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Analysis of Volatile Constituents from *Abelmoschus manihot* by GC-MS

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The volatile constituents from Flos *Abelmoschus manihot* were extracted from Flos *Abelmoschus manihot* by steam distillation and analyzed by GC-MS. A total of 47 compounds were successfully identified. The major components of the volatile constituents in Flos *Abelmoschus manihot* included hexadecane, 2,6,10,14-tetramethyl-hexadecane (37.83 %), pentacosane (13.49 %), 9-octyl-heptadecane (9.78 %), cedrol (3.07 %), 9,12,15-octadecatrienoic acid, methyl ester, (Z,Z,Z)- (2.79 %), tetratetracontane (2.78 %), cyclotetracosane (2.61 %) and hexadecanoic acid, methyl ester (2.32 %). The major components in volatile constituents were hydroxy compounds, acyclic alkanes, other arenes and sesquiterpenes.

Keywords: GC-MS, Flos *Abelmoschus manihot*, Volatile constituents, Acyclic alkanes, Hydroxy compounds.

INTRODUCTION

Flos *Abelmoschus manihot*, belonging to *Abelmoschus medicus*, the Malvaceae family, is an annual or perennial herb in China, which is widely distributed in Henan, Shandong, Sichuan, Fujian, Shaanxi, Anhui, Jiangsu, Guangxi, Yunnan and Jiangxi provinces in China. It has been taken orally for the treatment of strangurias and oedema as well as ulcer and scald caused by water and fire for external use. Researches showed that Flos *Abelmoschus manihot* exhibits notable therapeutic effect on renal disease syndrome, arthritis, cardiovascular disease, *etc.*[1]. In view of its prospects of pharmacology, scholars pay more and more attention to the active ingredients of Flos *Abelmoschus manihot*[2].

Flos *Abelmoschus manihot* contains various chemical ingredients including flavonoids, organic acids, steroids, volatile constituents, coumarins, long-chain aliphatic hydrocarbons and nitrogenous compounds. Most reports on the chemical constituents of Flos *Abelmoschus manihot* focused on flavonoids, whereas few studies have reported on volatile constituents. In our study, we conducted a qualitative analysis of volatile constituents of Flos *Abelmoschus manihot* produced in Anhui using GC-MS method[3,4]. We matched them with the NIST08 mass spectrometry database. The present work provides helpful information to further development and utilization of Flos *Abelmoschus manihot*.

EXPERIMENTAL

The dry flowers of *Abelmoschus manihot*, produced in Anhui, China, were bought from Huarong Tang Pharmaceutical Co., Ltd., Bozhou City, Anhui, in April, 2014. The materials were authenticated as *Abelmoschus manihot* (L.) Medic. by Prof. Liu Chun-Sheng, Department of Health and medicine, School of Pharmacy, Beijing University of Chinese Medicine.

Agilent 7890-5975 GC-MS (USA) and other instruments including round-bottomed flask, volatile oil extractor and electric heater were used for the experiments. The used chemical was acetone (Beijing Gao Hua Albert Chemicals Co., Ltd., China).

Extraction of volatile constituents: The flowers of *Abelmoschus manihot* (500 g) were pulverized and then placed in a steam distillation vessel and then connected volatile oil extractor to a reflux condenser. The sample was soaked for 1.5 h and extracted with water (5 L) for two times, 11 h each. Finally the volatile oil (0.6 mL) was obtained. The sample was dissolved in acetone prior to analysis. The yield was 0.11 % (w/w) for Flos *Abelmoschus manihot*.

Chromatographic conditions for GC-MS analysis: The GC conditions were as follows: HP-5MS 5 % phenyl methyl siloxane fused-silica capillary column (30 m × 0.25 mm, 0.25 μm). Samples (2 μL) were injected in splitless mode at 290 °C. The carrier gas was helium in constant-flow mode (1.0 mL/min). The temperature programming started at 95 °C for 1.5 min, increased to 190 °C for 1 min at a rate of 20 °C/min, then increased to 230 °C for 1 min at a rate of 5 °C/min and finally reached 290 °C for 20 min at a rate of 25 °C/min. Column pressure was 71 kPa.

