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# Changes in Volatile Compounds of Virgin Olive Oil Flavored with Essential Oils During Thermal and Photo-Oxidation

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## Abstract

The effects of four essential oils from peppermint (*Micromeria fruticosa*), oregano (*Origanum onites*), thyme (*Thymus vulgaris*), and laurel (*Laurus nobilis*) on the volatile compounds of olive oil were determined. The concentration of essential oils was 0.05% (v/w) and flavored olive oil samples were stored for 45 days at 60 °C and room temperature for thermal oxidation and photo-oxidation under fluorescent light, respectively. Control and flavored olive oils were analyzed after 15, 30, and 45th days to track the changes in their volatile compound contents using HS-SPME/GC-MS technique. Higher concentrations of diversified volatile components were detected under thermal oxidation conditions rather than photo-oxidation. According to thermal oxidation results, the E-2-hexenal values of control and flavored oils with peppermint essential oil were higher at the end of 30 days storage, while flavoring with essential oil of *Thymus vulgaris* resulted in the highest E-2-hexenal value for photo-oxidation. Results indicated that the main components of essential oil transferred into olive oil samples. Carvacrol was present in flavored oils with oregano and thyme. Eucalyptol and pulegone were determined as major components in flavored oils with laurel and peppermint essential oils, respectively. In both oxidation methods, these volatile components remained stable and little or no loss was observed.

**Keywords** Flavored olive oil · Essential oil · Volatile compounds · Thermal oxidation · Photo-oxidation · GC-MS

## Introduction

Extra virgin olive oil (EVOO) plays an important role in the Mediterranean diet. EVOO contains high amounts of fatty acids in addition to such minor components as phenolics, sterols, tocopherols, squalene, and volatile compounds. The positive impacts of minor compounds present in EVOO to human health have been evidenced by several clinical works (Francisco et al. 2019; Romani et al. 2019). Besides health benefits, EVOO has a unique aroma that originates from the mixture of volatile and non-volatile components. The quality of EVOO is mainly affected by its overall composition having such groups as fatty acids, sterols, tocopherols, and other minor compounds. Besides, volatile components also play an important role in the quality of EVOO in terms of the preferences of consumers (Genovese et al. 2019). The characteristic sensory attributes for EVOO are fruity and green, and these attributes are strongly related to C5 and C6 compounds which are formed by the enzymatic oxidation of olive oils, especially through the lipoxygenase pathway during olive oil processing (Angerosa et al. 2001; Kalua et al. 2007). The presence or absence of particular volatile compounds contributes to the overall EVOO aroma.

Consumers prefer functional foods that are beneficial to them. Consumption of these foods ensures some health benefits for some diseases (Sloan 2000). In recent years, new functional products based on olive oil flavored with herbs or spices have been placed on market shelves. Today, different types of flavored oils are available on the market. Vegetables, herbs, spices, mushrooms, fruits, nuts, and some aroma compounds have been used in flavored olive oil production (Sousa et al. 2015). Besides, most of these aromatizers are reported to increase the oxidative stability of oils (Gambacorta et al. 2007).

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The traditional infusion method is one of the several methods used in the production of flavored olive oil. The main disadvantage of this method is the long infusion times (up to months) at room conditions to successfully produce flavored olive oil. Besides, undesirable substances such as waxes can be also transferred from the aromatic plant to olive oil which may change the sensorial and stability attributes of olive oils during shelf life (Baiano et al. 2010). On the other hand, aromatic plants are mostly seasonal and not available throughout the year (Akçar and Gümüşkesen 2011). To avoid these problems, olive oil can be flavored by the use of the essential oils (Moldão-Martins et al. 2004) as an alternative approach. Although there are many studies using spices and herbs in aromatized oil production, the number of studies regarding the use of essential oils in flavoring olive oils is limited. Ayadi et al. (2009) studied physicochemical changes in flavored oils with rosemary, lavender, sage, menthe, basil, lemon, and thyme at thermal conditions (60 and 130 °C). Damechki et al. (2001) investigated the changes in the physicochemical characteristics and oxidative stability of olive oils flavored with oregano and rosemary. Moldão-Martins et al. (2004) studied response surface methodology (RSM) to determine the suitable ratio of *Mentha piperita* L. and *Thymus mastichina* subsp. *mastichina* essential oils in olive oil depending on sensory analysis. A study by Arcoleo et al. (2009) evaluated the effects of cold-pressed lemon essential oil on sensory properties of olive oil as well as the shelf life. Asensio et al. (2013) studied the effects of oregano essential oils on the oxidative stability of olive oils stored in dark and light conditions. In another study by Taoudiat et al. (2018), the effects of *Laurus nobilis* essential oil on the oxidative stability of olive oils packaged with brown and transparent glass or PET were investigated during photo-oxidation conditions.

This study aimed to determine the effects of such essential oils as peppermint (*Micromeria fruticosa*), oregano (*Origanum onites*), thyme (*Thymus vulgaris*), and laurel (*Laurus nobilis*) on the volatile components of olive oil during thermal and photo-oxidation.

## Materials and Methods

### Essential Oils and Preparation of Flavored Olive Oils

The four essential oils, peppermint (*Micromeria fruticosa*), oregano (*Origanum onites*), thyme (*Thymus vulgaris*), and laurel (*Laurus nobilis*) were purchased from the local markets in Turkey. The essential oils were kept at − 18 ± 0.5 °C in glass vials in a dark place. Flavored oils were prepared by adding essential oils (0.025 mL) to 50 g of olive oil samples. These oils were then transferred to transparent and dark glass bottles (100 cc) for thermal storage conditions and light-induced storage, respectively. Flavored oil samples are coded as follows: MF: flavored oil with peppermint essential oil, OO: flavored oil with oregano essential oil, TV: flavored oil with thyme essential oil, and LN: flavored oil with laurel essential oil.

### Gas Chromatography/Mass Spectrometry Analysis of Essential Oils

Essential oil compounds were characterized by a Shimadzu GC-2010 Plus model (Shimadzu Corporation, Kyoto, Japan) gas chromatograph equipped with a Shimadzu QP2010 Plus model mass spectrometer using electron impact. An electron ionization system with ionization energy of 70 eV was used within a scan range of 40-300 amu for GC-MS analysis. Separation of the compounds was achieved by using a Rxi 5Sil-MS-GC column (30 m × 0.25 mm ID × 0.25 μm) (Restek Corporation, Bellefonte, USA). Helium was used as the carrier gas with a flow rate of 1.61 mL/min. The temperatures of the ion source and the MS transfer line were 200 and 250 °C, respectively. The oven temperature program was initially set at 40 °C for 2 min, then raised to 250 °C at a rate of 4 °C/min, and finally held isothermal for 5 min. Then, 0.20 μL of each sample was injected into the equipment using split mode with a split ratio of 10:1. The compound identification was performed by the comparison of mass spectra and linear retention indexes (LRI) of the compounds with authentic standards and published data. Also, their mass spectra were also compared by such MS libraries as Wiley, NIST and FFNSC.

### Analysis of Volatile Compounds in EVOO Samples

The volatile fractions of EVOO samples were analyzed by headspace solid-phase microextraction/gas chromatography–mass spectrometer (HS–SPME/GC–MS). Two grams of sample was weighed in 20 mL screw cap vials and placed in a vial heater at 45 °C for 15 min of equilibration time. Volatile compounds were adsorbed onto an SPME fiber Carboxen/polydimethylsiloxane (CAR/PDMS; 75 μm) (Supelco, Bellefonte, USA). The sampling time was 45 min at 45 °C. The desorption of volatile compounds was completed by injecting to the GC operated under the same conditions as described above. GC/MS analyses of the volatiles were carried out by a Shimadzu GC-2010 Plus (Shimadzu Corporation, Kyoto, Japan) model gas chromatograph with a Rxi 5Sil-MS-GC column (30 m, 0.25 mm ID, 0.25 μm) (Restek, Bellefonte, USA) equipped with a Shimadzu QP2010 Plus (Shimadzu Corporation, Kyoto, Japan) mass spectrometer using electron impact (ionization energy: 70 eV). Retention indices of each volatile compound were calculated using a homologous series of C7-C30 *n*-alkanes injected under identical conditions with the samples. The components were identified regarding their LRI relative to

n-alkanes and matching data in such MS libraries as Wiley, NIST, FFNSC. Also, the fragmentation pattern of the mass spectra were compared with data published in the literature. The relative amounts of detected volatile compounds were expressed in arbitrary units (AU).

### Oxidation Methods

#### Thermal Oxidation

The flavored olive oil and control samples were heated to 60 °C in the oven. The volatile compounds which were released from oil samples were analyzed at the end of every 15 days up to the end of the 45th day of storage under accelerated thermal oxidation conditions.

#### Photo-Oxidation

The oil samples were placed in a lightbox (Test T742 LBD, Turkey) equipped with cool white fluorescent light sources. The fluorescent radiation intensity was about 3000 lx. Volatile compounds in the oil samples were analyzed at the end of every 15 days and the whole storage period for photo-oxidation was 45 days.

### Statistical Analysis

All samples were analyzed in duplicates. Data were expressed in terms of mean ± standard deviation. Significant differences were determined using ANOVA in combination with a Duncan test with a significance level of 95% by using Minitab (Version 16, Minitab Inc., USA) statistical software.

## Results and Discussion

### Essential Oil Composition

The compositions of essential oils from peppermint (*Micromeria fruticosa*), oregano (*Origanum onites*), thyme (*Thymus vulgaris*), and laurel (*Laurus nobilis*) analyzed by GC-MS were exhibited in the supplementary material. The major compound detected in oregano and thyme oil was carvacrol (69.95 and 57.28% for oregano and thyme oils, respectively). In a previous study (Iten et al. 2009), carvacrol was also reported as the major volatile constituent of essential oil obtained from *Thymus vulgaris.* It was determined that laurel essential oil contained two major compounds namely camphor (43.03%) and eucalyptol (1,8-cineol) (35.06%), followed by sabinene (4.21%), $\alpha$-terpinyl acetate (4.20%), $\alpha$-pinene (2.92%), and $\beta$-pinene (2.36%). Although the high eucalyptol content of *Laurus nobilis* essential oil agreed with data from the literature (Simić et al. 2004), some studies are reporting the low camphor content in *Laurus nobilis* (Jemâa et al. 2012; Shokoohinia et al. 2014) in contrast to our study. Results indicated that the most abundant compounds of peppermint (*Micromeria fruticosa*) oil were pulegone (65.30%) and *p*-menthone (14.49%). Pulegone was also reported as the dominant compound of *M. fruticosa* in previous studies (Güllüce et al. 2004; Arslan 2012).

