

## Comparison of Volatile Components in Essential Oil from Different Origin of *Atractylodes* spp.

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**ABSTRACT** : This study was carried out to compare the major volatile components in essential oil from different origin of *Atractylodes* spp. which is being traded as a crude herbal drug in Korean herbal markets. From the two *Atractylodes* of major volatile components were similarly detected such as the  $\beta$ -selinene,  $\beta$ -sesquiphellandrene, germacrene B, 2,7-dimethoxy-2-methylnaphthalene and 9-methoxy-2,3-dihydrofuro{3,2-q}coumarin. Among the volatile components, the major components were 2,7-dimethoxy-2-methylnaphthalene (40.98%), 9-methoxy-2,3-dihydrofuro {3,2-q} coumarin (15.74%), and  $\beta$ -sesquiphellandrene (1.98%) in both *Atractylodes*. As a results, It was found that the two *Atractylodes* were the same species which was being traded in the Korean herbal markets as the *A. japonica*, not to different species of *A. japonica* and *A. macrocephalla*, respectively.

**Key words** : *Atractylodes*, volatile components,  $\beta$ -sesquiphellandrene, 2,7-dimethoxy-2-methylnaphthalene, ethoxy-2,3-dihydrofuro{3,2-q}coumarin

### INTRODUCTION

*Atractylodes* are a perennial herbaceous plant belongs to the Compositae family and most found in mountain regions of Eastern asian countries. *Atractylodes* species of *A. japonica* is distributed in Korea and Japan but *A. macrocephalla* is found in China. *Atractylodes* are known as a Chinese crude herbal drug and are classified into two types, "Backchul" and "Changchul" in Korea. Backchul is the rhizome of *A. japonica* and *A. macrocephalla*. Changchul is the rhizome of *A. lancea*. (The 8th Korean Pharmacopeia, 2002). The Chinese pharmacopoeia, Backchul belongs to *A. macrocephalla* which is known from the same origin of *A. ovata* while Changchul is defined as different origin of *A.*

*lancea*, and *A. chinensis* (Kim *et al.*, 1998). *A. japonica* and *A. macrocephala* contained volatile components in essential oils and the major volatile components are sesquiterpenoids such as atractylon, atractylenolide (I, II, III), 3 $\beta$ -hydroxyatractylone, furaldehyde and 3 $\beta$ -acetoxyatractylon but the content levels are different between the two species (Bae, 2001; Takeda *et al.*, 1994). Rhizome of *Atractylodes* has been used for promoting digestion and protects stomach (Bae, 2001; Kim *et al.*, 1998; Hwang *et al.*, 1996). *A. japonica* and *A. macrocephala* have been used for various medical properties such as stimulate the sweating and preventing miscarriage. Consumption of *Atractylodes* has highly been increased for decades in the Korean crude herbal markets. The cultivation of *Atractylodes* was introduced into Korea

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in 1990s but it was not successful because of its inadaptability by different ecological conditions and severe infection of root rot disease (Cho *et al.*, 2001). Atractylodes of the two different origin can be identified by morphological figures such as the leaf form, flower color, and rhizome shape but it is impossible to distinguish the two species when the rhizome sliced to sell in the Korean herbal markets. Imported *A. macrocephala* is less valuable in Korea than *A. japonica* in the view of effectiveness. Recently, *A. macrocephala* ranked as the 2nd largest imported foreign crude herbal drug in Korea followed by licorice. *A. japonica* is being traded at 10 times higher price than *A. macrocephala* in the domestic herbal markets and *A. macrocephala* is being illegally traded either without attaching the origin label or mixed with the *A. japonica*. Vogue origin of atractylodes caused a big social problem and disordered crude herbal market functions in Korea, so discrimination of the two different origin of atrtylodes species is very important and imminent project for fair trading in Korea and for protecting Korean farmers. The aim of this study is to find out the differences between the two different origin of atractylodes species by comparing the major volatile components in essential oil of *A. japonica* and *A. macrocephala*.

