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RESEARCH NOTE

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Comparative Chemical Composition of Domestic and Imported *Chrysanthemum indicum* L. Flower Oils

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Abstract Volatile flavor compounds were isolated from the flowers of *Chrysanthemum indicum* L. (*gamguk*) produced in Korea and China by the hydro distillation, and were analyzed by gas chromatography-mass spectrometry (GC/MS). The yield of oils from Korean and Chinese *gamguk* were 2.0 and 0.5%(v/w), respectively. Sixty-three volatile compounds of Korean *gamguk* representing 89.28% of the total peak area were tentatively identified, including 35 hydrocarbons, 12 alcohols, 6 ketones, 3 esters, 5 aldehydes, 1 oxide, and 1 miscellaneous component. Thirty-six volatile components of Chinese *gamguk* constituted 58.15% of the total volatile composition were tentatively characterized, consisting of 19 hydrocarbons, 7 alcohols, 2 ketones, 2 esters, 4 aldehydes, 1 oxide, and 1 miscellaneous component. The predominant components of Korean oil were α-pinene, 1,8-cineol, and chrysanthenone. Whereas, camphor, α-curcumene, and β-sesquiphellandrene were the main aroma compounds of Chinese *gamguk*.

Keywords: *Chrysanthemum indicum* L. (*gamguk*), essential oil, hydro distillation, producing nation, gas chromatography/mass spectrometry

Introduction

Due to its unique aroma, *Chrysanthemum indicum* L. (*gamguk*) has been used as the mixed spices and food additives as the food masking agent in the region of Asia include India, and it has been used as the dish of the fried pancake named *gukwhajeon* in Korea, as the tea named *gukwhacha,* and alcoholic beverage called *gukwhajoo* (combined with the alcohol) in China, Japan, and Korea (1,2). Moreover, the whole body of the plants used in traditional folk medicine against woman's diseases, nephritis, and the treatment of neurological problem and headache since ancient times (2-5). Furthermore, recently it has been reported that it has evaluated to have central and peripheral analgesic properties, lowering blood pressure as well as anti-inflammatory activities (6,7). The essential oils of *Chrysanthemum* species have been studied by several researchers (8-10). However, there is no research on the essential oil of the imported aromatic medicinal plants. Recently, many food materials including vegetables and even some medicinal plants have been imported from foreign countries, especially from China. In this study, the analysis of volatile compounds from Chinese *gamguk*, in comparison with domestic *gamguk* was executed. We report the volatile aroma constituents from *gamguk* flower oils according to the regions of origin, and to evaluate and compare of those essential oils.

Materials and Methods

Plant materials *Chrysanthemum indicum* L. (*gamguk*) harvested in the fall of 2006 from Namwon (Jeollabuk-do) province, in the western part of Korea, was purchased at Gyeongdong herbal market (Seoul, Korea) in the spring of 2007. *Gamguk* produced in China was packaged in the fall of 2006, and imported by Jinhyung Inc., was purchased at Gyeongdong herbal market in the spring of 2007. These samples were kept at −70°C in air-tight bags until the analysis was carried out.

Separation of the aroma The dried flowers of *gamguk* were crushed for 10 sec by a blender (NJ-8060SM; NUC Electronics, Seoul, Korea) and 1 kg samples were extracted by hydro distillation extract method for 3 hr from setting using a Clevenger-type apparatus (Hanil Lab Tech Ltd., Incheon, Korea). The essential oils obtained were dried over anhydrous sodium sulfate overnight, measured and stored in hermetically sealed dark-glass containers in a freezer at −4°C until it was tested and analyzed by gas chromatography/mass spectrometry (GC/MS).

GC-MS analysis An Agilent 6890 gas chromatography/5973 mass selective detector (Agilent Co., Palo Alto, CA, USA) was employed. Analysis was carried out on a HP-5% phenyl-methylpolysiloxane (5MS) capillary column (30 m length×0.25 mm i.d.×0.25 μm film thickness; Agilent Co.) using a micro syringe. Helium gas was used as carrier gas at a flow rate of 1.0 mL/min. The oven temperature was maintained at 40°C for 5 min and then programmed to increase as follows: from 40 to 150°C at a rate of 3°C/min and holding at 150°C for 5 min, and then 150 to 220°C at a rate of 7°C/min and holding at 220°C for 5 min. The temperatures of the injector and detector were 250 and 280°C, respectively. The sample 0.1 μL, previously dissolved in methylene chloride, was injected in split mode with a split ratio of 10:1. The MS condition were: ionization energy of the mass selective detector was 70 eV, scanning interval 0.5 sec and detector voltage 1.2 kV, and the mass scanning ranges were recorded at *m/z* 33-330.

