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# Characteristics of Volatile Components from *Magnolia ovobata* Thunb. by Different Analysis Methods

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**ABSTRACT** : This study was carried out to establish an optimum method for identifying the volatile components of *Magnolia ovobata* Thunb. using the dynamic headspace (Purge & Trap) and simultaneous distillation and extraction (SDE) method. Between the two different identification analysis, the volatile components were more easily detected in the SDE than the Purge & Trap method. Among the identified volatile components, the 12 compounds were detected to have similar retention times and match quality within the 45 minutes in both identification methods. The maximum values of the major volatile components were detected differently by SDE and (Purge & Trap) method such as α-pinene (3.4, 18.2%), β-pinene (3.5, 10.3%), l-limonene (5.2, 15.4%). These results indicated that the Dynamic Headspace (Purge & Trap) was much more reliable method for identifying the volatile components of *Magnolia ovobata* Thunb. as compared to the SDE method.

***Key words*** : *Magnolia ovobata*, volatile components, α-pinene, β-pinene, limonene, β-caryophyllene

## INTRODUCTION

*Magnolia ovobata* Thunb. is a deciduous ornamental tree and is well grown in the central region of Korean peninsula (Bae, 2001). It was introduced from Japan in 1920s as a name of "hubak" and has been miscalled ever. A deciduous large tree, up to 22 m high has a smooth light rust-ash color and aromatic bark. The creamy-white flower blooms in May and emits a strong odoriferous scent. Petal numbers from 9 to 15, with the outer 3 being pale-green tinged pink on the outside, 6~12 cm wide, 8~10 cm log, which is 3 cm longer than inner part.

The red purple fruits ripen in autumn and it gives off sweet smells just like fresh baked bread. The fruit corns are 10~12 cm long, oblong, apex truncate and rounded base. Its pericarp has some several useful fatty acids, namely, palmitic (20.4%), steric (1.3%), oleic (53.3%), linoleic (20.4%), and linolenic (2.6%) (Anonymous, 2002). The quantity and quality of fatty acid composition is similar to peanut cultivar "Iksan 17" which contained palmitic (10.1%), steric (2.9%), oleic (53%), linoleic (29.3%), and linolenic acid (1.0%). The bark, consists of rough, thick and tight cylinders, has an aromatic taste and strong pungent smell. It is often used as a drug in Koran folk medicine. Specially, in the southern region of the country, the bark has been used for chest and abdominal pains, indigestion, diarrhea, cough, and sputum (Bae, 2001). Mass spectra are widely used in order to identify the peaks resulting from a chromatographic separation. The most common and available approach for knowing its structural information is the reference mass spectra database

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(Kang *et al.*, 1998). This study was investigated the volatile components from its aromatic flowers with the different two analysis methods, the dynamic headspace (here after called purge trap) and simultaneous distillation and extraction (SDE) method, to find the preferable isolation method in further studying the fragrancy of volatile components of natural resource plants.

## MATERIALS AND METHODS

### Plant materials

Sample was originally collected in the medicinal plant garden of the Division of Genseng & Medicinal Crop, National Institute of Crop Science, RDA during full flowering period in may 2002. Sample were dried under natural conditions and kept 4℃ refrigerator until use.

### Oil isolation

Air dried were subjected to simultaneous distillation and extraction method (SDE) for two hours using a clevenger apparatus, The distilled essential oil was collected and dried over dehydrate (sodium sulfate) and stored at 4℃, the colorless Oil yield was 0. 4% (w/w).

### GC/MS analysis

The analysis was carried out using a Hewlett Packard 5890 GC/MS system operating in El mode at 70 eV, equipped with a DB-5 capillary column (30 m ×0.25 ㎜; film thickness 0.25 ㎛). The initial temperature of the oven was 50℃ and subsequently raised to 230℃ with 3℃/min. rate. Helium was used as the carrier gas at the rate of 1 me $\text{min}^{-1}$.

