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DETERMINATION OF VOLATILE COMPOUNDS OF THE FIRST ROSE OIL AND THE FIRST ROSE WATER BY HS-SPME/GC/MS TECHNIQUES

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Abstract

Background: Rose water and rose oil are used in the perfume, cosmetic, pharmaceutical and food industries. The determination of volatile compounds in rose oil and rose water obtained from oil-bearing rose is highly important in terms of availability in the industry and in human health.
Materials and Methods: Twenty four and twenty six volatile compounds were determined in the first rose oil and in the first rose water. Thus, in this study, volatile compounds in the first rose oil and first rose water have been determined by HS-SPME/GC/MS (Headspace-Solid Phase Micro Extraction/Gas Chromatography Mass Spectrometry) techniques which were not published previously for determining volatile compounds in rose oil and rose water.
Results: Twenty four and twenty six volatile compounds were found in the first rose oil and in the first rose water, respectively. It was further discovered that both first rose oil and first rose water are rich in oxygenated monoterpenes and sesquiterpenes, with a third group of volatile compounds known as aliphatic hydrocarbons being found only in first rose oil. Citronellol contents of the first rose oil and rose water were found to be 43.40% and 40.13% respectively, whereas geraniol contents were 11.81% and 15.97%, respectively.
Conclusion: These findings suggest that HS-SPME/GC/MS is a suitable technique for the determination of volatile compounds of rose oil and rose water.

Keywords: Oil-bearing rose, *Rosa damascena*, HS-SPME/GC/MS, volatile compounds.

Introduction

Roses are considered as cut flower, garden plant and indoor plant in ornamental plants industry (Weiss, 1997). In addition, they have economic importance as a source of natural fragrances and aromas. Containing important aromatic and volatile compounds, they have a significant value in food, perfumes, cosmetics and pharmaceutical industries (Guterman et al., 2002; Kovacheva et al., 2010). Four major species of rose (*Rosa damascena* Mill, *Rosa gallica* L., *Rosa moshata* Herrm and *Rosa centifolia* L.) which are used in the production of essential oil are available throughout the world. Among these Rosa species, the most important one is *Rosa damascena* Mill. (Göktürk Baydar et al., 2004). *Rosa damascena* is more commonly grown in Turkey and Bulgaria. Oil-bearing rose has been grown in Isparta, Turkey since 1881 while it has been cultivated in Kazanlak, Bulgaria since 1664 (Baydar et al., 2008a).

Important industrial products such as oil, water, concrete and absolute are obtained from oil-bearing rose (Kurkcuoglu and Baser, 2003; Aycı et al., 2005). These are distillation and extraction products of freshly harvested rose flowers. *Rose oil* and *rose water* are obtained from steam distillation and *concrete* is derived from the extraction of solvent; whereas *absolute* is obtained from the re-extraction of concrete with the alcohol (Göktürk Baydar and Baydar, 2005; Baydar et al., 2008a; Kovacheva et al., 2010). In rose oil processing technology, fresh flowers of oil-bearing rose are distilled with steam. This is followed by oil production wich is obtained from the first distillation of rose flowers. The names 'first oil', 'direct oil', 'curude oil' or 'decanted oil' therefore derive from the fact that the oil was produced from first distillation of rose flowers. Oily water remaining in the boiler cup during this process is considered as 'first water' or 'bottom water'. Oil obtained through the second distillation of this water is referred to as 'second oil', 'indirect oil', 'cooked oil', or 'recovered oil'. Blended oil, which is obtained by combining first and second oil, is used commercially. Rose water offered for commercial use is obtained by re-distillation of oily water accumulated under rose oil during the distillation process (Başer, 1992; Tosun et al., 2002; Aycı et al., 2005; Baydar et al., 2008b).

