

Comparison of essential oil compositions of fresh and dried fruits of *Magnolia kobus* DC.

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ABSTRACT

The aim of this work was to determine the volatile constituents of fresh and dried (shade dried) fruits of *Magnolia kobus* DC. using gas chromatography-mass spectrometry (GC-MS). Essential oils from fresh and dried fruits of *M. kobus* were obtained by steam distillation and the yields were 0.81 and 1.73% (v/w), respectively. GC-MS analysis revealed the identification of seventeen components from both the fresh and dried fruits of *M. kobus*, which were mostly monoterpene hydrocarbons (62.8–71.8%). A comparison of oils from fresh and dried fruits exhibited the changes in the amounts of several constituents. Major components of the essential oil were α -pinene (26.7–31.6%), β -pinene (20.2–27.9%) and limonene (8.6–10.0%). In the dried sample, concentration of α -pinene, β -pinene, caryophyllene, camphene, and α -humulene contents were decreased. On the other hand, limonene, α -terpineol, bornyl acetate and terpinen-4-ol contents were increased in the dried sample. The present investigation clearly suggested that the drying treatment decreased the concentration of major components in the essential oil of *M. kobus* fruit.

INTRODUCTION

Magnoliaceae is one of the families of flowering plants and the genus *Magnolia* contains more than 128 species, with about two-thirds of species are distributed in East and Southeast Asia (Azuma *et al.*, 1999; Gottsberger *et al.*, 2012). Flower buds of different *Magnolia* species have been used for the treatments of nasal congestion with headache, sinusitis and allergic rhinitis (Jung *et al.*, 1998; Kim *et al.*, 2003). Kobayashi *et al.* (1998) also studied the antirheumatic and antiangiogenic activities of *Magnolia* flower.

Previous studies have reported that the essential oil from different parts of *Magnolia* taxa have a very wide distribution of monoterpene and sesquiterpene components (Barros *et al.*, 2012; Guerra-Boone *et al.*, 2013). *Magnolia kobus* DC is a medium sized deciduous tree native to Japan, also distributed in China and Korea. Flower buds of *M. kobus* are essential ingredients in the Chinese traditional medicine ‘Shin-I’, which is used for the treatments of headaches, and colds (Matsutani and Shiba, 1975). Previous chemical studies on *M. kobus* showed that it to be a source of bioactive terpenes and

lignans (Seo *et al.*, 2008). However, there is no information available on the essential oil composition of *M. kobus* fruit. Medicinal plants can be marketed as fresh or dried products depending on their use and consumer’s preference. Fresh herbs cannot be distributed in a profitable way to world-wide because they contain about 75–80% of water. So, it is essential to lower the water levels to less than 15% for the preservation of plant materials (Diaz-Maroto *et al.*, 2002a, 2002b). Drying is the most widely used preservation method to obtain high quality products. Previous studies have reported that drying processes inhibit the microbial growth, extend the shelf life of product, minimize packaging requirements and reduce shipping weights (Hossain *et al.*, 2010; Hamrouni-Sellami *et al.*, 2012; Kubra and Rao, 2012). Further, the drying processes minimize the loss of active components and delay their biological deterioration by reducing enzymatic activities. Dehydration of herbal plants can be achieved using different methods. In the various drying methods, natural drying (under shade) and hot air drying are the inexpensive and most commonly used methods (Soysal, 2004). The quality of volatile components in the aromatic plants is mainly depends on drying methods and biological characteristics of the plant species (Venskutonis, 1997). The essential oil components are more sensitive to drying process. Hence, it is more essential to understand the changes in the volatile oils from fresh and dried fruits of *M. kobus* in determining the potential of this oil for commercial utilization.

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The aim of this study was to compare the volatile oil compositions of fresh and dried (shade dried) fruits of *M. kobus*.

MATERIALS AND METHODS

Plant material

Fruits (unripen) of *Magnolia kobus* DC. were collected from Chuncheon, Gangwon province, Korea during the month of August 2013. The plant was authenticated and deposited in the Herbarium, Kangwon National University with voucher number KWNU 71688.