### Thermal Oxidation

The identification data of volatile compounds in olive oil samples during thermal treatment at 60 °C are shown in Table 1. During thermal oxidation of olive oil samples, 24 components were identified in the control sample, while flavored olive oils contained numerous volatile compounds. The number of identified compounds was 38 in LN and TV samples, followed by OO with 37 compounds and MF with a minimum number of identified components of 27.

Changes in the volatile compounds of olive oil samples are presented in Table 2. During the thermal oxidation process, the initial volatiles such as hexanal, E-2-hexenal, Z-2-penten-1-ol, Z-3-hexen-1-ol, E-2-hexen-1-ol, octen-3-ol, and Z-3-hexenyl acetate, which contribute to the aroma of olive oil, disappeared first and then appeared as volatile oxidation products as their amounts increased with the ongoing oxidation. In analyzed oils, E-2-hexenal and hexanal were the main volatile compounds determined during oxidation of olive oils as compared with the previously reported data for vegetable oils treated with heat (Kiralan and Ramadan 2016; Wang et al. 2019). The highest E-2-hexenal values in C and MF samples after 30 days storage were $140.45 \times 10^6$ AU and $187.10 \times 10^6$ AU, respectively, while E-2-hexenal content of LN reached a maximum value of $33.81 \times 10^6$ AU at the end of 15 days storage. After reaching the maximum level, E-2-hexenal content started to decrease in the samples mentioned above. However, there was a slight increase in OO and TV samples during thermal oxidation conditions. Although not as much as E-2-hexenal, hexanal also showed an increasing trend during storage. Among volatile aldehydes, the presence of other saturated compounds, such as pentanal, heptanal, octanal, and nonanal, were also detected. Pentanal appeared only in C, OO, and MF samples after 15 days of storage. There was an increase in heptanal and nonanal contents of all analyzed samples, while a similar trend was observed for octanal in samples except the TV sample. 3-Methyl-2-butenal, which is a branched aldehyde, was present in all samples through the oxidation process.

Alcohols such as pentanol, hexanol, and octanol were also detected in the analyzed samples. Hexanol, an aroma compound of olive oil, was detected in fresh samples and subsequently disappeared with ongoing oxidation. Pentanol and octanol were absent only in MF and LN samples, respectively.

**Table 1** Identified volatile compounds of olive oil samples at thermal and photo-oxidation experiments

| $KI^{a}_{cal}$ | $KI^{b}_{lit}$ | Method of identification[c] | Library match similarity (%) | Compound[b] | Thermal oxidation | | | | | Photo-oxidation | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | C | OO | MF | LN | TV | C | OO | MF | LN | TV |
| 624 | 656[1] | MS, KI | 96 | 2-Butenal | + | + | + | + | + | + | + | + | + | + |
| 696 | 696[1] | MS, KI | 95 | Pentanal | + | + | + | – | – | + | + | + | + | + |
| 751 | 751[2] | MS, KI | 96 | E-2-Pentenal | + | + | + | + | + | + | + | + | – | + |
| 753 | 761[3] | MS, KI | 96 | Pentanol | + | + | – | + | + | – | – | – | + | – |
| 767 | 767[2] | MS, KI | 93 | Z-2-penten-1-ol | + | – | – | – | – | + | – | – | – | – |
| 788 | 788[4] | MS, KI | 94 | 3-methyl-2-butenal | + | + | + | + | + | + | + | + | + | – |
| 801 | 802[1] | MS, KI | 96 | Hexanal | + | + | + | + | + | + | + | + | + | + |
| 814 | 806[5] | MS, KI | 98 | E-2-octene | – | – | + | + | + | – | – | + | + | – |
| 850 | 850[2] | MS, KI | 96 | E-2-hexenal | + | + | + | + | + | + | + | + | + | + |
| 853 | 853[6] | MS, KI | 95 | Z-3-Hexen-1-ol | + | + | + | + | + | + | + | + | + | + |
| 861 | 861[2] | MS, KI | 91 | E-2-Hexen-1-ol | + | + | + | + | + | + | + | + | + | + |
| 867 | 867[6] | MS, KI | 95 | Hexanol | + | + | + | + | + | + | + | + | + | + |
| 898 | 888[3] | MS, KI | 84 | 2-Heptanone | + | + | – | + | + | – | – | – | – | – |
| 900 | 900[2] | MS, KI | 97 | Nonane | – | – | – | + | + | – | – | – | + | + |
| 906 | 906[2] | MS, KI | 94 | Heptanal | + | – | – | – | – | + | + | + | + | + |
| 927 | 928[4] | MS, KI | 91 | α-Thujene | – | + | – | + | + | – | + | – | + | – |
| 933 | 933[2] | MS, KI | 97 | α-Pinene | – | – | – | + | + | – | – | – | + | + |
| 956 | 956[2] | MS, KI | 96 | E-2-Heptenal | + | + | + | + | + | + | + | + | + | + |
| 972 | 972[7] | MS, KI | 97 | Sabinene | – | – | – | + | – | – | – | – | + | – |
| 974 | 974[7] | MS, KI | 96 | β-Pinene | – | – | – | + | – | – | – | – | + | – |
| 978 | 978[3] | MS, KI | 97 | Octen-3-ol | + | + | + | + | + | + | – | – | + | + |
| 986 | 989[1] | MS, KI | 95 | 6-Methyl-5-hepten-2-one | – | + | – | – | + | + | + | + | + | + |
| 991 | 991[7] | MS, KI | 92 | β-Myrcene | – | + | – | + | + | – | + | + | + | + |
| 991 | 991[1] | MS, KI | 97 | 2-pentyl- Furan | – | + | – | + | + | – | – | – | – | – |
| 1006 | 1004[1] | MS, KI | 95 | Octanal | + | + | + | + | – | + | – | + | + | – |
| 1008 | 1008[2] | MS, KI | 93 | Z-3-Hexenyl acetate | + | + | + | + | + | + | + | + | + | + |
| 1013 | 1014[1] | MS, KI | 95 | E,E-2,4-Heptadienal | – | + | – | + | – | – | – | – | – | – |
| 1018 | 1015[7] | MS, KI | 96 | α- Terpinene | – | + | – | + | + | – | – | – | – | – |
| 1025 | 1023[1] | MS, KI | 97 | *p*- Cymene | – | + | + | + | + | – | + | + | + | + |
| 1030 | 1027[1] | MS, KI | 94 | Limonene | – | + | + | + | + | – | + | + | + | + |
| 1032 | 1030[7] | MS, KI | 96 | Eucalyptol | – | – | – | + | – | – | – | – | + | – |
| 1038 | 1036[8] | MS, KI | 92 | 3-Octen-2-one | + | + | + | + | + | + | – | + | + | – |
| 1058 | 1061[4] | MS, KI | 94 | γ- Terpinene | – | + | – | + | + | – | + | – | + | + |
| 1059 | 1057[8] | MS, KI | 96 | E-2-Octenal | + | + | + | + | + | + | – | + | + | – |
| 1072 | 1072[1] | MS, KI | 89 | 3,5-Octadien-2-one | – | + | – | – | + | – | – | – | – | – |
| 1076 | 1072[7] | MS, KI | 96 | Octanol | + | + | + | – | + | – | – | – | – | – |
| 1093 | 1090[9] | MS, KI | 91 | 2-Nonanone | + | + | – | – | + | – | – | – | – | – |
| 1101 | 1098[1] | MS, KI | 94 | Linalool | – | + | – | + | + | – | + | – | + | + |
| 1107 | 1107[2] | MS, KI | 95 | Nonanal | + | + | + | + | + | + | + | + | + | + |
| 1158 | 1164[10] | MS, KI | 97 | p-Menthone | – | – | + | – | – | – | – | + | – | – |
| 1163 | 1163[9] | MS, KI | 94 | E-2-Nonenal | + | + | + | + | + | – | – | – | – | – |
| 1174 | 1177[11] | MS, KI | 93 | Isopulegone | – | – | + | – | – | – | – | + | – | – |
| 1180 | 1184[11] | MS, KI | 96 | Terpinen-4-ol | – | + | – | + | + | – | + | – | + | + |
| 1198 | 1192[4] | MS, KI | 96 | α-Terpineol | – | – | – | + | + | – | + | – | + | + |
| 1218 | 1218[8] | MS, KI | 96 | E,E-2,4-nonadienal | – | + | – | – | – | – | – | – | – | – |
| 1241 | 1238[12] | MS, KI | 97 | Pulegone | – | – | + | – | – | – | – | + | – | – |
| 1265 | 1262[11] | MS, KI | 93 | E-2-decenal | + | + | + | + | + | + | + | – | + | + |
| 1267 | 1255[12] | MS, KI | 91 | Piperitone | – | – | + | – | – | – | – | + | – | – |
| 1288 | 1290[13] | MS, KI | 92 | Thymol | – | – | – | – | + | – | – | – | – | + |
| 1299 | 1298[13] | MS, KI | 97 | Carvacrol | – | + | – | – | + | – | + | – | – | + |
| 1322 | 1322[7] | MS, KI | 95 | E,E-2,4-Decadienal | – | + | + | + | + | – | – | – | – | – |
| 1347 | 1341[11] | MS, KI | 96 | Piperitenone | – | – | – | – | – | – | – | + | – | – |
| 1349 | 1345[14] | MS, KI | 97 | α-Terpinyl acetate | – | – | – | + | – | – | – | – | + | – |
| 1376 | 1364[15] | MS, KI | 93 | Piperitenone oxide | – | – | – | – | – | – | – | + | – | – |

[a] $KI_{cal}$ Kovats index calculated for Rxi 5Sil capillary column (30 m × 0.25 mm ID× 0.25 μm) installed on a GC equipped with a mass-selective detector

