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Character Impact Odorants of *Citrus* Hallabong [(*C. unshiu* Marcov × *C. sinensis* Osbeck) × *C. reticulata* Blanco] Cold-Pressed Peel Oil

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The volatile components of Hallabong ([*C. unshiu* Marcov × *C. sinensis* Osbeck] × *C. reticulata* Blanco) cold-pressed peel oil were quantitatively and qualitatively determined by use of two internal standards with GC, GC-MS, and GC−olfactometry. According to instrumental analysis by GC and GC-MS, limonene (90.68%) was the most abundant compound, followed by sabinene (2.15%), myrcene (1.86%), and γ-terpinene (0.88%). Flavor dilution (FD) factors of the volatile flavor components from Hallabong peel oil were determined by aroma extract dilution analysis. Furthermore, relative flavor activity was investigated by means of FD factor and weight percent. The highest FD factors were found for citronellal and citronellyl acetate, and δ-murollene showed a higher relative flavor activity. Results of sniff testing of the original oil and its oxygenated fraction revealed that citronellal, *cis*-β-farnesene, and citronellyl acetate were regarded as the character impact odorants of Hallabong peel oil, and citronellal gave the most odor-active character of Hallabong aroma.

KEYWORDS: ***Citrus* Hallabong [(*C. unshiu* Marcov × *C. sinensis* Osbeck) × *C. reticulata* Blanco]; gas chromatography−olfactometry; aroma extract dilution analysis; character impact odorants; citronellal**

INTRODUCTION

Fruits of the *Citrus* genus are important crops because of their nutritional and industrial uses. The genus *Citrus* consists of many species, all of which produce characteristic distinct flavor used in foods, perfumery, and cosmetics. In Korea, citrus fruits have long been used in traditional herbal medicine, especially for colds and coughs, and in bath products (*1*). Hallabong [(*C. unshiu* Marcov × *C. sinensis* Osbeck) × *C. reticulata* Blanco] is a new hybrid citrus crop in Korea and has been regarded as a citrus fruit with potential commercial value because of its attractive and pleasant aroma. The flesh of this fruit is juicy and has good taste with a sweet aroma. It matures in December in Jeju Province, an island off southeastern Korea. In 2001 the production of Hallabong in Korea was estimated to be 3901 tons. This fruit has a round shape with a neck, orange flesh and peel, and seedless flesh. Hallabong has a large diameter of 8−10 cm and an average weight of 350 g. This fruit has a sweet taste of 14−16 °Brix and an acids content of 1−1.2% (*2*).

Citrus hybrids are so variable as the result of hybridization of many fine-quality mandarins and sweet oranges, and many of these varieties are now being used sucessfully for juice production and as fresh fruit (*3*). In view of the commercial value and wide applications of these hybrid fruits, every essential characteristic information, especially about the flavor quality of the essential oil of these fruits, should be presented.

Gas chromatography−olfactometry (GC-O) of stepwise-diluted aroma extracts is a systematic approach to estimate the contribution of the most odor-active compounds presented in the overall odor by sniffing the GC effluent. Aroma extract dilution analysis (AEDA) is used for the detection of potent odorants in foods and is useful for determining the odor activity, quality, and potency of food aroma (*4*−*7*).

No attempt has been undertaken to identify the volatile components of Hallabong. In the present study, the quantitative and qualitative determination of Hallabong cold-pressed peel oil was carried out by GC, GC-MS, and GC-O, and their character impact odorants were elucidated by AEDA technique and sniffing test.

