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Characteristic Odor Components of Kumquat (*Fortunella japonica* Swingle) Peel Oil

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This study was conducted to determine the composition of kumquat (*Fortunella japonica* Swingle) cold-pressed peel oil and to determine which volatile components are primarily responsible for the aroma of this oil. Eighty-two compounds were identified in the oil by GC and GC-MS. The major compounds were limonene (93.73%), myrcene (1.84%), and ethyl acetate (1.13%). Flavor dilution (FD) factors and relative flavor activities (RFA) of volatile constituents were evaluated by aroma extract dilution analysis with gas chromatography−olfactometry (GC-O). Camphene, terpinen-4-ol, citronellyl formate, and citronellyl acetate showed high FD factors (≥5) and RFA (>20). Citronellyl formate and citronellyl acetate were regarded as the characteristic odor components of the kumquat peel oil from the results of FD factor, RFA, and GC-sniffing. Citronellyl acetate is considered to be the odor component most similar to kumquat by organoleptic evaluation with GC-O.

KEYWORDS: Kumquat (*Fortunella japonica* Swingle); cold-pressed peel oil; characteristic odor components; gas chromatography−olfactometry; aroma extract dilution analysis

INTRODUCTION

Citrus fruits have wide acceptance due to their attractive flavor and nutritional value. Citrus oils are widely used in the flavor industry, as they are used in a wide variety of food and beverage products. There are a great number of *Citrus* varieties widely distributed in the world. Most citrus fruits are extensively grown from the temperate zone to the tropical zone of both the north and south hemispheres. Kumquat (*Fortunella japonica* Swingle, Rosaceae), called gumgyul or gumgam in Korea, has a typical citrus flavor character. Kumquat is included in the *Citrus* genus, but it is a different species (*1*). Kumquat is believed to be native to China. The fruit varies in shape from round to slightly oval of ∼2 cm diameter, averaging ∼10 g in weight. Kumquat has long been used in traditional herbal medicine, especially for colds and coughs (*2*). The flesh is quite sour, and its peel is eaten together with the flesh. In 2001 the production of kumquat in Korea was estimated to be 3589 tons (*3*). Citrus essential oils are widely used in both the perfumery and food industries. A number of different types of citrus essential oils are available to the food industries (*1*).

There are only a few studies on rare citrus fruits, such as kumquat, in contrast with major citrus fruits, such as lemon and orange (*4*−*7*). Koyasako and Bernhard (*5*) reported 71 volatile compounds in California kumquat, of which *d*-limonene was the most abundant component, comprising 93% of the whole oil. The volatile compositions from Korean and Japanese kumquats were also reported (*6*, *7*). Kwag et al. (*7*) detected >40 volatile compounds in Korean kumquat, of which limonene and α-terpineol were the major components of total oil volatiles and a diethyl ether fraction of the oil, respectively. The characteristic odor components of peel oils from several citrus varieties have been reported by using gas chromatography−olfactometry (GC-O) and aroma extract dilution analysis (AEDA): for instance, citronellal, (*R*)-(+)-citronellal, 2-dodecenal, linalool, and octanol were characteristic odorants of *C. reticulata* Blanco × (*C. unshiu* Marcov × *C. sinensis* Osbeck), *C. sphaerocarpa* Tanaka, *C. grandis* Osbeck forma *Tosa*, and *C. tamurana* Hort. ex Tanaka cold-pressed peel oils, respectively (*8*−*11*). However, no detailed report on the characteristic odor components of kumquat elucidated by GC-O has been given until now. The aim of this work to determine the volatile constituents of kumquat peel oil and to elucidate the characteristic odor components of the oil by GC-O and AEDA techniques.

