
| 11 | 21.651 | *(E)*-2-Hexen-1-ol | 736 | 559 | 2318 | 1199 | 630 | 353 | 1957 | 859 | 264 | 183 |
| 12 | 24.993 | 1-Heptanol | 64.7 | ND | 24.4 | 16.6 | ND | 22.2 | 24.0 | ND | 68.6 | 65.0 |
| 13 | 30.518 | 1-Octanol | 172 | 100 | ND | 57.8 | 105 | 55.0 | ND | ND | ND | ND |
| 14 | 34.922 | 1-Nonanol | ND | ND | ND | ND | ND | 15.3 | 8.6 | ND | ND | ND |
| 15 | 35.462 | *(Z)*-3-Nonen-1-ol | 40.7 | ND | ND | ND | ND | 24.3 | 11.8 | ND | 85.0 | 43.4 |
| 16 | 43.303 | Benzyl alcohol | 107 | 56.0 | 61.4 | 26.1 | 125 | 51.6 | 35.8 | 17.6 | 142 | 59.2 |
| 17 | 44.828 | Phenylethyl Alcohol | 136 | 66.4 | 119 | 69.5 | 141 | 89.1 | 122 | 89.8 | 190 | 121 |
| 18 | 46.591 | 3-Octanol | ND | ND | 12.5 | ND | 96.0 | 37.1 | ND | ND | ND | ND |
| 19 | 51.862 | 2-Phenoxyethanol | 19.2 | ND | 11.1 | 7.3 | ND | ND | 10.4 | ND | 25.8 | 20.4 |
| | | **Total** | **5820.9** | **3678** | **6767.1** | **3294.8** | **5094.0** | **2587.5** | **5310.5** | **3038** | **5193** | **4108** |
| **Terpenes** | | | | | | | | | | | | |
| 1 | 13.787 | dl-Limonene | 128 | 77.3 | 109 | 73.2 | 337 | 143 | 59.7 | 35.0 | 127 | 59.0 |
| 2 | 14.813 | Styrene | 86.5 | ND | 31.3 | ND | 223 | 24.0 | 23.1 | ND | ND | ND |
| 3 | 16.137 | *β*-Ocimene | 2654 | 239 | 471 | 247 | 644 | 315 | 230 | 151 | 3645 | 570 |
| 4 | 30.162 | *α*-Copaene | 180 | 112 | 894 | 744 | 140 | 138 | 351 | 286 | 3022 | 3259 |
| 5 | 34.014 | *(E)-α*-Bergamotene | 138 | 86.2 | ND | ND | 124 | 80.1 | 9.2 | ND | ND | ND |
| 6 | 34.180 | *(Z,E)-α*-Farnesene | 179 | ND | ND | ND | ND | 62.0 | 111 | ND | ND | ND |
| 7 | 35.991 | *β*-Sesquiphellandrene | 104 | 73.2 | 125 | 76.9 | 143 | 114 | 44.6 | ND | 64.4 | ND |
| 8 | 39.670 | *α*-Muurolene | ND | ND | 121 | 67.1 | ND | ND | ND | ND | 402 | 272 |
| 9 | 40.680 | *α*-Farnesene | 387 | 98.8 | 841 | 230 | 4525 | 1675 | 4200 | 1240 | 451 | 138 |
| | | **Total** | **3857.2** | **686.7** | **2593.2** | **1438.5** | **6136** | **2551.8** | **5029.4** | **1711.3** | **7711.7** | **4297.3** |
| **Acids** | | | | | | | | | | | | |
| 1 | 22.891 | Acetic acid | 58.3 | 120 | 27.6 | 57.3 | 132 | 48.3 | 9.2 | 25.1 | ND | 257 |
| 2 | 28.055 | Propanoic acid | 67.7 | 57.3 | 23.1 | 64.7 | 305 | 55.2 | 43.3 | ND | ND | ND |
| 3 | 32.240 | Butanoic acid | 136 | 48.3 | 13.5 | ND | 186 | 14.0 | 38.8 | 12.3 | 73.3 | 60.8 |
| 4 | 36.649 | Pentanoic acid | 59.4 | 36.5 | 14.8 | 26.7 | 268 | 27.8 | 31.3 | 16.3 | 37.3 | 336 |

