ETHOS X APPLICATION REPORTS

Microwave Green Extraction of Natural Products

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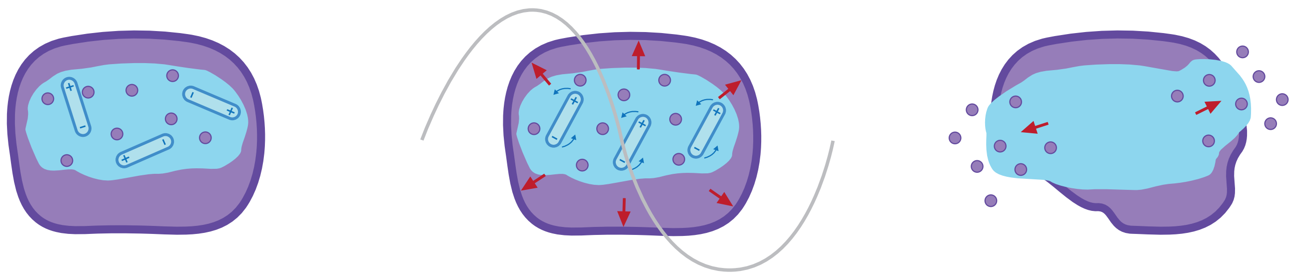
Introduction

The microwave heating is generated by two mechanisms: ionic conduction and dipole rotation. The electromagnetic field applied by the microwave irradiation (MW) causes the migration of ions of the solvent and the resistance of this ion conduction results in friction that heats the solution. The electromagnetic field produces also a rearrangement of dipoles, which contributes to the heating up of the solvent as well. This conversion of electromagnetic energy into thermal energy is peculiar and unique for microwave heating process. In conventional processes the energy is transferred to the material by convention, conduction and radiation phenomena from the external material, while in MAE the microwave energy leads to directly molecular interactions with the electromagnetic field and consequent heating up of the suscepting material[1].

The microwave extraction (MAE) process occurs as the result of changes in the cell structure caused by electromagnetic waves. Its process acceleration and high extraction yield may be the result of a synergistic combination of two transport phenomena: heat and mass gradients working in the same direction. In that sense MAE differs from the conventional extraction methods (solid-liquid or simply extraction) where the mass transfer occurs from the inside to the outside, although the heat transfer occurs from the outside to the inside of the substrate.



Microwave selective heating

Furthermore in MAE the dissipation of the heating takes place volumetrically inside the irradiated medium, while in conventional extraction the heat is transferred from the heating medium to the interior of the sample. There are six main steps that characterize the microwave extraction process conducted with a solvent: 1) penetration of the solvent into the solid matrix; 2) solubilization and/or break down of components; 3) transport of the solute out of the solid matrix by internal diffusion; 4) migration of the extracted solute from the external surface of the solid into the bulk solution by external diffusion; 5) movement of the extract with respect to the solid; 6) separation and discharge of the extract and solid[2]. Regarding the extraction process itself, three main subprocesses are known to take place and can be therefore pointed out: 1) desorption at an approximately constant velocity of the substrate from the outer surface of the particle (equilibrium phase); 2) after an intermediary transition phase, a mass transfer by internal diffusion and convection takes place in the solid-liquid interface; 3) in the last step the solute must overcame the interactions that bind it to the matrix and diffuse into the extracting solvent (external diffusion). This last step is considered as the limiting step of the process, since the extraction rate is really low and due to its irreversibility[3].

Important parameters in Microwave-Assisted Extraction

EFFECT OF EXTRACTION TIME

Extraction time represents another parameter to be taken into consideration during MAE. The period of heating is indeed an important factor in MAE, since a selective heating occurs and extraction times are very short (from a few minutes to a half-hour) in order to avoid thermal degradation or oxidation of target compounds, that might be sensitive against overheating[4]. The overheating depends on the dielectric properties of the solvent and it must be avoided especially in case of thermolable constituents. In that sense, when longer extraction time is required, multiple steps extraction cycles are the best solution to enhance extraction yields, avoiding long heating[4b, 5]. In flavonoids extraction from R. astragali for instance, it was found an increase in yield with time up to an exposure of 25 minutes and then the extraction yield started to decrease[5].