MATERIALS AND METHODS

Materials. Fresh kumquat (*F. japonica* Swingle), harvested in January 2003, was collected from a farm located in Jeolla province, Korea. The peel oil sample was prepared according to the cold-pressing method described by Choi and Sawamura (*12*) within 24 h of harvest. All of the fruits (∼5 kg) were sliced, and the albedo layer was separated from the flavedo. The peel oils were extracted by hand-pressing from the flavedo, and the peel oils were collected in a brine solution on ice. The oil extract was centrifuged at 4000*g* for 15 min at 4 °C. The supernatant was dehydrated with anhydrous sodium sulfate at 5 °C for 24 h and filtered. The oil was stored at −25 °C until analyzed. Authentic chemicals were obtained from Aldrich Chemical Co. (Milwaukee, WI),

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Table 1. Volatile Components Identified in Kumquat Peel Oil

no.	compound	retention index DB-Wax	retention index DB-5	w/w %	identification	odor description	FD factor (3^n)
1	ethyl acetate	900		1.13	RI,[a] MS[b]		
2	α-pinene[1g]	1035	933	0.39	RI, MS, Co-GC[c]	oily, green	7
3	camphene[2]	1082	953	tr[d]	RI, MS, Co-GC	sweet	6
4	undecane[2]	1112		0.01	RI, MS, Co-GC		
5	β-pinene	1123	981	0.04	RI, MS	dry, green	4
6	sabinene	1133	973	0.10	RI, MS	herbaceous	7
7	δ-3-carene	1161		0.01	RI, MS	herbaceous, green	2
8	myrcene[2]	1168	991	1.84	RI, MS, Co-GC	herbaceous, sweet	7
9	limonene[1]	1235	1039	93.73	RI, MS, Co-GC	lemon-like	7
10	γ-terpinene[3]	1261	1059	0.27	RI, MS, Co-GC	green, woody	4
11	*p*-cymene	1282	1027	0.03	RI, MS	green, fruity	3
12	terpinolene[4]	1294	1084	0.05	RI, MS, Co-GC	tree-like	3
13	tridecane[1]	1312	1291	0.02	RI, MS, Co-GC	fruity, green	3
14	tetradecane[1]	1399	1116	0.01	RI, MS, Co-GC	green	2
15	α-thujone	1431		0.01	RI, MS		
16	*cis*-limonene oxide	1458	1138	tr	RI, MS	citrus-like	3
17	α-cubebene	1466	1345	tr	RI, MS	sweet, fruity	2
18	menthone	1473		0.01	RI, MS		
19	*trans*-linalool furanoid oxide	1478	1172	0.18	RI, MS	fruity	5
20	citronellal[1]	1488	1161	tr	RI, MS, Co-GC	fruity	3
21	α-ylangene	1492		tr	RI, MS	fruity	2
22	pentadecane[1]	1503		0.02	RI, MS, Co-GC	herbaceous	2
23	*d*-camphor	1528	1229	tr	RI, MS	green, gaseous	2
24	β-cubebene[5]	1546	1018	0.01	RI, MS, Co-GC	fruity, green	2
25	linalool[2]	1553	1098	0.10	RI, MS, Co-GC	fruity	3
26	octanol[1]	1566	1072	0.01	RI, MS, Co-GC		
27	nonyl acetate[4]	1582	1302	0.01	RI, MS, Co-GC		
28	β-elemene[6]	1595	1393	0.03	RI, MS, Co-GC	fruity	7
