Changes in volatile compounds of Ayvalık (Edremit) and Uslu olive oils depending on conditions and time of storage

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Abstract

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Introduction. Volatile aromatic compounds present in olive oils extracted from Turkish olive cultivars including Edremit (Ayvalık) and Uslu were determined qualitatively.

Materials and methods. The olives were harvested from Akhisar/Manisa region, which is one of most important Turkish olive-growing locations, at almost the same maturity stage by hand. Harvested olives were put in case and nylon sacks and were stored under the same conditions until they analyzed. Determination of the volatile aroma compounds were done with the aim of Headspace Solid-Phase Microextraction (HS-SPME) and Gas Chromatography-Mass Spectrometry (GC-MS).

Results and discussion. 46 different volatile compounds were identified. The inappropriate storage conditions of olives had a negative impact on the aroma profiles of oils. The most abundant compounds were hexanal, α -farnesene, dimethylpalmitamine, and α -Farnesene, 2-hexanal, hexanal in olive oils extracted from Edremit (Ayvalık) and Uslu varieties, respectively. 1-Hexanol was mostly increased compound in Edremit olive oils extracted from olives stored at nylon sacks during 14 days. The increase of concentration of 2-hexanal during holding periods could be explained by the activity of the fungal enzymes in Lipoxigenase pathway of olive fruits. While 5-Hepten-2-one 6-methyl and 1-Butanol 3-methyl were not detected in Edremit and Uslu olive oils at initial day, they formed during both sack and box holding due to the microbial activity in olives. Three principal components (PCs) were extracted representing 81.27% of the total variance of olive oil samples extracted from Uslu cultivar and 80.14 % of the total variance of olive oil samples extracted from Edremit cultivar. The first PCs, PC1, PC2 and PC3 represented 45.15 and 41.31%, 21.90 and 21.39%, 14.21 and 17.43%, for Uslu and Edremit varieties, respectively.

Conclusions. It is recommended to store olives at 5 °C in air conditioning boxes for at least 30 days to reduce fungal development and to maintain the desired aroma.

Introduction

Olive fruit (*Olea europaea*) is one of most important products in Mediterranean countries and olive cultivation and processing has been carried out since the beginning of human civilization (Ozdemir et al. 2018). Olive and olive oil have superior nutritional properties due to high content of volatile and phenolic compounds, so their regular consumption could help in prevention of such diseases such as cardiovascular diseases, cancer and osteoporosis. Production of a high quality virgin olive oil requires the storing the olive fruit in proper holding conditions (Inarejos-García et al. 2009). In addition to the extraction process and storage, cultivar and cultivar applications, geographical origin, climatic conditions, degree of fruit ripening also affect the content and composition of volatile compounds in olive oils (Issaoui et al. 2010; Kesen et al. 2013). Several researchers have determined that the olive storage types and times affect the virgin olive oil quality as much as technological operations (Bouaziz et al. 2008; Inarejos-García et al. 2009; Pereira et al. 2002; Rizzo et al. 2011).

The presence of volatile and phenolic compounds directly influence and determine the quality of virgin olive oil. Volatile aromatic compounds are one of the most important factors for olive oil's quality and affect sensorial perception (Bayrak & Hu 2013). They are formed by the oxidation of oils with certain enzymes such as lipoxygenase (LOX pathway) (Cavalli et al. 2004). Olive oils possess more than 180 different aromas, and the majority of the volatile substances are presented by esters, aldehydes, hydrocarbons, ketones, and furans (Kesen et al. 2013). Servili et al (2003) studied the time of revealing of olive pastes to air during malaxation and found a positive correlation between air contact and the content of volatile compounds in olive oil including hexanal, 1-butanol, (Z)-3-hexen-1-ol, 1-penten-3-ol, and 2-methyl-1-butanol. Inappropriate olive fruit storage generally induce the activity of microorganisms that are responsible for unpleasant odours and formation of volatile compounds (Koprivnjak & Conte 2002).

Principal component analysis (PCA) is an statistical method that can be used to determine the content of triglycerides, sterols, phenolic compounds and volatile compounds to distinguish oils from different cultivars (Boskou 2007; de Fernandez et al. 2014; Yang et al. 2017). The aim of this study was to examine the effects of different holding times and types on volatile aroma compounds of oil samples obtained from different olive cultivars named "Edremit and Uslu" collected from Aegean province, Akhisar Region of Turkey by Gas Chromatography– Mass Spectroscopy (GC–MS) headspace technique. There is no detailed information that compares the volatile profiles of oils extracted from olives holded in unsuitable conditions that is already applied by some olive producers. For this reason, this study carried out is of importance.

Materials and methods

Materials

Olive fruits (*Olea europaea* L.) from the Edremit and Uslu varieties grown in the Akhisar area were harvested in the 2012/2013 crop season. Maturity index of olives, which was calculated according to the method of the International Olive Oil Council (IOOC, 2011), was 3.75 ± 0.35 and 4.50 ± 0.71 for Edremit and Uslu cultivars, respectively. According to this method, 100 fruits were randomly taken to assess their level of maturity by a subjective evaluation of the color of the olive skin and flesh. The olives were distributed into eight

groups according to the following characteristics: bright-green skin (group N 0), greenish-yellowish skin (group N 1), green skin with reddish spots (group N 2), reddish-brown skin (group N 3), black skin with white flesh (group N 4), black skin with < 50% purple flesh (group N 5), black skin with > 50% and < 100% purple flesh (group N 6), and black skin with 100% purple flesh (group N 7). Maturation indexes ranged from zero (intense green skin) to seven (black skin and 100% purple flesh). The maturity index was calculated by $\sum (N_i n_i) / 100$, where N is the group number and n is the olive amount in that group (Aydar et al. 2017). Air temperature and weather conditions during olives holded periods were shown in Table 1 (Turkish State Meteorological Service, 2012-2013). Olives were put into nylon sacks (60x90 cm) and plastic boxes (53x37x31 cm) and were hold inside them for 0, 7, 14 and 21 days. Olive oil samples symbolized as E: Edremit variety, U: Uslu variety, K: Holded in plastic boxes, and C: Holded in nylon sacks.

