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SUPERCRITICAL CO_2 EXTRACTION AND GC-MS ANALYSIS OF THE VOLATILE COMPONENTS FROM *Gastrodia elata*

Yan-Ni Zhang,[1,2] Xuan-Feng Yue,[1] and Zhe-Zhi Wang[1,2*]

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The steamed and dried tubers of *Gastrodia elata* Blume, which are called “Tianma” in China, have been officially listed in the Chinese Pharmacopoeia for a long time. Its tubers have been widely used for the treatment of rheumatism, neuralgia, epilepsy, paralysis, hemiplegia, lumbago, headache, and vertigo [1–3]. Many companies in China are promoting large-scale cultivation of *Gastrodia elata* Blume with clinical interest in mind [4]. Concomitant with the imminent large-scale cultivation and commercial exploitation of *Gastrodia elata* Blume, quality control during processing and storage is critical.

In recent years supercritical fluid extraction (SFE) has become an alternative to more conventional extraction procedures. With carbon dioxide, the most commonly used supercritical fluid, supercritical conditions are readily attained (critical temperature 31.05°C, critical pressure 72.9 atm; 1 atm = 101, 325 Pa). Carbon dioxide also has the additional advantages of being nonflammable, fairly nontoxic, cost-effective, and easily removed from the extract following decompression. The combined liquid-like solvating capabilities and gaslike transport properties of supercritical fluids make them particularly suitable for the extraction of diffusion-controlled matrices such as plant tissues [5–7].

The aim of this study is to explore the information on the volatile composition of the extract in *Gastrodia elata*.

Preliminary supercritical fluid extraction experiments were carried out to establish the best extraction procedures to give accurate and consistent results. At pressure of 27.5 MPa and temperature of 30°C for a static duration of 30 min, the extraction yield of the components was 0.20 (w/w).

Qualitative and Quantitative Analyses. The unambiguous identification of most of the compounds was done by comparing their fragmentation pattern in EI mass spectra with those in mass spectral databases (NIST05 and NIST05s); as for those without adequate assurance, identification was based on joint information from the EI and CI mass spectra. Some of them were further confirmed by comparing their fragmentation pattern in EI mass spectra with those of authentic compounds available in our laboratory or from literature data [8, 9]. Relative amounts (percent) of individual components were calculated based on GC peak areas without response factor correction.

Forty components, amounting to 96.23% of the volatile components, were identified. The results are listed in Table 1, where the compounds are arranged in the order of elution on the RTX-5MS silica capillary column. The compounds identified comprise organic acids (68.99%), alcohols (11.32%), esters (5.60%), aldehydes (5.39%), alkanes (3.26%), amines (1.39%), and ketones (0.28%). The fatty acids are found to be the main components (68.99%), in which octadecadienoic acid (32.28%) and hexadecanoic acid (30.08%) are dominant, and the acids also include two odd carbon long-chain fatty acids, pentadecanoic acid (0.81%) and heptadecanoic acid (0.77%).

Plant Material. *Gastrodia elata* Blume was collected in Nov. 2007 from Yunnan, China. The samples were dried in a shady place, and a voucher specimen was deposited in the School of Life Sciences, Shaanxi Normal University, Shaanxi, China. The dried sample was ground to a powder of a certain size.

Supercritical Fluid Extraction (SFE). Model SFT-100XW system (Supercritical Fluid Technologies, INC) was used for all the extractions. Supercritical fluid extractions were conducted at pressure of 27.5 MPa and temperature of 30°C for a static duration of 30 min after the variables were optimized by preliminary investigation. The supercritical CO_2 flow rate was approximately 1.0 mL/min (compressed).

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1) Key Laboratory of the Ministry of Education for Medicinal Resources and Natural Pharmaceutical Chemistry, Xi′an, shaaxi, 710062, P. R. China, fax: 86 29 85310282, e-mail: zzwang@snnu.edu.cn; 2) School of Life Sciences, Shaanxi Normal University; Xi′an 710062, China. Published in Khimiya Prirodnykh Soedinenii, No. 6, pp. 810–811, November–December, 2010. Original article submitted June 18, 2009.