Rose oil and rose water are among the most valuable raw materials of food, perfume, cosmetic, and pharmaceutical industries (Farooq et al., 2011; Rusanov et al., 2012). Rose water is used in religious ceremonies in some countries such as Iran (Mostafavi and Afzali, 2009). In the food industry, rose water and rose oil are used in the production of tea, jam, biscuits, cake, confectionery, and beverages due to their aromatic and volatile characteristics (Farooq et al., 2011; Rusanov et al., 2011). The cosmetics industry uses rose-oil in the production of tonic, cream, soaps, and other cosmetic products such as skin care lotion with antiseptic and refreshing effects (Rusanov et al., 2011; Baydar et al., 2007). In perfumery, rose oil serves as a fixator in perfume blends (Buccellato, 1980; Baydar et al., 2008a). Some rose preparations are used medicinally as tonics, mild laxatives and in treatment of sore throat, enlarged tonsils, cardiac troubles, eye disease as well as gall stones (Verma et al., 2011a). In addition, anti-HIV (Mahmood et al., 1996), antibacterial and analgesic (Basim and Basim, 2003), antispasmodic and hypnotic activities (Boskabady et al., 2006), antitussive, anti-inflammatory (Köse et al., 2007) and anxiolytic-like (Almeida et al., 2004) effects of rose extract/isolates have been reported. Furthermore, rose oil aroma has been identified to have positive effects on learning and memory (Köse et al., 2007).

Volatile compounds of oil-bearing rose products such as flower, oil, water, concrete and absolute have been identified in several studies (Baser, 1992; Bayrak and Akgül, 1994; Babu et al., 2002; Loghmani-Khouzani et al., 2007; Mostafavi and Afzali, 2009; Dobreva and Kovacheva, 2010; Aycı et al., 2005; Kurkcuoglu and Baser, 2003; Verma et al., 2011b; Naquvi et al., 2014; Koksal et al., 2015). The most significant volatile compounds of rose oil are monoterpene alcohols such as citronellol, geraniol, linalool, nerol and farnesol (Baser, 1992; Baydar and Göktürk Baydar, 2005; Baydar et al., 2013). In addition, it has been identified that hydrocarbons (nonadecane, eicosane, heneicosane and tricosane), alcohols, oxides, ethers (methyl eugenol), aldehydes (geranyl acetate and geranyl), esters, ketones, phenols (eugenol) are

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available within rose oil (Baser, 1992; Bayrak ve Akgül, 1994; Baydar et al., 2008b; Baydar et al., 2013). Other volatile compounds which contribute immensely to the volatile characteristic of rose oil include β-damascenone and β-ionene. However, they exist in low amounts (David et al., 2006; Baydar et al., 2013). More volatile compounds such as citronellol, nerol, geraniol, linalool and 2-phenethyl alcohol were found in rose water (Göktürk Baydar and Baydar, 2005; Baydar et al., 2007; Mostafavi and Afzali, 2009).

Volatile compounds of rose oil and rose water obtained from the first distillation have not been reported previously. Therefore, the aim of the present study is to identify volatile compounds of first rose oil and first rose water via hydro distillation method of oil-bearing rose. Moreover, since previous studies focused on identifying essential oils of rose oil and rose water by means of GC-FID, GC-MS or GC/GC-MS techniques, the present study aims at determining volatile compounds of first rose oil and first rose water by the use of HS-SPME/GC/MS (Headspace Solid Phase Micro Extraction/Gas Chromatography Mass Spectrometry) techniques. This is therefore the first study to report on the identification of volatile compounds of first rose oil and first rose water using HS-SPME/GC/MS techniques.

Materials and Method

First Rose Oil and First Rose Water Extraction

In the study, oil-bearing rose plants were grown in the Fruit Research Station-Isparta. Samples were collected in the early hours of the morning (between 05:00 and 08:00) and distilled once. Clevenger hydro-distillation apparatus was used in order to obtain rose oil. For this purpose, 500 g of roses were placed in 5 liter-Clevenger and after adding 1.5 liters of water, it was subjected to distillation for 2.5 hours by means of heating mantle. Essential oils obtained as a result of distillation process was recorded as % (v/w) and it was accepted as 'first rose oil' (Baydar et al., 2008a).