EFFECT OF MICROWAVE POWER AND EXTRACTION TIME

Similarly to the period of heating, microwave power and consequently temperature are factors that must be properly set in order to maximize the extraction efficiency, without causing the degradation of thermally sensitive compounds. It is known that in MAE temperature is controlled by incident microwave power that controls the amount of energy provided to the matrix, which is converted to heat energy in the dielectric material[2]. Microwave power is closely related to the extraction time required, the quantity of sample and of course to the type of sample we want to obtain the extract from. It acts as a driving force because it causes localized heating in plant matrix which allows the solute to diffuse out from the plant cells and to dissolve in the solvent. For that reason, an increase in the microwave power improves in general the extraction yield, ending up in shorter extraction time[4b, 6]. On the other hand it must be however taken into consideration that a high microwave power might also cause a rapid breakage of the cell wall, ending up with the leaching out both of the target compounds and of undesired impurities, in addition to causing the degradation of thermally sensitive compounds[7].

EFFECT OF THE SAMPLE CHARACTERISTICS

Another important parameter affecting the MAE process are the characteristics of the sample. It is well known that the higher the contact surface area is, the higher the extraction efficiency results, since finer particles allow a deeper penetration of the microwave[8]. A prior grinding and homogenizing of the samples are therefore strongly recommended. In some cases soaking of the dried plant material in the extracting solvent before MAE has also resulted in improved yield[7]. The moisture of the matrix acts as an extraction solvent as well, since it heats evaporates and causes internal ruptures of the cells, increasing though the extraction yield[9].

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EX00 - BASIL FRAGRANCES

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

Introduction

Ocimum basilicum L. (Lamiaceae), respectively, named basil, is an aromatic herb that has been used traditionally as a medicinal herb in the treatment of headaches, coughs, diarrhea, constipation, warts, worms and kidney malfunctions. It has a long history as culinary herb, thanks to its foliage adding a distinctive flavor to many foods. It is also a source of aroma compounds and essential oils containing biologically active constituents that possess insecticidal, nematicidal, fungistatic and antimicrobial properties[1].

[1] O. Politeo, M. Jukic, M. Milos, Food Chemistry 2007, 101, 379-385.

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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 basil 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 Basil, SFME						
Reactor	Weighted fresh raw material [g]*	Power [W]	Chiller		Volatile fraction [mL]	Yield [%]
			1kW	2.1kW		
Small	500	500	•		0.4	0.07
Medium	1580	1580		•	1.3	0.08
Large	3720	1800		•	3	0.08

Dry Basil, MWHD						
Reactor	Weighted dry soaked material [g]*	Power [W]	Chiller		Volatile fraction [mL]	Yield [%]
			1 kW	2.1kW		
Small	500	500	•		0.2	0.04
Medium	1580	1580		•	0.5	0.03
Large	3720	1800		•	1.5	0.04

*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 Basil 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 scalar amounts of sample due to three different reactor vessels (small, medium, large), complying with a high range of extraction-scale needs.

No.	Identified compound	Peak area (%)	RI[a] HP-20M	RI[a] HP-101
1	β-Pinene	0.1	-	949
2	Limonene	0.1	1180	1005
3	1,8-Cineole	4.0	1185	1006
4	Camphor	0.5	1477	1109
5	Linalool	28.6	1518	1092
6	Bornyl acetate	0.5	1545	1252
7	Terpinen-4-ol	0.7	1563	1154
8	α-Bergamotene	2.2	1564	1407
9	Caryophyllene	0.3	-	1385
10	Aloaromadendrene	0.1	-	1450
11	Estragole	21.7	1632	1177
12	α-Terpineol	1.0	1653	1176
13	Germacrene D	0.3	1673	1444
14	α-Humulene	0.2	-	1417
15	Carvone	0.4	1685	1207
16	β-Cubebene	0.5	1694	1059
17	β-Burbonene	t	-	1354
18	β-Elemene	0.3	-	1364
19	α-Cadinene	0.2	1716	1426
20	Calamenene	0.2	-	1483
21	α-Amorphene	1.0	1710	1479
22	β-Farnesene	0.2	-	1452
23	Δ-Cadinene	0.1	1724	1486
24	α-Bisabolene	0.1	-	1506
25	(Z)-Methyl cinnamate	1.6	1900	1281

Table 2. Chemical composition of basil essential oil

No.	Identified compound	Peak area (%)	RI[a] HP-20M	RI[a] HP-101
26	Methyl eugenol	3.1	1959	1378
27	(E)-Methyl cinnamate	14.3	2019	1364
28	Spatulenol	0.8	2066	-
29	Eugenol	5.9	2105	1368
30	Carvacrol	t	2118	1814
31	α-Cadinol	7.1	2120	1614
32	Torreyol	0.2	2173	-
33	Chavicol	0.7	-	-[b]
	Total	97.0		

–, not identified.
t, trace (<0.1%).
[a] Retention indices relative to C_8–C_{22} n-alkanes on polar HP-20M and apolar HP-101 column.
[b] Retention times is outside of retention times of homologous series of C_8–C_{22} n-alkanes (identified by MS).

