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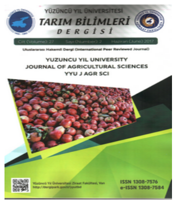

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Research Article

Extraction and Identification of Volatile Compounds in *Rosa laxa* Retz var *harputense* T. Baytop "Kişmiri rose"

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Abstract: The gardening of ornamental plants comes back to ancient times (Urartians). Nowadays the gardening of ornamental plants is done worldwide as in the Van provinces of Türkiye. Old and traditional gardens can be seen in different regions of this province naturally. Among these plants, *Rosa laxa* Retzius var. *harputense* T. Baytop (Kişmiri rose), is an exotic plant coming from Central Asia. Kişmiri rose has semi-double and miniature flowers and blooms for about five months in the ecological conditions of Van. It has not only highly decorative but also has a light, pleasant, and enthusiastic fragrance. As far as we know there is no previously published on the volatile profile of *Rosa laxa* Retz var. *harputense* (Kişmiri rose). For this purpose, in this study, "Headspace Solid Phase Micro Extraction Gas Chromatography-Mass Spectrometry" (HS/SPME/GC/MS) was applied for the detection of volatile compounds of Kişmiri rose flowers. A total of 31 compounds of Kişmiri rose were identified and quantified using Gas Chromatography-Mass Spectrometry (GC/MS). Among these identified compounds, phenyl ethyl alcohol (26.59%), cis-3-hexenyl acetate (18.573%) was detected as the major ones. According to the obtained results, it is concluded that our species has also a Chinese origin.

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1. Introduction

Roses are grown and consumed as food, perfumes, cosmetics, and pharmaceutical aspects and have a big potential for the ornamental plant industry (Guney, 2020). Native plants including *Rosa* species can be used for aesthetic studies besides functional landscape renewals such as biological repair works (Erzurumlu and Erzurumlu, 2021). The importance of roses is increasing recently worldwide because of containing natural fragrances and aromas, which make them an economically precious plant (Lavid et al., 2002; Kovacheva et al., 2010).

The rose plant, which has been used for medicinal purposes for over 5000 years, is mostly of Western Asian and partly European origin. It is common in Middle East and European countries, especially in Iran, Afghanistan, Türkiye, and Bulgaria. Due to the pharmacological effect of the Rose, it is accepted as an important medicinal and aromatic plant. Besides, it is also widely used in the food and cosmetic industry. Studies have shown that the leaves and fruits of rose have health-supporting properties due to their anti-cancer, anti-inflammatory, anti-mutagenic, anti-microbial, and anti-depressant properties.

R. damascena Mill., *R. gallica* L., *R. moshata* Herrm, and *R. centifolia* L. are the four main *Rosa* species that are used for the production of essential oil in the industry all over the world. Different significant products, including concrete, water, oil, and absolute, are extracted from oil-bearing rose (Koksal et al., 2015).

Previous reports demonstrated over 400 volatile compounds which are obtained from the floral scent of various rose cultivars. It is well known that the main components of perfumery rose oil are linalool, citronellol, nerol, and geraniol. Volatiles reported from *Rosa* spp are classified into five major compounds: terpenes, esters, alcohols, acids, and others (Lavid et al., 2002). So far, volatile compounds of *Rosa damascena* Mill. of rose oil have been studied by some research groups (Babu et al., 2002; Rezaei et al., 2003; Nowa et al., 2005; Koksal et al., 2015).

In recent research carried out in Van's old garden, a new garden rose was determined; *Rosa laxa* Retzius var. *harputense* T. Baytop. Because of prominent characteristics such as shape, color, and scent, it has been admired since ancient times (Alp and Koyuncu, 2008 and 2010). This study aimed to detect flavor profiles of newly signed rose genotypes using Headspace Solid Phase Micro Extraction "Gas Chromatography-Mass Spectrometry Techniques" (HS-SPME/GC/MS).

