MILESTONE
HELPING CHEMISTS

APPLICATION REPORT

EX04 - GARLIC FLAVORS

Garlic Microwave Hydrodiffusion and Gravity (MHG)

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.

rev.00/2016

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 naturl 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 Garlic (MHG)

Reactor	Weighted fresh raw material [g]*	Power [W]	Chiller		Total flavour extract [mL]	Total flavor extract yield [%]
			1kW	2.1kW		
Small	1000	1000	•		185	18.5
Medium	3160	1800		•	600	19
Large	7445	1800		•	1400	18.8

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

MILESTONE
HELPING CHEMISTS

APPLICATION REPORT

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.

rev.00/2016

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