The Mass Spectrometer conditions for the experiment were as follows: Electron-impact (EI) mode at 70 eV using single ion monitoring. The ion source temperature was 230 °C.

Mass scan range was from 100 to 800 amu and solvent delay was 5 min. NIST08 mass spectrometry database was used and compared with the standard spectrum to identify the components of every peak.

RESULTS AND DISCUSSION

Chemical identification of Flos *Abelmoschus manihot* was accomplished by GC-MS analysis. The total ion chromatogram of volatile constituents was obtained and shown in Fig. 1. Table-1 listed the compounds including the relative content, retention time, mean relative molecular weight (m.w.) and formula identified by GC-MS.

A total of 47 compounds were successfully identified. Table-1 showed that the main components of volatile constituents from Flos *Abelmoschus manihot* were hydroxy compounds, acyclic alkanes, other arenes and sesquiterpenes. After area normalization, the eight components with the highest relative content fraction (> 2 %) were identified as hexadecane, 2,6,10,14-tetramethyl-hexadecane (37.83 %), pentacosane (13.49 %), 9-octyl-heptadecane (9.78 %), cedrol (3.07 %), 9,12,15-octadecatrienoic acid,methyl ester, (Z,Z,Z)- (2.79 %), tetratetracontane (2.78 %), cyclotetracosane (2.61 %) and hexadecanoic acid, methyl ester (2.32 %)[5-10].

TABLE-1
VOLATILE CONSTITUENTS FROM Flos *Abelmoschus manihot*

No.	Compounds	m.w.	m.f.	Time (min)	Area (%)
1	3-Cyclohexene-1-methanol, α,α-4-trimethyl-	154	$C_{10}H_{18}O$	10.08	0.37
2	2,4-Decadienal, (E,E)-	152	$C_{10}H_{16}O$	12.20	0.25
3	1-(4-*tert*-Butylphenyl)propan-2-one	190	$C_{13}H_{18}O$	14.05	0.20
4	5,5,8a-Trimethyl-3,5,6,7,8,8a-hexahydro-2H-chromene	180	$C_{12}H_{20}O$	15.45	0.38
5	Nerolidol 2	222	$C_{15}H_{26}O$	16.08	0.21
6	Cedrol	222	$C_{15}H_{26}O$	16.99	3.07
7	Benzene, 1-methyl-4-(1,2,2-trimethylcyclopentyl)-, (R)-	202	$C_{15}H_{22}$	17.47	0.79
8	Naphthalene, 1,2,3-trimethyl-4-propenyl-, (E)-	210	$C_{16}H_{18}$	17.81	0.38
9	(4-Acetylphenyl)phenylmethane	210	$C_{15}H_{14}O$	17.96	0.26
10	2-Pentadecanone	226	$C_{15}H_{30}O$	18.04	0.45
11	2,4,6-Trimethoxyacetophenone	210	$C_{11}H_{14}O_4$	18.24	0.19
