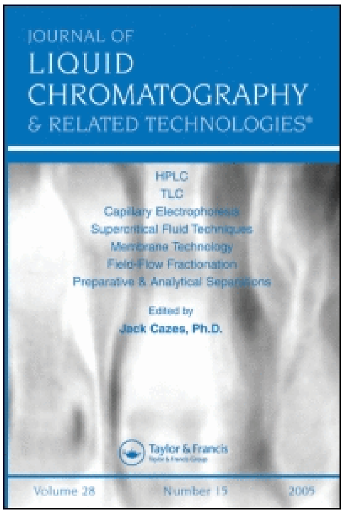

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Guoying Zhang[ab]; Jianya Ling[c]; Zhaojie Cui[a]
[a] School of Environmental Science and Engineering, Shandong University, Jinan, China [b] Shandon University of Traditional Chinese Medicine, Jinan, China [c] State Key Laboratory of Microbial Technology, Shandong University, Jinan, China

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Supercritical CO_2 Extraction of Essential Oil from *Dracocephalum tanguticum* Max-im and Analysis by GC-MS

Guoying Zhang
School of Environmental Science and Engineering, Shandong University, Jinan, China and Shandon University of Traditional Chinese Medicine, Jinan, China

Jianya Ling
State Key Laboratory of Microbial Technology, Shandong University, Jinan, China

Zhaojie Cui
School of Environmental Science and Engineering, Shandong University, Jinan, China

Abstract: The feasibility of active components of *Dracocephalum tanguticum* Max-im (DtM) extracted by supercritical CO_2 was discussed in this paper. The components of the essential oil from the DtM were analyzed by GC-MS. The optimal conditions of supercritical CO_2 extraction: pressure 20 MPa, extraction temperature 50°C, extraction time 75 min, CO_2 flow 1.0 $L \cdot min^{-1}$ were obtained. Seventy-six components from DtM were separated and identified by supercritical CO_2 extraction. There is an abundance of unsaturated fatty acid, saturated fatty acid, acid, alkane, and ester in the essential oil extracted from the DtM. The study established a good method for further studying medical applications and develoment of the DtM.

Keywords: *Dracocephalum tanguticum* Max-im, Essential oil, Supercritical fluid extraction, GC-MS

Address correspondence to Jianya Ling, State Key Laboratory of Microbial Technology, Shandong University, Jinan 250100, P. R. China. E-mail: lingjian-ya@sdu.edu.cn

INTRODUCTION

Dracocephalum tanguticum Max-im is an annual plant of the Labiate family. It is mainly distributed in Tibet, Qinghai, Sichuan and Gansu Province of China. It usually grows in the hillside, dry valley, between about $1900 \sim 4600$ m in height. The flowers and the leaves both blue. The Tibet doctors usually use it with other herbs to cure gastricism, hepatitis, swirl, arthritis, canker, et al. It mainly includes flavone, genin, essential oils, amino acids, and steroids.[1–2]

Supercritical CO_2 extraction of volatile oils from natural products has received great attention in recent years.[3–6] This is mainly due to the surprising dissolving power of the supercritical CO_2 extraction, which can be adjusted by controlling its pressure. Compared to steam distillation or extraction with organic solvents, supercritical CO_2 can avoid the problem of toxic residual solvents in the products, and the deterioration of the thermally labile components in the essential oils. Thus, the organoleptic characteristics of the extracts by supercritical CO_2 extraction can be remained in its original state.

In this paper, supercritical CO_2 was used to extract essential oils from *Dracocephalum tanguticum* Max-im and 76 chemical components of the essential oils were separated and identified by GC-MS.

EXPERIMENTAL

Plant Material

Dracocephalum tanguticum Max-im was collected from Tibet Municipality, China. After air drying in the shade for a week, 8 g of material was ground into a powder.

Supercritical CO_2 Extraction

The extraction was carried out using a supercritical fluid extraction system (Applied Separation Co., America). The supercritical grade carbon dioxide (99.99%) was supplied by Jinan gas factory of Shandong Province of China.

The 8 g sample was added to a 10 mL extraction cell. Then, the cell was placed in the oven. The extraction process was optimized at a pressure of 20 MPa, temperature 50°C; 15 min of static extraction was followed by 60 min of dynamic extraction, supercritical CO_2 at a flow rate of $1.0\,L \cdot min^{-1}$ in the dynamic extraction procedure. The extract was collected in chloroform and stored in a refrigerator before GC-MS analysis.

