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Original Article

Volatile Compounds of New Promising Dried Apricot (*Prunus armeniaca* L.) Genotypes

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Abstract

Turkey has rich wild apricot populations and all Turkish apricot cultivars were previously selected among wild apricots. On this background for apricot breeding, six new late flowering dried apricot genotypes were taken under study, along with wide spread cv. 'Hacihaliloglu'; all genotypes were examined in terms of volatile compounds using Headspace-Solid Phase Micro Extraction - Mass Spectrometry (HSSPME/GC/MS) techniques. The most important volatiles of apricot genotypes were aldehydes, alcohols, esters, terpenes, ketones and acids. Among these compounds, ethanol, hexanal, 3-carene, squalene, acetic acid, tetradecaonic acid, pentadecaonic acid, octadecaonic acid, n- hexadecaonic acid and 1-hdroxy-2-propanone were present in all genotypes studied at certain levels. In general, total concentrations of aroma compounds were higher in some promising genotypes under study than within 'Hacihaliloglu' cultivar, except total alcohol compound (53.33%). Volatile compounds, particularly esters, were the major contributors to fruity, floral and pleasant fruit flavours. The highest esters' compound contents were detected in 'N95' (9.2%) and 'N57' (2.18%) genotypes, while 'Hacihaliloglu' had 1.61% ester compounds. Lacton (γ-decalactone) was a key aroma compound of apricot. γ-decalactone was detected ranging between 0.4-1.13% in all genotypes, except cv. 'Hacihaliloglu'. The hereby obtained results showed that the volatile composition depended largely upon the apricot genotypes, moment of harvest, growing conditions and cultural applications that may all affect fruit quality. These results represent valuable starting points for apricot breeding programs.

Keywords: GC/MS, Headspace, SPME, volatile compounds, *Prunus armeniaca*

Introduction

Turkey is a major producer of fresh and dry apricot in the world, with an annual production of 780,000 tons, followed by Iran, Uzbekistan, Algeria and Italy (FAO, 2013). As a leading producer, 85% of the total dried apricots are produced in Turkey. A significant portion of fresh and 90-95% of dried apricots of Turkey are produced in Malatya region (Ercisli, 2009). Most of the fresh apricot cultivars are grown in the Southern and the Western regions (Mediterranean, Aegean and Marmara regions) of Central Anatolia, while the dried cultivars are mainly grown in the Malatya regions (Gokbulut and Karabulut, 2012).

'Hacihaliloglu' cultivar, with 24-26% soluble solid content, is the main apricot cultivar for commercial drying and comprises approximately 70% of dried apricot production in Turkey (Yilmaz *et al*., 2012).

The significant factor for growing fresh and dried apricot in Turkey is late spring frost. Therefore, the aim of most breeding programs in Turkey and the other apricot producer countries are focused on developing new cultivars that have late flowering period (high chilling requirements) (Ercisli, 2009; Marek *et al*., 2013).

In fruit, flavour occurs due to the different ration mixture of a few different aromatic ingredients. Flavour is one of the most important aspects of fruit quality and helps for the determination of differences among varieties (Solís-Solís *et al*., 2007; Kaczmarska *et al*., 2015; Motalebipour *et al*., 2015). Aroma of variety takes uttermost importance for customers. Therefore aromatic components are important for customer to accept and prefer new varieties (Azodanlou *et al*., 2003). Up to now, the aroma compounds tests of apricot from different

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regions of the world have been carried out and studied by many researchers (Greger and Schieberle, 2007; Lo Bianco *et al*., 2010; Xi *et al*., 2016). Ethyl acetate, hexyl acetate, limonene, 6-methyl-5-hepten-2-one, menthone, E-hexen-2-al, linalool, b-ionone and c-decalactone have been determined as important aromatic components in apricot (Guichard and Souty, 1988; Takeoka *et al*., 1990; Guillot *et al*., 2006). Xi *et al*. (2016) reported that β-ionone, γ-decalactone, sucrose and citrate are the key characteristic flavour factors in apricot fruits, contributing to consumer acceptance.

