MILESTONE
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APPLICATION REPORT

EX15 - ROSEMARY FLAVORS

Rosemary Microwave Hydrodiffusion and Gravity (MHG)

Introduction

Rosemary (Rosmarinus officinalis L.) is a perennial herb with fragrant evergreen needle-like leaves. It is native to the Mediterranean region and it has been cultivated for a long time. It belongs to the Lamaiaceae family, which comprises up to 200 genera and about 3500 species. The leaves are evergreen, with dense short woolly hairs. Rosemary has been a significant herb since antiquity, although rosemary is more familiar to contemporary Westerners as a kitchen herb used to add a spicy or slightly medicinal flavor to some foods, it was traditionally used as an antiseptic, astringent, and food preservative before the invention of refrigeration. Rosemary's antioxidant properties are still used to extend the shelf life of prepared foods. Rosemary is also known medicinally for its powerful antioxidant activity, its antibacterial and antimutagenic properties, and as a chemopreventive agent. Besides the therapeutic application, the essential oil is widely applied in the cosmetic industry producing various Cologne waters, bathing essences, hair lotions and shampoos and as a component of disinfectants and insecticides [1].

[1] N. Bousbia, M. Abert Vian, M. Ferhat, E. Peticolas, B. Meklati, F. Chemat, Food Chemistry 2009, 14, 355-362.

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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 Rosemary (MHG)						
Reactor	Weighted fresh raw material [g]*	Power [W]	Chiller		Total flavour extract [mL]	Total flavor extract yield [%]
			1kW	2.1kW		
Small	500	500	•		267	53.3
Medium	1580	1580		•	855	54.1
Large	3720	1800		•	2001	53.8

Dry Rosemary (MHG)						
Reactor	Weighted dry soaked material [g]*	Power [W]	Chiller		Total flavour extract [mL]	Total flavor extract yield [%]
			1 kW	2.1kW		
Small	500	500	•		180	36
Medium	1580	1580		•	540	34.2
Large	3720	1800		•	1330	35.8

*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 Rosemary, mainly to the water content of Rosemary 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 Rosemary. 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.

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APPLICATION REPORT

EX16 - ROSEMARY FRAGRANCES

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

Introduction

Rosemary (Rosmarinus officinalis L.) is an important herb on the world food and aromatherapy market. Natural antioxidants such as those present in rosemary essential oil may be an alternative source for compounds capable of protecting lipids in foods. The essential oil secreted by glandular trichomes is mainly located in leaves. Rosemary essential oil is also used as an antibacterial and antifungal agent. Nevertheless, it has been noted that these activities often depend on the origin of the rosemary plant and the method of extraction. Since both of these quality parameters can greatly influence the chemical composition of rosemary oil, Solvent-Free Microwave Extraction (SFME) as upcoming extraction techniques have been reported for the extraction of fragrances from Rosemary [2].

[2] N. Tigrine-Kordjani, B. Meklati, F. Chemat, International Journal of Aromatherapy 2006, 16, 141-147.

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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 Rosemary 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 Rosemary, SFME						
Reactor	Weighted fresh raw material [g]*	Power [W]	Chiller		Volatile fraction [mL]	Yield [%]
			1kW	2.1kW		
Small	500	500	•		3.2	0.63
Medium	1580	1580		•	10.3	0.65
Large	3720	1800		•	26.8	0.72

Dry Rosemary, MWHD						
Reactor	Weighted dry soaked material [g]*	Power [W]	Chiller		Volatile fraction [mL]	Yield [%]
			1 kW	2.1kW		
Small	500	500	•		1.9	0.38
Medium	1580	1580		•	6.0	0.38
Large	3720	1800		•	14.9	0.4

*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 Rosemary 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.

N°	Compound[a]	SFME (%)	RI[b]	RI[c]
	Monoterpene hydrocarbons	68.56		
1	Tricyclene	0.26	921	1011
2	α-Pinene	44.05	936	1023
3	Camphene	6.14	951	1103
4	Verbenene	0.77	955	1121
5	β-Pinene	2.61	980	1109
6	Myrcene	1.94	995	1149
7	α-Phellandrene	0.31	995	1165
8	γ-3-Carene	0.08	1014	1290
9	α-Terpinene	0.86	1020	1083
10	para-Cymene	1.27	1025	1250
11	Limonene	5.48	1030	1206
12	γ-Terpinene	3.08	1052	1251
13	Terpinolene	1.71	1092	1287
	Oxygenated monoterpenes	24.87		
14	Linalool	2.00	1106	1538
15	α-Campholenal	1.24	1122	1471
16	Camphor	7.82	1149	1514
17	Pinocarvone	1.33	1160	1548
18	Borneol	2.57	1173	1679
19	Terpin-4-ol	2.07	1184	1590
20	α-Terpineol	0.77	1198	1677
21	Verbenone	6.37	1207	1696
22	Geraniol	0.70	1279	1828

Table 3. Chemical composition of Rosmarinus officinalis essential oils obtained by SFME.

N°	Compound[a]	SFME (%)	RI[b]	RI[c]
	Sesquiterpene hydrocarbons	1.91		
23	E-Caryophyllene	0.95	1425	1470
24	α-Humulene	0.42	1450	1657
25	γ-Curcumene	0.04	1469	1738
26	β-Bisabolene	0.43	1508	1714
27	β-Sesquiphellandrene	0.07	1519	1776
	Oxygenated sesquiterpenes	0.26		
28	Caryophyllene oxide	0.10	1570	1977
29	α-Bisabolol	0.16	1684	2022
	Other oxygenated compounds	1.03		
30	Bornyl acetate	0.81	1263	1579
31	Methyl eugenol	0.12	1397	2032
32	Z-Methyl jasmonate	0.10	1635	2349
Extraction time (min)		180		
Yield (%)		0.35±0.07		
Total oxygenated compounds		26.16		
Total non-oxygenated compounds		70.47		

[a] Essential oil compounds sorted by chemical families and percentages calculated by GC–FID on non-polar HP5MSTM capillary column.
[b] Retention indices relative to C_5–C_{28} n-alkanes calculated on non-polar HP5MS™ capillary column.
[c] Retention indices relative to C_5–C_{28} n-alkanes calculated on polar Carbowax™-PEG capillary column.

Table 3 (continued).