Small commercial hydro-distillation apparatus (Dünder) was used to obtain oil water. Fifteen kilograms (15 kg) of fresh rose flowers were placed in a 100 liter stainless steel bowl followed by addition of 45 l of water and then subjected to distillation for 2.5 hours. Rose oil obtained at the end of distillation process was collected and separated. Residual water was then accepted as 'first rose water' (Baydar et al., 2008a).

SPME Analysis of Volatile Compounds

A supelco fiber holder (Bellefonte, PA-USA) and a 100 µm polydimethylsiloxane (PDMS) coated fused-silica fiber were used due to the most suitable fiber for adsorbing volatiles compound from the rose oil and first distilation water. Prior to first extraction, the fiber was conditioned in the GC injector port at 250°C for 1h according to manufacturer's recommendation. HS-SPME (Headspace-Solid Phase Micro Extraction) technique was used in the extraction of the volatile compounds. The samples were homogenized with saturated sodium chloride (1g) for HS-SPME and 5 ml of sample for each extraction was placed into a 100 ml glass vial. In HS-SPME analysis, the PDMS fibre was inserted into the headspace of the glass vial and PDMS fibre was immersed into the sample for 30 mins at 30°C. During this time, experimental samples were stirred with a magnetic stirrer. After equilibration, the fibre was removed from the sample and the analytes were thermally desorbed in the injector port of the GC/MS instrument for analysis. Thermal desorption in the injector glass liner at 250°C, for 10 min. The analyses were carried out in triplicate.

GC/MS Analysis

Aroma compounds of the samples were analysed by GC-MS (Gas Chromatography Mass Spectrometry). A Perkin Elmer Clarus apparatus equipped with CPSil5CB (25 m x 0.25 mm i.d., 0.4 µm film thickness) fused-silica capillary column was used. The flow rate of helium as carrier gas was 1 ml/min. The injector temperature was 250°C, set for splitless injection. The column temperature was 6 °C//5°C/min//260°C for 20 min. Mass spectra were taken at 70 eV. Mass range was between m/z 30-425. A library search was carried out using the Wiley GC-MS Library and Flavor Library of Essential Oil Constituents. The mass spectra were also compared with those of reference compounds and confirmed with the aid of retention indices from published sources. Relative percentage amounts of the separated compounds were calculated from total ion chromatograms by the computerized integrator.

Results and Discussions

Volatile compounds and chemical class, retention time, and the percentage of volatile compounds which were determined by HS-SPME/GC/MS technique in the first rose oil (F-RO) and first rose water (F-RW) obtained through first distillation process in oil-bearing rose (*Rosa damascene* Mill.) are presented in Table 1. While 24 volatile compounds were determined in the first rose oil, 26 volatile compounds were identified in the first rose water.

In the analyses made in terms of the first rose oil, dominant volatile compounds were determined to be citronellol (43.40%), geraniol (11.81%), heneicosane (6.53%), pentadecane (4.53%), methyleugenol (4.04%), eugenol (3.82%), and phenol (2.85%). Besides, such compounds as farnesyl acetate, azulene, trans-caryophyllene, nonadecane, δ-3-carene, E-15-heptadecenal, geranyl acetate, β-pinene, heptadecane, cis-rose oxide, α-caryophyllene, α-guaiene, tetracosane which are among the volatile compounds in rose oil were determined to be taken into consideration. In addition, such compounds as elemol, nerolidol, farnesol, octadecane were found in a trace amount (Table 1).