| | | | Concentration (µg/kg)[b] | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | LRI[a] | Compounds | SU | | ME | | AY | | GE | | BE | |
| **Acids** | | | **2014** | **2015** | **2014** | **2015** | **2014** | **2015** | **2014** | **2015** | **2014** | **2015** |
| 5 | 41.315 | Hexanoic acid | 152 | 127 | 31.6 | 116 | 305 | 98.2 | 106 | ND | 178 | 2522 |
| 6 | 46.472 | *(E)*-3-Hexenoic acid | ND | ND | 4.7 | ND | ND | ND | 10.2 | ND | 106 | 86.2 |
| 7 | 46.977 | *(E)*-2-Hexenoic acid | ND | ND | ND | ND | ND | ND | 110 | 69.0 | ND | ND |
| 8 | 50.099 | Octanoic acid | 41.3 | 28.4 | 21.5 | 23.1 | ND | 33.4 | 18.1 | ND | 45.8 | 96.7 |
| 9 | 52.912 | Nonanoic acid | 80.6 | 73.8 | 23.9 | 60.0 | 266 | 74.2 | 28.4 | ND | 27.9 | 75.7 |
| 10 | 55.405 | Decanoic acid | 48.8 | ND | 12.2 | 17.6 | ND | ND | 5.9 | ND | 33.0 | 20.8 |
| 11 | 58.212 | Benzoic acid | 78.6 | 40.0 | 19.5 | ND | ND | ND | 13.4 | ND | 65.4 | 17.9 |
| 12 | 59.672 | Dodecanoic acid | ND | ND | 31.2 | 19.5 | ND | ND | 22.5 | ND | 83.4 | 19.6 |
| 13 | 62.984 | Tetradecanoic acid | ND | 121.8 | ND | 45.5 | ND | ND | ND | ND | 141 | 56.6 |
| 14 | 68.587 | Hexadecanoic acid | 309 | 734 | 145 | 143 | 1104 | 234 | 153 | 248 | 375 | 259 |
| | | **Total** | **1031.6** | **1388** | **368.9** | **574.1** | **2564** | 585.2 | 588.9 | 371.1 | 1166.4 | 3809.1 |
| **Volatile Phenols** | | | | | | | | | | | | |
| 1 | 42.389 | Guaiacol | 43.5 | ND | 8.9 | ND | 46.0 | 29.8 | 55.8 | ND | ND | ND |
| 2 | 47.903 | Phenol | 24.0 | ND | 6.7 | 6.6 | 33.3 | 12.3 | 11.6 | ND | 25.7 | 17.0 |
| 3 | 50.128 | *p*-Cresol | ND | ND | ND | 10.8 | 27.6 | ND | ND | ND | ND | 13.2 |
| 4 | 52.746 | 4-Ethyl-phenol | 21.4 | ND | 13.0 | 11.6 | ND | 22.1 | 10.6 | ND | 96.4 | 84.6 |
| 5 | 56.367 | 2.4-Di-tert-butylphenol | 40.8 | ND | 16.8 | 14.9 | 86.1 | 60.6 | 9.1 | ND | 40.6 | ND |
| | | **Total** | **129.7** | **0.0** | **45.4** | **43.8** | **192.9** | 124.8 | 87.2 | 0.0 | 162.7 | 114.8 |
| **Ketones** | | | | | | | | | | | | |
| 1 | 4.897 | 3-Methyl-3-buten-2-one | 586 | 139 | 127 | 72 | 391 | 152 | 191 | 88.0 | ND | ND |
| 2 | 4.919 | 2-Pentanone | ND | ND | ND | ND | ND | ND | 2149 | 1200 | ND | ND |
| 3 | 5.827 | 1-Penten-3-one | 620 | 401 | 659 | 407 | 701 | 220 | ND | ND | 1484 | 1090 |
| 4 | 6.373 | 2-Methyl-3-buten-2-one | ND | ND | ND | ND | 284 | 184 | ND | ND | ND | ND |
| 5 | 33.047 | Acetophenone | 30.3 | 13.0 | 14.0 | ND | 40.1 | 15.3 | 7.1 | ND | ND | ND |
| | | **Total** | **1236** | **553** | **800** | **479** | **1415** | **571** | **2347** | **1288** | **1484** | **1090** |
| **Esters** | | | | | | | | | | | | |
| 1 | 15.852 | Hexyl acetate | 122 | 62.2 | 407 | 214 | 234 | 166 | 204 | 144 | ND | ND |
| 2 | 18.078 | *(Z)*-3-Hexenyl acetate | ND | ND | 2382 | 598 | 3540 | 1295 | 3443 | 875 | ND | ND |
| 3 | 38.442 | Methyl salicylate | 118 | 68.3 | 61.4 | 32.0 | 268 | 39.3 | ND | ND | 183.0 | 137.0 |
| | | **Total** | **240.2** | **130.6** | **2850.7** | **844.3** | **4042.1** | **1500.7** | **3646.7** | **1019.1** | **183.0** | **137.0** |
| **Lactones** | | | | | | | | | | | | |
| | 34.655 | *γ*-Caprolactone | ND | ND | 13.51 | ND | ND | ND | 25.9 | 18.4 | 148 | 70.0 |
| **Hydrocarbons** | | | | | | | | | | | | |
| | 6.860 | 3-Ethyl-1.5-octadiene | 1359 | 652 | 937 | 567 | 1309 | 665 | 446 | 240 | 1659 | 958 |
| **Furans** | | | | | | | | | | | | |
| | 14.635 | 2-Pentylfuran | ND | ND | ND | ND | ND | ND | ND | ND | 57.9 | 59.4 |
| | **General Total** | | **34569.5** | **15282** | **29097.7** | **13519.0** | **34809.1** | **14026.0** | **20947.8** | **8677.8** | **45364** | **31990** |

