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Research Article

**THE EFFECT OF EXTRACTION APPARATUS ON THE  
CHEMICAL COMPOSITION OF ESSENTIAL OIL FROM THE  
ENDEMIC DAMASK ROSE (*ROSA DAMASCENE* MILL) WITH  
AN ANTIOXIDANT EVALUATION****Mohammed H. Geesi<sup>1\*</sup>**Department of Chemistry, College Of Science and Humanities, Prince Sattam Bin Abdulaziz  
University, P.O. Box 83, Al-Kharj 11942, Saudi Arabia.**Abstract:**

*Rosa damascene Mill is one of the most important Rosa species and has an attractive flavor and fragrance. This research study appraises the effect of extraction techniques on the chemical composition and antioxidant attributes of essential oil from Rosa damascene Mill harvested from the Taif Region in Saudi Arabia. The essential oil extracted was done using stainless steel and copper-based distillation apparatus. A GC-MS analysis of the extracted essential oil revealed the presence of 20 and 28 chemical components, with citronellyl isobutyrate (22.44%) and citronellol (38.57%) identified as the principal components when stainless steel and copper apparatus was used for the purpose of extraction, respectively. The antioxidant activity of the tested essential oil from the Damask rose exhibited a concentration-dependent DPPH radical scavenging activity ( $R^2 = 0.980$ ) with  $IC_{50}$  1.68  $\mu\text{g/mL}$ .*

**Keywords:** *Rosa damascene Mill, Antioxidant, Essential oils, GC-MS***\* Corresponding author:****Mohammed H. Geesi<sup>1</sup>**Department Of Chemistry,  
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**INTRODUCTION:**

The *Rosa* genus from the *Rosaceae* family comprises 200 species and approximately 18,000 cultivars [1]. The *Rosa* species known as *Rosa damascena* Mill is one of the most important *Rosa* species.[2] It is also known as “Gol-e-Mohammadi” in Persian. *Rosa damascene* is an aromatic plant famous for its high-value essential oil[3-4]. *R. damascene* originated from Damascus, Syria, where it grows as a wild plant. Rose species are also distributed throughout the Middle East, China, India, Europe, and America, and are cultivated in other parts of the world for their high-quality rose essential oils [1-5]. A relatively high-quality rose essential oil is produced in the Taif Region in Saudi Arabia and in Bulgaria[5-6]. Moderate temperatures and humid air are the two main factors contributing to the production of high-quality rose essential oils when flowering. A temperate climate and an altitude of 300-1800 m are important requirements for the growth of this type of rose [7]. Rose genus essential oils and volatile products have been used as folk medicines due to their wide range of pharmacological activity [4]. Essential rose oil is economically important with a variety of applications in the perfume, cosmetics and scent industries [1]. Various rose products are traditionally used for heart disease, eye disease, sore throat and tonsillitis, and as laxatives and a main ingredient in other medicines [8-9]. Recently, *Rosa damascena* Mill was reported to have potential antioxidant, analgesic, antibacterial, hypnotic and antispasmodic effects [10-12]. Citronellol, nonadecane, geraniol, nerol, heneicosane, tricosane,

citronellal, citral, carvone, citronellyl acetate, eugenol, ethanol, farnesol, nonanol, nonanal, phenylacetaldehyde, phenylmethyl acetate, phenyl ethyl alcohol and phenyl geraniol are the main components which have been identified in rose oil by different agro-ecological varieties [13-17].The production of high-quality rose essential oil has a long history in the western province of Taif Region in Saudi Arabia, which has had a strong financial output for many years[5-8]. Saudi Arabia has a very low annual rainfall and the groundwater is highly saline; although *R. damascene* is well adapted to a wide range of environmental conditions, the composition and quality of its oil are affected by agro-ecological conditions [18-19]. The aim of the present work is to evaluate the effect of extraction apparatus on the chemical composition and antioxidant activity of hydro-distilled rose essential oil.

**EXPERIMENTAL:****Materials and methods**

The rose oil was given to us by Mr. Rashed Husain Al-Qorashei, who is the owner of the Rashed Husain Al-Qorashei factory, which produces the original fragrance of Taif roses. Freshly cut Damask roses (*Rosa damascene* Mill) were collected from the Taif Region in Saudi Arabia; then these roses were exposed for extraction using stainless steel and copper-based hydrodistillation apparatus with cooking gas as a heat source on an industrial level (Fig 1). The Damask rose material was exposed to hydro-distillation for approximately 10 h using a Clevenger-type apparatus (Fig 2).



