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Research Article

Qualitative and quantitative study of rose oil from different regions of Saudi Arabia using hydro distillation technique for extraction and their comparison

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Abstract

One of the basic utilizations of rose flowers is the production of essential oil for the medicinal, cosmetic, food industry, perfumery, and some other purposes. In this research article, the two samples of *Rosa damascena* var. trigintipetala from two different places Alhada and Alshifa of Taif region of Saudi Arabia were studied for the yield of oil using the hydro-distillation technique of extraction along with the quality of oil in relation to the proportion of citronellol and geraniol. Also, the comparison of fresh flowers with non-fresh (used) flowers were also made to consider the effectiveness of extraction techniques. The fresh samples from Alhada (A1) were found to give double the yield (0.5 ml/kg) of essential oil than samples from Alshifa (B1) (0.25 ml/kg). The fresh samples from Alshifa were found to contain a better amount of citronellol (17.55 %) and geraniol (1.014%) than Alhada which indicate that the oil production is more in the plane and hot climatic conditions while the geraniol and citronellol content is influenced by high altitude and cool climate. Alhada samples showed the presence of 34 compounds including alkanes, monocyclic, and acyclic isoprenoids, and out of these 25 were identified. The compounds ranged from C_8-C_{23} while Alshifa samples possess 52 compounds and out of which 43 were identified ranging from C_{10} -C_{23}. The extraction of oil from exhausted flowers suggests that the extraction techniques used on a commercial scale in the Taif region need to be refined to increase the production and eventually the revenue.

1. Introduction

Roses are woody plants with creeping, bushy, shrub like, or climbing habits. Stems are often thorny, creeping, climbing, or erect, with alternating leaves, 3 to 20 cm long, and carrying 5 to 7 often serrated leaflets. Rosa is one of the major economically important genera of ornamental horticulture and the area under cultivation continues to expand [1]. Its common name **is** given to the thorny shrubs and climbing vines of the genus Rosa in the Rosaceae family [2]. The flowers vary in size and shape and are usually large and showy, in colors ranging from white to yellows and reds [3]. More than 200 Rosa species have been recorded throughout the world, but only a few of them have found application as essential oil crops [4]. Identification of Rose species is crucial to maximize revenue and protect the crop from vulnerable diseases. The main distinguishing methods to identify high-quality Rose species used so

far to date are morphology and Genotyping. Some researchers regard Genotyping as the most precise method of identification. Global floriculture production statistics says that the worldwide market for the flower is expected to grow roughly 6.3% over the next five years, reaching $57.4 Billion USD in 2024, up from $42.4 Billion USD in 2019 [5]. The top 10 countries which are well known for exporting the flowers are listed in Table 1.

Table 1. Top 10 countries for exporting flowers for bouquet.

Rank	Exporter	Flower Bouquet export (US$)	2018-2019 (%)
1	Netherlands	$4.6 billion	+1.8%
2	Colombia	$1.4 billion	-3.7%
3	Ecuador	$879.8 million	+3.3%
4	Kenya	$709.4 million	+23.4%
5	Ethiopia	$241.3 million	+1.1%
6	Belgium	$153.4 million	+1.7%
7	China	$119.9 million	+10.9%
8	Malaysia	$113.1 million	-2.9%
9	Italy	$105.7 million	-2.5%
10	Belarus	$82.6 million	+16.4%
	Middle East		
21	Turkey	$35 million	+5%
41	Morocco	$7,9 million	+9.5%
53	Egypt	$3,039 million	-12.9%
74	Iran	$274,000	-98.6%
88	Oman	$64,000	-65.8%
89	Tunisia	$58,000	-87.8%
99	Syria	$17,000	+88.9%
101	Kuwait	$10,000	-58.3%
109	Saudi Arabia	$5,000	-97.6%

Rose cultivation in Saudi Arabia has a traditional history of more than 300 years, the rose oil and rose water have been produced mainly for use in local cultural and religious activities as well as in the local market of perfumery and cosmetics. The Taif rose, cultivated in Saudi Arabia, is a subspecies of *R. damascena* (*Rosa* × *damascena* trigintipetala) with a very close resemblance to the famous Kazanlak rose of Bulgaria (*Rosa damascena* Miller var. trigintipetala Dieck). It is believed that the Taif rose was brought to Saudi Arabia from the Balkans by Turks in the XIV century [6]. The valley of Taif is located in the southwestern region of the Kingdom of Saudi Arabia. It represents a highland (1700-2500 m. altitude) with favorable temperatures and plentiful groundwater, well-established irrigation systems, and fine topsoil. The flowering season is in March-April. In the western part of the valley, the roses are cultivated in two rose-growing micro-regions Alshifa and Alhada, of Taif. The traditional technology of breeding, propagation, and processing is preserved unchanged for centuries [7].