[a] LRI: Linear retention index calculated on DB-WAX capillary column; [b]Concentration. Results are the means of three repetitions as µg/kg Identification. Standardt deviation of all aroma compounds was below 10%. AY: Ayvalık, ME: Memecik, GE: Gemlik, SU: Sarı Ulak and BE: Beylik. Experiments were conducted 3 times.

inant alcohols in the ME EVOO (2318 and 1199 µg/kg); while 1-hexenol was found to be the most abundant alcohol in the GE EVOO (1618 and 1033 µg/kg) for both years.

***Terpenes.*** Terpenes (dl-Limonene, styrene, β-ocimene, α-copaene, (E)-α-bergamotene, (Z,E)-α-farnesene, β-sesquiphellandrene, α-muurolene and α-farnesene) were found to be the third most abundant group of volatile compounds in the samples, with total amounts of 6136 µg/kg, 2593.2 µg/kg, 5029.4 µg/kg, 3857.2 µg/kg and 7711.7 µg/kg in 2014, and 2551.8 µg/kg, 1438.5 µg/kg, 1711.3 µg/kg, 686.7 µg/kg and 4297.3 µg/kg in the AY, ME, GE, SU and BE EVOO from 2015, respectively. A total of 7, 7, 8, 8 and 6 terpenes and 8, 6, 4, 6 and 5 terpenes were found in the AY, ME, GE, SU and BE EVOO from 2014 and 2015, respectively. The highest amounts of terpenes were determined in the BE EVOO from both years. In the AY (4525 and 1675 µg/kg) and GE (4200 and 1240 µg/kg) EVOO α-farnesene was determined

to be the prominent terpene from both years. α-Copaene was found as the highest terpene for the ME (894 and 744 µg/kg) and for the BE (3022 and 3259 µg/kg) EVOO from 2014 and 2015. β-ocimene was identified as the highest terpene for the SU EVOO (2654 and 239 µg/kg) from both years. These terpenes were also detected in Turkish VOOs (Kaftan and Elmaci, 2011; Kesen *et al.,* 2013; Guclu *et al.,* 2016), Greek VOOs (Issaoui *et al.,* 2015), Tunisian VOOs (Ben Brahim *et al.,* 2018) and Iranian VOO (Amanpour *et al.,* 2016). Kelebek *et al.* (2015) reported that terpenes mostly affected the varieties of VOOs. The results of our study support this assertion.

***Acids.*** Fourteen acid components were found in the studied samples. A total of 7, 11, 13, 10 and 11 acids and 8, 10, 6, 11 and 12 acids were detected in the AY, ME, GE, SU and BE EVOO from 2014 and 2015, respectively. Kesen *et al*. (2013) determined acetic acid, nonanoic acid, and decanoic acid as the major acids in AY, GE, ME VOOs, respectively. Acetic acid was found in the highest concentration in Mari VOO by Amanpour *et al.* (2016), and acetic acid was identified as a major acid in Tunisian and Sicilian VOOs by Baccouri *et al.* (2008). In our study, the most representative acid was hexadecanoic acid with 1104 and 234 µg/kg, 145 and 143 µg/kg, 153 and 248 µg/kg and 309 and 734 µg/kg, in the AY, ME, GE and SU EVOO from 2014 and 2015, respectively. Hexadecanoic acid (375 µg/kg) and hexanoic acid (2522 µg/kg) were the highest acids in the BE EVOO from 2014 and 2015, respectively.