The aim of the hereby research was to compare in terms of volatile compounds six new selected apricot genotypes, promising by the fact they all have especially late bud break (high chilling requirement) and high °Brix values that is a key factor for dried apricots; the new cultivars under study were compared with the well-known and intensely spread dried apricot cv. 'Hacihaliloglu'. For this reason the concentrations and types of volatiles of selected new apricot genotypes were analyzed by Headspace Solid Phase Micro Extraction, Gas Chromatography and Mass Spectrometry (HS-SPME/GC-MS), which is a rapid and reliable technique.

Materials and Methods

The study was conducted on six apricot genotypes which were selected from Nevşehir region, among rich apricot seedling population at an average altitude of 1,285 meter, during the 2014-2015 growing season. Cultivar 'Hacihaliloglu', the most spread Turkish dried cultivar was used as a control. Thus the selected genotypes were compared with 'Hacihaliloglu' cultivar in terms of volatile compounds. Fruits were picked at commercial maturation stages (date 27th of July) in 2015 and brought to laboratory in cold chain quickly.

Twenty (20) apricot fruits per cultivar were prepared for analysis. Apricots were washed with distilled water, the stone was removed and discarded, and the fruits were cut in small pieces. Fruit samples were extracted using 250 g of fruits in each replicate. The fruit flesh was homogenized in a food processor and 1 g of the homogenate was diluted with 1 ml of 5M calcium chloride saturated aqueous solution and immediately headspace sampling was conducted on 85 μm fused silica fibers coated with polydimethylsiloxane / divinylbenzene (CAR/PDMS) (Supelco).

Volatile compounds were analyzed by Headspace Solid Phase Micro Extraction Technique (HS-SPME) on an automatic HS-40 head space auto sampler (Perkin Almer GC with split splitless inlet MSD system). Extraction was done for 40 min at room temperature during the extraction in the headspace auto sampler. HP-Innovax (30 m × 0.25 mm × 0.25 μm) fused-silica capillary column was used. Helium (1 ml/min) was used as a carrier gas. The injector temperature was 220 °C, set for splitless injection. The oven conditions were set to 60 °C for 5 min and then the temperature was increased at a rate of 4 °C/min followed by increasing to 240 °C at the same rate. Thermal desorption was allowed for 1.5 min. The detector temperature was 100 °C.

The components were identified by comparison of mass spectra and retention time data with those of authentic samples and complemented with an identified by doing a NIST, Wiley, Flavour library search of the acquired mass spectral data.

Results and Discussion

In total, 56 volatile compounds were detected in the six new apricot genotypes and cv. 'Hacihaliloglu'. Among the detected compounds, there were 13 alcohols, 9 aldehydes, 6 esters, 7 terpenoids, 16 acids and 5 ketones (Table 1).

As can be seen from the Table 1, the highest concentration of total alcohol compound was found in cv. 'Hacihaliloglu' (53.23%). The total aroma concentration of cv. 'Hacihaliloglu' was lower than those obtained for the others genotypes under study. According to Solís-Solís *et al*. (2007), aroma of the apricots is not connected to the total concentration of volatile compounds, even though they impact on volatile fraction. Takeoka *et al*. (1990) reported that the some volatile compounds, even if in low concentration level, have greater impact on apricot aroma. Cv. 'Hacihaliloglu' has moderate level volatile concentration thus it is consumed as dried.

Alcohol and aldehydes of six carbon atoms are responsible for the herbaceous odour of some fruits (Gómez and Ledbetter, 1997). Thirteen alcohol compounds were found in apricots and among them only the ethanol was detected in all genotypes. 'Hacihaliloglu' cultivar had higher total alcohol compound than those other selected apricot genotypes under study. The lowest concentration was detected in 'N95' genotype, with 27.08%. The most abundant alcohol compound was detected as ethanol in all genotypes, ranging between 23.55-50.81%. Compared to previous obtained data, concentration of the ethanol was found to be considerably in high level range from 34.21-50.81% among 56 important aroma compounds (Greger and Schieberle, 2007).

Nine aldehyde compounds were detected in the current study, but only hexenal was detected in all genotypes, while benzaldehyde was detected only in 'N82' cultivar (0.03%). Moreover, 'N95' fruit samples gave the highest quantities of total aldehydes (40.79%) of all the genotypes tested. Among the seven genotypes and cultivar, aldehyde was found to be after alcohol compounds, the second major aroma compound with a range from 21.51% in 'N97' to 40.79% in 'N95' (Table 1). Genotype 'N93' (36.4%) and 'N95' (33.94%) had the highest concentration of hexanal. The cv. 'Hacihaliloglu' took third place with 31.4% value among all genotypes under study, in terms of hexanal content. The concentration of aldehydic compounds (hexanal concentrations) determined in the current study was remarkably different than previous reported values (Greger and Schieberle, 2007).