[b] $KI_{lit}$ Kovats index reported in literature

[c] Methods of identification: MS mass spectrum comparison using Wiley, NIST, and FFNSC libraries, KI: Kovats index in agreement with literature value ([1] Araújo et al. 2007; [2] Cevik et al. 2016; [3] Wu et al. 2014a; [4] Petrović et al. 2017; [5] Mallia et al. 2008; [6] Karagoz et al. 2017; [7] Koutidou et al. 2017; [8] Liu et al. 2017; [9] Wu et al. 2014b; [10] Bishr and Salama 2018; [11] Stojanović et al. 2019; [12] Padalia et al. 2011; [13] Santos et al. 2015; [14] Lei et al. 2010; [15] Singh et al. 2020)

**Table 2** Changes in volatile compounds of olive oil samples during thermal oxidation

| Compound | Storage (days) | C | OO | MF | LN | TV |
|---|---|---|---|---|---|---|
| 2-Butenal | 0 | 0.14 ± 0.04b | 0.00 ± 0.00d | 0.00 ± 0.00c | 0.00 ± 0.00 | 0.00 ± 0.00b |
| | 15 | 0.70 ± 0.07ab | 0.58 ± 0.02c | 0.62 ± 0.03b | 1.66 ± 1.34 | 0.08 ± 0.04b |
| | 30 | 0.71 ± 0.41ab | 1.71 ± 0.22b | 0.46 ± 0.01b | 1.74 ± 0.33 | 0.36 ± 0.00a |
| | 45 | 1.06 ± 0.16a | 2.04 ± 0.08a | 1.44 ± 0.18a | 1.70 ± 0.00 | 0.43 ± 0.05a |
| Pentanal | 0 | 0.00 ± 0.00 | 0.00 ± 0.00 | 0.00 ± 0.00c | – | – |
| | 15 | 2.25 ± 0.19 | 1.40 ± 0.01 | 1.58 ± 0.02a | – | – |
| | 30 | 0.00 ± 0.00 | 0.00 ± 0.00 | 0.85 ± 0.04b | – | – |
| | 45 | 0.00 ± 0.00 | 0.00 ± 0.00 | 0.00 ± 0.00c | – | – |
| E-2-Pentenal | 0 | 0.10 ± 0.01c | 0.00 ± 0.00d | 0.00 ± 0.00c | 0.00 ± 0.00b | 0.00 ± 0.00c |
| | 15 | 0.84 ± 0.04b | 0.82 ± 0.01c | 0.63 ± 0.21b | 0.73 ± 0.23a | 0.12 ± 0.01b |
| | 30 | 0.20 ± 0.08c | 1.10 ± 0.0b | 0.16 ± 0.01c | 1.07 ± 0.08a | 0.33 ± 0.03a |
| | 45 | 0.99 ± 0.04a | 1.48 ± 0.06a | 1.45 ± 0.01a | 0.95 ± 0.02a | 0.36 ± 0.01a |
| Pentanol | 0 | 0.00 ± 0.00b | 0.00 ± 0.00b | – | 0.25 ± 0.03 | 0.00 ± 0.00c |
| | 15 | 0.63 ± 0.15b | 0.29 ± 0.03ab | – | 0.50 ± 0.32 | 0.00 ± 0.00c |
| | 30 | 0.39 ± 0.39b | 0.77 ± 0.04a | – | 0.86 ± 0.30 | 0.13 ± 0.00b |
| | 45 | 1.42 ± 0.30a | 0.97 ± 0.51a | – | 0.69 ± 0.09 | 0.42 ± 0.01a |
| Z-2-penten-1-ol | 0 | 0.06 ± 0.00 | – | – | – | – |
| | 15 | 0.00 ± 0.00 | – | – | – | – |
| | 30 | 0.00 ± 0.00 | – | – | – | – |
| | 45 | 0.00 ± 0.00 | – | – | – | – |
| 3-Methyl-2-butenal | 0 | 0.00 ± 0.00b | 0.00 ± 0.00c | 0.00 ± 0.00c | 0.00 ± 0.00b | 0.00 ± 0.00b |
| | 15 | 0.37 ± 0.14a | 0.39 ± 0.03b | 0.16 ± 0.08b | 0.48 ± 0.28a | 0.00 ± 0.00b |
| | 30 | 0.14 ± 0.08b | 0.79 ± 0.04a | 0.12 ± 0.01bc | 0.52 ± 0.01a | 0.18 ± 0.04a |
| | 45 | 0.55 ± 0.01a | 0.76 ± 0.06a | 0.84 ± 0.05a | 0.55 ± 0.01a | 0.15 ± 0.01a |
| Hexanal | 0 | 4.34 ± 0.08b | 2.38 ± 0.12d | 3.45 ± 0.05b | 2.15 ± 0.09c | 5.62 ± 0.47a |
| | 15 | 17.15 ± 2.35a | 12.27 ± 0.06a | 13.96 ± 1.44a | 6.26 ± 0.43b | 1.38 ± 0.00c |
| | 30 | 4.06 ± 1.71b | 11.29 ± 0.20b | 5.07 ± 0.29b | 8.02 ± 0.51a | 2.27 ± 0.04b |
| | 45 | 11.24 ± 0.21a | 8.91 ± 0.00c | 14.84 ± 0.87a | 6.55 ± 0.23b | 2.65 ± 0.09b |
| E-2-octene | 0 | – | – | 2.51 ± 0.07 | 1.32 ± 0.18 | 3.87 ± 0.54 |
| | 15 | – | – | 0.00 ± 0.00 | 0.00 ± 0.00 | 0.00 ± 0.00 |
| | 30 | – | – | 0.00 ± 0.00 | 0.00 ± 0.00 | 0.00 ± 0.00 |
| | 45 | – | – | 0.00 ± 0.00 | 0.00 ± 0.00 | 0.00 ± 0.00 |
| E-2-hexenal | 0 | 4.38 ± 0.03b | 1.15 ± 0.04d | 1.58 ± 0.06b | 1.46 ± 0.12 | 1.04 ± 0.13a |
| | 15 | 3.67 ± 0.15b | 2.88 ± 0.08a | 5.12 ± 0.67b | 33.81 ± 0.33 | 1.11 ± 0.14a |
| | 30 | 140.45 ± 10.77a | 2.37 ± 0.08b | 187.10 ± 5.87a | 1.62 ± 0.45 | 0.70 ± 0.03b |
| | 45 | 1.04 ± 0.10b | 1.62 ± 0.17c | 2.26 ± 0.31b | 1.33 ± 0.33 | 0.31 ± 0.01c |
| Z-3-Hexen-1-ol | 0 | 1.86 ± 0.07a | 0.62 ± 0.00a | 0.85 ± 0.06b | 0.72 ± 0.07 | 0.83 ± 0.13a |
| | 15 | 0.91 ± 0.04b | 0.68 ± 0.06a | 1.21 ± 0.03a | 0.00 ± 0.00 | 0.31 ± 0.01b |
| | 30 | 0.00 ± 0.00 | 0.16 ± 0.01b | 0.00 ± 0.00c | 0.00 ± 0.00 | 0.00 ± 0.00c |
| | 45 | 0.00 ± 0.00 | 0.00 ± 0.00c | 0.00 ± 0.00c | 0.00 ± 0.00 | 0.00 ± 0.00c |
| E-2-hexen-1-ol | 0 | 0.29 ± 0.01 | 0.20 ± 0.01a | 0.19 ± 0.00a | 0.19 ± 0.01 | 0.28 ± 0.07 |
| | 15 | 0.00 ± 0.00 | 0.04 ± 0.01b | 0.11 ± 0.00b | 0.00 ± 0.00 | 0.04 ± 0.00 |
| | 30 | 0.00 ± 0.00 | 0.00 ± 0.00c | 0.00 ± 0.00c | 0.00 ± 0.00 | 0.00 ± 0.00 |
| | 45 | 0.00 ± 0.00 | 0.00 ± 0.00c | 0.00 ± 0.00c | 0.00 ± 0.00 | 0.00 ± 0.00 |
| Hexanol | 0 | 0.32 ± 0.02a | 0.17 ± 0.01a | 0.18 ± 0.04a | 0.17 ± 0.00 | 0.39 ± 0.11 |
| | 15 | 0.16 ± 0.03b | 0.06 ± 0.01b | 0.14 ± 0.01a | 0.00 ± 0.00 | 0.04 ± 0.00 |
| | 30 | 0.00 ± 0.00 | 0.00 ± 0.00c | 0.00 ± 0.00b | 0.00 ± 0.00 | 0.00 ± 0.00 |
| | 45 | 0.00 ± 0.00 | 0.00 ± 0.00c | 0.00 ± 0.00b | 0.00 ± 0.00 | 0.00 ± 0.00 |
| 2-Heptanone | 0 | 0.00 ± 0.00b | 0.00 ± 0.00c | – | 0.00 ± 0.00c | 0.00 ± 0.00 |
| | 15 | 0.00 ± 0.00b | 0.00 ± 0.00c | – | 0.13 ± 0.10bc | 0.00 ± 0.00 |
| | 30 | 0.23 ± 0.23b | 0.36 ± 0.04b | – | 0.40 ± 0.18ab | 0.00 ± 0.00 |
| | 45 | 1.38 ± 0.77a | 0.56 ± 0.01a | – | 0.54 ± 0.04a | 0.27 ± 0.10 |
| Nonane | 0 | – | – | – | 1.35 ± 0.08 | 2.88 ± 0.57 |
| | 15 | – | – | – | 0.00 ± 0.00 | 0.00 ± 0.00 |
| | 30 | – | – | – | 0.00 ± 0.00 | 0.00 ± 0.00 |
| | 45 | – | – | – | 0.00 ± 0.00 | 0.00 ± 0.00 |
| Heptanal | 0 | 0.03 ± 0.01b | 0.00 ± 0.00c | 0.00 ± 0.00c | 0.00 ± 0.00b | 0.00 ± 0.00d |
| | 15 | 0.45 ± 0.17ab | 0.36 ± 0.00b | 0.42 ± 0.04b | 0.30 ± 0.12ab | 0.05 ± 0.00c |
| | 30 | 0.49 ± 0.33ab | 0.76 ± 0.04a | 0.22 ± 0.01bc | 0.62 ± 0.28a | 0.08 ± 0.01b |
| | 45 | 1.06 ± 0.46a | 0.79 ± 0.09a | 1.40 ± 0.20a | 0.43 ± 0.01ab | 0.18 ± 0.01a |
| α-Thujene | 0 | – | 0.09 ± 0.00b | – | 0.07 ± 0.01a | 0.37 ± 0.04 |
| | 15 | – | 0.13 ± 0.01a | – | 0.06 ± 0.00a | 0.00 ± 0.00 |
| | 30 | – | 0.00 ± 0.00c | – | 0.00 ± 0.00b | 0.00 ± 0.00 |
| | 45 | – | 0.00 ± 0.00c | – | 0.00 ± 0.00b | 0.00 ± 0.00 |
| α-Pinene | 0 | – | – | – | 0.14 ± 0.04c | 0.08 ± 0.01 |