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Characterization of volatile components The components of the hydro distilled oils from *gamguk* were tentatively identified by means of comparison of their retention indexes (RIs) on a HP-5MS capillary column, which were determined relative to the retention time of a homologous series of *n*-alkane with linear interpolation with those of authentic compounds. The constituents were also identified by comparison of their RIs with those of other essential oils which had been identified earlier (11). The RIs of the compounds, determined using *n*-alkanes [Alkane Standard Solution (04070, 04071), (C_8-$C_{20,}$ C_{21-40}), Standard for GC, Fluka, Buchs, Switzerland] as external references, were compared with the published data (12,13). Especially several compounds were identified with those of the literature (14-17), and identification based on coinjection with authentic compounds (Acros; Sigma-Aldrich, St. Louis, MO, USA). The relative amount of individual components from the oil are expressed as peak area % relative to total peak area from the based on the ratio of the peaks obtained from the mass total ion chromatogram, and also marked quality percentage of the volatile flavor compounds from the GC/MS data.

Results and Discussion

The list of detected compounds in the hydro distilled oils from 2 different produced nations of *gamguk* flowers with their retention time, retention indices, relative peak area percentage, quality percentage, and percentage amounts of compound classes are given in Table 1 and 2. The data are mean values of triplicates. There were some differences observed in composition of the essential oils from *gamguk* flower according to the producing region. *Gamguk* flower oils were tentatively characterized, 89.28 and 58.15% of the total peak area. The color of the 2 *gamguk* oils seemed to have difference, Korean was light golden yellow, Chinese was strong yellow. The yield of the essential oils from *gamguk* flower was 2.0 and 0.5%(v/w), respectively.

Chemical composition of domestic *gamguk* oil As shown in Table 1 and 2, 63 volatile flavor compounds representing 89.28% of the total peak area were tentatively identified, including 35 hydrocarbons (48.75%), 12 alcohols (19.92%), 6 ketones (15.31%), 3 esters (4.61%), 5 aldehydes (0.43%), 1 oxide (0.22%), and 1 miscellaneous component (0.04%). The major constituents characterized from *gamguk* were α-pinene, 1,8-cineol, and chrysanthenone (>10%). Followed by germacrene D, (-)-sinularene, bisabolene, bornyl acetate, borneol, and β-elemene (>3%). Monoterpene hydrocarbons were 14 [α-pinene, tricyclene, camphene, sabinene, β-pinene, myrcene, α-phellandrene, α-terpinene, *p*-cymene, *cis*-ocimene, γ-terpinene, *(E)*-β-ocimene, α-terpinolene, and γ-terpinolene], and 20 sesquiterpene hydrocarbons [α-copaene, β-elemene, α-longipinene, α-humulene, calarene, β-caryophyllene, germacrene D, α-farnesene, *allo*-aromadendrene, zingiberene, α-muurolene, β-sesquiphellandrene, γ-cadinene, germacrene B, α-curcumene, β-bisabolene, (-)-sinularene, *(E)*-β-farnesene, *cis*-α-bisabolene, and eremophillene]. Among them, the most abundant volatile flavor of *gamguk*, α-pinene was described as the pine tree aroma and flavor, and it is very important starting material for perfume industry (19). This volatile flavor compound was also characterized in high contents of the *Juniperus oxycedrus* ssp. *oxycedrus* (27.40%) berry and wood oils from Lebanon, and it has reported antioxidant and hypoglycemic activities (20). It is so valuable and beneficial that the major abundant components from Korean *gamguk*, its physicochemical and biofunctional properties would be the useful and important material for the industrial and medicinal purposes. It could be used for chemotherapy and immunotherapy. There were 12 terpenic alcohol compounds included 1,8-cineol, *(E)*-chrysanthenol, iso pinocarveol, borneol, terpinen-4-ol, endoborneol, *(E)*-charveol, eugenol, spathulenol, cedrol, vulgarol B, and α-bisabolol. Among them, 1,8-cineol, this volatile flavor compound detected abundant in this study, was also found in *Chrysanthemum boreale* Makino, not detected in German chamomile (21,22). This compound, is called as eucalyptol or cajeputol, was known to the important volatile flavor of mugwort (*Artemisia asictica* Nakai), a member of the Compositae family also, and also reported that has the anti cancer and anti microbial effect (23). There were 6 ketone compounds, consisting of camphor, filifolone, chrysanthenone, menthone, pinocarvone, and carvone, and 3 esters included *(E)*-sabinene hydrate, bornyl acetate, and *(E)*-carvyl acetate. And also there were 5 aldehyde compounds were detected by GC/MS, consisting of *(E)*-2-hexanal, safranal, benzealdehyde, campholen acetaldehyde, and phenyl acetaldehyde. Chrysanthenone which belonging to the ketone was also detected in *C. boreale* Makino, not detected in German chamomile tea (21). This compound was known as the main volatile flavor compound of Compositae genus essential oil, however it has been reported that was not found in *Artemisia asictica* Nakai even though same Compositae family (24). *Gamguk* flower has some oriental volatile aroma flavor notes, it is known that this scent comes from chrysanthenone and bornyl acetate, and chrysanthenone was known as the fresh volatile flavor component of pine tree aroma (19). It was also detected in *Zanthoxylum piperitum* A.P. DC. produced in Korea (25).

