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The Volatile Composition of Kiyomi Peel Oil (*Citrus unshiu* Marcov.×*C. sinensis* Osbeck) Cultivated in Korea

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Abstract

The volatile composition of Kiyomi peel oil cultivated in Korea was studied by using gas chromatography and gas chromatography-mass spectrometry. The peel oil from the Kiyomi fruit was prepared by using a cold-pressing extraction method. Among the 65 components quantified in Kiyomi oil, 25 terpene hydrocarbons and 40 oxygenated compounds were identified, with peak weight percentages measuring 94.5% and 4.9%, respectively. Limonene was the predominant compound (87.5%), followed by myrcene (2.4%), sabinene (0.9%), α-pinene (0.8%), β-sinensal (0.8%), (*Z*)-β-farnesene (0.7%), neryl acetate (0.6%), valencene (0.5%), α-farnesene (0.5%), and α-sinensal (0.5%). A unique characteristic of the volatile profile of the Kiyomi oil was the proportion of aldehydes (2.7%), which resulted from the relative abundance of α- and β-sinensal. Another unique characteristic of the Korean Kiyomi oil was its relative abundance of β-sinensal, (*Z*)-β-farnesene, neryl acetate, valencene, α-sinensal and nootkatone. Valencene and α- and β-sinensal were regarded as the influential components of Korean Kiyomi peel oil.

Key words: Kiyomi oil, *Citrus unshiu*×*C. sinensis*, volatile composition, limonene, sinensal, nootkatone

INTRODUCTION

Citrus aroma is regarded as an essential industrial material due to its familiarity and general appeal. Essential oils of citrus and their aroma components are used in foods, beverages, perfumeries, cosmetics and even medicine and aromatherapy. Thus knowledge of the volatile profiles of citrus varieties is important for the cultivation and production of commercially valuable fruits.

There are many *Citrus* species, including *Citrus* hybrids, throughout the world and these citrus fruits are considered as staple products. Among the many kinds of citrus fruits, Kiyomi was bred by crossing Satsuma mandarin (*Citrus unshiu* Marcov) and Trovita orange (*Citrus sinensis* Osbeck) in Japan in 1949 (1). This fruit was brought from Japan to Korea in 1978 (2). Kiyomi looks similar to mandarin because of its thin and smooth peel, but its size is slightly larger and its average weight is 200～250 g. The flesh is very juicy and has a sweet and pleasant taste like that of orange (1). Kiyomi is harvested from February to April in Korea, making it more popular in the spring than other sweet citrus fruits such as mandarins or oranges (3). The popularity of Kiyomi is also evident in the recent changes in Korean consumer preferences. Kiyomi together with Shiranui (Hallabong) and Setoka is regarded by consumers as a commercial citrus crop and has begun to replace Satsuma mandarin in Korea (2-5). Moreover the popularity of Kiyomi is so strong that it is commonly referred to as 'Kiyomi orange' in Japan (4).

The volatile composition of Kiyomi was briefly reported with those of 97 other kinds of citrus fruits by Sawamura (6). Recently, Song et al. also reported the volatile profile of the Kiyomi peel oil (4). Both reports were related to the Japanese Kiyomi. From the results of these reports, it has been suggested that following limonene, the major components of the Japanese Kiyomi peel oil are myrcene, sabinene, octanal, α-pinene, β-phellandrene and neryl acetate (4,6). However, the volatile composition of Kiyomi cultivated in Korea was not provided even though Kiyomi is a commercially valuable and popular fruit in both Japan and Korea.

The aim of this study is to investigate both qualitative and quantitative properties of the volatile composition of Kiyomi peel oil cultivated in Korea. The results may be valuable to provide information for understanding the aroma characteristic of the Korean Kiyomi peel oil and for its use in foods, beverages, perfumeries and other fragrance products.

MATERIALS AND METHODS

Materials and chemicals

Mature Kiyomi fruit harvested in February 2006 was

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obtained from Jeju Fruit Experimental Station in Jeju, Korea. Authentic standard chemicals were purchased from Tokyo Kasei Kogyo, Japan; Nacalai Tesque Inc., Japan; Aldrich Chemical Co., USA, and Fluka Fine Chemicals, Switzerland for identification of the oil components.

Preparation of cold-pressed oil

Ten kilograms of Kiyomi fruit was used for preparation of the cold-pressed oil, which was performed by using the method previously described (7). The fruit was cut into 4 or 6 pieces, and then the mesocarp and albedo layers were peeled off the flavedo. The peel oil was isolated by hand-pressing the flavedo and collected in a brine solution on ice. The oil emulsion was centrifuged at 4000 rpm for 15 min at 4°C. To remove any juice or water, the supernatant was dehydrated with anhydrous sodium sulfate at 5°C for 24 hrs and filtered. The yield of oil was 1.8 mL/kg fruit. The oil samples were stored at -25°C until needed for analysis.