MATERIALS AND METHODS

Materials. Fresh Hallabong [(*C. unshiu* Marcov × *C. sinensis* Osbeck) × *C. reticulata* Blanco], which was harvested in December 2001, was provided by the Citrus Experiment Station Rural Development Administration, Jeju Province, Korea. The peel oil sample was prepared according to the cold-pressing method described by Choi and Sawamura (*8*) within 24 h of harvest. All of the fruits (∼5 kg) were sliced, and the mesocarp and albedo layers were peeled from the flavedo. The peel oils were extracted by hand-pressing the flavedo, and the peel oils were collected in 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. The yield of cold-pressed oil was 1.02% of the flavedo by weight. Authentic chemicals for co-injection in GC and MS were obtained from reliable commercial sources as follows: Aldrich Chemical Co. (Milwaukee, WI), Sigma Chemical

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Table 1. Volatile Flavor Components Identified in the Cold-Pressed Peel Oil of Hallabong

no.	compound	linear retention index DB-Wax	linear retention index DB-5	peak % (w/w)	identification	ref[a]	odor description	FD factor (3^n)
1	ethyl acetate	898		tr[b]	RI,[c] MS,[d] GC-O[e]	*18*		
2	α-pinene	1034	933	0.53	RI, MS, Co-GC,[f] GC-O	*18*, *19*	sweet, green	5
3	camphene	1077	953	0.01	RI, MS, Co-GC, GC-O	*18*, *19*	sweet, fruity	1
4	β-pinene	1124	981	0.10	RI, MS, Co-GC, GC-O	*18*, *19*	sweet, green	3
5	sabinene	1134	973	2.15	RI, MS, Co-GC, GC-O	*18*, *19*	sweet	4
6	myrcene	1168	991	1.86	RI, MS, Co-GC, GC-O	*18*, *19*	sweet, fruity	6
7	limonene	1234	1039	90.68	RI, MS, Co-GC, GC-O	*18*, *19*	green, citrus-like	6
8	*cis*-β-ocimene	1245	1043	0.16	RI, MS, Co-GC, GC-O	*18*, *19*	citrus-like	4
9	γ-terpinene	1262	1059	0.88	RI, MS, Co-GC, GC-O	*18*, *19*	herbaceous, fruity, sweet	5
10	*p*-cymene	1282	1027	0.02	RI, MS, Co-GC, GC-O	*18*, *19*	fruity, sweet	6
11	terpinolene	1297	1084	0.11	RI, MS, Co-GC, GC-O	*18*, *19*	fruity	3
12	tridecane	1312	1291	0.01	RI, MS, Co-GC, GC-O	*20*, *21*	citrus-like, fruity	2
13	tetradecane	1399	1116	0.06	RI, MS, Co-GC, GC-O	*18*, *19*, *21*	mild herbaceous, sweet	1
14	*cis*-linalool furan oxide	1453	1070	tr	RI, Co-GC, GC-O	*19*, *21*	sweet	1
15	(+)-*cis*-limonene oxide	1458	1138	0.01	RI, MS, Co-GC	*18*, *19*, *21*		
16	(−)-α-cubebene	1463	1345	tr	RI, MS, Co-GC, GC-O	*19–21*	mild waxy, woody	1
17	(+)-*trans*-limonene oxide	1470	1139	0.02	RI, MS, Co-GC, GC-O	*18*, *19*, *21*	mild green	4
18	menthone	1474		0.03	RI, MS, Co-GC, GC-O	*18*, *21*	fresh, green	4
19	*trans*-linalool furan oxide	1478	1172	0.02	RI, Co-GC, GC-O	*18*, *19*, *21*	fresh, green, fruity	5
20	citronellal[g]	1485	1161	0.19	RI, MS, Co-GC, GC-O	*18*, *20*, *21*	citrus peel-like	7
21	pentadecane	1504		0.50	RI, MS, Co-GC, GC-O	*18*, *20*, *21*	mild green	1
22	decanal	1538	1229	tr	RI, MS, Co-GC, GC-O	*18–21*	herbaceous	4
23	β-cubebene	1546	1018	0.02	RI, MS, Co-GC, GC-O	*18–21*	fruity, citrus-like	5
24	linalool	1554	1098	0.47	RI, MS, Co-GC, GC-O	*18–21*	fruity, sweet, green	5
25	octanol	1566	1072	0.01	RI, MS, Co-GC, GC-O	*18–21*	green	3
26	linalyl acetate	1569	1261	tr	RI, MS, Co-GC, GC-O	*18–21*	sweet, fruity	3
27	nonyl acetate	1585	1302	0.01	RI, MS, Co-GC, GC-O	*18–21*	fruity, sweet	5
28	β-elemene	1595	1393	0.01	RI, MS, Co-GC, GC-O	*18–21*	waxy, herbaceous	5
