GC-MS Analysis on Volatile Oil from Branches of *Picrasma quassioides*

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Abstract: **Objective** To analyze the volatile oil from the branches of *Picrasma quassioides*. **Methods** Volatile oil was extracted from the branches of *P. quassioides* by steam distillation. GC-MS method was used to analyze the components. **Results** A total of 49 compounds were isolated, and 46 compounds were successfully identified, which represented over 98% of the total oil composition. The major components of the volatile oil in the branches of *P. quassioides* included caryophyllene (12.83%), 12-oxabicyclo[9.1.0]dodeca-3,7-diene,1,5,5,8-tetramethyl-[1R-(1*R**,3*E*,7*E*,11-*R**)] (12.29%), 1-hexanol (9.96%), naphthalene, 1,2,3,5,6,7,8,8a-octahydro-1,8a-dimethyl-7-(1-methyletheyl)-[1*S*-(1a,7a,8aa)] (7.32%), aromadendrene oxide-(2) (6.69%), and α-caryophyllene (3.88%). **Conclusion** The major components in volatile oil are terpenoids, hydroxy compounds, and other acyclic alkane compounds.

Key words: GC-MS; hydroxy compounds; *Picrasma quassioides*; terpenoids; volatile oil

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Introduction

Picrasma quassioides (D. Don) Benn., belonging to the Simaroubaceae family, is a traditional medicinal plant in China (Pharmacopeia Committee of P. R. China, 2010). *P. quassioides* is distributed in the provinces of Hebei, Shanxi, Henan, Shandong, Jiangsu, Jiangxi, Hunan, Hubei, Shaanxi, Gansu, Sichuan, Yunnan, Guangdong, and Guangxi, China. The deciduous shrub or the small tree has good adaptability, and often grows on the hillside, valley, forest edge, and along the sinuous mountain stream, or other places with moist fertile soil (Committee of Chinese Herbacology, 2004).

The branches and leaves of *P. quassioides* could be used in Chinese materia medica (CMM) for the treatment of cold, sore throat, dysentery, eczema, scabies, hypertension, child pneumonia, and bites caused by insects or snakes (Pharmacopeia Committee of P. R. China, 2010). Researches have shown that *P. quassioides* exhibits antitumor, blood flow-promoting, and antiviral effects (Zhao *et al*, 2011; Gu, Wang, and Wang, 2011). *P. quassioides* contains various chemical ingredients including terpenes, triterpenes, volatile oil, alkaloids, sterols, saponins, and coumarins (Committee of Chinese Herbacology, 2004; Chen *et al*, 2007). Most reports on the chemical constituents of *P. quassioides* focused on alkaloids and quassinoids, whereas few studies have reported on volatile oil. There is only one report on the volatile oil of *P. quassioides* growing in the south of Guizhou province (Yang *et al*, 2011). We investigate the volatile oil in *P. quassioides* from Beijing using GC-MS method, with the aim to illustrate the difference of the volatile oil in *P. quassioides* from two habitats. The present work provides the helpful information to further study chemical-ecological type of components in *P. quassioides.*

Materials and methods

Plant materials

The fresh branches of *Picrasma quassioides* (D. Don) Benn. were collected from Beijing on August 11,

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2011, and dried at room temperature. The materials were identified by Prof. LIN Yu-lin and HUANG Lin-fang in Institute of Medicinal Plant Development (IMPLAD). A voucher specimen (BJ2011-01) has been deposited in the herbarium of IMPLAD, China.

Instruments and reagents

Varian 430GC—300MS (USA) and other instruments including conical flasks, volatile oil extractor, electric heater, injector, and 0.45 μm microporous membrane filter were used for the experiments. The chemicals used include ethyl ether (Beijing Chemical Works, China), NaCl, and anhydrous sodium sulfate (Xilong Chemical Industry, Shantou, China).

Extraction of volatile oil

The plant material was pulverized, passed through 24-mesh sieve, and then placed in a steam distillation vessel. The sample was soaked for 4 h and subjected to hydro-distillation using Clevenger-type apparatus for 8 h. NaCl was added until saturation was reached. Diethyl ether was then used as the solvent to extract the oil for three times. The extraction was enriched to 2 mL. The sample was dried by anhydrous Na_2SO_4, filtered by a microporous membrane, stored in a sealed container, and refrigerated prior to analysis.