In the analyses made in the first rose water, dominant volatile compounds were determined to be citronellol (40.13%), geraniol (15.97%), benzoic acid 2-hydroxy-.3-methyl butyl ester (6.82%), carbamic acid methyl ester (5.03%), geranyl acetate (4.02%), methyleugenol (3.33%), linalool (3.21%), and eucalyptol (2.82%). Moreover, eugenol, phenethyl alcohol, caryophyllene oxide, phthalic acid, isobutyl octyl ester, camphor, citronellyl acetate, lavandulyl acetate, azulene, cis-rose oxide, farnesol, borneol, phenol, terpinen-4-ol, δ-cadinene, β-fenchyl alchol and dodecanoic acid in F-RW were found in a remarkable rate. Nerolidol and nonadecane were found in a trace amount (Table 1).

The percentage of class compositions of F-RO and F-RW of *R. damascena* volatile compounds were given in Table 2. Six monoterpenes (62.08%), eight sesquiterpenes (9.59%), six aliphatic hydrocarbons (15.83%), only eugenol (3.82%) as benzenoid compound and three other compounds (8.68%) were determined in the first rose oil. Besides, twelve monoterpenes (72.93%), five sesquiterpenes (4.55%), one aliphatic hydrocarbon (0.17%), phenethyl alcohol (2.01%) and eugenol (2.25%) as benzenoid compounds and six other compounds (18.9%) were determined in the first rose water.

While two monoterpene hydrocarbons (MH) were found in the first rose oil, this was not the case in the first rose water. The most intense group determined in both F-RO and F-RW was oxygenated monoterpenes (OM). Four OM (58.49%) were found in the first rose oil, whereas twelve OM (72.93%) were identified in the first rose water (Table 2). Citronellol and geraniol available in OM group are the most volatile compounds in not only the first rose oil but also the first rose water (Table 1).

Table 1: The percentage of volatile compounds of fist rose oil (F-RO) and first rose water (F-RW) of *Rosa damascena*.

No	*Compound*	*Class*	*RT*[x]	*F-RO (%)*	*F-RW (%)*
1	Carbamic acid methyl ester	OC	2.454	nd[y]	5.03
2	Eucalyptol	OM	8.804	nd	2.82
3	Linalool	OM	10.882	nd	3.21
4	Phenethyl alcohol	BC	11.450	nd	2.01
5	Cis-Rose oxide	OM	11.088	1.50	0.91
6	Camphor	OM	12.145	nd	1.76
7	δ-3-Carene	MH	12.483	1.83	nd
8	Borneol	OM	12.912	nd	0.82
9	β-Pinene	MH	12.924	1.77	nd
10	Terpinen-4-ol	OM	13.190	nd	0.50
11	β-Fenchyl alchol	OM	13.650	nd	0.47
12	Citronellol	OM	14.924	43.40	40.13
13	Geraniol	OM	15.613	11.81	15.97
14	Lavandulyl acetate	OM	16.332	nd	1.14
15	Citronellyl acetate	OM	18.054	nd	1.18
16	Eugenol	BC	18.423	3.82	2.25
17	Geranyl acetate	OM	18.936	1.78	4.02
18	Methyleugenol	OC	19.613	4.04	3.33
19	Phenol	OC	20.652	2.85	0.62
20	Trans-caryophyllene	SH	21.758	2.12	nd
21	α-Guaiene	SH	21.993	0.98	nd
22	α-Caryophyllene	SH	22.163	1.29	nd
23	Pentadecane	AH	23.021	4.53	nd
24	Azulene	SH	23.238	2.28	1.05
25	Elemol	OS	24.193	tr[z]	nd
26	Nerolidol	OS	24.326	tr	tr
27	Caryophyllene oxide	OS	24.356	nd	1.97
28	Dodecanoic acid	OC	25.129	nd	0.36
29	δ-Cadinene	SH	25.800	nd	0.50
30	Heptadecane	AH	27.341	1.69	nd
31	Farnesol	SA	27.534	tr	0.87
32	Farnesyl acetate	SE	28.090	2.44	nd
33	Octadecane	AH	29.316	tr	nd
34	[†]Benzoic acid 2-hydroxy-..….	OC	29.504	nd	6.82
35	[††]Phthalic acid isobutyl..….	OC	30.815	nd	1.92
36	E-15-Heptadecenal	OC	31.214	1.79	nd
37	Heneicosane	AH	38.077	6.53	nd
38	Nonadecane	AH	41.600	2.01	tr
39	Tetracosane	AH	44.000	0.87	nd
	Total identified			99.31	99.65