29	β-caryophyllene	1608	1428	0.04	RI, MS, Co-GC, GC-O	*18–21*	fruity, sweet	5
30	terpinen-4-ol	1616	1178	tr	RI, MS, Co-GC, GC-O	*18–21*	sweet, herbaceous	4
31	*l*-menthol	1626		tr	RI, MS, Co-GC, GC-O	*21*	fresh, green, cool	4
32	γ-elemene	1636		0.01	RI, MS, Co-GC, GC-O	*19*, *20*	green, oily	3
33	*cis*-β-farnesene[g]	1648		0.03	RI, MS, Co-GC, GC-O	*19–21*	green, citrus-like	5
34	citronellyl acetate[g]	1663	1357	0.04	RI, MS, Co-GC, GC-O	*18*, *20*, *21*	citrus-like, oily	7
35	*trans*-β-farnesene	1674		0.01	RI, MS, Co-GC, GC-O	*19–21*	oily, fruity, citrus-like	6
36	α-humulene	1680	1444	tr	RI, MS, Co-GC, GC-O	*18–21*	oily, fruity	4
37	δ-muurolene	1684		tr	RI, MS, Co-GC, GC-O	*18*, *21*	oily	5
38	decyl acetate	1691	1408	tr	RI, MS, Co-GC, GC-O	*18*, *20*, *21*	oily, fruity	3
39	neral	1695	1235	tr	RI, MS, Co-GC, GC-O	*18*, *19*, *21*	oily, citrus-like	3
40	terpinyl acetate	1700		0.05	RI, MS, GC-O	*18*, *21*	waxy	6
41	α-terpineol	1711	1185	0.12	RI, MS, Co-GC, GC-O	*18–21*	oily, fruity	5
42	dodecanal	1718	1401	0.01	RI, MS, Co-GC, GC-O	*18–21*	oily, herbaceous	5
43	germacrene D	1722		0.03	RI, MS, GC-O	*18–21*	oily, green	3
44	valencene	1726	1490	0.04	RI, MS, Co-GC, GC-O	*18*, *21*	oily, green	3
45	bicyclogermacrene	1738		0.11	RI, MS, GC-O	*18*, *21*	green	3
46	*cis*-linalool pyran oxide	1750		0.81	RI, Co-GC, GC-O	*21*	green, citrus-like	3
47	*trans*−2-undecenal	1761		0.03	RI, MS, Co-GC, GC-O	*21*	sweet, green	3
48	citronellol	1771	1435	0.15	RI, MS, Co-GC, GC-O	*18*, *19*, *21*	sweet, citrus-like	5
49	sesquiphellandrene	1780	1149	tr	RI, MS, GC-O	*18–21*	sweet, fruity, herbaceous	4
50	cumin aldehyde	1789		0.06	RI, MS, Co-GC, GC-O	*18*, *19*, *21*	green	3
51	perilla aldehyde	1797	1271	tr	RI, MS, Co-GC, GC-O	*19–21*	sweet, herbaceous	2
52	octadecane	1805		0.01	RI, MS, Co-GC, GC-O	*17*, *21*	sweet, fruity	3
53	methyl laurate	1813		0.04	RI, MS, Co-GC, GC-O	*21*	sweet, fruity	1
54	tridecanal	1824	1503	tr	RI, MS, Co-GC, GC-O	*19–21*	sweet, fruity	2
55	*p*-mentha-1-en-9-yl acetate	1834		0.01	RI, MS, GC-O	*18*, *21*	herbaceous, fruity	2
56	isopiperitone	1841		0.01	RI, MS, GC-O	*20*, *21*	sweet, fruity	1
57	*cis*-carveol	1846	1230	0.01	RI, MS, Co-GC, GC-O	*18*, *19*, *21*	citrus-like, fruity	1
58	geraniol	1862		0.01	RI, MS, Co-GC, GC-O	*18–21*	sweet	3
59	*trans*-2-dodecenal	1872	1462	tr	RI, MS, Co-GC, GC-O	*21*	citrus-like, oily	1
60	*trans*-carveol	1876		tr	RI, MS, Co-GC, GC-O	*19*, *21*	green, oily	2
61	perilla alcohol	1892		0.01	RI, MS, Co-GC	*18*, *21*		
62	dehydrocarveol	1941		tr	RI, MS, GC-O	*21*	oily, herbaceous	1
63	*p*-mentha-1-en-9-ol	1945	1486	tr	RI, MS, Co-GC, GC-O	*21*	fruity, herbaceous	2
64	tetradecenal	1989		tr	RI, MS, Co-GC	*21*		
65	caryophyllene oxide	1999	1573	0.01	RI, MS, Co-GC, GC-O	*21*	sweet, fruity	1
66	*cis*-nerolidol	2010	1565	tr	RI, MS, Co-GC, GC-O	*19*, *20*	waxy	2
67	methyl tetradecanoate	2034		tr	RI, MS, Co-GC, GC-O		waxy	1
68	*trans*-dodec-2-enol	2040		0.02	RI, MS, GC-O	*21*	oily	4
69	*trans*-nerolidol	2054	1539	0.01	RI, MS, Co-GC, GC-O	*19*, *20*	waxy	4
70	globulol	2061		tr	RI, MS	*18*		
71	octanoic acid	2083		0.01	RI, MS	*19*		
72	elemol	2089	1547	0.01	RI, MS, Co-GC, GC-O	*21*	green	1
73	viridiflorol	2102		tr	RI, MS, GC-O	*19*, *20*	sweet, green	1
74	cedrenol	2113	1604	0.01	RI, MS, Co-GC, GC-O	*19*, *20*	fruity	2