**Table 1.** Conditions of Gas Chromtography/Mass spectrometry for analyzing the volatile components of *Magnolia ovobata* Thunb.

| | |
|---|---|
| Instrument | HP 5890 GC/HP 5973 MSD |
| Column | DB-5 (crossed linked methyl siloxan, 30 m× 0.25 ㎝×0.33 ㎛) |
| Inject temp | 250℃ |
| Oven temp | 50℃ (3 min.)→ 8℃/min.→ 230℃ (3 min.) |
| Auxiliary temp | 230℃ |
| Carrier gas (flow rate) | He(0.8 ㎖/min.) |
| Split ratio | 1:10 |
| Ionization mode | Electric impact (70 eV) |
| Ms source temp | 230℃ |

### Identification

Chemical constituents was identified by comparing of their relative retention times and mass spectrum with those obtained from authentic samples and Willey library.

## RESULTS AND DISCUSSION

The two different analysis methods, the Purge & Trap and simultaneous distillation and extraction (SDE) method were used. The 43 volatile components were identified on the SDE method but 25 compounds were detected on the Purge & Trap instrument (Table 2, 3). Usually, volatile components of plants can be detested during the retention time from 10 to 50 minutes. This result showed slight differences as the α-Pinene and nonadecane were 9.80 and 94.94, respectively, which were not common at retention times observed in plants. A total of 43 volatile components were detected the SDE method where 12 compounds were selected as the major components, which were compared with the Purge & Trap method. The volatile components of the SDE method were mostly monoterpene and sesquiterpene derivatives. α-Pinene (3.4%), β-pinene (3.6%), α-terpineol (2.3%), myrcene (0.7%), limonene (5.2%), camphene (2.1%), bornyl acetate (5.3%), were the monoterpene. β-Caryophyllene (4.4%) and caryophyllene oxide (4.6%) were detected as the sesquiterpene derivatives. The compounds with high content were bornyl acetate (5.3%), limonene (5.2%), bornyl (5.1%), β-Caryophyllene (4.4%), β-asarone (4.6%), and T-muurolol (3.8%), Among these components, limonene which has been well-known for anticancer and β-asarone for its antifungal effects, were the concerned components for further studying. The volatile components identified using of the Purge & Trap were also mostly mono-and sesquiterpene derivatives. The content of most monoterpene was higher more than two times as compared to the content identified using SDE. Especially, the content of α-pinene (18.2%), β-pinene (10.3%), myrcene (2.5%), limonene (15.4%) and camphene (6.8%) were higher

