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Composition of volatile oil from the leaves of *Uncaria sessilifructus* Roxb.

Wang Dong-Mei and Huang Lin-Fang*
Institute of Medicinal Plant Development, Chinese Academic of Medical Science, Peking Union Medical College, Beijing 100193, China

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ABSTRACT

The chemical compositions of the volatile oil from the leaves of *Uncaria sessilifructus* Roxb., obtained by hydrodistillation, was analyzed by gas chromatography – mass spectrometry. A total of 70 compounds were identified representing 97.62% of total oil composition. Major components of the volatile oil were esters (30.45%)> alkane (12.45%)> olefine aldehyde (11.69%)> alkene (11.02%)> Ketene (8.06%), the large concentrations of the chemical composition is methyl salicylate, fumaricacid, tetradecyl trans-hex-3-enyl ester, squalene, bicyclo[2.2.1]heptane analysis based on principal components of *Uncaria sessilifructus* Roxb. The results showed that established GC-MS analysis method can comprehensively reflect the compositions of the volatile oil from the leaves of *Uncaria sessilifructus* Roxb. , and provide scientific evidences for further exploitation.

INTRODUCTION

Uncaria sessilifructus Roxb. is hooks of stems plants which belong to the genus Uncaria of Rubiaceae. As a original kind of sources of the Chinese drug "Gou-teng", It was recorded in Chinese Pharmacopoeia (2010 Edition, Volume I). The dried hooks generally have been used as spasmolytics, analgesics, and sedatives for symptoms associated with nervous disorders (Yano *et al.* 1991; Lee *et.al.* 1999a.; Lee *et al*. 1999b.). One of the primary uses of several Uncaria species and the traditional medicines derived from them is the treatment of hypertension (Aisaka *et al.* 1985). As far as investigation of our literature could ascertain.The chemical compositions of the volatile oil from the leaves of *Uncaria sessilifructus* Roxb has been no reported except for the early work that reported the presence of the hooks of stems (Phillipson *et al*. 1978), such as pentacyclic triterpenoids, mostly of the ursane-type A–E (2–34 through 2–38). In this work we report the chemical composition of volatile oil from the leaves of *Uncaria sessilifructus* Roxb. To our knowledge this is the first time to characterize the volatile oil components of *Uncaria sessilifructus* Roxb.

* *Corresponding Author*
Institute of Medicinal Plant Development, Chinese Academic of Medical Science, Peking Union Medical College, Beijing 100193, China.
Tel:86-10-57833197

MATERIAL AND METHODS

Plant material

The leaves of *Uncaria sessilifructus* Roxb. were collected from the Mountain on the northern slope of the peak at 500 m above sea level in Yunnan Province of China during December 2011 and identified by Prof. LIN Yu lin and Huang Lin fang in the Institute of Medicinal Plant Development, Chinese Academic of Medical Science, Peking Union Medical College, Beijing (China). A voucher specimen was deposited in the herbarium of the department.

Preparation of volatile oil by hydrodistillation

The volatile oils were extracted from 100 g of previously air-dried and powdered leaves and mixed with 800 mL of distilled water. The extraction was carried out by steam distillation in a Clevenger apparatus for 10 h (Demirci *et al.* 2008). Ethyl ether was used to extract essential oil from the water phase for three times. The ethyl ether fraction was dehydrated over anhydrous sodium sulphate and filtered. After concentration by rotary evaporation at room temperature (25 °C), the resulting volatile extract was kept at 4 °C for further analysis.

GC/MS analysis

The essential oils were diluted in diethyl ether (20 μL in 1 mL) and analyzed with Varian 431 GC-300 MS fitted with a HP-

5 MS fused silica capillary column, which with a (5% phenyl)-polymethylsiloxane stationary phase, film thickness of 0.25 μm, a length of 30 m, and an internal diameter of 0.25 mm was used for separation. Helium was used as a carrier gas with a flow rate of 1.0 mL/min; detector's temperature was 280 °C, interface temperature was 250 °C. Sample (1 μL) was injected into the injector with a split ratio of 10:1. The GC oven temperature program was used as follows: 40 °C initial temperature, hold for 2 min; increased at 2 °C/min to 100 °C, hold for 5 min; increased 5 °/min to 150 °C, hold for 10 min; increased 10 °C/min to 280 °C, hold for 10 min. Mass spectra: electron impact (EI+) mode, 70 ev and ion source temperature 230 °C. Mass spectra were recorded over 40-550 a.m.u range. Identification of the compounds was based on comparison of the corresponding mass spectra with data from The MS library [NIST database]. The relative amount (RA) of each oil component is expressed as percent peak area relatively to the total peak area.