Table 1

**Air temperature and weather conditions during olives holded periods
(Turkish State Meteorological Service, 2012-2013)**

MANISA/AKHISAR	Air temperature (°C)	Weather
21.12.2012 (harvest-first extraction)	7	Snowy
1th day	8	Sunny
2th day	8	Sunny
3th day	7	Sunny
4th day	12	Sunny
5th day	16	Sunny
6th day	15	Partly cloudy
28.12.2012 (7th day-second extraction)	14	Stormy
8th day	13	Stormy
9th day	12	Stormy
10th day	13	Stormy
11th day	13	Some clouds
12th day	13	Some clouds
13th day	13	Some clouds
04.01.2013 (14th day-third extraction)	10	Foggy
15th day	11	Some clouds
16th day	10	Some clouds
17th day	6	Partly cloudy
18th day	6	Partly cloudy
19th day	3	Partly cloudy
20th day	5	Partly cloudy
11.01.2013 (21th day-fourth extraction)	8	Stormy

Methods

Oil extraction

A laboratory scale Abencor extraction system consisted of a small-quantity mill (MC2 Ingenieria Sistemas, Seville, Spain) equipped with a mixer (Kitchen Aid Mixer 4lt Model 5KSM45 220-240VN 50-60 HZ 250W, USA), a basket centrifuge (Marelli Motore Asincrono Trifase Tipo NR90S2) and a metal crusher was used to extract oil from olive fruits. The malaxation process was performed at 35 ± 1 °C for 60 min for Edremit variety and at 35 ± 1 °C for 90 min for Uslu variety, and the oil separation was carried out by decanter. Extracted olive oils were filtered and then kept at 4°C in amber glass bottles until analysis.

Volatile compound analysis

Sample Preparation. An 8 mL of oil was put in a 20 mL glass headspace sample vial and to attain a final 3 ppm concentration, 27 μ L and 24 μ L of internal standard solutions (butyl acetate and 1-nonanol, respectively) were added to each vial. The mixture was shaken carefully and allowed to equilibrate for 1 hour in the dark at ambient temperature before the analysis.

HS-SPME procedures. The SPME coated by polydimethylsiloxane (PDMS) fiber at 100 μ m thickness and 23 gauge was used in this study. It was purchased from Supelco and thermal conditions provided according to the manufacturer's recommendations before first use. The samples of oils were heated to 40 °C for 10 min before revealing the SPME fiber to the headspace of the sample. Headspace sampling/extraction took 30 min with continuous stirring (250 rpm). The samples were analyzed in duplicate and as a blank sample (empty glass vial) was used before and after each analysis.

GC-MS analysis. The analytical system was constituted from A Gerstel MPS autosampler (Gerstel, Baltimore, MD, USA) installed to an Agilent 6890 N model Gas Chromatography (Little Falls, DE, USA) paired with an Agilent 5975 mass selective detector. The software was MSD ChemStation (Agilent). SPME injections were carried on a splitless mode using a SPME injection sleeve (0.75 mm I.D) at 250 °C for 350 sec meanwhile thermal desorption of analyses was occurred in DB-Wax column (60m $\times$ 0.25mmI.D., 0.25 μ m film thickness) (J&W Scientific, Folsom, CA, USA). Gas helium was used as a carrier with a total flow of 1.2 mL $\cdot$ min⁻¹. The initial temperature 40 °C was kept for 1.0 min, followed by an increased to 200 °C at a rate of 6 °C min⁻¹ and kept at this temperature for 5 min, then raised to 250 °C at a rate of 8 °C min⁻¹. Lastly, the the temperature was retained 250 °C for 10 min. The total cycle time was 48.92 min. The MS detector was handled in scan mode (mass range 30–500) and the transfer line to the MS system was retained at 250 °C. The identification of the compounds was carried out by comparing (i) the Kovats indices (KI) based on a homologous series of even numbered n-alkanes (C₈–C₂₀), with those of standard compounds and by comparison with the data of literature, and (ii) by MS data received from NIST library (NIST/EPA/NIH Mass Spectral Library with Search Program, data version NIST 05, software version 2.0d).

Statistical analysis

XLSTAT (Addinsoft SARL, NY, USA) for Microsoft Excel (Microsoft, Redwood, WA) was used to perform ANOVA. In order to identify the variations of headspace components and analyze the composition data in different samples of oil, principal component analysis (PCA) was performed by SCAN software (Minitab Inc., State College, PA, USA). The software autoscaled the content values before the statistical analysis, i.e. each variable was subtracted by the mean value and the result was divided by its standard deviation.

Results and discussion

Principal volatile compounds are commonly found in great sensory quality virgin olive oil that are synthesized by biogenic pathways of the olive fruit such as fatty acid or amino acid metabolism and LOX pathway (Morales 2005). In spite of that, especially storing types and conditions, climatic conditions are very effective on producing of disagreeable volatiles compounds (Gomes da Silva et al. 2012). In olive fruits stored in batches, under high humidity conditions, the most abundant deuteromycetes such as several species of genus *Aspergillus*, together with ascomycetes, *Penicillium notatum* are occurred. These microorganisms have the capacity to oxidise free fatty acids and produce volatile compounds such as methyl ketones (Morales 2005).

In this study, 46 different volatile compounds were identified during different holding times of olive oils extracted from Edremit and Uslu varieties (Table 2, 3 and 4).