**Table 2.** Identification of the volatile components in essential oil of *Magnolia ovobata* Thunb. using GC/MS.

| Peak no. | Retention time | Volatile components | Peak | Match (%) | Contents (%) |
|---|---|---|---|---|---|
| 1 | 9.80 | α-pinene | 162 | 97 | 3.43 |
| 2 | 10.85 | camphene | 98 | 98 | 2.08 |
| 3 | 13.21 | β-pinene | 172 | 97 | 3.55 |
| 4 | 14.49 | mycrene | 3 | 97 | 0.06 |
| 5 | 17.12 | benzene | 48 | 96 | 1.01 |
| 6 | 17.75 | limonene | 247 | 95 | 5.21 |
| 7 | 20.85 | *trans*-sabinene hydrate | 59 | 98 | 1.25 |
| 8 | 23.52 | cis-sabinene | 5 | 98 | 0.16 |
| 9 | 24.00 | linalool | 132 | 96 | 2.74 |
| 10 | 25.21 | 2-cyclohexen | 1 | 97 | 0.03 |
| 11 | 27.03 | l-camphene | 113 | 98 | 2.37 |
| 12 | 29.18 | 1-borneol | 243 | 91 | 5.14 |
| 13 | 29.95 | terpinen-4-ol | 92 | 97 | 1.94 |
| 14 | 31.06 | β-terpineol | 106 | 91 | 2.25 |
| 15 | 35.96 | gerany acetate | 80 | 83 | 1.69 |
| 16 | 36.40 | chavicol | 150 | 98 | 3.09 |
| 17 | 38.33 | bornyl acetate | 251 | 99 | 5.33 |
| 18 | 44.78 | geranyl acetate | 26 | 91 | 0.55 |
| 19 | 47.00 | β-caryophyllene | 205 | 99 | 4.36 |
| 20 | 49.00 | β-selinene | 88 | 98 | 1.86 |
| 21 | 51.57 | α-selinene | 73 | 98 | 1.54 |
| 22 | 51.90 | oxetane | 6 | 91 | 0.13 |
| 23 | 53.37 | calacorene | 53 | 46 | 1.13 |
| 24 | 54.31 | caryophyllene oxide | 14 | 80 | 0.29 |
| 25 | 56.85 | humulene epoxide | 221 | 81 | 4.68 |
| 26 | 58.21 | β-asarone | 45 | 81 | 0.93 |
| 27 | 59.54 | bicyclo(4.4.0)dec-1-en | 219 | 99 | 4.61 |
| 28 | 60.12 | t-muurolol | 67 | 95 | 1.41 |
| 29 | 61.08 | 5-oxatric | 181 | 96 | 3.79 |
| 30 | 62.27 | nonadecene | 678 | 70 | 1.40 |
| 31 | 73.67 | palmitic acid | 14 | 97 | 0.28 |
| 32 | 77.05 | eicosane | 38 | 99 | 0.77 |
| 33 | 78.38 | *trans*-2-nonadecane | 20 | 98 | 0.38 |
| 34 | 79.78 | 10-heneicosane | 17 | 83 | 0.32 |
| 35 | 81.68 | 11-hydroxy-7.8.9.10-tetrahydrobebzo(b)xa | 9 | 99 | 0.19 |
| 36 | 82.22 | heneicosane | 15 | 83 | 0.31 |
| 37 | 83.11 | linoleic acid | 110 | 97 | 2.32 |
| 38 | 84.60 | magnolol | 18 | 96 | 0.36 |
| 39 | 86.90 | N-docosane | 9 | 95 | 0.16 |
| 40 | 87.87 | *trans*-2-nonadecane | 13 | 97 | 0.27 |
| 41 | 92.28 | cis-2-nonadecane | 4 | 91 | 0.07 |
| 42 | 92.81 | cis-2-nonadecane | 6 | 87 | 0.11 |
| 43 | 94.94 | nonadecane | 135 | 91 | 2.85 |

**Table 3.** Identification of the volatile components from dried flowers of *Magnolia ovobata* Thunb. using the Purge & Trap.

| Peak no. | Retention time | Components | Peak | Match (%) | Contents (%) |
|---|---|---|---|---|---|
| 1 | 7.18 | hexanal | 39 | 90 | 1.06 |
| 2 | 9.08 | flomamidinium acetate | 6 | 74 | 0.14 |
| 3 | 10.04 | 3-hexen-1-ol | 17 | 50 | 0.42 |
| 4 | 13.98 | α-phellandrene | 28 | 90 | 0.78 |
| 5 | 14.39 | α-pinene | 593 | 97 | 18.17 |
| 6 | 15.23 | camphene | 226 | 98 | 6.82 |
| 7 | 17.18 | β-pinene | 339 | 97 | 10.33 |
| 8 | 18.53 | myrcene | 86 | 95 | 2.54 |
| 9 | 19.22 | α-phellandrene | 17 | 86 | 0.41 |
| 10 | 20.11 | α-terpinene | 22 | 98 | 0.58 |
| 11 | 20.70 | benzene, 1-methy1-3-(1-methylethy1) | 137 | 96 | 4.17 |
| 12 | 21.08 | 1-limonene | 502 | 98 | 15.40 |
| 13 | 21.22 | 1,8 cineol | 59 | 95 | 1.70 |
| 14 | 23.25 | $\sqrt{\ }$-terpinene | 29 | 96 | 0.86 |
| 15 | 25.43 | benzene, 1-methy1-4-[1-methylethenyl] | 22 | 98 | 0.63 |
| 16 | 26.57 | α-terpinolene | 80 | 95 | 2.42 |
| 17 | 26.80 | cyclopropyl carbind | 9 | 25 | 0.23 |
| 18 | 29.34 | camphore | 33 | 98 | 0.92 |
| 19 | 31.09 | endo-Borneol | 79 | 90 | 2.38 |
| 20 | 36.73 | carvol | 45 | 95 | 1.31 |
| 21 | 39.81 | bornyl acetate | 40 | 90 | 1.19 |
| 22 | 45.75 | propanoic acid | 26 | 72 | 0.72 |
| 23 | 48.68 | β-caryophyllene | 103 | 99 | 3.13 |
| 24 | 49.88 | *trans*-α-bergamotene | 18 | 90 | 0.50 |
| 25 | 50.82 | α-Humulene | 24 | 97 | 0.71 |
| 26 | 51.36 | *trans*-β-farnesene | 18 | 91 | 0.47 |
| 27 | 55.35 | delta-cardinene | 16 | 95 | 0.42 |

