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# Extraction of essential oils from damask rose using green and conventional techniques: Microwave and ohmic assisted hydrodistillation versus hydrodistillation



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#### ABSTRACT

Damask rose is cultivated as the main species used in the production of rose water and relevant essential oils (EOs) of rose for its fragrance and therapeutic applications. Since the flowers are highly perishable, the extraction of oil should be done rapidly in order to produce rose EO successfully and optimally. Solvent free microwave-assisted extraction (SFME) and ohmic assisted hydrodistillation (OAHD) are advanced and green distillation techniques, which utilize microwave, and ohmic heating processes respectively for the extraction of EOs. In this study, SFME and OAHD of EOs were performed on fresh flowers of damask rose, and the findings were compared with the traditional hydrodistillation (HD). The findings of EQ analysis indicated considerable alterations in EOs compounds extracted by SFME and OAHD methods compared with the traditional HD. To reach the desirable temperature of extraction (nearly 100 °C) and to get the first EO droplets' evaporation with steam, the mixture was heated for 2.50  $\pm$  0.29 min in SFME and for 17.33  $\pm$  0.33 min in OAHD, while for the HD the heating had to take more than 40 min (42.66  $\pm$  0.32 min). The total extraction time of EOs extracted from damask rose by using the green extraction procedures (SFME and OAHD) were compared with HD extraction method. Extraction by SFME occurred much earlier than extraction by OAHD. Also in this study, the analysis of EOs indicated that the chemical profile of damask rose may fluctuate quantitatively with respect to the constituents and structure of the extracted molecules. This would depend on the system of extraction that influences the characteristics of the EOs.

#### 1. Introduction

The damask rose (*Rosa damascena* Mill.) is considered as a major species of rose on which extractions are performed to yield of EOs, products of rose water and perfume of rose in industries (*Rusanov et al.*, 2012; Karami et al., 2012). Two major rose oil production areas recognized widely in the world are rose cultivations in Isparta of Turkey and Kazanlik of Bulgaria (Baydar et al., 2008). In addition to those regions, the Fars province of Iran is another leading producer of damask rose where 8598 t of flowers are produced annually in 6149 ha of gardens (Iranian Ministry of Agriculture, 2014). Generally, EO yield of damask rose flowers are very little (0.03–0.04%) and the fresh top-quality rose flowers are harvested in the early hours of morning for highest EO yields (Baydar and Baydar, 2005). Traditionally, the full-bloom phase is the most appropriate period of plant growth when considering the purpose of obtaining extraction of rose oil or rose water. Nonetheless, recent investigations indicate that the yield of rose

oil distilled from buds of rose is the same or greater in amount for the same flower material weight when compared to distillation performed on full-bloomed flowers (Rusanov et al., 2012). Various methods are employed for isolating oils from different materials of plant. It may seem comparatively easy to separate such oils, but the composition of oils could differ substantially, based on the extraction method that is applied (Anitescu et al., 1997; Cassel et al., 2009). Technologies of traditional extraction are often inconvenient and energy-consuming. Concerns pertaining to cost and the environment have demanded reductions in CO<sub>2</sub> emissions. This encourages researchers to find substitutes and equipment based on their cost-effective criteria. The equipment and method need to be sustainable, and to have better potentials of making products with similar principal features. Green technology is becoming popular recently. Microwave-assisted extraction (MAE) and OAHD, for example, have optimum heating mechanisms, they are cost-effective, and exhibit better performance when atmospheric conditions prevail. Shorter durations of time are required by

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MAE and OAHD for extraction to take place effectively. MAE and OAHD are capable of producing higher extraction yields when viewed in comparison with conventional extraction methods (Farahnaky et al., 2010; Chan et al., 2011; Gavahian et al., 2011, 2013). Damask rose industries partly focus on which extraction technology can be more optimum as a better option compared with traditional methods. Both OAHD and SFME are technologies that have received considerable attention recently in the industry. Both have similar advantages including the reduction in process time. Therefore, the aims of this study were to compare the extraction yield, time of extraction, and the EO composition from fresh flowers of damask rose obtained by OAHD and SFME methods in comparison to those obtained by the HD method.