Gas chromatography (GC)

GC analysis was carried out using a Jeol Agilent Technologies 6890N JMS-Q1000 gas chromatograph equipped with a flame ionization detector. The used capillary column was a DB-Wax column, 60 m × 0.25 mm with a film thickness of 0.25 μm (J & W Scientific, Folsom, CA). The column temperature was programmed from 70°C (2 min) to 230°C (20 min) at a rate of 2°C/min. The injector and detector temperatures were 250°C. Nitrogen was used as the carrier gas at a flow rate of 1.0 mL/min. The split ratio was 1:100 and a sample of 1 μL was injected.

Gas chromatography-mass spectrometry (GC-MS)

A Jeol Ultra Quad MS coupled with JMS-Q1000GC and 7683B Series Auto Sampler & Injector was used for GC-MS analysis. The column type, size and temperature were the same as in GC analysis. The split ratio was 1:100 and a sample of 0.2 μL was injected. The MS conditions were as follows: injector temperature, 250°C; ionization voltage, 70 eV; ion source temperature, 250°C; mass range, 35 to 350 *m/z*.

Identification of components

Identification was performed by GC-MS analysis: the retention indices of each peak were compared with those of standard compounds on a DB-Wax column; mass spectra for each peak were paralleled with those in the data library of the GC-MS equipment. Data for individual components were identified by comparison of both mass spectra and GC retention times with those of authentic compounds previously analyzed. The retention indices were also determined for all constituents by using a homologous series of *n*-alkanes (C_7-C_{27}).

Quantitative GC analysis was executed using two internal standards, heptanol and methyl myristate (5,7). The quantity ratios of the two internal standards and oil sample were 1:1:150, respectively. The weight percentage of each compound in the oil sample was calculated with response factors to the flame ionization detector (5,7). The quantitative measurements were made in triplicate.

RESULTS AND DISCUSSION

The individual volatile components of Kiyomi cold-pressed peel oil are presented in Table 1 and its chromatogram is shown in Fig. 1. The components were listed in order of elution on the DB-wax column and a classification was based on these functional groups. Major components for each functional group of Korean Kiyomi oil and their weight percentages are summarized in Table 2, together with those of the peel oils of Japanese Kiyomi, Shiranui (*Citrus unshiu* Marcov. × *C. sinensis* Osbeck × *C. reticulata* Blanco) and Satuma mandarin (*Citrus unshiu* Marcov. forma *Miyagawa-wase*).

Sixty-five components accounting for 99.4% of the total were identified in the Kiyomil cold-pressed peel oil. Twenty-five terpene hydrocarbons and 40 oxygenated compounds were identified and the respective peak weight percentages were 94.5% and 4.9%. The monoterpene hydrocarbons representing 92.2% of the total oil, was the predominant volatile group in Kiyomi oil, followed by aldehydes (2.7%). Volatile profile of Kiyomi peel oil was summarized by functional groups in the classification of Table 1.

Hydrocarbons

Among the 13 monoterpene hydrocarbons identified in Kiyomi oil, limonene was the most prominent (87.5%): it is well known to generally be the principal component in citrus oil (8). In the previous report, the proportion of limonene in Japanese Kiyomi oil was 92% (4). It has been reported that the contents of limonene in the oils from Korean Shiranui and Satuma mandarin were lower than those from Japan (5,9, Table 2). The next most plentiful monoterpene hydrocarbons in Kiyomi peel oil were myrcene (2.4%), sabinene (0.9%), α-pinene (0.8%) and β-phellandrene (0.4%). The monoterpene composition was similar to that of Japanese Kiyomi oil (4).