The characteristics of a particular rose variety can only be fully understood by considering the rose class to which it belongs. There are many ways to classify roses, but the most common is the system now used by the American Rose Society [8], which uses the following categories (Table 2)

Table 2. Category of roses according to American Rose Society.

Major Category	Subcategory	Examples
Modern Garden Roses	Climbing Roses	Altissimo
	Grandiflora Roses	Cherry Parfait
	Hybrid Tea Roses	Dark Night
	Groundcover Roses	Flower Carpet Amber
	Polyantha Roses	The Fairy
	Rambler Roses	American Pillar
	Shrub Roses	Peachy Knock Out
	Miniature Roses	Lemon Delight
Old Garden Roses	Damask Roses	Duc de Cambridge
	Gallica Roses	Charles de Mills
	Tea Roses	Safrano
	Centifolia Roses	Petite de Hollande
	Alba Roses	Pompon Blanc Parfait

According to oil production and its quality *Rosa damascena* (Damask rose) such as Roses growing in Taif, Saudi Arabia also, *R. gallica* and *R. centifolia* are the most important species, producing high-value aromatic oil, which is used in the pharmaceutical, flavor, and fragrance industries [9]. The essential oil from Damask rose is the most highly valued on the world markets [10]. The rose petals generally contain very little essential oil in comparison with other essential oil plants. Generally, 1 kg of rose oil can be obtained from 3000-4000 kg of rose petals. Roses (*Rosa* spp.) have a reputation for being difficult to grow, and some hybrid tea roses truly are prima donnas. Many rose varieties, however, are hardy, long-lived plants that can grow in a variety of conditions, including full sun to partial shade. Most rose bushes need at least six to eight hours of sunlight each day to bloom and perform well [11]. Taif has always been known for its many valleys, qualitative irrigation systems, also known as very fertile land. This is one reason it is the center of an agricultural area known

for its vegetables and crops; more specifically its grapes and honey. Because of its cooler climate conditions, these temperatures attribute to the perfect conditions for growing '*Rosa damascena* var. trigintipetala', and 'The Taif Rose' [12]. Distillation is the most widely used and most economical method of extracting rose essential oils. In distillation, the rose flowers are heated in stills, either by placing it in water which is brought to the boiling point, or passing steam through it. The essential oil molecules and steam are carried along a pipe and channeled through a cooling tank (condenser), where they return to the liquid form (extract) and are collected in a special tank (Florentine). The emerging liquid is a mixture of oil and water, Essential oils which are lighter than water will float on the surface in the Florentine flask [13]. The major volatile oil contents include terpene alcohols citronellol/geraniol (C/G) ratios 45 to 72%., the phenols eugenol (1%) and methyl eugenol, the aldehydes citral, up to 3% phenylethyl., traces of sesquiterpenes have also been found with azulene-like qualities, it also contains about 2% of sesquiterpene alcohol and farnesol. Some ingredients are also important for perfumeries such as esters, nerol, the nonyl aldehyde, and trace constituents, such as carvone and rose-furan [14]. The Physical and chemical properties of rose oil as described by Tobyn and Denham [14] are: color: rose oil is a pale yellow with an occasional green tinge, it has a sweet, yet spicy floral aroma, solidify at temperatures between 16-23 °C, congealing point: +18°C to 23.5°C, specific gravity: +At 20°C /15°C: .856 to .870, optical rotation: -1o to -4o, refractive index: At 25°C: 1.452 to 1.466, solubility in alcohol: 90% alcohol showed slightly soluble with turbid mixture, ester value, mg KOH/g from 7,0 to 24, relative density, g/cm3 from 0,848 to 0,880.

2. Materials and methods

2.1 Plant Material

Samples (3 kilograms each) of rose, *Rosa damascena* (Damask rose) were collected from two different locations within Taif farms, Saudi Arabia. The samples were authenticated by a taxonomist in the Herbal garden affiliated with the Department of Pharmacognosy, College of Pharmacy, King Saud University. The validity and health of the rose samples were checked by an engineer from the Directorate, Ministry of Environment, Water and Agriculture (MEWA), Saudi Arabia. Voucher specimens were deposited in the herbarium of the college for future needs. Samples were refrigerated just after the collection and were processed spontaneously in the lab. The samples (3 kilograms each) of exhausted flowers were taken from distilleries of respective regions [Alhada (A2) & Alshifa (B2)].

