MILESTONE
HELPING CHEMISTS

APPLICATION REPORT

EX10 - MINT FLAVORS

Mint Microwave Hydrodiffusion and Gravity (MHG)

Introduction

The mint species have a great importance, both medicinal and commercial. Indeed, leaves, flowers and stems of Mentha spp. are frequently used in herbal teas or as additives in commercial spice mixtures for many foods to offer aroma and flavor. In addition, Mentha spp. has been used as a folk remedy for treatment of nausea, bronchitis, flatulence, anorexia, ulcerative colitis, and liver complaints due to its antinflammatory, carminative, antiemetic, diaphoretic, antispasmodic, analgesic, stimulant, emmenagogue, and anticatharrhal activities. Commercially, the most important mint species are peppermint (M. x piperita), spearmint (M. spicata), and corn mint (M. canadensis). From these species, corn mint is cultivated only because of oil production. Peppermint oil is one of the most popular and widely used essential oils, mostly because of its main components menthol and menthone. Corn mint is the richest source of natural menthol (Sharma and Tyagi 1991; Shasany et al. 2000). Carvone-scented mint plants, such as spearmint, are rich in carvone and are widely used as spices and cultivated in several countries. Peppermint oil is used for

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flavouring pharmaceuticals and oral preparations, such as toothpastes, dental creams, and mouth washes. It is also used as a flavouring agent in cough drops, chewing gums, confectionery and alcoholic liqueurs. It is used in medicines for internal use. Its pleasant taste makes it an excellent gastric stimulant [1].

[1] H. Hajlaoui, N. Trabelsi, E. Noumi, M. Snoussi, H. Fallah, R. Ksouri, A. Bakhrouf, World Journal of Microbiology and Biotechnology 2009, 25, 2227-2238.

Why to choose Microwave Flavor set-up?

The patented and revolutionary Microwave Hydrodiffusion and Gravity (MHG) system pays the way to new flavoring products which were impossible to be obtained with the ancient extraction concepts. Percolation, solvent extraction ecc… were inefficient and environmental-unfriendly methods of flavor extraction. MHG is going to improve the efficiency and the quality of flavoring products.

- New natural flavors
- Fast extraction
- No thermal degradation
- No solvent

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 MHG techniques (see the “Microwave Extraction Techniques” section for theory and principle).

Results and experimental procedure

The MHG technique is suitable for both dry and fresh raw material, see the “Quick start guide” for a list of easy and sequential setting-up operations (*Table 1*).

Fresh Mint (MHG)						
Reactor	Weighted fresh raw material [g]*	Power [W]	Chiller		Total flavour extract [mL]	Total flavor extract yield [%]
			1kW	2.1kW		
Small	500	500	•		125	25
Medium	1580	1580		•	382	24.2
Large	3720	1800		•	960	25.8

Dry Mint (MHG)						
Reactor	Weighted fresh dry soaked material [g]*	Power [W]	Chiller		Total flavour extract [mL]	Total flavor extract yield [%]
			1 kW	2.1kW		
Small	500	500	•		110	22
Medium	1580	1580		•	302	19.1
Large	3720	1800		•	810	21.8

*Time, Power
≤ 1800 g: Power(W) = Weight(g) for 20 min.
> 1800g: Power = 1800W for 40min
Chiller settings:
≤ 900g, 1 kW Chiller
> 900g, 2.1 kW Chiller

Table 1

Important remarks

Please take into account that the interaction between microwaves and raw material is based on the water content. The optimized method (power and time) depends on the type of Mint, mainly to the water content of Mint itself. Please use the reported parameter as general application note to start to optimize your own method. Be careful, that using an excess power might cause burning of your sample.

Please take care to seal properly the glass reactor during the installation of the flavors set-up according to the manual, to avoid loss of vapor during extraction.

Conclusion

A newly and cleaner design process for extraction of flavors was developed in this study: MHG. This green process has been studied and tested using Mint. This new system was developed to date indicate that microwave extraction process of flavors offers important advantages over antiquated extraction techniques, namely, shorter extraction times, less energy consuming, lower costs as well as new flavoring products. The MHG system offers furthermore 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.