29	β-caryophyllene[4]	1604	1428	tr	RI, MS, Co-GC	citrus-like, fresh	3
30	terpinen-4-ol	1612	1178	0.01	RI, MS	green, fruity, citrus-like	7
31	citronellyl formate[7e]	1629		tr	RI, MS, Co-GC	fruity, sweet, citrus-like, kumquat-like	5
32	γ-elemene[6]	1636		tr	RI, MS, Co-GC	fruity, dry	2
33	*p*-mentha-2,8-diene-1-ol	1642		0.01	RI, MS	fresh, fruity	2
34	menthol[7]	1651		0.02	RI, MS, Co-GC	woody	3
35	citronellyl acetate[4e]	1666	1357	0.01	RI, MS, Co-GC	citrus-like, kumquat-like	6
36	*trans*-β-farnesene[6]	1674		0.01	RI, MS, Co-GC	sweet, fruity	3
37	α-humulene[6]	1681	1444	0.01	RI, MS, Co-GC	citrus-like	4
38	δ-muurolene	1684		tr	RI, MS	fruity	3
39	decyl acetate[1]	1691	1408	tr	RI, MS, Co-GC	fruity, green	1
40	neral	1695	1235	tr	RI, MS	fruity, sweet	1
41	terpinyl acetate	1700		0.01	RI, MS	fruity, sweet	3
42	α-terpineol[1]	1712	1185	0.37	RI, MS, Co-GC	resinous	3
43	dodecanal[8]	1718	1401	0.01	RI, MS, Co-GC	dry, green	3
44	valencene	1727	1490	0.02	RI, MS	green, herbaceous	3
45	bicyclogermacrene	1736		0.04	RI, MS	herbaceous, dry	6
46	*l*-carvone	1740		0.02	RI, MS	dry, herbaceous	4
47	*cis*-linalool pyranoid oxide	1748		0.03	RI, MS	herbaceous, fresh	2
48	*trans*-2-undecenal	1758		0.13	RI, MS	dry, herbaceous	3
49	geranyl acetate	1766		0.01	RI, MS	dry, herbaceous	1
50	citronellol[4]	1771	1435	0.01	RI, MS, Co-GC	sweet, herbaceous	2
51	sesquiphellandrene	1780	1149	tr	RI, MS	mild, herbaceous	2
52	cumin aldehyde	1789		0.04	RI, MS	sweet, fresh	2
53	octadecane[7]	1805		tr	RI, MS, Co-GC		
54	carvone oxide	1818		0.02	RI, MS	sweet	1
55	tridecanal[8]	1824	1503	0.02	RI, MS, Co-GC	fresh, green	4
56	*p*-mentha-1-en-9-yl acetate	1834		0.07	RI, MS	fresh	5
57	*cis*-carveol	1847	1230	tr	RI, MS		
58	geraniol[1]	1861		0.05	RI, MS, Co-GC	herbaceous	2
59	perilla acetate	1905		0.02	RI, MS		
60	tetradecanal[2]	1924		tr	RI, MS, Co-GC	fresh, herbaceous	1
61	*p*-mentha-1-en-9-ol	1946	1486	tr	RI, MS	fresh, herbaceous	3
62	caryophyllene oxide	1990	1570	0.01	RI, MS		
63	*cis*-nerolidol	2008	1565	tr	RI, MS	mild, herbaceous	2
64	*trans*-dodec-2-enol	2041		tr	RI, MS	fruity	3
65	*trans*-nerolidol	2054	1539	tr	RI, MS	fruity	2
66	octanoic acid	2084		0.02	RI, MS	fruity	1
67	elemol	2090	1547	tr	RI, MS	fruity	2
68	cedrenol	2110	1604	tr	RI, MS	fruity	1
69	spathulenol	2133		0.01	RI, MS	herbaceous	3
70	eugenol[1]	2172	1351	tr	RI, MS, Co-GC	dry, herbaceous	2
71	nonanoic acid[7]	2193		0.01	RI, MS, Co-GC	herbaceous	2
72	γ-eudesmol	2208		tr	RI, MS		
73	α-cadinol	2217		tr	RI, MS		
74	isothymol	2222		tr	RI, MS,		