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), injector temperature at 250 ℃, helium as the carrier gas, column pressure of 72 kPa, injection volume (*n*-hexane solution) of 2 μL, and flow rate of 1.2 mL/min. The temperature programming started at 50 ℃ for 2 min, increased to 120 ℃ for 2 min at a rate of 4 ℃/min, then increased to 220 ℃ for 5 min at a rate of 2 ℃/min, and finally reached 250 ℃ for 5 min at a rate of 5 ℃/min.

The MS conditions for the experiment were as follows: EI, ion source temperature at 23 ℃, electron energy of 70 eV, interface temperature at 270 ℃, solvent delay for 2 min, and mass scan range of 30—500 amu. NIST mass spectral database was used and compared with the standard spectrum to identify the components of every peak.

Results and discussion

The total ion chromatogram of volatile oil components was obtained (Fig. 1). Table 1 listed the compounds including the relative content, retention time, mean relative molecular weight (MW), and formula indentified by GC-MS.

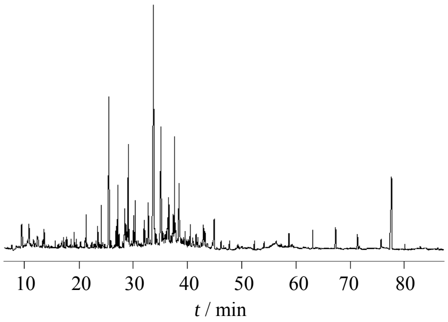

Fig. 1 GC-MS total ion chromatogram of volatile oil in branches of *P. quassioides*

A total of 46 compounds were successfully identified, which accounted for 98.5% of the total oil composition. Table 1 showed that the main components of volatile oil from the branches of *P. quassioides* were terpenoids, hydroxy compounds, and other acyclic alkane compounds. After performing area normalization, the eleven components with the highest relative content fraction (> 2%) are caryophyllene (12.83%), 12-oxabicyclo[9.1.0]dodeca-3,7-diene,1,5,5,8-tetramethyl-, [1*R*-(1*R**,3*E*,7*E*,11-*R**)] (12.29%), 1-hexanol (9.96%), naph-thaalene,1,2,3,5,6,7,8,8a-octahydro-1,8a-dimethyl-7-(1-methyletheyl)-[1*S*-(1a,7a,8aa)] (7.32%), aromadendrene oxide-(2) (6.69%), α-caryophyllene (3.88%), 6,10-dodecatrien-3-ol,3,7,11-trimethyl-[*S*-(*Z*)]-a (2.89%), naphthalene,1,2,3,4,4-a,5,6,8a-octahydro-4a,8-dimethyl-2-(1-methylethylidene)-(4a*R*-*trans*) (2.8%), furan,2-pentyl (2.86%), 2(3H)-naphthalenone,4,4a,5,6, 7,8-hexahydro-4,4a-di-methyl-6-(1-methylethenyl) (2.12%), and cyclohexane,1-ethenyl-1-methyl-2,4-bis(1-methylethenyl)-[1*S*-(1a,2b,4b)] (2.11%).

In this study, the volatile oil in the branches of *P. quassioides* from Beijng was first analyzed using the GC-MS method. A total of 46 compounds were identified. Yang *et al* (2011) investigated the volatile oil in the branches of *P. quassioides* from Guizhou and found that the terpenoids and their derivatives were the primary constituents. Most of the terpenoids and their derivatives were the important natural medicinal chemical constituents with wide biological activities (Yang, 2005; Sun, 1988). We compared the differences between the two populations of this species originated

