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Component Analysis of Volatile Oil from *Chrysanthemum morifolium* by Supercritical Carbon Dioxide Extraction

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In this study, the volatile oil was extracted from *Chrysanthemum morifolium* by supercritical carbon dioxide extraction. Ninety three peaks were separated and 26 kinds of compounds were identified by GC-MS and their relative contents were determined by area normalization method. The main components in *Chrysanthemum morifolium* are monoterpenes hydrocarbons, sesquiterpenes hydrocarbons and their oxo-derivatives, *etc*.

Keywords: *Chrysanthemum morifolium*, Volatile oil, Supercritical carbon dioxide extraction, GC-MS.

INTRODUCTION

Chrysanthemum morifolium (also known as feverfew), *Chuzhou chrysanthemum*, *Bo-chrysanthemum* and *Florists Chrysould likehemum* are generally considered as the four famous Chinese chrysanthemum. *Chrysanthemum morifolium* is rich in the north of zhejiang province and especially in Tongxiang City[1]. It is a special variety which has the homology of medicine and food in the family of chrysanthemum and also is one of the well-known traditional chinese medicine. *Chrysanthemum morifolium* has many beneficial effects such as dispelling wind and heat, improving eyesight and detoxicating. Modern pharmacological studies have shown that it also can resist inflammation[2], resist virus[3], resist tumour[4] and resist aging[5].

The volatile oil is one of the major bioactive components in *Chrysanthemum morifolium*. At present, the main extraction methods of volatile oil from *Chrysanthemum morifolium* are direct distillation extraction method[6,7] and ultrasonic extraction method[8], *etc*. And the major analysis methods are GC-MS[9] and GC × GC/TOFMS[10], *etc*. Whereas, the component analysis of volatile oil in *Chrysanthemum morifolium* by supercritical carbon dioxide extraction has not been reported yet.

In this study, the volatile oil in *Chrysanthemum morifolium* extracted by supercritical carbon dioxide extraction (the extraction rate of volatile oil was 5.92 %) was analysed with GC-MS and then identified 26 kinds of compounds and determined the relative percentage of each component by area normalization method.

EXPERIMENTAL

Supercritical carbon dioxide extraction device (model: HL-0.5/50 MPaIIIA; Hangzhou Huali Pump Industry Co., Ltd); GC-MS(model: 6890N/5975; Agilent); Electronic analytical balance(model: AB204-N); *Chrysanthemum morifolium* (Hangzhou Chinese Medicine Factory); Diethyl ether (analytical grade; Hangzhou Chemical Reagent Co., Ltd); CO_2 (purity ≥ 99 %; Hangzhou Minxing Industry and Trade Co., Ltd).

Extraction of volatile oil: Put 100 g *Chrysanthemum morifolium* powder in the extraction kettle, opened the main pump, added to the required pressure and regulated the flow rate of CO_2. Started to extract circularly when the preset temperature of the refrigerating device, separation and extraction kettle were reached. Kept extracting 2 h under the condition of extraction pressure 20 MPa, extraction temperature 55 °C and the flow rate of CO_2 10 kg/h. The extraction rate of volatile oil was 5.92 %.

Chromatogram conditions: HP-5 quartz capillary column, 30 m × 0.25 mm (length × inner diameter), thickness of stationary phase 0.25 μm, injection temperature 280 °C. Temperature-increasing procedure: started at 60 °C, maintained for 2 min, increased to 100 °C at 10 °C/min, maintained for 2 min, then to 180 °C at 2 °C/min, maintained for 10 min, then to 280 °C at 10 °C/min, maintained for 10 min. He as carrier gas, its flow rate is 0.9 mL/min, pressure 7.23 kPa and injection 0.1 μL volume.

MS conditions: The spectrometers were operated in electron-impact mode, the scan range was 50-550 amu,

detection electric pressure 1576 V, the ionization energy was 70 eV. The ionization source temperature was 230 °C and the solvent delay was 3 min.

Component analysis of volatile oil: Analyzed the components of volatile oil from *Chrysanthemum morifolium* according to the above-mentioned method, obtained the mass spectrogram by scanning to each peak of the total ion flow chart as shown in Fig. 1, the components of volatile oil were identified as shown in Table-1 with the help of a spectral library search system and some relevant mass spectra informations, then determined the relative percentage of each component by area normalization method as shown in Table-1.

RESULTS AND DISCUSSION

The volatile oil from *Chrysanthemum morifolium* was extracted by supercritical carbon dioxide with 5.92 % extraction rate. We isolate and analyze the chemical components of the volatile oil by capillary column gas chromatography, determine the relative percentage of each component by area normalization method, identify its chemical compounds by GC-MS, isolate 93 peaks and identify 26 compounds which comprise 56.09 % of the total. The main ingredients in *Chrysanthemum morifolium* are monoterpenes hydrocarbons, sesquiterpenes hydrocarbons and their oxo-derivatives, *etc*.