## MATERIALS AND METHODS

### Plant materials

*A. japonica* was collected in the experimental field of the Division of Ginseng & Medicinal Crops, National Institute of Crop Science, RDA as the control in 2003. *A. macrocephala* was collected in the local herbal markets of Euseong (Kyungbuk), and Keumsan (Chungnam) where the Chinese origin.

### Oil isolation

Essential oils were extracted by simultaneous distillation and extraction method (SDE) for two hours using a clevenger apparatus. The distilled essential oils were collected and dried over dehydrate (sodium sulfate) and stored at 4°C. The yield of colorless oils were 0.06~0.30% (w/w). The essential oil was kept at 4°C until use.

### GC/MS analysis

The analysis was carried out at French–Korean Aromatics R&D Center, Yongin Korea, using a Hewlett Packard 6890/HP 5973 GC/MS system operating in EI mode at 70 eV, equipped with a HP–Innowax capillary column (50 m×0.32 mm; film thickness 0.5 µm). The initial temperature of the oven was 70°C and subsequently raised to 230°C with 3°C/min. rate. Helium was used as the carrier gas at the rate of 1.3 ml min.

### Identification

Chemical constituents were identified by comparing of their relative retention times and mass spectrum with those obtained from authentic samples and Willey library in computer of National Institute of Crop Science (NICS).

## RESULTS AND DISCUSSION

The major volatile components in essential oil from different origin of atractylodes showed interesting results between the two different species (Table 1 & Fig. 1.). Several terpenoides were detected such as the  $\beta$ -selinene (0.88%),  $\beta$ -sesquiterphellandrene (0.86%), germacrene B (1.53%), valencene (4.24%), 2,7-dimethoxy-3-6-dimethylnaphthalene (21.67%), and 9-methoxy-2,3-dihydrofuro {3,2-q} coumarin (17.39%) from the *A. japonica*. But in case of *A. macrocephala* only 2,7-dimethoxy-3-6-dimethylnaphthalene (27.02%) and 4H-Furl {3,2-c} {1} benzopyran-4-one (18.37%) were detected as the major components from the cultivation of NICS. *A. macrocephala* was introduced into National Institute of Crop Science, RDA, Korea in mid 1990s and it had required long time to adapt Korean ecological conditions. However, we know that it has still got a genetic heritage of quality variations without changing on it. When certain species moved from habitat to different ecological conditions the quality heritage was changed but after obtaining proper habitat conditions, the quality heritage changed with original figures (Hornok *et al.*, 1993). So we assumed that *A. macrocephala*'s ecological conditions of China are similar to those of

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Korea, especially Suwon region. In order to find the differences of volatile components in essential oil of *Atractylodes* from Euseong, and Keumsan herbal markets, showed the same volatile components in both regions which means that we assumed the same species were being traded as *A. japonica* (Table 2~3, Fig. 2~3). Volatile components, from *A. japonica* were over two times than *A. macrocephala* such as berkheyaradulen (1.23, 0.72%),  $\alpha$ -selinene (1.21, 0.73%),  $\beta$ -caryophyllene (1.33, 0.79%),  $\alpha$ -Humulene (0.77, 0.49%), and  $\beta$ -sesquiphellandrene (1.98, 0.40%). The major compounds of *A. japonica* were 2,4-dicyclohexyl-1-1-buten-3-yne (41.11%) and  $\beta$ -sesquiphellandrene (1.69%). This result showed that the *Atractylodes* of Euseong and Keumsan were being traded the same species as the *A. japonica*, not two different origin of *Atractylodes* compare to the result of control components (Table 1). *A. japonica*