2. Material and Methods

2.1. Material

Flowers of *Rosa laxa* Retzius var. *harputense* T. Baytop that are grown in a traditional garden were used as a material in the study. *R. laxa* var. *harputense* is a shrubby plant of 1-4 meters in height. The flowers are milky white with a base of lemon yellow. Flowers have a very pleasant fragrance and can be formed both singly or in 2-6 groups, and are semi-double with about 2.5 cm diameter. The blooming period may last from May to October, based on the ecological conditions. The blooming period of *R. laxa* var. *harputense* in Van province is about 5 months where the flowers were collected for analysis. Flowers of used rose are highly decorative with a pleasant fragrance (Alp and Koyuncu, 2008). Flower samples were taken at the blooming time (June 2014) in Van province.

2.2. Methods

2.2.1. HS-SPME analysis of volatile compounds

The extraction of the volatile compounds was performed via HS-SPME technique. Fresh flowers were homogenized with 5M calcium chloride to release volatiles through HS-SPME. Polydimethylsiloxane/Divinylbenzene (PDMS/DVB) with 60 µm coated fused-silica fiber was used for absorbing the volatile compounds. The fiber was immersed through headspace and incubated for 30 min at 30ºC while extraction of volatiles was continued by stirring with a magnetic stirrer. After equilibration, the fiber was injected into the GC/MS port for 10 min set at 250ºC to thermally desorb the volatile compounds. The analyses were carried out in triplicate.

2.2.2. GC/MS analysis

A Perkin Elmer GC/MS (Clarus 600) equipped with HP-5 MS (30 m × 0.25 mm × 0.25 µm), fused-silica capillary column was used. The carrier gas was helium, with a flow rate of 1 ml/min. The injector temperature was 250ºC. The initial column temperature was set at 60ºC by increasing 5 ºC/min to 260 ºC and remained for 20 min. The mass spectra for the identification of volatiles were screened through Wiley and NIST GC-MS Libraries. The percentage of related picks for each identified compound was calculated using the total ion chromatograms equipped with the computerized integrator.

3. Results and Discussions

Gas chromatography is a popular basic analytical technique for analyzing volatile compounds in plants due to its efficiency, versatility, and sensitivity. However, different types of plant compounds only can be analyzed by GC when they are vaporized in normal conditions or can be vaporized at a suitable temperature. Solid Phase Micro Extraction (SPME) is also a fast and solventless technique for analyzing the volatiles between the headspace above the sample and a stationary phase coated on a fused-silica fiber (Zarifikhosroshahi et al., 2022). However, the volatile extraction and analysis methods may affect the apparent overall aroma composition. Moreover, new compounds can be formed before and during the analysis is possible (Kafkas et al., 2012; Guney et al., 2015; Keles and Erturk, 2021). The geographic and climatic conditions of the Van Region have revealed a unique garden culture, and R. laxa var. harputense grown in Van has taken an important place because of the smell in traditional Van garden culture. The percent of Volatile compounds in R. laxa var. harputense determined by HS-SPME/GC/MS technique are presented in Table 1.

Table 1. The percentage of volatile compounds of *R. laxa* var. *harputense*

Retention Time	Compounds	Content (%)
	Esters	
8.05	cis-3-hexenyl acetate	18.57
1.74	Methyl acetate	9.99
15.27	Benzyl acetate	9.19
9.28	Hexyl acetate	1.88
22.16	Methyl 9-octadecenoate	1.16
14.45	Citronellol acetate	0.93
2.18	Ethyl acetate	0.69
20.31	Methyl pentadecanoate	0.22
15.92	β-Phenethyl formate	0.11
	∑ Esters	**42.74**
	Alcohols	
17.24	Phenylethyl alcohol	26.59
16.83	Benzyl Alcohol	8.68
10.53	1-Hexanol	1.96
7.42	1-Penten-3-ol	0.06
14.22	Levomenthol	0.56
7.01	3-Hexenol	0.14
	∑Alcohols	**37.99**
	Aldehydes	
8.41	2-Hexenal	0.28
5.73	Hexanal	0.24
	∑Aldehydes	**0.52**
	Terpenes	
4.12	a-Phellandrene	0.59
13.707	Caryophyllene	0.27
19.88	a-Cadinol	0.18
7.92	D-Limonene	0.16
6.54	a-Pinene	0.16
8.92	3-Carene	0.07
	∑Terpenes	**1.43**
	Acids	
2.78	Formic acid	0.18
	Others	17.14