Table 1 (Continued)

no.	compound	linear retention index DB-Wax	linear retention index DB-5	peak % (w/w)	identification	ref[a]	odor description	FD factor (3^n)
75	spathulenol	2129		tr	RI, MS, GC-O	*19*	fruity, herbaceous	2
76	eugenol	2175	1351	tr	RI, MS, Co-GC, GC-O	*18*	herbaceous	1
77	γ-eudesmol	2185		0.02	RI, MS, GC-O	*18*, *19*	sweet, waxy	2
78	nonanoic acid	2202		tr	RI, MS, Co-GC, GC-O	*18*	green	2
79	α-cadinol	2211		tr	RI, MS, GC-O	*20*	green, waxy, woody	2
80	β-eudesmol	2246	1654	0.01	RI, MS, Co-GC, GC-O	*18*, *19*	green, woody	3
81	*trans*,*trans*-farnesyl acetate	2283		tr	RI, MS, GC-O	*19*, *20*	oily, waxy	3
82	cinnamyl alcohol	2300	1312	0.01	RI, MS, Co-GC, GC-O	*19*, *20*	oily	4
83	limonenediol	2334		tr	RI, MS, GC-O	*21*	green, waxy	3
84	*trans*,*trans*-farnesol	2371	1722	tr	RI, MS, Co-GC, GC-O	*19*	oily	4
85	nerol oxide	2385		tr	RI, MS, GC-O	*21*	oily	3
86	octadecanal	2400		tr	RI, MS, Co-GC, GC-O	*20*	oily	3
87	undecanoic acid	2407	1490	tr	RI, MS, Co-GC, GC-O	*20*	oily	3
88	undecanal	2444		0.01	RI, MS, Co-GC, GC-O	*20*	oily	3
	hydrocarbons							
	aliphatics (4)[h]			0.58				
	monoterpenes (10)			96.5				
	sesquiterpenes (13)			0.30				
	aldehydes							
	aliphatics (8)			0.05				
	terpenes (4)			0.25				
	alcohols							
	aliphatics (2)			0.03				
	monoterpenes (14)			0.78				
	sesquiterpenes (11)			0.06				
	ketones (2)			0.04				
	esters (10)			0.15				
	oxides and epoxides (7)			0.87				
	acids (3)			0.01				
	total			99.62				