Extraction of essential oil

For the comparison of essential oil composition of fresh and dried fruits, a portion of the fresh fruits was extracted and analyzed immediately and equal portion of fruits dried under shade at 25 ± 2 °C for 1 week, then extracted and analyzed. The volatile oil was isolated by steam distillation (1 kg fruits) for 1 h. The lighter than water, slightly yellow oils were dried over anhydrous Na_2SO_4 and stored under refrigeration ($+4$ °C). Oil yields were determined on an oven-dry weight basis (48 h in 65 °C).

Chemical composition of essential oil

GC-MS analysis was performed with a Varian CP3800 gas chromatography equipped with a VF-5MS polydimethylsiloxane capillary column (30×0.25 mm \times 0.25 μm) and a Varian 1200L mass detector (Varian, CA, USA). Helium was used as a carrier gas at the rate of 1 mL/min. Oven temperature was kept at 50 °C for 5 min initially, and then raised with rate of 5 °C min to 250 °C. The injected volume of essential oil was 10 μL with a split ratio of 1:10. The injector temperature was set at 250 °C. The mass spectra were recorded in the electrospray ionization mode at 70 eV in a scan range of 50 - 600 m/z.

Identification of components

The components of the volatile oils from fresh and dried samples were identified by comparison of their retention indices to n-alkanes (C_9 – C_{18}) and their mass spectral fragmentation pattern with those reported in the literature (Adams, 2007), and stored in the MS libraries (Wiley 275 and National Institute of Standards and Technology (NIST, 3.0))²⁰.

RESULTS AND DISCUSSION

The volatile oils were isolated from fresh and dried fruits of *M. kobus* by steam distillation. The higher oil yield was obtained from dried fruits ($1.73 \pm 0.21\%$) followed by fresh fruits ($0.81 \pm 0.04\%$) on a dry weight basis. The chemical composition of fresh and dry fruits of *M. kobus* was determined using GC-MS techniques. The fresh fruits of *M. kobus* revealed the separation of 12 different compounds. Whereas, essential oil obtained from dry fruits of *M. kobus* revealed the detection of 16 different components. The area percentage and retention indices of the

identified compounds are presented in Table 1. The fresh and dried fruits contained complex mixture consisting mainly by monoterpene hydrocarbons (62.8–71.8%), sesquiterpene hydrocarbons (5.9–10.9%), oxygenated monoterpenes (8.9–13.2%) and oxygenated sesquiterpenes (5.7 – 8.3%) (Fig. 1). A comparison of oils from fresh and dried fruits revealed the changes in the amounts of several constituents, especially in mono- and sesquiterpene hydrocarbons. α -Pinene (26.7–31.6%) was the most abundant component followed by β -pinene (20.2–27.9%) and limonene (8.6–10.0%) in both the fresh and dried fruits of *M. kobus*. Other quantitatively important components were α -terpineol (3.2–4.8%), caryophyllene (3.9–8.1%), camphene (3.3–3.7%), bornyl acetate (1.7–3.2), eucalyptol (2.9%), α -humulene (2.0–2.8).

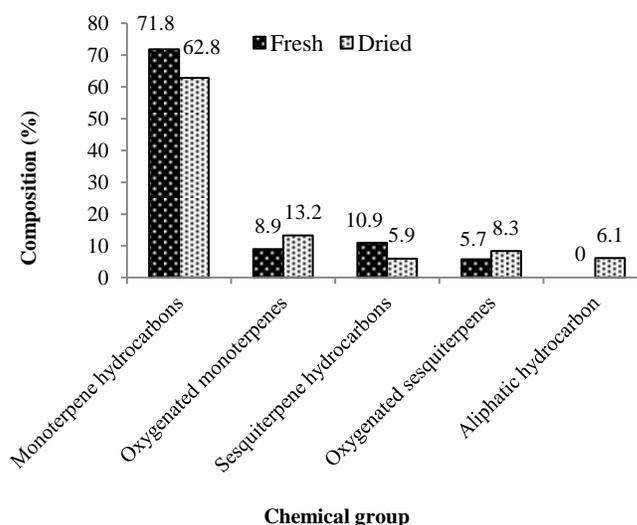


Fig. 1: Percentage concentration of different chemical groups in the essential oils from fresh and dried fruits of *Magnolia kobus*.

