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Chemical Composition of the Essential Oil and Petroleum Ether Extract from Korea Pine Needle Leaves of *Cedrus deodara*

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The composition of the pine needles leaves of *Cedrus deodara* essential oil and petroleum ether extract of Gwangjin-gu region of Seoul, Korea were obtained by hydrodistillation and extraction, respectively analyzed by gas chromatography-mass spectrometry (GC-MS). The essential oil yield was (0.20 %, w/w) of leaves. Thirty eight and forty components comprising 99.2 and 99.7 % of the total peak area were identified in the essential oil and petroleum ether extract, respectively. Essential oil major components were dominated by: α-pinene (2.9 %), α-myrcene (9.9 %), dl-limonene (3.9 %), *trans*-caryophyllene (4.2 %), α-humulene (3.4 %), linalyl propionate (10.9 %), δ-cadinene (2.7 %), caryophyllene oxide (2.8 %), 1-dodecanol (2.9 %), α-cadinol (3.9 %), t-muurolol (4.4 %), dodecanoic acid (12.9 %). Petroleum ether extract major components were dominated by: butyl acetate (33.9 %), 4-allyloxy-2-methyl penta-en-2-ol (6.2 %), 2,2-dimethyl pentanal (2.4 %), α-pinene (2.9 %), 2-methyl-5-phenyl-5-pentanonenitrile (2.7 %), benzoic acid (2.7 %), ethyl ester of dodecanoic acid (6.1 %), butyl ester of 5-oxohexanethioic acid (2.5 %), caryophyllene oxide (6.8 %). The identity of components of essential oil and petroleum ether extract was confirmed on the basis of retention time, mass and supplemented library of NIST, USA. The essential oil and petroleum ether extract constituents are identified for the first time in Korean *C. deodara* pine needle leaves.

Keywords: *Cedrus deodara*, Pinaceae, Essential oil and petroleum ether extract composition, Major and minor components.

INTRODUCTION

Cedrus deodara (Pinaceae family, common name: Himalayan cedar) is widely distributed in the world. The wood oil called Himalayan cedar wood oil or deodar cedarwood oil was once locally used in medicine and is now used as a raw material wood oil have been studied[1,2]. It is renowned because of its ornamental property and also because that its leaves (called pine needle) are widely used for flavoring foods, beverages and many medicinal purposes. It is important to note that many species of pine needles have been processed to many kinds of manufactured products including pine needles powders, pine needles wine and pine needles tea[3]. For centuries throughout eastern Asia, various part of the pine tree, such as pine needles, pine cones, pine cortices and pine pollen have been widely used for promoting health as folk medicine or as food[4,5].

Natural products of plant origin are still a major part of traditional medicinal system in developing countries. There is also a resurgence of interest in herbal medicine in Western countries as an alternative source of drugs often for in traceable diseases such as rheumatoid arthritis[6,7]. Since the volatile oil is one of the important constituents of the wood of *C. deodara*[6], it was used for the pharmacological investigations[7]. There are a lot of reports on the essential oil composition, larvicidal and antimicrobial activities essential oil and plant based extracts, which has a potential application both in the food and in medicinal industries[2,8]. There are several studies reporting the pharmacological and phytochemical studies on *C. deodara*. Spasmolytic activity[9], antioxidant activity[10], anticancer activity[11] have been attributed to extracts from the barks of the plants. Chemical investigations of *C. deodara* have revealed the presence of sesquiterpenes[12], glycosides[13] and phenols[14,15].

There are few reports concerning essential oil composition of leaves, in which the identifies of several mono- and sesquiterpenes[1,7,8], of pine needles leaves of *C. deodara* other than Korea. Nevertheless, no information is available on the essential oil composition of *C. deodara* pine needles of Korea. In the present work, we have investigated for the first time the essential oil and petroleum ether extract composition of *C. deodara*. The main aim of this study was therefore to determine the constituents of the essential oil and petroleum ether extract of *C. deodara* leaves growing in Korea.

EXPERIMENTAL

Fresh leaves (pine needles) of *C. deodara* were collected from Gwangjin-gu region of Konkuk University campus in Seoul, Korea in March 2011. The plant was identified by botanist and voucher specimen (CD-PNL-2011) has been deposited in the herbarium of the Department of Applied Life Science, Konkuk University.

Extraction of essential oil: Fresh leaves pine needles *C. deodara* (500 g) was subjected to hydrodistilled in Clevenger apparatus for 5-6 h to give yellow essential oil. The resulting essential oil was obtained in a yield of 0.20 % w/w after drying over anhydrous sodium sulphate, which was stored at 4 °C until analysis.