***Volatile Phenols.*** Five volatile phenols, namely guaiacol, phenol, p-cresol, 4-ethyl-phenol and 2,4-di-tert-butylphenol, were identified in the studied EVOOs. They are generally responsible for the bitter and pungent attributes of VOOs (Amanpour *et al.,* 2016). A total of 4, 4, 4, 4 and 3 volatile phenols and 4, 4, 0, 0 and 3 volatile phenols were determined in the AY, ME, GE, SU and BE EVOO from 2014 and 2015, respectively. The highest total volatile phenols were found in the AY EVOO (192.9 and 124.8 µg/kg), followed by the BE EVOO (162.7 and 114.8 µg/kg), from 2014 and 2015, respectively. Guaiacol, phenol and 4-ethyl-phenol were detected in Turkish VOOs (Kesen *et al.,* 2013) and Iranian VOO (Amanpour *et al.,* 2016).

***Ketones.*** A total of 4, 3, 3, 3 and 1 ketones and 4, 2, 2, 3 and 1 ketones were identified in the AY (1415 and 571 µg/kg), ME (800 and 479 µg/kg), GE (2347 and 1288 µg/kg), SU (1236 and 553 µg/kg) and BE (1484 and 1090 µg/kg) EVOO from 2014 and 2015, respectively. 3-methyl-3-buten-2-one was detected in the AY, ME, GE and SU EVOO from both years. 2-pentanone was found only in the GE EVOO from both years. 1-penten-3-one was determined in the AY, ME, SU and BE EVOO from both years. 2-methyl-3-buten-2-one was identified only in the AY EVOO from both years. Acetophenone were detected in the AY and SU EVOO from both years and in the ME and GE EVOO from 2014. It was reported by Kalua *et al.* (2007) that especially the short ketones are responsible for the positive sensory attributes in VOOs.

***Esters.*** A total of 3, 3, 2, 2 and 1 esters were determined in the AY, ME, GE, SU and BE EVOOs from both years. Hexyl acetate, (Z)-3-hexenyl acetate and methyl salicylate esters were determined in the studied EVOOs. The highest total esters were found in the AY EVOO with 4042.1 and 1500.7 µg/kg, followed by the GE EVOO with 3646.7 and 1019.1 µg/kg and the ME EVOO with 2850.7 and 844.3 µg/kg, from 2014 and 2015, respectively. Esters are accountable for the pleasant fruity and flowery odor of the olive fruits (Kelebek *et al.,* 2015). These compounds were also detected in the AY, ME, GE VOOs in previous studies by Kesen *et al.* (2013), Karagoz *et al.* (2017), Guclu *et al.* (2016) and in Jemri, Touffehi and Fakhari OOs by Ben Brahim *et al.* (2018). Our results are in agreement with these studies.

***Lactones, hydrocarbons, furans.*** The other minor volatile compounds in the samples were lactones, hydrocarbons and furans. Lactone (γ-caprolactone) was identified in the ME, GE and BE EVOO; hydrocarbon (3-ethyl-1,5-octadiene) was detected in the all EVOO samples and furan (2-pentylfuran) was determined only in the BE EVOO. Kesen *et al.* (2014) reported that lactones contribute to the characteristic fruity odors of VOOs.

### 3.4. Aroma active compounds of samples

Table 3 shows the results of AACs detected using AEDA, as well as their FD values and odor descriptions. AAC odor intensities were measured as FD factors and ranged from 4 to 2048. Aromatic extracts of the samples revealed a total of 29 AACs. Aromatic extracts of AY, ME, GE, SU, and BE EVOOs contained a total of 22, 21, 18, 22 and 21 AACs, respectively.