showed little higher volatile components contents than *A. macrocephala* such as  $\alpha$ -selinene (0.73, 0.44%),  $\beta$ -selinene (1.94, 1.56%)  $\beta$ -caryophyllene (0.58, 0.40%),  $\alpha$ -Humulene (0.57, 0.44%),  $\beta$ -sesquiphellandrene (1.98, 0.40%) and Gamacrene B (4.25, 3.36%) (Table 3). The major volatile components were 2,7-dimethoxy-2-methylnaphthalene (38.00, 40.98%) and 9-methoxy-2,3-dihydrofuro{3,2-q} coumarin (8.82, 15.74%) in both species. This result showed that the same species as *A. japonica* were being traded in the Euseong and Keumsan herbal markets. The same components were also detected as the  $\beta$ -selinene,  $\beta$ -sesquiphellandrene, Germacrene B, 2,7-dimethoxy-2-methylnaphthalene and 9-methoxy-2,3-dihydrofuro {3,2-q} coumarin. As above results, it was assumed that either the *A. japonica* or mixed the *A. japonica* with *A. macrocephala* have been traded as *Atractylodes* in the Korean Herbal markets.

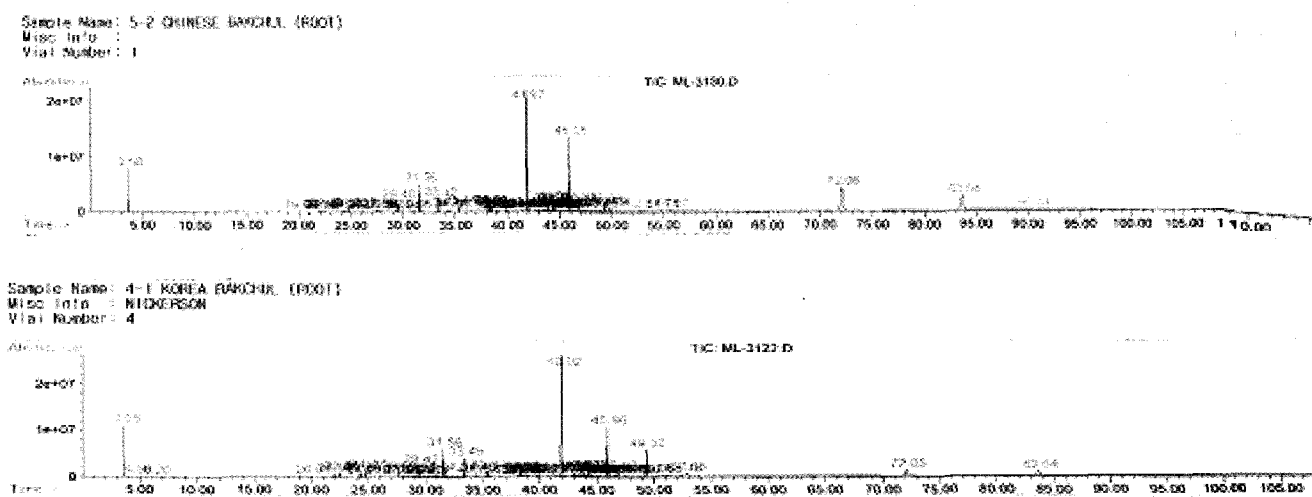


Fig. 1. Chromatography of volatile components in essential oil from different origin of *A. macrocephala* (above) and *A. japonica* (below) cultivated in NICS.

Table 1. Identification of the volatile components in essential oil from different origin of *Atractylodes* cultivated in NICS.

<i>A. japonica</i>			<i>A. macrocephala</i>		
R.T	Components	(%)	R.T	Components	(%)
29.41	$\beta$ -selinene	0.88	-	-	-
30.96	$\beta$ -sesquiphellandrene	0.86	-	-	-
31.57	Valencene	4.24	-	-	-
33.43	Germacrene B	1.53	-	-	-
41.96	2,7-dimethoxy-3,6-dimethylnaphthalene	21.67	41.97	2,7-dimethoxy-3,6-dimethylnaphthalene	27.02
45.98	9-methoxy-2,3-dihydrofuro{3,2-q}coumarin	17.39	72.22	4H-Fur{3,2-c}1}benzopyran-4-one	18.37