GC–MS Analysis

The analysis was performed using a Shimadzu 2010 Series Chromatograph equipped with a DB-1 capillary column (30 m × 0.25 mm, film thickness 0.25 μm) and a mass spectrometer selective detector. Analytical conditions were: oven temperature programmed from 50°C (hold for 2 min) to 250°C at a rate of $5°C \cdot min^{-1}$; injector temperature 270°C; interface temperature 270°; sample injection 0.1 μL, split ratio 30:1; helium carrier gas $1.1\ mL \cdot min^{-1}$; ionization source 70 eV; mass range 30–400 amu.

RESULTS AND DISCUSSION

The essential oil extracted from *Dracocephalum tanguticum* Max-im by supercritical CO_2 was a light green liquid in a yield of 2.3% (w/w). Figure 1 is the total iron chromatography and Table 1 shows the chemical components and content of the essential oil. Seventy-four components were characterized, representing 97.4% of the total essential oil.

The results of this study indicated that there are variable ingredients in *Dracocephalum tanguticum* Max-im. The main components are gamma-linolenic acid (11.06%), tetracosane (8.19%), hexatriacontane (10.95%), tetracontane (9.89%), tetratriacontane (5.99%). Among the components, ackanes account for 50% of the total identified essential oil.

Some other components, in a relatively high concentration, are germacrone (3.67%), linolenic acid, methyl ester (3.4%), tau-cadinol (2.92%), cedrene (2.13%), palmitic acid (2.00%), perilla alcohol (1.3%), isomenthol (1.19%), farnesol (1.14%), beta-elemenone (1.12%), linalyl acetate (1.08%), and palmitone (1.01%).

Components in relatively low amounts occupy 11.60% of the total essential oil.

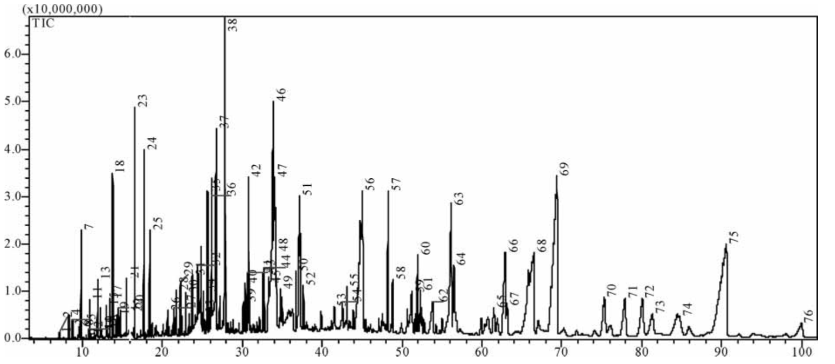

Figure 1. Total ion current chromatogram of the essential oils from *Dracocephalum tanguticum* Max-im by Supercritical CO_2.

Table 1. Analytical results of chemical constituents of essential oils from *Dracocephalum tanguticum* max-im by supercritical CO_2

No.	Compound	Molecular formula	Molecular weight	Relative content (%)
1	alpha-Thujene	$C_{10}H_{16}$	136	0.03
2	alpha-Pinene	$C_{10}H_{16}$	136	0.04
3	Sabinene	$C_{10}H_{16}$	136	0.07
4	beta-Pinene	$C_{10}H_{16}$	136	0.12
5	beta-Myrcene	$C_{10}H_{16}$	136	0.09
6	beta-Cymene	$C_{10}H_{14}$	134	0.06
7	Eucalyptol	$C_{10}H_{18}O$	154	0.55
8	D-Limonene	$C_{10}H_{16}$	136	0.02
9	Ocimene	$C_{10}H_{16}$	136	0.02
10	gamma-Terpinene	$C_{10}H_{16}$	136	0.04
11	Z-beta-Terpineol	$C_{10}H_{18}O$	154	0.19
12	cis-Linalool	$C_{10}H_{18}O_2$	170	0.02
13	Eucarvone	$C_{10}H_{14}O$	150	0.55
14	E-Pinane	$C_{10}H_{18}$	138	0.05
15	trans-Pinocarveol	$C_{10}H_{16}O$	152	0.18
16	cis-Verbenol	$C_{10}H_{16}O$	152	0.04
17	alpha-Linalool	$C_{10}H_{18}O_2$	170	0.22
18	Isopinocamphone	$C_{10}H_{16}O$	152	1.19
19	Myrtenal	$C_{10}H_{14}O$	150	0.09
20	alpha-Terpineol	$C_{10}H_{18}O$	154	0.15
21	Isopulegol acetate	$C_{12}H_{20}O_2$	196	0.28
22	(+)-Pinanediol	$C_{10}H_{18}O_2$	170	0.09
23	Linalyl acetate	$C_{12}H_{20}O_2$	196	1.08
24	(−)-trans-Pinocarvyl acetate	$C_{12}H_{18}O_2$	194	0.83
25	Myrtenyl acetate	$C_{12}H_{18}O_2$	194	0.58
26	delta-Guaiene	$C_{15}H_{24}$	204	0.12
27	Caryophyllene	$C_{15}H_{24}$	204	0.14
28	gamma-Elemene	$C_{15}H_{24}$	204	0.36
29	alpha-Caryophyllene	$C_{15}H_{24}$	204	0.29
30	alpha-Cubebene	$C_{15}H_{24}$	204	0.25
31	Isoledene	$C_{15}H_{24}$	204	0.28
32	Elemol	$C_{15}H_{26}O$	222	0.26
33	Ledol	$C_{15}H_{26}O$	222	0.38
34	Globulol	$C_{15}H_{26}O$	222	0.24
35	beta-Elemenone	$C_{15}H_{22}O$	218	1.12
36	Cedrene	$C_{15}H_{24}$	204	2.13
37	tau-Cadinol	$C_{15}H_{26}O$	222	2.92
38	Germacrone	$C_{15}H_{22}O$	218	3.67
39	cis-Z-alpha-Bisabolene epoxide	$C_{15}H_{24}O$	220	0.18
40	Caryophyllene oxide	$C_{15}H_{24}O$	220	0.33
41	(+)-2-Isopropenyl-2-Carene	$C_{13}H_{20}$	176	0.30