**Table 2** (continued)

| Compound | Storage (days) | C | OO | MF | LN | TV |
|---|---|---|---|---|---|---|
| | 15 | – | – | – | 0.34 ± 0.01a | 0.08 ± 0.01 |
| | 30 | – | – | – | 0.26 ± 0.06ab | 0.03 ± 0.04 |
| | 45 | – | – | – | 0.21 ± 0.02bc | 0.02 ± 0.00 |
| E-2-Heptenal | 0 | 0.05 ± 0.01c | 0.00 ± 0.00d | 0.00 ± 0.00c | 0.00 ± 0.00b | 0.00 ± 0.00c |
| | 15 | 1.62 ± 0.38bc | 2.39 ± 0.11c | 0.62 ± 0.11c | 4.09 ± 2.46a | 0.23 ± 0.04c |
| | 30 | 2.14 ± 1.29b | 7.28 ± 0.34b | 1.66 ± 0.16b | 6.10 ± 0.21a | 1.60 ± 0.27b |
| | 45 | 7.28 ± 0.16a | 8.39 ± 0.23a | 9.76 ± 0.54a | 6.42 ± 0.35a | 2.10 ± 0.03a |
| Sabinene | 0 | – | – | – | 0.42 ± 0.11ab | – |
| | 15 | – | – | – | 0.75 ± 0.14a | – |
| | 30 | – | – | – | 0.42 ± 0.25ab | – |
| | 45 | – | – | – | 0.30 ± 0.04b | – |
| β-Pinene | 0 | – | – | – | 0.11 ± 0.03b | – |
| | 15 | – | – | – | 0.33 ± 0.06ab | – |
| | 30 | – | – | – | 0.38 ± 0.10a | – |
| | 45 | – | – | – | 0.35 ± 0.12ab | – |
| Octen-3-ol | 0 | 0.05 ± 0.01c | 0.06 ± 0.02d | 0.23 ± 0.01c | 0.00 ± 0.00b | 0.19 ± 0.01 |
| | 15 | 0.00 ± 0.00bc | 0.20 ± 0.03c | 0.96 ± 0.07a | 0.00 ± 0.00b | 0.11 ± 0.01 |
| | 30 | 0.29 ± 0.20b | 0.37 ± 0.02a | 0.37 ± 0.03b | 0.37 ± 0.16a | 0.11 ± 0.01 |
| | 45 | 0.70 ± 0.02a | 0.27 ± 0.03b | 0.98 ± 0.05a | 0.36 ± 0.12a | 0.18 ± 0.00 |
| 6-Methyl-5-hepten-2-one | 0 | – | 0.00 ± 0.00b | – | – | 0.00 ± 0.00 |
| | 15 | – | 0.24 ± 0.05a | – | – | 0.00 ± 0.00 |
| | 30 | – | 0.18 ± 0.01a | – | – | 0.08 ± 0.02 |
| | 45 | – | 0.17 ± 0.01a | – | – | 0.00 ± 0.00 |
| β-Myrcene | 0 | – | 0.91 ± 0.02b | – | 0.32 ± 0.07 | 0.00 ± 0.00 |
| | 15 | – | 1.82 ± 0.18a | – | 0.00 ± 0.00 | 1.08 ± 0.10 |
| | 30 | – | 0.00 ± 0.00c | – | 0.00 ± 0.00 | 0.76 ± 0.16 |
| | 45 | – | 0.00 ± 0.00c | – | 0.00 ± 0.00 | 0.00 ± 0.00 |
| 2-pentyl-furan | 0 | – | 0.00 ± 0.00b | – | 0.00 ± 0.00c | 0.00 ± 0.00 |
| | 15 | – | 0.00 ± 0.00b | – | 0.00 ± 0.00c | 0.00 ± 0.00 |
| | 30 | – | 2.91 ± 0.07ab | – | 1.93 ± 0.35b | 0.00 ± 0.00 |
| | 45 | – | 5.31 ± 2.14a | – | 3.90 ± 0.19a | 1.75 ± 0.19 |
| Octanal | 0 | 0.00 ± 0.00 | 0.00 ± 0.00c | 0.00 ± 0.00b | 0.00 ± 0.00 | – |
| | 15 | 0.43 ± 0.36 | 0.30 ± 0.09c | 0.32 ± 0.04b | 0.72 ± 0.59 | – |
| | 30 | 0.61 ± 0.49 | 0.79 ± 0.01b | 0.37 ± 0.04b | 0.80 ± 0.06 | – |
| | 45 | 1.88 ± 1.18 | 1.43 ± 0.28a | 2.63 ± 0.51a | 0.70 ± 0.01 | – |
| Z-3-Hexenyl acetate | 0 | 0.19 ± 0.01 | 0.16 ± 0.02b | 0.12 ± 0.00 | 0.15 ± 0.02 | 0.00 ± 0.00 |
| | 15 | 0.25 ± 0.18 | 0.31 ± 0.10a | 0.26 ± 0.00 | 0.48 ± 0.40 | 0.13 ± 0.01 |
| | 30 | 0.00 ± 0.00 | 0.00 ± 0.00c | 0.00 ± 0.00 | 0.00 ± 0.00 | 0.00 ± 0.00 |
| | 45 | 0.00 ± 0.00 | 0.00 ± 0.00c | 0.00 ± 0.00 | 0.00 ± 0.00 | 0.00 ± 0.00 |
| E.E-2.4-Heptadienal | 0 | – | 0.00 ± 0.00d | – | 0.00 ± 0.00 | – |
| | 15 | – | 0.47 ± 0.03a | – | 0.59 ± 0.48 | – |
| | 30 | – | 0.26 ± 0.04b | – | 0.18 ± 0.01 | – |
| | 45 | – | 0.14 ± 0.01c | – | 0.00 ± 0.00 | – |
| α- Terpinene | 0 | – | 0.36 ± 0.06a | – | 0.08 ± 0.01 | 0.77 ± 0.01a |
| | 15 | – | 0.21 ± 0.01b | – | 0.00 ± 0.00 | 0.25 ± 0.02b |
| | 30 | – | 0.00 ± 0.00c | – | 0.00 ± 0.00 | 0.00 ± 0.00c |
| | 45 | – | 0.00 ± 0.00c | – | 0.00 ± 0.00 | 0.00 ± 0.00c |
| *p*-cymene | 0 | – | 3.59 ± 0.59b | 0.56 ± 0.08a | 2.25 ± 0.49a | 13.86 ± 1.14a |
| | 15 | – | 5.67 ± 0.11a | 0.12 ± 0.01b | 1.61 ± 0.51ab | 3.29 ± 0.20b |
| | 30 | – | 2.73 ± 0.03bc | 0.00 ± 0.00b | 0.83 ± 0.59b | 2.30 ± 0.04bc |
| | 45 | – | 1.48 ± 0.96c | 0.00 ± 0.00b | 0.48 ± 0.01b | 0.91 ± 0.03c |
| Limonene | 0 | – | 0.98 ± 0.03a | 0.54 ± 0.07a | 0.94 ± 0.20a | 3.68 ± 0.54 |
| | 15 | – | 0.79 ± 0.06b | 0.61 ± 0.05a | 0.61 ± 0.17ab | 0.28 ± 0.01 |
| | 30 | – | 0.00 ± 0.00c | 0.00 ± 0.00b | 0.31 ± 0.22b | 0.21 ± 0.01 |
| | 45 | – | 0.00 ± 0.00c | 0.00 ± 0.00b | 0.21 ± 0.01b | 0.00 ± 0.00 |
| Eucalyptol | 0 | – | – | – | 2.89 ± 0.81b | – |
| | 15 | – | – | – | 5.35 ± 0.18a | – |
| | 30 | – | – | – | 4.52 ± 0.81ab | – |
| | 45 | – | – | – | 4.13 ± 0.08ab | – |
| 3-Octen-2-one | 0 | 0.00 ± 0.00b | 0.00 ± 0.00b | 0.00 ± 0.00b | 0.00 ± 0.00b | 0.00 ± 0.00c |
| | 15 | 0.23 ± 0.01b | 0.25 ± 0.04b | 0.11 ± 0.04b | 0.38 ± 0.04a | 0.09 ± 0.03b |
| | 30 | 0.27 ± 0.12b | 0.64 ± 0.04a | 0.18 ± 0.00b | 0.56 ± 0.11a | 0.22 ± 0.01a |
| | 45 | 0.86 ± 0.15a | 0.79 ± 0.17a | 0.73 ± 0.23a | 0.57 ± 0.09a | 0.29 ± 0.05a |
| γ-Terpinene | 0 | – | 1.05 ± 0.18a | – | 0.14 ± 0.00 | 1.83 ± 0.11 |
| | 15 | – | 0.92 ± 0.01a | – | 0.00 ± 0.00 | 0.54 ± 0.05 |