RESULTS AND DISCUSSION

After extraction of essential oils by hydrodistillation, GC/MS was used to identify the volatile compounds in it. The volatile compositions of the leaves of *Uncaria sessilifructus* Roxb. are listed in Table 1.

Table. 1: Compositions of the volatile oil from the leaves of *Uncaria sessilifructus* Roxb.

NO.	compound	RA%
Alcohol		
1.	.alpha.-Methyl-.alpha.-[4-methyl-3-pentenyl]oxiranemethanolC	0.03
2.	yclohexanol,2,6-dimethyl-	0.03
3.	6,10,14-Trimethyl-pentadecan-2-ol	0.09
4.	1-Heneicosanol	0.47
5.	Isophytol	2.88
6.	Phytol	1.81
Enol		
7.	1,6-Octadien-3-ol,3,7-dimethyl-	0.03
8.	2-Cyclohexen-1-ol,2-methyl-5-(1-methylethenyl)-,cis-	0.13
9.	3-Cyclohexene-1-methanol,alpha,alpha,4-trimethyl-,(S)-	0.33
10.	2,6-Octadien-1-ol,3,7-dimethyl-,(E)-	0.69
11.	1,6,10-Dodecatrien-3-ol,3,7,11-trimethyl-,(E)-	0.81
12.	Selina-6-en-4-ol	0.09
13.	3,7,11,15-Tetramethyl-2-hexadecen-1-ol	0.74
14.	Eugenol	0.22
Aldehyde		
15.	Nonanal	0.19
16.	Tetradecanal	0.93
17.	Hexadecanal	2.31
18.	Pentadecanal	3.31
Olefine aldehyde		
19.	2-Undecenal	0.13
20.	9,17-Octadecadienal,(Z)-	3.24
21.	Cis,cis,cis-7.10.13-Hexadecatriena	5.52
22.	2-Decenal,(E)-	0.65
23.	2,4-Decadienal,(E,E)-	2.15
Alkane		
24.	Hentriacontane	0.97
25.	Pentadecane	0.91
26.	Hexadecane	1.06
27.	Tritetracontane	1.12
28.	Heneicosane	1.49
29.	Tetracosane	1.48
30.	Bicyclo[2.2.1]heptane	5.51
Alkene		
31.	2-Buten-1-0ne,1-(2,6,6-triehtyl-1,3-cyclohexadien-1-yl)-,(E)-	1.85
32.	Caryophyllene	0.23
33.	Caryophyllene oxide	0.53
34.	8-Heptadecene	0.93
35.	Squalene	5.43
36.	10-Heneicosene	1.05
37.	2-Hexadecene,3,7,11,15-tetramethyl-,[R-[R*,R*-(E)]]-	1.00
Aromatic hydrocarbon		
38.	1,2,4-Triazolidine-3,5-dione,1-(bicyclo[3.2.1]oct-2-en-4- yl)-4-phenyl-	0.14
39.	Naphthalene,1,2-dihydro-1,1,6-trimethyl-	0.29
40.	Naphthalene,decahydro-4a-methyl-1-methylene-7-(1-methylethenyl)-,[4aR-(4a.alph.,7.alpha.,8a.beta.]- b)	Trace
41.	Benzene,1,2,3,4-tetramethyl-2H-Benzopyran,3,4,4a,5,6,8a-hexahydro-2,5,5,8a-tetramethyl-(2.alpha.,4a.alpha.,8a.alpha.)-	0.14
42.	2H-Benzopyran,3,4,4a,5,6,8a-hexahydro-2,5,5,8a-tetramethyl-(2.alpha.,4a.alpha.,8a.alpha.)-	0.14
43.	Benzene,1,2,3,4-tetramethyl-	0.01
44.	1,2,4-Triazolidine-3,5-dione,1-(bicyclo[3.2.1]oct-2-en-4-yl)-4-phenyl-	0.14
45.	Benzenemethanamine,N-ethyl-N-Phenyli	1.15