Table 2*
Volatile compounds isolated from Edremit (Ayvalık) and Uslu oils

Codes	Compounds	tr (min)	KI _E	KI _R	Odor
A1	Hexanal (44)	9.31	1103.5	1108	Fatty; fruity; green
A2	1-Propanol 2-methyl (43)	9.31	1103.0	1103	Sweet; musty odor
A3	Pyridine (79)	11.62	1205.5	1193	Sour; fishy
A4	1-Butanol 2-methyl (57)	11.99	1221.5	1220	Natural
A5	1-Butanol 3-methyl (70)	12.00	1222.0	1224	Fusel; alcohol; sweet; fruity
A6	Limonene (68)	12.00	1222.0	1221.5	Herbaceous; minty
A7	2-Hexenal (E) (41)	12.50	1243.6	1238	Green type flavor
A8	Cyclodecane methyl (55)	12.82	1257.4	1260	
A9	2-Butanone 3-hydroxy (45)	14.10	1313.1	1314	Butter; creamy
A10	Tridecane (57)	14.15	1315.3	1300	Floral
A11	5-Hepten-2-one 6-methyl (43)	15.20	1361.8	1361	Oily; herbaceous; green
A12	1-Hexanol (56)	15.37	1369.3	1362	Herbal

Table 2* (continue)					
Codes	Compounds	tr (min)	KI _E	KI _R	Odor
A13	3-Hexen-1-ol (Z) (41)	16.14	1403.5	1406	Green
A14	Nonanal (57)	16.50	1420.1	1422	Apple; coconut; grape; grapefruit; lemon
A15	2-Hexen-1-ol (E) (57)	16.60	1424.8	1420	Apple; banana; orange; green; wine-like;
A16	Acetic acid (45)	17.74	1477.4	1477	Pungent; sour; vinegar-like odor
A17	Cycloisosalivene (147)	18.43	1509.8	1522	
A18	n.i. (119)	19.61	1566.9	n.d.	
A19	Dimethyl sulfoxide (63)	20.38	1604.3	1603	Butter; alliaceous (onion; garlic)
A20	Alpha-Bergamotene (119)	20.67	1619.1	1609	Woody
A21	Decanoic acid methyl ester (74)	20.74	1622.7	1624	Oily; fruity; wine- like
A22	Cycloheptanone 2-methylene (43)	20.98	1634.9	n.d.	
A23	β -Farnesene (69)	21.85	1679.2	1674	Apple; lavender; lime; green; woody;
A24	8-Heptadecene (69)	23.00	1739.6	1718	
A25	Eremophilene (161)	23.31	1756.2	1744	
A26	α -Farnesene (93)	23.75	1779.7	1778	Woody
A27	β -Sesquiphellandrene (69)	24.30	1809.6	1782	Herbal
A28	Dodecanoic acid methyl ester (74)	24.73	1833.7	1834	Coconut; creamy; soapy; waxy
A29	Hexanoic acid (60)	25.50	1876.9	1874	Cheese; fatty; sour
A30	Phenylethyl alcohol (91)	26.85	1955.1	1946	Honey; floral; rose
A31	2,6-Bis(1,1-dimethylethyl)-4- (1-oxopropyl)phenol (233)	26.90	1958.0	n.d.	
A32	4,6-Heptadienoic acid, 3,3,6- trimethyl methyl ester (109)	27.12	1971.0	n.d.	
A33	n.i. (159)	27.29	1981.0	n.d.	
A34	3-Buten-2-ol, 2-methyl (71)	27.49	1992.7	n.d.	Herbal
A35	1-Dodecanol (55)	27.53	1995.1	1984	Coconut; honey; soapy; waxy; earthy; fatty
A36	Dimethylpalmitamine (58)	28.03	2017.4	n.d.	
A37	Methyl tetradecanoate (74)	28.37	2031.7	2032	Honey; fatty

Table 2* (continue)					
Codes	Compounds	tr (min)	KI _E	KI _R	Odor
A38	Phenol (94)	28.45	2035.0	2035	Sweet; tarry odor
A39	Nerolidol 2 (69)	28.86	2052.2	2052	Apple; green; woody; citrus; rose
A40	2(3H)-Furanone dihydro-5-propyl (γ -lactone (85)	29.10	2062.3	2068	Waxy; creamy; coconut character;
A41	Benzoic acid 2-methoxy. methyl ester (135)	29.69	2087.0	2088	
A42	2H-Pyran-2-one. 6-hexyltetrahydro (99)	32.70	2213.2	2215	Fruity; sweet
A43	Hexadecanoic acid methyl ester (74)	32.75	2215.3	2214	Waxy
A44	n.i. (71)	33.80	2259.3	n.d.	
A45	9-Octadecenoic acid methyl ester (oleic acid) (55)	37.51	2414.8	2424	Fatty
A46	p-Isopropenylphenol (134)	37.76	2425.2	n.d.	

*KI_E. experiment value of Kovats index (KI);

*KI_R. reference value of KI.

*The odor descriptors were obtained from SAFC "Flavors and Fragrances. European Ed. Catalogue 2009".

Table 3*

Normalized peak area of volatile compounds isolated from Edremit (Ayvalık) oils in two different packaging at initial and after 7, 14 and 21 days of ripening