(*continued*)

Table 1. Continued

No.	Compound	Molecular formula	Molecular weight	Relative content (%)
42	Perilla alcohol	$C_{10}H_{16}O$	152	1.30
43	alpha-Limonene diepoxide	$C_{10}H_{16}O_2$	168	0.37
44	(+)-Isomenthol	$C_{10}H_{20}O$	156	0.58
45	Isomenthol	$C_{10}H_{20}O$	156	1.19
46	Palmitic acid	$C_{16}H_{32}O_2$	256	2.00
47	gamma-Linolenic acid	$C_{20}H_{34}O_2$	306	11.06
48	trans-Oleic acid	$C_{18}H_{34}O_2$	282	0.34
49	Eicosanoic acid	$C_{20}H_{40}O_2$	312	0.39
50	Phytol	$C_{20}H_{40}O$	296	0.40
51	Linolenic acid, methyl ester	$C_{19}H_{32}O_2$	292	3.40
52	Octadecanoic acid	$C_{18}H_{36}O_2$	284	0.26
53	Heneicosane	$C_{21}H_{44}$	296	0.35
54	Vitamin E	$C_{29}H_{50}O_2$	430	0.28
55	Docosane	$C_{22}H_{46}$	310	0.44
56	Tetracosane	$C_{24}H_{50}$	338	8.19
57	Pentacosane	$C_{25}H_{52}$	352	1.92
58	Hexacosane	$C_{26}H_{54}$	366	0.47
59	Heptacosane	$C_{27}H_{56}$	380	0.92
60	Farnesol	$C_{15}H_{26}O$	222	1.14
61	Octadecanal	$C_{18}H_{36}O$	268	1.03
62	Octacosane	$C_{28}H_{58}$	394	1.13
63	Nonacosane	$C_{29}H_{60}$	408	3.67
64	Triacontane	$C_{30}H_{62}$	422	1.69
65	1-Triacontanol	$C_{30}H_{62}O$	438	0.61
66	Hentriacontane	$C_{31}H_{64}$	436	2.90
67	Dotriacontane	$C_{32}H_{66}$	450	0.67
68	Tetratriacontane	$C_{34}H_{70}$	478	5.99
69	Hexatriacontane	$C_{36}H_{74}$	506	10.95
70	Un			1.40
71	Un			1.29
72	1,30-Triacontanediol	$C_{30}H_{62}O_2$	454	1.48
73	gamma-Sitosterol	$C_{29}H_{50}O$	414	0.93
74	11-Decyl-tetracosane	$C_{34}H_{70}$	478	0.81
75	Tetracontane	$C_{40}H_{82}$	562	9.89
76	Palmitone	$C_{31}H_{62}O$	450	1.01

Un: unidentified.

There are no articles on supercritical fluid extraction of essential oil from *Dracocephalum tanguticum* Max-im till now. The analytical results suggest that Tibet doctors make good use of this rare traditional Chinese medicine. This study also established a good method for further studying medical applications and development of the DtM.

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