12	Phenanthrene	178	$C_{14}H_{10}$	19.40	1.01
13	3-Octadecyne	250	$C_{18}H_{34}$	19.91	0.44
14	2-Pentadecanone, 6,10,14-trimethyl-	268	$C_{18}H_{36}O$	19.97	0.67
15	Phthalic acid, isobutyl octyl ester	334	$C_{20}H_{30}O_4$	20.22	0.85
16	Oxirane, tridecyl-	226	$C_{15}H_{30}O$	20.47	0.30
17	2-Heptadecanone	254	$C_{17}H_{34}O$	20.74	0.52
18	5,9,13-Pentadecatrien-2-one, 6,10,14-trimethyl-, (E,E)-	262	$C_{18}H_{30}O$	20.88	0.53
19	Hexadecanoic acid, methyl ester	270	$C_{17}H_{34}O_2$	21.03	2.32
20	4H-Cyclopenta[def]phenanthrene	190	$C_{15}H_{10}$	21.16	0.31
21	Dibutyl phthalate	278	$C_{16}H_{22}O_4$	21.43	0.54
22	*n*-Hexadecanoic acid	256	$C_{16}H_{32}O_2$	21.49	0.46
23	Nerolidol 1	222	$C_{15}H_{26}O$	22.27	0.39
24	1-Naphthalenepropanol, α-ethenyldecahydro-α,5,5,8a-tetramethyl-2-methylene-, [1S-[1α(S*),4aβ,8aα]]-	290	$C_{20}H_{34}O$	22.79	1.14
25	Fluoranthene	202	$C_{16}H_{10}$	22.95	1.24
26	9,12-Octadecadienoic acid (Z,Z)-, methyl ester	294	$C_{19}H_{34}O_2$	23.07	1.07
27	9,12,15-Octadecatrienoic acid, methyl ester, (Z,Z,Z)-	292	$C_{19}H_{32}O_2$	23.14	2.79
28	Valeric acid, undec-2-enyl ester	254	$C_{16}H_{30}O_2$	23.21	0.95
29	Phytol	296	$C_{20}H_{40}O$	23.32	0.49
30	1-Penten-3-one, 1-(2,6,6-trimethyl-1-cyclohexen-1-yl)-	206	$C_{14}H_{22}O$	23.37	0.57
31	Pyrene	202	$C_{16}H_{10}$	23.58	1.51
32	*cis*-9-Tetradecen-1-ol	212	$C_{14}H_{28}O$	23.77	0.23
33	Ethyl 9,12,15-octadecatrienoate	306	$C_{20}H_{34}O_2$	23.90	0.30
34	Heptadecane	240	$C_{17}H_{36}$	24.29	0.56
35	Octadecane	254	$C_{18}H_{38}$	25.37	1.01
36	4,8,12,16-Tetramethylheptadecan-4-olide	324	$C_{21}H_{40}O_2$	25.89	0.19
37	Tetracosane	338	$C_{24}H_{50}$	26.42	1.17
38	Pentacosane	352	$C_{25}H_{52}$	27.44	13.49
39	Tetratetracontane	618	$C_{44}H_{90}$	28.38	2.78
40	Behenic alcohol	326	$C_{22}H_{46}O$	29.10	0.35
41	Nonadecyl trifluoroacetate	380	$C_{21}H_{39}O_2F_3$	29.18	1.11
42	Hexadecane, 2,6,10,14-tetramethyl-	282	$C_{20}H_{42}$	29.40	37.83
43	Octacosane	394	$C_{28}H_{58}$	30.42	1.95
44	Cyclotetracosane	336	$C_{24}H_{48}$	31.50	2.61
45	Heptadecane, 9-octyl-	352	$C_{25}H_{52}$	31.72	9.78
46	Eicosane	282	$C_{20}H_{42}$	33.24	0.83
47	Tetratriacontane	478	$C_{34}H_{70}$	35.12	1.17