**Table 2** (continued)

| Compound | Storage (days) | C | OO | MF | LN | TV |
|---|---|---|---|---|---|---|
| | 30 | – | 0.00 ± 0.00b | – | 0.00 ± 0.00 | 0.00 ± 0.00 |
| | 45 | – | 0.00 ± 0.00b | – | 0.00 ± 0.00 | 0.00 ± 0.00 |
| E-2-Octenal | 0 | 0.00 ± 0.00b | 2.61 ± 0.42b | 0.00 ± 0.00c | 0.00 ± 0.00b | 0.00 ± 0.00c |
| | 15 | 0.31 ± 0.11b | 6.66 ± 0.15a | 0.09 ± 0.01c | 1.58 ± 1.27ab | 0.00 ± 0.00c |
| | 30 | 2.65 ± 2.21ab | 0.00 ± 0.00c | 2.34 ± 0.26b | 4.19 ± 2.38a | 1.06 ± 0.13b |
| | 45 | 5.53 ± 1.67a | 0.00 ± 0.00c | 8.03 ± 0.66a | 4.04 ± 0.28a | 3.09 ± 0.01a |
| 3,5-Octadien-2-one | 0 | – | 0.00 ± 0.00c | – | – | 0.00 ± 0.00c |
| | 15 | – | 0.38 ± 0.02a | – | – | 0.19 ± 0.04b |
| | 30 | – | 0.33 ± 0.03a | – | – | 0.29 ± 0.04a |
| | 45 | – | 0.19 ± 0.04b | – | – | 0.00 ± 0.00c |
| Octanol | 0 | 0.00 ± 0.00 | 0.00 ± 0.00 | 0.00 ± 0.00b | – | 0.00 ± 0.00 |
| | 15 | 0.00 ± 0.00 | 0.00 ± 0.00 | 0.00 ± 0.00b | – | 0.00 ± 0.00 |
| | 30 | 0.00 ± 0.00 | 0.00 ± 0.00 | 0.25 ± 0.04b | – | 0.00 ± 0.00 |
| | 45 | 0.40 ± 0.016 | 0.27 ± 0.12 | 0.75 ± 0.29a | – | 0.25 ± 0.02 |
| 2-Nonanone | 0 | 0.00 ± 0.00 | 0.00 ± 0.00b | – | – | 0.00 ± 0.00 |
| | 15 | 0.00 ± 0.00 | 0.00 ± 0.00b | – | – | 0.00 ± 0.00 |
| | 30 | 0.00 ± 0.00 | 0.10 ± 0.04b | – | – | 0.00 ± 0.00 |
| | 45 | 0.40 ± 0.011 | 0.36 ± 0.06a | – | – | 0.07 ± 0.03 |
| Linalool | 0 | – | 1.06 ± 0.31ab | – | 0.12 ± 0.01 | 1.32 ± 0.00a |
| | 15 | – | 1.45 ± 0.04a | – | 0.00 ± 0.00 | 1.04 ± 0.03b |
| | 30 | – | 0.72 ± 0.01b | – | 0.00 ± 0.00 | 0.70 ± 0.04c |
| | 45 | – | 0.24 ± 0.03c | – | 0.00 ± 0.00 | 0.17 ± 0.00d |
| Nonanal | 0 | 0.08 ± 0.01 | 0.15 ± 0.01c | 0.00 ± 0.00b | 0.13 ± 0.00b | 0.00 ± 0.00 |
| | 15 | 1.02 ± 0.06 | 1.29 ± 0.16b | 0.51 ± 0.02b | 1.17 ± 0.13a | 0.46 ± 0.14 |
| | 30 | 1.14 ± 0.57 | 1.68 ± 0.07b | 0.58 ± 0.04b | 1.16 ± 0.04a | 0.71 ± 0.06 |
| | 45 | 2.68 ± 1.76 | 2.37 ± 0.46a | 3.28 ± 0.64a | 1.18 ± 0.05a | 0.62 ± 0.06 |
| p-Menthone | 0 | – | – | 0.25 ± 0.05c | – | – |
| | 15 | – | – | 1.49 ± 0.11a | – | – |
| | 30 | – | – | 0.37 ± 0.04c | – | – |
| | 45 | – | – | 0.66 ± 0.13b | – | – |
| E-2-Nonenal | 0 | 0.00 ± 0.00 | 0.00 ± 0.00b | 0.00 ± 0.00c | 0.00 ± 0.00c | 0.00 ± 0.00c |
| | 15 | 0.00 ± 0.00 | 0.00 ± 0.00b | 0.00 ± 0.00c | 0.00 ± 0.00c | 0.00 ± 0.00c |
| | 30 | 0.72 ± 0.66 | 0.44 ± 0.02b | 0.35 ± 0.09b | 0.59 ± 0.36b | 0.09 ± 0.03b |
| | 45 | 1.66 ± 1.32 | 1.71 ± 0.36a | 2.02 ± 0.04a | 1.37 ± 0.01a | 0.60 ± 0.03a |
| Isopulegone | 0 | – | – | 0.17 ± 0.02b | – | – |
| | 15 | – | – | 0.67 ± 0.06a | – | – |
| | 30 | – | – | 0.00 ± 0.00c | – | – |
| | 45 | – | – | 0.00 ± 0.00c | – | – |
| Terpinen-4-ol | 0 | – | 0.15 ± 0.06b | – | 0.58 ± 0.21 | 0.11 ± 0.00b |
| | 15 | – | 0.24 ± 0.00a | – | 0.61 ± 0.16 | 0.14 ± 0.00a |
| | 30 | – | 0.13 ± 0.01b | – | 0.33 ± 0.25 | 0.11 ± 0.01b |
| | 45 | – | 0.00 ± 0.00c | – | 0.21 ± 0.01 | 0.00 ± 0.00c |
| α-Terpineol | 0 | – | – | – | 0.20 ± 0.06a | – |
| | 15 | – | – | – | 0.26 ± 0.03a | – |
| | 30 | – | – | – | 0.00 ± 0.00b | – |
| | 45 | – | – | – | 0.00 ± 0.00b | – |
| Pulegone | 0 | – | – | 5.05 ± 0.71b | – | – |
| | 15 | – | – | 12.19 ± 0.47a | – | – |
| | 30 | – | – | 1.97 ± 0.18c | – | – |
| | 45 | – | – | 2.85 ± 0.57c | – | – |
| E.E-2,4-nonadienal | 0 | – | 0.00 ± 0.00c | – | – | – |
| | 15 | – | 0.00 ± 0.00c | – | – | – |
| | 30 | – | 0.08 ± 0.01b | – | – | – |
| | 45 | – | 0.21 ± 0.01a | – | – | – |
| E-2-Decenal | 0 | 0.00 ± 0.00 | 0.00 ± 0.00c | 0.00 ± 0.00b | 0.00 ± 0.00b | 0.00 ± 0.00 |
| | 15 | 0.00 ± 0.00 | 0.09 ± 0.01c | 0.00 ± 0.00b | 0.40 ± 0.31b | 0.00 ± 0.00 |
| | 30 | 1.99 ± 1.89 | 0.87 ± 0.04b | 1.18 ± 0.42b | 1.69 ± 1.31ab | 0.00 ± 0.00 |
| | 45 | 3.37 ± 2.68 | 4.31 ± 0.40a | 3.72 ± 0.90a | 2.74 ± 0.10a | 2.15 ± 0.31 |
| Piperitenone | 0 | – | – | 0.00 ± 0.00 | – | – |
| | 15 | – | – | 0.23 ± 0.06 | – | – |
| | 30 | – | – | 0.39 ± 0.10 | – | – |
| | 45 | – | – | 0.00 ± 0.00 | – | – |
| Thymol | 0 | – | – | – | – | 0.26 ± 0.10a |
| | 15 | – | – | – | – | 0.06 ± 0.01b |
| | 30 | – | – | – | – | 0.08 ± 0.01b |

**Table 2** (continued)

| Compound | Storage (days) | C | OO | MF | LN | TV |
|---|---|---|---|---|---|---|
| | 45 | – | – | – | – | 0.00 ± 0.00b |
| Carvacrol | 0 | – | 2.55 ± 1.23ab | – | – | 2.29 ± 0.52ab |
| | 15 | – | 4.16 ± 0.00a | – | – | 2.67 ± 0.11a |
| | 30 | – | 3.79 ± 0.26ab | – | – | 2.69 ± 0.21a |
| | 45 | – | 2.30 ± 0.09b | – | – | 0.77 ± 0.01b |
| E.Z-2,4-Decadienal | 0 | – | 0.00 ± 0.00c | 0.00 ± 0.00c | 0.00 ± 0.00c | 0.00 ± 0.00c |
| | 15 | – | 0.00 ± 0.00c | 0.00 ± 0.00c | 0.00 ± 0.00c | 0.00 ± 0.00c |
| | 30 | – | 0.47 ± 0.06b | 0.18 ± 0.06b | 0.42 ± 0.06b | 0.13 ± 0.07b |
| | 45 | – | 1.08 ± 0.25a | 0.51 ± 0.01a | 0.91 ± 0.04a | 0.34 ± 0.02a |
| α-Terpinyl acetate | 0 | – | – | – | 0.73 ± 0.30 | – |
| | 15 | – | – | – | 1.14 ± 0.24 | – |
| | 30 | – | – | – | 0.91 ± 0.40 | – |
| | 45 | – | – | – | 0.94 ± 0.05 | – |

Results (mean ± standard deviation) are expressed as means of total ion current (TIC) area units ($\times 10^{-6}$)

Mean values within each column for the same storage time followed by the same letters are not significantly different at $P < 0.05$

The levels of these alcohols were increased at the later stages of oxidation.

Ketones such as E-2-octene, 2-heptanone, 2-nonane, 6-methyl-5-hepten-2-one, 3-octen-2-one, and 3,5-octadien-2-one were detected in the headspace of the analyzed oils, and some of these components are known to be linked with oxidation processes.

Except for E-2-hexenal, 2-butenal, E-2-pentenal, E-2-heptenal, E-2-octenal, E-2-nonenal, and E-2-decenal were also detected. Especially, E-2-heptenal, E-2-octenal, and E-2-decenal were detected as the most abundant 2-alkenals in olive oil samples during heating. The contents of E-2-heptenal and E-2-decenal were similar in control and flavored oil samples except TV sample which exhibited lower values than those of other samples. A similar trend for E-2-octenal was observed; however, the highest value of this compound in OO was $6.66 \times 10^6$ AU after 15 days of storage, and then an immediate decrease was observed. In literature, the presence of 2-alkenals in olive oil samples treated at high temperatures was also evidenced by other studies (Gómez-Alonso et al. 2004; Issaoui et al. 2011; Poyato et al. 2014).