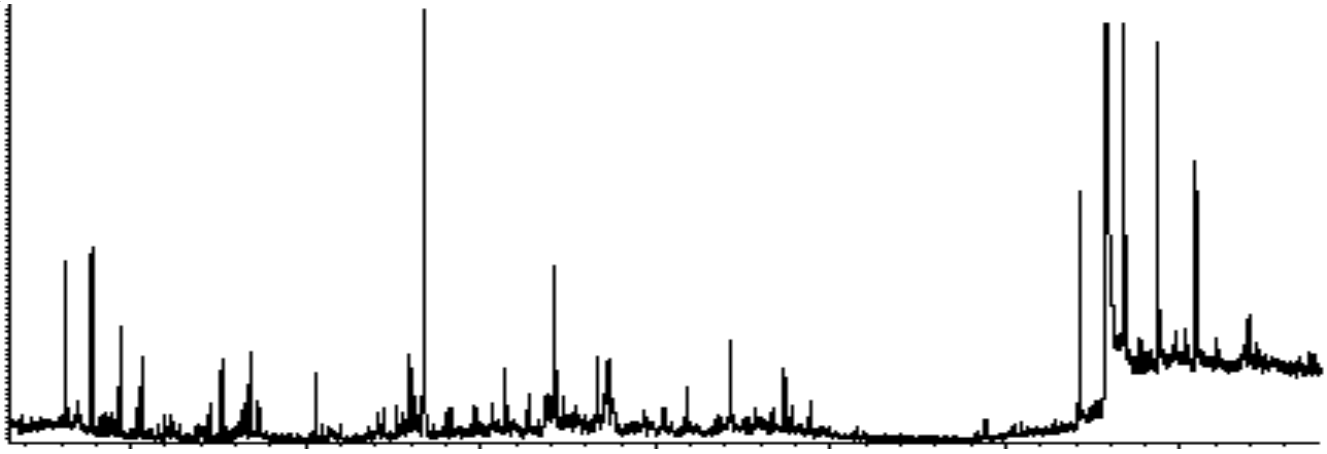

Fig.1. Total ion chromatogram of the volatile oil from *Chrysanthemum morifolium*

TABLE-1
MAIN CHEMICAL COMPONENTS OF VOLATILE OILS FROM *Chrysanthemum morifolium*

No.	Retention time (min)	Name of component	m.f.	Weight (%)
1	6.285	3-Methyl-2-cyclohexen-1-one	$C_7H_{10}O$	2.09
2	7.699	6-Methyl-5-hepten-2-one	$C_8H_{14}O$	2.68
3	7.762	4-Ethyl-1,2-dimethyl-benzene	$C_{10}H_{14}$	0.25
4	9.441	1,2,4,5-Tetramethyl-benzene	$C_{10}H_{14}$	2.05
5	10.425	1,2,3,4-Tetramethyl-benzene	$C_{10}H_{14}$	1.35
6	10.657	1-Ethyl-4-(1-methylethyl)-benzene	$C_{11}H_{16}$	1.56
7	12.049	Undecane	$C_{11}H_{24}$	0.98
8	14.496	2,6-Dimethyl-decane	$C_{12}H_{26}$	0.74
9	15.197	β-Elemene	$C_{15}H_{24}$	1.94
10	20.545	β-Guaiene	$C_{15}H_{24}$	1.70
11	25.877	1,4-Dimethyl-7-[1-methylethyl]-azulen-2-ol	$C_{15}H_{18}O$	2.11
12	26.106	1H-3α, 7-Methanoazulene-8-methanol2,3,4,7,8,8α-hexahydro-3,6,8-trimethyl-,(3R,3αS,7S,8R,8αR)-	$C_{15}H_{24}O$	2.10
13	26.752	2,6-Di-tert-butyl-4-methylphenol	$C_{15}H_{24}O$	14.8
14	28.265	l5,2.2.0[1,6]lUndecan-3-ol, 2-methylene-6,8,8-trimethyl-tricyclo	$C_{15}H_{24}O$	1.11
15	31.411	Santalol	$C_{15}H_{24}O$	1.88
16	32.716	1,4-Methanoazulen-9-ol,decahydro-1,5,5,8α-tetramethyl-(1R,3αR,4S,8αS,9S)-	$C_{15}H_{26}O$	1.28
17	34.762	10α-Endesm-11-en-1α-ol-5α,7αH	$C_{15}H_{26}O$	1.08
18	35.378	3-Methyl-2-butenoic acid 2,7-dimethyloct-7-en-5-yn-4-ylester	$C_{15}H_{22}O_2$	1.24
19	36.659	8s,14-Cedran-diol	$C_{15}H_{26}O_2$	2.31
20	41.740	Hexadecane	$C_{16}H_{34}$	1.37
21	45.727	2,6,10-Trimethyl-tetradecane	$C_{17}H_{36}$	0.54
22	47.346	Eicosane	$C_{20}H_{42}$	1.77
23	48.823	Retinal	$C_{20}H_{28}O$	0.91
24	64.256	Heneicosane	$C_{21}H_{44}$	3.86
25	70.291	3-Ethyl-5-(2-ethylbutyl)-octadecane	$C_{26}H_{54}$	0.44
26	70.874	Heptacosane	$C_{27}H_{56}$	3.95

as shown in Table-1. In addition, several components of volatile oil from *Chrysanthemum morifolium* have the pharmaceutical activity, β-elemene has the effect of resisting tumour[11], retinal may be the mediator between the retina and sclera[12], *etc*. It is significative to analyze the volatile oil from *Chrysanthemum morifolium* which provides a scientific reference for comprehensive study of the chemical composition from *Chrysanthemum morifolium* and promote the efficient development of *Chrysanthemum morifolium*.

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