Figure 1: The collected roses inside the stainless steel (left) and copper apparatus (right)



Figure 2. The stainless steel (left) and copper (right) apparatus used for extracting the *Rosa damascene* Mill

The collected oil (Fig. 3) was dried over anhydrous sodium sulfate ( $\text{Na}_2\text{SO}_4$ , 99%) in our laboratory in a desiccator; then it was filtered and preserved at  $-4^\circ\text{C}$  for the experimental work.



Figure 3: The rose oil

#### ***GC-MS characterizations of Rosa damascene Mill essential oil***

The GC-MS analysis of the *Rosa damascene* Mill was performed using a QP 2010/Ultra mass spectrophotometer (Shimadzu, Tokyo, Japan) with

AOC. 20i Auto-Injector. The components present in the rose essential oils were analyzed using a capillary column ( $30\text{ m} \times 0.25\text{ mm}$ ;  $0.25\text{ }\mu\text{m}$ ) with fused silica

Rtx-5 MS (RESTEK, Bellefonte, PA, USA) added. 1:4 v/v of oil and methanol were mixed and injected into the auto-injector at a temperature of 290 °C. Further, the components of the essential oil were authenticated by comparing the mass spectral data available in the NIST spectral library by co-injecting the pure standard [20-21].

### RESULTS AND DISCUSSION:

#### *Chemical composition of hydro-distilled essential oil from the Damask rose (Rosa damascene Mill)*

The fresh aerial parts of the Damask rose collected from the south western Taif Regions in Saudi Arabia were subjected to hydrodistillation using a stainless steel and copper pan. The Damask rose essential oils were analyzed using a GC-MS chromatogram On

using two different metal apparatus (stainless steel and copper) for the extraction, variations in the chemical composition and major components were found. When copper was used as an extraction apparatus, 28 components were detected, while only 20 components were identified using the stainless steel apparatus for extracting the rose oil (Tables 1 and 2). There was a noticeable difference in the chemical composition of the essential oil with regard to the extraction apparatus. Citronellol (**1**) (38.57%) and citronellyl isobutyrate (**2**) (22.44%) were found to be the principal components when the copper and stainless steel apparatus was used for the purpose of extraction (Fig. 4).

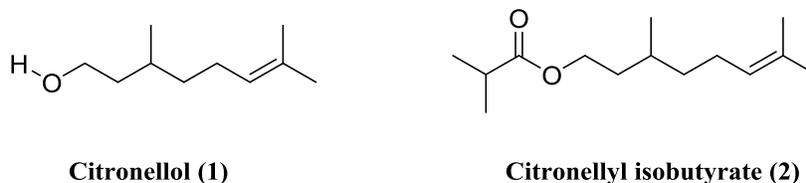


Figure 4. The structures of citronellol (1) and citronellyl isobutyrate (2)

The other main components detected also exhibited a significant variation with regard to the extraction apparatus. *Trans*-geraniol emerged as the second most valuable chemical component detected, with contributions of 19.20% for the stainless steel apparatus and 10.65% for the copper-based hydro-distillation apparatus (Fig. 5).

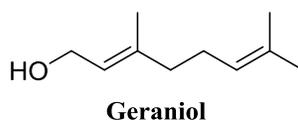


Figure 5: The structure of geraniol

Considerable differences were found in some chemical components such as  $\alpha$ -Pinene and  $\beta$ -Pinene during both extractions. The amount of  $\alpha$ -Pinene detected was 10.89% for the stainless steel derived extraction, whereas only 1.57% was detected when the copper apparatus was used for the extraction. Similarly, the amounts of  $\beta$ -Pinene were found to be 1.89 and 0.44%, respectively, in the stainless steel and copper directed extractions. Linalool and  $\beta$ -linalool were also detected at levels of 6.84% and 8.35% when copper and stainless steel were used for the extraction, respectively (Fig. 6) and (Fig. 7).