[†]Benzoic acid 2-hydroxy-. 3-methyl butyl ester
[††]Phthalic acid isobutyl octyl ester
[x]RT: Retention time
[y]nd: Correct isomer not determined
[z]tr: Trace (<0.2%).

On one hand, the total rate of four sesquiterpene hydrocarbons (SH) in the first rose oil was found to be considerable with 6.67%; on the other, just two SH (1.55%) were determined in the first rose water. Two oxygenated sesquiterpenes (OS) were found in both first rose oil and first rose water. While the total ratio of OS was determined to be 0.33% in the first rose oil, it was found to be 2.14% in the first rose water. As sesquiterpene alcohols, only farnesol was found in F-RO and F-RW. In addition, farnesyl acetate (2.44%) was only found in F-RO as sesquiterpene ester (Table 2).

Table 2: The percentage of class composition of first rose oil (F-RO) and first rose water (F-RW) of *Rosa damascena* volatile compounds

Class composition	Abbreviations of class composition	Total contents of F-RO (%)	Total contents of F-RW (%)
Monoterpene hydrocarbons	MH	3.59	nd
Oxygenated monoterpenes	OM	58.49	72.93
Sesquiterpenes hydrocarbons	SH	6.67	1.55
Oxygenated sesquiterpenes	OS	0.33	2.14
Benzenoid compounds	BC	3.82	4.26
Aliphatic hydrocarbons	AH	15.83	0.17
Sesquiterpene alcohols	SA	0.15	0.87
Sesquiterpene esters	SE	2.44	nd
Other compounds	OC	8.68	18.09

Furthermore, in the analyses, carbamic acid methyl ester, methyleugenol, phenol, dodecanoic acid, benzoic acid 2-hydroxy- 3-methyl butyl esters, phthalic acid isobutyl octyl ester, E-15-heptadecenal were determined. Among them, methyleugenol was found at a remarkable rate in both F-RO and FRW. Also, the ratios of carbamic acid methyl ester and benzoic acid 2-hydroxy- 3-methyl butyl ester, which were only in F-RW, were found to be important (Table 1).

Volatile compounds identified as common in both first rose oil and first rose water is presented in Fig 1. The ratios of volatile compounds which are common in the first rose oil and first rose water were determined to be generally higher in F-RO except for geraniol, geranyl acetate and farnesol. In other respects, δ-3-carene, β-pinene, trans-caryophyllene, α-guaiene, α-caryophyllene, pentadecane, elemol, heptadecane, farnesyl acetate, octadecane, E-15-heptadecenal, heneicosane, tetracosane are volatile compounds which were only determined in F-RO (Table 1). Unlike the first rose oil, volatile compounds available in the first rose water are carbamic acid methyl ester, eucalyptol, linalool, phenethyl alcohol, camphor, borneol, terpinen-4-ol, β-fenchyl alchol, lavandulyl acetate, citronellyl acetate (Table 1).

