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CHEMICAL INVESTIGATION OF THE VOLATILE COMPONENTS OF SHADE-DRIED PETALS OF DAMASK ROSE (*ROSA DAMASCENA* MILL.)

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Abstract - Roses are always appreciated because of their inimitable aroma, many uses and of course their beauty. In addition to the different damask rose (*Rosa damascena* Mill.) products (oil, water, concrete, absolute, gulkand etc.), its dried petals are also used for various health purposes. The hydrodistilled volatile oil and water of shade-dried damask rose petals were investigated by GC and GC-MS. The predominant components of tThe essential oil and rose water were aliphatic hydrocarbons (56.4 and 46.3%), followed by oxygenated monoterpenes (14.7 and 8.7%). The main aliphatic hydrocarbons of the essential oil and rose water were heneicosane (19.7 and 15.7%), nonadecane (13.0 and 8.4%), tricosane (11.3 and 9.3%) and pentacosane (5.3 and 5.1%) while the content of 2-phenyl ethyl alcohol was 0.4% and 7.1% in the essential oil and rose water, respectively. The chemical composition of the dried rose petal volatiles is quite different from fresh flower volatiles.

Key words: Rosa damascena Mill. var. 'Noorjahan', shade dried petals, essential oil, rose water, composition, aliphatic hydrocarbons

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INTRODUCTION

The damask rose (*Rosa damascena* Mill.) is the most important rose species used to produce rose oil, water, concrete and absolute which are valuable and important base materials for the perfume and cosmetic industry (Ayci et al., 2005). The total production of rose oil is approximately 5 metric tons, with Bulgaria and Turkey being the major producers followed by Morocco, Egypt, China, Russia, Iran and India. At present, mainly four species of rose are used for the production of rose products of perfume quality: *Rosa damascena, Rosa moschata* Herrm, *Rosa centifolia* and *Rosa gallica* (Tucker and Maciarello, 1988). However, rose oil obtained from *Rosa damascena* is traditionally preferred (Shawl and Adams, 2009). In the Indian system of medicine, various rose preparations are used as an astringent, tonic, mild laxative, antibacterial agent and in treatment of sore throat, enlarged tonsils, cardiac troubles, eye disease, gall stones, for their cooling effect and as a vehicle for other medicines (Hunt, 1962; Kaul, 1998; Schweisheimer, 1961). Essential oil from the rose is reported to have analgesic and antispasmodic effects (Basim and Basim, 2003; Libster, 2002). In addition, anti-HIV, anti bacterial and hypnotic activities of rose extract/isolates have been reported (Basim and Basim, 2003; Mahmood et al., 1996; Rakhshandah et al., 2007).

The fresh damask rose petals possess a very small quantity of essential oil. One kg of rose oil can be obtained from about 3,000 kg of rose petals (Baser, 1992). Because of the low oil content and the lack of natural and synthetic substitutes, rose oil is one of the most expensive essential oil in the world markets. The chemical composition of rose oil, rose water, concrete and absolute has been investigated in India and abroad (Agarwal et al., 2005; Ayci et al., 2005; Aydinli and Tutas, 2003; Eikani et al., 2005; Gupta et al., 2000; Lawrence, 2003; Shawl and Adams, 2009). Essential oil composition is varied over the flower stages, flower parts, and the harvesting period (Mihailova et al., 1997; Verma et al., 2011).

In addition to the various uses of rose oil, dried rose petals are also used for various purposes. Its intake as food has an important medicinal use as it can solve problems of the digestive system (Haghighi et al., 2008). In addition to this, dried rose petals are also be used for skin care and the preparation of Gul-e-Roghan for making hair oils. A literature survey revealed that significant work has been done on the damask rose: however, information on the chemical composition of dried rose petals is meagerlimited. Therefore, the aim of the present study was to characterize the volatile aroma chemicals that remaining in rose petals even after complete shade drying.

MATERIALS AND METHODS

Plant material and isolation of volatile components

Fresh flowers of *Rosa damascena* var. 'Noorjahan' were collected in the month of May, 2009, from an experimental field of the Central Institute of Medicinal and Aromatic Plants, Research Centre, Purara, Uttarakhand. The experimental site is located at an elevation of 1,250 m and has a temperate climate. The flowers were shade-dried at room temperature till moisture removal had taken place (79.3%). 60 g shade-dried rose petals were hydrodistilled with 1.5 l of water for 4 h to prepare 800 ml of rose water. The rose water was extracted with hexane to trap the present compounds. The organic layer was separated and dried over anhydrous sodium sulphate to remove residual moisture, if any. The solvent was evaporated under reduced pressure (35°C) to obtain a concentrated volatile fraction. In the second part, 145 g of rose petals were hydrodistilled in a Clevenger apparatus for 3 h to obtain the essential oil. The rose water volatiles and essential oil obtained in this way were stored at -5°C prior to analysis.

