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Preparation and Chemical Analysis of Volatile Oil in Seven-lobed leaf type of *Ficus hirta* Vahl.

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Abstract. To analyze the chemical constituents of volatile oil in *Ficus hirta* Vahl. of seven-lobed leaf type. The volatile chemical components of extracts were analyzed using GC-MS, and the relative contents of various chemical components in the volatile oils were calculated by area normalization method. The volatile oils from *Ficus hirta* Vahl. of seven-lobed leaf type were detected to contain 50 chemical components respectively. The main components are compounds such as esters, aldehydes, alcohols, coumarins, and organic acids, and some terpenoids, alkaloids, phenols, and ketones are also present. The method is simple and efficient, and can be used for preliminary identification of volatile oil components from *Ficus hirta* Vahl. of seven-lobed leaf type.

1. Introduction

Ficus hirta Vahl. is a common herbal medicine grows in the south of the Five Ridges that belongs to the family of Moraceae. It is mainly distributed in temperate and subtropical regions of Asia, especially in south China, and the most widely artificial planting area is Heyuan of Guangdong province[1-2]. Most of them are shrubs while few are small arbor. The whole stem, peel and leaf contain emulsion, and the aroma exists in its root and bark. Its components include organic acids, amino acids, terpenes, alkaloids, coumarins, etc. *Ficus hirta* Vahl. has the effects of improving digestive and respiratory system, enhancing immune function, and anti-inflammatory[3-7]. In TCM(Traditional Chinese Medicine) it is used as a medicinal plant for nephritis, hepatitis, mastitis, bruises, injuries, rheumatism, cough and promotion of lactation during childbirth[8]. *Ficus hirta* Vahl. is a medicinal and edible plants, it is used for cooking soup in Guangdong and has the fragrance of coconut milk. It has the effects of invigorating spleen, eliminating dampness, relieving cough and moistening lung. Its application has a long history and its health-care function is widely recognized[9-10].

The shape of the leaves of the *Ficus hirta* Vahl. includes single leaf, three-lobed leaf, five-lobed leaf and seven-lobed leaf. Although the leaf type of *Ficus hirta* Vahl. is different, there are not distinguished when being raised seedlings or applied. In general, there is only one leaf type on one *Ficus hirta* Vahl. plant, and the odors of different *Ficus hirta* Vahl. with the same picking conditions are different. The odour of Seven-lobed leaf type of *Ficus hirta* Vahl. was significantly different from



that of the other *Ficus hirta Vahl.* and has the strongest aromatic smell. It was speculated that the chemical composition and percentage content of volatile oil in different leaf types of *Ficus hirta Vahl.* were different, and no reports have been related to the chemical composition of volatile oil in Seven-lobed leaf type of *Ficus hirta Vahl.*. In order to provide a theoretical basis for the development and utilization of Seven-lobed leaf type of *Ficus hirta Vahl.*, the chemical constituents of the volatile oil were analyzed by GC-MS technique in this study.

2. Instruments, Samples and Reagents

2.1. Instruments

DSQII Gas Chromatography-Mass Spectrometry (Seymour Fisher Company, USA); Sartorius MA35M moisture measuring instrument (Guangzhou Botai Science and Technology instrument Co., Ltd); YP 3001N electronic balance (Shanghai Jing Branch Instrument Co., Ltd.); Sartorius One Over Ten-thousand Analytical Balance (Beijing Sartorius Scientific Instruments Co., Ltd.); DFT-200 Portable High Speed Universal Crusher (Wenling Linda Machinery Co., Ltd.).

2.2. Samples and Reagents

Seven-lobed leaf type of *Ficus hirta Vahl.* (The year of the plant is 4.5 years. it was collected by Heyuan city Jinyuan green life co., ltd. in the GAP industrial planting demonstration base of *Ficus hirta Vahl.* in Heyuan city, Guangdong province, and it was certified as genuine by Lin Zhiyun senior experimenter of Department of Traditional Chinese Medicine, Guangdong University of Pharmacy); Cyclohexane is of analytical grade (Tianjin Zhiyuan Chemical Reagent Co., Ltd.).

3. Methods and Results

3.1. Volatile Oil Extraction

3.1.1. Sample Solution Configuration. Pulverizing freshly picked *Ficus hirta Vahl.* of seven-lobed leaf type, taking 300g of medicinal materials with the same amount after water content is converted, put into a 5000ml round bottom flask, add five times the amount of water, soak for 0.5h, and then extract for 1.5h. The collected distillate was added with 6ml cyclohexane for extraction, transferred to a separatory funnel, the upper oil phase was collected, and the remaining 2ml was blown with nitrogen.

