

Preparation and Chemical Analysis of Volatile Oil in Ficus Hirta

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Abstract. Subcritical fluid extraction technology was used to collect the extract of Ficus hirta, and the volatile oil was obtained by molecular distillation. The chemical composition of the volatile oil was characterized by gas chromatography-mass spectrometry (GC-MS), and the relative content of each component in the volatile oil was obtained by GC-MS peak area normalization analysis. Subcritical extraction technology combined with molecular distillation method is not only can be very good in the preparation of volatile oil Ficus hirta, but also can preserved its rich fragrance; Ficus hirta volatile oil identified a total of 29 kinds of substances, of which the higher levels of components of caproic acid, pelargonic acid, psoralen, caprylic acid. This method is simple and feasible, what's more, it can be used to prepare the composition of the volatile oil from Ficus hirta preliminary identification.

1. Introduction

Medicinal Ficus hirta, which is comes from Southern China, is the mulberry plants Ficus hirta Vahl dry roots [1]. Ficus hirta is a medicinal and edible plants [2], with the spleen dampness, lungs cough effect [3]. Ficus hirta are mainly produced in Guangdong, Guangxi, Hunan and other places. Due to the fact that it has the high economic value, at present, Heyuan and other places in Guangdong have a large area of artificial cultivation [4].

Subcritical fluid extraction is based on the principle of similar compatibility of organic matter, the target ingredient in the material is transferred to the liquid extractant and the extractant is separated from the product by evaporation under reduced pressure^[5]. Molecular distillation, also known as short range distillation, is a fast, efficient, non-polluting separation and concentration technique. It is also considered as a common method for the separation and purification of natural products. It is especially suitable for the separation of high boiling point, high viscosity and heat sensitive materials. It is widely used in the separation of volatile oil of Chinese herbal medicine and other oils [6,7]. Furthermore, it has broad application prospects. However, the molecular distillation technology used in the research of Volatile oil of Ficus hirta has not been reported at home and abroad. In this study, Ficus hirta subcritical extracts were purified by molecular distillation to obtain the volatile oil. The constituents were analyzed by GC-MS, which provided a scientific basis for the development and utilization of the volatile oil of Ficus hirta.



2. Instruments, samples and reagents

2.1. Instruments

D-70 molecular distillation (Guangzhou Green and edge Biological Technology Co., Ltd.); YLJ-3 subcritical extraction unit (Guangzhou Green and edge Biological Technology Co., Ltd.); YP 3001N electronic balance (Shanghai Jing Branch Instrument Co., Ltd.); TRACE DSQ GC-MS (The United States Fenigen mass spectrometry company); DFT-200 portable high-speed grinder (Wenling Lin Machinery Co., Ltd.); Sartorius hundred thousandth electronic balance (Beijing Sartorius Instrument System Co., Ltd.).

2.2. Samples and reagents

Ficus hirta (Provided by Heyuan Jinyuan Green Life Co., Ltd., and it was identified as the dry root of *Ficus hirta* Vahl .by Moraceae *Ficus* plant by the Professor Liu Jizhu of Department of Traditional Chinese Medicine, Guangdong University of Pharmacy); Ethyl acetate is of analytical grade (Tianjin Zhiyuan Chemical Reagent Co., Ltd.); Butane (Food grade).

3. Methods and results

3.1. Volatile oil extraction

3.1.1. Subcritical extraction. The *Ficus hirta* crushed and passed through a 20 mesh screen. The smashing pinch *Ficus hirta* powder placed in the extraction tank, capped with screws. When the extraction pressure is reduced to -0.1MPa, the butane from the storage tank pumped into the extraction tank; after extraction, turn on the compressor for solvent recovery and desolventizing to collect the *Ficus hirta* subcritical extract. Extract is a yellow semi-solid fat.

3.1.2. Molecular distillation. Turn on the condenser, condensate temperature 30 °C after filling the *Ficus hirta* sub-critical extraction into the the molecular distillation unit feed bottle. After cooling the system, the work is set to 1 Pa degree of vacuum, with a distillation temperature of 70 °C. Pressure stability, vacuum and temperature have reached the set value, the film speed adjust 500r/min, and open the feed valve. At a feed temperature of 70 °C, molecular distillation was started, and fraction 1, fraction 2 and fraction 3 were collected.

3.1.3. Sample solution configuration. 20 μ l of *Ficus hirta* molecular distilled volatile oil (component 3) was placed in an 1ml volumetric flask. After dissolution and dilute with ethyl acetate, shake it well and filter it with 0.22 μ m filter, the filtrate stored in the liquid vial for GC-MS analysis as the test solution.

