IDENTIFICATION AND CHARACTERISATION OF VOLATILE COMPOUNDS DETERMINED BY HS/GC-MS TECHNIQUE IN PULP OF 'ABBAS' FIG (Ficuscarica L.) VARIETY

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This study was carried out to determine the aromatic profiles of volatile compounds in the pulp of 'Abbas' fig variety by SPME headspace methods for (CAR/PDMS, PDMS, CAR/PDMS/DVB) procedure for GC/MS (Gas Chromatography-Mass Spectrometry) technique. The volatile compounds are known to contain different biochemical substances that can be classified as aldehydes, alcohols, esters, terpenic compounds and other compounds. According to the results of current study, pulp of the 'Abbas' fig variety was found to include twenty-five different volatile compounds. The dominant volatile compounds in dried figs are aldehydes and esters. Total esters were found to be higher than other classes of compounds. Among other compounds, detected in volatiles of the pulps were 1-hydroxy-2-propanone (CAS), isocaproic acid, benzaldehyde, 2-ethyl-Hexanol, and pentyl-alpha-furoate.

Keywords: Fig, Volatile compounds, Aroma profile, SPME, GC/MS.

INTRODUCTION

Fruits are a large horticultural group and include a lot of species and numerous cultivars, genotypes, accessions, etc., occurring in most parts of the world as cultivated, semi-wild and wild. All these three groups are important genetic resources of biodiversity, which support the life system on earth (Okatan, 2018; Mertoğlu et al., 2019; Gundesli et al; 2019; Usanmaz et al., 2019). Figs (Ficuscarica L.) are among the oldest known fruit trees that reported to date back to ancient times. Fig trees belong to the genus Ficus, Moraceae family. Figs are subtropical fruits with high adaptability to different climatic conditions and are suitable for growing in many parts of the world including temperate climates and Mediterranean countries. Total fig production throughout the world was about 1.15 million tons in 2017, produced on about 315.53 ha area. Turkey is the top country in terms of fig production with a share of 26%, and is followed by Egypt and Morocco with 15% and 12%, respectively (FAOSTAT, 2017; TURKSAT, 2018). The yearly average fig production in Turkey is about 300.000 tons and the genetic fig resources of Turkey is very rich (Calıskan and Polat, 2012). Fig fruitscan be used both fresh and dried. Due to the scientifically accepted health benefits of its fruits, the domestic and international trade of fresh figs has been increasing. The figs cultivation is widely carried in the Marmara, Black Sea, Southeast Anatolia, Aegean and Mediterranean regions of Turkey (Gozlekci et al., 2011).

According to TUIK's data, the total area devoted for fig production in Turkey is 50.330 ha with approximately 10.7 million fig tree where about 9.7 million are fruit bearing. Aegean Region has the highest fig production in Turkey (Table 1). The Eastern Marmara Region and the Mediterranean Region follow this region. 'Sari Lop' and 'Bursa Siyahi' are the most important fig varieties grown in Turkey. Among all, 'Sari Lop'variety is the only one being exported as dried figs. Moreover, 'Bursa Siyahi' is accepted as the best variety for fresh consumption. The current production trend is export-oriented in Turkey. Furthermore, there are different local fresh fig varieties in different regions of Turkey (Polat and Ozkaya, 2005; Gozlekci et al., 2011). Among these, the 'Abbas' fig variety is highly cultivated in Kahramanmaraş province and constitute a large part of the fig production in that area. 'Abbas' fig is preferred as table fig for fresh consumption due to its size and taste. It has an important share in markets throughout local provinces of Eastern Mediterranean, Central Anatolia, Southeast Anatolia and Eastern Anatolia.

Turkey has many fig varieties adapted to different climatic regions. Fig is considered as the symbol of a "healthy and long life". It is an excellent source of minerals and many biochemical compounds including phenolic compounds,

Provinces	2014		2015		2016		2017		2018	
	Production	%								
Aydın	184.548	61,46	186.124	61,92	182.775	59,84	185.412	60,65	186.346	60,80
Izmir	35.883	11,95	38.753	12,89	43.741	14,32	42.576	13,93	45.652	14,89
Bursa	29.189	9,72	22.541	7,50	25.734	8,42	25.456	8,33	26.385	8,61
Mersin	6.773	2,26	8.426	2,80	7.202	2,36	7.425	2,43	7.693	2,51
Kahramanmaras	4.210	1,40	4.520	1,50	5.350	1,75	6.210	2,03	6.000	1,95
Hatay	6.123	2,04	6.244	2,08	6.585	2,16	6.495	2,12	3.756	1,23
Gaziantep	3.207	1,07	2.870	0,95	2.913	0,95	2.235	0,73	1.992	0,65
Other provinces	30.349	10,11	31.122	10,36	31.150	10,20	36.090	9,78	34.675	9,36
Toplam	300.282	100,00	300.600	100,00	305.450	100,00	305.689	100,00	306.499	100,00