**Table 4.** Differences of the major volatile components content between the two analysis methods in *Magnolia ovobata* Thunb.

| Volatile | Purge & Trap | | | SDE** | | |
|---|---|---|---|---|---|---|
| Components | *R.T | Quality (%) | Content (%) | R.T | Quality (%) | Content (%) |
| α-pinene | 14.39 | 97 | 18.17 | 9.80 | 97 | 3.43 |
| camphene | 15.23 | 98 | 6.82 | 10.85 | 98 | 2.18 |
| β-pinene | 17.18 | 97 | 10.33 | 13.21 | 97 | 3.55 |
| myrcene | 18.53 | 95 | 2.54 | 14.49 | 97 | 0.67 |
| benzene, 1-methylethyl | 20.70 | 96 | 4.17 | 17.12 | 96 | 1.01 |
| 1-limonene | 21.08 | 98 | 15.40 | 17.75 | 95 | 5.21 |
| 1.8 cineol | 21.22 | 95 | 1.70 | -*** | - | - |
| α-terpinolene | 26.57 | 95 | 2.42 | 31.06 | 91 | 2.25 |
| borneol | 31.09 | 90 | 2.38 | 29.18 | 91 | 5.14 |
| carvol | 36.73 | 95 | 1.31 | - | - | - |
| bornyl acetate | 39.81 | 90 | 1.19 | 38.33 | 99 | 5.33 |
| β-caryophyllene | 48.68 | 99 | 3.13 | 47.00 | 99 | 4.36 |

* Retention time. ** Steam distillation method. *** Non-detected.

several times in the Purge & Trap method. However, the content of bornyl acetate (1.2%), was lower three times. comparing the sesquiterpene content of the Purge & Trap to SDE method, β−caryophyllene (3.1%) and −humulene content was less in the Purge & Trap (SDE). The content of α−Pinene (18.2%), limonene (15.4%), β−pinene (10.3%), camphene (6.8%), β−Caryophyllene (3.1%) and myrcene (2.5%) were conspicuously higher in the Purge & Trap. The same components were identified with both Purge & Trap and SDE mehod were α−Pinene, β−pinene, α−terpinene, myrcene, limonene, camphore, bornylacetate and β−Caryophyllene. However, the magnolol compound in bark which is well−known for anti−germs effect (Bae, 2001), was also detected at the retention time 86.90 in the SDE. In the isolation spectrum of chromatography, the separation was shown more clearly and exactly in the Purge & Trap than the SDE method. Meanwhile, the volatile components were easily isolated in the SDE method