Essential oil and rose water volatiles analyses

Gas chromatography (GC) analyses of the rose petal volatiles was carried out on a Nucon gas chromatograph model 5765 equipped with FID and DB-5 (30 m × 0.32 mm; 0.25 µm film coating) fused silica capillary column. Oven temperature programming was done from 60-230°C at 3°C/min. Hydrogen was the carrier gas at 1.0 ml/min. The injector and detector temperatures were 220°C and 230°C, respectively. The injection volume was 0.02 µl neat (syringe: Hamilton 1.0 µl capacity, Alltech USA) and the split ratio was 1:30. Gas chromatography-mass spectrometry (GC-MS) analysis of the essential oil sample was carried out on a PerkinElmer AutoSystem XL GC interfaced with a Turbomass Quadrupole Mass Spectrometer fitted with an Equity-5 fused silica capillary column (60 m × 0.32 mm i.d., film thickness 0.25 µm) The oven temperature was programmed from 60-210°C at 3°C/min using helium as the carrier gas at 1.0 mL/min. The injector temperature was 210°C, injection volume 0.1 µl prepared in *n*-hexane (dilution 10%), split ratio 1: 40. MS were taken at 70 eV with a mass scan range of 40-450 amu and scan rate 1 sec with an interscan delay of 0.5 sec.

Identification and quantification of the components

Constituents were identified on the basis of a Retention Index (RI), determined with reference to a homologous series of *n*-alkanes, C_9-C_{24}, under identical experimental conditions, co-injection with standards (Aldrich and Fluka) or known essential oil constituents, MS Library search (NIST/EPA/NIH version 2.1 and WILEY registry of MS data 7th edition), by comparing with the MS literature data (Adams, 2007). The relative amounts of individual components were calculated based on the GC peak area (FID response) without using a correction factor.

Table 1. Chemical composition of the volatile components of shade-dried petals of *Rosa damascena* var. 'Noorjahan'

RI	Compound	Class	Content (%)	
			A	**B**
938	α-Pinene	MH	0.1	0.3
948	Benzaldehyde	BC	t	0.1
981	β-Pinene	MH	-	0.1
989	β-Myrcene	MH	0.2	0.2
1019	α-Terpinene	MH	t	t
1023	*p*-Cymene	BC	0.6	0.6
1047	(*Z*)-β-Ocimene	MH	t	-
1053	(*E*)-β-Ocimene	MH	0.3	-
1082	Terpinolene	MH	0.1	0.1
1099	Linalool	OM	0.5	0.7
1103	*n*-Nonanal	OA	0.4	0.4
1106	2-Phenylethyl alcohol	BC	0.4	7.1
1150	Citronellal	OM	0.1	0.2
1158	Nerol oxide	OM	0.2	0.1
1162	Isomenthone	OM	0.2	0.3
1175	Terpinen-4-ol	OM	0.1	0.2
1187	α-Terpineol	OM	0.1	0.2
1228	Citronellol	OM	7.1	2.2
1233	Nerol	OM	0.1	-
1236	Neral	OM	t	0.1
1252	Geraniol	OM	4.1	2.5
1259	Linalyl acetate	OM	t	t
1272	Geranial	OM	0.1	t
1274	Citronellyl formate	OM	0.2	0.3
1300	Geranyl formate	OM	0.5	1.5
1319	Methyl geranate	OM	t	-
1343	δ-Elemene	SH	0.1	-
1352	Citronellyl acetate	OM	0.1	0.3
1369	Neryl acetate	OM	0.4	-
1380	Geranyl acetate	OM	0.8	0.1
1396	β-Elemene	SH	0.1	-
1400	Methyl eugenol	BC	t	0.1
1416	β-Caryophyllene	SH	t	-
1424	β-Copaene	SH	0.1	0.2
1439	α-Guaiene	SH	-	0.1
1442	(*Z*)-β-Farnesene	SH	0.3	0.1
1450	α-Humulene	SH	0.3	0.2
1472	Geranyl propionate	OM	t	-
1476	Germacrene-D	SH	t	-
1488	β-Selinene	SH	0.1	-
1496	α-Selinene	SH	t	t
1500	Pentadecane	AH	-	0.2
1502	α-Bulnesene	SH	0.4	0.1
1522	δ-Cadinene	SH	-	0.1
1524	Citronellyl butyrate	OM	0.1	-
1577	Caryophyllene oxide	OS	0.1	0.1
1600	Hexadecane	AH	0.1	0.4
1619	10-*epi*-γ-Eudesmol	OS	0.1	0.1