APPLICATION REPORT

EX11 - MINT FRAGRANCES

Mint Solvent-Free Microwave Extraction (SFME) and Microwave Hydrodistillation (MWHD)

Introduction

The mint species have a great importance, both medicinal and commercial. Indeed, leaves, flowers and stems of Mentha spp. are frequently used in herbal teas or as additives in commercial spice mixtures for many foods to offer aroma and flavor. In addition, Mentha spp. has been used as a folk remedy for treatment of nausea, bronchitis, flatulence, anorexia, ulcerative colitis, and liver complaints due to its antinflammatory, carminative, antiemetic, diaphoretic, antispasmodic, analgesic, stimulant, emmenagogue, and anticatharrhal activities. Commercially, the most important mint species are peppermint (M. x piperita), spearmint (M. spicata), and corn mint (M. canadensis). From these species, corn mint is cultivated only because of oil production. Peppermint oil is one of the most popular and widely used essential oils, mostly because of its main components menthol and menthone. Corn mint is the richest source of natural menthol (Sharma and Tyagi 1991; Shasany et al. 2000). Carvone-scented mint plants, such as spearmint, are rich in carvone and are widely used as spices and

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cultivated in several countries. Peppermint oil is used for flavouring pharmaceuticals and oral preparations, such as toothpastes, dental creams, and mouth washes. It is also used as a flavouring agent in cough drops, chewing gums, confectionery and alcoholic liqueurs. It is used in medicines for internal use. Its pleasant taste makes it an excellent gastric stimulant [1].

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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 Mint 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 the "Microwave Extraction Techniques" section 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 2*).

Fresh Mint, SFME						
Reactor	Weighted fresh raw material [g]*	Power [W]	Chiller		Volatile fraction [mL]	Yield [%]
			1kW	2.1kW		
Small	500	500	•		4.8	0.98
Medium	1580	1580		•	16	1.01
Large	3720	1800		•	37.2	1.0

Dry Mint, MWHD						
Reactor	Weighted dry soaked material [g]*	Power [W]	Chiller		Volatile fraction [mL]	Yield [%]
			1 kW	2.1kW		
Small	500	500	•		4.3	0.86
Medium	1580	1580		•	13.7	0.87
Large	3720	1800		•	30.9	0.83

*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 2

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 Mint 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.

	Fresh leaves SFME (g Kg^{-1})	Dried leaves MWHD (g Kg^{-1})
α-Pinene	4.0	7.4
Sabinene	4.6	6.3
β-Pinene	7.2	10.8
2-Thujene	15.9	14.7
3-Octanol	-	2.6
Limonene	67.5	8.67
1,8-Cineole	24.8	3.45
(E)-β-Ocimene	6.3	5.2
(Z)-β-Ocimene	3.4	2.0
γ -Terpinene	3.6	1.1
3-Carene	8.1	11.1
p-Menth-1-en-8-ol	-	1.4
(–)-4-Terpineol	7.1	2.6
t-Dihydrocarvone	18.9	17.3
c-Carveol	7.1	3.3
t-Carveol	-	2.3
D-Carvone	602.7	601.7
Piperitone	4.9	8.2
t-Carvone oxide	2.8	4.0
c-Carvone oxide	-	4.2
Dihydroedulan II	-	1.0
Dihydroedulan I	-	1.5
Isolimonene	5.8	-
Dihydrocarvyl acetate	-	3.2
t-Carveyl acetate	-	1.8
β-Bourbonene	29.7	24.7
β-Elemene	4.2	5.5
Isocaryophyllene	-	1.1

Table 3. Main components ofM. spicata L. var. rubra EO yield (g kg^{-1})

	Fresh leaves SFME (g Kg^{-1})	Dried leaves MWHD (g Kg^{-1})
β-Caryophyllene	44.8	42.3
α-Caryophyllene	3.9	3.8
(+)-Epi-bicyclosesquiphellandrene	9.8	9.0
Germacrene D	17.3	16.1
Bicyclogermacrene	4.4	3.5
α-Muurolene	-	1.3
Calamenene	9.3	5.6
Caryophyllene oxide	16.8	3.0
1,4-Cadinadiene	5.9	2.2
τ -Muurolol	4.1	1.2
Total	945.0	954.0

Table 3 (continued).