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Effect of solvents on the composition of *Rosa x damascena* concrete oil in multistage solvent extraction

K. Sofiya*, K. Anbalagan, A. Richa Elson, I. Fazal Fathima and M. Vignesh Kumar

*Department of Chemical Engineering, SRM Institute of Science and Technology, Kattankulathur- 603203, Tamil Nadu, India. *E-mail: sofiyak@srmist.edu.in,ksofiya26@yahoo.co.in*

Abstract

Four different solvents, ethyl acetate, ethanol, petroleum ether, and hexane, were used for the multistage solvent extraction of rose concrete oil from the aromatic plant species of *Rosa x damascena*. The components present in the concrete oils were analyzed using Gas Chromatography-Mass Spectrometer. After the multistage solvent extraction process, the solvent was removed by using a rotary vacuum evaporator. Methyl alpha d-glucopyranoside, 5-hydroxy methyl furfural, 2,3-butanediol, and ethyl-d glucopyranoside were the major components identified using ethyl acetate ethanol, hexane, and petroleum ether as a solvent, respectively. The phenyl ethyl alcohol and 5-hydroxymethyl furfural were identified as the repeated components in all four solvents. The solvent ethanol showed a different composition when compared to the other three solvents. A high yield was obtained when ethanol was used as a solvent. The type of solvent used significantly impacts the compositions of the concrete oil of *Rosa x damascena*.

Keywords: *Rosa x damascena*, organic solvents, composition analysis, multistage solvent extraction

Introduction

Rosa x damascena has multiple applications in the food, pharmaceutical, flavour, and fragrance industries (Mostafavi and Afzali, 2009; Akram *et al.*, 2020; Thakur and Kumar, 2021). Conventional and non-conventional techniques are used to obtain various products of *Rosa x damascena* (Younis *et al.*, 2008; Jirovetz *et al.*, 2005; Mariana and Marilena, 2016). The products obtained from *Rosa x damascena* are rose oil, rose water, rose concrete, rose absolute, rose ott, and essential oil (Mahboubi, 2016).

There is a continued demand for these quality products, and the need for these products will increase in the future (Pal, 2013). The rose concrete and rose absolute, produced from fresh oil-bearing flowers of *Rosa x damascena* plant species, have essential applications in the cosmetic and perfumery industries (Ayci *et al.*, 2005; Erbaş and Baydar, 2016). The solvent extraction process mainly produces the rose concrete oil, and after treating it with alcohol, the obtained absolute is used in the perfume industry (Erbaş and Baydar, 2016). The advantage of solvent extraction over distillation is that it involves minimum temperature, is inexpensive, and results in a higher yield (Younis *et al.*, 2007; Dupuy *et al.*, 2011). Soxhlet solvent extraction and multistage solvent extractions (Aydinli and Tutaş, 2003) usually extract natural components. Sometimes, the temperature conditions may change the characteristics of the products. There is a continued debate on which solvent must be selected to get *Rosa x damascena* compositions. The selection of solvents plays a vital role in the extraction process and depends on factors such as selectivity, cost, solubility, *etc.* (Zhang *et al.*, 2018). The solvent's polarity also plays an essential role in the extraction process; usually, polar solvents dissolve the polar material while non-polar solvents dissolve the non-polar material (Zhang *et al.*, 2018; Efthymiopoulos *et al.*, 2018). Many researchers carried out studies on the use of polar and non-polar solvents (Erbaş and Baydar, 2016) to enhance the extraction yield and quality of essential rose oil. The selection of an appropriate solvent for the extraction of natural essential oil, its impacts, the efficiency of the extraction method, the effect of the extraction method on yield and quality, variation of oil content, and new developments in the field are continued to be the areas of much research interest. The present research work mainly concentrates on the multistage solvent extraction of rose concrete oil from *Rosa x damascena* using four different solvents, namely ethyl acetate, ethanol (Chemat *et al.*, 2012), hexane (El-Sharnouby *et al.*, 2021), and petroleum ether (Zhang *et al.*, 2012) to study the impact of the type of solvent on the concrete oil compositions. To the best of the author's knowledge, data on the components of *Rosa x damascena* using multistage solvent extraction is not available in the literature. Among the four chosen solvents, ethyl acetate is polar; ethanol has polar and non-polar characteristics; hexane and petroleum ether are non-polar solvents. After the multistage solvent extraction, the rotary vacuum evaporator was used to remove the solvent. The compositions of the concrete oil were analyzed by Gas Chromatography-Mass Spectrometry (GC-MS). We have determined the concrete oil's physical properties and the yield from the rose petals' mass ratio to the quantity of concrete oil obtained. The main components present in the four concrete oils were analyzed and reported.