2.2 Solvents and reagents

All solvents used were HPLC grade and purchased from Sigma (or another authentic vendor). Reagents of chemical synthesis grade were used and purchased from the most authentic available local vendor.

2.3 Methods

2.3.1 Hydro-distillation by Clevenger

500 g of fresh roses from each region (A and B) were extracted by distillation to give Oils (A1 and B1). Moreover, 500 g of non-fresh roses from the two regions were also extracted to give oils (A2 and B2). All samples were compared with two standard oils from the same regions (purchased from Alhada and Alshifa).

2.3.2 Gas chromatography-mass spectrometry (GC/MS)

The GC/MS analysis was performed on an Agilent 7890A GC System Plus gas chromatography coupled with G3171A Mass Selective Detector (Agilent Technologies). Silica capillary columns—an ultra-inert non-polar DB-5ms UI with 30 m column length, 0.25 mm i.d., 0.25 μm film thickness, were used. Helium (99.999%) was used as a carrier gas at a constant flow rate of 1.2 mL/min, with 0.2 μL injection volume in a split less mode. Mass selective detector operated in electron impact ionization (EI) mode at 70 eV electron energy, the ion source temperature was set to 230°C, and the quadrupole temperature was 150°C. The mass scan range was 30-450 m/z.

The sample of rose oil's standards A and B and their samples of different origins were prepared in hexane. The prepared samples were injected as per the above chromatographic condition. The chromatographic condition was optimized concerning the temperature, flow of carrier gas, and injection mode. The best results were resolved at a 1.2 mL flow rate and in split-less mode. The inlet temperature was tuned and fixed to 60°C. The temperature ramping was also analyzed and set to obtain a complete separation [15].

3. Results and discussion

As far as the production of rose oil is concerned Turkey and Bulgaria are among the top producers in the world. The possibility of 96.92% of rose flowers can be processed using modern techniques. The characteristic components of rose oil are acyclic monoterpene alcohols, geraniol (up to 75%), citronellol (20%) and nerol (20%), and long-chain hydrocarbons like nonadecane or heneicosane (up to 10%) [16]. The oil samples from the two places were collected and compared for their essential oil content. The yield of oil from fresh samples of Alhada (A1) was found to be 0.5 ml/kg while from fresh samples of Alshifa (B1) 0.25 ml/kg. However, the oil production from non-fresh roses of Alhada (A2) was found to be 0.22 ml/kg while the counterpart (B2) from Alshifa was 0.05 ml/kg.

As far as the citronellol and geraniol contents are concerned the Alshifa samples took the lead. The fresh samples (B1) of Alshifa contain citronellol and geraniol at 17.55% and 2.942%, respectively while non-fresh samples contain 2.229% of citronellol and 0.132% geraniol (Table 3). The fresh Alhada samples contain 16.75% citronellol and 1.014% geraniol while the non-fresh sample possesses 2.229% citronellol and 0.132% geraniol (Table 4).

There are differences related to the presence of some unfamiliar compounds in trace amounts at the region of 8-15 mins and some compounds are missing in one sample and present in another sample. Therefore, the climate impact is one of the major factors for the diverse chemical profile of rose oil samples.

In comparing both samples, Alhada was found to be better than Alshifa for citronellol content. As far as oil production is concerned Alshifa was found to be more productive than Alhada (Table 5). The citronellol content in A2 samples indicates the scope for improvement in oil extraction techniques. Also, the higher citronellol content in Alhada samples indicates that cold and higher altitude is a favorable factor for citronellol production in roses. As far as the quality of the oil is concerned, both samples A1 and B1 exhibited only 50% of the quality of their respective standards (A and B) in terms of citronellol. The geraniol content in Alshifa samples was found to be