3.2. GC-MS Conditions

3.2.1. Gas Chromatography Conditions. The GS system used chromatographic separation was achieved by using a fused capillary column DM-5MS, length (30m×0.25mm×0.25um). The inlet temperature was 250°C. The initial column temperature was kept at 60 °C for 3min, and it was raised to 180 °C at 10°C/min for 8min, and then raised to 230 °C at 15°C/min for 5min. The carrier gas was high purity helium with a flow rate of 1.0 mL / min. The volume sample is 1.0µl.

3.2.2. Mass Spectrometry Conditions. The interface temperature between chromatography and mass spectrometry is 230°C. Electron bombardment source is used as ionization mode. The monitoring method is full scanning. The scanning range is 35-500. The ionization energy is 70eV. Solvent delay 4 min. The ion source temperature is 250°C.

3.3. Results

In order to obtain the total ion chromatogram, the volatile oil of the *Ficus hirta Vahl.* of seven-lobed leaf type were analyzed by GC-MS (Figure 1). The results and mass spectral information is automatically retrieved by a computerized data processing system. The components of Volatile oil were identified and compared with the standard mass spectrometer library (INST2005), and the relative content of each component was calculated by the peak area normalization method.

RT: 0.00 - 31.37

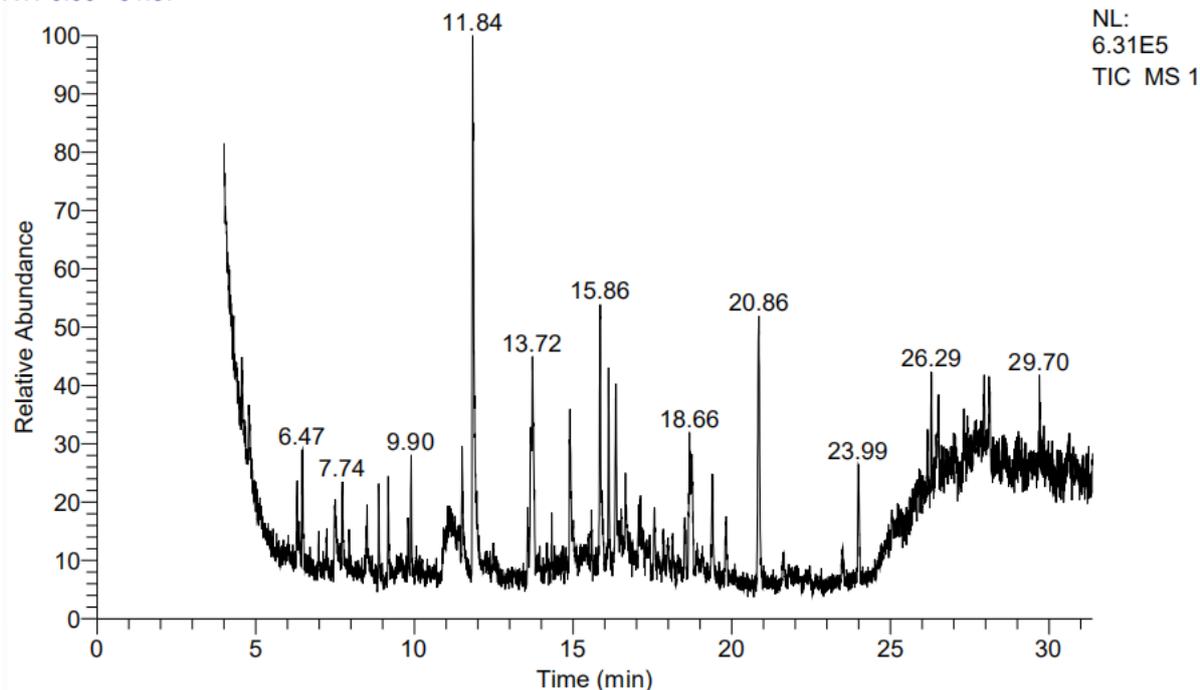


Figure 1. Total ion chromatogram of the volatile oil of the seven-lobed *Ficus hirta* Vahl.

Table 1. Chemical Analysis of Volatile Oil from the Seven-lobed *Ficus hirta* Vahl.