Materials and methods

Raw material (*Rosa x damascena*): The raw material, cultivated species of *Rosa x damascena*, was procured from the Dindugal district of Tamil Nadu in South India. The seller was a regular supplier to the nearby perfumery industry. A perennial shrub, the hybrid plant is also known as Damask Rose. It blooms all the year round and bears fragrant flowers which are used to commercialize the rose products.

Multistage solvent extraction and GC-MS analysis: For the multistage solvent extraction process, 500 grams of rose petals were taken, along with 1000 mL of solvent, divided into four portions (250 mL each). The ground petals were first agitated in 250mL of solvent, and the extract was then collected. Then used materials were exposed to 250 mL of solvent for a second time, and the portion obtained was mixed with the first extract. The procedure was carried out four times to bring all possible material from the flower to the solvent phase. The solvent was separated from the intermediate by a rotary vacuum evaporator (Heidolph) at 60ºC.

The density (specific gravity bottle), viscosity (Ostwald viscometer), refractive index (Abbe's refractometer), and pH were determined for the said four concrete oils. The GC-MS was used to analyze the compositions of obtained oils. Shimadzu QP2010-Plus column was used for the GC-MS analysis. The operating conditions of the HP-5MS capillary column were at 65ºC for 2-3 mins and later at 225ºC. Helium was used as the carrier gas at a speed of 1ml/min. Electron ionization was the mass spectrometry method used. The findings of the components were represented as a graph depicting the peaks and retention times.