Ester		0.55
46	Methyl salicylate	0.15
47.	Hexanoic acid,3-hexenyl ester,(Z)-	0.15
48.	Nonanoic acid,trimethylsilyl ester	0.72
49.	2-Hexenoic acid,butyl ester,(E)	1.52
50.	2-2-Hexenoic acid,2-hexenyl ester,(E,E)-	1.77
51.	3-Hexen-1-ol benzoate	2.38
52.	2-Oxobicyclo(3.2.2)nona-3,6-dien-1-yl benzoate	0.93
53.	6,9-Heptadecadiene	0.46
54.	Cis-3 Hexenyl salicylate	0.61
55.	Acetic acid,chloro-,octadecyl eser	2.42
56.	Phthalic acid,isobutyl4-octyl ester	0.81
57.	Hexadecanoic acid, methyl ester	2.06
58.	Dibutyl phthalate	0.29
59.	(Z)-14-Tricosenyl formate	5.62
60.	Fumaric acid,tetradecyl trans-hex-3-enyl ester	4.78
61.	1,2-Benzenedicarboxylic acid ,mono(2-ethylhexyl)ester	trace
62.	i-Propyl9.12.15-Octadecatrienoate	5.23
63.	Terephthalic acid, di(2-ethylhi-Propy l9.12.15 –Octadecatri enoateexyl) ester	
Ether		
64.	4,8,12,16-Tetramethylheptadecan-4-olide	2.98
Ketone		
65.	Phytol, trimethylsilyl ether	2.56
Ketene		
66.	3-Buten-2-one,4-(2,6,6-trimethyl-2-cyclohexen-1-yl)-	3.98
67.	3-buten-2-one,4-(2,6,6-trimethyl-1-cyclohexen-1-yl)	2.05
68.	2-Pentadecaone,6,10,14-trimethyl-	1.75
69.	2-Pentacosanone	0.27
Carboxylic acid		
70.	Propanoiacid,2-methyl-,1-(1,1-dimethylethyl)-2-methyl-1,3- propanediyl ester	3.28
total		97.62

The GC-MS analyses indicated that the oil is a complex mixture of 70 identified compounds which represent about 97.62% of the total oil (Table 1), including alcohols, enol, aldehydes, olefine aldehyde, esters, ether, ketene, carboxylic acids, alkanes and ketones are detected in the volatile oil of *Uncaria sessilifructus* Roxb leaf. The major component were in a decreasingorder, esters (30.45%) >alkane (12.45%) >olefin-aldehyde (11.69%) >alkene (11.02%)> ketene (8.06%). Fumaric acid, tetradecyl trans-hex-3-enyl ester, methyl salicylate, bicyclo[2.2.1]heptane, squalene are the major volatile compounds, which have anti-inflammatory, anti-arthritic, anti-diabetic, antiulcer, hypolipidemic, antiatherosclerotic, anti-HIV and cytotoxic activities (Safayhi and Sailer,1997). Several flavonoids isolated from the leaves of *Uncaria sessilifructus* Roxb. are used to treat blood capillary ailments.

Some aldehydes were also identified. Aldehydes are often important allelochemicals. Benzaldehyde is an attractant for some insects and repellent to others depending on its concentration (Cosse and Baker1998; Bargmann *et al*.1993; Nuttley *et al*.2001). It possesses antitermitic activity and can be a synergist to other allelochemicals (Chang and Cheng 2002). Benzoic acid and its derivatives possess antibacterial and antifungal activity and could have defensive functions (Terreaux *et al*. 1998). The presence of esters, ethers and the hydrocarbons can be used as taxonomic characteristics of Uncaria species.

CONCLUSION

Volatile oil compounds of the leaves from *Uncaria sessilifructus* Roxb. were analysed using GC-MS technique.

Volatile oil composed of mainly of alcohols, enol, aldehydes, olefine aldehyde, esters, ether, ketene, carboxylic acids, alkanes and ketones et.al. Further research on the diversity among species is in progress. It is helpful to explain the difference in the volatile oil composition. As a traditional Chinese medicine, the stems and branches with hook of Uncaria plants have been used for ages. Recent studies found that leaves of Uncaria had the similar chemical composition with stems. But in pharmaceutical industry, Uncaria leaf has not been exploited yet, which have resulted in a waste of resources. Our result will provide scientific evidences for sustainable development and utilization of *Uncaria sessilifructus* Roxb.

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