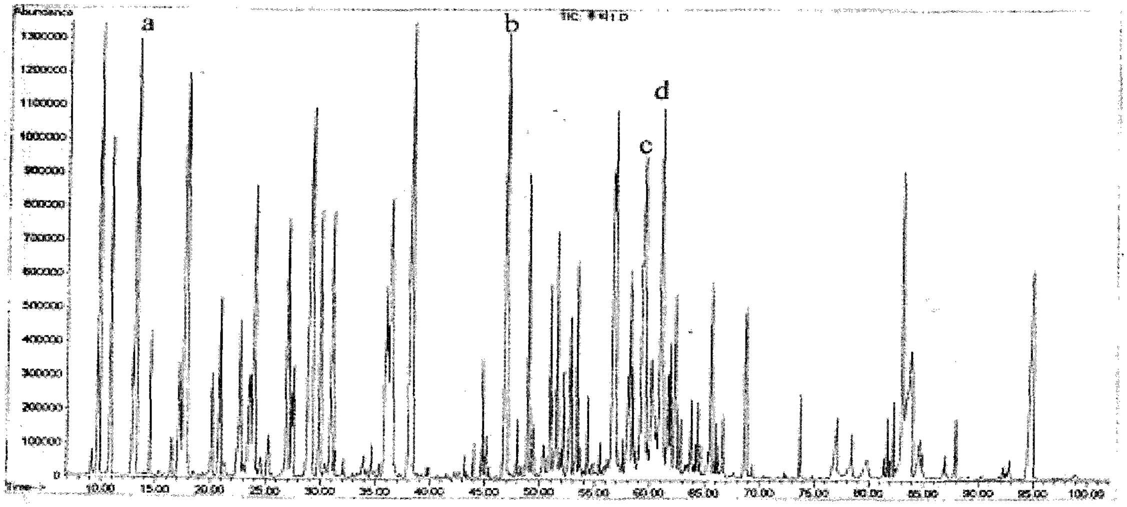

**Fig. 1.** Total ion chromatogram spectrum of GC/MS in essential oil of *Magnolia ovobata* thunb. (a: β-pinene, b: β-cary ophyllene, c: β-asarone, d:t-muurolol).

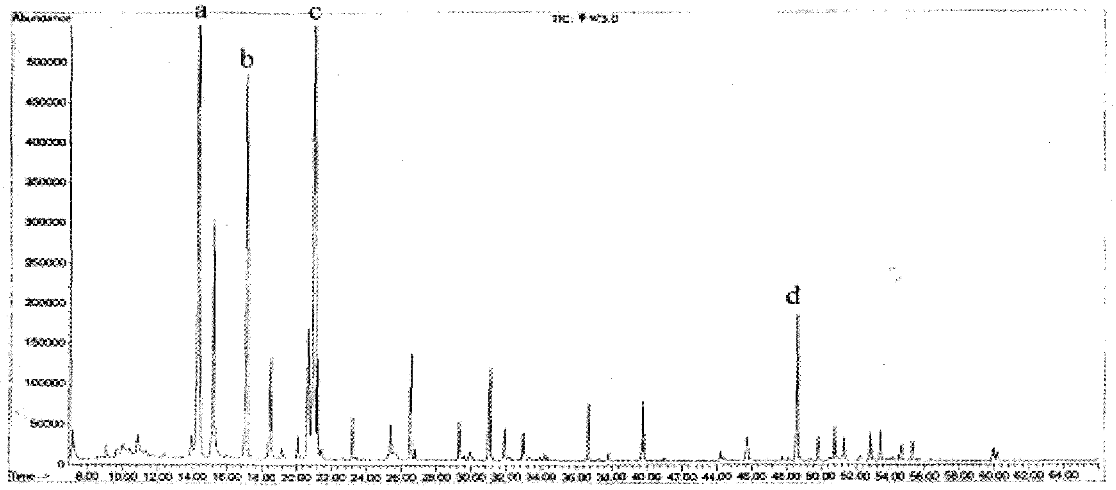

**Fig. 2.** Total ion chromatogram spectrum of Dynamic headspace (Purge & Trap) of *Magnolia ovobata* thunb. (a:β-pinene, b:β-pinene, c:1-limonene, d:β-caryophyllene).

than the Purge & Trap method in *Magnolia ovobata* Thunb. (Fig. 1 & 2). This result was opposite to the study of Lee *et al.* (1997) where the Purge & Trap was found as an adequate analysis method for low polar volatile components than the SDE method and other solvent extraction methods.

## LITERATURE CITED

**Lee JG, Kwon Yj, Jang Hj, Kim OC, Choi YH** (1997) A comparison of different extraction methods for volatile components of Anise (*Pimpinella anisum* L.) Agricultural Chemistry and Biotechnology. Vol, 40(2), 144-147.

**Hong CU, Kim CS, Kim NG, Kim YH** (2001) Composition of essential oil of *Tagetes minuta* L. Korean J. Medicinal Crop Sci., 9(2) : 108-115

**Bae KH** (2001) The medicinal plants of Korea. 3rd Edition Published by Kyohak Co. LTD.

**Anonymous** (2002) Fatty composition of *Magnolia ovobata* Thunb. Research Report on the Division of Industrial Crop, National Crop Experiment Station.

**Kang KH, Roh BS, Seo JH, Heur WD** (1998) Principal of Food Analysis. Published by SungKyunKwan University.