[a] Reference number where identified earlier. [b] Trace, <0.005% (weight percentage). [c] Identification based on retention index. [d] Identification based on comparison of mass spectra. [e] Identification based on gas chromatography–olfactometry. [f] Identification based on co-injection with authentic compounds. [g] Hallabong-like odor compounds perceived at the sniffing port. [h] Number of identified compounds.

Co. (St. Louis, MO), PolyScience Co. (Nile, IL), AccuStandard, Inc. (New Haven, CT), Theta Co. (Newtown Square, PA), and Wako Pure Chemical Industries (Osaka, Japan). Some chemicals were provided by Bolak Co., Ltd. (Osan, Korea) and French-Korean Aromatics (Youngin, Korea).

Silica Gel Column Chromatography. The whole volatile concentrate was fractionated into nonpolar and polar compound fractions on a silica gel column (25 cm × 2 cm i.d.) packed with Wako gel Q-23 (Wako Pure Chemical Industries) (*9*). About 3 g of Hallabong peel oil was applied. The nonpolar and polar compound fractions were eluated with *n*-hexane (1 L) and diethyl ether (0.5 L), respectively. Each fraction was concentrated under reduced pressure at room temperature.

GC and GC-MS. An Agilent 6890N gas chromatograph equipped with a DB-Wax (60 m × 0.25 mm i.d., film thickness = 0.25 μm) fused silica capillary column (J&W Scientific, Folsom, CA) and a flame ionization detector (FID) was used. 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. A nonpolar column was also used for analysis: a DB-5 fused silica column (30 m × 0.25 mm i.d., film thickness = 0.25 μm, J&W Scientific). 1-Heptanol and methyl myristate were used as internal standards for quantitative analysis of Hallabong peel oil. The ratio of Hallabong peel oil for the two internal standards was 150:1:1. The weight percent of each peak was calculated according to the correlation factor to the FID (*10*). An oil sample of 1 μL was injected, and the injector split ratio was 50:1.

Gas chromatography combined 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.

Identification of Components. Components were identified by comparing their LRIs and matching their mass spectra with those of reference compounds in the data system of the Wiley library and NIST Mass Spectral Search program (ChemSW. Inc., NIST 98 version database) connected to a Varian Saturn 2000R MS. Whenever possible, the volatile flavor components were matched by co-injection with authentic compounds.

Sniffing Test by 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., Mülheim, Germany) including an olfactory detector port, an olfactory intensity device, and a humidifier was imployed for GC-O. The oven condition 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 1:10.

AEDA. The cold-pressed Hallabong peel oil was stepwise 3-fold diluted with acetone until the sniffer could not detect any significant odor in a run (*6*, *11*); aliquots of the dilutions were evaluated by two assessors. The highest dilution at which an individual component could be detected was defined as the flavor dilution (FD) factor for that odorant. On the basis of the AEDA results, relative flavor activity was calculated using the following equation (*7*, *12*): relative flavor activity = $\log 3^n/S^{0.5}$, where n is the FD factor and S is the weight percent of a component.