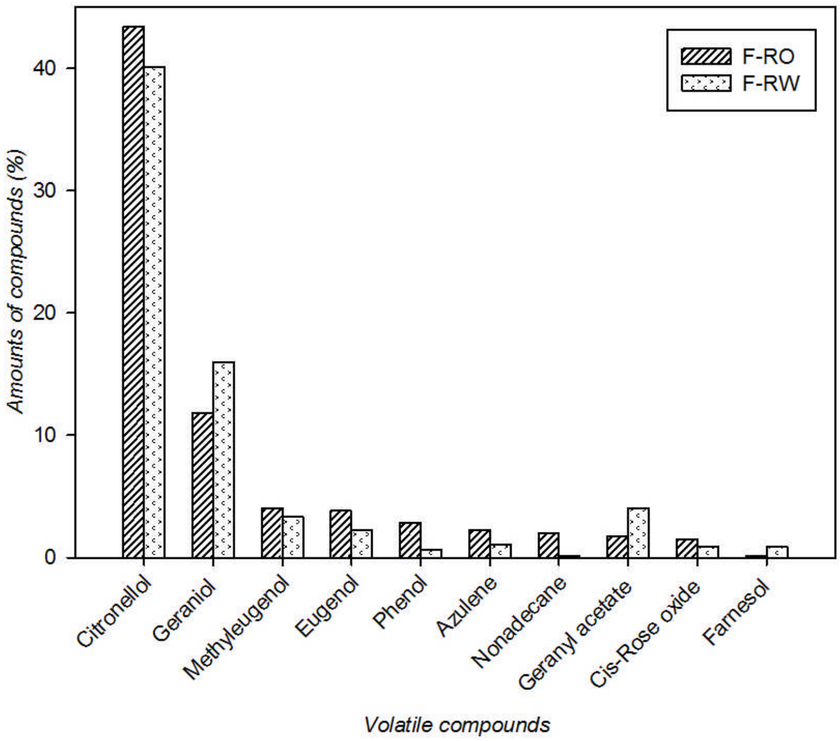

Figure 1: The common volatile compounds of first rose oil (F-RO) and first rose water (F-RW)

So far, in the studies carried out in order to determine the volatile compounds in rose oil, GC-MS, GC/GC-MS or GC-FID techniques have been widely utilized (Babu et al., 2002; Baydar et al., 2008b; Baydar et al., 2013; Naquvi et al., 2014). Unlike previous studies, our study reflects the results of HS-SPME/GC/MS techniques. Despite the use of a different method for the determination of volatile compounds, the results of the present study are in agreement with those reported in literature. Depending on the GC/FID and GC/MS analyses made in the Turkish rose oil, the most important volatile compounds were determined to be monoterpene alcohols such as linalool, citronellol, nerol and geraniol; long-chain hydrocarbons such as nonadesan, nonadesen, eicosane, heneicosane and tricosane; oxides and ethers such as methyleugenol; esters and aldehydes such as geranyl acetate, and geranial and phenols such as eugenol (Baydar et al., 2013). In the current study, some of the volatile compounds like citronellol, geraniol, heneicosane, pentadecane, methyleugenol, eugenol and geranyl acetate which are available in general in rose oil were determined in F-RO in this study while some of those like linalool and nerol reported previously were not identified. Moreover, volatile compounds which were determined in rose oil in previous studies such as citronellol, geraniol, geranyl acetate, methyl eugenol, linalool, and eugenol were found in F-RW which has high rate oil content, in our study.

Loghmani-Khouzani et al. (2007) revealed that the dominant compounds were β-citronellol (14.5-47.5%) nonadecane (10.5-40.5%) geraniol (5.5-18%) and heneicosane (7-14%) in rose oil obtained from the oil-bearing rose plants grown in different regions of Iran using GC-MS technique. In parallel with these results, citronellol (43.40%), geraniol (11.81%), and heneicosane (6.53%) were also determined as dominant compounds on first rose oil in our study. In addition, unlike previous studies, although HS-SPME/GC/MS technique was used in our study so as to determine volatile compounds in the *first rose oil,* the content of three dominant compounds (citronellol, geraniol, and heneicosane) were found to be similar with previous studies. This shows that HS-SPME/GC/MS techniques can be used for the determination of volatile compounds in rose oil. Besides, in the analysis performed in the *first rose water*, citronellol (40.13%) and geraniol (15.97%) were found to be major compounds in our study and also similar with previous studies on rose-oil. On the other hand, previous studies reported that, 2-phenethyl alcohol, citronellol, nerol, geraniol and linalool were observed in the second distilled rose water (Mostafavi and Afzali, 2009; Baydar and Göktür-Baydar, 2005). In addition to this, the second distilled rose water was reported to contain a high proportion of 2-phenethyl alcohol (Göktürk Baydar and Baydar, 2005). Being the 'first distilled rose water' and containing high proportion of oil, our study validates the fact that phenethyl alcohol content is low. Water solubility of phenethyl alcohol regarded as the essential compound of rose water is quite high and thus it was found at a higher rate in the second distilled rose water (Baydar et al., 2008b).