One of the most important AACs which affects the overall composition of VOO is aldehydes. Ten odorants were defined as aroma active aldehydes

**Table 3.** Aroma-active compounds in Ayvalık, Memecik, Gemlik, Sarı Ulak and Beylik Extra Virgin Olive Oils from 2014 and 2015 harvest years

| Year | No | Compound | RT[a] | Odor description[b] | FD factor | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | AY | ME | GE | SU | BE |
| 2014 | 1 | *α*-Pinene | 7.30 | Plant | - | - | 16 | - | - |
| 2015 | | | | | - | - | 16 | - | - |
| 2014 | 2 | Hexanal | 9.47 | Green-cut grass | 1024 | 512 | 256 | 1024 | 2048 |
| 2015 | | | | | 512 | 256 | 32 | 512 | 2048 |
| 2014 | 3 | *(E)*-2-Pentenal | 11.20 | Fresh-plant | - | 8 | - | 4 | 4 |
| 2015 | | | | | - | 16 | - | 4 | 8 |
| 2014 | 4 | (Z)-3-Hexenal | 11.69 | Fresh-cut grass | - | 128 | - | - | 1024 |
| 2015 | | | | | - | 32 | - | 128 | 16 |
| 2014 | 5 | 3-Hexenal | 11.90 | Pleasant-cut grass | - | 128 | - | 256 | 1024 |
| 2015 | | | | | - | 32 | - | 4 | 16 |
| 2014 | 6 | 1-Penten-3-ol | 12.80 | Herbal-green | 128 | 128 | 32 | 256 | 128 |
| 2015 | | | | | - | 32 | 32 | 128 | 128 |
| 2014 | 7 | 3-Penten-2-ol | 13.18 | Herbal-fruity | - | 16 | 8 | 32 | - |
| 2015 | | | | | 16 | 16 | 128 | 32 | - |
| 2014 | 8 | Heptanal | 13.72 | Green-oily | - | - | - | 32 | - |
| 2015 | | | | | - | - | - | - | 16 |
| 2014 | 9 | dl-Limonene | 14.21 | Floral-citrusy | 64 | - | - | - | - |
| 2015 | | | | | 32 | - | 32 | 32 | - |
| 2014 | 10 | *(E)*-2-Hexenal | 15.25 | Cut grass-green | 1024 | 2048 | 512 | 2048 | 2048 |
| 2015 | | | | | 1024 | 512 | 128 | 512 | 512 |
| 2014 | 11 | *β*-Ocimene | 16.93 | Fruity-leafy | 32 | 64 | 32 | 256 | 512 |
| 2015 | | | | | 16 | 32 | 32 | 32 | 64 |
| 2014 | 12 | Hexyl acetate | 17.92 | Fruity-plant | 32 | 64 | 32 | - | - |
| 2015 | | | | | 16 | 32 | 64 | - | - |
| 2014 | 13 | Octanal | 18.50 | Oily-floral | - | 8 | - | 64 | - |
| 2015 | | | | | 16 | - | - | 32 | 32 |
| 2014 | 14 | Unknown | 19.45 | Oily-fruity | - | - | - | - | - |
| 2015 | | | | | 128 | - | - | - | - |
| 2014 | 15 | *(Z)*-3-Hexenyl acetate | 19.98 | Fruity-green | - | 1024 | 1024 | - | - |
| 2015 | | | | | - | 512 | 512 | - | - |
| 2014 | 16 | *(Z)*-2-Penten-1-ol | 20.33 | Green-oily | 32 | 32 | 8 | 64 | 64 |
| 2015 | | | | | - | - | 8 | 64 | - |
| 2014 | 17 | 1-Hexanol | 21.99 | Floral-herbal | 128 | 256 | 512 | 64 | 64 |
| 2015 | | | | | 64 | 64 | 512 | 128 | 64 |
| 2014 | 18 | *(Z)*-3-Hexen-1-ol | 23.62 | Herbal-cut grass | 512 | 512 | 128 | 512 | 256 |
| 2015 | | | | | 128 | 128 | 64 | 512 | 1024 |
| 2014 | 19 | Nonanal | 23.89 | Oily-citrusy | 64 | - | - | 128 | 64 |
| 2015 | | | | | 32 | - | - | 64 | 64 |
| 2014 | 20 | *(E,E)*-2.4-Hexadienal | 23.96 | Oily | 4 | - | - | - | 16 |
| 2015 | | | | | - | - | - | - | 16 |
| 2014 | 21 | *(E)*-2-Hexen-1-ol | 24.99 | Grassy-cool | 64 | 1024 | 1024 | 512 | 128 |
| 2015 | | | | | 128 | 32 | 512 | 512 | 64 |
| 2014 | 22 | *(E,E)*-2.4-Heptadienal | 27.89 | Oily | 16 | - | - | - | 16 |
| 2015 | | | | | 8 | 8 | - | - | 16 |
| 2014 | 23 | *α*-Copaene | 29.25 | Sweet-fruity | - | 256 | 128 | - | 256 |
| 2015 | | | | | - | - | 64 | - | 256 |
| 2014 | 24 | 1-Octanol | 32.70 | Fruity-green | 32 | 16 | - | 32 | - |
| 2015 | | | | | 16 | - | - | 32 | - |
| 2014 | 25 | *α*-Farnesene | 41.46 | Floral-green plant | 1024 | 512 | 1024 | 64 | 16 |
| 2015 | | | | | 128 | 128 | 256 | - | - |
| 2014 | 26 | Hexanoic acid | 45.90 | Buttery-cheesy | 64 | - | | - | 32 |
| 2015 | | | | | - | - | - | 32 | - |
| 2014 | 27 | Guaiacol | 46.20 | Olive paste | - | - | 32 | 32 | |
| 2015 | | | | | 16 | - | - | - | - |
| 2014 | 28 | Benzyl alcohol | 47.03 | Floral | 64 | 32 | 16 | 64 | 64 |
| 2015 | | | | | 32 | 16 | 16 | 32 | - |
| 2014 | 29 | Phenylethyl alcohol | 48.28 | Floral | 64 | 64 | 64 | 64 | 128 |
| 2015 | | | | | 32 | 32 | 32 | 32 | - |

