Journal of Oleo Science
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doi : 10.5650/jos.ess19296
J. Oleo Sci. **69**, (6) 643-648 (2020)

JOURNAL OF OLEO SCIENCE
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NOTE

Composition and Monthly Changes of the Volatile Constituents in the Sour Hetsuka-daidai Citrus Peel

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Abstract: Sour citrus are prized for their flavor and fragrance. This work identified the components of the peel oil of Hetsuka-daidai (*Citrus* sp. *hetsukadaidai*), a special sour citrus that is native to the southern part of the Osumi peninsula, Kagoshima, Japan. These compounds were compared to those identified from the peels of six other major sour citrus: lime (*Citrus latifolia*), lemon (*Citrus limon*), Yuzu (*Citrus junos*), Kabusu (*Citrus aurantium*), Kabosu (*Citrus sphaerocarpa*), and Sudachi (*Citrus sudachi*). Peel oil contents were analyzed for the duration of four months during harvest season to investigate the differences in peel oil/fragrance during ripening. These results could facilitate the development of preferred flavor and scent profiles using local species.

Key words: citrus, essential oils, GC-MS, monthly change, Hetsuka-daidai

1 Introduction

Sour citrus fruits are widely cultivated for use as food and beverages and in fragrances throughout the world. Lemon and lime (particularly Mexican and Tahiti lime) are among the most widely known in the world[1, 2], while Yuzu, Kabosu and Sudachi are popular in Japan. There are however other rare species of citrus that have been cultivated and utilized by the local communities. Hetsuka-daidai is one such species and is bred only in the Hetsuka Community in Kagoshima Prefecture, Japan[3−5]. This citrus was the 57th product to receive a Geographical Indication (GI) certificate from the Japanese ministry of Agriculture, Forestry and Fisheries[6]. The essential oils of its peel are widely used in Japanese food industries. To keep pace with this demand, the production of Hetsuka-daidai was recently estimated to be 50 tons/year. Hetsuka-daidai is smaller (globose fruit; *ca* 5 cm in horizontal diameter) than daidai (Kabusu) with thinner and smoother skin. Although the odor of the citrus has been likened to lime, the fruit and peel of these two species are distinct and will thus confer unique properties to the products derived from their use. The purpose of this work was to identify and characterize the volatile components in the Hetsuka-daidai peel, using gas chromatography-mass spectrometry (GC-MS), and to compare this profile with those of the six other major sour citrus: lime, lemon, Yuzu, Kabusu, Kabosu and Sudachi. In addition, monthly changes to the volatile compositions of the Hetsuka-daidai peel were evaluated to determine how they fluctuated based on the season. Characterization of the essential oils from this citrus peel will provide important information for the development of new products in the food and cosmetic industries.

2 Experimental Procedures

2.1 Plant samples

Immature and mature fruits of Hetsuka-daidai were col-

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Accepted February 21, 2020 (received for review November 20, 2019)
Journal of Oleo Science ISSN 1345-8957 print / ISSN 1347-3352 online
http://www.jstage.jst.go.jp/browse/jos/ http://mc.manusriptcentral.com/jjocs

lected from citrus farms in the Kishira area, Kagoshima, Japan, between September and December 2018. Two immature sour citrus, Kabusu and Kabosu, were collected from the Toso Orchard of the Experimental Farm, Faculty of Agriculture, Kagoshima University, and three immature sour citrus, lemon, Yuzu and Sudachi, were collected from farms at the Division of Citrus Research, Institute of Fruit Tree and Tea Science, NARO in October 2018. Tahiti lime was collected from the Toso Orchard of the Experimental Farm, Faculty of Agriculture, Kagoshima University in September 2019. Each species was picked from the same tree and peels were obtained manually or with the aid of a peeler. The fresh peels were stored at −80℃ prior to extraction.

2.2 Extraction of essential oils

Essential oils were extracted from the citrus peels (250～300 g) by steam distillation[7], immediately after harvesting. Each extraction was repeated 4-5 times. The distilled oils were stored under nitrogen at −30℃ in sealed tubes prior to analysis. All the isolated oils were pale yellow in color.

2.3 Gas chromatography-mass spectrometry (GC-MS) analysis

GC-MS analysis of the essential oils was performed on a 7890A/5975C GC/MSD system (Agilent Technologies, CA, USA) equipped with a DB-WAX capillary column (J&W Scientific Inc., CA, USA; 30 m×0.25 mm (i.d.), 0.25 μm film thickness). The injection port was heated to 250℃. The column was heated to 50℃ for 5 min and the temperature was increased at 10℃/min to a final temperature of 240℃, which was maintained for 10 min. The MS conditions were as follows: ionization voltage 70 eV; emission current 40 mA; mass range 50–800 amu; scan rate 1.0 scan/s. Helium was used as the carrier gas at a flow rate of 1 mL/min. The split ratio was 50:1. Each analysis was performed in triplicate.