Number	Retention (min)	Compound	Molecular	Relative molecular	Relative content(%)
1	11.84	(E,E)-2,4-Decadienal	C ₁₀ H ₁₆ O	152	9.2
2	13.72	Tetradecamethyl-Cycloheptasiloxane	C ₁₄ H ₄₂ O ₇ Si ₇	518	8.3
3	20.85	Diisobutyl phthalate	C ₁₆ H ₂₂ O ₄	278	6.15
4	15.85	Hexadecamethyl-Cyclooctasiloxane	C ₁₆ H ₄₈ O ₈ Si ₈	592	4.49
5	18.68	Acetic acid, (1,2,3,4,5,6,7,8-octahydro-3,8,8-trimethylnaphth-2-yl)methyl ester	C ₁₆ H ₂₆ O ₂	250	3.78
6	16.35	7-(1,1-dimethylethyl)-3,4-dihydro-1(2H)-naphthalenone	C ₁₄ H ₁₈ O	202	3.38
7	14.91	mellein	C ₁₀ H ₁₀ O ₃	178	2.81
8	16.12	(-)- α -acoradiene	C ₁₅ H ₂₄	204	2.76
9	24.00	Dibutyl phthalate	C ₁₆ H ₂₂ O ₄	278	2.31
10	26.51	3-ethyl-5-(2-ethylbutyl)octadecane	C ₂₆ H ₅₄	366	2.06
11	19.39	1,4-methanoazulen-9-ol, decahydro-1,5,5,8 α -tetramethyl-(1R,3 α R,4S,8 α S,9S)-	C ₁₅ H ₂₆ O	222	2.04
12	17.12	9-Isopropyl-1-methyl-2-methylene-5-oxatricyclo[5.4.0.0(3,8)]undecane	C ₁₅ H ₂₄ O	220	1.95
13	6.47	2-Pentylfuran	C ₉ H ₁₄ O	138	1.87
14	6.3	Octamethyl-Cyclotetrasiloxane	C ₈ H ₂₄ O ₄ Si ₄	296	1.69
15	29.96	1,1,3,3,5,5,7,7,9,9,11,11,13,13,15,15-hexadecamethyl-octasiloxane	C ₁₆ H ₅₀ O ₇ Si ₈	578	1.67

Number	Retention (min)	Compound	Molecular	Relative molecular	Relative content(%)
16	25.88	1-Monolinoleoylglycerol trimethylsilyl ether	C ₂₇ H ₅₄ O ₄ Si ₂	498	1.65
17	28.12	6-AH-cAMP	C ₁₆ H ₂₄ N ₆ O ₆ P	427	1.65
18	11.05	Dodecamethyl-cyclohexasiloxane	C ₁₂ H ₃₆ O ₆ Si ₆	444	1.63
19	8.5	Nonanal	C ₉ H ₁₈ O	142	1.59
20	7.5	phenylacetaldehyde	C ₈ H ₈ O	120	1.49
21	9.18	1-methyl-4-(1-methylethyl)-2-cyclohexen-1-ol	C ₁₀ H ₁₈ O	154	1.46
22	9.9	3,6-Dimethyl-2,3,3a,4,5,7a-hexahydrobenzofuran	C ₁₀ H ₁₆ O	152	1.39
23	26.29	1-Nonadecene	C ₁₉ H ₃₈	266	1.26
24	19.82	Isopropyl Myristate	C ₁₇ H ₃₄ O ₂	270	1.19
25	4.79	pentan-2-ylcyclopropane	C ₈ H ₁₆	112	1.16
26	17.57	2-(4a,8-Dimethyl-1,2,3,4,4a,5,6,7-octahydro-naphthalen-2-yl)-prop-2-en-1-ol	C ₁₅ H ₂₄ O	220	1.14
27	7.73	cis-3,7-Dimethyl-2,6-octadienol	C ₁₀ H ₁₈ O	154	1.11
28	17.99	(-)-Caryophyllene-(II)	C ₁₅ H ₂₄	204	1.06
29	25.24	9,12,15-Octadecatrienoic acid, 2,3-bis[(trimethylsilyl)oxy]propyl ester, (Z,Z,Z)	C ₂₇ H ₅₂ O ₄ Si ₂	496	1.03
30	27.32	Ethyl iso-allocholate	C ₂₆ H ₄₄ O ₅	436	1.03
31	8.88	1-methyl-4-(1-methylethyl)-2-cyclohexen-1-ol	C ₁₀ H ₁₈ O	154	1
32	18.53	β-Guaiene	C ₁₅ H ₂₄	204	0.94
33	21.63	1-Hexadecanol, 2-methyl	C ₁₇ H ₃₆ O	256	0.92
34	17.41	2-Hydroxy-8-pyridin-2-yl-oct-7-enenitrile	C ₁₃ H ₁₆ N ₂ O	216	0.85
35	18.14	2-methyl-4-(2,6,6-trimethyl-1-cyclohexen-1-yl)-2-Butenal	C ₁₄ H ₂₂ O	206	0.79
36	9.81	Alpha-Terpineol	C ₁₀ H ₁₈ O	154	0.79
37	23.49	Octadecamethyl-Cyclononasiloxane	C ₁₈ H ₅₄ O ₉ Si ₉	666	0.77
38	26.17	Yohimbine	C ₂₁ H ₂₆ N ₂ O ₃	354	0.76
39	17.85	6-(1,3-Dimethyl-buta-1,3-dienyl)-1,5,5-trimethyl-7-oxa-bicyclo[4.1.0]hept-2-ene	C ₁₅ H ₂₂ O	218	0.71
40	7.23	4-methylcyclohex-3-ene-1-carbaldehyde	C ₈ H ₁₂ O	124	0.67
41	14.32	2,4-Di-tert-butylpheno	C ₁₄ H ₂₂ O	206	0.65
42	16.5	1H-Cyclopenta[a]pentalen-7-ol, decahydro-3,3,4,7a-tetramethyl-, acetate	C ₁₇ H ₂₈ O ₂	264	0.62
43	12.48	cholic acid ethyl ester	C ₂₆ H ₄₄ O ₅	436	0.58
44	27.43	Dibutyl sebacate	C ₁₈ H ₃₄ O ₄	314	0.57
45	18.91	5-butylpiperidine-2-carboxylic acid	C ₁₀ H ₁₉ NO ₂	185	0.54
46	15.58	Globulol	C ₁₅ H ₂₆ O	222	0.51