Table 2 (continued).

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EX02 - CITRUS PEEL FRAGRANCES

Citrus Peel Solvent-Free Microwave Extraction (SFME) and Microwave Hydrodistillation (MWHD)

Introduction

Citrus essential oils are the most widely used essential oils in the world. They are obtained as by-products of the citrus processing. They are used as aroma flavor in many food products, including alcoholic and non-alcoholic beverages, candy, gelatins. In pharmaceutical industries they are employed as flavoring agents to mask unpleasant tastes of drugs. In perfumery and cosmetic, they are used in many preparations. The traditional way to extract essential oils is by cold pressing the citrus peels. The oil is present in oil sacs or oil glands located at different depths in the peel and the cuticles of the fruit. Peel and cuticle oils are removed mechanically by cold pressing and since cold pressing yields a watery emulsion, this emulsion is then centrifuged to separate out the essential oil. Distillation is also used in some countries as an economical way to recover the oils. During distillation, the Citrus peels exposed to boiling water or steam, release their essential oils through evaporation. Researchers in many universities are working on novel techniques that could lead to compact, safe,

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efficient, energy saving, and sustainable extraction processes. Solvent-Free Microwave Extraction (SFME) as upcoming extraction techniques have been reported for the extraction of fragrances and flavors from citrus peel[3].

[3] N. Bousbia, M. Abert Vian, M. Ferhat, B. Meklati, F. Chemat, Journal of Food Engineering 2009, 90, 409-413.

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 cistrus peel 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 Citrus peel, SFME						
Reactor	Weighted fresh raw material [g]*	Power [W]	Chiller		Volatile fraction [mL]	Yield [%]
			1kW	2.1kW		
Small	500	500	•		4.9	1.0
Medium	1580	1580		•	16	1.0
Large	3720	1800		•	48.4	1.3

Dry Citrus peel, MWHD						
Reactor	Weighted dry soaked material [g]*	Power [W]	Chiller		Volatile fraction [mL]	Yield [%]
			1 kW	2.1kW		
Small	500	500	•		2.8	0.56
Medium	1580	1580		•	9	0.57
Large	3720	1800		•	22.7	0.61

*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 Citrus peel 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 scalar amounts of sample due to three different reactor vessels (small, medium, large), complying with a high range of extraction-scale needs.

No.	Compounds[a]	R.I.[b]	R.I.[c]	SFME
	Monoterpenes			**92.76**
1	Pinene<Alpha->	926	1023	1.75
2	Pinene<Beta->	974	1109	15.35
3	Myrcene<Beta->	988	1165	1.33
4	Carene<Delta-3-> 1101	1101	1290	0.20
5	Limonene	1030	1206	65.25
6	Terpinene<Gamma->	1103	1285	8.08
	Oxygenated Monoterpenes			**92.76**
7	Linalool	1125	1538	0.18
8	Citron ellal	1167	1478	0.05
9	Terpin-4-ol	1191	1590	0.42
10	Terpineol<Alpha->	1203	1677	0.56
11	Nerol	1237	1781	0.49
12	Neral	1268	1670	0.68
13	Geraniol	1271	1828	0.60
14	Geranial	1284	1714	0.89
	Sesquiterpenes			**1.06**
15	Elemene<Beta->	1373	1583	-
16	Caryophellene<E->	1391	1594	0.18
17	Bergamotene<Alpha-Trans->	1437	1577	0.28
18	Humulene<Alpha->	1450	1657	0.04
19	Farnesene<(E)-Beta->	1453	1650	0.02
20	Germacrene D	1477	1696	-
21	Valencene	1488	1705	0.04
22	Bisabolene<(Z)-Alpha->	1498	1761	0.03
23	Bisabolene(Beta-)	1508	1718	0.44

Table 3. Chemical compositions of essential oils from citrus peel obtained by SFME

No.	Compounds[a]	R.I.[b]	R.I.[c]	SFME
	Oxygenated Sesquiterpenes			**0.03**
24	Elemol	1540	1381	-
25	Nerolidol<E->	1555	2026	-
25	Bisabolol<Alpha->	1684	2212	0.03
27	Nootkatone	1799	2250	-
	Other oxygenated compounds			**0.86**
28	Nonanal<N->	1126	1400	0.06
29	Citronellyl Acetate	1342	1645	0.04
30	Neryl Acetate	1351	1706	0.19
	Extraction time (min)			180
	Yield (%)			0.8
	Total oxygenated compunds (%)			4.78
	Total non oxygenated compounds (%)			93.82

[a] Essential oil compounds sorted by chemical families and percentages calculated by GC-FID on non-polar HP5MS™ capillary column.
[b] Retention indices calculated on non-polar HP5MS™ capillary column.
[c] Retention indices calculated on polar Carbowax™-PEG capillary column.