Gas chromatography-mass spectrometry analysis of essential oil: Samples of essential oil were diluted in hexane (spectroscopic grade) and analyzed in a Finnigan Focus GC/ Finnigan Focus DSQ MS system (Thermo Co., Germany) apparatus equipped with VB-WAX bonded9 PEG capillary column (30 m × 0.25 mm internal diameter, 0.25 μm film thickness). Helium (1 mL/min) was used as a carrier gas. Sample volume was injected in the split mode 10 μL (split less). The injector was kept at 150 °C. The column was maintained at 50 °C for 10 min and then programmed to 200 at 2 °C and held for 0.5 h at 200 °C. Detector temperature was held at 250 °C. The MS was operated in EI mode at 70 eV in the *m/z* range 25-350.

The identification of the compounds was performed by matching their recorded mass spectra of the GC-MS data system. Quantitative data were obtained from electronic integration peak areas and comparing their retention time and mass spectra library with those found in the literature and supplemented by the Wiley (Wiley 7th mass spectral library) & NIST MS Search 2 (National Institute of Standards and Technology) GC-MS libraries. The essential oil composition of *C. deodara* is summarized in Table-1.

Extraction of petroleum ether extract: The fresh leaves of *C. deodara* (100 g) after drying in oven at 35-40 °C for 4 h, after crushing immersed in petroleum ether (500 mL, 35-60 °C) for overnight at room temperature and then the supernatant

TABLE-1
CHEMICAL COMPOSITION OF THE ESSENTIAL OIL OF *Cedrus deodara leaves*

Retention time	Compound	Percentage
4.50	Butyl acetate	0.8
5.11	α-Pinene	2.9
7.30	α-Myrcene	9.9
8.41	dl-Limonene	3.9
8.71	α-Thujene	0.9
13.24	α-Terpinolene	1.9
32.86	*trans*-Caryophyllene	4.2
33.56	3-Cyclohexen-1-ol 4-methyl-1-(1-methylethyl)	1.1
36.69	α-Humulene	3.4
38.28	Napthalene 1,2,3,4,4α,5,6,8α-octahydro-7-methyl-4-methylene-1-(1-methylethyl)	2.4
39.80	Linalyl propionate	10.9
40.23	Napthalene 1,2,4α,5,6,8α-hexahydro-4,7-dimethyl-1-(1-methylethyl)	1.9
41.59	Napthalene 1,2,3,4, 4α,5,6,8α-octahydro-7-methyl-4-methylene-1-(1-methylethyl)	0.9
41.92	δ-Cadinene	2.7
44.07	Myrtenol	0.9
47.37	Benzenemethanol 4-(1-methylethyl)-	0.8
49.47	α-Calacorene	0.9
52.34	Caryophyllene oxide	2.8
53.94	1-Dodecanol	2.9
54.93	12-Oxabicyclo dodeca3,7-diene 1, 5, 5, 8-tetramethyl-	0.7
56.05	1-Napthalenol decahydro-4α-methyl-8-methylene-2-(1methylethyl)-	1.7
56.21	Caryophyllenyl alcohol	0.6
59.78	2-(4α, 8-Dimethyl-1, 2, 3, 4, 4α, 5, 6, 7-octahydronapthalen-2-yl)-prop-2-en--ol	0.8
60.90	Napthalene 2,3,6-trimethyl	1.5
62.13	t-Cadinol	1.5
62.88	α-Cadinol	3.9
63.48	1-Napthalenol 1,2,3,4,4α, 7,8,8α-octahydro-1,6-dimethyl-4-(1-methylethyl)-	1.5
64.35	1,6,10,14-Hexadecatetraen-3-ol 3,7,11,15-tetramethyl	1.5
64.92	t-Muurolol	4.4
66.09	Cembrene	1.2
67.29	Ledene oxide	1.3
67.99	2-Hydroxy-2,4,4-trimethyl-3-(3-methylbuta-1,3-dienyl)cyclohexanone	0.9
69.09	Isoaromadendrene expoxide	1.0
70.77	Alloaromadendrene oxide	1.2
77.01	Dodecanoic acid	12.9
81.52	Phytol	1.2
84.96	Tetradecanoic acid	1.8
95.47	Hexadecanoic acid	1.8

was concentrated under vacuum to yield (1.25 g) of the extract, which was small sample dissolved in hexane (spectroscopic grade) and prepare sample after filtration for GC-MS analysis.

Gas chromatography-Mass spectrometry analysis of petroleum ether extract: Samples of petroleum ether extract were diluted in hexane (spectroscopic grade) and analyzed in a Finnigan Focus GC/Finnigan Focus DSQ MS system (Thermo Co., Germany) apparatus equipped with Vesteck rtx-50 capillary column (30 m × 0.25 mm internal diameter, 0.25 µm film thickness). The other conditions are same as in case of essential oil. The petroleum ether extract composition of *C. deodara* is summarized in Table-2.