Chemical composition of imported *gamguk* oil Thirty-six volatile flavor compounds of Chinese *gamguk* constituted 58.15% of the total composition were tentatively identified, consisting of 19 hydrocarbons (25.83%), 7 alcohols (9.24%), 2 ketones (14.18%), 2 esters (4.23%), 4 aldehydes (1.89%), 1 oxide (2.78%), and 1 miscellaneous component (Table 1). Among them, camphor (14.10%) was the most abundant compound, followed by α-curcumene (7.67%) and β-sesquiphellandrene (7.17%). Camphor is a saturated ketone having camphene skeleton, and is obtained industrially via the pinene, camphene, and isoborneol (26). α-Curcumene, the second abundant flavor compound of the Chinese oil, was also detected in the essential oils of the umbels and fruit of *Prangos uloptera*, an indigenous Iranian medicinal plant (10.65%) (27). There were 15 compounds of the terpene hydrocarbons in the oils of *gamguk* from China, monoterpenes were 7 [α-thujene, α-pinene, camphene, sabinene, myrcene, α-terpinene, and γ-terpinene] and taking 1.65%, and sesquiterpenes were 8 [β-elemene, α-humulene, *(E)*-β-farnesene, germacrene D, α-curcumene, zingiberene, β-bisabolene, and β-sesquiphellandrene] and making up 23.69% of the total peak area. There were