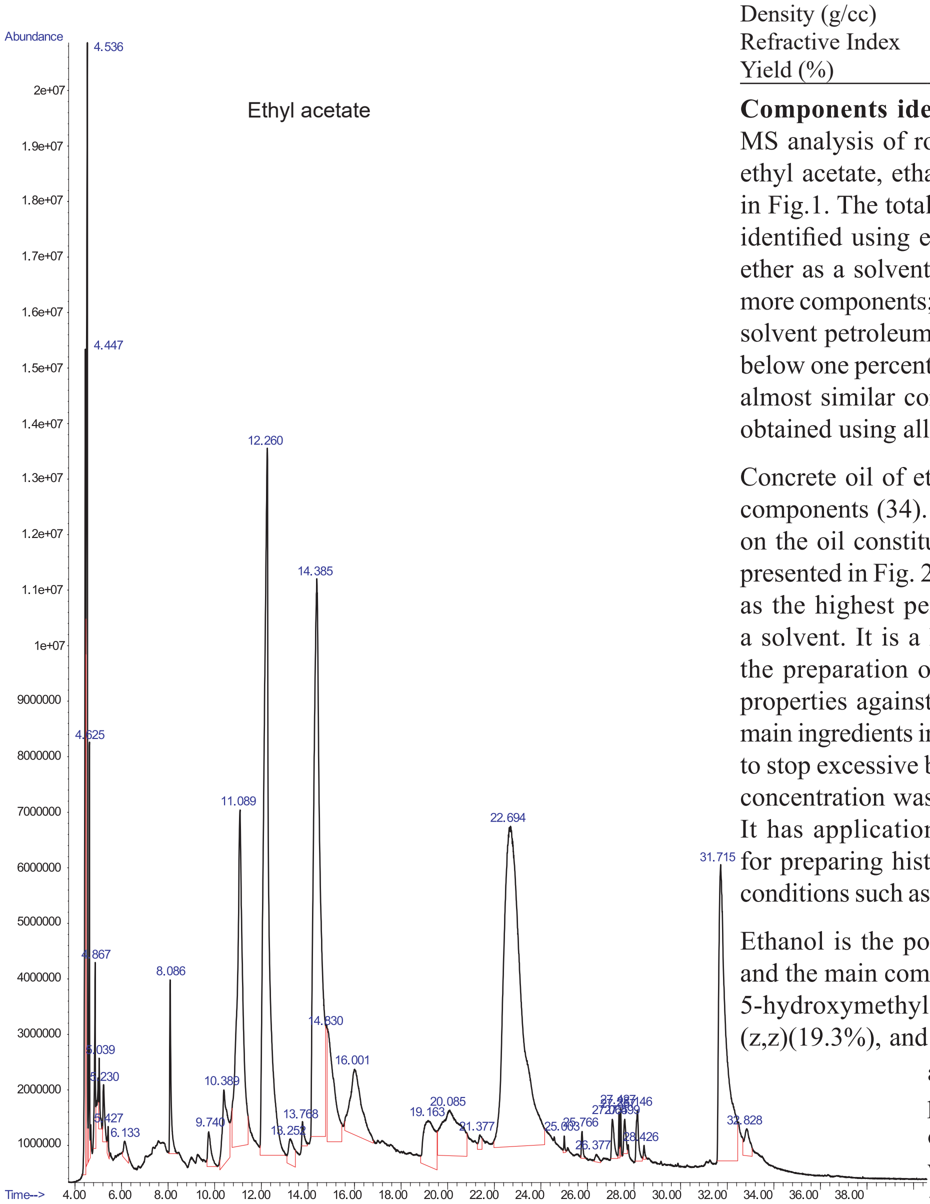

Fig. 1a. Chromatogram of GC-MS analysis of concrete oil for ethyl acetate.

Results and discussion

Physical properties of concrete oil: The rose concrete oil's physical properties are listed in Table 1. Table.1 shows that the solvent ethanol yield is higher (98.2%) than the other solvents. It may be due to the property of the ethanol to dissolve both polar and non-polar components. The pH value indicates the neutral behavior of the concrete oil, even after extracted with the solvent. The value of density and refractive index indicates the quality of the oil.

Table.1. pH, density, refractive index, and yield of the four different concrete oils

Properties	Concrete oil (Reddish in colour)			
	Petroleum ether	Ethyl acetate	Hexane	Ethanol
pH	6.30	6.83	6.92	6.38
Density (g/cc)	1.04	1.08	1.02	0.96
Refractive Index	1.35	1.34	1.36	1.37
Yield (%)	69.30	67.70	72.60	98.20

Components identified by GC-MS: The results of the GC-MS analysis of rose concrete oil using four different solvents, ethyl acetate, ethanol, hexane, and petroleum ether, are shown in Fig.1. The total components of around 34, 25, 26, and 28 are identified using ethyl acetate, ethanol, hexane, and petroleum ether as a solvent, respectively. The solvent ethyl acetate gave more components; On the other hand, ethanol yielded higher. The solvent petroleum ether extracted many constituents, which are below one percent. The solvents hexane and petroleum ether had almost similar constituents. Many medicinal compounds were obtained using all four solvents.

Concrete oil of ethyl acetate showed the maximum number of components (34). The quality of the rose oil primarily depends on the oil constituents; the primary constitutions identified are presented in Fig. 2. Methyl-β-D-galactopyranoside was obtained as the highest percentage (23.383%) when ethyl acetate was a solvent. It is a hexose, and it has its applications mainly in the preparation of medicines. This component has antitumor properties against pancreatic cancer cells. It is also one of the main ingredients in Chinese herbal medicine, Panax ginseng, used to stop excessive bleeding. The next component with the highest concentration was 4H-Pyran-4-one, at a percentage of 15.11%. It has applications in the Millard reaction as an antioxidant for preparing histidine, which is essential for treating medical conditions such as arthritis, allergic reactions, ulcers, and anemia.

Ethanol is the polar solvent used to extract rose concrete oil, and the main components identified are presented in Fig. 2. The 5-hydroxymethylfurfural (26.1%), 9,12-Octadecadienoic acid (z,z)(19.3%), and phenyl ethyl alcohol (18.7%) were identified as the main components with the highest portions. The 5-hydroxymethylfurfural is an organic compound containing a furan ring with alcohol and an aldehyde functional group. 5-Hydroxymethylfurfural is classified as a taste enhancer and food improvement product in the