A total of six esters were found in the volatile fraction of the samples, whereas 'N95' (9.2%) and 'N57' (2.18%) cultivars had the highest concentration of esters. The most abundant acetic acid, hexyl ester, was detected in 'N95' (4.71%), while the lowest ester content was found in 'N93' (0.1%). The concentration of the esters was measured in relatively lower levels in previous studies on apricot fruits (Takeoka *et al*., 1990; Gokbulut and Karabulut, 2012). Gokbulut and Karabulut (2012) determined ester concentration as 1.4% in cv. 'Hacihaliloglu'. Ester concentration obtained in the current study for the well-known cultivar (1.61%) was in agreement with the data reported by Gokbulut and Karabulut (2012). The highest ester content was detected in 'N95' genotype. Volatile composition and content depended largely on genetic background.

Table 1. Volatile profiles of six new apricot genotypes and cultivar 'Hacihaliloglu' detected by HS-SPME/GC-MS (%)

Compound	R1	Apricot new genotypes under study and 'Hacihaliloglu' cultivar						
		'N97'	'HH'	'N95'	'N82'	'N57'	'N93'	'N91'
Alcohol								
Ethanol	1.54	34.21	49.88	23.5	48.95	42.83	47.37	50.81
2-Propanol	4.41	N.d.	N.d.	N.d.	0.37	N.d.	N.d.	N.d.
1-Nonanol	4.62	0.43	1.77	N.d.	N.d.	N.d.	N.d.	0.18
1-Butanol	4.78	0.37	N.d.	2.51	1.06	N.d.	0.97	0.67
1-Pentanol	5.56	N.d.	0.15	N.d.	N.d.	N.d.	0.11	N.d.
2-Heptanol	7.26	N.d.	0.1	N.d.	N.d.	N.d.	N.d.	N.d.
1-Hexanol	8.49	2.79	1.17	N.d.	0.08	1.03	0.25	0.67
2-Hexen-1-ol	9.77	2.41	N.d.	0.94	N.d.	5.17	0.18	N.d.
3-Hepten-1-ol.(E)	9.96	N.d.	0.16	N.d.	N.d.	N.d.	N.d.	N.d.
1-Hexanol.2-ethyl	11.57	N.d.	N.d.	0.05	N.d.	N.d.	N.d.	0.17
Terpineol	15.85	0.34	N.d.	0.08	0.27	N.d.	N.d.	N.d.
Phyogallol	25.04	0.14	N.d.	N.d.	N.d.	N.d.	N.d.	N.d.
Phytol	13.01	0.34	N.d.	N.d.	N.d.	N.d.	0.34	N.d.
Total Alcohol		**41.03**	**53.23**	**27.08**	**50.73**	**49.03**	**49.22**	**52.5**
Aldehyde								
Hexanal	2.40	20.2	31.49	33.94	17.38	25.81	36.4	27.57
2-Hexenal	4.58	0.54	N.d.	N.d.	N.d.	1.11	N.d.	N.d.
Acetaldehyde	5.59	N.d.	N.d.	N.d.	1.79	N.d.	N.d.	N.d.
3-Hexen-1-ol.acetate.(Z)-	7.02	N.d.	N.d.	2.49	N.d.	N.d.	N.d.	N.d.
Nonanal	8.82	0.19	N.d.	N.d.	N.d.	N.d.	N.d.	0.44
2-Octenal	9.85	N.d.	0.27	4.36	N.d.	N.d.	0.27	0.5
Furfural	11.00	0.3	N.d.	N.d.	1.95	N.d.	0.11	0.25
benzaldehyde	12.14	N.d.	N.d.	N.d.	0.03	N.d.	0.08	N.d.
2-Furancarboxaldehyde	35.97	0.28	0.35	N.d.	1.97	0.8	0.69	1.43
Total Aldehyde		**21.51**	**32.11**	**40.79**	**23.12**	**27.72**	**37.55**	**30.19**