RESULTS AND DISCUSSION

Volatile Components of Hallabong Peel Oil. In the GC and GC-MS analyses of the Hallabong peel oil, 88 compounds,

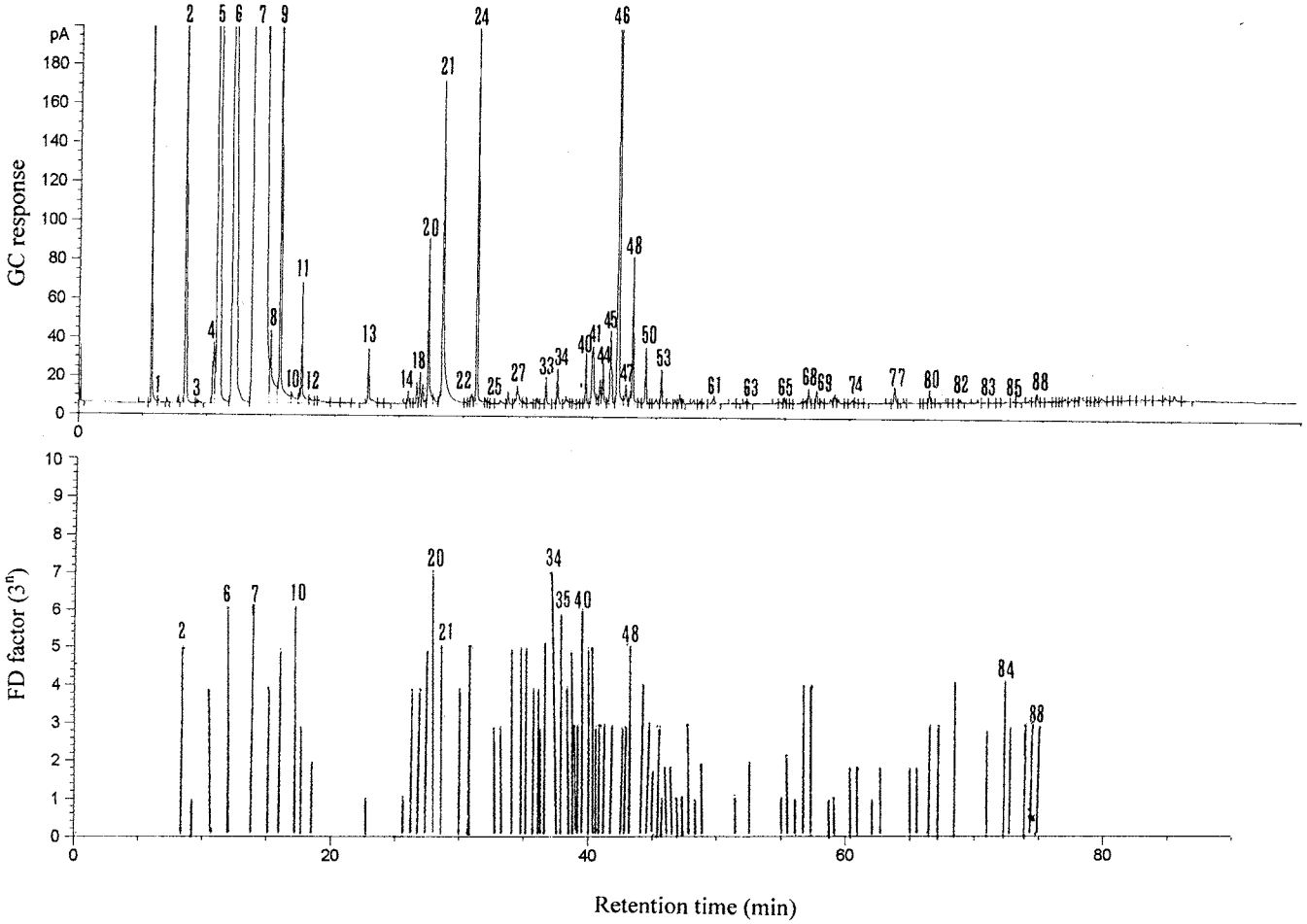

Figure 1. Gas chromatogram (top) and FD chromatogram (bottom) of odor-active volatiles of Hallabong cold-pressed peel oil.