#### 2. Materials and methods

#### 2.1. Plant materials

Damask rose fresh flowers were obtained from cultivated populations in their full flowering stage in Layzangan (Darab, Fars Province, southern Iran). The plant species was specified and authenticated by A. Khosravi, a plant taxonomist at Shiraz University, Shiraz, Iran. A specimen of voucher was related with the herbarium.

#### 2.2. Solvent free microwave-assisted extraction (SFME)

SFME was performed in a modified way as described by Wang et al. (2006). The EOs were obtained from fresh flowers of damask rose by distillation without the use of solvents for 25 min using a Clevenger type apparatus (Fig. 1). One hundred grams of damask rose samples were placed in a modified microwave oven (ME343, Samsung, Korea, 230, 1550 W; variable in 110 W increments, 2450 MHz). The microwave oven operated at 1000 W during the first 3 min and then at 400 W for 22 min. All EOs from the sample were extracted in this period. Over anhydrous sodium sulphate, the oil was dried and conserved in brown sealed vials at 4 °C for further use.

#### 2.3. Ohmic assisted hydrodistillation (OAHD)

OAHD was carried out employing an ohmic distillatory tool and platinum electrodes in the Department of Food Science and Technology of Shiraz University, Shiraz, Iran (Fig. 1). Processing variables like temperature, processing time, and power consumption were controlled applying a software application coupled with a Wattmeter to record the ohmic apparatus input power to check the software data. OAHD was done at 220 V, 50 Hz, and at differing current rates in accordance with the time of process (Farahnaky et al., 2010). To eliminate water, the extracted EOs were dried on anhydrous sodium sulphate and kept at 4 °C in amber vials for further trials.

#### 2.4. Hydrodistillation (HD)

HD was performed as SFME, applying a heater (FINTECH; Korea; and 500 W). The proportions and volume of the operated container were alike those employed for MAHD. Fresh flowers of the damask rose (100 g) and 500 mL distilled water were put in a round bottom flask and linked to a Clevenger-type device. After boiling, HD was completed for three h. The sample's oil yield was estimated on a moisture free basis. Over anhydrous sodium sulphate, the oil was dried and was stored in sealed brown vials at 4 °C.

### 2.5. Physical constants

Specific gravity of the EOs from the fresh petal of damask rose was estimated based on Food Chemical Codex (FCC) (FCC, 1996) at 25  $^{\circ}$ C.

#### 2.6. Analysis of the oil

GC analysis was done on over Agilent 7890-A GC with the help of a Flame Ionization Detector (FID) using a HP-5 fused silica capillary column (30 m  $\times$  0.32 mm i.d.; film thickness 0.25 µm; J & W Scientific, Folsom). The temperatures of injector and detector were, respectively, 250 and 280 °C. As a carrier gas, nitrogen was applied at 1 mL/min flow rate, and in the split mode, 0.1 µL of EO samples diluted in *n*-hexane were injected. The temperature of oven was programmed from 60 °C to 210 °C with a gradient of 4 °C/min, and then increased to 240 °C with a gradient of 20 °C/min and isothermally kept for 8.5 min. A split ratio of 1:50 was maintained.

The same Agilent gas chromatograph as above coupled with a mass spectrometer detector (Model 5975 C) and equipped with a HP-5 MS fused silica capillary column ( $30 \text{ m} \times 0.25 \text{ mm}$  i.d.; film thickness 0.25 µm; J & W Scientific, Folsom) was used to conduct the GC/MS analysis. Helium was employed as a carrier gas. The source of ion and interface temperatures of 230 °C and 280 °C were employed, respectively. 70 eV ionization voltage was applied. The range of mass was set between 45 and 550 amu. The temperature of oven programme was as the temperature used for GC-FID. The temperature-programmed Retention Indices (RIs) of EO components were calculated using a mixture of *n*-alkanes ( $C_8$ - $C_{25}$ ) and the same chromatographic conditions as above and compared with those expressed in previous studies (Adams, 2007). In addition, their mass spectra and those contained in mass spectral libraries (Willey/ChemStation data system and NIST 08/ National Institute of Standards and Technology) were compared. The percentages of relative region estimated via FID were utilized for quantification with no application of the correction parameters.



Fig. 1. Solvent free microwave-assisted extraction (A); Ohmic assisted hydrodistillation (B) apparatuses set-up.

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