Table 1. Comparison of volatile compounds from domestic and imported *gamguk*

Compounds	RI[1)]	RI[2)]	QA%[3)]	QA%[4)]	PA%[5)]	PA%[6)]	Method of ID[7)]
(E)-2-Hexanal	823	826	97	95	0.01	0.05	RT, MS/RI
Tricyclene	903	891	96	96	0.01	0.04	RT, MS/RI
α-Thujene	927	-	94	-	0.03	-	RT, MS/RI
α-Pinene	939	940	96	96	0.06	14.63	RT, MS/RI
Camphene	945	945	98	98	0.40	0.40	RT, MS/RI
Benzealdehyde	957	955	94	93	0.09	0.04	RT, MS/RI
Sabinene	973	967	96	97	0.41	1.24	RT, MS/RI
β-Pinene	-	968	-	97	-	0.23	RT, MS/RI[a)]
1,2,4-Trimethyl benzene	-	983	-	90	-	0.06	RT, MS
1-Octen-3-ol	975	-	90	-	0.44	-	RT, MS
6-Methyl-5-hepten-2-one	980	-	91	-	0.11	-	RT, MS
Myrcene	988	986	69	94	0.04	1.17	RT, MS/RI
α-Phellandrene	-	993	-	95	-	0.49	RT, MS/RI
α-Terpinene	1,009	1,004	98	98	0.26	0.58	RT, MS/RI[b)]
p-Cymene	-	1,010	-	95	-	0.01	RT, MS/RI
1,8-Cineol	1,022	1,020	98	98	1.81	10.71	RT, MS/RI
cis-Ocimene	-	1,026	-	97	-	0.04	RT, MS/RI
Phenylacetaldehyde	1,030	1,028	87	91	0.05	0.05	RT, MS
(E)-β-Ocimene	-	1,038	-	97	-	0.03	RT, MS/RI
γ-Terpinene	1,056	1,046	97	97	0.45	0.87	RT, MS/RI[*]
(E)-Sabinene hydrate	-	1,056	-	94	-	0.77	RT, MS/RI[c)]
α-Terpinolene	-	1,097	-	98	-	0.30	RT, MS
α-Campholene aldehyde	-	1,100	-	83	-	0.14	RT, MS
Filifolone	-	1,113	-	90	-	2.24	RT, MS
(E)-Chrysanthenol	-	1,129	-	78	-	1.17	RT, MS
Chrysanthenone	-	1,140	-	93	-	10.01	RT, MS
Isopinocarveol	-	1,150	-	78	-	1.55	RT, MS
Camphor	1,150	1,152	98	98	14.10	2.64	RT, MS/RI
Menthone	-	1,155	-	83	-	0.03	RT, MS/RI[d)]
Pinocarvone	-	1,171	-	69	-	1.19	RT, MS
Borneol	1,186	1,186	95	87	2.47	3.02	RT, MS/RI[a)]
Terpinen-4-ol	1,190	1,192	96	97	3.22	2.41	RT, MS/RI[*]
Endoborneol	-	1,195	-	63	-	0.07	RT, MS
α-Terpineol	1,198	-	83	-	1.11	-	RT, MS
Dodecane	1,200	-	93	-	0.37	-	RT, MS
γ-Terpinolene	-	1,204	-	78	-	2.04	RT, MS
Methyl chavicol	-	1,207	-	95	-	0.14	RT, MS/RI*
(E)-Chaveol	-	1,229	-	98	-	0.29	RT, MS
Cuminic aldehyde	1,233	-	97	-	1.74	-	RT, MS
Carvone	1,258	1,248	94	97	0.08	0.07	RT, MS/RI
Bornyl acetate	1,319	1,289	99	98	4.06	3.64	RT, MS/RI
(E)-Carvyl acetate	-	1,295	-	94	-	0.20	RT, MS/RI
Sabinyl acetate	1,349	-	72	-	0.17	-	RT, MS
Eugenol	1,353	1,350	98	97	0.08	0.14	RT, MS/RI
α-Copaene	-	1,375	-	99	-	0.23	RT, MS/RI
β-Elemene	1,383	1,382	99	99	0.71	3.18	RT, MS/RI
Safranal	-	1,384	-	86	-	0.15	RT, MS
α-Logipinene	-	1,391	-	69	-	0.09	RT, MS
β-Caryophyllene	-	1,440	-	99	-	0.81	RT, MS/RI
Calarene	-	1,448	-	97	-	0.04	RT, MS
α-Farnesene	-	1,455	-	89	-	0.06	RT, MS/RI
α-Humulene	1,487	1,467	98	98	0.27	0.13	RT, MS/RI
allo-Aromadendrene	-	1,473	-	99	-	0.06	RT, MS/RI

Table 1. Continued

Compounds	RI[1)]	RI[2)]	QA%[3)]	QA%[4)]	PA%[5)]	PA%[6)]	Method of ID[7)]
(E)-β-Farnesene	1,498	1,480	97	98	3.06	1.87	RT, MS/RI
α-Muurolene	-	1,481	-	91	-	0.28	RT, MS/RI
Germacrene D	1,502	1,494	98	99	0.88	5.25	RT, MS/RI
α-Curcumene	1,504	1,496	98	96	7.67	1.80	RT, MS/RI
Germacrene B	-	1,505	-	98	-	0.80	RT, MS/RI
Zingiberene	1,513	1,511	94	93	3.19	2.70	RT, MS
(-)-Sinularene	-	1,514	-	69	-	3.95	RT, MS
β-Bisabolene	1,526	1,516	99	98	0.74	3.95	RT, MS/RI
γ-Cadinene	-	1,520	-	97	-	0.13	RT, MS/RI
β-Sesquiphellandrene	1,542	1,532	98	98	7.17	1.19	RT, MS/RI[d)]
cis-α-Bisabolene	-	1,543	-	98	-	0.08	RT, MS
Spathulenol	-	1,567	-	95	-	0.68	RT, MS/RI
Caryophyllene oxide	1,590	1,570	96	99	2.78	0.22	RT, MS/RI
α-Cedrol	1,598	1,596	76	78	0.11	0.02	RT, MS
Eremophilene	-	1,603	-	90	-	0.02	RT, MS
α-Bisabolol	-	1,651	-	90	-	0.37	RT, MS/RI[c)]
Vulgarol B	-	1,740	-	89	-	0.03	RT, MS/RI[a)]
Hexadecanoic acid	1,898	-	96	-	tr	-	RT, MS/RI
14 β-Pregnane	2,210	-	78	-	tr	-	RT, MS