Codes	Compounds (m/z)	Normalised peak area (mean±SD. n=2)						
		E ₀	E _C			E _K		
			E _{7C}	E _{14C}	E _{21C}	E _{7K}	E _{14K}	E _{21K}
A1	Hexanal (44)	(251±11) ^a	(29.74±0.14) ^b	n.d. ^c	n.d. ^c	(9±2) ^c	n.d. ^c	n.d. ^c
A2	1-Propanol 2-methyl (43)	n.d. ^b	n.d. ^b	(9±2) ^a	(10.6±0.3) ^a	n.d. ^b	n.d. ^b	n.d. ^b
A3	Pyridine (79)	n.d. ^b	(32.7±0.4) ^a	n.d. ^b	n.d. ^b	n.d. ^b	n.d. ^b	n.d. ^b
A4	1-Butanol 2-methyl (57)	n.d. ^c	(1.7±0.2) ^{bc}	(4.0±1.0) ^a	(5.0±0.7) ^a	(1.32±0.10) ^{bc}	(1.43±0.04) ^{bc}	(1.9±0.2) ^b
A5	1-Butanol 3-methyl (70)	n.d. ^e	(1.8±0.3) ^{cd}	(4.0±0.2) ^b	(5.59±0.04) ^a	(1.24±0.08) ^d	(1.73±0.11) ^{cd}	(2.06±0.09) ^c
A6	Limonene (68)	(2.70±0.06) ^{bc}	(40.2±0.4) ^a	(1.95±0.12) ^{cd}	(0.80±0.07) ^e	(2.8±0.2) ^b	n.d. ^f	(1.9±0.2) ^d
A7	2-Hexenal (E) (41)	(14.0±1.3) ^a	(0.43±0.09) ^b	(0.82±0.03) ^b	n.d. ^b	n.d. ^b	n.d. ^b	n.d. ^b
A8	Cyclodecane methyl (55)	(12.25±0.12) ^d	(17.66±0.07) ^c	(22.8±0.6) ^a	(21.6±1.3) ^{ab}	(19.3±0.14) ^{bc}	(9.6±0.5) ^e	(17.4±0.8) ^d
A9	2-Butanone 3-hydroxy (45)	n.d. ^b	n.d. ^b	(5.22±0.03) ^a	(5.85±0.13) ^a	n.d. ^b	(1.7±0.5) ^b	(1.8±1.1) ^b
A10	Tridecane (57)	(2.20±0.04) ^a	(2.0±0.2) ^a	(1.9±0.4) ^a	(2.9±0.6) ^a	(2.51±0.11) ^a	(1.9±1.0) ^a	(1.7±0.5) ^a
A11	5-Hepten-2-one. 6-methyl (43)	n.d. ^b	(2.0±0.3) ^{ab}	(1.2±0.3) ^{ab}	(2.6±0.4) ^a	(0.42±0.11) ^b	(0.48±0.02) ^b	(1.19±0.02) ^{ab}
A12	1-Hexanol (56)	(33±2) ^f	(236±9) ^d	(503±11) ^a	(268±3) ^c	(356±4) ^b	(119±13) ^e	(144.3±0.2) ^e
A13	3-Hexen-1-ol (Z) (41)	(13.87±0.11) ^e	(52±2) ^b	(60.5±1.4) ^a	(51.9±0.8) ^b	(49.4±0.9) ^{bc}	(30±3) ^d	(44.1±0.4) ^c
A14	Nonanal (57)	(8.7±1.5) ^b	(3.0±0.4) ^{bc}	(54.9±1.4) ^a	(3.9±0.4) ^{bc}	(4.32±2.03) ^{bc}	(1.6±0.8) ^c	n.d. ^c
A15	2-Hexen-1-ol (E) (57)	n.d. ^d	(19.3±0.5) ^c	(53.6±0.5) ^a	n.d. ^d	(35±3) ^b	n.d. ^d	(18.2±0.7) ^c
A16	Acetic acid (45)	(14.1±0.2) ^a	(10.5±1.1) ^a	(11±2) ^a	(12±2) ^a	(8±2) ^a	(7±6) ^a	(7.5±0.7) ^a
A17	Cycloisotativene (147)	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a
A18	n.i. (119)	(3.0±0.3) ^a	(2.0±0.7) ^a	(4.41±0.12) ^a	(4.5±0.2) ^a	(6.2±1.5) ^a	(4.2±0.2) ^a	(5.8±0.2) ^a
A19	Dimethyl sulfoxide (63)	(3.0±0.2) ^a	(0.8±0.3) ^b	(1.18±0.01) ^b	(3.7±0.7) ^a	(0.5±0.2) ^b	(0.75±0.14) ^b	(0.74±0.04) ^b
A20	α-Bergamotene (119)	(1.99±0.01) ^b	(2.3±0.3) ^b	(2.32±0.01) ^b	(2.25±0.12) ^b	(4.2±0.9) ^a	(1.63±0.05) ^b	(3.0±0.2) ^{ab}
A21	Decanoic acid methyl ester (74)	n.d. ^b	n.d. ^b	n.d. ^b	(31.5±0.9) ^a	n.d. ^b	n.d. ^b	n.d. ^b
A22	Cycloheptanone 2-methylene (43)	n.d. ^c	n.d. ^c	(3.1±0.4) ^b	(5.1±0.2) ^a	n.d. ^c	n.d. ^c	n.d. ^c

Table3* (continue)