Compare to the previous reports, there are some differences in the volatile compounds of rose oil. In general, the content of monoterpenes, sesquiterpenes, and aliphatic hydrocarbons were reported to be high. Baydar et al. (2008b) reported that acyclic monoterpene alcohols, long-chain hydrocarbons, sesquiterpene hydrocarbons, esters and aldehydes were the major groups in Turkish rose oil. Kazaz et al. (2009) stated that the main compound groups of Turkish rose oil are monoterpene alcohols and hydrocarbons. However, Nequvi et al. (2014) found out two monoterpenes (0.44%), nineteen sesquiterpenes (44.5%), and twenty-nine aliphatic compounds (54.47%) in the rose oil through GC-GC/MS technique in India conditions. It is normal that there are differences among the results found by different researchers when determining the compounds of rose oil. As is known, the quantity and quality of rose oil are influenced by factors such as genotype, region, climate, soil, and altitude characteristics, cultivation techniques, collection time, the waiting duration as well as the process of distillation and extraction (Babu et al., 2002; Misra et al., 2002; Saffari et al., 2004; Baydar and Göktürk-Baydar, 2005; Baydar et al., 2008b, Dobreva and Kovacheva, 2010; Farooq et al., 2011; Kazaz et al., 2009; Kazaz et al., 2010; Baydar et al., 2013). Although there are some differences in terms of various volatile compounds, our results are parallel with the literature. Similar to previous studies, our study determined the major groups in the first rose oil which are oxygenated monoterpenes, aliphatic hydrocarbons, and sesquiterpenes. In first rose water, monoterpenes, sesquiterpenes and benzenoid compounds are the main groups of volatile compounds.

Conclusion

In the present study, it was found that 24 volatile compounds were identified in first rose oil of oil-bearing rose (*Rosa damascene* Mill.) while 26 volatile compounds were determined in first rose water of oil-bearing rose by HS-SPME/GC/MS techniques. It was determined that dominant volatile compounds of rose oil are citronellol, geraniol, heneicosane, pentadecane, methyleugenol, eugenol and phenol. On the other hand, dominant volatile compounds of first rose water were identified to be citronellol, geraniol, benzoic acid 2-hydroxy-.3-methyl butyl ester, carbamic acid methyl ester, geranyl acetate, methyleugenol, linalool and eucalyptol. It was pointed out that first rose oil is rich in terms of monoterpenes, sesquiterpenes and aliphatic hydrocarbons while monoterpenes and sesquiterpenes are dominant in first rose water. Oxygenated monoterpenes (OM) have been particularly found to be prominent in monoterpenes. It was determined that the first rose water is much richer than rose oil in terms of OM number and total content of OM. Oxygenated monoterpenes contents of the first rose water and first rose oil were determined to be 72.9% and 58.49%, respectively, and OM numbers were found as 4 and 12, respectively. Furthermore, 10 common compounds, mainly citronellol and geraniol, were determined in both F-RO and F-RW. Citronellol contents in the first rose oil and first rose water were determined as 43.40% and 40.13% respectively, while geraniol content was found to be 11.81% and 15.97% respectively. This study indicates that HS-SPME/GC/MS techniques, which were not used in previous studies, are appropriate for determining volatile compounds in first rose oil and water.

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