MILESTONE
HELPING CHEMISTS

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APPLICATION REPORT

EX14 - ROSE FRAGRANCES

Rosa Damascena Mill. Solvent-Free Microwave Extraction (SFME) and Microwave Hydrodistillation (MWHD)

Introduction

Roses have been used since the earliest times in rituals, cosmetics, perfumes, medicines and aromatherapy. A great variety of garden roses also exist, which are bred less for fragrance and more for color and shape. Even with the high price of roses and the advent of organic synthesis, rose oils are still the most widely used essential oils in perfumery. In fact, because of the labor-intensive production process and the low content of oil in rose blooms, rose oil is very expensive and is often called as 'liquid gold'. For the production of essential rose oil as well as rose extracts, the two rose species most often used are Rosa damascena (pink damask rose) and Rosa centifolia (light pink cottage rose). Whereas the former is predominantly used for rose oil production, the oil of the latter is usually extracted with solvents such as petroleum ether or n-hexane in order to obtain rose concrete. Rose concrete, the result of solvent extraction, is mainly composed of fragrance-related substances, and contains large quantities of paraffins, fatty acids, fatty acid methyl esters, di- and tri-terpenic compounds and pigments. The concrete can be therefore processed through Solvent-Free Microwave Extraction (SFME) as upcoming extraction technique for the extraction of essential oil to eliminate non-volatile compounds such as paraffins, shorten extraction time, reduce organic solvent consumption, improve extraction yield, enhance quality of the extract, prevent pollution and reduce sample preparation costs [1].

[1] M. Mohamadi, T. Shamspur, A. Mostafavi, Journal of Essential Oil Research 2012, 25, 55-61.

Why to choose Microwave Fragrances set-up?

The standard method is the Clavenger method, which was published for the first time in 1928. According to that method, the essential oil from Rosa Damascena Mill. can be extracted by hydrodistillation or steam distillation. These techniques take several hours of heating which may cause degradation of thermolabile compounds present in the starting plant material and therefore odor deterioration. The patented and innovative Microwave Hydrodistillation (MWHD) and Solvent-free Microwave Extraction (SFME) techniques allow the production of essential oils with higher quality.

- High quality frangrances
- No thermal degradation
- Fast extraction
- High purity, no artifacts

Instrumentation and Principles of Operation

A very efficient extraction process can be achieved thanks to the selective heating of microwaves to materials through molecular interactions with the electromagnetic field via conversions of electromagnetic energy into thermal energy. The high quality fragrance were obtained through the SFME or MWHD techniques. (See Cookbook for theory and principle).

Results and experimental procedure

The SFME and the MWHD techniques are respectively suitable for fresh and dry raw materials. See the “Quick start guide” for a list of easy and sequential setting-up operations (*Table 1*).

Fresh Rosa Damascena Mill., SFME						
Reactor	Weighted fresh raw material [g]*	Power [W]	Chiller		Volatile fraction [mL]	Yield [%]
			1kW	2.1kW		
Small	400	400	•		0.4	0.1
Medium	1264	1264		•	1.8	0.14
Large	2978	1800		•	3.9	0.13

*Time, Power
The extractions were carried out till complete recovery of the fragrance
≤ 1800 g: Power(W) = Weight(g).
> 1800g: Power = 1800W
Chiller settings:
≤ 900g, 1 kW Chiller
> 900g, 2.1 kW Chiller

Table 1

Important remarks

The system is developed with an automatic recirculation of the distilled water. This allows to manage extraction power and time to match your own specific requirements. Please take care to seal properly the glass reactor during the installation of the fragrances set-up according to the manual, to avoid loss of vapor during extraction.

Conclusion

In this study, we propose state-of-the-art processes for extraction of essential oils from Dry Rosa Damascena Mill. through SFME and MWHD. It is the unique modern concept of the antiquated Clavenger method, highly accelerating the isolation process, without causing changes in the volatile oil composition. The efficiency of the new techniques SFME and MWHD are considerably higher than the conventional procedure, if we take into account short distillation times required, cost and energy used and cleanliness of the process. An added-value feature is the possibility to work with different scalar matrices amounts due to three different volumes of the reactor vessels (small, medium, large), complying with a high range of extraction-scale needs.

Compound	RI[a]	SFME (%[b])
Monoterpenes		**0.6**
α-Pinene	858	0.2
Sabinene	896	tr[c]
β-Pinene	898	0.1
β-Myrcene	916	0.1
α-Terpinene	937	tr
Limonene	949	tr
γ-Terpinene	977	tr
α-Terpinolene	1003	tr
Oxygenated monoterpenes		**49.3**
Rosefuran	1009	tr
Linalool	1017	0.4
trans-Rose oxide	1023	0.3
Citronellal	1056	tr
Neroloxide	1061	tr
4-Terpineol	1081	0.1
β-Citronellol	1157	34.7
Geraniol	1173	9.1
Eugenol	1262	1.5
Geranyl acetate	1596	0.6
Methyl eugenol	1309	2.3
Neryl acetate	1926	0.2
Sesquiterpenes		**4.1**
β-Bourbonene	1302	0.2
trans-caryophyllene	1326	0.5

Table 2. Qualitative and quantitative composition of rose essential oils obtained by SFME

Compound	RI[a]	SFME (%[b])
α-Guaiene	1340	0.4
α-Humulene	1348	0.5
Germacrene D	1368	1.4
β-Selinene	1369	0.1
Caryophyllen (1l)	1378	0.1
α-Selinene	1379	-
δ-Guaiene	1381	0.4
(*E,E*)-α-Farnesene	1385	0.1
δ-Cadinene	1394	tr
Ledene	1518	0.3
Oxygenated sesquiterpenes		**2.2**
Elmol	1419	tr
Nerolidol	1437	0.1
Caryophyllene oxide	1446	tr
γ-Eudesmol	1505	0.1
β-Eudesmol	1516	0.2
cis-Farnesol	1555	1.8
Hydrocarbons		**31.4**
Pentadecane	1388	0.2
Hexadecane	1492	0.1
8-Heptadecene	1547	0.2
Heptadecane	1569	1.8
cis-9-Tricosene	1630	0.2
Octadecane	1669	0.4
(*Z*)-5-Nonadecene	1737	3.5
Nonadecane	1765	15.1
(*E*)-9-Eicosene	1811	0.6
Eicosane	1836	1.2
9-Nonadecene	1882	0.2
1-Nonadecene	1894	0.2
n-Heneicosan	1910	6.0
Docosan	1972	0.1
cis-9-Tricosene	2029	0.2
Tricosane	2039	0.9
Tetracosane	2099	tr
Pentacosane	2160	0.2
n-Heptacosane	2274	0.2
Other oxygenated compounds		**0.6**
Phenylethyl alcohol	813	0.4
Heptanal	1011	tr

Table 2 (continued).

Compound	RI[a]	SFME (%[b])
Tetradecanal	1024	0.1
Nonanal	1485	tr
Total oxygenated compounds		**52.1**
Total non-oxygenated compounds		**36.1**
Extraction time (min)		**210**
Oil yield (w/w%)		**0.018**

[a]*Retention indices relative to C6–C27 n-alkanes on HP-1MS column.*
[b]*%, relative percentage obtained on HP-1 column using GC/MS detector.*
[c]*tr, 60.05. *Significant at p60.05 based on F-value determined by analysis of variance (ANOVA). **Significant at ≤60.01 based on F-value determined by ANOVA. CV, coefficient of variation.*

Table 2 (continued).