Codes	Compounds (m/z)	Normalised peak area (mean±SD. n=2)						
			E _C			E _K		
		E ₀	E _{7C}	E _{14C}	E _{21C}	E _{7K}	E _{14K}	E _{21K}
A23	(E)-β-Fanese (69)	(9.1±0.3) ^b	(10.2±0.5) ^b	(11.2±0.3) ^b	(12.1±1.0) ^{ab}	(19±5) ^a	(7.4±0.2) ^b	(14.0±0.4) ^{ab}
A24	8-Heptadecene (69)	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a
A25	Eremophilene (161)	(0.79±0.03) ^d	(2.62±0.03) ^{bc}	(5.3±0.4) ^a	(5.0±0.2) ^a	(3.7±1.0) ^{abc}	(2.02±0.07) ^{cd}	(3.8±0.2) ^{ab}
A26	α-Farnesene (93)	(150±20) ^a	(88±2) ^{bcd}	(136±8) ^{ab}	(120±4) ^{abc}	(110±40) ^{abc}	(41±2) ^d	(62.8±0.2) ^{cd}
A27	β-Sesquiphellandrene (69)	(8.7±1.0) ^{ab}	(8.3±0.6) ^{ab}	(7.3±0.6) ^{ab}	(8.20±0.14) ^{ab}	(14±5) ^a	(4.5±0.2) ^b	(8.3±0.4) ^{ab}
A28	Dodecanoic acid methyl ester (74)	n.d. _b	n.d. _b	n.d.	(27.0±0.3) ^a	n.d. _b	(0.8±0.3) ^b	(0.43±0.2) ^{ob}
A29	Hexanoic acid (60)	(17±6) ^a	(10±2) ^{ab}	(16.9±1.3) ^a	(20.9±0.2) ^a	(4±2) ^b	(1.7±1.0) ^a	(3±2) ^b
A30	Phenylethyl alcohol (91)	(5±3) ^c	(12±5) ^{abc}	(18±5) ^{ab}	(19.6±0.7) ^a	(9.4±0.3) ^{abc}	(4.9±0.8) ^c	(7.1±0.4) ^{bc}
A31	2.6-Bis(1.1-dimethylethyl)-4-(1-oxopropyl) phenol (233)	(15±7) ^a	(9.32±0.08) ^{ab}	(10.4±0.5) ^{ab}	(10.7±0.8) ^{ab}	(9.74±0.05) ^{ab}	(0.9±0.2) ^b	(10.1±0.5) ^{ab}
A32	4.6-Heptadienoic acid 3.3.6-trimethyl. methyl ester (109)	n.d. ^a	(2.1±1.1) ^a	(20±15) ^a	(1.4±0.6) ^a	(7±5) ^a	n.d. ^a	(1.6±0.9) ^a
A33	n.i. (159)	n.d. ^c	(0.96±0.05) ^{bc}	(1.2±0.4) ^{ab}	(1.65±0.14) ^a	n.d. ^c	n.d. ^c	n.d. ^c
A34	3-Buten-2-ol 2-methyl (71)	n.d. ^b	(6±5) ^{ab}	(11±7) ^{ab}	(3.1±0.5) ^{ab}	(13.1±0.9) ^a	(1.43±0.07) ^{ab}	(2.8±0.3) ^{ab}
A35	1-Dodecanol (55)	(8±5) ^a	n.d. ^a	(1.5±0.3) ^a	n.d. ^a	(10±4) ^a	n.d. ^a	(0.8±0.5) ^a
A36	Dimethylpalmitamine (58)	(80±70) ^a	n.d. ^a	n.d. ^a	n.d. ^a	(5±4) ^a	n.d. ^a	n.d. ^a
A37	Methyl tetradecanoate (74)	n.d. ^b	n.d. ^b	n.d. ^b	(27±2) ^a	n.d. ^b	n.d. ^b	n.d. ^b
A38	Phenol (94)	(2.8±0.9) ^a	(2.3±0.2) ^a	(2.66±0.10) ^a	(3.44±0.09) ^a	(2.4±0.2) ^a	(3.5±0.4) ^a	(2.21±0.02) ^a
A39	Nerolidol 2 (69)	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a
A40	2(3H)-Furanone. dihydro-5-propyl (85)	(3.1±0.8) ^b	(4.0±1.2) ^b	(7.0±0.5) ^a	(8.65±0.05) ^a	(4.14±0.13) ^b	(2.8±0.3) ^b	(3.2±0.3) ^b
A41	Benzoic acid. 2-methoxy. methyl ester (135)	(5±2) ^a	(1.9±0.6) ^{ab}	(2.10±0.09) ^{ab}	(1.3±0.4) ^{ab}	(2.5±0.3) ^{ab}	n.d. ^b	(0.96±0.05) ^{ab}

Table 3* (continue)								
Codes	Compounds (m/z)	Normalised peak area (mean±SD. n=2)						
		E ₀	E _C			E _K		
			E _{7C}	E _{14C}	E _{21C}	E _{7K}	E _{14K}	E _{21K}
A42	2H-Pyran-2-one, 6-hexyltetrahydro (99)	(4±2) ^a	(5±3) ^a	(3±2) ^a	(2.0±0.2) ^a	(2.0±0.7) ^a	n.d. ^a	n.d. ^a
A43	Hexadecanoic acid methyl ester (74)	(9±4) ^b	(5±4) ^b	(12±2) ^b	(33±5) ^a	(5±4) ^b	(5.0±0.3) ^b	(6±2) ^b
A44	n.i. (71)	(1.3±0.4) ^a	(12±10) ^a	(30±20) ^a	(14.9±1.3) ^a	(5±3) ^a	n.d. ^a	(2.2±0.2) ^a
A45	9-Octadecenoic acid methyl ester (55)	(5.77±0.08) ^b	(4±3) ^b	(18.0±0.3) ^a	(15±5) ^{ab}	(6±7) ^b	(4.5±0.7) ^b	(8±2) ^b
A46	<i>p</i> -Isopropenylphenol (134)	(1.7±0.4) ^b	(1.6±0.8) ^b	(2.2±0.8) ^b	(1.8±0.3) ^b	(2.4±0.3) ^b	(11±3) ^a	(2.40±0.09) ^b

* Peak areas are normalised respect to internal standard

¹n.d. not detected.

^{a-f} Values in the same row with different superscript letters differ significantly ($p < 0.05$).

Data showed mean of two independent tests (mean ±sd).

Table 4*

Volatile compounds (AU x10⁴) isolated from Uslu oils in two different packaging at initial and after 7, 14 and 21 days of stages of ripening