representing 99.62% of the total oil, were characterized. Eighty-two peaks were confirmed by sniffing with GC-O. The detected constituents from the Hallabong peel oil are shown in **Table 1**, together with their peak percentage (w/w) based on the DB-Wax column and classification based on functional groups. The data are mean values of triplicates. The components are listed in order of their elution on the DB-Wax column. The gas chromatogram and the FD chromatogram of Hallabong peel oil are shown in **Figure 1**.

Limonene (90.68% of total oil) was the most abundant compound, followed by sabinene (2.15%), myrcene (1.86%), and γ-terpinene (0.88%). Monoterpene hydrocarbons were predominant in Hallabong peel oil. The Hallabong peel oil contained 0.58% aliphatic hydrocarbons and 0.3% sesquiterpene hydrocarbons of total oil. Among them, pentadecane and bicyclogermacrene were the most abundant components, and their peak percentages (w/w) were 0.5 and 0.11% of total oil, respectively. Among the oxygenated compounds, 27 alcohols were identified. Monoterpene alcohols accounted for 0.78%; linalool (0.47%), citronellol (0.15%), and α-terpineol (0.12%) were the major components. Citronellal (0.19%) was the principal aldehyde component of Hallabong volatiles. Two ketones (0.04%), 10 esters (0.15%), and 3 acids (0.01%) were detected from this oil. Seven oxides and epoxides were identified in Hallabong oil, and *cis*-linalool pyranoxide (0.81%) was the major component.

AEDA. AEDA is a human bioassay for determining the odor activity of each compound in a mixture by sniffing the GC effluent through a series of dilutions. Each volatile component is separated by GC, and the odors are determined at a sniffing port of the GC−olfactometer. Odor descriptions for compounds detected with GC-O are given in **Table 1**. As shown in this table, the range of FD factors of each peak was between 1 and 7.

Citronellal and citronellyl acetate showed the highest FD factor of 7, and myrcene, limonene, *p*-cymene, *trans*-β-farnesene, and terpinyl acetate showed FD factors of 6. The odor-active volatiles (FD $\geq$ 5) in Hallabong peel oil are given in **Table 2**. Very small peaks such as *p*-cymene, *trans*-linalool furan oxide, β-cubebene, nonyl acetate, β-elemene, β-caryophyllene, *cis*-β-farnesene, citronellyl acetate, *trans*-β-farnesene, δ-murollene, and dodecanal on the gas chromatogram were detected as predominant peaks (FD factors of 5 and 6) on the FD chromatogram of **Figure 1**. The higher FD factors were often related to the aroma-active compounds. These compounds having high FD factors were predominant peaks on the FD chromatogram, but some of them did not have the characteristic flavor of Hallabong by the sniffing test. The high FD factors of these compounds may be caused by their high contents in Hallabong fruit.

The GC-O technique of AEDA is based on the determination of odor-threshold values of the volatile components eluted from the GC column (*5*, *13*). The higher FD factors are often related to the top note of the aroma. However, the FD factor also depends on the concentration. Therefore, the relative flavor activity was also determined as a more realistic expression because the FD factor does not always coincide with characteristic odor-active compounds. Limonene is the most predominant component (222.1 mg/kg of fresh weight), and its FD factor is as high as 6. However, limonene showed a low relative flavor