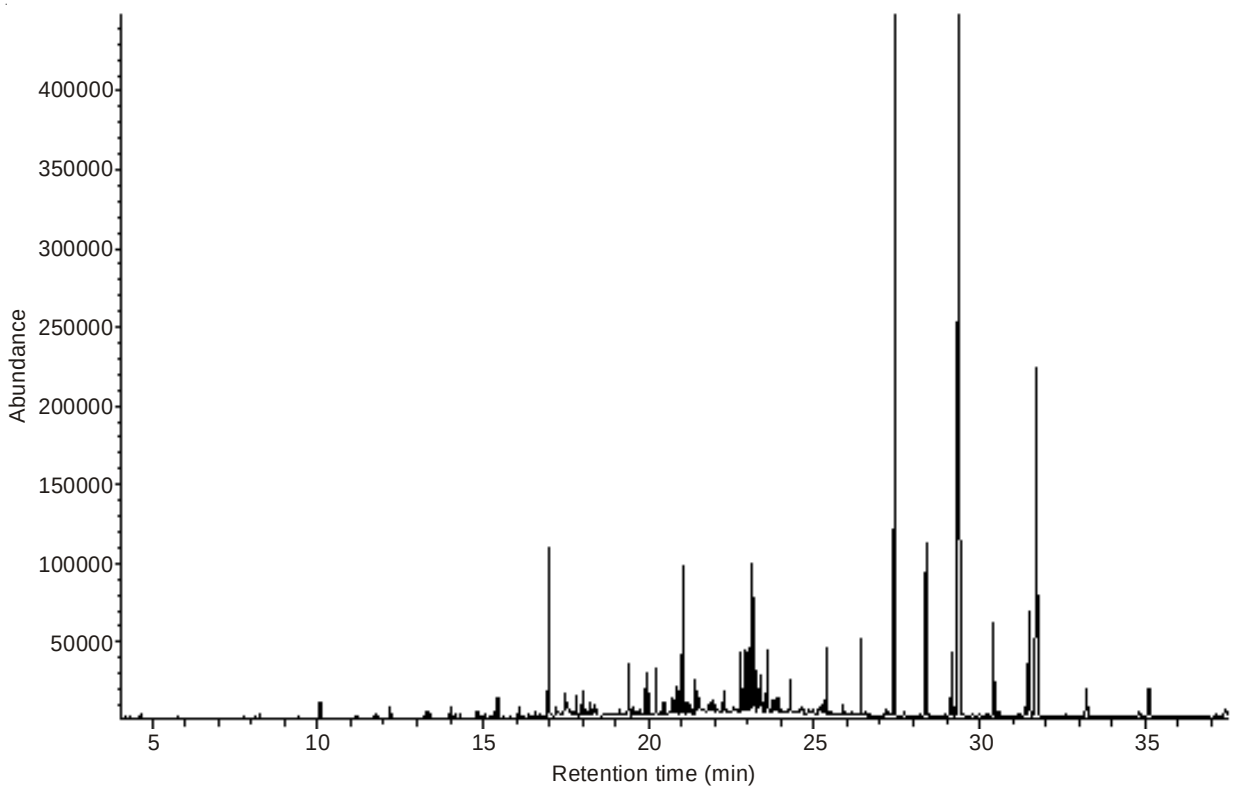

Fig. 1. Total ion chromatogram of the volatile constituents from Flos *Abelmoschus manihot* by GC-MS

Conclusion

In this study, we conducted a qualitative analysis of volatile constituents from Flos *Abelmoschus manihot* using GC-MS method, which would contribute to provide reference for the development and utilization of Flos *Abelmoschus manihot*. The structures of these compounds were diverse, most of which were acyclic alkanes, arenes, sesquiterpenes and hydroxy compounds. Flos *Abelmoschus manihot* was riched in hexadecane, 2,6,10,14-tetramethyl-hexadecane (29.40 min), which has antioxidant and antibacterial effects and can be used for the treatment of skin disease such as scald caused by water and fire, seborrheic alopecia and so on. This study laid the foundation for the research on pharmacology and new topical preparations of Flos *Abelmoschus manihot*.

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REFERENCES

1. H. Liu, W. Zhang and H. Chen, *J. Medical Colleges of PLA*, **27**, 334 (2012).
2. C. Njume, A.J. Afolayan, E. Green and R.N. Ndip, *Int. J. Antimicrob. Agents*, **38**, 319 (2011).
3. M.Z.M. Salem, H.M. Ali, N.A. El-Shanhorey and A. Abdel-Megeed, *Asian Pacif. J. Trop. Med.*, **6**, 785 (2013).
4. J.Z. Al-Kalaldeh, R. Abu-Dahab and F.U. Afifi, *Nutr. Res.*, **30**, 271 (2010).
5. H. Lin-fang, W. Zeng-hui and C. Shi-lin, *Chinese Herbal Medicines*, **5**, 73 (2013).
6. M.A. Hossain, M.D. Shah and M. Sakari, *Asian Pacif. J. Trop. Med.*, **4**, 637 (2011).
7. H.Y. Gong, W.H. Liu, G.Y. Lv and X. Zhou, *Rev. Bras. Farmacogn.*, **24**, 25 (2014).
8. Y. Wang, L. Chang, X. Zhao, X. Meng and Y. Liu, *J. Tradit. Chin. Med.*, **32**, 459 (2012).
9. L. Jun, T. Yu-zeng, S. Bao-ya, et al., *Chinese Herbal Medicines*, **4**, 63 (2011).
10. V. Soumya, Y.I. Muzib, P. Venkatesh and K. Hariprasath, *Chin. J. Nat. Med.*, **12**, 677 (2014).