Table 1 (Continued)

no.	compound	retention index DB-Wax	retention index DB-5	w/w %	identification	odor description	FD factor (3^n)
75	α-bisabolol	2228		tr	RI, MS		
76	*β*-sinensal	2238		tr	RI, MS		
77	*β*-eudesmol	2248	1654	tr	RI, MS		
78	*trans,trans*-farnesyl acetate	2282		tr	RI, MS		
79	cinnamyl alcohol[g]	2306	1312	tr	RI, MS, Co-GC		
80	*cis,trans*-farnesol	2355		tr	RI, MS	sweet, fruity	2
81	*trans,trans*-farnesol	2355	1722	tr	RI, MS	mild, herbaceous	2
82	undecanoic acid	2421	1490	tr	RI, MS		
hydrocarbons							
aliphatics (5)[f]				0.06			
monoterpenes (10)				96.46			
sesquiterpenes (12)				0.12			
aldehydes							
aliphatics (4)				0.16			
terpenes (4)				0.04			
alcohols							
aliphatics (2)				0.01			
monoterpenes (12)				0.57			
sesquiterpenes (11)				0.01			
ketones (4)				0.04			
esters (10)				1.26			
oxides and epoxides (5)				0.24			
acids (3)				0.03			
total (82)				99			

[a] Identification based on retention index. [b] Identification based on comparison of mass spectra. [c] Identification based on co-injection with authentic compounds. [d] Trace, <0.005% (w/w %). [e] Kumquat-like odor compounds perceived at the sniffing port. [f] Numbers of identified compounds. [g] Sources of authentic chemicals: 1, Wako Pure Chemical Industries; 2, Aldrich Chemical Co.; 3, Funakoshi Co., Ltd.; 4, Tokyo Kasei Kogyo Co.; 5, Fluka Fine Chemicals; 6, Bolak Co.; 7, French-Korean Aromatics; 8, Theta Co.; 9, Nacalai Tesque Inc.

Fluka Fine Chemicals (Buchs, Switzerland), Funakoshi Co., Ltd. (Tokyo, Japan), Nacalai Tesque Inc. (Kyoto, Japan), Theta Co. (Newtown Square, PA), Tokyo Kasei Kogyo Co. (Tokyo, Japan), Wako Pure Chemical Industries (Osaka, Japan), Bolak Co., Ltd. (Osan, Korea), and French-Korean Aromatics (Youngin, Korea).

GC. An Agilent 6890N gas chromatograph equipped with a flame ionization detector (FID) was used. A polar (DB-Wax) column and a nonpolar (DB-5) column (60 m × 0.25 mm i.d., film thickness = 0.25 m, J&W Scientific, Folsom, CA) were used for GC analysis. The column temperature was programmed from 70 °C (2 min) to 230 °C (20 min) at a program rate of 2 °C/min. The injector and detector temperatures were 250 °C. Nitrogen was the carrier gas at a flow rate of 1 mL/min and a linear velocity of 22 cm/s. The linear retention indices (LRIs) were calculated for all volatile components using a homologous series of *n*-alkanes (C_7−C_{29}) under the same GC conditions.

n-Heptanol and methyl myristate were used as internal standards for quantitative analysis of kumquat oil. The ratio of kumquat oil for the two internal standards was 150:1:1. The weight percentage of each peak was calculated according to the correlation factor to the FID (*13*). An oil sample of 1 μL was injected, and the injector split ratio was 50:1.

GC-MS. Gas chromatography combined with mass spectrometry was used for identifying the volatile components that had been detected. The analysis was carried out with a Varian Saturn 2000R 3800 GC (Walnut Creek, CA) linked with a Varian Saturn 2000R MS. The oven condition, injector and detector temperatures, and column (DB-Wax) were the same as those given above for the Agilent 6890N GC. Oil samples of 0.2 μL were injected, and the split ratio was 34:1. Helium was the carrier gas at a flow rate of 1.1 mL/min and a linear velocity of 38.7 cm/s. MS conditions were as follows: ionization voltage, 70 eV; ion source temperature, 250 °C; mass range, *m*/*e* 40−350.

Identification of Components. Individual components were identified by comparing their LRIs and matching their mass spectra with those of reference compounds in the data system of Wiley library and NIST Mass Spectral Search Program (ChemSW. Inc., NIST 98 version database) connected to a Varian Saturn 2000R MS. Other identifications were made by comparison of both mass spectrum and their GC retention data with those of authentic compounds previously analyzed and stored in the data system. The volatile flavor components were also matched by co-injection with authentic compounds whenever available data were not obtained from retention indices and mass spectra.

GC-O. An Agilent 6890N GC equipped with a DB-Wax fused-silica capillary column (60 m × 0.53 mm i.d., film thickness = 1 μm, J&W Scientific), FID, and olfactometer (Gerstel GmbH & Co., Muelheim, Germany) including an olfactory detector port, an olfactory intensity device, and a humidifier was employed for GC-O. The oven conditions and injector and detector temperatures were the same as those given above for the GC. The carrier gas was nitrogen, and the split ratio was 10:1.