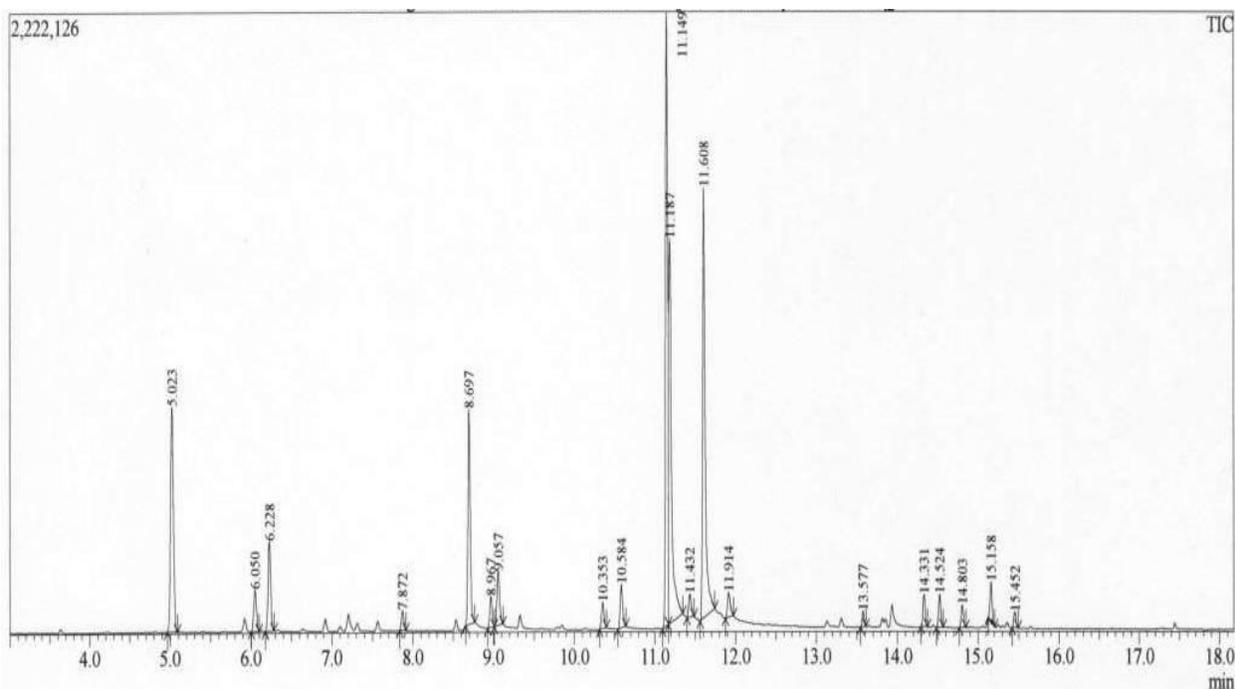


Figure 6. Typical GC-MS chromatogram of the Damask rose (*Rosa damascene* Mill) essential oil obtained from the extraction using the stainless steel apparatus

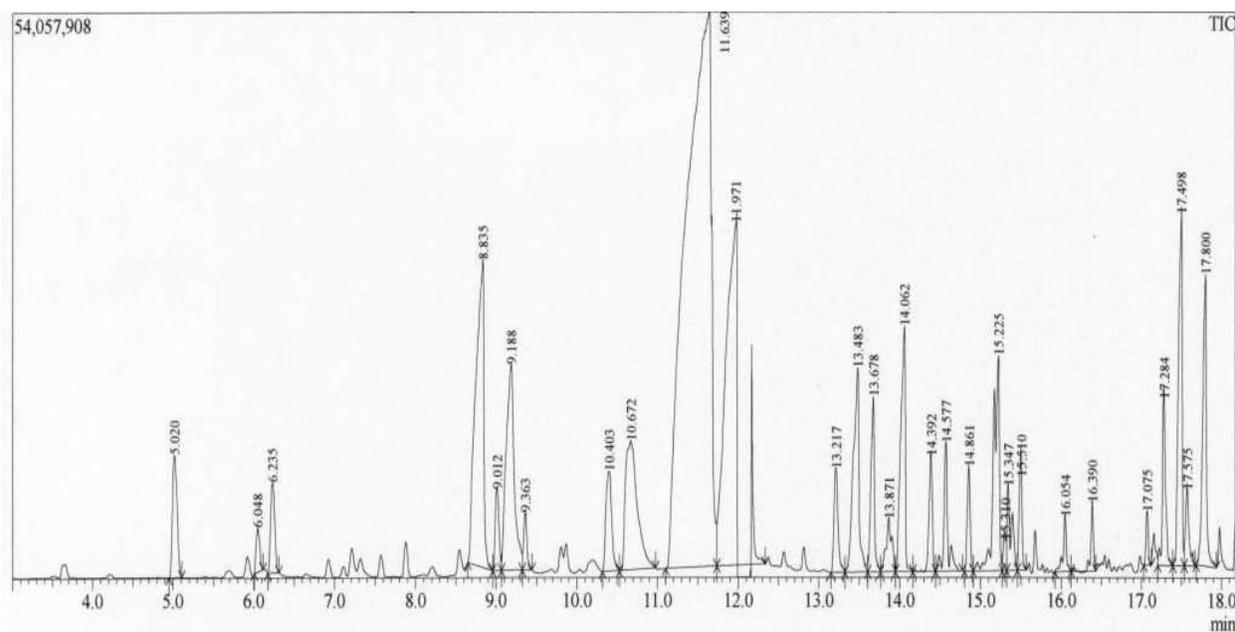


Figure 7. Typical GC-MS chromatogram of Damask rose (*Rosa damascene* Mill) essential oil obtained from the extraction using the copper apparatus