Table 3 (continued).

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EX03 - FRANKINCENSE FRAGRANCES

Hydrodistillation (MWHD)

Introduction

Gum resins from Boswellia species, also known as frankincense, have been used as a major ingredient in Ayurvedic and Chinese medicine to treat a variety of health-related conditions. Both frankincense chemical extracts and essential oil prepared from Boswellia species gum resins exhibit anti-neoplastic activity, and have been investigated as potential anti-cancer agents. The anti-cancer activity is mediated through multiple signaling pathways. In addition, frankincense essential oil overcomes multicellular resistant and invasive phenotypes of human breast cancer cells. Fast and green Extraction of Frankincense essential oil turns out therefore to be extremely important. This essential oil is obtained from a resin from the bark of a shrub originally from the area surrounding the Red Sea, in Somalia and Arabia. To collect the resin, fine incisions are made in the bark, and drops of sap appear and dry in large, odorous yellow droplets[1].

[1] X. Ni, M. Suhail, Q. Yang, A. Cao, K.-M. Fung, R. Postier, C. Woolley, G. Young, J. Zhang, H.-K. Lin, BMC Complementary and Alternative Medicine 2012, 12.

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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 Frankincense 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) technique allows 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*).

Frankincense, MWHD						
Reactor	Weighted dry material + added water [g]*	Power [W]	Chiller		Volatile fraction [mL]	Yield [%]
			1 kW	2.1kW		
Small	1050	1050	•		5.5	0.52
Medium	3318	1800		•	17.6	0.53
Large	7816	1800		•	43.8	0.56

*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 Frankincense resin 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 scalar amounts of sample due to three different reactor vessels (small, medium, large), complying with a high range of extraction-scale needs.

RRI	Compounds	Boswellia rivae (%)
1032	α-Pinene	5.3
1035	α-Thujene	1.3
1076	Camphene	0.1
1118	β-Pinene	0.6
1132	Sabinene	1.2
1159	δ-3-Carene	9.6
1187	o-Cymene	2.5
1203	Limonene	14.8
1213	1,8-Cineole	0.3
1266	(E)-β-Ocimene	0.4
1278	m-Cymene	0.4
1280	p-Cymene	2.9
1424	o-Methylanisol	0.2
1430	α-Thujone	0.1
1439	γ-Campholene aldehyde	0.1
1444	2,5-Dimethylstyrene	0.2
1450	trans-Linalool oxide	0.1
1451	β-Thujone	0.7
1458	cis-1,2-Limonene epoxide	4.6
1468	trans-1,2-Limonene epoxide	0.5
1474	trans-Sabinene hydrate	0.9
1478	cis-Linalool oxide	0.1
1498	(E)-β-Ocimene epoxide	0.2
1499	α-Campholene aldehyde	0.9
1536	Pinocamphone	0.3
1553	Linalool	0.2
1556	cis-Sabinene hydrate	0.9
1565	8,9-Limonene epoxide-I	0.6
1571	8,9-Limonene epoxide-II	0.6
1586	Pinocarvone	0.5
1597	Bornyl acetate	0.4
1600	β-Elemene	0.3
1611	Terpinen-4-ol	1.4
1524	trans-Dihydrocarvone	0.2
1639	trans-p-Mentha-2,8-diene-1-ol	3.9
1642	Thuj-3-en-10-al	0.2
1648	Myrtenal	0.5
1651	Sabinaketone	0.2
1657	Umbellulone	0.1
1663	cis-Verbenol	0.5
1664	trans-Pinocarveol	2.2
1678	cis-p-Mentha-2,8-diene-1-ol	0.9
1683	trans-Verbenol	6.8
1700	p-Mentha-1,8-diene-4-ol	0.4
1706	α-Terpineol	1.4
1709	α-Terpinyl acetate	1.0
1720	trans-Sabinol	0.3
1725	Verbenone	4.3
1751	Carvone	1.6
1804	Myrtenol	0.7
1811	trans-p-Mentha-1(7),8-diene-2-ol	0.3
1845	trans-Carveol	2.5
1856	m-Cymen-8-ol	3.1
1864	p-Cymen-8-ol	2.0
1882	cis-Carveol	0.7
1896	cis-p-Mentha-1(7),8-diene-2-ol	0.2
1949	Piperitenone	0.6
2113	Cumin alcohol	0.1
2198	Thymol	0.1
2239	Carvacrol	0.1
	Total	88.1