The members of 2,4-alkadienals group namely 2,4-heptadienal, 2,4-nonadienal, and 2,4-decadienal were not identified in the control sample. However, E,E-2,4-nonadienal was identified only in OO sample, while E,E-2,4-heptadienal was identified in both OO and LN samples. Besides, E,E-2,4-decadienal was identified in all flavored samples after 30 days of storage. Although some vegetable oils include small amounts of alkadienals, oxidized oils—in particular—contain higher amounts of these compounds whose concentrations can be related to fatty acids. Oils that are rich in oleic acid were reported to contain these compounds in fewer levels than high linoleic acid-containing oils (Wang et al. 2019).

During thermal oxidation, only one furan namely 2-pentylfuran, a typical degradation compound of linoleic acyl groups, formed in OO, LN, and TV samples. At the end of 45 days of storage, the content of this compound in OO, LN, and TV samples reached up to 5.31, 3.90, and $1.75 \times 10^6$ AU, respectively. The presence of 2-pentylfuran was also reported in the headspace of the edible oil samples during oxidation (Morales et al. 1997; Xu et al. 2017). Our results were in good agreement with Issaoui et al. (2011) stating that 2-pentylfuran was absent in heated olive oils, while this compound appeared in thyme-flavored olive oil during thermal oxidation.

Other compounds such as terpenes, which are not normally present in non-oxidized virgin olive oil, were detected at varying concentrations in flavored oils. α-Thujene, α-pinene, sabinene, β-pinene, β-myrcene, α-terpinene, *p*-cymene, limonene, eucalyptol, γ-terpinene, linalool, *p*-menthone, 4-terpineol, α-terpineol, thymol, carvacrol, isopulegone, pulegone, piperitenone, and α-terpinyl acetate were identified in analyzed samples. These are major compounds present in essential oils and are transferred from essential oil to olive oil samples. Issaoui et al. (2011) also reported that some compounds (e.g., carvacrol, α-thujene, α-pinene, myrcene, α-terpinene, γ-terpinene, and 4-terpineol) with aromatic and antioxidant properties were released to olive oil. Carvacrol is the major component of *Origanum onites* and *Thymus vulgaris* essential oils and it was detected at high levels in OO and TV samples. This volatile compound showed oxidative stability against thermal oxidation, and a similar trend was also reported for thyme-flavored olive oils at 100 °C (Issaoui et al. 2011). Pulegone, which was transferred from *Micromeria fruticosa* essential oil, was also detected in the MF sample. Pulegone was the major component of *M. fruticosa* essential oil (65.30%). Among terpenes, *p*-cymene was detected in all flavored oils with a good transfer behavior to olive oil samples. The highest concentration of *p*-cymene was $13.86 \times 10^6$ AU in the TV sample, while its lowest level ($0.56 \times 10^6$ AU) was

**Table 3** Changes in volatile compounds of olive oil samples during photo-oxidation

| Compound | Storage (days) | C | OO | MF | LN | TV |
|---|---|---|---|---|---|---|
| 2-Butenal | 0 | 0.14 ± 0.04b | 0.00 ± 0.00c | 0.00 ± 0.00 | 0.00 ± 0.00c | 0.00 ± 0.00b |
| | 15 | 0.06 ± 0.03c | 0.03 ± 0.02c | 0.07 ± 0.01 | 0.03 ± 0.01bc | 0.03 ± 0.01ab |
| | 30 | 0.08 ± 0.01bc | 0.09 ± 0.01b | 0.14 ± 0.03 | 0.09 ± 0.01b | 0.09 ± 0.02ab |
| | 45 | 0.24 ± 0.01a | 0.42 ± 0.01a | 0.76 ± 0.62 | 0.82 ± 0.04a | 0.13 ± 0.07a |
| Pentanal | 0 | 0.00 ± 0.00c | 0.00 ± 0.00c | 0.00 ± 0.00c | 0.00 ± 0.00c | 0.00 ± 0.00b |
| | 15 | 0.66 ± 0.04a | 0.40 ± 0.01a | 0.85 ± 0.11a | 0.43 ± 0.06ab | 0.32 ± 0.01a |
| | 30 | 0.61 ± 0.03a | 0.39 ± 0.01a | 0.86 ± 0.01a | 0.50 ± 0.03a | 0.34 ± 0.05a |
| | 45 | 0.50 ± 0.01b | 0.29 ± 0.00b | 0.59 ± 0.08b | 0.33 ± 0.02b | 0.00 ± 0.00b |
| E-2-Pentenal | 0 | 0.10 ± 0.01a | 0.00 ± 0.00b | 0.00 ± 0.00c | – | 0.00 ± 0.00b |
| | 15 | 0.05 ± 0.01b | 0.03 ± 0.01a | 0.06 ± 0.01b | – | 0.03 ± 0.01a |
| | 30 | 0.04 ± 0.00b | 0.03 ± 0.00a | 0.05 ± 0.00b | – | 0.04 ± 0.01a |
| | 45 | 0.04 ± 0.00b | 0.00 ± 0.00b | 0.10 ± 0.00a | – | 0.00 ± 0.00b |
| Pentanol | 0 | – | – | – | 0.25 ± 0.03 | – |
| | 15 | – | – | – | 0.00 ± 0.00 | – |
| | 30 | – | – | – | 0.00 ± 0.00 | – |
| | 45 | – | – | – | 0.00 ± 0.00 | – |
| Z-2-penten-1-ol | 0 | 0.06 ± 0.00a | – | – | – | – |
| | 15 | 0.06 ± 0.03a | – | – | – | – |
| | 30 | 0.04 ± 0.01ab | – | – | – | – |
| | 45 | 0.00 ± 0.00b | – | – | – | – |
| 3-Methyl-2-butenal | 0 | 0.00 ± 0.00b | 0.00 ± 0.00 | 0.00 ± 0.00b | 0.00 ± 0.00 | – |
| | 15 | 0.00 ± 0.00b | 0.00 ± 0.00 | 0.00 ± 0.00b | 0.00 ± 0.00 | – |
| | 30 | 0.02 ± 0.01b | 0.00 ± 0.00 | 0.05 ± 0.01b | 0.03 ± 0.01 | – |
| | 45 | 0.14 ± 0.01a | 0.25 ± 0.03 | 0.53 ± 0.32a | 0.41 ± 0.11 | – |
| Hexanal | 0 | 4.34 ± 0.08a | 2.38 ± 0.12a | 3.45 ± 0.05a | 2.15 ± 0.09 | 5.62 ± 0.47a |
| | 15 | 1.78 ± 0.21b | 0.93 ± 0.18b | 2.05 ± 0.04bc | 1.03 ± 0.15 | 0.83 ± 0.03b |
| | 30 | 1.66 ± 0.08b | 0.92 ± 0.06b | 1.73 ± 0.13c | 1.05 ± 0.15 | 0.73 ± 0.01b |
| | 45 | 1.67 ± 0.11b | 1.02 ± 0.01b | 2.47 ± 0.33b | 1.66 ± 0.35 | 0.00 ± 0.00c |
| E-2-octene | 0 | – | – | 2.51 ± 0.07a | 1.32 ± 0.18 | – |
| | 15 | – | – | 0.01 ± 0.00b | 0.00 ± 0.00 | – |
| | 30 | – | – | 0.00 ± 0.00b | 0.05 ± 0.00 | – |
| | 45 | – | – | 0.00 ± 0.00b | 0.00 ± 0.00 | – |
| E-2-hexenal | 0 | 4.38 ± 0.03a | 1.15 ± 0.04c | 1.58 ± 0.06c | 1.46 ± 0.12a | 1.04 ± 0.13b |
| | 15 | 2.32 ± 0.20b | 1.77 ± 0.26a | 3.09 ± 0.19a | 1.74 ± 0.22a | 1.87 ± 0.02b |
| | 30 | 2.11 ± 0.03b | 1.62 ± 0.02ab | 2.47 ± 0.18b | 1.63 ± 0.04a | 1.68 ± 0.04b |
| | 45 | 1.57 ± 0.14c | 1.37 ± 0.07bc | 1.48 ± 0.25c | 0.78 ± 0.08b | 54.97 ± 13.10a |
| Z-3-Hexen-1-ol | 0 | 1.86 ± 0.07 | 0.62 ± 0.00 | 0.85 ± 0.06a | 0.72 ± 0.07a | 0.83 ± 0.13a |
| | 15 | 0.82 ± 0.21 | 0.56 ± 0.24 | 0.74 ± 0.15ab | 0.60 ± 0.19a | 0.55 ± 0.01b |
| | 30 | 0.80 ± 0.07 | 0.57 ± 0.12 | 0.94 ± 0.09a | 0.54 ± 0.03a | 0.59 ± 0.01b |
| | 45 | 0.62 ± 0.04 | 0.37 ± 0.00 | 0.46 ± 0.13b | 0.20 ± 0.04b | 0.00 ± 0.00c |
| E-2-hexen-1-ol | 0 | 0.29 ± 0.01a | 0.20 ± 0.01a | 0.19 ± 0.02a | 0.19 ± 0.01c | 0.28 ± 0.07a |
| | 15 | 0.13 ± 0.02b | 0.09 ± 0.04b | 0.17 ± 0.01a | 0.10 ± 0.02b | 0.10 ± 0.01b |
| | 30 | 0.13 ± 0.01b | 0.08 ± 0.01b | 0.13 ± 0.01ab | 0.09 ± 0.01b | 0.09 ± 0.01b |
| | 45 | 0.08 ± 0.01c | 0.05 ± 0.01b | 0.08 ± 0.04b | 0.00 ± 0.00a | 0.00 ± 0.00b |
| Hexanol | 0 | 0.32 ± 0.02a | 0.17 ± 0.01a | 0.18 ± 0.04 | 0.17 ± 0.00a | 0.39 ± 0.11a |
| | 15 | 0.18 ± 0.01b | 0.10 ± 0.04b | 0.18 ± 0.03 | 0.11 ± 0.03ab | 0.09 ± 0.00b |
| | 30 | 0.18 ± 0.01b | 0.08 ± 0.01b | 0.15 ± 0.04 | 0.10 ± 0.04b | 0.07 ± 0.01b |
| | 45 | 0.10 ± 0.01c | 0.00 ± 0.00c | 0.09 ± 0.05 | 0.00 ± 0.00c | 0.00 ± 0.00b |
| Nonane | 0 | – | – | – | 1.35 ± 0.08 | 2.88 ± 0.57 |
| | 15 | – | – | – | 0.00 ± 0.00 | 0.00 ± 0.00 |
| | 30 | – | – | – | 0.00 ± 0.00 | 0.00 ± 0.00 |
| | 45 | – | – | – | 0.00 ± 0.00 | 0.00 ± 0.00 |
| Heptanal | 0 | 0.03 ± 0.01 | – | – | – | – |
| | 15 | 0.00 ± 0.00 | – | – | – | – |
| | 30 | 0.00 ± 0.00 | – | – | – | – |
| | 45 | 0.00 ± 0.00 | – | – | – | – |
| α-Thujene | 0 | – | 0.09 ± 0.00a | – | 0.07 ± 0.01a | – |
| | 15 | – | 0.08 ± 0.02a | – | 0.05 ± 0.01b | – |
| | 30 | – | 0.06 ± 0.00a | – | 0.03 ± 0.00b | – |
| | 45 | – | 0.00 ± 0.00b | – | 0.00 ± 0.00c | – |
| α-Pinene | 0 | – | – | – | 0.14 ± 0.04b | 0.08 ± 0.01b |
| | 15 | – | – | – | 0.26 ± 0.01a | 0.09 ± 0.01b |
| | 30 | – | – | – | 0.23 ± 0.00a | 0.09 ± 0.01b |
| | 45 | – | – | – | 0.22 ± 0.00a | 0.13 ± 0.00a |
| E-2-Heptenal | 0 | 0.05 ± 0.01a | 0.00 ± 0.00c | 0.00 ± 0.00 | 0.00 ± 0.00c | 0.00 ± 0.00c |