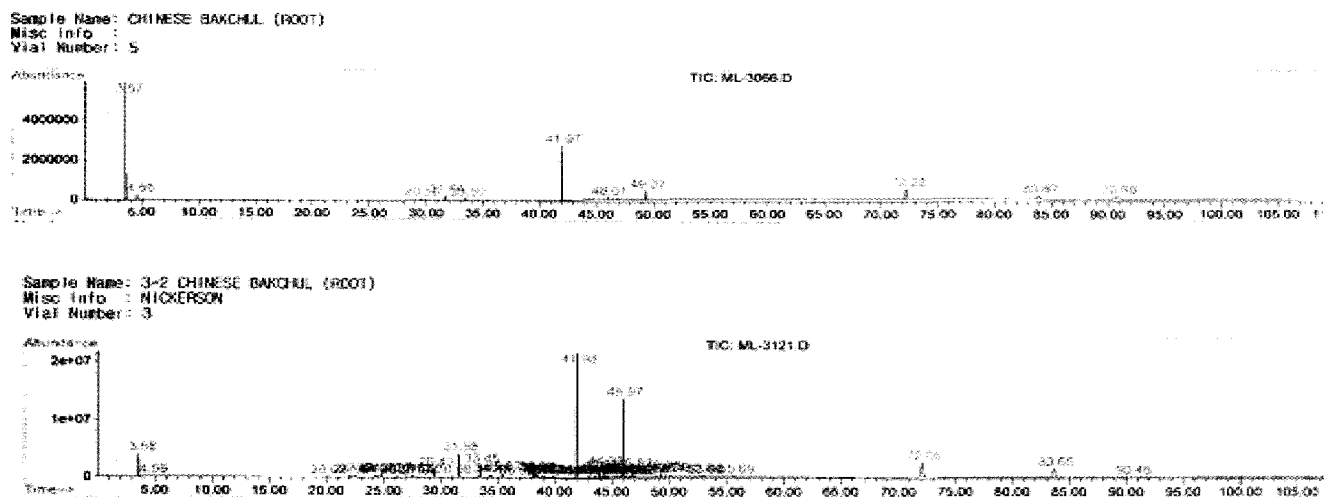


Fig. 2. Chromatography of volatile components in essential oil of *A. japonica* (above) and *A. macrocephala* (below) collected in Euseong herbal market.

Table 2. Identification of the volatile components in essential oils of atractylodes collected in Euseong herbal market.

<i>A. japonica</i>			<i>A. macrocephala</i>		
R.T	Components	(%)	R.T	Components	(%)
22.45	Berkheyaradulen	1.23	22.47	Berkheyaradulen	0.72
24.27	$\alpha$ -selinene	1.21	24.28	$\alpha$ -selinene	0.73
24.77	$\beta$ -caryophyllene	1.33	24.78	$\beta$ -caryophyllene	0.79
27.52	$\alpha$ -Humulene	0.77	27.53	$\alpha$ -Humulene	0.49
29.42	$\beta$ -sesquiphellandrene	1.98	29.43	$\beta$ -selinene	1.69
30.97	$\beta$ -sesquiphellandrene	0.72	30.98	$\beta$ -sesquiphellandrene	0.40
42.02	2,4-dicyclohexy1-1-buten-3-yne	41.11	-	-	-