Sesquiterpene hydrocarbons accounted for 2.3% of the total oil with 12 components identified. Among the sesquiterpene hydrocarbons, (*Z*)-β-farnesene was quantified as the largest percentage (0.7%), followed by valencene

Table 1. Volatile components of Kiyomi peel oil cultivated in Korea

No	Compound	Retention Index	Peak weight % (w/w)[1)]	Identification[2)]
Monoterpene hydrocarbons				
1	α-pinene	1042	0.8	RI, MS
2	camphene	1089	*	RI, MS
3	β-pinene	1128	**	RI, MS
4	sabinene	1134	0.9	RI, MS
5	myrcene	1169	2.4	RI
6	α-phellandrene	1174	**	RI, MS
7	α-terpinene	1188	*	RI, MS, Co-GC
8	limonene	1222	87.5	RI, MS
9	β-phellandrene	1224	0.4	RI, MS
11	(*Z*)-β-ocimene	1246	*	RI, MS
12	γ-terpinene	1255	**	RI, MS, Co-GC
13	(*E*)-β-ocimene	1266	0.2	RI
14	terpinolene	1295	**	RI, MS, Co-GC
	Total amount (number)		92.2 (13)	
Sesquiterpene hydrocarbons				
23	α-copaene	1498	**	RI, MS, Co-GC
25	β-cubebene	1540	*	RI, MS
30	β-caryophyllene	1589	0.1	RI, MS
35	(*Z*)-β-farnesene	1665	0.7	RI, MS, Co-GC
36	(*E*)-β-farnesene	1668	0.1	RI, MS
37	α-humulene	1678	**	RI, MS, Co-GC
41	germacrene D	1715	0.2	RI, MS, Co-GC
42	valencene	1721	0.5	RI, MS
43	α-muurolene	1725	**	RI
47	α-farnesene[3)]	1755	0.5	RI, MS
49	*d*-cadinene	1771	0.1	RI
50	sesquiphellandrene	1775	0.1	RI
	Total amount (number)		2.3 (12)	
	Total hydrocarbons		94.5 (25)	
Alcohols				
16	6-methyl-5-hepten-2-ol	1316	*	RI, MS
26	linalool	1552	0.2	RI, MS
28	octanol	1559	**	RI, MS, Co-GC
32	thujyl alcohol	1628	**	RI, MS
40	α-terpineol	1701	0.3	RI, MS, Co-GC
52	nerol	1811	**	RI, MS, Co-GC
54	*cis*-carveol	1846	*	RI, MS, Co-GC
57	perillyl alcohol	2001	0.1	RI, MS, Co-GC
58	(*E*)-nerolidol	2053	**	RI, MS, Co-GC
59	cedrol	2121	*	RI, MS, Co-GC
61	β-eudesmol	2242	0.1	RI
	Total amount (number)		0.7 (11)	
Aldehydes				
15	octanal	1297	0.3	RI, MS
17	nonanal	1399	0.1	RI, MS, Co-GC
22	citronellal	1484	**	RI, MS, Co-GC
24	decanal	1501	0.3	RI, MS, Co-GC
31	undecanal	1601	0.1	RI, MS, Co-GC
33	(*E*)-2-decenal	1644	**	RI, MS
39	neral	1696	**	RI, MS
45	geranial	1737	0.3	RI, MS
51	perill aldehyde	1791	0.1	RI, MS, Co-GC

Table 1. Continued

No	Compound	Retention Index	Peak weight % (w/w)[1)]	Identification[2)]
53	2,4-decadienal[4)]	1814	**	RI, MS
55	(*E*)-2-dodecenal	1864	0.2	RI, MS, Co-GC
60	β-sinensal	2226	0.8	RI, MS
62	α-sinensal	2330	0.5	RI, MS
63	heptadecanal	2338	*	RI, MS
	Total amount (number)		2.7 (14)	
Esters				
20	*trans*-sabinene hydrate	1472	**	RI
21	octyl acetate	1482	**	RI, MS, Co-GC
27	linalyl acetate	1557	*	RI, MS, Co-GC
29	bornyl acetate	1588	0.1	RI
34	citronellyl acetate	1662	0.1	RI, MS, Co-GC
38	decyl acetate	1682	0.2	RI, MS, Co-GC
44	neryl acetate	1734	0.6	RI, MS, Co-GC
48	geranyl acetate	1761	0.2	RI, MS, Co-GC
64	(*E*)-(*E*)-farnesyl acetate	2401	**	RI
	Total amount (number)		1.2 (9)	
Ketones and oxides				
10	1,8-cineole	1226	*	RI, MS
18	*trans*-limonene oxide	1454	**	RI, MS, Co-GC
19	*cis*-limonene oxide	1468	**	RI, MS, Co-GC
46	carvone	1739	0.1	RI, MS, Co-GC
56	caryophyllene oxide	1983	**	RI
65	nootkatone	2524	0.2	RI, MS, Co-GC
	Total amount (number)		0.3 (6)	
	Total oxygenated compounds		4.9 (40)	
	Total volatile compounds		99.4 (65)	

[1)]*Peak weight quantified less than 0.005%; **peak weight quantified> 0.005%< 0.05%. [2)]RI, identification based on retention index of DB-Wax; MS, identification based on comparison of mass spectra; Co-GC, identification based on co-injection with authentic standards. [3)]Correct geometrical isomer not identified. [4)]Correct isomer not identified.