**Table 3** (continued)

| Compound | Storage (days) | C | OO | MF | LN | TV |
|---|---|---|---|---|---|---|
| | 15 | 0.20 ± 0.02b | 0.16 ± 0.01bc | 0.29 ± 0.00 | 0.17 ± 0.02bc | 0.14 ± 0.03bc |
| | 30 | 0.47 ± 0.03c | 0.46 ± 0.01b | 0.75 ± 0.01 | 0.50 ± 0.06b | 0.47 ± 0.03a |
| | 45 | 1.34 ± 0.08d | 2.01 ± 0.23a | 3.06 ± 2.49 | 3.35 ± 0.23a | 0.19 ± 0.11b |
| Sabinene | 0 | – | – | – | 0.42 ± 0.11b | – |
| | 15 | – | – | – | 0.68 ± 0.01a | – |
| | 30 | – | – | – | 0.61 ± 0.04a | – |
| | 45 | – | – | – | 0.52 ± 0.04ab | – |
| β-Pinene | 0 | – | – | – | 0.11 ± 0.03b | – |
| | 15 | – | – | – | 0.20 ± 0.01a | – |
| | 30 | – | – | – | 0.18 ± 0.01a | – |
| | 45 | – | – | – | 0.17 ± 0.01a | – |
| Octen-3-ol | 0 | 0.05 ± 0.01b | – | – | 0.00 ± 0.00 | 0.19 ± 0.01a |
| | 15 | 0.00 ± 0.00c | – | – | 0.00 ± 0.00 | 0.21 ± 0.02a |
| | 30 | 0.02 ± 0.00c | – | – | 0.00 ± 0.00 | 0.23 ± 0.01a |
| | 45 | 0.10 ± 0.01a | – | – | 0.30 ± 0.02 | 0.00 ± 0.00b |
| 6-Methyl-5-hepten-2-one | 0 | 0.00 ± 0.00d | 0.00 ± 0.00d | 0.00 ± 0.00 | 0.00 ± 0.00d | 0.00 ± 0.00c |
| | 15 | 0.10 ± 0.01c | 0.12 ± 0.01c | 0.00 ± 0.00 | 0.09 ± 0.01c | 0.11 ± 0.01b |
| | 30 | 0.19 ± 0.03b | 0.18 ± 0.00b | 0.00 ± 0.00 | 0.14 ± 0.01b | 0.17 ± 0.01a |
| | 45 | 0.31 ± 0.01a | 0.32 ± 0.01a | 0.54 ± 0.18 | 0.33 ± 0.03a | 0.00 ± 0.00c |
| β-Myrcene | 0 | – | 0.91 ± 0.22b | 0.00 ± 0.00c | 0.32 ± 0.07ab | 0.00 ± 0.00d |
| | 15 | – | 1.32 ± 0.17a | 0.21 ± 0.01a | 0.39 ± 0.05a | 2.02 ± 0.09a |
| | 30 | – | 1.14 ± 0.04ab | 0.23 ± 0.04a | 0.32 ± 0.06ab | 1.73 ± 0.10b |
| | 45 | – | 0.85 ± 0.06b | 0.13 ± 0.02b | 0.19 ± 0.01b | 0.27 ± 0.11c |
| Octanal | 0 | 0.00 ± 0.00 | – | 0.00 ± 0.00 | 0.00 ± 0.00 | – |
| | 15 | 0.00 ± 0.00 | – | 0.00 ± 0.00 | 0.00 ± 0.00 | – |
| | 30 | 0.00 ± 0.00 | – | 0.00 ± 0.00 | 0.00 ± 0.00 | – |
| | 45 | 0.03 ± 0.00 | – | 0.09 ± 0.02 | 0.10 ± 0.04 | – |
| Z-3-Hexenyl acetate | 0 | 0.19 ± 0.01a | 0.16 ± 0.02 | 0.12 ± 0.00c | 0.15 ± 0.02 | 0.00 ± 0.00 |
| | 15 | 0.19 ± 0.01a | 0.18 ± 0.01 | 0.23 ± 0.04a | 0.15 ± 0.04 | 0.19 ± 0.00 |
| | 30 | 0.20 ± 0.01a | 0.17 ± 0.00 | 0.20 ± 0.01ab | 0.17 ± 0.01 | 0.19 ± 0.00 |
| | 45 | 0.15 ± 0.01b | 0.16 ± 0.00 | 0.15 ± 0.03bc | 0.11 ± 0.01 | 0.00 ± 0.00 |
| p-Cymene | 0 | – | 3.59 ± 0.59a | 0.56 ± 0.08a | 2.25 ± 0.49a | 13.86 ± 1.14a |
| | 15 | – | 2.85 ± 0.16ab | 0.05 ± 0.01b | 2.08 ± 0.04a | 4.46 ± 0.01b |
| | 30 | – | 2.88 ± 0.05ab | 0.04 ± 0.00b | 1.99 ± 0.04a | 4.65 ± 0.11b |
| | 45 | – | 2.40 ± 0.05b | 0.00 ± 0.00b | 1.22 ± 0.17b | 1.42 ± 0.47c |
| Limonene | 0 | – | 0.98 ± 0.03a | 0.54 ± 0.07a | 0.94 ± 0.20a | 3.68 ± 0.54a |
| | 15 | – | 0.37 ± 0.04b | 0.43 ± 0.01ab | 0.80 ± 0.08a | 0.48 ± 0.01b |
| | 30 | – | 0.40 ± 0.06b | 0.42 ± 0.02ab | 0.84 ± 0.07a | 0.52 ± 0.01b |
| | 45 | – | 0.18 ± 0.05c | 0.34 ± 0.07b | 0.42 ± 0.11b | 0.13 ± 0.18b |
| Eucalyptol | 0 | – | – | – | 2.89 ± 0.81 | – |
| | 15 | – | – | – | 3.90 ± 0.01 | – |
| | 30 | – | – | – | 3.55 ± 0.19 | – |
| | 45 | – | – | – | 3.49 ± 0.07 | – |
| 3-Octen-2-one | 0 | 0.00 ± 0.00 | – | 0.00 ± 0.00 | 0.00 ± 0.00 | – |
| | 15 | 0.00 ± 0.00 | – | 0.00 ± 0.00 | 0.00 ± 0.00 | – |
| | 30 | 0.00 ± 0.00 | – | 0.00 ± 0.00 | 0.00 ± 0.00 | – |
| | 45 | 0.04 ± 0.01 | – | 0.07 ± 0.04 | 0.09 ± 0.01 | – |
| γ-Terpinene | 0 | – | 1.05 ± 0.18a | – | 0.14 ± 0.04a | 1.83 ± 0.11a |
| | 15 | – | 1.09 ± 0.11a | – | 0.10 ± 0.00a | 1.58 ± 0.01b |
| | 30 | – | 0.96 ± 0.01a | – | 0.10 ± 0.01a | 1.52 ± 0.04b |
| | 45 | – | 0.41 ± 0.04b | – | 0.00 ± 0.00b | 0.38 ± 0.06c |
| (E)-2-Octenal | 0 | 0.00 ± 0.00 | – | 0.00 ± 0.00 | 0.00 ± 0.00 | – |
| | 15 | 0.00 ± 0.00 | – | 0.00 ± 0.00 | 0.00 ± 0.00 | – |
| | 30 | 0.00 ± 0.00 | – | 0.00 ± 0.00 | 0.00 ± 0.00 | – |
| | 45 | 0.03 ± 0.01 | – | 0.11 ± 0.10 | 0.27 ± 0.17 | – |
| Linalool | 0 | – | 1.06 ± 0.31a | – | 0.12 ± 0.01b | 1.32 ± 0.00b |
| | 15 | – | 1.03 ± 0.04ab | – | 0.14 ± 0.00a | 1.51 ± 0.01a |
| | 30 | – | 0.86 ± 0.04ab | – | 0.10 ± 0.00c | 1.27 ± 0.01b |
| | 45 | – | 0.61 ± 0.02b | – | 0.00 ± 0.00d | 0.45 ± 0.10c |
| Nonanal | 0 | 0.08 ± 0.01b | 0.15 ± 0.01c | 0.00 ± 0.00c | 0.13 ± 0.00 | 0.00 ± 0.00c |
| | 15 | 0.17 ± 0.04ab | 0.18 ± 0.02bc | 0.17 ± 0.03b | 0.16 ± 0.03 | 0.20 ± 0.01a |
| | 30 | 0.20 ± 0.02a | 0.20 ± 0.01b | 0.20 ± 0.01b | 0.22 ± 0.00 | 0.23 ± 0.01a |
| | 45 | 0.24 ± 0.06a | 0.30 ± 0.01a | 0.26 ± 0.02a | 0.28 ± 0.02 | 0.08 ± 0.04b |
| p-Menthone | 0 | – | – | 0.25 ± 0.05b | – | – |
| | 15 | – | – | 0.52 ± 0.01a | – | – |