Table 1. Percentage chemical composition of hydro-distilled essential oil using the stainless steel extraction apparatus from the Damask rose (*Rosa damascene* Mill)

Essential oil constituent	RT <sup>a</sup>	RI <sup>b</sup>	% Composition
$\alpha$ -Pinene	5.023	948	10.89
$\beta$ -Pinene	6.050	943	1.89
$\beta$ -Myrcene	6.228	958	3.88
$\gamma$ -Terpinen	7.872	998	0.80
$\beta$ -Linalool	8.697	1082	8.35
Rose oxide	8.967	1114	1.24
$\beta$ -Phenethyl alcohol	9.057	1136	2.47
Terpinin-4-ol	10.353	1137	1.08
$\alpha$ -Terpeniol	10.584	1143	1.78
Citronellyl isobutyrate	11.149	1501	22.44
Neryl acetate	11.187	1352	18.22
$\beta$ -Citral	11.432	1174	1.02
<i>trans</i> -Geraniol	11.608	1228	19.20
Citral	11.914	1174	1.19
$\beta$ -Myrcene	13.577	958	0.51
Caryophyllene	14.331	1494	1.15
$\alpha$ -Guaiene	14.524	1490	1.18
$\alpha$ -Caryophyllene	14.803	1579	0.82
$\beta$ -copaene	15.158	1216	1.34
Azulene	15.452	1490	0.55

Table 2. Percentage chemical composition of hydro-distilled essential oil using the copper extraction apparatus from the Damask rose (*Rosa damascene* Mill)

Essential oil constituent	RT <sup>a</sup>	RI <sup>b</sup>	% Composition
$\alpha$ -Pinene	5.023	948	1.57
$\beta$ -Pinene	6.048	943	0.44
$\beta$ -Pinene	6.235	943	1.03
Linalool	8.833	1082	6.84
Rose oxide	9.012	1114	0.85
Phenethyl alcohol	9.187	1136	4.60
Rose Oxide	9.363	1114	0.50
Terpinine-4-ol(4-Terpinol)	10.403	1137	1.59
$\alpha$ -Terpeneol	10.672	1143	4.20
Citronellol	11.640	1179	38.57
Geraniol	11.970	1228	10.65
Rhodinol	13.217	1302	1.14
$\rho$ -Eugenol	13.483	1392	3.23
Geranyl acetate	13.678	1352	1.69
$\beta$ -Bourbonene	13.871	1339	0.84
Eugenol methyl ether	14.062	1361	3.05
Caryophyllene	14.392	1494	1.05
$\alpha$ -Guaiene	14.577	1490	1.38
$\alpha$ -Humulene	14.861	1579	0.83
$\beta$ -copaene	15.225	1339	3.37
2-Butenoic acid	15.310	1443	0.32
$\alpha$ -Farnesene	15.347	1458	0.99
Azulene	15.510	1490	0.92
(6 <i>E</i> -Nerolidol	16.054	1564	0.50
<i>n</i> -Cetane	16.390	1612	0.50
$\gamma$ -Eudesmole	17.075	1626	0.33
( <i>Z</i> )-7-Hexadecenal	17.283	2044	1.52
<i>n</i> -Ecosaine	17.497	1711	3.70
Tetraprenol	17.573	1710	0.76
<i>trans</i> -Farnesol	17.800	1710	3.04

<sup>a</sup>Chemical compounds are listed in their elution order from the Rtx-5 MS column.

<sup>b</sup>Retention indices relative to C9–C24 *n*-alkanes in the Rtx-5 MS column.

RT= Retention time

RI = Retention index

Nd: Not detected

Moreover, despite major differences in their chemical components, the minor components of the tested rose oils were also notably variable in relation to the type of extraction apparatus. A number of constituents detected in the steel vessel hydrodistilled essential oil were found to be absent in the copper-based vessel hydrodistilled oil. Therefore, it was concluded that the nature of the extraction apparatus/vessel has an important effect on the yield and chemical components of the isolated essential oils. Previously,  $\beta$ -Citronellol (30.24%) and *trans*-geraniol (20.62%) were reported to be major components of the essential oil from the Bulgarian *Rosa Damascene* Mill [6]. In one research study, essential oil from the Indian rose was found to contain citronellol with nerol (38.63%) and geraniol (26.50%) as its main components [17]. The results of the chemical composition of the *Rosa Damascene* Mill essential oil in the present analysis are reasonably comparable to those reported in the earlier literature [19-22].