Codes	Compounds (m/z)	Normalised peak area (mean±SD. n=2)						
				U _C		U _K		
		U ₀	U _{7C}	U _{14C}	U _{21C}	U _{7K}	U _{14K}	U _{21K}
A1	Hexanal (44)	(140.5±4.6) ^a	(51.4±0.2) ^b	n.d. ^c	n.d. ^c	(52.5±0.8) ^b	n.d. ^c	n.d. ^c
A2	1-Propanol 2-methyl (43)	n.d. ^c	n.d. ^c	(17.54±0.02) ^a	(18.1±0.2) ^a	n.d. ^c	(12.2±1.4) ^b	(13.8±0.08) ^b
A3	Pyridine (79)	n.d. ^b	n.d. ^b	n.d. ^b	n.d.	n.d. ^b	n.d. ^b	(7.1±0.3) ^a
A4	1-Butanol 2-methyl (57)	(1.9±0.05) ^c	(2.9±1.2) ^{bc}	(11.69±0.14) ^a	(5.0±0.7) ^b	(3.1±0.2) ^{bc}	(1.23±0.08) ^c	(2.12±0.07) ^{bc}
A5	1-Butanol 3-methyl (70)	(2.09±0.04) ^d	(3.5±0.4) ^d	(12.8±0.6) ^a	(5.5±0.3) ^c	(3.2±0.2) ^d	(9.3±0.6) ^b	(2.21±0.02) ^d
A6	Limonene (68)	(0.82±0.01) ^b	(2.42±0.06) ^a	(1.02±0.02) ^b	(1.14±0.04) ^b	(3.00±0.02) ^a	(1.10±0.14) ^b	(1.83±0.02) ^{ab}
A7	2-Hexenal (E) (41)	(146±3) ^a	(28.3±0.9) ^b	(2.0±1.1) ^c	(1.00±0.00) ^c	(37±6) ^b	n.d. ^c	n.d. ^c
A8	Cyclodecane methyl (55)	(16.3±0.3) ^a	(15.1±0.4) ^a	(10.34±0.14) ^c	(10.9±1.1) ^c	(11.2±0.2) ^{bc}	(13.1±0.3) ^b	(11.3±0.2) ^{bc}
A9	2-Butanone 3-hydroxy (45)	(4.02±0.11) ^d	(9.6±0.5) ^{cd}	(12.1±0.2) ^{bcc}	(17.37±0.08) ^b	(9.0±0.7) ^{cd}	(39±4) ^a	(8.6±0.4) ^{cd}
A10	Tridecane (57)	(3.53±0.01) ^a	(4±2) ^a	(2.44±0.08) ^a	(2.3±0.3) ^a	(2.02±0.08) ^a	(3.5±0.8) ^a	(2.32±0.04) ^a
A11	5-Hepten-2-one. 6-methyl (43)	(1.64±0.04) ^{de}	(1.4±0.5) ^e	(3.0±0.3) ^c	(3.32±0.09) ^{bc}	(2.6±0.2) ^{cd}	(4.52±0.12) ^a	(4.02±0.06) ^{ab}
A12	1-Hexanol (56)	(46.8±0.9) ^d	(186±2) ^a	(91.9±1.2) ^c	(51.3±0.2) ^d	(114±2) ^b	(18.7±0.2) ^f	(34.3±0.3) ^e
A13	3-Hexen-1-ol (Z) (41)	(5.00±0.09) ^a	(4.3±0.3) ^{ab}	(2.5±0.8) ^{bcd}	(2.0±0.4) ^{cd}	(3.8±0.2) ^{abc}	n.d. ^e	(1.2±0.2) ^{de}
A14	Nonanal (57)	(6.2±0.2) ^a	(3±2) ^b	(2.8±0.6) ^b	(1.3±0.5) ^b	(1.65±0.12) ^b	(1.9±0.5) ^b	(1.75±0.09) ^b
A15	2-Hexen-1-ol (E) (57)	(30.8±0.2) ^a	(128±2) ^a	(45.1±0.8) ^b	(23.95±0.11) ^c	(71±3) ^a	(1.1±0.3) ^a	(22.6±0.8) ^a
A16	Acetic acid (45)	(21.8±0.5) ^a	(19±2) ^a	(11.6±0.8) ^a	(16.4±0.4) ^a	(13.3±0.9) ^a	(15.01±0.08) ^a	(13±8) ^a
A17	Cycloisotavene (147)	(1.27±0.04) ^a	(1.81±0.04) ^a	(2.66±0.05) ^a	(2.82±0.05) ^a	(1.66±0.14) ^a	(3.0±0.3) ^a	(2.8±0.2) ^a
A18	n.i. (119)	(2.32±0.07) ^a	(2.4±0.2) ^a	(1.49±0.08) ^b	(1.69±0.05) ^b	(1.66±0.01) ^b	(1.9±0.2) ^b	(1.7±0.1) ^b
A19	Dimethyl sulfoxide (63)	(13.3±0.3) ^b	(2.0±0.3) ^d	(14.7±0.2) ^a	(14.0±0.4) ^{ab}	(2.1±0.3) ^d	(2.2±0.5) ^d	(4.75±0.13) ^c
A20	α-Bergamotene (119)	(2.32±0.01) ^a	(2.35±0.03) ^a	(1.62±0.07) ^{bc}	(1.7±0.2) ^{bc}	(1.42±0.13) ^c	(1.96±0.01) ^{ab}	(1.7±0.2) ^{bc}

Table 4* (continue)

Codes	Compounds (m/z)	Normalised peak area (mean±SD. n=2)						
		U ₀	U _C			U _K		
			U _{7C}	U _{14C}	U _{21C}	U _{7K}	U _{14K}	U _{21K}
A21	Decanoic acid methyl ester (74)	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a
A22	Cycloheptanone 2-methylene (43)	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a
A23	(E)-β-Farnesene (69)	(6.70±0.18) ^{ab}	(7.1±0.7) ^a	(3.8±0.3) ^{cd}	(4.8±0.5) ^{bcd}	(3.27±0.14) ^d	(5.5±0.2) ^{abc}	(4.7±0.9) ^{cd}
A24	8-Heptadecene (69)	(7.64±0.10) ^a	(7±2) ^a	(2.7±0.4) ^b	(3.1±0.5) ^b	(2.7±0.3) ^b	(3.6±0.3) ^b	(3.7±0.6) ^b
A25	Eremophilene (161)	(10.7±0.4) ^a	(13.8±1.2) ^b	(12.3±0.4) ^{bc}	(11.9±1.0) ^{bc}	(9.6±1.0) ^c	(17.7±0.3) ^a	(11.0±1.3) ^{bc}
A26	α-Farnesene (93)	(614±2) ^a	(570±90) ^a	(219.70±0.05) ^b	(230±30) ^b	(200±20) ^b	(259±6) ^b	(210±20) ^b
A27	β-Sesquiphellandrene (69)	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a
A28	Dodecanoic acid. methyl ester (74)	(1.5±0.01) ^a	(0.7±0.2) ^{ab}	n.d. ^b	n.d. ^b	(0.91±0.13) ^{ab}	(1.11±0.13) ^a	n.d. ^b
A29	Hexanoic acid (60)	(20.4±0.4) ^a	(15.4±1.3) ^b	(8±2) ^{cd}	(3.4±0.7) ^{ef}	(9.9±0.5) ^c	(4.1±0.2) ^{de}	n.d. ^f
A30	Phenylethyl alcohol (91)	(6.20±0.12) ^b	(6.7±0.3) ^b	(80±30) ^a	(28.6±0.6) ^b	(6.9±0.2) ^b	(23.4±0.8) ^b	(18.2±0.8) ^b
A31	2,6-Bis(1,1-dimethylethyl)-4-(1-oxopropyl)phenol (233)	(9.70±0.13) ^{ab}	(10.6±0.7) ^a	(10.06±0.04) ^{ab}	(9.10±0.11) ^b	(9.4±0.4) ^{ab}	(10.3±0.5) ^{ab}	(8.95±0.070) ^b
A32	4,6-Heptadienoic acid 3,3,6-trimethyl. methyl ester (109)	n.d. ^b	(5±3) ^{ab}	(7.8±1.4) ^a	(1.4±0.6) ^b	n.d. ^b	(0.94±0.08) ^b	(1.4±0.5) ^b
A33	n.i. (159)	(7.72±0.13) ^a	(3±2) ^b	(4.54±0.10) ^{ab}	(2.4±0.5) ^{ab}	(1.4±0.6) ^b	(2±2) ^b	(2.3±0.2) ^{ab}
A34	3-Buten-2-ol. 2-methyl (71)	(3.31±0.10) ^a	n.d. ^b	n.d. ^b	n.d. ^b	n.d. ^b	n.d. ^b	n.d. ^b
A35	1-Dodecanol (55)	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a
A36	Dimethylpalmitate (58)	(8.2±0.2) ^a	(8.1±0.6) ^a	(16±5) ^a	(12±6) ^a	(4.0±0.4) ^a	(6.8±0.9) ^a	(7.3±0.7) ^a
A37	Methyl tetradecanoate (74)	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a	n.d. ^a
A38	Phenol (94)	(3.43±0.07) ^b	(2.2±0.4) ^c	(9±2) ^a	(6.18±0.09) ^b	(2.0±0.5) ^c	(3.43±0.08) ^{bc}	(3.08±0.09) ^{bc}
A39	Nerolidol 2 (69)	(54±2) ^a	(21±10) ^b	(15±2) ^{bc}	(3.3±0.8) ^c	(3.0±1.4) ^c	(7.0±0.2) ^{bc}	(2.59±0.04) ^c
A40	2(3H)-Furanone dihydro-5-propyl (85)	(8.07±0.12) ^b	(9±2) ^b	(25±9) ^a	(5.9±0.7) ^b	(4.6±0.3) ^b	(12.44±0.04) ^{ab}	(4.21±0.04) ^b