Table 1. Continued

RI	Compound	Class	Content (%)	
1673	Tetradecanol	OA	0.1	-
1700	Heptadecane	AH	0.6	0.5
1720	(2*E*,6*E*)-Farnesol	OS	0.4	0.3
1800	Octadecane	AH	0.2	0.9
1885	1-Nonadecene	AH	1.6	0.8
1900	Nonadecane	AH	13.0	8.4
1975	1-Eicosene	AH	0.1	0.1
2000	Eicosane	AH	2.5	2.4
2100	Heneicosane	AH	19.7	15.7
2200	Docasane	AH	1.1	1.4
2300	Tricosane	AH	11.3	9.3
2400	Tetracosane	AH	0.9	1.1
2500	Pentacosane	AH	5.3	5.1
	Class composition			
	Monoterpene hydrocarbons (MH)		0.7	0.7
	Oxygenated monoterpenes (OM)		14.7	8.7
	Sesquiterpenes hydrocarbons (SH)		1.4	0.8
	Oxygenated sesquiterpenes (OS)		0.6	0.5
	Benzenoid compounds (BC)		1.0	7.9
	Aliphatic hydrocarbons (AH)		56.4	46.3
	Oxygenated aliphatics (OA)		0.5	0.4
	Total identified		75.3	65.3

RI: Retention indices calculated on DB-5 column; **A:** hydrodistilled essential oil of shade dried rose petals
B: hexane extract of rose water prepared from shade-dried rose petals

RESULTS AND DISCUSSION

The hydrodistillation of shade-dried rose petals gave 0.12% essential oil. The essential oil and hexane extract of rose water were analyzed by GC and GC-MS and the results are summarized in Table 1. A total of fifty-seven components, representing 75.3% of the essential oil, and forty-eight components representing 65.3% of the rose water, were identified. The rose oil and water both were dominated by aliphatic hydrocarbons (56.4 and 46.3%, respectively). The representative aliphatic hydrocarbons of the essential oil and rose water were heneicosane (19.7 and 15.7%), nonadecane (13.0 and 8.4%), tricosane (11.3 and 9.3%), pentacosane (5.3 and 5.1%) and eicosane (2.5 and 2.4%). Oxygenated monoterpenes were only 14.7% and 8.7% in the essential oil and rose water, respectively. The main oxygenated monoterpenes of the rose oil were citronellol (7.1%), geraniol (4.1%), geranyl acetate (0.8%), linalool (0.5%), geranyl formate (0.5%) and 2-phenyl ethyl alcohol (0.4%). However, the main oxygenated monoterpenes of the rose water were 2-phenyl ethyl alcohol (7.1%), geraniol (2.5%), citronellol (2.2%), geranyl formate (1.5%) and linalool (0.7%).

As far as the essential oil and rose water compositions of fresh flowers of *R. damascena* are concerned, Verma et al. (2011) found citronellol (15.9-35.3%), geraniol (8.3-32.2%), nerol (4.0-9.6%), nonadecane (4.5-16.0%) and heneicosane (2.6-7.9%) to be the major components of the essential oil, while 2-phenyl ethyl alcohol (66.2% - 80.7%), citronellol (1.8% - 5.5%) and geraniol (3.3% - 7.9%) were the major components of the rose water. This indicated that fresh and dried rose petals differ considerably in the composition of their volatile components. Good quality rose oil should possess a higher amount of monoterpene alcohols and a lower amount of alkanes (Baser, 1992). However, this criterion was not fulfilled by the volatile oil/extracts of the dried rose flowers/petals. Thus, on the basis of this study it could be concluded that the volatile oil or water of shade-dried rose petals cannot be substituted by rose oil and water prepared from fresh flowers. However, shade-dried rose petals can be utilized as a good source of aliphatic hydrocarbons.

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