AEDA. The cold-pressed peel oil of kumquat was stepwise 3-fold diluted with acetone until the sniffer could not detect any significant odor in a run (*8*−*11*), and aliquots of the dilutions were evaluated by three sufficiently trained assessors. The highest dilution at which an individual component could be detected was defined as the flavor dilution (FD) factor for that odorant. The FD factor was expressed as a power of 3. On the basis of the AEDA results, relative flavor activity (RFA) was calculated using the equation (*8*, *11*)

$$\text{relative flavor activity} = \log 3^n/S^{0.5}$$

where *n* is the FD factor and *S* is the weight percentage of a component.

RESULTS AND DISCUSSION

Constituents of Kumquat Peel Oil. The detected constituents from the cold-pressed peel oil of kumquat are listed in **Table 1**, together with their weight percentages. The components are listed in order of their elution on the DB-Wax column. Eighty-two components, representing 99% of the total oil, were identified in the kumquat peel oil. The oil contained 27 hydrocarbons (96.64%), 8 aldehydes (0.2%), 25 alcohols (0.59%), 4 ketones (0.04%), 10 esters (1.26%), 5 oxides and epoxides (0.24%), and 3 acids (0.03%). The peel oil is characterized by a large presence of monoterpenes (96.46%).

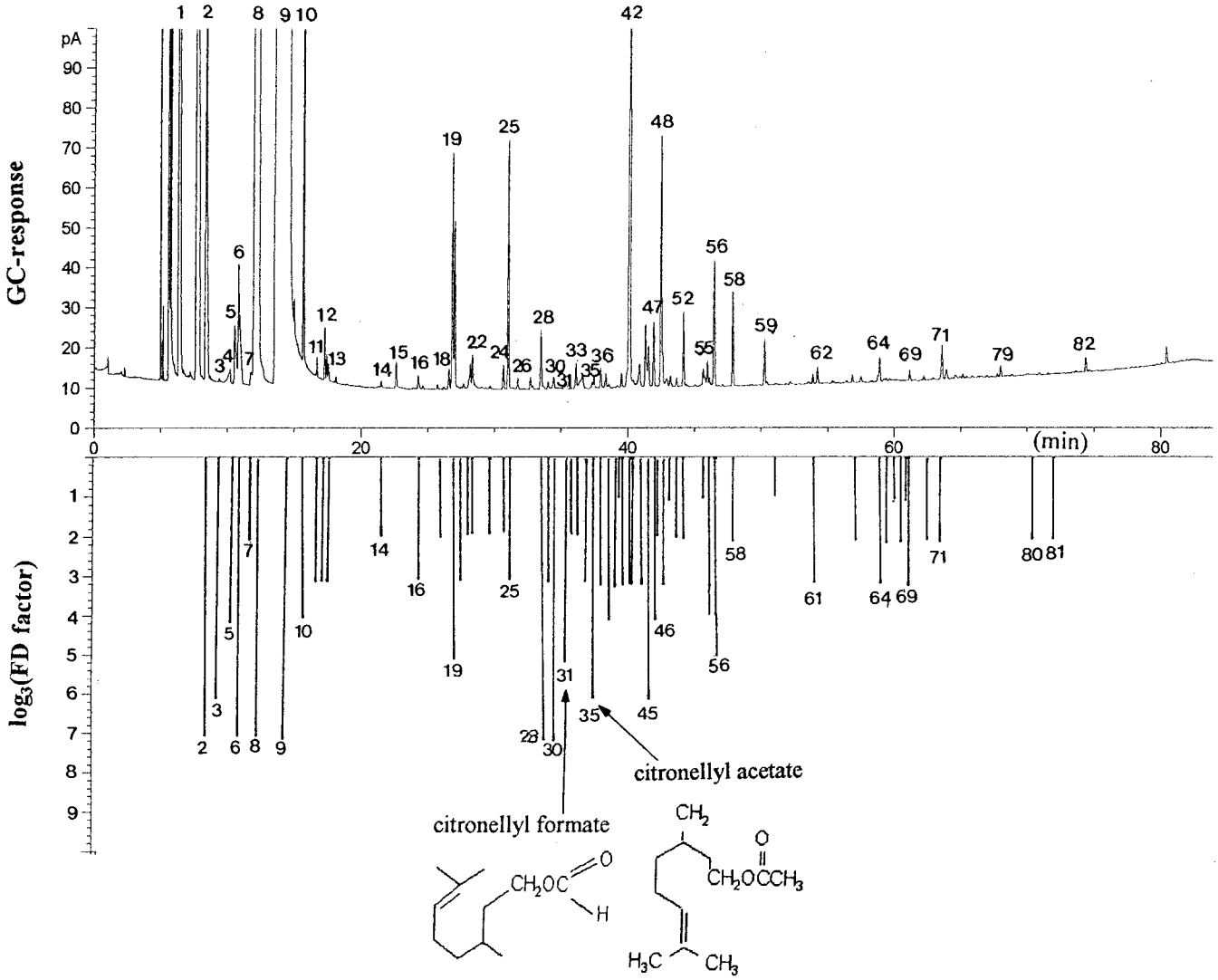

Figure 1. Gas (top) and FD chromatogram (bottom) of kumquat peel oil.

Table 2. Odor-Active Compounds (FD Factor ≥ 5) of Kumquat Peel Oil As Detected by GC-O

peak[a]	compound	concentration in the peel (mg/kg of fresh wt)	FD factor (3^n)	RFA
2	α-pinene	0.94	7	5.38
3	camphene	0.01	6	56.9
6	sabinene	0.24	7	10.8
8	myrcene	4.51	7	2.46
9	limonene	230	7	0.34
19	*trans*-linalool furanoid oxide	0.44	5	5.62
28	*β*-elemene	0.08	7	18.9
30	terpinen-4-ol	0.02	7	33.4
31	citrorellyl formate[b]	0.01	5	36.7
35	citronellyl acetate[b]	0.03	6	27.7
45	bicyclogermacrene	0.1	6	14.3
56	*p*-mentha-1-en-9-yl acetate	0.17	5	9.02