Esters								
Acetic acid.butyl ester	2.28	N.d.	N.d.	1.9	N.d.	N.d.	N.d.	N.d.
Acetic acid.hexyl ester	5.64	0.31	N.d.	4.71	N.d.	0.33	N.d.	0.5
Ketogluconic acid.methyl ester	20.66	0.06	N.d.	N.d.	0.03	0.23	N.d.	N.d.
Hexanedioic acid.dibutyl ester	30.82	0.1	0.05	0.21	0.04	0.78	N.d.	0.09
Benzoic acid.hexyl ester	39.94	0.05	0.92	0.41	N.d.	0.84	0.1	0.4
1.2-benzenedicarboxylic acid.mono(2-ethylhexyl)ester	44.62	N.d.	0.64	1.97	1.42	N.d.	N.d.	N.d.
Total Esters		**0.52**	**1.61**	**9.2**	**1.49**	**2.18**	**0.1**	**0.99**
Terpenes								
a-Myrecene	1.89	3.92	N.d.	N.d.	N.d.	N.d.	N.d.	N.d.
Benzene.1.3-dimethyl	2.94	2.39	N.d.	N.d.	N.d.	N.d.	N.d.	N.d.
D-limonene	3.53	5.88	0.13	N.d.	N.d.	5.25	N.d.	N.d.
Camphene	11.47	N.d.	N.d.	N.d.	N.d.	0.17	N.d.	N.d.
3-Carene	12.84	1.1	0.43	0.11	1.74	3.21	0.5	0.44
Phenanthrene.2-methoxy	11.10	5.74	N.d.	N.d.	N.d.	N.d.	N.d.	N.d.
Squalene	43.04	0.41	0.18	2	0.59	0.62	0.59	0.39
Total Terpenes		**19.44**	**0.74**	**2.11**	**2.33**	**9.25**	**1.09**	**0.83**
Acids								
Ethylphosphonic acid	5.27	0.16	0.77	N.d.	0.59	N.d.	N.d.	N.d.
Acetic aid	11.20	4.19	2.68	2.53	5.23	2.22	2.29	3.49
Formic acid	12.52	0.44	0.3	N.d.	1.02	0.14	N.d.	0.39
Propanoic acid	13.01	0.32	N.d.	N.d.	N.d.	N.d.	0.27	0.36
Hexanoic acid	14.77	N.d.	0.27	0.24	0.17	0.41	0.6	0.12
Butanoic acid	14.78	0.06	0.09	N.d.	N.d.	N.d.	N.d.	N.d.
butanoic acid.2-methyl	15.56	0.04	N.d.	0.21	0.47	N.d.	0.7	N.d.
Hexanoic acid.2-methyl	20.98	N.d.	N.d.	N.d.	N.d.	0.7	N.d.	N.d.
Pentanoic acid	20.99	0.31	N.d.	0.26	N.d.	N.d.	N.d.	0.05
Tetradecaonic acid	38.20	0.73	0.93	1.29	0.66	0.65	0.76	0.64
Pentadecanoic acid	39.76	0.46	0.32	0.56	0.47	0.33	0.43	0.34
n-Hexadecenoic acid	41.25	3.12	3.37	5.24	5.86	2.82	3.3	3.96
Hexadecanoic acid.Z-11-	41.76	0.86	N.d.	0.33	0.15	0.55	N.d.	0.31
Z-11-Tetradecenoic acid	41.76	N.d.	N.d.	N.d.	N.d.	0.46	0.13	N.d.
Octadecanoic acid	44.07	1.36	0.65	1.51	0.26	0.65	0.64	1.07
Oleic acid	44.48	0.48	N.d.	0.41	N.d.	N.d.	N.d.	N.d.
Total Acids		**12.53**	**9.38**	**12.58**	**14.88**	**8.93**	**9.12**	**10.73**

Ketones								
3-hydroxy-2-Butanone	7.32	0.25	N.d.	0.15	0.58	0.11	0.26	0.5
1-Hydroxy-2-Propanone.	7.70	1.11	0.88	2.81	3.12	0.62	0.28	1.17
2H-Pyran-2-one	8.47	N.d.	N.d.	2.7	N.d.	N.d.	N.d.	N.d.
Furyl hydroxymethyl ketone	25.38	0.24	N.d.	N.d.	0.85	N.d.	N.d.	N.d.
γ-Decalactone	28.37	0.96	N.d.	0.62	0.92	0.18	0.4	1.13
Ketones Total		**2.56**	**0.88**	**6.28**	**5.47**	**0.91**	**0.94**	**2.8**