 Table 1. Fig production in the leading provinces of Turkey

Source: (TURKSAT, 2018)

vitamin A, vitamin C, minerals and organic acids. It is an exotic fruit and rich in reducing fiber, mineral and polyphenol content which make figs as an important component of the Mediterranean diet (Slavin, 2006; Trichopoulou et al., 2006; Aksoy et al., 2007; Calışkan and Polat, 2011; Mujić et al., 2014; Eskimez et al., 2019). Therefore, the intensity and diversity of the volatile constituents in fig are very important as the source of flavor for people. Especially in human nutrition, flavour of the fruit plays a significant and major role in the choice of the fruit that with human senses. The fruit aroma plays an important role on the fruit flavour and is determined by several compounds with very various biochemical natures. It is usually accepted that the varietal aroma of figs is mainly localized in the pulp and is less in the skin and juice (Solomon et al., 2006; Gozlekci et al., 2011; Mujić et al., 2014). However, the distribution of volatile compounds in plants varies between varieties due to different climatic characteristics and genetic differences. There are very limited studies about the volatile compounds of figs throughout the world. Recently, the importance of the volatile compounds in the aromatic profiles is increasing and development of rapid analysis techniques making it remarkable to determine the volatile compounds for different fruits. Solid-phase micro-extraction (SPME) technique is among these important techniques which is relatively easy for the determination of the fruit volatiles (Kafkas et al., 2005a,b; Dong et al., 2006; Cuevas-Glory et al., 2007; Gul and Tekeli, 2019). In addition, SPME is a sensitive, reliable, simple and solvent-free for identifying aroma substances. This method consists of a fused silica fiber coated with a polymeric constant excess that is fed to a liquid or gas sample (Hawthorne et al., 1992; Grison et al., 2002; Kafkas et al., 2005b; Mujić et al., 2014). The aromatic profiles of the fruits is also known to highly affected by various factors including genetic characteristics, biotic and abiotic stress factors, cultural practices, fertilization, irrigation, planting systems, ecological conditions and soil (Kafkas et al., 2009). Recently, improvement of flavor and aroma profiles has been considered in breeding programs too.But, there are very few studies regarding the concentration of aroma compounds in

different fig varieties (Nunes *et al.*, 2008; Oliveira *et al.*, 2010 Gözlekci *et al.*, 2011; Mujić *et al.*, 2014). Therefore, present research focused on the determination of the aromatic components which define the fragrance and aroma of the 'Abbas' fig variety.

MATERIALS AND METHODS

Fruits of present study were collected from the Kahramanmaraşcity, which is located between 37° 43' north longitudes and 37° 8' east latitudes with an altitude of 900 m. The climatic data of the region was obtained from the General Directorate of Meteorology. Average Temperature is 16.9 °C. Minimum and maximum temperatures are 11.4 and 22.9 °C, respectively. The average monthly rainfall of the region is 725.4 mm (Anonymous, 2018).

Fig. fruits of 'Abbas' variety were collected from the TAGEM (General Directorate of Agricultural Research and Policy)'s project named as "fig selection project" (Figure 1-3). Fruits were h and harvested at the commercial maturity in July 2018 and immediately transferred to the Instrumental Analysis Laboratory at the University of Çukurova, Faculty of Agriculture, Department of Horticulture using cold chain. Totally 3 replications were used in the current study where 25 fruits found in each replication. Fresh fig pulp was obtained experimentally and used for analysis (Figure 2). Pulp tissue was obtained from fresh fruits and homogenized with deionized water in equal portions and the diluted homogenate was stored at -20 ° C until used for volatile analysis. Some important fruit, leaf and tree characteristics of fig variety described by Anonymous (2001) (Table 2).