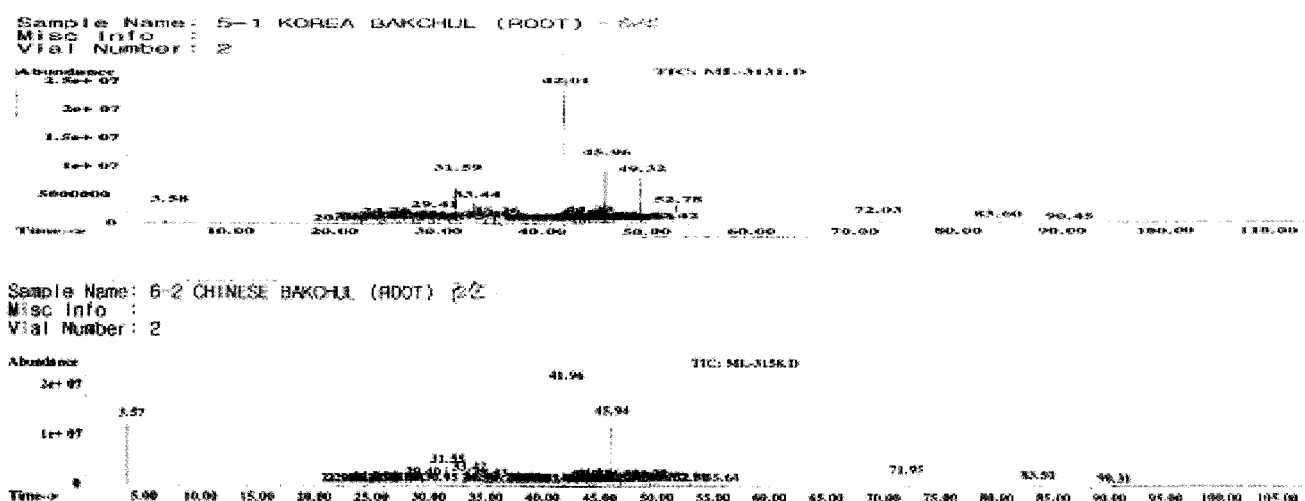


Fig. 3. Chromatography of volatile components in essential oil of *A. japonica* (above) and *A. macrocephala* (below) collected in Keumsan herbal market.

**Table 3.** Identification of the volatile components in essential oils of *atractylodes* collected in Keumsan herbal market.

<i>A. japonica</i>			<i>A. macrocephala</i>		
R.T	Components	(%)	R.T	Components	(%)
24.26	$\alpha$ -selinene	0.73	24.26	$\alpha$ -selinene	0.44
24.76	Trans( $\beta$ )-Caryophyllene	1.22	24.76	Trans-Caryophyllene	0.70
26.09	Gamma-elemene	0.54	26.09	Gamma-elemene	0.16
27.51	$\alpha$ -Humulene	0.57	27.50	$\alpha$ -Humulene	0.44
27.95	Gamma-curcumene	0.34	27.60	$\beta$ -Selinene	0.28
29.41	$\beta$ -Selinene	1.94	9.40	$\beta$ -Selinene	1.56
30.95	$\beta$ -Sesquiphellandrene	0.58	30.95	$\beta$ -Sesquiphellandrene	0.40
33.44	Garmacrene B	4.25	33.42	Garmacrene B	3.36
35.35	9,10-Dehydro-isolongifolene	1.10	35.33	9,10-Dehydro-isolongifolene	1.22
38.33	Caryophyllene oxide	0.28	38.30	Caryophyllene oxide	0.44
42.01	2,7-dimethoxy-3,6-dimethylnaphthalene	38.00	41.96	2,7-dimethoxy-3,6-dimethylnaphthalene	40.98
44.33	$\alpha$ -eudesmol	1.11	44.31	$\alpha$ -eudesmol	1.29
45.96	3-dihydrofuro(3,2-q)coumarin	8.82	45.94	9-Methoxy-2,3-dihydrofuro(3,2-q)coumarin	15.74
49.32	3,5-dimethoxy-2-methylnaphthalene	7.79	49.70	3,5-dimethoxy-2-methylnaphthalene	0.47
72.03	5-dimethylcyclohex-2ene	3.41	83.50	5-dimethylcyclohex-2ene	3.39
83.60	Isolantolactonoid butenolide A.	1.73	90.31	Isolantolactonoid butenolide A.	0.59

This study showed a similar result with Seong *et al.* (2003). Who discriminated the angelica species which was imported from China by morphological figures with microtom assay and develop to distinguish method the evulation the inner part of angelica specise. Comparing of the volatile components in essential oil is also one of useful method to distinguish the different origin of *atractylodes* species in Korea for fair trading in domestic crude herbal markets and protecting the Korean farmers who has cultivated the *atractylodes*.

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