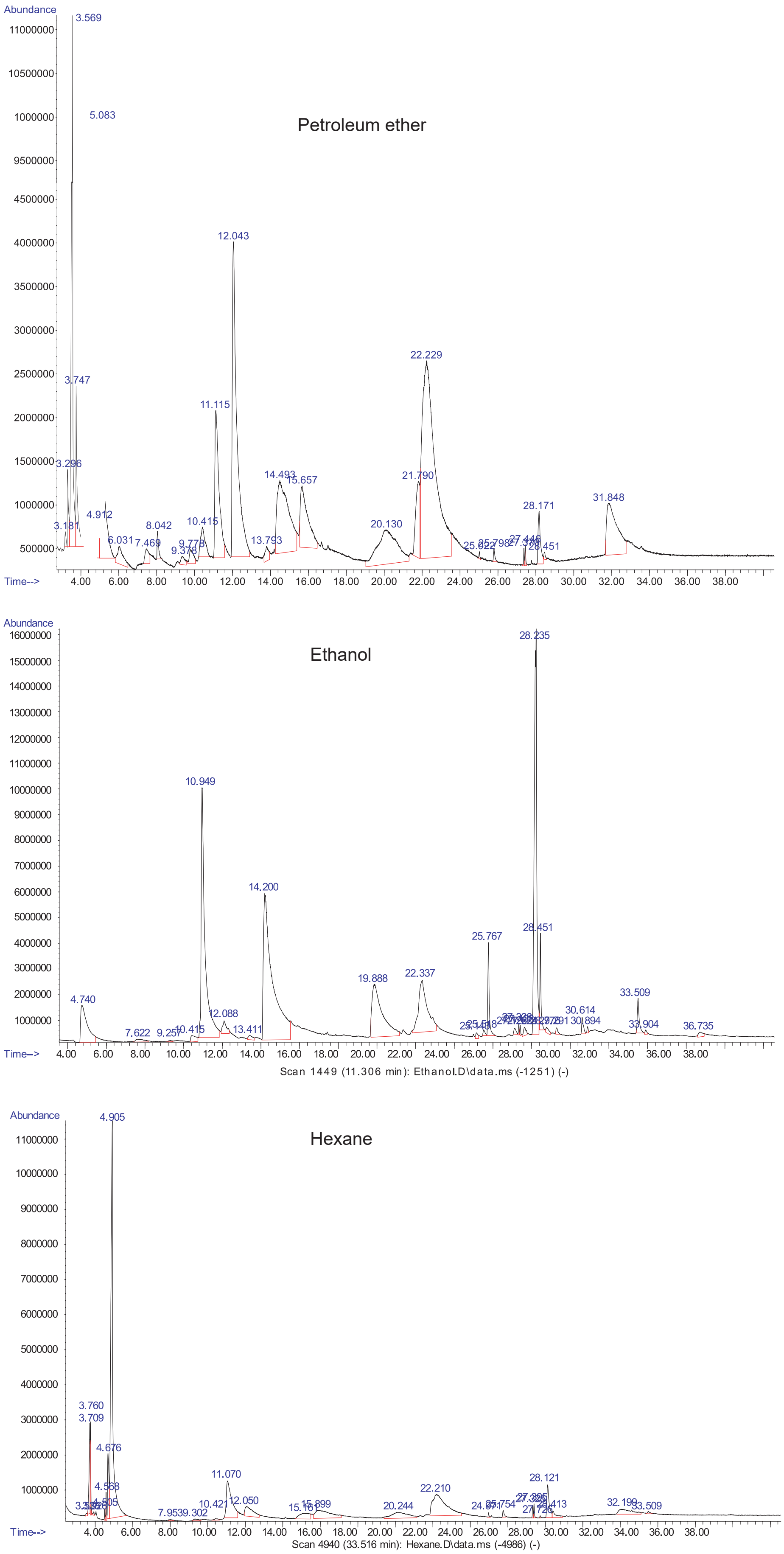

Fig 1b. Chromatogram of GC-MS analysis of concrete oil for the different solvents: petroleum ether, ethanol and hexane.

food industry; it is utilized as a food additive as a biomarker and flavouring agent for a food product. It has the characteristics to inhibit sickle cell growth during medical treatment and, it is the primary component used to treat sickle cell diseases. It has applications in the perfume industry due to its pleasant smell.

Hexane is the most efficient solvent used for essential oil extraction, according to Younis *et al.* (2008), due to its higher extraction efficiency. Some new components were identified, which were not reported in this concrete oil early (El-Sharnouby *et al.*, 2021). The main constituents are presented in Fig. 2. They need to be studied further to understand their medicinal properties. Rose oil is believed to have components with anti-cancer properties used in the medical field. 2,3- Butanediol is a component found in 42.8 percent of the samples (principal component). It is soluble in ketones, alcohols, and ether. It has applications in the manufacturing of perfumes and pharmaceuticals.

Petroleum ether is another solvent that belongs to the ether group, which is widely used in the extraction process and can extract both polar and non-polar components. The major component in the extraction process using petroleum ether was methyl α d glucopyranoside in the percentage of 23.01% (Fig. 2). It has been utilized in skin therapy because it provides relief from the problem of skin roughness which was caused by UV radiation.

