Evaluation of Some Postharvest Storage Approaches on Essential Oil Characteristics of Fresh Organic Damask Rose (*Rosa damascena* Mill.) Flowers

Maryam Mirzaei 1, Nima Ahmadi 1-*, Fatemeh Sefidkon 2, Abdolali Shojaeiyan 1 and Alireza Mazaheri 3

1 Department of Horticultural Sciences, Tarbiat Modares University, Tehran 336-14115, Iran; maryammirzaeem@gmail.com (M.M.); shoja_a@yahoo.com (A.S.)
2 Research Institute of Forests and Rangelands, Tehran 13185-116, Iran; sefidkon@rifr-ac.ir
3 R & D Department, Golkaran Essential Oils and Herbal Extracts Company, Kashan 1178, Iran; mazaheri_58@yahoo.com
* Correspondence: ahmadin@modares.ac.ir; Tel.: +98-21-4829-2088; Fax: +98-21-4829-2200

Academic Editors: Varit Srilaong, Mantana Buanong, Chalermchai Wongs-Aree, Sirichai Kanlayanarat and Douglas D. Archbold

Received: 4 December 2015; Accepted: 27 July 2016; Published: 30 December 2016

**Abstract:** Damask rose (*Rosa damascena* Mill.) is an economically important species in the Rosaceae family for rose oil and rose water production, obtained exclusively from freshly-gathered Damask rose flowers. Iran is famous for producing organic rose essential oil, mainly used in the perfume industry and for folk medicines due to its healing properties. Due to their high perishability, postharvest handling of the flowers prior to oil extraction are a key point in rose essential oil production. In this study, postharvest handling conditions of Damask rose flowers were evaluated for the effect on yield and quality of the extracted essential oils. Organically-grown flowers were stored under different conditions included packaging in polyethylene bags and immersing into water containers, and then held at 25 ± 2 °C or 4 ± 1 °C for 1, 2, or 3 days. Identification of the chemical composition of the essential oils was performed by GC and GC/MS. The results indicated that petal storage in water resulted in the highest essential oil content and quality, and storage in polyethylene bags resulted in the greatest loss, compared to those from unstored petals. The results provide a reference for interested groups such as producers and consumers who are concerned about Damask rose flower storage and subsequent oil extraction.

**Keywords:** essential oil characteristics; organic damask rose; post-harvest handling

1. Introduction

*Rosa damascena* Mill., commonly known as the oil-bearing rose, is one of the most important Rosa species cultivated for production of rose oil and rose water [1]. Rose oil, one of the most expensive base materials in the flavor and perfume industries, is characterized by a high percentage of monoterpenic alcohols including citronellol, nerol, geraniol, linalool, and phenyl ethyl alcohol. These components are the main contributors to the perfume value of rose oil [2]. According to the international standard of rose essential oil [3], rose oil requires 20%–34% citronellol, 15%–22% geraniol, and 8%–15% nonadecane as the main compounds [3]. As harvested Damask rose flowers are highly perishable, postharvest handling of the flowers prior to oil extraction is a vital step in rose essential oil production. Due to the huge volume of Damask rose flowers delivered to the factories during the harvest period, it often takes a long time before flowers are eventually extracted and distilled. Excessive temperatures inside the stacks of harvested flowers caused by their high respiration rate results in losses of essential oil quantity and quality [2]. Providing optimal storage conditions throughout postharvest handling
could reduce losses of yield and quality of essential oils [4,5]. The high value of the essential oil of Damask rose is an important reason for finding proper storage techniques for the flower crop after harvest. Therefore, the objectives of this study were to determine the effects of ambient and reduced temperatures as well as different storage durations and surrounding mediums on the yield and chemical components of organic Damask rose flowers throughout a postharvest handling period of 3 days.

2. Experimental Section

2.1. Plant Material

Organically-grown fresh Damask roses petals were handpicked from a Rosarium located in Mashhad-e Ardehal, Kashan, Iran, and transported immediately to the laboratory. The essential oils obtained from unstored petals (0 day storage duration), which were hydrodistilled shortly after harvest, were considered as the reference point in order to determine the effect of storage conditions on essential oil content and oil composition.

2.2. Storage Conditions

The petals were stored under the following conditions: packaged using sealed polyethylene (PE) bags (60 µm thick), or immersed in water inside sealed plastic containers (20 cm × 25 cm × 30 cm), and then held at 25 °C (25 ± 2 °C, 45% ± 5% RH) or 4 °C (4 ± 1 °C, 90% ± 5% RH) for 1, 2 and 3 days.