TABLE 1. Compounds Identified in the Volatile Components from *Gastrodia elata* by SFE

Compound	T, min	Relative cont., %	Compound	T, min	Relative cont., %
Benzaldehyde, 4-hydroxy-	8.036	0.15	Tetradecanoic acid	12.725	0.53
Tridecane	8.428	0.13	Pentadecanoic acid	14.041	0.81
Tetradecane	9.213	0.11	Hexadecanoic acid, methyl ester	15.040	0.45
Pentadecanal	10.415	0.25	Cyclopentadecanone, 2-hydroxy-	15.314	0.76
Hexadecane	10.868	0.19	Hexadecanoic acid	15.675	30.08
Cyclohexyl-3-ethoxybutan-2-one	11.300	0.28	Phthalic acid, butyl undecyl ester	15.834	0.19
Benzenamine, *N*-phenyl-	11.380	1.39	Isopropyl palmitate	16.721	0.17
Heptadecane	11.597	0.36	Heptadecanoic acid	17.445	0.77
1-Hexacosanol	11.703	0.22	Tridecanal	18.070	0.20
Heneicosane	11.788	0.49	9,12-Octadecadienoic acid, methyl ester	18.196	1.76
Hexanedioic acid, bis(2-methylpropyl) ester	11.885	0.15	Octadecadienoic acid	19.123	32.28
Octadecane, 1-chloro-	12.908	0.14	9-Hexadecenal	19.186	5.04
2-Isopropyl-5-methyl-1-heptanol	12.041	0.12	6,9-Pentadecadien-1-ol	19.253	10.25
Hexadecane, 2,6,10,14-tetramethyl-	12.123	0.31	9-Octadecenoic acid, methyl ester, (*E*)-	19.370	1.57
Octadecane, 1-(ethenyloxy)-	12.207	0.14	Octadecanoic acid	19.629	4.16
Dodecanoic acid	12.289	0.15	8,10-Hexadecadien-1-ol	19.815	0.55
Propylphosphonic acid, fluoroanhydride, 4-methylcyclohexyl ester	12.347	0.36	1,2-Benzenedicarboxylic acid, bis(2-methyl propyl) ester	20.414	0.56
2-Naphthalenemethanol	12.403	0.18	11-Eicosenoic acid, methyl ester	21.794	0.27
Pentadecane, 2,6,10,14-tetramethyl-	12.516	0.17	Erucic acid	22.967	0.21
Oxalic acid, isobutyl undecyl ester	12.646	0.12	Triacontane, 11,20-didecyl-	23.155	0.21

GC/MS Analysis. The analysis of the extract was performed on GC/MS (Shimadzu GC/MS-QP2010, Japan). The components were separated on an RTX-5MS fused-silica capillary column (30 m length, 0.25 mm diameter, 0.25 m film thickness) coated with 5% diphenyl and 95% dimethylpolysiloxane.

GC/MS (EI) Conditions. Helium was used as the carrier gas (1.2 mL/min). A 2 μL sample was injected into the column with the split ratio set at 50:1 and injector port temperature at 250°C. The GC program was initiated by a column temperature set at 80°C and a temperature increase to 200°C and 220°C at a rate of 10°C/min and 2°C/min, respectively, which was then kept constant at 220°C for 2 min. The temperatures of the ion source and interface were 200°C and 250°C, respectively. The mass spectrometer was operated in full scan mode with mass range ranging from *m/z* 10 to 800.

GC/MS (CI) Conditions. The PCI and NCI mass spectra were recorded on the same apparatus equipped with the same column and corresponding ionization chemical source. Ionizing gas: methane (CH_4); other experimental conditions were the same as those in the EI analysis.

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REFERENCES

1. H. J. Kim, K. D. Moon, D. S. Lee, and S. H. Lee, *J. Ethnopharmacol.*, **84**, 95 (2003).
2. H. Z. Zheng, Z. H. Dong, and J. She, *Modern Study of Traditional Chinese Medicine*, Xue Yuan Press: Beijing, China, 1997.
3. C. L. Hsieha, C. H. Changa, S. Y. Chiang, T. C. Li, N. Y. Tanga, C. Z. Pona, C. T. Hsiehb, and J. G. Lina, *Life Sci.*, **67**, 1185 (2000).

4. S. L. Yang, J. Lan, and J. T. Xu, *Chin. Trad. Herb. Drugs*, **31**, 66 (2000).
5. M. Khajeh, Y. Yamini, N. Bahramifar, F. Sefidkon, and M. Reza Pirmoradei, *Food Chem.*, **91**, 639 (2005).
6. W. Q. Guan, S. F. Li, R. X. Yan, S. K. Tang, and C. Quan, *Food Chem.*, **101**, 1558 (2007).
7. Y. Yamini, M. Khajeh, E. Ghasemi, M. Mirza, and K. Javidnia, *Food Chem.*, **108**, 341 (2008).
8. Y. Masada, *Analysis of Essential Oils by Gas Chromatography and Mass Spectrometry,* John Wiley and Sons. Inc., New York, 1976.
9. R. P. Adams, *Identification of Essential Oil Components by Gas Chromatography/ Quadrupole Mass Spectroscopy,* Allured Publishing Co., Illinois, 1995.