In all four concrete oils, phenyl ethyl alcohol and 5-hydroxymethyl furfural were commonly found irrespective of the solvent type. Phenyl ethyl alcohol is usually found in all the essential oils, mainly in rose oil at higher concentrations. It is an organic compound, is slightly soluble in water, and has a pleasant floral fragrance. It also has excellent antiseptic and antimicrobial properties.

The multistage solvent extraction of rose concrete oil from *Rosa x damascena* was carried out using four solvents: ethyl acetate, ethanol, hexane, and petroleum ether. The solvent ethyl acetate extracted more components than the other three solvents. The solvent ethanol yielded higher than other solvents, but it gave less number of components. Hexane and petroleum ether yielded almost similar components. More components were obtained with petroleum ether, most of which were below one percent. Phenyl ethyl alcohol and hydroxymethylfurfural were obtained in all four solvents, irrespective of

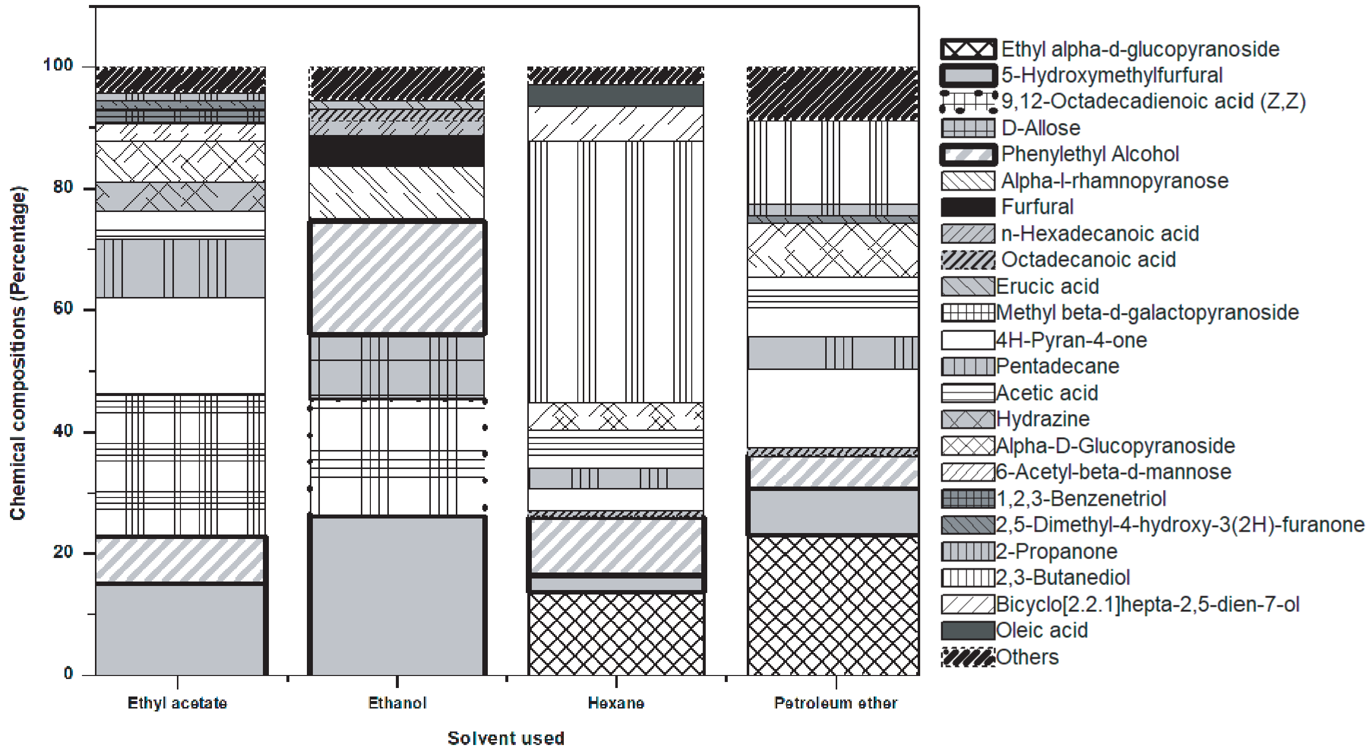

Fig. 2. Major components in multistage extraction of *Rosa x damascena* using four different solvents ethyl acetate, ethanol hexane, and petroleum ether.

the solvent characteristics. The study concluded that the solvent characteristics significantly influenced the compositions of the concrete oil extracted from *Rosa x damascena*.