Table 2. The composition of Boswellia Rivae essential oil

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EX05 - GARLIC FRAGRANCES

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

Introduction

Garlic (Allium sativum) belongs to a group of dietary supplements that lessen the incidence of cardiovascular and cerebrovascular diseases by reducing cholesterol concentration. The beneficial effect of garlic on health confirm several studies which were showed that garlic has been evaluated for lowering blood pressure, cholesterol and glucose concentration, reduce blood lipids as well as for the prevention of arteriosclerosis and cancer. The biological activities of garlic including antibacterial, parasiticidal, antithrombotic, antioxidant and antidiabetic actions have been known for a long time. The unique flavor and health-promoting functions of garlic are generally attributed to its rich content of sulfur-containing compounds: alliin, g-glutamylcysteine and their derivatives.

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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 garlic 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 Garlic, SFME						
Reactor	Weighted fresh raw material [g]*	Power [W]	Chiller		Volatile fraction [mL]	Yield [%]
			1kW	2.1kW		
Small	1000	1000	•		2	0.20
Medium	3160	1800		•	8.2	0.26
Large	7445	1800		•	22.3	0.30

*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 Garlic 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 scalar amounts of sample due to three different reactor vessels (small, medium, large), complying with a high range of extraction-scale needs.

Compounds	LRI_{HP1}	LRI_{INNO}	SFME (% ± SD)	Identification methods
Dimethyl sulfide	-	750	tr	SM, LRI
Dimethyl disulfide	731	1099	tr	SM, LRI, Std
Methyl ethyl disulfide	818	1119	0.5	SM, LRI
Diallyl sulfide	840	1150	0.5	SM, LRI, Std
Methyl allyl disulfide	894	1290	0.8 ± 0.1	SM, LRI, Std
(Z)-prop-1-enyl methyl disulfide	917	1251	tr	SM, LRI
(E)-prop-1-enyl methyl disulfide	919	1275	0.1	SM, LRI
Dimethyl trisulfide	941	1340	0.2	SM, LRI, Std
Diallyl disulfide	1056	1491	26.3 ± 0.9	SM, LRI, Std
Allyl (Z)-prop-1-enyl disulfide	1073	1390	2.9 ± 0.1	SM, LRI
Allyl (E)-prop-1-enyl disulfide	1082	1415	7.7 ± 0.1	SM, LRI
Ally methyl trisulfide	1115	1601	7.9 ± 0.1	SM, LRI, Std
Methyl (E)-propenyl trisulfide	1138	-	0.1	Tentative
3-vinyl-(4H)-1,2-dithiin	1156	1735	1.1	SM, LRI
Unknown 1	1169	-	0.1	-
2-vinyl-(4H)-1,3-dithiin	1178	1830	3.1	SM, LRI
Diallyle trisulfide	1285	1825	39.7 ± 0.8	SM, LRI, Std
Propyl propenyl trisulfide[d]	1290	1781	0.3	-
Allyl propenyl trisulfide	1300	1798	2.0	SM, LRI
3,5-diethyl-1,2,4-trithiolane	1321	1788	0.2	SM, LRI
Unknown 2	1348	2011	0.4	-
Diallyl tetrasulfide	1508	-	1.5	SM, LRI, Std
2,4-dimethyl-5,6-dithia-2,7-nonadienal	1718	> 2400	0.9 ± 0.1	SM, LRI
2,4-dimethyl-5,6-dithia-2,7-nonadienal	1730	> 2400	0.4 ± 0.1	SM, LRI
Unknown 3	1775	-	0.1	-