Totally, 24 volatile compounds, including 9 ester compounds, 6 alcohol compounds, 2 aldehyde compounds, 6 terpene compounds, 1 acid, and other compounds, were detected in Rose kesmiri (Table 1). Alcohols were detected as the major compounds, and the highest value was obtained from phenyl ethyl alcohol (26.59%) and benzyl alcohol (8.68%), respectively. Hethelyi et al., (2010) pointed out that phenyl ethyl alcohol was the principal component of fragrant rose flowers among the 13 rose varieties.

They also implied that the main compounds in white and pink rose varieties were phenyl ethyl alcohol and orcinol dimethyl ether. Phenylethanols show antioxidant, antiviral, and anti-inflammatory properties (Zarifikhosroshahi et al., 2021). The role of antioxidant capacity in the prevention of diseases such as cancer, cellular aging, cardiovascular diseases, and inflammation is proven (Keles, 2020; Gundesli et al., 2021, Ergun, 2021). Rusanov et al., (2011a) demonstrated that the main compound on flower extract of five oil-bearing rose genotypes was phenyl ethyl alcohol (7.99-8.44%) and followed by nonadecane, heneicosane, 9-nonadecene, heptecosane, tricosane, nonacosane, beta-citronellol, nerol, trans geraniol, n-heptedecane, pentacosane. Antonelli et al., (1997) reported that significant differences were seen in the composition of the major components in 24 old rose cultivars. Except for five cultivars, 2-phenyl ethanol was an almost major component in roses. Similarly, in this paper, we detected phenyl ethyl alcohol as the major component of *R. laxa* var. *harputense* of fresh petals. Piccone et al., (2004) also implied that 2-phenyl ethanol was the major volatile emitted of *Rosa damascena* var *semperflorens*. Rusanov et al., (2011b) demonstrated that major volatile compounds of *R. damascene* flower were designated by β-citronellol, trans-geraniol, phenyl ethyl alcohol, heneicosane, nonadecane, heptedecane. Similar results to previous papers were obtained in this study. However, various compounds were also detected in this paper. These differences can be due to using various extraction techniques. However, the aromatic compounds in ornamental plants also varies depending on the climatic conditions and the genotype (Georgieva et al., 2022).

Esters are one of the most valuable flavor compounds and cis-3-hexenyl acetate, Methyl acetate, Benzyl acetate, and acetic acid hexyl ester were identified as major ester compounds. Volatile components identified in the alcohols were determined to be phenyl ethyl alcohol, 1-hexanol, Levomenthol, 1-penten-3-ol, benzyl alcohol, 3-hexenol.

The results obtained in this study are in agreement with Joichi et al., (2005) in which Chinese rose species and varieties ascertained cis-3-hexenyl acetate and Citronellol acetate. The obtained results revealed that our species might come from China or Chinese origin. This study is important due to the first report in our country on *R. laxa* var *harputense*.

Conclusion

Rosa is a kind of cut-flower used in the cosmetic industry due to having a strong odor. As far as we know, there is no study on the volatile compounds of Kismiri Rose. Different numbers of volatile compounds were detected in Kismiri Rose flowers from Türkiye. Phenylethyl alcohol, cis-3-hexenyl acetate, and benzyl alcohol were the main aroma components for the studied rose flower. Due to increasing demands for cosmetic product besides natural ones versus synthetic perfumes, investigating flower scenes from naturally grown plants are of interest. However, agricultural aspects need plants that have various applications, such as medicine and landscape. Therefore, this study may be a light for achieving these purposes in the future because it is unavoidable to transform our genetic resources into economic values and to ensure the sustainable use of these resources.

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