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 (30 $\times$ 0.25 mm, 3 μ m). The inlet temperature and the interface temperature were 250 °C and 230 °C, respectively. The carrier gas was 99.99% high purity Helium with a flow rate of 1.0mL/min. The sample of volume was 1.0 μ l, which was injected on to the column, with a column pressure of 80kPa. The injection mode was splitless. Oven temperature program was initially set at 40 °C for 2 min, then ramped at 10 °C /min to 70 °C, then ramped at 10 °C /min to 280 °C and held for 10 min.

3.2.2. Mass spectrometry conditions. The mass spectrometer ion source was an EI source and the ion source temperature was 200 °C; the electron multiplier voltage was 520V; the full ion scan gap was 1.0s and the scan rate was 1000 amu/s.

3.3. Results

3.3.1. *Ficus hirta* molecular distillation. Molecular distillation gets three fractions. It is a brown paste at room temperature and light in weight for a single component. Light Component 2 is a brownish-yellow paste and light in odor; cold trap component 3 is a yellow liquid and is rich in odor.

3.3.2. *Ficus hirta* volatile oil GC-MS analysis. In order to obtain the total ion chromatogram, the volatile oil of the *Ficus hirta* 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 (NIST08), and the relative content of each component was calculated by the peak area normalization method.

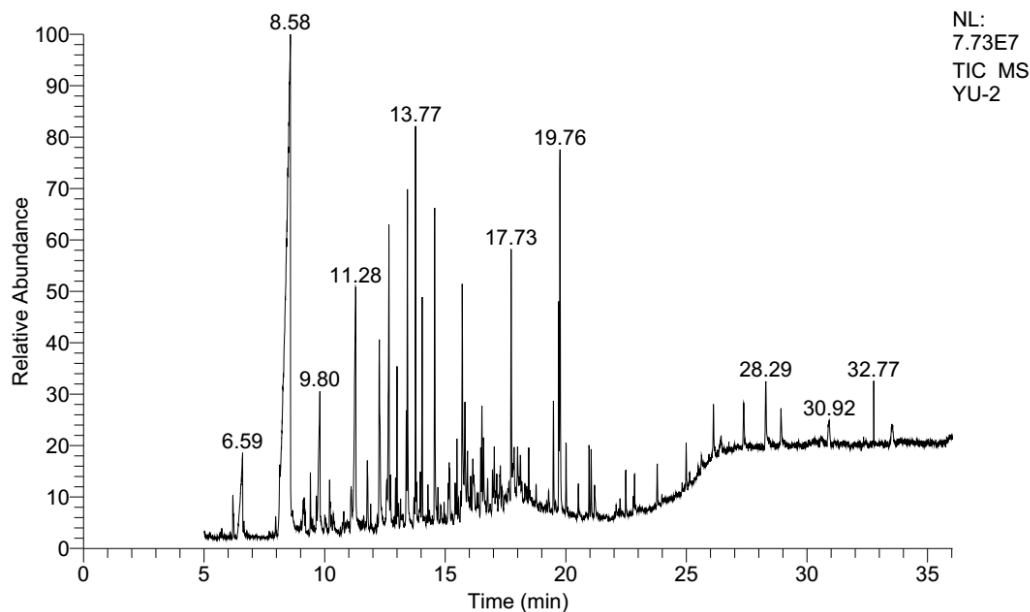


Figure 1. The total ion chromatogram of the *Ficus hirta* Vahl molecular distillation of volatile

Table 1. Component analysis of the Ficus hirta molecular distillation of volatile oil