[a] Compounds are listed in order of their elution time from a HP-1 column. Compositional values less than 0.1% are denoted as traces (tr). Presence of a compound is indicated by its GC-FID percentage with S.D., absence is indicated by "-".
[b] RI = retention indices are determined on HP-1 and INNOWAX column using the homologous series of n-alkanes (C6-C24).
[c] S.D. = standard deviation.
[d] Correct isomer not identified
tentative: tentatively identified by MS and RI without standard compound co-injection
Unknown 1: 186 (M+., 7.1) ; 162(23.2) ; 100 (15) ; 97 (28.3) ; 73(50.1) ; 60 (16.0) ; 56(15.6) ; 59 (28.2) ; 57 (31.8) ; 45 (45.5) ; 41(100).
Unknown 2: 178 (M+., 3.4) ; 172(16.6) ; 170 (100) ; 128(42.3) ; 106(25.3) ; 73(12.3) ; 64(62.3) ; 59(17.3) ; 45(26.8) ; 42 (11.7) ; 41(31).
Unknown 3: 179(M+. , 5.6) ; 147(89.0) ; 106(10.5) ; 105(33.4) ; 75(14.6) ; 73(98.7) ; 64 (14.8) ; 57(10,7) ; 47 (15.4) ; 45(29.7) ; 41(100).

Table 3. Essential oil from garlic (Allium sativum oil) obtained by SFME

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APPLICATION REPORT

EX07 - GINGER FRAGRANCES

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

Introduction

Due to its composition in valuable natural compounds, ginger represents a suitable matrix for extraction of essential oil as well as many other bioactive compounds. It contains indeed products of interest such as essential oils (1–4%), phenolics (gingerols and 6-shogaol, 1–2%), and total carbohydrates (60–75%). Ginger, and more specifically rhizomes are variously used as food product or in traditional medicine. In the food industry, rhizomes are mainly used for spices or condiments (fresh or dried), candy or as juice after cold mechanical pressing. Due to the fact that mechanical pressing does not alter the chemical composition of the pressed product, this process provides huge amounts of press cake still containing high amounts of bioactive compounds, which is currently considered as waste. The novelty of the extraction of these products through MHG and SFME relies on the extraction of compounds achieved without addition of solvent or water. The only water used in the process is the matrix water naturally present in the plant cells [1].

[1] M. Navarro-Jacotet, N. Rombaut, S. Deslis, A. Fabiano-Tixter, F. Pierre, A. Bily, F. Chemat, Green Chemistry 2016, 18, 3016.

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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 Ginger 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 Ginger, SFME

Reactor	Weighted fresh raw material [g]*	Power [W]	Chiller		Volatile fraction [mL]	Yield [%]
			1kW	2.1kW		
Small	1000	1000	•		0.7	0.08
Medium	3160	1800		•	2.2	0.07
Large	7445	1800		•	5.2	0.07

*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 Ginger 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 scalar amounts of sample due to three different reactor vessels (small, medium, large), complying with a high range of extraction-scale needs.

		GR	GP	0.6 W g^{-1}	0.8 W g^{-1}	1.0 W g^{-1}	1.2 W g^{-1}	1.4 W g^{-1}	1.6 W g^{-1}	1.8 W g^{-1}
Essential oil *Yield (g per 100 g fresh plant material). Major compounds (%)*		0.2	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.2
	β-Pinene	1.2	1.0	2.3	2.6	2.4	2.6	2.3	2.4	2.2
	Camphene	4.3	3.8	9.1	10.3	9.2	10.0	9.1	9.4	9.1
	Sabinene	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
	Sulcatone	0.0	0.8	1.2	2.8	3.3	3.2	3.0	3.2	2.9
	Myrcene	0.6	0.6	0.0	1.4	1.4	1.4	1.3	1.3	1.1
	α-Phellandrene	0.2	0.1	0.2	0.3	0.3	0.3	0.3	0.3	0.1
	Limonene	0.9	0.9	1.7	1.9	1.9	1.9	1.7	1.8	1.7
	β-Phellandrene	4.6	4.2	8.7	10.4	10.3	10.2	9.7	10.0	8.6
	Terpinolene	0.1	0.1	0.1	0.1	0.3	0.3	0.3	0.3	0.2
	Linalol	0.2	0.2	0.3	0.4	0.4	0.4	0.4	0.4	0.4
	Borneol	0.5	0.6	0.8	0.9	1.0	0.9	1.0	1.0	1.1
	α-Terpineol	0.2	0.3	0.4	0.5	0.5	0.5	0.5	0.5	0.6
	Citronellol	0.1	0.3	0.2	0.5	0.4	0.3	0.4	0.4	0.8
	Neral	1.7	0.5	0.4	1.3	1.5	1.7	1.5	1.5	1.3
	Geraniol	0.1	0.2	0.1	0.3	0.3	0.2	0.2	0.2	0.6
	Geranial	3.3	1.0	0.6	1.9	2.2	2.6	2.3	2.5	2.3
	Geranyl acetate	0.3	0.1	0.4	0.2	0.2	0.2	0.2	0.2	0.2
	α-Curcumene	3.5	13.9	17.0	7.6	7.2	6.6	7.0	6.8	9.9
	Germacrene D	1.6	1.3	0.1	1.3	1.4	1.4	1.4	1.4	0.7
	Zingiberene	35.7	25.2	18.4	23.2	24.0	24.0	25.1	24.3	18.4
	α-Farnesene	6.5	6.5	6.3	5.4	5.5	5.5	5.7	5.5	5.7
	β-Bisabolene	5.7	6.8	0.0	4.8	4.7	4.6	4.8	4.7	5.4
	β-Sesquiphellandrene	12.1	13.9	12.3	9.9	9.9	9.7	10.2	9.8	10.4
Antioxidants *Total content (g per 100 g plant material DW). Major compounds (g per 100 g plant material DW)*		1.17	0.90	0.57	1.24	1.06	1.18	1.22	1.37	1.18
	6-Gingerol	0.77	0.58	0.31	0.81	0.65	0.79	0.81	0.92	0.79
	8-Gingerol	0.15	0.11	0.07	0.14	0.11	0.14	0.14	0.17	0.14
	10-Gingerol	0.23	0.19	0.11	0.18	0.19	0.19	0.19	0.21	0.19
	6-Shogaol	0.02	0.02	0.08	0.11	0.10	0.08	0.09	0.08	0.08