Number	Retention (min)	Compound	Molecular	Relative molecular	Relative content (%)
47	7.95	2,7-Diphenyl-1,6-dioxopyridazino[4,5:2',3']pyrrolo[4',5'-d]pyridazine	C ₂₀ H ₁₃ N ₅ O ₂	355	0.49
48	6.99	(4-methyl-1-propan-2-ylcyclohex-3-en-1-yl) acetate	C ₁₂ H ₂₀ O ₂	196	0.46
49	14.17	1-[3-(2,6,6-Trimethyl-cyclohex-2-enyl)-4,5-dihydro-3H-pyrazol-4-yl]-ethanone	C ₁₄ H ₂₂ N ₂ O	234	0.45
50	25.06	9,12,15-Octadecatrienoic acid	C ₂₇ H ₅₂ O ₄ Si ₂	496	0.37

4. Discussion

This experiment used steam distillation to extract volatile oil. Under the action of saturated steam infiltration and diffusion, the low boiling point oil in *Ficus hirta Vahl.* was separated first. When the steam flow rate is increased, the slightly higher boiling point oil is distilled out, thus achieving efficient extraction of volatile oil.

According to the result of research, the *Ficus hirta Vahl.* of seven-lobed leaf type volatile oil contains 50 compounds, of which the main components are esters (27.88%), aldehydes (23.74%), alcohols (19.57), coumarins (8.40%), organic acids (7.94%). In addition, there are also some terpenoids, alkaloids, phenols, ketones and so on, and more than 5.0% are (E,E)-2,4-Decadienal(9.2%), Tetradecamethyl-Cycloheptasiloxane (8.3%), Phthalic acid, isobutyl 4-octyl ester (6.15%). (E,E)-2,4-Decadienal is an edible spice (incense), Commonly used as food additives. Tetradecamethyl-cycloheptasiloxane is used in cosmetic and personal care products. Diisobutyl phthalate is mainly used as plasticizer for PVC.

Seven-lobed leaf type of *Ficus hirta Vahl.* has a unique and intense fragrance, and the results show that there are 12 aroma compounds with a total content of 27.92%, including 2-Pentylfuran (1.87%), phenylacetaldehyde (1.49%). Cis-3, 7-Dimethyl-2,6-octadienol (1.11%) Nonanal (1.59%), Alpha-Terpineol (0.79%), 3, 6-min-2, 3, 3 a, 4, 5, 7 a -hexahydrobenzofuran (1.39%), (E, E)-2, 4-Guaiene (9.2%), Globulol (0.51%), β -Guaiene (0.94%), Diisobutyl phthalate (6.15%), Dibutyl phthalate (2.31%), Dibutyl sebacate (0.57%). There are many terpenoids in the volatile oil, mainly monoterpenes and sesquiterpenes. These identified components have the effects of anti-inflammatory and antiviral activities, and they are also natural plant essence so that can be used in the development of essence products, and considered to be added to food or cosmetics to improve the smell of products.

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