MILESTONE
HELPING CHEMISTS

APPLICATION REPORT

EX12 - ORANGE PEEL FLAVORS

Orange peel Microwave Hydrodiffusion and Gravity (MHG)

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.

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 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 Orange peel (MHG)

Reactor	Weighted fresh raw material [g]*	Power [W]	Chiller		Total flavour extract [mL]	Volatile fraction [mL]	Total flavor extract yield [%]
			1kW	2.1kW			
Small	500	500	•		185	9.5	37
Medium	1580	1580		•	599	30	38
Large	3720	1800		•	1432	70.5	38.5

Dry Orange peel (MHG)

Reactor	Weighted dry soaked material [g]*	Power [W]	Chiller		Total flavour extract [mL]	Volatile fraction [mL]	Total flavor extract yield [%]
			1 kW	2.1kW			
Small	500	500	•		150	6.2	30
Medium	1580	1580		•	540	24	34.2
Large	3720	1800		•	1330	59	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 Orange fruit, mainly to the water content of Orange fruit 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 orange peel. 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

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.

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 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).