MILESTONE
HELPING CHEMISTS

APPLICATION REPORT

EX17 - THYMUS FRAGRANCES

Thymus vulgaris Solvent-Free Microwave Extraction (SFME) and Microwave Hydrodistillation (MWHD)

Introduction

Thymus vulgaris L. (common thyme), a member of the Labiatae family, is an aromatic/medicinal plant of increasing economic importance for North America, Europe, North Africa and Asia. Thyme is one of many aromatic plants that has been utilized in variety of food products to provide a flavor specific to this herb. Studies indicating the antiseptic, carminative, antimicrobial, and antioxidative properties of thyme have also been published. From the medicinal point of view, thyme has been used as a culinary herb and also as a herbal medicine. The essential oils of thyme are responsible for the typical spicy aroma of the plant leaves. These oils are stored in glandular peltate trichomes situated on both sides of the leaves. Results published on the chemical composition of thyme oil revealed that most of the oil was produced from flowering plants. In the plant's life cycle, the oil production is usually at its highest level during this period. For T. vulgaris and T. pulegioides, such finding was reported in the early 1960s [1].

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[1] M. Golmakani, K. Rezaei, Food Chemistry 2008, 109, 925-930.

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 Thymus vulgaris 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 1*).

Fresh Thymus vulgaris, SFME						
Reactor	Weighted fresh raw material [g]*	Power [W]	Chiller		Volatile fraction [mL]	Yield [%]
			1kW	2.1kW		
Small	400	400	•		1.3	0.32
Medium	1264	1264		•	4.4	0.35
Large	2978	1800		•	11	0.37

Dry Thymus vulgaris, MWHD						
Reactor	Weighted dry soaked material [g]*	Power [W]	Chiller		Volatile fraction [mL]	Yield [%]
			1 kW	2.1kW		
Small	400	400	•		0.6	0.16
Medium	1264	1264		•	1.9	0.15
Large	2978	1800		•	5.4	0.18

*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 Thymus vulgaris 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°	RT[b] (min)	Compound	I_K^c	Relative peak area[a] (%) SFME
1	16.05	α-Thujene	930	0.53 ± 0.02
2	16.53	α-Pinene	938	0.86 ± 0.01
3	17.31	Camphene	952	0.53 ± 0.01
4	19.04	1-Octen-3-ol	983	2.64 ± 0.31
5	19.60	β-Myrcene	993	1.30 ± 0.17
6	19.91	3-Octanol	999	0.19 ± 0.03
7	20.39	α-Phellandrene	1008	0.18 ± 0.01
8	20.71	Δ-3-Carene	1014	0.09 ± 0.01
9	21.12	α-Terpinene	1021	1.73 ± 0.14
10	21.73	ρ-Cymene	1033	17.57 ± 0.78
11	21.99	1,8-Cineole	1037	1.31 ± 0.12
12	23.55	γ-Terpinene	1066	8.54 ± 0.02
13	23.91	Trans-Sabinene hydrate	1073	0.94 ± 0.05
14	24.95	Terpinolene	1093	0.27 ± 0.05
15	25.59	Linalool	1105	2.43 ± 0.27
16	29.20	Borneol	1176	1.11 ± 0.21
17	29.69	Endo-Borneol	1185	1.41 ± 0.21
18	29.80	Terpinen-4-ol	1187	0.63 ± 0.16
19	31.20	α-Terpineol	1216	0.17 ± 0.00
20	32.34	Methyl thymylether	1240	0.14 ± 0.07
21	34.32	Geraniol	1281	0.39 ± 0.07
22	35.90	Thymol	1315	40.20 ± 3.03
23	36.50	Carvacrol	1328	6.84 ± 0.68

Table 2. Chemical compositions of essential oils obtained by solvent-free microwave extraction (SFME) of thyme aerial parts

N°	RT[b] (min)	Compound	I_K[c]	Relative peak area[a] (%) HD
24	38.22	Thymyl acetate	1366	0.16 ± 0.03
25	41.36	β-Caryophyllene	1438	2.86 ± 0.27
26	42.72	α-Humulen	1470	0.64 ± 0.22
27	42.98	Geranyl acetate	1477	0.42 ± 0.09
28	45.44	Δ-Cadinene	1536	0.40 ± 0.05
29	48.17	Caryophyllene oxide	1604	1.42 ± 0.21
Total peak area (%)			94.31	95.91
Total extraction time (min)			120	240
Yield (%)			2.39 ± 0.06%	2.52 ± 0.00%

[a] Mean ± SD (n = 2).
[b] Retention time.
[c] Kova´ts Retention Index (I_K) relative to C_9–C_{18} n-alkanes on the HP-5MS column.

Table 2 (continued).