Table 4* (continue)

Codes	Compounds (m/z)	Normalised peak area (mean±SD. n=2)						
		U ₀	U _C			U _K		
			U _{7C}	U _{14C}	U _{21C}	U _{7K}	U _{14K}	U _{21K}
A41	Benzoic acid. 2-methoxy. methyl ester (135)	(18.0 ±0.5) ^a	(7.4±0.5) ^b	(3.7±1.3) ^c	(1.2±0.2) ^d	(2.79±0.04) ^{cd}	(2.73±0.02) ^{cd}	(1.7±0.2) ^{cd}
A42	2H-Pyran-2-one. 6-hexyltetrahydro (99)	(14.83 ±0.50) ^a	(11 ±5) ^{ab}	(10.8 ±0.8) ^{ab}	(1.8 ±0.2) ^c	(2.1 ±0.6) ^c	(5.5 ±0.3) ^{bc}	(1.3 ±0.2) ^c
A43	Hexadecanoic acid methyl ester (74)	(5.30 ±0.08) ^a	(4.6 ±0.8) ^a	(60 ±60) ^a	(2.8 ±0.4) ^a	(2.7 ±0.3) ^a	(3.23 ±0.10) ^a	(4.1 ±1.4) ^a
A44	n.i. (71)	n.d. ^b	(1.4 ±0.5) ^{ab}	(5.7 ±0.4) ^a	n.d. ^b	n.d. ^b	(7.6 ±1.2) ^a	n.d. ^b
A45	9-Octadecenoic acid methyl ester (55)	(4.63±0.14) ^a	(7±4) ^a	(15±6) ^a	(3.03±1.85) ^a	(3.8±1.0) ^a	(3.3±1.0) ^a	(6±2) ^a
A46	<i>p</i> -Isopropenylphenol (134)	(2.16±0.07) ^a	(2.5±0.8) ^a	(2.1±0.3) ^a	(1.90±0.05) ^a	(1.6±0.3) ^a	(2.14±0.10) ^a	(1.90±0.05) ^a

¹n.d. not detected.

Data showed mean of two independent tests (mean ±sd).

^{a-c} Values in the same row with different superscript letters differ significantly ($p < 0.05$).

The storage of olives in inappropriate conditions had a negative impact on the aroma profiles of oils. Uslu olive oil was characterized with high concentrations of terpene volatile compound such as α -Farnesene (614±2) and aldehydes as 2-hexenal (146±3) and hexanal (140.5±4.6). In contrast, Edremit olive oil had lower concentrations of α -Farnesene and 2-hexenal, which were determined as 150±20 and 14±1.3, respectively. The increase of concentration of 2-hexenal (E) during holding periods could be explained by the activity of the fungal enzymes in LOX pathway of olive fruits (Schnurer et al. 1999). However, the concentration of hexanal (251±11) in Edremit olive oil was immediately increased after extraction. In this study, from alcohol compounds 1-Hexanol and 3-Hexen-1-ol were found as the most potent volatiles of the Edremit oils, which concentration increased over holding time in both holding types. In Uslu olive oils, the concentration of 1-hexanol also increased significantly ($p < 0.05$) during the holding time as observed for Edremit oil. The concentration of 3-Hexen-1-ol in Uslu oil during holding time did not increase in contrast to Edremit oil. Lower values of hexanal, hexanol, cis-3-hexenol and higher values of methanol, ethanol and 2-methyl butanol (fusty) in samples from plastic bags were also observed by other researchers (Koprivnjak & Conte 2002). The presence of hexanol was in virgin olive oils also reported (Haddada et al. 2007; Krichene et al. 2010; Manai et al. 2008). However, Kaftan (2007) did not identify hexanol in Memecik olive oils of 2005 and 2006 harvesting seasons. These differences between the findings can be related to the activity of alcohol dehydrogenase enzyme. As seen from Table 3 and Table 4, phenylethyl alcohol was also detected and its content increased during holding periods in both type of olive oils. Its occurrence was due to the degradation of phenylalanine (Issaoui et al. 2009).