Table 1 Chemical constituents of volatile oil in branches of *P. quassioides*

No.	Compounds	MW	Formula	*t* / min	Area / %
1	1-hexanol	102	$C_6H_{14}O$	5.65	9.96
2	2-pentylfuran	138	$C_9H_{14}O$	9.17	2.86
3	cyclohexene,1-methyl-4-(1-methylethenyl)-(_S_)-	136	$C_{10}H_{16}$	10.46	1.54
4	henzeneacetaladehyde	120	C_8H_8O	11.26	0.65
5	1,6-octadien-3-ol,3,7-dimthyl-	154	$C_{10}H_{18}O$	13.14	0.22
6	nonanal	142	$C_9H_{18}O$	13.27	1.04
7	2-nonenal,(_E_)-	140	$C_9H_{16}O$	15.26	0.33
8	decanal	156	$C_{10}H_{20}O$	16.84	0.42
9	1-cyclohexenne-1-carboxaldehyde,4-(1-methylethyl)-	152	$C_{10}H_{16}O$	17.33	0.54
10	2-cyclohexen-1-ol,2-methyl-5-(1-methyleyhenyl)-,_cis_-	152	$C_{10}H_{16}O$	17.46	0.33
11	2-cycloheven-1-one,2-methyl-5-(1-methylethenyl)-,(_S_)-	150	$C_{10}H_{14}O$	18.27	0.34
12	2-decenal,(_E_)-	154	$C_{10}H_{18}O$	18.83	0.53
13	bicyclo[3.3.1]nonan-1-ol	140	$C_9H_{16}O$	19.17	0.39
14	2-methoxy-4-vinylphenol	150	$C_9H_{10}O_2$	20.78	0.17
15	2,4-decadienal	152	$C_{10}H_{16}O$	20.96	1.46
16	1-methyl-2,4-bis(1-methylethylidene)-1-vinylcyclohexane	204	$C_{15}H_{24}$	21.20	0.13
17	copaene	204	$C_{15}H_{24}$	23.10	1.17
18	cyclohexane,1-ethenyl-1-methyl-2,4-bis(1-methylethenyl)-,[1_S_-(1a,2b,4b)]-	204	$C_{15}H_{24}$	23.78	2.11
19	caryophyllene	204	$C_{15}H_{24}$	25.22	12.83
20	naphthalene,1,2,3,4,4a,5,6,8a-octahydro-4a,8-dimethyl-2-(1-methylethenyl)-[2_R_-(2a,4aa,8ab)]	204	$C_{15}H_{24}$	26.48	1.14
21	aromadendrene	204	$C_{15}H_{24}$	26.64	0.69
22	α-caryophyllene	204	$C_{15}H_{24}$	26.87	3.88
23	1,6-cyclodecadiene,1-methyl-5-methylene-8-(1-methylethyl)-,[s-(_E_,_E_)]-	204	$C_{15}H_{24}$	28.10	2.00
24	spiro[5.5]undec-2-ene,3,7,7-trimethyl-11-methylene-,(−)-	204	$C_{15}H_{24}$	28.24	1.04
25	naphthalene,1,2,3,4,4a,5,6,8a-octahydro-4a,8-dimethyl-2-(1-methylethenyl)-[2_R_-(2a,4aa,8ab)]	204	$C_{15}H_{24}$	28.48	0.54
26	naphthalene,1,2,3,5,6,7,8,8a-octahydro-1,8a-dimethyl-7-(1-methylethenyl)-,[1_S_-(1a,7a,8aa)]	204	$C_{15}H_{24}$	28.78	7.32
27	naphthalene,decahydro-4a-methyl-1-methylene-7-(1-methylethenyl)-,[4a_R_-(4aa,7a,8ab)]	204	$C_{15}H_{24}$	28.80	0.42
28	naphthalene,1,2,4a,5,6,8a-hexahydro-4,7-dimethylethyl-1-(1-methylethyl)-(1.alpha,4a.alpha.8a.alpha)	204	$C_{15}H_{24}$	29.00	0.50
29	trans-.alpha-bergamotene	204	$C_{15}H_{24}$	29.34	0.32
30	4,4-dimethyl-3-(3-methylbut-3-enylidene)-2-methylenebicyclo[4.1.0]heptane	202	$C_{15}H_{22}$	29.76	2.05
31	naphthalene,1,2,3,4,4a,5,6,8a-octahydro-4a,8-dimethyl-2-(1-methylethylidene)-,(4a_R_-trans)	204	$C_{15}H_{24}$	30.00	2.86
32	caryophylleneoxide	220	$C_{15}H_{24}O$	31.61	1.14
33	1-oxaspiro[2.5]octane,5,5-dimethyl-4-(3-methyl-1,3-butadienyl)-	206	$C_{15}H_{22}O$	31.80	1.44
34	1,6,10-dodecatrien-3-ol,3,7,11-trimethyl-,[_S_-(_Z_)]-	222	$C_{15}H_{26}O$	32.47	2.89
35	12-oxabicyclo[9.1.0]dodeca-3,7-diene,1,5,5,8-tetramethyl-,[1_R_-(1_R_*,3_E_,7_E_,11_R_*)]-	220	$C_{15}H_{24}O$	34.80	12.29
36	tetracyclo[6.3.2.0(2,5).0(1,8)]tridecan-9-ol,4,4-dimethyl-	220	$C_{15}H_{24}O$	36.28	3.08
37	1H-3a,7-Methanoazulen-5-ol,octahydro-3,8,8-trimethyl-6-methylene-	220	$C_{15}H_{24}O$	37.07	1.36
38	aromadendrene oxide-(2)	220	$C_{15}H_{24}O$	37.34	6.69
39	isoarmadendrene epoxide	220	$C_{15}H_{24}O$	38.12	2.53
40	2-naphthalenol,2,3,4,4a,5,6,7-octahydro-1,4a-dimethyl-7-(2-hydroxy-1-methylethyl)	220	$C_{15}H_{26}O$	39.26	0.90
41	pentadecanal-	226	$C_{15}H_{30}O$	40.22	0.82
42	aromadendreneoxide-(1)	220	$C_{15}H_{24}O$	42.66	1.31
43	aceticacid,3-hydroxy-6-isopropenyl-4,8a-dimethyl-1,2,3,5,6,7,8,8a-octahydronaphthalen-2-ylester	278	$C_{17}H_{26}O_3$	42.86	1.01
44	2(3H)-naphthalenone,4,4a,5,6,7,8-hexahydro-4,4a-dimethyl-6-(1-methylethenyl)-	218	$C_{15}H_{22}O$	44.57	2.12