HS-GC/MS Analysis: The juice was obtained from the fruit pulp of each fruit. The volatile compounds were analyzed with a HS/GC/MS (producer, country). Fruits were separated shell fruit and homogenized using fruit crush blender. 10 g samples were then weighted and 0.5 g NaCl was put in these samples and incubated at 30 ° C for 20 minutes. The SPME (Solid Phase Microextraction) fibers 100 um PDMS (Polydimethylsiloxane; red) and 85 um CAR/PDMS (Carboxen/Polydimethylsiloxane; light blue) were then compared (Kafkas *et al.* 2005b) (Figure 4). The aroma compounds were determined using GC-MS equipped HP-Innowax (30 mx 0.25 mm i.d., 0.25 mm thickness). The initial oven temperature was adjusted at 60 °C and increased to 260 °C with t 5 °C/min and then held at the same temperature for 40 minutes. The injector was taken 70 eV with the range 30-400 megahertz. The cass spectra were also compared with those of the reference compounds using the commercial Wiley, Nist and Flavor GC - MS Libraries and confirmed by the retention indices obtained and percentage amounts of the separated compounds were determined by total ionization from computerized chromatograms (Kafkas *et al.*, 2005b).

Table 2. Some	important	fruit,	leaf	and	tree
charact	eristics of 'Al	bas' fig	variert		

Characteristics	Value / Characteristic
Average Fruit weight (g)	80.16
Average Fruit length (mm)	48.25
Average Fruit diameter (mm)	55.22
Average Neck length(mm)	9.57
Ostiole width (mm)	5.31-8.92
*Fruit pulp cavity	Small
*Fruit skin ground color	Yellow-green
*Fruit pulp internal color	Dark-red
Fruit skin cracks	Minute cracks
Beginning of fruit maturation	15 July (very early)
Breba: fruits mature	21 May (early)
Main crop: fruit mature	end of July
Harvest period	21-40 days (medium)
Tree growth habit	Spreading
Tendency to from suckers of tree	low
Tree vigor	Medium
Shoot length (cm)	10.42-20.65 (medium)
Shoot width (mm)	9.58-14.55 (medium)
Shoot colour	Grey
Leaf shape	Base calcarate, lobes latate
Number of lobes leaf	three
Leaf peiole length (cm)	5.2-9.8 (long)
Leaf Width (cm)	15.5-21.5
Leaf Length (cm)	18.2-23.8
Leaf area (LXW) (cm ²)	400-550 (large)
*Soluble solid Content (%)	High (16.2-20.5)
Water soluble dry matter (%)	21

*Fruit, leaf and tree characteristics length were determined in this study, soluble solid content, fruit cavity, fruit skin ground color and fruit pulp internal color values were obtained from Anonymous (2001)

RESULTS AND DISCUSSION

Some pomological and phenological features of 'Abbas' female fig variety were summarized in Table 2. According to the results obtained, it was determined that the peak period of maturation happens at the end of July. The pomological and phenological characteristics of this variety were found to be different from many varieties and have predominant features than the others, i.e.: 'Sari Lop'' and 'Sultan Selim'' as

compared with those of Gozlekci et al. (2011). It was also previously reported that the pomological and phenological features of the fig varieties ('Karabakunya', 'Sari Lop' and 'Sultan Selim') may significantly vary (Gozlekci et al., 2011). In this present study, a total of 25 volatile compounds were determined from the pulps of 'Abbas' local fig variety by using different SPME fibers including blue and red and blue at GC-MS techniques and the results are given Table 3-9 and Figure 4-6. Total 8 aldehydes, 4 terpenes, 5 esters, 5 alcohols and 3 ketones were identified in the current study. According to the results, aldehydes and terpenes were found to be the major volatiles in fig fruits (Table 3 and Table 5). Previous studies showed that the fig fruits had higher free volatile compounds in the pulps than in other parts (Oliveira et al., 2010; Gözlekci et al., 2011; Mujić et al., 2014). The compounds 1-hydroxy-2-Propanone (CAS), Phenol, 2,6-bis (1,1-dimethyl ethyl)-4-methyl- (CAS), Isocaproic acid, pentyl-, alpha-Furoate, and benzaldehyde were more abundant in the pulp of fig fruits of current study (Figure 4 and 5). Gözlekci et al. (2011) and Mujić et al. (2014) were previously reported similar results with the present study.

Table 3. Aldehydes compositions (relative content, %) of 'Abbas' fig (*Ficus carica* L.) cultivar: RT: Retention time (min).