RESULTS AND DISCUSSION

Chemical constituents of the essential oil: The yellow essential oil of *C. deodara* was obtained in a 0.20 % yield. The constituents identified by GC-MS analysis in order of elution of VB-WAX bonded capillary column are presented in Table-1. The major components were identified as: α-pinene (2.9 %), α-myrcene (9.9 %), dl-limonene (3.9 %), *trans*-caryophyllene (4.2 %), α-humulene (3.4 %), linalyl propionate (10.9 %), δ-cadinene (2.7 %), caryophyllene oxide (2.7 %), 1-dodecanol (2.9 %), α-cadinol (3.9 %), *t*-muurolol (4.4 %), dodecanoic acid (12.9 %) in essential oil. The oil was dominated by variety of monoterpenes, sesquiterpenes and acids. However, the comparison of our results with literature shows some qualitative and quantitative differences in the composition of *C. deodara*[1] and *C. atlantica*[16] leaf oil. So far, no reports are availabvle on essential oil composition of *C. deodara* leaves of Korea. To our best of knowledge, this is the first report of *C. deodara* leaves oil. The identification of the compounds was performed by matching their recorded mass spectra of the GC-MS data system. Quantitative data were obtained from electronic integration peak areas and comparing their retention time and mass spectra library with those found in the literature and supplemented library. Other methods of identification and took help of by comparing mass data with data of library[17,18].

TABLE-2
CHEMICAL COMPOSITION OF THE PETROLEUM ETHER EXTRACT OF *Cedrus deodara leaves*

Retention time	Compound	Percentage
3.80	Butyl acetate	33.9
3.97	2-Methyl-4-nonanol	0.52
4.08	Hydroperoxide 1-methylbutyl	0.9
4.36	α-Pinene	0.6
4.49	Penta-en-2-ol 4-allyloxy-2-methyl	6.2
4.68	2,2-Dimethyl pentanal	2.4
4.91	α-Pinene	2.9
5.14	7,7-Dimethyl-tetracyclo heptane	1.4
5.41	2-Methyl-5-phenyl-5-pentanonenitrile	2.7
6.17	Undecanal	0.5
6.27	2-Butoxyethyl acetate	0.5
6.65	Octanoic acid	0.9
6.80	[1S-(1α,2α,3α)]-Bicyclo hept-3-ene-2-ol 4,6,6-trimethyl	1.8
7.31	Benzoic acid	2.7
7.52	Bicyclo hept-2-ene-2-carboxaldehyde 6,6-dimethyl	0.8
7.73	Bicyclo heptan-2-ol 1,7,7-trimethyl acetate	1.2
7.97	α-Copaene	1.9
8.12	2-Hexadecen-1-ol 3,7,11,15-tetramethyl	1.6
8.25	*trans*-2-Undecenoic acid	0.7
8.53	1,4-Methanoazulene decahydro 4,8,8-trimethyl-9-methylene	1.2
8.94	Hexadecane	0.9
9.16	Naphthalene 1, 2, 4α, 5, 6, 8α -hexahydro-4,7-dimethyl-1-(1-methylethyl)-	1.0
9.29	Dodecanoic acid methyl ester	0.5
9.42	α-Muurolene	1.4
9.92	Dodecanoic acid ethyl ester	6.0
10.64	5-Oxohexanethioic acid butyl ester	2.5
11.08	1,3,5-Triazine-2,4-diamine 6-chloro-N-ethyl	1.6
11.21	Caryophyllene oxide	6.8
11.39	4,6,6-Trimethyl-2-(3-methylbuta-1,3,-dienyl)-3-	1.6
11.68	12-Oxabicyclo dodeca-3,7-diene 1,5,5,8-tetramethyl	1.1
12.09	Tetracyclo tridecane-9-ol 4,4-dimethyl	1.9
12.41	Octadecane 1-chloro	1.2
12.54	Isoaromadendrene epoxide	0.8
13.03	4α, α′-Methyl-3,4,4α, 5,6,7,8αα′-hexahydronaphthalen-1-(2H)7 (8H)-dione	0.9
13.94	Docosane	0.6
15.70	Dodecanoic acid hex-3-enyl ester	1.4
17.41	Limonen-6-ol, pivalate	1.4
19.23	Heptacosane	1.2
19.55	1-Cyclohexene-1-carboxylic acid 4-(1,5-dimethyl-3-oxohexyl)-methyl ester	0.9
20.76	7 α-Isopropyl-4,5-dimethyloctahydroinden-4-yl) methanol	1.2

Chemical constituents of petroleum ether extract: The constituents identified by GC-MS analysis in order of elution of Vesteck rtf-50 capillary column are presented in Table-2. The major components were identified as: butyl acetate (33.9 %), 4-allyloxy-2-methyl penta-en-2-ol (6.2 %), 2,2-dimethyl pentanal (2.4 %), α-pinene (2.9 %), 2-methyl-5-phenyl-5-pentanonenitrile (2.7 %), benzoic acid (2.7 %), ethyl ester of dodecanoic acid (6 %), butyl ester of 5-oxohexanethioic acid (2.5 %), caryophyllene oxide (6.8 %), respectively in petroleum ether extract. The oil was dominated by esters, acids and terpenes. The identification of compounds is same as in essential oils. To our best of knowledge this is the first report of identified components in petroleum ether extract of *C. deodara* leaves.

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