Table 2. Most Odor-Active Volatiles (FD ≥ 5) in Cold-Pressed Peel Oil of Hallabong As Detected by GC-O

peak[a]	compound	concn (μg/kg of fresh wt)	FD factor (3^n)	rel flavor activity
2	α-pinene	1288	5	3.3
6	myrcene	4565	6	2.1
7	limonene	222071	6	0.3
8	*cis*-β-ocimene	387	5	6.0
9	γ-terpinene	2159	5	2.5
10	*p*-cymene	61	6	18.2
19	*trans*-linalool furan oxide	57	5	15.6
20	citronellal	475	7	7.6
21	pentadecane	1220	5	3.4
23	β-cubebene	45	5	17.7
24	linalool	1141	5	3.5
27	nonyl acetate	22	5	25.4
28	β-elemene	33	5	20.6
29	β-caryophyllene	94	5	12.2
33	*cis*-β-farnesene	70	5	14.1
34	citronellyl acetate	89	7	17.6
35	*trans*-β-farnesene	34	6	24.3
37	δ-muurolene	10	5	36.6
40	terpinyl acetate	112	6	13.4
41	α-terpineol	293	5	6.9
42	dodecanal	17	5	28.5
48	citronellol	365	5	6.2

[a] Peak numbers correspond with peak numbers in **Table 1**.

activity of 0.3, which means it has little importance in Hallabong flavor. *p*-Cymene, *trans*-linalool furan oxide, β-cubebene, nonyl acetate, β-elemene, *cis*-β-farnesene, citronellyl acetate, *trans*-β-farnesene, δ-muurolene, terpinyl acetate, and dodecanal showed high relative flavor activity (≥10). On the basis of an estimation of relative flavor activity, these compounds were regarded as important contributors to Hallabong flavor. These results suggest that minor components often contribute significantly to characteristic flavor.

Sniff testing is used not only for AEDA but also for expressing the aroma character of each component. Organoleptic response to a compound, in general, depends on its concentration (*14*). The FD factor or relative flavor activity has proven to be a useful criteria for reconstruction of the original aroma from odor-active compounds detected by AEDA. The concept of relative flavor activity used in this study was defined as a new odor unit calculated by use of FD factors instead of the odor threshold values. However, the FD factor and relative flavor activity often have no relation to the aroma character of a compound (*14*). In other words, even if the FD factor of one compound is not comparatively high, it often contributes primarily to the original odor of the food (*15*). Thus, the sniffing test of the original essential oil by on-line GC is an effective means of determining character impact odorants of an aroma. As shown in **Table 1**, citronellal (peak 20), *cis*-β-farnesens (peak 33), and citronellyl acetate (peak 34) were estimated as having a Hallabong-like odor by the sniff test. This result was also confirmed in the analysis of an oxygenated fraction prepared from cold-pressed Hallabong peel oil by silica gel column chromatography.

In this study the character impact odorants of Hallabong were screened by FD factor in the first instance, and then relative flavor activity was adopted. However, the relative flavor activity data are not sufficient to assess the degree of actual contribution of a compound to original aroma. The use of relative flavor activity is not for the determination of characteristic flavor components but for consideration of relative contribution in flavor activity. Thus, the sniffing test of the original cold-pressed oil of Hallabong was adopted by on-line GC. Citronellal and citronellyl acetate, which were evaluated as Hallabong-like odors by the sniffing test, had the highest FD factors of 7. *cis*-β-Farnesene, having an FD factor of 5 and a relative flavor activity of 14.1, was also regarded as an odor-active compound of Hallabong peel oil. As a result of careful sniff testing, citronellal had the odor most simlar to Hallabong, although its relative flavor activity is low at 7.6. From these experiments it is concluded that comprehensive evaluation of the flavor should be accomplished by simultaneous chemical and sensory analyses.

It was well-known that the characteristic flavor of grapefruit is nootkatone and its derivatives (*16*). Citral is known to be the typical aroma of lemon (*17*). *Citrus* Hallabong has a characteristic sweet flavor and plenty of juice, much like sweet orange and ponkan. This fruit is regarded as the finest citrus of Jeju in Korea. The results reported here suggest that citronellal, *cis*-β-farnesene, and citronellyl acetate were regarded as the character impact odorants of Hallabong peel oil, and citronellal gave the most odor-active character of Hallabong aroma.

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