(To be continued)

(Continued Table 1)

No.	Compounds	MW	Formula	*t* / min	Area / %
45	4-(3,3-dimethyl-but-1-ynyl)-4-hydroxy-2,6,6-trimethylcyclohex-2-enone	234	$C_{15}H_{22}O_2$	45.94	0.47
46	1,2-benzenedicarboxylic acid, butyl octyl ester	334	$C_{20}H_{30}O_4$	47.38	0.53
			Identified		98.35
47	dibutylphthalate	278	$C_{16}H_{22}O_4$	51.95	0.55
48	heptadecane	240	$C_{17}H_{36}$	53.85	0.32
49	pentadecane	212	$C_{15}H_{32}$	58.40	0.78
			Unidentified		1.65

form Guizhou and Beijing. The major constituents in the branches of *P. quassioides* from Guizhou were *trans*-caryophyllene (5.530%), α-bergamotene (9.997%), *trans*-β-farnesene (7.994%), cuminol (15.416%), and β-bisabolene (12.733%) (Yang *et al*, 2011). However, from a chemosystematic point of view, each species has its own characteristic type of volatile oil. Cuminol (15.416%) and β-bisabolene (12.733%) were the predominant in the sample from Guizhou, whereas these two constituents did not exist in the sample from Beijing (Yang *et al*, 2011). The essential oil from Beijing was rich in caryophyllene (12.83%) and 12-oxabicyclo[9.1.0]dodeca-3,7-diene, 1,5,5,8-tetramethyl-[1*R*-(1*R**,3*E*,7*E*,11-*R**)] (12.29%); Whereas, the both two were the minor constituents in the sample from Guizhou. The result showed that the *P. quassioides* samples from Beijing and Guizhou differed in essential compositions, which implied the different chemical-ecological types. However, further tests are needed to confirm this finding.

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