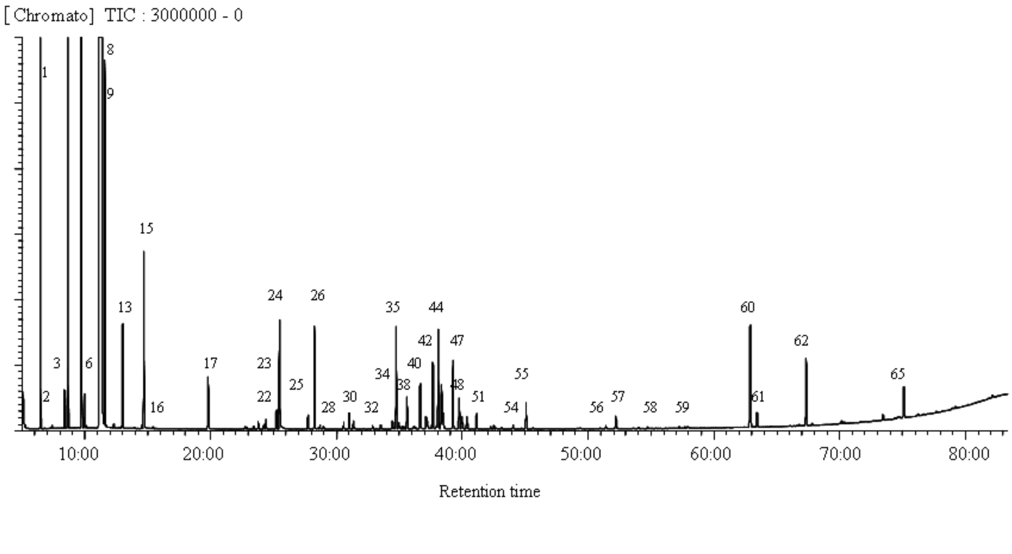

Fig. 1. Total ion chromatogram of Kiyomi peel oil (*Citrus unshiu* Marcov.×*C. sinensis* Osbeck) cultivated in Korea.

(0.5%) and α-farnesene (0.5%). According to previous reports, the portion of (*Z*)-β-farnesene was less than 0.1% in the peel oils of sweet citrus such as Ponkan, Kimikan, Shiranui and mandarins (5,9-11); however, it was relatively abundant in Kiyomi oils, not only from Korea but also from Japan (4). Both α- and β-farnesene were present as low as 0.05% in the peel oil of Satsuma mandarin, a parent of Kiyomi (9). α-Farnesene is seldom

Table 2. Major volatile components of Korean Kiyomi oil and comparisons with those of Shiranui and Satuma mandarin oils

	Kiyomi		Shiranui[3)]		Satuma mandarin[4)]	
	Korean	Japanese[2)]	Korean	Japanese	Korean	Japanese
α-pinene	0.8[1)]	0.5	0.6	0.7	0.9	0.7
sabinene	0.9	0.9	3.6	1.7	0.1	0.1
myrcene	2.4	2.4	2.4	2.6	2.1	2.1
limonene	87.5	92.0	86.4	91.8	86.7	87.6
(*Z*)-β-farnesene	0.7	0.3	nd[5)]	nd	**	**
valencene	0.5	*	*	0.1	**	0.1
α-farnesene	0.5	0.1	1.3	0.7	nd	nd
linalool	0.2	0.2	1.2	0.1	0.7	0.4
β-sinensal	0.8	0.2	nd	nd	nd	nd
α-sinensal	0.5	0.1	nd	nd	nd	nd
neryl acetate	0.6	0.5	0.1	**	**	0.1
nootkatone	0.2	0.1	**	0.1	nd	nd

[1)]Each value is the peak weight (%); *peak weight quantified less than 0.005%; **peak weight quantified> 0.005%< 0.05%.
[2)]Reference 4. [3)]Reference 5. [4)]Reference 9. [5)]nd: not detected.

identified in the peel oils of sweet and sour citrus (4-7, 9-23); however, it is abundant in the oils of some sweet citrus; for example, in Shiranui oil it is present at the level of 0.7～1.3%, which is a child of Kiyomi (5). Valencene has not been regarded as chief sesquiterpene component in common citrus oils (4-7,9-23) because of its low content at the level of less than 0.1%. From these results, the content ratios of (*Z*)-β-farnesene, valencene and α-farnesene seemed to be an important factor for the identification of Korean Kiyomi oil (Table 2). Especially, valencene was suggested as an influential component characterizing the flavor of Korean Kiyomi, which aroma described as like ‘green’, ‘woody’, and ‘herbaceous’ (21).