References

Akram, M., M. Riaz, N. Munir, N. Akhter, S. Zafar, F. Jabeen, M. Ali Shariati, N. Akhtar, Z. Riaz, S. H. Altaf, M. Daniyal, R. Zahid, and F. Said Khan, 2020. Chemical constituents, experimental and clinical pharmacology of *Rosa damascena*: A literature review. *J. Pharm. Pharmacol.,* 72: 161-174.

Ayci, F., M. Aydinli, O. A. Bozdemir, and M. Tutaş, 2005. Gas chromatographic investigation of rose concrete, absolute and solid residue. *Flavour Fragra. J.,* 20: 481-486.

Aydinli, M. and M. Tutaş, 2003. Production of rose absolute from rose concrete. *Flavour Fragra. J.,* 18: 26-31.

Chemat, F., M. A. Vian, and G. Cravotto, 2012. Green extraction of natural products: Concept and principles. *Int. J. Mol. Sci.,* 13: 8615-8627.

Dupuy, A., V. Athes, J. Schenk, U. Jenelten, and I. Souchon, 2011. Solvent extraction of highly valuable oxygenated terpenes from lemon essential oil using a polypropylene membrane contactor: Potential and limitations. *Flavour Fragra. J.,* 26: 192-203.

Efthymiopoulos, I., P. Hellier, N. Ladommatos, A. Russo-Profili, A. Eveleigh, A. Aliev, A. Kay, and B. Mills-Lamptey, 2018. Influence of solvent selection and extraction temperature on yield and composition of lipids extracted from spent coffee grounds. *Ind. Crop. Prod.,* 119: 49-56.

El-sharnouby, M. E., M. M. Montaser, and S. M. Abdallah, 2021. Oil and flower production in *Rosa damascena* trigintipetala dieck under salinity stress in Taif region, Saudi Arabia. *Sustainability,* 13: 1-8.

Erbaş, S. and H. Baydar, 2016. Variation in scent compounds of oil-bearing rose (*Rosa damascena* Mill.) produced by headspace solid-phase microextraction, hydrodistillation, and solvent extraction. *Rec. Nat. Prod.,* 10: 555-565.

Jirovetz, L., G. Buchbauer, A. Stoyanova, A. Balinova, Z. Guangjiun and M. Xihan, 2005. Solid-phase microextraction/gas chromatographic and olfactory analysis of the scent and fixative properties of the essential oil of *Rosa damascena* L. from China. *Flavour Fragra. J.,* 20: 7-12.

Mahboubi, M. 2016. *Rosa damascena* as holy ancient herb with novel applications. *J. Tradit. Complement. Med.,* 6: 10-16.

Mariana, P. and R. Marilena, 2016. Rose essential oil extraction from fresh petals using synergetic microwave & ultrasound energy: chemical composition and antioxidant activity assessment. *J. Chem. Chem. Eng*., 10: 136-142.

Mostafavi, A. and D. Afzali, 2009. Chemical composition of the essential oils of *Rosa damascena* from two different locations in Iran. *Chem. Nat. Compd.,* 45: 110-113.

Pal, P.K. 2013. Evaluation, genetic diversity, recent development of distillation method, challenges and opportunities of *Rosa damascena*: A Review. *J. Essent. Oil-Bear. Plants*, 16: 1-10.

Thakur, M. and R. Kumar, 2021. Light conditions and mulch modulate the damask rose (*Rosa damascena* Mill.) yield, quality, and soil environment under mid-hill conditions of the western Himalaya. *Ind. Crop. Prod*., 163: 113-317.

Younis, A., M. Khan, A. Khan, A. Riaz, and M. Pervez, 2007. Effect of different extraction methods on yield and quality of essential oil from four rosa species. *Floriculture Ornamental Biotech.,* 1: 73-76.

Younis, A., A. Riaz, M.A. Khan, A.A. Khan, and M.A. Pervez, 2008. Extraction and identification of chemical constituents of the essential oil of *Rosa* species. Acta Hort., 766: 485-492.

Zhang, Q.W., L.G. Lin, and W.C. Ye, 2018. Techniques for extraction and isolation of natural products: A comprehensive review. *Chin. Med.,* 13: 1-26.

Zhang, X., H. Gao, L. Zhang, D. Liu and X. Ye, 2012. Extraction of essential oil from discarded tobacco leaves by solvent extraction and steam distillation, and identification of its chemical composition. *Ind. Crop Prod.,* 39: 162-169.

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