2.3. Essential Oil Extraction

The essential oil was extracted from 250 g petals by hydrodistillation with 750 mL of distilled water using a 4 L Clevenger-type apparatus for each experimental replication. The distillation time was 1.5 h and the oil content was measured as percentage (v/w%). In the case of the water storage method, the water used for storage was also hydrodistilled.

2.4. Characterization of Essential Oil Components

2.4.1. Gas Chromatography (GC)

The essential oils were analyzed on a Thermo-UFM (Ultra-fast model, Milan, Italy) gas chromatograph using a 10 m × 0.1 mm capillary column PH-5 (nonpolar), with an inner surface with a stationary phase of 5% dimethyl siloxane phenyl. The oven temperature was held at 60 °C for 3 min, programmed to 285 °C at the rate of 80 °C/min, and detector (FID) and injection chamber temperatures were 280 °C. Helium was used as the carrier gas with inlet pressure of 0.5 mL/min.

2.4.2. Gas Chromatography-Mass Spectrometry (GC-MS)

The GC-MS analysis was performed using a Varian 3400 gas chromatograph connected to a mass spectrometer model Saturn II, using an ion trap system having ionization energy of 70 eV, with a DB-5 column (semi-polar), with dimensions of 30 m × 0.25 mm, and a film thickness 0.25 mm. Gas pressure was 2.46 kg/cm², column temperature was 40 to 250 °C with a rate of increase of 3 °C/min, and the injection chamber temperature and the transfer line were set at 260 °C and 270 °C, respectively. The carrier gas was helium with a linear velocity of 31.5 cm/s, and a split ratio of 1:60. A scan time of 1 s with a mass range 40–300 were used. Spectra identification were carried out using retention indices and by injection of normal hydrocarbons (C7-C25) under the same conditions and confirmed with the Wiley 275-L library and the data reported in the literature [6–8].

2.5. Data Analysis

The experiment was carried out as a factorial with the factors of storage duration, petal storage medium, and storage temperature in a randomized complete block design (RCBD). There were
three replications of essential oil extraction from the petals for every treatment combination. Analysis of data from the essential oil content was performed using the general linear model (GLM) of SAS 9.2 [9]. Mean values were compared at the 5% \((p \leq 0.05)\) and 1% \((p \leq 0.01)\) levels of significance using Fisher’s Least Significant Difference (LSD) test. There were three replications of every treatment combination. Component analyses of the oils were performed by injecting a mix of the three replications of each treatment combination on both GC and GC/MS.

3. Results and Discussion

3.1. Essential Oil Contents

The petal essential oil content was significantly affected by storage condition and duration, but not temperature alone. There was a significantly greater content of essential oil stored in water at 4 °C and 25 °C at 1, 2 and 3 day compared to petals stored in PE bags (Figure 1). Storage of flowers in water recorded a significant increase \((p \leq 0.01)\) of 16%, 19% and 23% in essential oil content after 1, 2, and 3 day, respectively, over the unstored petals with an average value of 0.069 (dry weight%). This increase could be explained by considering the limitation of O\(_2\) inside the containers along with the presence of water that may have caused fermentation, as it has been indicated that rose petals produce more essential oil with a fermentation period after harvest [10]. The mean essential oil content obtained from petals stored in PE for 1 day were close to the value at harvest (unstored petals) but were significantly lower after 2 or 3 day of storage. Cold-stored flowers in water contained higher oil content than those in PE at the same storage temperature, which might be explained by the ability of water to maintain volatile oil content of the petals during the storage period, while the permeability of the PE bags might result in losses from volatilization of essential oils, particularly with the extension of the storage period.

![Figure 1. Mean essential oil content (v/w%, fresh weight basis) of organic Damask rose under different storage conditions. PE: (Polyethylene bag); W: (Water).](image)

3.2. The Essential Oil Components

The changing patterns of the essential oil components under the storage conditions indicated that the chemical profile of rose oil throughout storage of petals in PE bags was affected more than storage in water under either 25 °C or 4 °C storage conditions, with a higher degradation of geraniol in favor of citronellol at 25 °C (Table 1). Storage of petals in water impacted the maintenance of the major essential oil compounds. The component analyses of oils from 2 and 3 day samples indicated that storage of the petals in the cold water (4 °C-W) maintained the rose oil characteristics better than the other handling method, with approximately 38% lower levels of long-chain aliphatic hydrocarbons after 1 day of storage, which resulted in improvement of the oil quality. A possible reason for the rise of monoterpene alcohols might be ascribed to the proper conditions for the action of enzymes, such as \(\beta\)-glucosidase involved in hydrolysis and liberation of monoterpene alcohols from their glycosidic forms [11].
Table 1. Damask rose essential oil components under different storage conditions.