[1)]RI is retention index; *gamguk* imported from China.
[2)]*Gamguk* produced in Korea (18).
[3)]QA% means quality%, (average, n=3) from the MS data imported from China.
[4)]QA%, produced in Korea.
[5)]PA% is peak area %, average (n=3) of the relative percentage of the peak area in the MS total ion chromatogram from Chinese *gamguk*; tr, trace; mean value <0.01%.
[6)]PA%, produced in Korea.
[7)]ID: Method of identification based on reference no.12,13. Tentative identification index was performed as follows: mass spectrum (MS) was identical with that of Wiley mass spectral database (2001, Hewlett Packard Co., Palo Alto, USA). RI: retention index was consistent with that of the literature. [a)]Identification based on reference no.14; [b)]Identification based on reference no.15; [c)]Identification based on reference no.16; [d)]Identification based on reference no.17. *Identification based on co-injection with authentic compounds compounds (Acros, Sigma-Aldrich, St. Louis, MO, USA).

7 terpenic alcohol compounds in Chinese *gamguk* flower oil consisting of 1,8-cineol, borneol, terpinen-4-ol, α-terpineol, eugenol, α-cedrol, and 1-octen-3-ol. There were 2 ketone compounds in Chinese *gamguk* with camphor and carvone, and 2 esters included bornyl acetate and sabinyl acetate. And also there were 4 aldehyde compounds detected by GC/MS in this study, consisting of *(E)*-2-hexanal, benzealdehyde, phenyl acetaldehyde, and cuminic aldehyde (Table 2).

Table 2. Relative constitution by functional group of *gamguk*

Functional group	No. of peak		% of peak area	
	I[1)]	II[2)]	I[1)]	II[2)]
Hydrocarbon	35	19	48.75	25.83
Aldehyde	5	4	0.43	1.89
Ester	3	2	4.61	4.23
Alcohol	12	7	19.92	9.24
Ketone	6	2	15.31	14.18
Oxide	1	1	0.22	2.78
Miscellaneous	1	1	0.04	tr
Total	63	36	89.28	58.15

[1)]Essential oil of Korean *gamguk* flowers by hydro-distillation-GC/MS.
[2)]Essential oil of Chinese *gamguk* flowers by hydro-distillation-GC/MS.
[3)]Average (n=3) of the relative percentage of the peak area in the MS total ion chromatogram.

Some differences were observed in composition of the essential oils of *gamguk* depending upon the producing nations. In the volatile compounds of those from Korea and China, terpene hydrocarbons were predominant class of compounds with a ratio of 45.91 and 25.34%, respectively. Plant essential oils are highly enriched with terpenoids. They have inhibitory action against micro-organisms by disturbing membranes (28). *(E)*-2-Hexanal, tricyclene, α-pinene, camphene, benzealdehyde, sabinene, myrcene, α-terpinene, 1,8-cineol, phenyl acetaldehyde, γ-terpinene, camphor, borneol, terpinen-4-ol, carvone, bornyl acetate, eugenol, β-elemene, α-humulene, *(E)*-β-farnesene, germacrene D, α-curcumene, zingiberene, β-bisabolene, β-sesquiphellandrene, caryophyllene oxide, and α-cederol were the common terpene hydrocarbon compounds in both *gamguk*. α-Pinene (14.63%) was the most volatile flavor compound of *gamguk* in Korea. However it was small amount in the Chinese *gamguk*. Korean *gamguk* contains more volatile flavor compounds and larger peak area % of total peak area than that of China. Sixty-three and 36 volatile flavor compounds of *gamguk* produced in Korea and China were identified, respectively. The yield of Korean oil was 4 times more than that of Chinese, and representing 89.28 and 58.15% of the total peak area, respectively. The predominant components of Korean oil were α-pinene, 1,8-cineol, and chrysanthenone. Whereas, camphor, α-curcumene, and β-sesquiphellandrene were the main aroma components of Chinese *gamguk*. Our experimental results show that the quality and functional properties of Korean *gamguk* seemed to be more superior than that of China, and suggests a plausible utilization of the essential oils from *gamguk* produced in Korea as a specific purpose and commercial source.

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