Number	Retention time (min)	Compound	Molecular formula	Relative molecular mass	Relative content (%)
1	6.58	Pentanoic acid	C ₅ H ₁₀ O ₂	102	2.94
2	8.56	Hexanoic acid	C ₆ H ₁₂ O ₂	116	34.37
3	9.41	4-Hexanolide	C ₆ H ₁₀ O ₂	114	0.79
4	9.79	Heptanoic acid	C ₇ H ₁₄ O ₂	130	3.28
5	10.21	Nonanal	C ₉ H ₁₈ O	142	0.94
6	11.10	2-Octenoic acid,(E)-	C ₈ H ₁₄ O ₂	142	1.79
7	11.28	Octanoic Acid	C ₈ H ₁₆ O ₂	144	5.69
8	12.27	2-Coumaranone	C ₈ H ₆ O ₂	134	3.53
9	12.66	Nonanoic acid	C ₉ H ₁₈ O ₂	158	5.25
10	13.00	Benzene, 1-methoxy-4-(1-propenyl)-	C ₁₀ H ₁₂ O	148	1.89
11	13.43	Bicyclo[3.3.2]decan-9-one	C ₁₀ H ₁₆ O	152	4.49
12	13.77	Bicyclo[2.2.2]octane,1-methoxy-4-methyl-	C ₁₀ H ₁₈ O	154	3.89
13	14.05	gamma-Nonanolace	C ₉ H ₁₆ O ₂	156	2.94
14	14.56	Vanillin	C ₈ H ₈ O ₃	152	3.66
15	15.17	5-Hepten-3-yn-2-ol,6-methy	C ₁₁ H ₁₈ O	166	1.45
16	15.48	Eudesma-3,7(11)-diene	C ₁₅ H ₂₄	204	1.42
17	15.81	b-Guaiene	C ₁₅ H ₂₄	204	1.77
18	15.92	Naphthalene,1,2,3,4,4a,5,6,8a-octahydro-4a,8-dimethyl-2-(1-methylethenyl)-, (2R,4aR,8aR)-	C ₁₅ H ₂₄	204	1.14
19	16.15	3-O-Acetyl-8-O-tigloylingol	C ₂₇ H ₃₈ O ₈	490	1.39
20	16.52	Mellein	C ₁₀ H ₁₀ O ₃	178	2.55
21	17.03	Caryophyllene oxide	C ₁₅ H ₂₄ O	220	1.64
22	17.28	1-Cyclohexanone,3,3-dimethyl-2-[5-methoxy-3-methyl-2-pentenylidene]-	C ₁₅ H ₂₄ O ₂	236	1.17
23	18.00	5-Methoxy-2,2,6-trimethyl-1-(3-methyl-but-1,3-dienyl)-7-oxa-bicyclo[4.1.0]heptane	C ₁₅ H ₂₄ O ₂	236	1.31
24	18.11	Octadecane,3-ethyl-5-(2-ethylbuty)-	C ₂₆ H ₅₄	366	1.36
25	19.75	Psoralen	C ₁₁ H ₆ O ₃	186	5.98
26	20.01	Diisobutyl phthalate	C ₁₆ H ₂₂ O ₄	278	0.77
27	20.97	Dibutyl phthalate	C ₁₆ H ₂₂ O ₄	278	0.70
28	21.05	8-(4-Chlorophenylthio)guanosine-3',5'-cyclic monophosphorothioate, Rp-isomer	C ₁₆ H ₁₅ Cl N ₅ O ₆ PS ₂	502	0.66
29	28.29	Erucylamide	C ₂₂ H ₄₃ N O	337	1.23

4. Discussion

Volatile oil obtained when steaming *Ficus hirta* with higher temperature, and heated a long time, it will cause the volatile oil decomposition of certain components or polymerization. The molecular distillation of volatile oil due to the fact that the distillation temperature is low, *Ficus hirta* heated time is short, easy to remove of butane, etc., which can retain the volatile oil well in the active ingredient is not destroyed, and it can preserve ingredients that are thermally unstable and susceptible to oxidation. It can preserve the peculiar aroma of the *Ficus hirta* in the volatile oil well.

Determination of Molecular *Ficus hirta* volatile oil samples, 29, of which the main component is long-chain organic acids (50.38%), coumarin (8.53%), and ether (7.67%) using GC-MS technique. In addition, there are saturated long-chain alkanes, aldehydes, short-chain organic acids and so on.

It shows a total ion flow diagram of *Ficus hirta* molecular distillation volatile oil (Figure 1). As can be seen from the figure, the baseline is uneven. As the temperature rises, the baseline tends to increase.

This may be due to the subcritical extraction of volatile oils containing some high-boiling substances that needed further isolation and identification.

The result of GC-MS analysis of molecular distillation was compared with steam distillation^[8]. The contents of long-chain organic acids were significantly increased, but the content of aldehydes and ketones decreased significantly, meanwhile, the molecular weight of the volatile oil of volatile oil was obviously higher than that of water vapor.

In this experiment, the extraction and separation of Volatile Oil from *Ficus hirta* through subcritical extraction-molecular distillation aims to find out an effective new way to obtain volatile oil. Compared with the traditional methods, this method had the advantages merits of low temperature, no pollution, etc., and has broad application prospects.

5. Acknowledgements

This work was financially supported by the joint project of the Department of Science and Technology of Guangdong Province and the Guangdong Provincial Academy of Chinese Medical Sciences (2016A020226038).

6. References

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