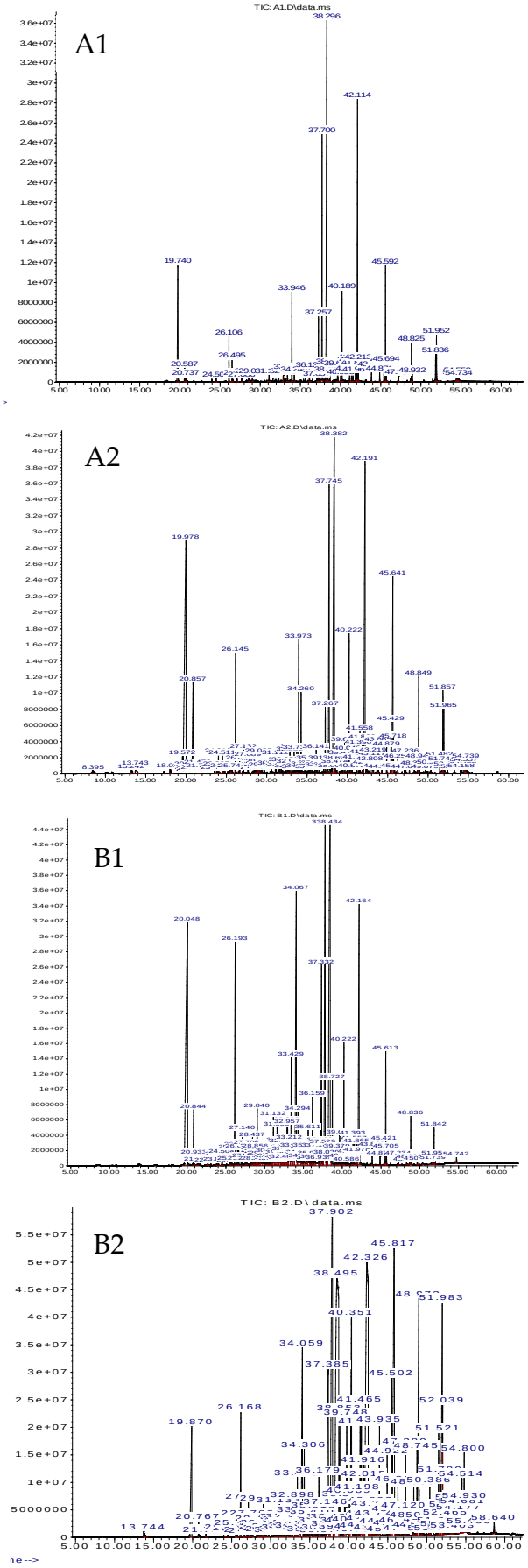

Figure 1. Comparative analysis of all the four samples of fresh as well as exhausted rose flowers

Table 3. Rose Oil Standard A, Sample A1 and Sample A2.

S.No.	Components	Standard (%) A	Sample (%) A1	Sample (%) A2	R.T.	Mol. Wt.
1	Citronellol	30.212	17.55	8.541	20.259	156.151
2	Phenethyl alcohol	4.351	0.32	0.453	13.885	122.073
3	(E)-Geraniol	16.118	0.222	2.942	21.255	154.136
4	*n*-Nonadecane	10.269	27.735	17.227	38.358	268.313
5	*n*-Heneicosane	3.572	16.095	12.197	42.129	296.344
6	Z-5-Nonadecene	3.398	11.482	8.049	37.723	266.297
7	*n*-Eicosane	0.936	0.208	2.627	40.201	282.329
8	α -Caryophyllene	0.537	0.173	0.335	27.721	204.188
9	*n*-pentadecane	0.729	0.223	0.31	29.031	212.25
10	*n*-heptadecane	2.125	3.452	2.475	33.986	240.282
11	Methyl eugenol	1.875	1.849	2.5	26.17	178.099
12	Farnesyl alcohol	1.73	0.34	1.294	34.295	222.198
13	β -Fenchol	2.648		0.228	18.091	154.136
14	cis-Geraniol	2.17		0.863	19.676	154.136
15	(-)-Germacrene D	1.034		0.077	28.453	204.188
16	*n*-Tricosane	0.706		4.522	45.596	324.376
17	β -Caryophyllene	0.697		0.24	26.668	204.188
18	t-Muurolol	0.455		0.417	32.888	222.198
19	(+-)-Linalool	2.797		0.16	13.342	154.136
20	Geranyl acetate	1.655			25.512	196.146
21	3-Allyl-2-methoxyphenol	1.988			24.568	164.084
22	4-Terpineol	0.677			17.284	154.136
23	α -Guaiene	0.672			27.149	204.188
24	Linalool L	2.691			13.416	154.136
25	Nerol	0.509			24.843	154.136

Table 4. Rose Oil Standard B, Sample B1 and Sample B2.

S.No.	Components	Standard (%) B	Sample (%) B1	Sample (%) B2	R.T.	Mol. Wt.
1	Citronellol	28.49	16.75	2.229	20.036	156.151
2	Phenethyl alcohol	3.168		0.132	13.788	122.073
3	(E)-Geraniol	18.484	1.014	0.132	21.066	154.136
4	*n*-Nonadecane	8.634	17.191	15.923	38.283	268.313
5	*n*-Heneicosane		6.676	13.792	42.089	296.344
6	Methyl eugenol	1.176	4.672	1.229	26.124	178.099
7	*n*-heptadecane	2.156	8.395	3.616	33.963	240.282
8	*n*-Eicosane	0.737	1.768	3.766	40.189	282.329
9	Z-5-Nonadecene	3.823	9.969	9.591	37.7	266.297
10	β -Caryophyllene	0.654	0.293	0.172	26.645	204.188
11	α -Caryophyllene	0.593	0.299	0.174	27.704	204.188
12	(-)-Germacrene D	0.546	0.398	0.118	28.436	204.188
13	*n*-Tricosane	0.482	1.713	7.687	45.585	324.376
14	t-Muurolol	0.317	0.759	56.70%	32.882	222.198
15	n-Octadecane	0.202	0.935	0.608	36.138	254.297
16	β - Elemene	0.195	0.232	7.90%	25.747	204.188