[a]RT: Retention Time on DB-WAX capillary column; [b]Odor description as perceived by panelists during olfactometry. AY: Ayvalık, ME: Memecik, GE: Gemlik, SU: Sarı Ulak and BE: Beylik. Experiments were conducted 3 times.

(Table 3). The most dominant was (E)-2-hexenal, which had a cut grass-green odor and an FD factor ranging from 128 to 2048. The first year, FD factor was determined at the highest level with 2048 in ME, SU and BE EVOOs. AY EVOO followed it with an FD factor of 1024. The second year, it was determined in AY EVOO with the highest 1024 FD factor. The FD factor was determined as 512 in ME, SU and BE EVOO s. (E)-2-hexenal was followed by hexanal with a green-cut grass odor and an FD factor ranging from 128 to 2048. In 2014 and 2015, the FD factor was determined at the highest level with 2048 in BE EVOO. Guth *et al*. (1991) states that (E)-2-hexenal contributes to the aroma of VOOs with its strong odor. Solinas *et al*. (1988) also suggests that (E)-2-hexenal can be used for distinguishing a monovariatel VOO. Other aldehydes are (E)-2-pentenal, (Z)-3-hexenal, 3-hexenal, heptanal, octanal, nonanal. (E,E)-2.4-hexadienal and (E,E)-2.4-heptadienal were determined to impart fresh-plant, fresh-cut grass, pleasant-cut grass, green-oily, oily-floral, oily-citrusy, oily and oily odors, respectively. The detection threshold of aldehydes is low. It is known that aldehydes have a significant effect which can change the general properties of VOOs, even at low detection thresholds and low concentrations (Kesen *et al.,* 2013). It can be seen from previous studies that the detected aldehydes are commonly found in many VOOs (Guth *et al.,* 1991). The results are consistent with the studies performed.

Alcohols are the second most important aroma active compounds which influence the VOO's overall composition. Aldehydes have a higher sensory value than alcohols. The FD factors of the samples varied from 8 to 1024. As aroma active alcohols, 10 odorants were detected in the samples (Table 3). Among them, (E)-2-hexen-1-ol, (Z)-3-hexen-1-ol and 1-hexanol were the most dominant with grassy-cool, herbal-cut grass and floral-herbal odors and an FD factor ranging from 32 to 1024. In 2014, (E)-2-hexen-1-ol was determined at the highest level in ME and GE EVOOs with an FD factor of 1024. It was followed by SU EVOO with an FD factor of 512. In 2015, the highest FD value was determined for GE and SU EVOOs with an FD factor of 512. The (Z)-3-hexen-1-ol aroma-active compound was determined at the highest level with 512 FD factor in AY, ME and SU EVOOs from 2014. In 2015, it was determined at the highest level in BE EVOO with an FD factor of 1024. The results are in accordance with other studies (Kesen *et al*., 2014; Amanpour *et al.,* 2016).