Although higher yield obtained from dried fruits, the concentration of major components such as α -pinene, β -pinene and caryophyllene was comparatively decreased than fresh fruits. Singh *et al.* (2010) reported that the variations in dried fruits may be due to the absence of aromatic ring and presence of two conjugated double bonds which may undergo oxidation or polymerization very easily. On the other hand, limonene, α -terpineol and bornyl acetate contents were increased in the dried samples. Additionally, caryophyllene oxide (6.0%), heptadecane (6.1%), Cymene (1.8%), terpinen-4-ol (1.1%), and α -thujene (0.8%) were detected in the essential oil of dried fruit. Further, α -eudesmol (3.5%) was detected only in the fresh sample. Azuma *et al.* (1999) studied the composition of floral scent of *M. kobus* and identified 36 different volatile components. The major components in the floral scents were linalool and its oxides, limonene, *cis* and *trans-p*-ocimene, benzaldehyde, benzyl alcohol, benzyl cyanide, and 2-aminobenzaldehyde. Barros *et al.* (2012) studied the volatile constituents of the unripe fruits of *Magnolia ovata* (A. St.-Hil.) Spreng. from two plant populations (A and B). The essential oil from sample A was rich in sesquiterpenes, mainly spathulenol

(19.3%), whereas the oil from sample B exhibited a predominance of aliphatic compounds, mainly hexadecanoic acid (52.0%).

Table 1: Essential oil composition identified from fresh and dried fruits of *Magnolia kobus*

No.	Compound name	RI ^a	Composition (%)		Identification
			Fresh	Dried	
1	α -Thujene	930	-	0.8	MS, RI
2	α -Pinene	939	31.6	26.7	MS, RI
3	Camphene	954	3.7	3.3	MS, RI
4	β -Pinene	979	27.9	20.2	MS, RI
5	Cymene	1026	-	1.8	MS, RI
6	Limonene	1029	8.6	10.0	MS, RI
7	Eucalyptol	1031	2.9	2.9	MS, RI
8	Fenchone	1086	1.1	1.2	MS, RI
9	Terpinen-4-ol	1177	-	1.1	MS, RI
10	α -Terpineol	1188	3.2	4.8	MS, RI
11	Bornyl acetate	1285	1.7	3.2	MS, RI
12	β -Caryophyllene	1419	8.1	3.9	MS, RI
13	α -Humulene	1454	2.8	2.0	MS, RI
14	Caryophyllene oxide	1583	-	6.0	MS, RI
15	α -Eudesmol	1653	3.5	-	MS, RI
16	Humulene epoxide II	1608	2.2	2.3	MS, RI
17	Heptadecane	1700	-	6.1	MS, RI
			71.8	62.8	
Monoterpene hydrocarbons					
Oxygenated monoterpenes			8.9	13.2	
Sesquiterpene hydrocarbons			10.9	5.9	
Oxygenated sesquiterpenes			5.7	8.3	
Aliphatic hydrocarbon			-	6.1	
Total identified			97.3	96.3	
Oil yield (v/w DW)			0.81	1.73	

^aRI: Retention indices reported in the literature.

Retention indices relative to C₈-C₁₈ n-alkanes on the HP-5MS capillary column.

Several authors have reported that the influence of drying methods on yield and composition of essential oil in many aromatic plants. In the present study, results of essential oil composition are in general agreement with previous studies exhibit the drying process produces qualitative and quantitative changes between the fresh and dried samples (Dong *et al.*, 2011; Doymaz, 2011; Tarhan *et al.*, 2011). Based on the drying method and plant species, several changes occur in the yield and composition of essential oil samples (Kubra and Rao, 2012; Calin-Sanchez *et al.*, 2012; Rahimmalek and Goli, 2013). The drying process facilitates the transportation, storage and handling of the plant materials. Further, drying process prevents some important biochemical reactions that can alter organoleptic characteristics of the plants (Díaz-Maroto *et al.*, 2003; Hamrouni-Sellami *et al.*, 2012). Aromatic plants are most sensitive to drying processes that increase the biological deterioration. Therefore, determining an appropriate drying method is very important to obtain high quality essential oil components.

CONCLUSION

In the present study, the results revealed the identification of 19 different volatile components from fresh and dried fruits of *M. kobus* with α -pinene and β -pinene as major ones. This study indicated that the drying influences the essential oil yield and composition of *M. kobus* fruits. For industrial utilization, standardization of drying method is a fundamental requirement for

achieving a high quality product. The studies related to optimization of drying methods for *M. kobus* fruit are under progress.

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CONFLICT OF INTEREST

Authors declare that they have no conflict of interest.

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