N.d: not detected

Seven terpens were detected in the seven apricot genotypes and cultivar, but only 3-carane was detected in all the genotypes, while a-myrecene (3.92%), benzene, 1.3-dimethyl (2.39%) and phenanthrene.2-mothoxy (5.74%) were detected only in 'N97' genotype. Also, camphane compound was detected only in 'N57' (0.17%) cultivar. 'Hacihaliloglu' apricot cultivar was found to have the lowest concentration in terms of terpenes (3-carane (0.43%), squalene (0.18%) and d-limonene (0.13%)). 'N97' and 'N57' cultivars had the highest total terpen concentrations with values of 19.44% and 9.25%, respectively. Among the terpenes identified, d-limonene has been reported to be responsible for fruity and citrus character of fruits (Guillot *et al*., 2006). This compound was detected only in fruits of 'N97', cv. 'Hacihaliloglu' and 'N57'. This may be due to the high affinity of the SPME fiber to these compounds. The hereby obtained results are in agreement with those reported by Solís-Solís *et al*. (2007). In a previous study (Majoros *et al*., 2008), 3-carane and d-limonene had been reported as aroma components of fresh and dried apricots. These compounds were also detected in the current study.

Two acidic volatile compounds, N-hexadeceonic and acetic acid were detected as the most abundant acids in all genotypes and cultivar, followed by octadecanoic, tetradecanoic, and pentadecanoic acid. The highest acid concentrations were detected in 'N82' (14.88%) and 'N95' (12.58%) cultivars, while 'N57' (8.93%) had the lowest acid concentration.

In total, five ketons were detected in all apricot genotypes and among them 'N95' (6.28%) and 'N82' (5.475) were found to be the richest in terms of ketones. The cultivar 'Hacihaliloglu' had the lowest value (0.88%). Also 1-hdroxy-2-propanone was detected in all genotypes and cultivar. However, 3-hydroxy-2-butanone and γ-decalactone were detected in all genotypes, except cv. 'Hacihaliloglu'. γ-decalactone is reported as an important aroma compound of apricot (Xie *et al*., 2016). Moreover, lactone is suggested to be responsible for the sweet and fruity sensory properties of fresh and dried apricot (Greger and Schieberle, 2007). Among the ketones quantified, γ-decalactone had the highest proportion in 'N91' (1.13%) and 'N97' (0.96%). The most abundant ketone was 2H-Pyran-2-one that was found to be the highest in fruits of 'N95' (2.7%).

The concentrations of volatile compounds are depending on a many physical and biochemical changes during the harvesting period. The apricot fruits were harvested based on the soluble solid content (SSC), fruit firmness and fruit color (Hegedus *et al*., 2012), but according to the experience and advises of the local farmers they may not be at the same ripening degree. Moreover, the growing conditions and cultural applications are affecting fruit quality and these differences were indicated by some researchers (Guillot *et al*., 2006; Solís-Solís *et al*., 2007).

Conclusions

Volatile compounds of six new apricot genotypes and one cultivar were analysed using SPME-GC-MS technique. Volatiles of apricot cultivars were composed mainly of aldehydes, alcohols, esters, terpenes and acids. In total, 56 volatile compounds were detected in the fruits of tested genotypes and cultivar. The concentrations of the total volatile compounds of the 'Hacihaliloglu' cultivar were mainly due to alcohol compounds, which were higher than those obtained for the other selected apricot genotypes under study. Among the six apricot selections, 'N95' had higher volatile content compared with cv. 'Hacihaliloglu'. Moreover, the highest aldehydes (40.79%), esters (9.2%) and ketones (6.28%) concentrations were detected in 'N95' genotype. Also, the second highest acid concentration was detected in 'N95' genotype (12.58%). In terms of terpenes and ketones, all genotypes had higher concentration than cv. 'Hacihaliloglu'. These differences may be caused by different genetic background of the genotypes. This work represents one of the first study describing volatile compounds of apricots in Turkey.

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