#### **Total phenolic compounds (TPCs) and DPPH radical scavenging**

The concentration of total phenolics present in the *Rosa damascene* Mill essential oil was found to be 0.20 mg GAE/100g). A fairly similar TPC result was observed in the *Rosa Damascene* Mill extract [23]. In another research study, the TPCs were found to be higher than in our present data [24]. The DPPH radical scavenging power of the tested *Rosa Damascene* Mill essential oil was compared with standard ascorbic acid at different concentrations. The DPPH scavenging was evaluated by selecting oil concentrations within the range of 2.5-200  $\mu\text{g/mL}$ , while ascorbic acid showed similar activity at concentrations of 0.0195-5  $\mu\text{g/mL}$ . The results of both the test and the standard were found to be concentration dependent ( $R^2 = 0.980$ ) (Tables 3 and 4, Fig. 8). The  $\text{IC}_{50}$  values of the essential oil and ascorbic acid were found to be 147.75 and 1.68  $\mu\text{g/mL}$  (Tables 3 and 4). The DPPH scavenging ability of the *Rosa damascene* Mill may be attributed to the main component isolated in the oil such as citronellol and citronellyl isobutyrate [24].

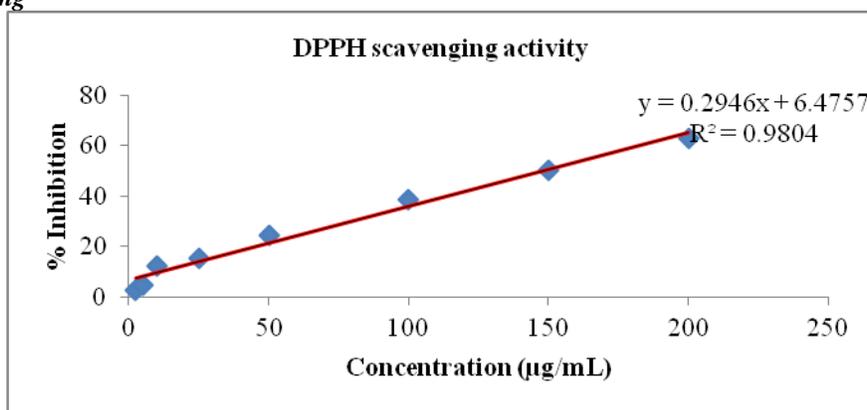


Figure 8. DPPH scavenging activity of the *Rosa Damascene* Mill

Table 3. % DPPH scavenging activity of the Damask rose (*Rosa damascene* Mill) essential oil

Conc. ( $\mu\text{g/ml}$ )	% DPPH Scavenging ( <i>Rosa damascene</i> Mill)	$\text{IC}_{50}$ $\mu\text{g/mL}$
2.5	2.580645	147.75
5	4.516129	
10	12.25806	
25	15.48387	
50	24.51613	
100	38.70968	
150	50.32258	
200	63.22581	

Table 4. % DPPH scavenging activity of ascorbic acid

Conc. ( $\mu\text{g/ml}$ )	% DPPH Scavenging Activity Ascorbic acid	IC <sub>50</sub> $\mu\text{g/mL}$
0.0195	0.769230	1.68
0.039	2.307692	
0.078	3.076923	
0.156	3.846153	
0.312	18.461538	
0.625	40.769230	
1.25	83.846153	
2.5	93.846153	
5	94.615384	

**CONCLUSION:**

The essential oil of the Damask rose (*Rosa damascena* Mill) obtained using different metal-based hydrodistillation apparatus exhibited different chemical compositions. A total of 20 chemical components were detected when a stainless steel-based apparatus was used for the extraction, while 28 compounds were identified when a copper-based apparatus was used. Differences were also observed in the amount and nature of the principal components. Citronellyl isobutyrate (22.44%) and citronellol (38.57%) emerged as the main components when the stainless steel and copper apparatus were employed for the extraction, respectively. Overall, the chemical composition of the tested rose oils varied significantly with regard to the extraction mode. The antioxidant activity of the tested oils was comparable to those reported in the literature. The tested oils can be explored for nutraceutical applications.

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