<table>
<thead>
<tr>
<th>Factors of Variance</th>
<th>Compound</th>
<th>Phenyl Ethyl Alcohol</th>
<th>cis-Rose Oxide</th>
<th>Dihydrolinalool</th>
<th>Citronellol</th>
<th>Neat</th>
<th>Geraniol</th>
<th>Geranial</th>
<th>Citronellyl Acetate</th>
<th>E-β-Damascone</th>
<th>n-Heptadecane</th>
<th>E,E-Farnesyl Acetate</th>
<th>Nonadecane</th>
<th>n-Eicosane</th>
<th>n-Heneicosane</th>
<th>n-Tricosane</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time</td>
<td>Storage conditions</td>
<td>RI</td>
<td>1109</td>
<td>1113</td>
<td>1135</td>
<td>1228</td>
<td>1240</td>
<td>1255</td>
<td>1269</td>
<td>1355</td>
<td>1416</td>
<td>1693</td>
<td>1728</td>
<td>1902</td>
<td>2003</td>
<td>2102</td>
</tr>
<tr>
<td>0 day</td>
<td>Unstored petals</td>
<td>1.3</td>
<td>0.3</td>
<td>0.8</td>
<td>35.5</td>
<td>13.3</td>
<td>16.8</td>
<td>3.1</td>
<td>0.4</td>
<td>0.2</td>
<td>2.3</td>
<td>0.2</td>
<td>17.9</td>
<td>1.4</td>
<td>8.1</td>
<td>1.2</td>
</tr>
<tr>
<td>1 day</td>
<td>4 °C-PE</td>
<td>1.0</td>
<td>0.3</td>
<td>0.8</td>
<td>42.5</td>
<td>3.9</td>
<td>13.0</td>
<td>0.3</td>
<td>-</td>
<td>-</td>
<td>2.3</td>
<td>0.2</td>
<td>17.9</td>
<td>1.4</td>
<td>8.1</td>
<td>1.2</td>
</tr>
<tr>
<td></td>
<td>4 °C-W</td>
<td>1.3</td>
<td>0.3</td>
<td>0.9</td>
<td>38.2</td>
<td>21.5</td>
<td>13.4</td>
<td>2.9</td>
<td>0.2</td>
<td>0.3</td>
<td>2.5</td>
<td>0.9</td>
<td>9.1</td>
<td>0.5</td>
<td>1.0</td>
<td>0.3</td>
</tr>
<tr>
<td></td>
<td>25 °C-PE</td>
<td>1.6</td>
<td>0.4</td>
<td>1.3</td>
<td>47.4</td>
<td>16.5</td>
<td>9.8</td>
<td>5.8</td>
<td>-</td>
<td>0.3</td>
<td>2.6</td>
<td>0.4</td>
<td>8.8</td>
<td>0.3</td>
<td>4.1</td>
<td>0.8</td>
</tr>
<tr>
<td></td>
<td>25 °C-W</td>
<td>1.1</td>
<td>0.7</td>
<td>0.9</td>
<td>42.3</td>
<td>11.4</td>
<td>12.6</td>
<td>2.0</td>
<td>0.1</td>
<td>0.4</td>
<td>2.9</td>
<td>0.7</td>
<td>20.5</td>
<td>1.0</td>
<td>6.3</td>
<td>0.9</td>
</tr>
<tr>
<td>2 days</td>
<td>4 °C-PE</td>
<td>0.9</td>
<td>0.4</td>
<td>0.8</td>
<td>45.6</td>
<td>12.5</td>
<td>10.8</td>
<td>1.6</td>
<td>-</td>
<td>0.4</td>
<td>2.6</td>
<td>0.5</td>
<td>18.9</td>
<td>2.1</td>
<td>12.3</td>
<td>2.2</td>
</tr>
<tr>
<td></td>
<td>4 °C-W</td>
<td>1.7</td>
<td>0.5</td>
<td>1.4</td>
<td>48.2</td>
<td>15.6</td>
<td>8.1</td>
<td>6.2</td>
<td>-</td>
<td>0.3</td>
<td>2.8</td>
<td>0.7</td>
<td>7.3</td>
<td>0.4</td>
<td>0.6</td>
<td>0.8</td>
</tr>
<tr>
<td></td>
<td>25 °C-PE</td>
<td>1.2</td>
<td>0.3</td>
<td>0.5</td>
<td>36.5</td>
<td>18.7</td>
<td>10.0</td>
<td>2.0</td>
<td>0.2</td>
<td>0.1</td>
<td>2.6</td>
<td>0.5</td>
<td>12.8</td>
<td>0.4</td>
<td>1.7</td>
<td>0.5</td>
</tr>
<tr>
<td></td>
<td>25 °C-W</td>
<td>1.1</td>
<td>1.1</td>
<td>1.1</td>
<td>43.4</td>
<td>10.2</td>
<td>11.8</td>
<td>1.2</td>
<td>-</td>
<td>0.6</td>
<td>3.3</td>
<td>0.4</td>
<td>22.2</td>
<td>1.1</td>
<td>5.0</td>
<td>1.1</td>
</tr>
<tr>
<td>3 days</td>
<td>4 °C-PE</td>
<td>1.0</td>
<td>0.3</td>
<td>0.7</td>
<td>47.4</td>
<td>7.4</td>
<td>9.0</td>
<td>0.6</td>
<td>-</td>
<td>2.9</td>
<td>-</td>
<td>20.7</td>
<td>2.7</td>
<td>14.3</td>
<td>4.1</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4 °C-W</td>
<td>0.7</td>
<td>0.4</td>
<td>1.0</td>
<td>35.6</td>
<td>16.4</td>
<td>15.4</td>
<td>3.4</td>
<td>-</td>
<td>0.4</td>
<td>3.1</td>
<td>-</td>
<td>7.6</td>
<td>0.6</td>
<td>2.2</td>
<td>0.4</td>
</tr>
<tr>
<td></td>
<td>25 °C-PE</td>
<td>1.9</td>
<td>0.6</td>
<td>0.8</td>
<td>36.5</td>
<td>12.8</td>
<td>5.2</td>
<td>5.9</td>
<td>-</td>
<td>0.2</td>
<td>3.4</td>
<td>0.8</td>
<td>12.6</td>
<td>0.4</td>
<td>3.6</td>
<td>0.7</td>
</tr>
<tr>
<td></td>
<td>25 °C-W</td>
<td>1.0</td>
<td>1.5</td>
<td>1.4</td>
<td>46.7</td>
<td>9.2</td>
<td>10.0</td>
<td>0.9</td>
<td>-</td>
<td>0.8</td>
<td>3.9</td>
<td>0.1</td>
<td>23.6</td>
<td>1.2</td>
<td>4.6</td>
<td>1.3</td>
</tr>
</tbody>
</table>