Table 4. (*continued*)

S.No.	Components	Standard (%) B	Sample (%) B1	Sample (%) B2	R.T.	Mol. Wt.
17	E-farnesol	0.135	0.187	0.048	30.582	222.198
18	α -Selinene	0.126	0.256	0.079	28.848	204.188
19	9-Eicosene	0.121	0.697	1.45	39.657	280.313
20	Behenic alcohol	0.113	0.287	1.088	41.551	326.355
21	*n*-Hexadecane	0.111	0.528	0.119	31.589	226.266
22	α -Guaiene	0.567	0.463		27.131	204.188
23	Eugenol	1.867	0.193		24.528	164.084
24	1-Nonadecene	0.124	0.227		41.86	266.297
25	(Z,E)-Farnesol	0.153		0.256	33.769	222.198
26	γ-Eudesmol	0.115		0.129	32.327	222.198
27	Farnesyl alcohol	1.344		0.642	34.272	222.198
28	Geraniol ester	1.232			25.472	196.146
29	Nerol	5.617			19.67	154.136
30	α -Terpineol	2.804			18.062	154.136
31	β-Linalool	5.549			13.29	154.136
32	n-Pentadecane	0.718			29.025	212.25
33	β -Myrcene	1.559			8.392	136.125
34	4-Terpineol	0.647			17.261	154.136
35	d-Limonene	0.461			10.086	136.125
36	β -Citral	0.404			21.547	152.12
37	α-Pinene	0.369			10.326	136.125
38	8-Heptadecene	0.351			33.403	238.266
40	cis-Citral	0.182			20.299	152.12
41	Nerol acetate	0.166			24.82	196.146
42	*n*-Eicosane	0.157			48.823	282.329

better than Alhada which signals that the geraniol production is more in the plane area and in warm climates rather than in cold climates and high altitudes. If we compare the geraniol contents in samples A1 and A2, again we need to improve the extraction techniques.

The other constituents which are abundant include phenethyl alcohol, nonadecane, *n*-henicosane, *n*-heptadecane, methyl eugenol, *n*-eicosane, farnesol etc. Standard A and B were obtained from the local industry after the rose flower extraction, and the used flower A2 and B2 were again extracted through the mentioned extraction procedure. The GC chromatogram of samples A2 and B2 (Figure 1) itself reveal the presence of significant number of constituents in reasonable quantity. As far as the oil production (% yield) is concerned the samples from plane and comparatively hotter region (Alhada-B) give better yield than samples from high altitude and cooler climate (Alshifa-A).

Table 5. Comparative yield of rose oil from four different samples

Sl. no.	Sample code	Weight	Oil qty (mL)	Yield (%)
1.	A1 …(Fresh-Alhada)	500g	0.250	0.05%
2.	A2 …(Used- Alhada)	500g	0.110	0.022%
3.	B1 …(Fresh-Alshifa)	500g	0.125	0.025%
4.	B2 …(Used- Alshifa)	500g	0.025	0.005%

4. Conclusions

The substantial presence of the essential components in the rose oil samples indicates that the extraction procedure of industry requires refinement to avoid wastage. Taif regions possessed a similar quality of oils with minimal differences especially in the region of the fatty acids 8-15 mins in the GC-MS chromatogram. This difference may be valuable to build a unique fingerprint for different Taif regions oils. The best Rose cultivar that produced the heaviest weight (and subsequently the better oil yield) is

Alshifa samples, however, in terms of citronellol content Alhada samples produced better compliance. The distillation waste was found to contain a lot of valuable content especially fatty acids which were found to be abundant and easy to be extracted.

Authors' contributions

Conceptualization, N.A.S. and M.F.A.; Methodology, B.A.S. and A.A.J.; Investigation, A.H. and O.N.; Writing- original Draft Preparation, N. A. S. and A. H.; Writing- Review & Editing, M.F.H and B.A.S.; Supervision, A.A.J. and N. A.S.; Funding Acquisition, N. A. S. and M.F.A.

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Conflicts of interest

The authors declare no conflict of interest.

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