Previous studies focused on relationship between irradiation or weather conditions and content of volatile compounds in olive oil before harvesting. However, no studies determined the effect of weather conditions on the content of volatile compounds in olive oil after harvesting. Servili et al. (2007) found that the presence of several volatile compounds, such as hexanal, (E)-2-hexenal, and other lipoxygenase derivative products positively correlated with the irrigation rate. In our study hexanal was not detected between 7-14 days when weather was stormy and heavy rain in both two variety of olive oils, while dimethylpalmitamine was not detected in stormy weather only in Edremit variety olive oils. The content of 2-hexen-1-ol significantly increased in Edremit olive oils holded in nylon sacks between 7-14 days. However, it was not detected in oils holded in plastic boxes. Similarly, the content of cycloheptanone 2-methylene significantly increased in Edremit olive oils holded in nylon sacks between 14-21 days (cloudy weather), however it was not detected in Uslu and Edremit oils holded in plastic boxes. While decrease in α -Farnesene in Edremit olive oils holded in nylon sacks between 0-7 days (sunny weather) was significant, this decrease was not so great in Uslu olive oils holded in nylon sacks during the days of 0-7.

Hexanal, which is responsible for fatty, fruity, green odor perceptions and contributed to pleasant sensory perceptions, was formed due to activity of aldehyde-lyase enzyme (Table 2). Therefore, the content of hexanal has been correlated with the highest overall grading of virgin olive oil (Morales & Aparicio 1999). In this study, the concentration of hexanal decreased more in oils obtained from Edremit variety holded at cases compared to oils extracted from Edremit variety holded at nylon sacks during 7 days. It decreased to 29.74 ± 0.14 and to 9 ± 2 in nylon sacks and case, respectively. While the ratio of hexanal to nonanal was lower than 2, the oil was evaluated as rancid which detects the beginning of oxidation (Kesen et al. 2013). Therefore, according to the results of 14th and 21th days, it has seen that hexanal did not occur in the oil because of it is under the detectable limits. This indicated that hexanal/nonanal ratio was below 2. However, the decrease in hexanal content was not significantly different in oils obtained from Uslu variety olives which were holded for 7 days in sacks and cases ($p > 0.05$). Acetic acid which is responsible for pungent, sour, vinegar-like odor can be formed by microorganisms growing on or in the olives during storage and are undesirable for olive oils (Davis 2007). Because of the production of acetic acid, *Acetobacter* could develop and cause vinegary defect. Acetic acid concentration in this study was not effected significantly by holding time and holding type in both olive oils during 21 days of storage ($p > 0.05$). While 5-Hepten-2-one 6-methyl and 1-Butanol 3-methyl volatiles compounds were not detected in Edremit and Uslu olive oils at initial day, they formed during both sack and box holding due to the activity of the microorganisms in olives (Table 3 and Table 4). The occurrence of 5-Hepten-2-one 6-methyl can be explained by the presence of bacteria from the genus *Pseudomonas* which are capable to degradate terpenic alcohols (Morales 2005).

PCA was used to highlight the data and to determine the relationships between the content of volatile compounds and olive oil variety during different holding times and types (Figure 1–3).

Figure 1. PCA plots of volatile compounds of Uslu variety olive oils.

Figure 2. PCA plots of volatile compounds of Edremit variety olive oils

Figure 3. Score plots of two first components applied to volatile compounds of olive oils extracted from Uslu and Edremit cultivars stored at case and nylon sacks until 21 days

PCA permitted a reduction of volatile compounds found in olive oil to three principal components (with eigenvalues >1). PCA results showed that 5 components were found (eigenvalues >1) for both olive varieties (Figure 4), but eigenvalues of two components were lower than the calculated percentile 95% they were dropped and only the first 3 components which eigenvalues were greater than percentile 95% were retained. Yang et al. (2017) used PCA and AHC chemometric analysis to determine the sensitive oxidation marker compounds, including (E, E)-2,4-heptadienal marker, by lipid oxidation in the n-3 PUFA rich oils. They found that oxidized volatiles did not rise linearly during oxidation and volatile compounds of lipid oxidation were not clearly identifiable with PCA analysis (Yang et al. 2017).

Three PCs were extracted represent 81.27 % of the total variance of olive oil samples extracted from Uslu cultivar and 80.14 % of the total variance of olive oil samples extracted from Edremit cultivar. The first principal components PC1, PC2 and PC3 represented 45.15% and 41.31%, 21.90% and 21.39%, 14.21% and 17.43%, respectively for Uslu and Edremit varieties. First component was mainly defined by A11, A17 and A2 with positive correlation and second component mainly by A42 and A33 (with a high negative correlation) in olive oils obtained from Uslu varieties (Figure 1). On the other hand, first component was mainly defined by A22, A40 and A43 and second component mainly by A19, A1 and A7 with a high positive correlation in olive oils extracted from Edremit varieties (Figure 2).

Figure 4. Eigenvalues of volatile compounds of Uslu and Edremit olive oils obtained by PCA

Conclusions

Principal component analysis (PCA) was used to evaluate the effect of different types of olive packaging (plastic box and nylon socks) and different period of olive storage in these packaging on oil aroma profiles and to find the relationships between volatile compounds and olive oil varieties. Content of hexanal decreased significantly in oils obtained from Edremit variety during 7 days of holding in case. However, the holding type had no effect on the hexanal content in Uslu olive oils. PC1, PC2 and PC3 identified the 81.27 % of the total variance of olive oil samples extracted from Uslu cultivar and 80.14% of the total variance of olive oil samples extracted from Edremit cultivar. The content of acetic acid, which is an undesirable compound in virgin olive oils, was not changed significantly in both two oils depending on holding type or time.

To provide high quality of olive oils conditions for its storage should be controlled. The recommended storage conditions for olives is about 5 °C in air conditioning boxes for at least 30 days to reduce development of fungi and protect aroma profiles of obtained oils from changes.

It's needed more studies on the investigation of olive oils aroma profile for reveal the effects of harvesting time and method, olive varieties, climatic factors, storage conditions, and extraction techniques.

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