Alcohols and aldehydes

The Kiyomi peel oil was not especially rich in alcohols, accounting for 0.7%. α-Terpineol (0.3%) of 11 alcohol components was dominant and the next was linalool (0.2%). α-Terpineol and linalool are commonly detected in *Citrus* oil and linalool is known to be an essential component in the fragrance products (24). In Kiyomi peel oils including Japanese Kiyomi oil, linalool was identified as being not very abundant (4), unlike the oils of other sweet citrus fruits such as oranges and mandarins, in which linalool accounts for than 0.4% of the total oil (4-7,9-23).

The aldehyde group containing 14 compounds occupied the highest portion of oxygenated compounds in Kiyomi oil at the level of 2.7%. The higher content of aldehydes in Kiyomi oil resulted from the relative abundance of β-sinensal (0.8%) and α-sinensal (0.5%), followed by octanal (0.3%), decanal (0.3%), and geranial (0.3%). Among the aldehyde components, α- and β -sinensal are usually absent or only present in trace amounts in citrus oils (4-7,9-23). Furthermore, neither compound was detected in the oils of Shiranui, a child of Kiyomi (4); however, α- and β-sinensal were rich enough to be characteristic compounds of Kiyomi peel oil. Aroma characteristics of α- and β sinensal were reported as odor description of ‘woody’, ‘green’, and ‘grassy’ (10,24).

Esters and ketones

Nine esters were identified in Kiyomi peel oil and the content of this group was relatively high at the level of 1.2%, in contrast to that in common citrus oils (0.1～0.2%) (4-7,9-23). Neryl acetate, accounting for 0.6%, was remarkably plentiful in esters of Kiyomi peel oil. In the second place decyl acetate and geranyl acetate were main components, at the level of 0.2%.

According to the previous report, octanal and neryl acetate were major oxygenated components of the peel oil from Japanese Kiyomi, accounting for more than 0.5% (4). Also, β-sinensal (0.8%), neryl acetate (0.6%) and α-sinensal (0.5%) were principal in oxygenated compounds of the peel oil from Korean Kiyomi. From these results neryl acetate was estimated as an essential flavor component characterizing the volatile profile of Kiyomi oil because of its richness (Table 2). Although neryl acetate is not found in most citrus oils or occurs at a trace levels lower than 0.05% (6,7,9-16), its aroma was reported as a citrus-like odor (22,24).

Nootkatone was also comparatively abundant among oxygenated compounds in Korean Kiyomi oil like that of Japanese Kiyomi oil (4, Table 2), at 0.2%. This result may support that nootkatone is an important volatile component discriminating Kiyomi from other oranges and mandarins including Satsuma mandarin, a parent of Kiyomi (4,6,9,19,20, Table 2). Nootkatone was known to be a key compound of pummelo species (19) with aroma characteristics of sweet, fruity and citrusy (23,24).

Table 3. Comparison of the constitution of functional groups

	Kiyomi		Shiranui[3)]		Satuma mandarin[4)]	
	Korean	Japanese[2)]	Korean	Japanese	Korean	Japanese
Hydrocarbons	94.5[1)]	97.1	96.2	98.8	97.1	97.5
Alcohols	0.7	0.3	1.8	0.2	1.0	1.4
Aldehydes	2.7	1.5	1.6	0.7	0.3	0.7
Esters	1.2	0.5	0.2	0.1	0.2	0.3

[1)]Each value is the weight percent of each functional group. [2)]Reference 4. [3)]Reference 5. [4)]Reference 9.

Characteristics of volatile composition of Kiyomi oil

As shown in Table 3, the respective contents of alcohols, aldehydes and esters detected in Korean Kiyomi oil were twice as high as those of Japanese Kiyomi oil (4). In the peel oils of Shiranui, which is a child of Kiyomi, the levels of these oxygenated functional groups were also comparatively higher in Korean oil (5). The abundance of aldehydes (2.7%) and esters (1.2%) was another characteristic of the Korean Kiyomi peel oil.

A significant feature of Korean Kiyomi oil was suggested from the relative abundance of β-sinensal, (*Z*)-β-farnesene, neryl acetate, valencene, α-sinensal and nootkatone.

Neryl acetate and nootkatone were determined to be key flavor components characterizing the volatile composition of Kiyomi oil because of their high proportions.

Valencene and α- and β-sinensal were considered as the influential volatile components of the Korean Kiyomi peel oil.

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