Table 3. Volatile compounds and antioxidants extracted from ginger plant material by SFME

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APPLICATION REPORT

EX09 - LAVANDER FRAGRANCES

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

Introduction

Lavender is one of the most useful medicinal plants. Commercially, the lavender provides several important essential oils to the fragrance industry, including soaps, colognes, perfumes, skin lotions and other cosmetics. In food manufacturing, lavender essential oil is employed in flavouring beverages, ice cream, candy, baked goods and chewing gum. The essential oils of Lavandula species are obtained by steam distillation of the fresh flowering spikes. Oil quality is assessed by oil chemical composition and by the organoleptic opinion. In addition, a large range of medical uses for this plant have also been reported. These include antispasmodic, sedative, antihypertensive, antiseptic, healing and anti-inflammatory properties, all of which render it highly appreciated in phytotherapy and aromatherapy. In food manufacturing, lavander essential oil has been employed in flavoring beverages, ice cream, baked goods and chewing gum [1].

[1] F. Chemat, M. Lucchesi, J. Smadja, L. Favretto, G. Colnaghi, F. Visinoni, Analytica Chimica Acta 2006, 555, 157-160.

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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 Lavander 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 Lavander, SFME						
Reactor	Weighted fresh raw material [g]*	Power [W]	Chiller		Volatile fraction [mL]	Yield [%]
			1kW	2.1kW		
Small	1000	1000	•		2.5	0.25
Medium	3160	1800		•	6.7	0.21
Large	7445	1800		•	15	0.22

Dry Lavander, MWHD						
Reactor	Weighted dry soaked material [g]*	Power [W]	Chiller		Volatile fraction [mL]	Yield [%]
			1 kW	2.1kW		
Small	1000	1000	•		1.7	0.17
Medium	3160	1800		•	6.6	0.21
Large	7445	1800		•	14.2	0.19

*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 flavors 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 Lavander 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 scalar amounts of sample due to three different reactor vessels (small, medium, large), complying with a high range of extraction-scale needs.

No.	Compounds	R.R.I.[a]	MWHD[b] (%)
	Monoterpenes		3.54
1	α-Thujene	907	0.08
2	α-Pinene	914	0.51
3	Camphene	933	0.32
4	Sabinene	963	0.14
5	β-Pinene	968	0.59
6	β-Myrcene	985	0.50
7	3-Carene	1008	0.22
8	Limonene	1024	tr.
9	(Z)-β-Ocimene	1031	0.33
10	(E)-β-Ocimene	1040	0.37
11	γ-Terpinene	1050	0.09
12	Terpinolene	1078	0.37
	Oxygenated monoterpenes		78.29
13	1,8-Cineole	1027	7.23
14	Sabinene hydrate-cis	1058	0.66
15	Linalool	1099	47.82
16	Camphor	1137	11.82
17	Borneol	1161	4.15
18	Terpin-4-ol	1174	5.94
19	ρ-Cymen-8-ol	1179	tr.
20	α-Terpineol	1186	0.68
	Sesquiterpenes		2.77
21	α-Bergamotene cis	1400	0.10
22	β-Caryophyllene	1412	1.28

Table 3. Yields, extraction times, grouped compounds and chemical compositions of essential oils obtained by MWHD from lavender flowers