RI: Retention Index; * Relative percentage of each component in relation to total essential oil; W: water; PE (polyethylene).
4. Conclusions

As the water used for the storage of the flowers was then used in the hydrodistillation, water, particularly at low temperatures, might be an effective storage method for oil-bearing rose petals after harvest to maintain essential oil content and component quantities close to the content of freshly-harvested flowers. These results are useful for choosing the best storage techniques for Damask rose flowers prior to processing.

Acknowledgments: The authors would like to express their appreciation to Barij Essence Company for their support and for providing facilities for this study.

Author Contributions: Maryam Mirzaei, Nima Ahmadi, Fateme Sefidkon and Abdolali Shojaeiyan designed the research; Maryam Mirzaei, Nima Ahmadi and Fateme Sefidkon performed the research; Nima Ahmadi, Fateme Sefidkon, Alireza Mazaheri and Abdolali Shojaeiyan contributed analysis tools/materials/reagents; Maryam Mirzaei analyzed the data; Maryam Mirzaei and Nima Ahmadi wrote the paper.

Conflicts of Interest: The authors declare no conflict of interest.

References

2. Baydar, H.; Baydar, N. The effects of harvest date, fermentation duration and Tween 20 treatment on essential oil content and composition of industrial oil rose (Rosa damascena Mill.). *Ind. Crops Prod.* **2005**, *21*, 251–255. [CrossRef]