Aldehydes and alcohols are affected according to the region where the olive is grown, especially *cis*-3-hexenal, *cis*-3-hexenol, hexanal, hexanol, *trans*-2-hexenal, *trans*-3-hexenol and *trans*-2-hexenol. (Vicchi *et al*., 2003; Žanetić *et al.*, 2021).

Five terpenes were determined in the study: α-farnesene, β-ocimene, α-copaene, dl-Limonene and α-pinene (Table 3). α-farnesene (floral, green-plant odor) was detected as the highest aroma-active terpene with an FD factor of 1024. The first year, it was found to be the highest in AY and GE EVOOs; the second year it was found at its highest in GE EVOO. β-ocimene was determined to have a fruity-leafy odor with an FD factor of ≤ 512. In 2014, all the samples were determined to have an FD factor which ranged from 32 to 512. In 2015, the FD factor decreased, and ranged from 16 to 64. α-Farnesene aroma-active compound was previously determined in Kilis Yağlık Turkish VOO by Kesen *et al*. (2014), and has also been determined as a key odorant in Moroccan green olives (Iraqi *et al.,* 2005).

Esters are associated with sweet and fruity sensory properties. Two esters, hexyl acetate with fruity-plant odor and (Z)-3-hexenyl acetate with fruity-green odor, were identified. (Z)-3-hexenyl acetate was detected only in ME and GE EVOOs. The FD factor was ≤ 1024 in 2014, and ≤ 512 in 2015. Hexyl acetate was detected in AY, ME and GE EVOOs (Table 3). Žanetić *et al.* (2021) stated that hexyl acetate caused significant differences in the differentiation of Dalmatian monovariatel EVOO.

Hexanoic acid was detected as a butter-cheesy odor in AY and BE EVOOs with an FD factor of ≤ 64 and only in 2015 (Table 3).

The most powerful AACs in the extracts were identified using the FD factor for AY EVOO hexanal, (E)-2-hexenal and α-farnesene (FD:1024) in 2014 and (E)-2-hexenal in 2015, for ME OO (E)-2-hexenal (FD:2048) in 2014 and (E)-2-hexenal (FD: 512) in 2015, for GE EVOO (Z)-3-hexenyl acetate, (E)-2-hexen-1-ol and α-farnesene (FD:1024) in 2014 and (Z)-3-hexenyl acetate, (E)-2-hexen-1-ol and 1-hexanol (FD: 512) in 2015, for SU EVOO (E)-2-hexenal (FD:2048) in 2014 and hexanal, (E)-2-hexenal, (Z)-3-hexen-1-ol and (E)-2- hexen-1-ol (FD:512) in 2015, for BE EVOO hexanal and (E)-2-hexenal (FD:2048) in 2014 and hexanal in 2015.

### 3.5. Principal component analysis

Total volatile compounds were used to construct the PCA models for the EVOOs of from different varieties of olives. The PCA model was formed with 4 components. PCA score plot and biplot are illustrated in Figure 3 based on 2 main components. The first major component explains 46.7% of the total variance, and the second major component explains 21.9% of the total variance. When the classification pattern of EVOO samples was examined, it was seen that EVOOs obtained in the first crop season were grouped and separated from the 2nd crop season except for the BE variety of EVOOs. The PCA biplot was used to establish the relationship between the varieties and total volatiles in the EVOOs.

Total ES is negatively correlated on PC1, while ALC, TER, PHE, KET, ES and HYD are positively correlated on PC2. SU1, ME1, GE1 and AY1 were characterized by T-ALC, T-ES, T-TER, T-PHE and T-KET groups of volatile compounds. T-ALD, T-TER, T-LAC and T-HYD characterized BE1 EVOOs, while T-AC and T-FU characterized BE2 EVOOs. Total volatile compounds were not found to be effective for the characterization of SU2, GE2 or AY2 EVOOs.