[a] Peak numbers correspond with compound numbers in **Table 1**. [b] Characteristic odor components of kumquat peel oil perceived at the sniffing port.

Limonene (93.73%) was the most abundant compound, followed by myrcene (1.84%) and ethyl acetate (1.13%). The proportion of limonene in kumquat was higher than those in other citrus fruits such as yuzu (78%), lemon (65%), Tahiti lime (52%), mandarin (86−90%), Korean satsuma mandarin (83−89%), orange (88−90%), and hyuganatsu (80−82%) (*7*, *14*−*17*).

Characteristic Odor Components of Kumquat. The olfactory profile of kumquat peel oil was characterized by fruity, sweet, and citrusy top notes, slowly emerging green and woody notes, and an oily note in the lasting undertone. The odor activity of each compound in a mixture identified by sniffing the GC effluent through a series of dilution was determined by AEDA technique. The aroma characters of eluting compounds from capillary GC are also described in this study (**Table 1**). The highest dilution of individual components detected by assessors was defined as its FD factor, and it was expressed as a power of 3.

GC-O techniques such as AEDA are based on the determination of odor-threshold values of the volatile components eluted from the GC column (*18*−*20*). The FD factor for a compound is the ratio of its concentration in the initial extract to its concentration in the most diluted extract in which odor was detected by GC-O. The higher FD factors were often related to the aroma-active compounds and to the top note of the aroma. However, the FD factor does not always coincide with characteristic odor components. A high FD factor of a compound may be caused by its high content in the sample. The RFA, based on the results of quantitative analysis and AEDA, was calculated in addition to the FD factor in this study. The RFA is also an important factor in the assessment of characteristic aroma together with the FD factor (*8*, *9*, *11*).