No.	Compounds	R.R.I.[a]	MWHD[b] (%)
23	α-Santalene	1414	0.15
24	(E)-β-Farnesene	1453	0.63
25	Sesquiterpene 1	1474	0.61
	Oxygenated sesquiterpenes		0.29
26	Caryophyllene oxide	1573	0.11
27	α-Bisabolol	1677	0.18
	Other oxygenated compounds		15.01
28	Octan-3-one	977	0.78
29	Octan-3-ol	990	0.26
30	Dihydromyrcenol	1063	0.34
31	n.i.	1141	0.37
32	n.i.	1188	2.00
33	n.i.	1232	0.43
34	Linalool acetate	1254	10.74
35	Geranyl acetate	1377	0.08
	Yield (%)		8.86
	Total extraction time (min)		10
	Heating time from 20 to 100 °C (min)		5
	Real extraction time (min)		5

tr., trace; n.i., non-identified.
[a] R.R.I., relative retention indices relative to C_8–C_{22} n-alkanes on SBP5™ capillary column.
[b] MWHD, microwave hydrodistillation.

Table 3 (continued).

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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].

[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 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).

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APPLICATION REPORT

EX13 - ORANGE PEEL FRAGRANCES

Orange Peel Solvent-Free Microwave Extraction (SFME) and Microwave Hydrodistillation (MWHD)

Introduction

Orange essential oil is used to confer the aroma and flavor of orange to a wide variety of products, such as carbonated drinks, ice-creams, cakes, air-fresheners, perfumes and so on. They are also being used in the design of new products, to which they add aroma and flavor 2, 3 and 4. Another application takes advantage of the germicide properties of some of their components. In that sense, a small amount of d-limonene was very effective in the germicide treatment of waste waters. Carotenoid pigments, found in orange extracts, are important for health, not only because of their nutritional value as precursors of vitamin A, but also because of their antioxidant potential. They also seem to have certain anticarcinogenic properties. These compounds are used in food coloring [1].

[1] B. Mira, M. Blasco, A. Berna, S. Subirats, Journal of Supercritical Fluids 1999, 14, 95-104.

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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 Orange peel 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 Orange peel, SFME						
Reactor	Weighted fresh raw material [g]*	Power [W]	Chiller		Volatile fraction [mL]	Yield [%]
			1kW	2.1kW		
Small	500	500	•		4.9	1.0
Medium	1580	1580		•	16	1.0
Large	3720	1800		•	69	1.9

Dry Orange peel, MWHD						
Reactor	Weighted dry soaked material [g]*	Power [W]	Chiller		Volatile fraction [mL]	Yield [%]
			1 kW	2.1kW		
Small	500	500	•		2.8	0.56
Medium	1580	1580		•	9	0.57
Large	3720	1800		•	23.1	0.62

*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 Orange peel 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	I[a]	SFME (%)
Monoterpenes			
1	α-Pinene	928	0.60
2	Sabinene	968	0.23
3	β-Myrcene	988	1.81
4	3-Carene	1001	tr
5	δ-3-Carene	1007	0.05
6	Limonene	1034	96.20
7	α-Terpinolene	1087	0.01
Oxygenated monoterpenes			
8	Linalool	1093	0.17
9	Trans-Limonene oxide	1135	0.02
10	Citronellal	1150	0.02
11	β-Citronellol	1227	0.06
Sesquiterpene			
12	α-Copaene	1372	0.01
13	β-Cubebene	1382	tr
14	β-elemene	1386	0.01
15	Caryophyllene (E)	1417	0.01
16	α-Humulene	1452	0.05
17	Germacrene-D	1479	tr
18	Valencene	1490	0.01
19	Germacrene-A	1503	0.01
20	δ-Cadinene	1520	0.01

Table 3. Chemical composition of orange EOs obtained by SFME

N°	Compound	I[a]	SFME (%)
Oxygenated sesquiterpenes			
21	Caryophyllene oxide	1589	tr
22	Cis, trans -Farnesol	1694	-
23	α-Sinensal	1754	tr
Other oxygenated compounds			
24	Decanal	1203	0.16
25	n-Dodecanal	1404	0.02
Extraction time (min.)		6	120
Yield (%)		5.43	5.45

tr. tracesb0.01%.
[a] I, Retention indices relative to C5–C28 n-alkanes calculated on non-polar HP5MS capillary column. Percentages calculated by GC–FID on non-polar HP5MS capillary column. Essential oil compounds sorted by chemical families.

Table 3 (continued).

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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

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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).

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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).

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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].

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

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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 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).