**Table 3** (continued)

| Compound | Storage (days) | C | OO | MF | LN | TV |
|---|---|---|---|---|---|---|
| | 30 | – | – | 0.54 ± 0.01a | – | – |
| | 45 | – | – | 0.48 ± 0.04a | – | – |
| Isopulegone | 0 | – | – | 0.17 ± 0.02b | – | – |
| | 15 | – | – | 0.35 ± 0.04a | – | – |
| | 30 | – | – | 0.35 ± 0.00a | – | – |
| | 45 | – | – | 0.26 ± 0.08ab | – | – |
| Terpinen-4-ol | 0 | – | 0.15 ± 0.06 | – | 0.58 ± 0.21 | 0.11 ± 0.00b |
| | 15 | – | 0.20 ± 0.01 | – | 0.70 ± 0.04 | 0.24 ± 0.01a |
| | 30 | – | 0.18 ± 0.01 | – | 0.63 ± 0.01 | 0.21 ± 0.01a |
| | 45 | – | 0.16 ± 0.02 | – | 0.43 ± 0.06 | 0.09 ± 0.01b |
| α-Terpineol | 0 | – | 0.00 ± 0.00b | – | 0.20 ± 0.06 | 0.00 ± 0.00b |
| | 15 | – | 0.07 ± 0.02a | – | 0.29 ± 0.01 | 0.10 ± 0.02a |
| | 30 | – | 0.07 ± 0.03a | – | 0.27 ± 0.02 | 0.09 ± 0.01a |
| | 45 | – | 0.08 ± 0.00a | – | 0.20 ± 0.02 | 0.00 ± 0.00b |
| Pulegone | 0 | – | – | 5.05 ± 0.71c | – | – |
| | 15 | – | – | 15.52 ± 0.75a | – | – |
| | 30 | – | – | 12.83 ± 0.14ab | – | – |
| | 45 | – | – | 10.22 ± 2.33b | – | – |
| Dec-2(E)-enal | 0 | 0.00 ± 0.00 | 0.00 ± 0.00 | – | 0.00 ± 0.00 | 0.00 ± 0.00 |
| | 15 | 0.00 ± 0.00 | 0.00 ± 0.00 | – | 0.00 ± 0.00 | 0.00 ± 0.00 |
| | 30 | 0.03 ± 0.01 | 0.00 ± 0.00 | – | 0.00 ± 0.00 | 0.00 ± 0.00 |
| | 45 | 0.03 ± 0.04 | 0.11 ± 0.01 | – | 0.17 ± 0.04 | 0.07 ± 0.00 |
| Piperitone | 0 | – | – | 0.00 ± 0.00 | – | – |
| | 15 | – | – | 0.06 ± 0.02 | – | – |
| | 30 | – | – | 0.05 ± 0.01 | – | – |
| | 45 | – | – | 0.00 ± 0.00 | – | – |
| Thymol | 0 | – | – | – | – | 0.26 ± 0.10a |
| | 15 | – | – | – | – | 0.06 ± 0.01b |
| | 30 | – | – | – | – | 0.05 ± 0.01b |
| | 45 | – | – | – | – | 0.00 ± 0.00b |
| Carvacrol | 0 | – | 2.55 ± 1.23b | – | – | 2.29 ± 0.52b |
| | 15 | – | 3.16 ± 0.51ab | – | – | 3.72 ± 0.19a |
| | 30 | – | 3.76 ± 0.39ab | – | – | 3.88 ± 0.25a |
| | 45 | – | 4.71 ± 0.11a | – | – | 3.33 ± 0.57ab |
| Piperitenone | 0 | – | – | 0.00 ± 0.00c | – | – |
| | 15 | – | – | 0.44 ± 0.06a | – | – |
| | 30 | – | – | 0.40 ± 0.01ab | – | – |
| | 45 | – | – | 0.33 ± 0.05b | – | – |
| α-Terpinyl acetate | 0 | – | – | – | 0.73 ± 0.30 | – |
| | 15 | – | – | – | 1.16 ± 0.04 | – |
| | 30 | – | – | – | 1.20 ± 0.19 | – |
| | 45 | – | – | – | 1.10 ± 0.00 | – |
| Piperitenone oxide | 0 | – | – | 0.00 ± 0.00b | – | – |
| | 15 | – | – | 0.12 ± 0.02a | – | – |
| | 30 | – | – | 0.10 ± 0.01a | – | – |
| | 45 | – | – | 0.09 ± 0.00a | – | – |

Results (mean ± standard deviation) are expressed as means of total ion current (TIC) area units ($\times 10^{-6}$)

Mean values within each column for the same storage time followed by the same letters are not significantly different at $P < 0.05$

analyzed in MF. Eucalyptol (1,8-cineol), which is a major component of *L. nobilis* essential oil (35.06%), was only detected in the LN sample. Also, eucalyptol remained stable during the thermal oxidation process.

## Photo-Oxidation

According to the headspace analysis of oil samples, more than 20 volatile compounds were formed during storage (Table 1). The highest number of identified compounds was 33 in the LN sample, while it was the minimum in the control sample with 20 components. The identified volatile compounds in olive oils during photo-oxidation are presented in Table 3. The major volatile compounds were hexanal and E-2-hexenal before the storage period. These compounds generally decreased during photo-oxidation in all samples with one exception; TV had the highest value of E-2-hexenal ($54.97 \times 10^6$ AU) at the end of 45 days of storage. The amount of E-2-heptenal increased with prolonged storage time under photo-oxidation conditions. At the end of 45 days of storage, the

level of E-2-heptanal increased up to $3.35 \times 10^6$ AU in the LN sample, while it reached only $0.19 \times 10^6$ AU in the TV sample. E-2-heptenal was considered as a photo-oxidation product in a previous report demonstrating that it was formed and reached higher levels as oxidation occurred in such oils as apricot and plum oils which are also rich in oleic acid like olive oil (Kiralan et al. 2018). As similar to our results, Kanavouras et al. (2004) demonstrated that E-2-heptenal was more abundant in olive oil stored in the light. Such aldehydes as 2-butenal, pentanal, E-2-pentenal, 3-methyl-2-butenal, heptanal, E-2-octenal, nonanal, and E-2-decenal were also identified in the headspace of oil samples. Among these compounds, 2-butenal, pentanal, and nonanal appeared as volatile oxidation compounds in all oil samples during photo-oxidation. E-2-pentenal, 3-methyl-2-butenal, and E-2-decenal did not form in LN, TV, and MF. Heptanal was only identified in the control sample and not detected in any other group. Most of these volatile compounds increased with photo-oxidation and their levels reached the maximum at the end of the storage period.

Alcohols including pentanol, Z-3-hexen-1-ol, E-2-hexen-1-ol, hexanol, and octen-3-ol were determined in the analyzed samples. The concentrations of pentanol, Z-3-hexen-1-ol, E-2-hexen-1-ol, and hexanol were decreased or these compounds disappeared during storage. The level of octen-3-ol increased in two photo-oxidized samples, C and LN, at the end of 45 days of storage, while this compound disappeared in the TV sample at the end of the storage period.

Regarding ketones, E-2-octene, 6-methyl-5-hepten-2-one, and 3-octen-2-one were identified. E-2-octene was the only detected volatile compound in two samples (MF and LN) at the beginning and this compound also disappeared at the end of the oxidation process. A similar trend was observed in a previous study by Lee and Min (2010). The researchers reported that 2-octene decreased dramatically in the linoleic acid system stored under light. 6-Methyl-5-hepten-2-one was detected as a result of the oxidation and the level of this volatile compound was higher in oil samples at the end of storage, except TV sample. 3-Octen-2-one was present in C, MF, and LN samples only at the end of the storage. Lee and Min (2010) reported that 3-octen-2-one was only identified in one sample mixed with chlorophyll during light storage.

$\alpha$-Thujene, $\alpha$-pinene, sabinene, $\beta$-pinene, $\beta$-myrcene, *p*-cymene, limonene, eucalyptol, $\gamma$-terpinene, linalool, *p*-menthone, 4-terpineol, $\alpha$-terpineol, thymol, carvacrol, isopulegone, pulegone, piperitone, piperitenone, piperitenone oxide, and $\alpha$-terpinyl acetate were identified as terpene compounds in samples. Eucalyptol was only identified in the LN sample at levels ranging between $2.89–3.90 \times 10^6$ AU. Similarly, carvacrol also remained stable during storage. *p*-Menthone, isopulegone, pulegone, piperitone, piperitenone, and piperitenone oxide were also identified in the MF sample and most of them were stable during oxidation. Besides, the concentrations of other compounds such as $\alpha$-thujene, $\alpha$-pinene, sabinene, $\beta$-pinene, $\beta$-myrcene, 4-terpineol, *p*-cymene, and $\alpha$-terpineol did not significantly change in contrast to the main volatile compounds found in essential oils during oxidation.

## Conclusion

Flavored oils have been used in such food processing techniques as cooking where heat is available. Besides, the oxidative effects of light have been studied on the valuable volatile compounds which contribute to the aroma of olive oils as well as antioxidant activity. To our best knowledge, no literature reports were available regarding the effects of thermal and photo-oxidation conditions on the volatile compounds of olive oils flavored with essential oils. Results indicated that E-2-hexenal formed at higher concentrations in oils during both oxidation processes. Besides, E-2-heptenal was another important volatile oxidation product and could be used as an indicator for thermal and photo-oxidation. On the other hand, the characteristic volatile compounds that were transferred from essential oils exhibited stable behavior against the oxidation mechanism which was a result of their antioxidant properties. The outcomes of this study are thought to be useful for the improvements in the gourmet oil industry as well as further research in this field.

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## Compliance with Ethical Standards

**Conflict of Interest** Sunduz Sezer Kiralan declares that she has no conflict of interest. Sermin Goksu Karagoz declares that she has no conflict of interest. Gulcan Ozkan declares that she has no conflict of interest. Mustafa Kiralan declares that he has no conflict of interest. Onur Ketenoglu declares that he has no conflict of interest.

**Ethical Approval** Not applicable.

**Informed Consent** Informed consent not applicable.

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