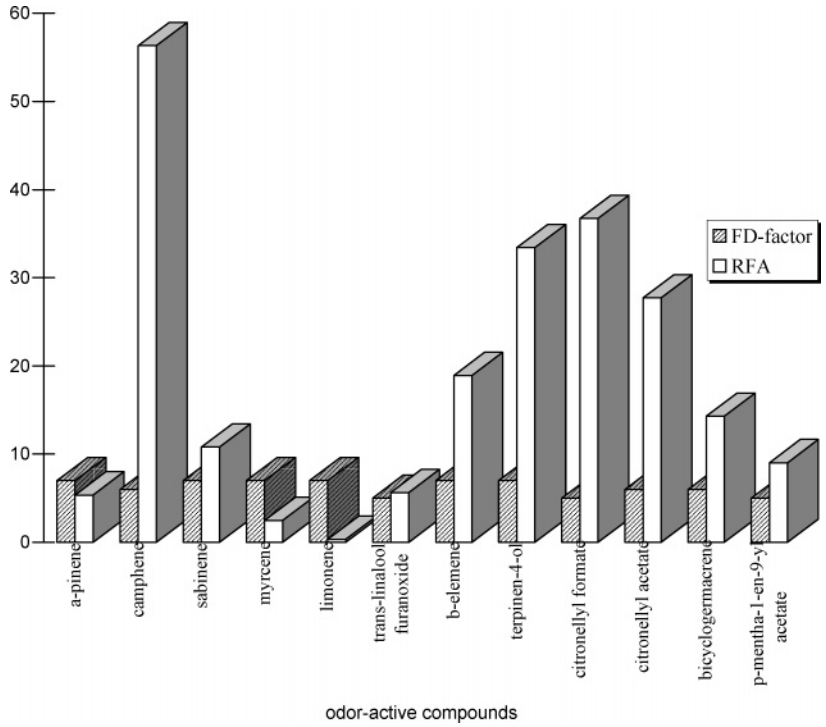

Figure 2. FD factors and RFA of odor-active compounds of kumquat peel oil.

The range of the FD factors of volatile constituents detected by AEDA was between 1 and 7 (**Table 1** and **Figure 1**). α-Pinene, sabinene, myrcene, limonene, *β*-elemene, and terpinen-4-ol showed the highest FD factors of 7, and camphene, citronellyl acetate, and bicyclogermacrene showed high FD factors of 6. The odor-active volatiles (FD factor ≥ 5) in kumquat oil are given in **Table 2** and **Figure 2**. Limonene is the most predominant component in the kumquat peel (230 mg/kg of fresh wt), and its FD factor is as high as 7. However, limonene showed a low RFA at 0.34, which means it has little importance in kumquat aroma. However, the concentrations of camphene, terpinen-4-ol, citronellyl formate, and citronellyl acetate were very low in the kumquat peel (≤0.03 mg/kg of fresh wt), but showed high FD factors (≥5) and RFA (>20). These four odorants are regarded as important aroma compounds of kumquat. These results suggest that minor components with low concentrations could contribute significantly to the characteristic aroma as reported previously (*8*−*11*, *21*).

The FD factor or RFA has proven to be a useful criterion for reconstruction of the original aroma from odor-active compounds detected by AEDA (*11*, *22*). However, the FD factor and RFA often have no relationship to the aroma character of each compound (*8*, *10*). The use of RFA is not for identifying characteristic odor components, but for consideration of relative contributions to odor activity. Therefore, a sniffing test of the original kumquat oil by on-line GC is adopted finally to determine characteristic odorants of kumquat aroma. Organoleptic evaluation by GC-sniffing was particularly enforced to several compounds with high FD factors (≥5) and/or high RFA (≥20). Citronellyl formate (FD factor = 5) and citronellyl acetate (FD factor = 6) were evaluated as kumquat-like odors by GC-sniffing. Among these esters, citronellyl acetate showed the odor most similar to kumquat, although its RFA (27.7) is lower than that of citronellyl formate (36.7).

As reported earlier in other *Citrus* species (*8*, *9*), limonene, despite being generally a major proportion of peel oil, had no importance in the flavor impact of their characteristic aroma. In this study, a minor ester compound, citronellyl acetate, is considered to be a characteristic odor component of kumquat peel oil.

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