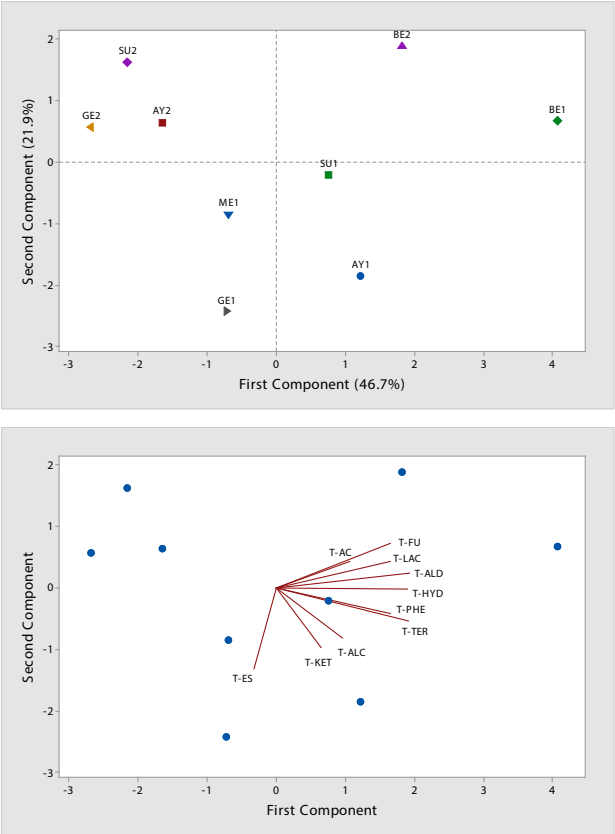


**Figure 3.** Principal component analysis score and biplot of Ayvalık, Memecik, Gemlik, Sarı Ulak and Beylik Extra Virgin Olive Oils according to total volatiles. AY: Ayvalık, ME: Memecik, GE: Gemlik, SU: Sarı Ulak and BE: Beylik

## 4. CONCLUSIONS

In the present study, the key odorants of EVOOs obtained from five different varieties grown in three different regions in Turkey were investigated. This work is the first study in which the aroma composition, key odorants and sensory properties of Turkish EVOOs were investigated in detail in terms of two different harvest seasons with a three-phase centrifuge system. All samples were classified as EVOO based on the results of the quality parameters. According to the ANOVA results, the difference between the averages of the quality parameter results was not significant at the 95% confidence level. A total of 52, 57, 51, 57 and 54 volatile compounds were identified and characterized in the studied EVOOs. Alcohols and aldehydes were determined to be the most dominant volatile compounds both qualitatively and quantitatively in the samples. According to the AEDA results, based on the FD factor, the strongest aroma-active compounds detected in the extracts were hexanal, with the cut green-grass odor, *(E)*-2-hexenal, with cut green-grass notes, and *(E)*-2-hexen-1-ol, which was associated with the odor of grassy-cool. Although the abundant compounds were similar and included mostly aldehydes, their FD factors varied for each cultivar and displayed differences in the aroma of the investigated Turkish EVOOs. The results show that AY has the highest FD value of 1024 with hexanal, *(E)*-2-hexenal and *α*-farnesene. ME has the highest FD value for 2048 with *(E)*-2-hexenal. GE has the highest FD value of 1024 with *(Z)*-3-hexenyl acetate, *(E)*-2-hexen-1-ol and *α*-farnesene. SU has the highest FD value of 2048 with *(E)*-2-hexenal. BE has the highest FD value of 2048 with *(E)*-2-hexenal and hexanal. The sensory and principal component analyses displayed clear discrimination of samples and according to the spider graphs, none of the samples had off-flavor attributes. The sensory aspects of EVOOs studied in the current work differed slightly according to harvest year, especially in bitterness, leaves and pungent parameters.

## ACKNOWLEDGMENTS

The Republic of Turkey Ministry of Agriculture and Forestry General Directorate of Agricultural Research and Policies (TAGEM) supported this project, and all authors who contributed to the project and publication are gratefully acknowledged (Number: TAGEM/HSGYAD/15A05/P01/75).

## ABBREVIATIONS

Flavor Dilution (FD), Virgin Olive Oil (VOO), Extra Virgin Olive Oil (EVOO), Gas Chromatography-Olfactometry (GC-O), Aroma-Active Compound (AAC), Aroma Extract Dilution Analysis (AEDA), Ayvalık (AY), Memecik (ME), Gemlik (GE), Sarı Ulak (SU), Beylik (BE), QP (Quality Parameter), Free Fatty Acidity (FFA), Peroxide Value (PV), FID (Flame Ionization Detector).

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