	Ketention time (mm),		
R.T.	Compounds name	Light Blue	Red
	Aldehydes	(CAR/PDMS)	(PDMS)
4.753	Capronaldehyde	9,30	0
9.181	Benzaldehyde	11,34	0
13.723	Nonanal	1,21	0
16.857	Decanal (CAS)	0	2,37
20.669	Deca-2(E),4(E)-dienal	0	2,77
21.353	<cis-> 8-Undecenal</cis->	1,53	9,77
22.021	2-methyl-Undecanal	1,36	0
22.616	Lauric aldehyde	0	2,39
	Total Aldehyde	24,74	17,30

Table 4. Keton compositions (relative content, %) of 'Abbas' fig (*FicuscaricaL*) cultivar

	Abbas ng (<i>Ficuscurica</i> L.) cultival				
R.T.	Compounds name	Light Blue	Red		
	Ketons	(CAR/PDMS)	(PDMS)		
3.33	1-hydroxy-2-Propanone, (CAS)	3,31	0		
3.81	3-hydroxy-2-Butanone, (CAS)	3,43	0		
24.96	delta-Decalactone	0	5,95		
	Total Keton	6,74	5,95		

Table 5. Terpens compositions (relative content, %) of 'Abbas' fig (*FicuscaricaL*.) cultivar

R.T.	Compounds name	Light Blue	Red
	Terpens	(CAR/PDMS)	(PDMS)
8.33	Alpha-Pinene	1,15	0
10.75	delta-3-Carene	1,31	0
11.31	D-Limonene	0	3,10
23.06	trans-Caryophyllene	0	3,05
	Total Terpens	2,46	6,15

	Abbas' fig (<i>Ficuscarica</i> L.) cultivar			
R.T.	Compounds name	Light Blue	Red	
	Esters	(CAR/PDMS)	(PDMS)	
8.99	isobutyl-Butyrate	2,00	0	
20.46	Nonyl acetate	0	2,89	
21.08	pentyl-, alpha-Furoate	2,31	14,70	
22.02	allyl-Pelargonate	0	11,75	
27.45	decyl-Butyrate	1,41	12,36	
	Total Ester	5,72	41,70	

 Table 6. Esters compositions (relative content, %) of

 'Abbas' fig (*Eiguscarical*) cultivar

Table 7. Alcohols and other compositions (relative content, %) of 'Abbas' fig (*Ficus carica* L.) cultivar

R.T.	Compounds name	Light Blue	Red
	Alcohols	(CAR/PDMS)	(PDMS)
11.42	2-ethyl-Hexanol	4,24	5,70
12.19	Nonylol	1,74	0
15.65	2(E)-Nonenol	0	2,62
15.78	Epoxylinalol	0	5,27
25.39	Phenol, 2,6-bis(1,1-	0	15,30
	dimethylethyl)-4-methyl- (CAS)		
	Total Alcohol	5,98	28,89

Table 8. Acid and compositions (relative content, %) of 'Abbas' fig (*Ficus carica* L.) cultivar

R.T. Compounds name	Light Blue	Red
Alcohols	(CAR/PDMS)	(PDMS)
10.26 Acid		
Isocaproic acid	23,25	0
Total Acid	23,25	0
10.91 Caprolactoneoxim	14,61	0
34.70 2-Benzothiazolamine, N-ethyl-	0	7,55
25.95 D-(+)-Lactose	6,87	0
Other componds	16.81	16.95

Table 9. Volatile compounds of 'Abbas' fig (*Ficus carica* L.) cultivar

	L.) cultival		
R.T.	Compounds name	Light Blue	Red
	Aldehydes	(CAR/PDMS)	(PDMS)
4.75	Capronaldehyde	9,30	0
9.18	Benzaldehyde	11,34	0
13.72	Nonanal	1,21	0
16.86	Decanal (CAS)	0	2,37
20.67	Deca-2(E),4(E)-dienal	0	2,77
21.35	<cis-> 8-Undecenal</cis->	1,53	9,77
22.02	2-methyl-Undecanal	1,36	0
22.62	Lauric aldehyde	0	2,39
	Total Aldehyde	24,74	17,30
	Ketons		
3.33	1-hydroxy-2-Propanone, (CAS)	3,31	0
3.81	3-hydroxy-2-Butanone, (CAS)	3,43	0
24.96	delta-Decalactone	0	5,95
	Total Keton	6,74	5,95



Figure 1. General view of the research parcel

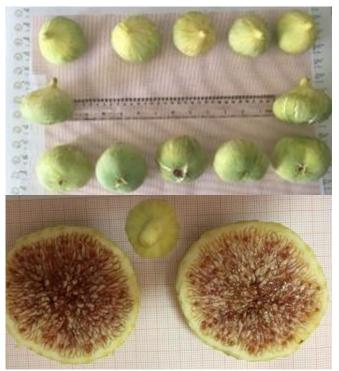


Figure 2. General view of the 'Abbas' fig variety

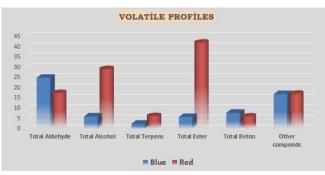


Figure 3. Total comparison of volatiles of fig of variety "Abbas" fruits using various SPME fibers

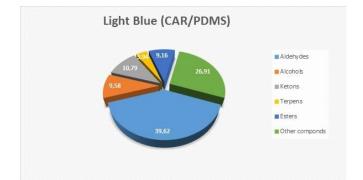


Figure 4. Percentage of the Volatile Compounds of Fig variety using Light Blue SPME fibers

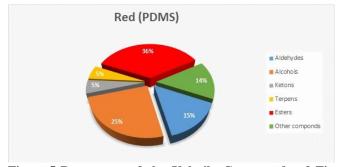


Figure 5. Percentage of the Volatile Compounds of Fig variety using Red SPME fibers

As seen in Figure 4, the percentage of the volatile compounds ranged from 17.30% to 24.74% for aldehydes, from 5.98% to 28.89% for alcohols, from 5.95% to 37.74% for ketons, from 2.46% to 6.15% for terpenes, from 23.25% for acids and from 5.72% to 41.70% for esters (Figure 3-5). As shown in Table 4 and 7, The amounts of esters, aldehydes, acids, alcohols, terpenes, ketones from aroma compounds determine very different variations. Similar results were previously reported by Kafkas et al. (2016) for aroma compounds. In our study, red (36%) and light blue (39.62%) fiber were found to be high percent of the ester composition (Figure 4 and 5). Gözlekci et al. (2011) previously conducted a study with different fig varieties and reported significantly less esters concentration in the fruit pulp than the results of current study. In another study, Kafkas and Kafkas (2016) reported that ester composition determined when red (37.22%) and blue (28.09%) and similar to our results. The authors also stated that the percentages of the esters identified varied with the fibers used. In our study, it was determined that aldehyde percentages were very high when blue and red fibers were used. Similar results were obtained by the Kafkas et al. (2016) for the 'Rubygem' strawberries. In this study, as suggested by some researchers, aldehydes were the most abundant and higher percentages (Kim et al., 2008; Gözlekci et al., 2011; Mujić et al., 2014).

In the current study, alcohols were found to comprise about 9.14 to 30.44% of the volatiles. 2-ethyl-Hexanol, Nonylol 2(E)-Nonenol, Epoxylinalol, Phenol, and 2,6-bis (1,1-dimethylethyl)-4-methyl- (CAS) were detected as alcohol (Table 7). Among the alcohols phenol, 2,6-bis(1,1-dimethylethyl)-4-methyl- (CAS) was found to have the highest proportion. In different studies, carried with different fruit types, alcohols were found to contribute to a little proportion of the aromas (Larsen and Watkins, 1995; Mujić *et al.*, 2014). 1-hydroxy-2-Propanone (CAS), 3-hydroxy-2-Butanone (CAS) delta-Decalactone was detected as Keton compounds and the highest terpenes were determined when light blue fiber used (Table 9; Figure 4-6).

As seen in Table 6, Alpha-Pinene, delta-3-Carene, D-Limonene and trans-Caryophyllene were detected as terpene compounds when using red fiber (Figure 3-5). Among the alcohols Caryophylleneterpenes was dominant and found to had the highest proportion (Table 6). Previous studies indicated that Caryophyllene and Limonene were the main volatile compounds for different fig cultivars (Oliveira 2010; Steliopoulos et al., 2002; Gözlekci et al., 2011). Kim et al.(2008) reported that terpenes share a small portion of the fig aroma compounds but they may had animportant effect on the antibacterial characteristics of the fruits. In this sudy, specially terpenes such as D-Limonene and trans-Caryophyllene were the most abundant and high percentage of aldehyde, as similar to previous studies (Gözlekci et al.,2011). Other volatiles of the current study are: Caprolactone oxime, 2-Benzothiazolamine and N-ethyl- and D-(+)-Lactose were found in trace amounts in some samples (Table 9). Moreover, it was previously reported that low levels of these volatile were naturally occurring in many foods, such as fruits (Russo et al., 2017).

Conclusions: In recent years, there has been an increasing interest from both consumers and researchers on figs due to their wide range of contributions to healthy living. According to the Authors knowledge, this is the first study to compare the aromatic profile of volatiles in 'Abbas' fig variety. The aroma profiles of fig were characterised with HS-SPME by GC-MS. Results showed that there area total of 25 volatile compounds in the pulp of 'Abbas' fig variety which were identified by various SPME fibers (red and light blue). These compounds can be used to characterize and distinguish fig varieties on aromatic criteria. The variety found to has different volatile profile patterns as compared with others and in general fruit pulp was found to be more diverse. The major components in the fig pulp were found to be esters and aldehydes.

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