2.4 Identification of volatile components

The volatile components were identified on the basis of the retention indices (RI) determined by co-injection with a reference set of a homologous series of *n*-alkanes (C7～C40 Saturated Alkanes Standard (Supelco)) under identical experimental conditions. Compound identification was confirmed by comparing the mass spectra with those of the National Institute of Standards and Technology (NIST) library[8] and a homemade MS library of pure substances and components of known essential oils, as well as by comparing their RI with literature values.

3 Results and Discussion

3.1 Volatile constituents

In the immature Hetsuka-daidai oil collected in October, 45 volatile compounds, accounting for 99.62% of the integrated peak area, were identified (Table 1). The essential oil contained high amounts of monoterpene hydrocarbons (14 components, 95.26%). Among these, limonene (69.31%) was the major compound in the oil. Other notable monoterpenes were γ-terpinene (15.71%), β-myrcene (2.49%), α-pinene (1.92%) and β-pinene (1.49%). Further components in the oil were identified as five monoterpene alcohols (1.67%), ten sesquiterpenes (1.56%), four aldehydes (0.28%), eight other alcohols (0.25%), two ketones (0.32%) and two acetates (0.16%). Linalool was the principal monoterpene alcohol in the oil. Other compounds were present in minor quantities. In comparison, lime had 52 components (14 monoterpene hydrocarbons, 80.63%; seven monoterpene alcohols, 3.47%; 12 sesquiterpene hydrocarbons, 2.92%; 19 others, 12.31%). Lemon contained 41 components (14 monoterpene hydrocarbons, 92.14%; eight monoterpene alcohols, 2.21%; four sesquiterpene hydrocarbons, 0.47%; 15 others, 4.93%). Yuzu oil was composed of 39 compounds (12 monoterpene hydrocarbons, 93.63%; six monoterpene alcohols, 3.17%; 13 sesquiterpene hydrocarbons, 2.11%; eight others, 0.64%). The oil from Kabusu peels contained 38 compounds (11 monoterpene hydrocarbons, 92.70%; five monoterpene alcohols, 2.35%; four sesquiterpene hydrocarbons, 0.32%; 18 others, 2.11%). Kabosu oil was comprised of 42 compounds (12 monoterpene hydrocarbons, 95.25%; five monoterpene alcohols, 0.44%; ten sesquiterpene hydrocarbons, 2.01%; 15 others, 2.02%). Sudachi had 52 compounds in its oil (13 monoterpene hydrocarbons, 88.59%; seven monoterpene alcohols, 1.02%; 14 sesquiterpene hydrocarbons, 7.21%; 18 others, 1.93%). Ketones, in particular carvone, were detected at higher levels in Hetsuka-daidai than in the other sour citrus oils except for Sudachi, while aldehydes were virtually absent in the Hetsuka-daidai oil. Although the fragrance of Hetsuka-daidai is said to be similar to that of lime[6], but the fragrances of the essential oils of both citrus peels are completely different. In fact, the composition pattern of the Hetsuka-daidai oil was considerably different from that of lime; in particular, the content of β-pinene in Hetsuka-daidai (1.49%) was much lower than in lime (9.81%). Therefore, it would be expected that Hetsuka-daidai smells less pine-like than lime, as β-pinene has a woody, pine-like aroma. Moreover, compounds such as terpineols, neral, geranial, and neryl acetate, which greatly contribute to the classic lime scent, were also considerably lower in Hetsuka-daidai than in lime. Thus, despite associations between Hetsuka-daidai and lime fragrances, the composition of their oils are notably distinct. Among the six sour citrus, the composition pattern of Hetsuka-daidai oil was most similar to that

Table 1 The volatile constituents of the oils of Hetsuka-daidai peels over a four-month collection period with lime, lemon, Yuzu, Kabusu, Kabosu, and Sudachi peels[a].

No.	RI	Compounds	Hetsuka－daidai				lime	lemon	Yuzu	Kabusu	Kabosu	Sudachi
			Sep.	Oct.	Nov.	Dec.						
		Monoterpene hydrocarbons										
1	1017	α-Pinene	1.94	1.92	1.85	1.83	1.67	2.32	1.84	0.86	0.81	1.78
2	1021	α-Thujene	0.62	0.62	0.61	0.60	0.40	0.54	0.44	−	0.13	0.29
3	1057	Camphene	−[c]	−	−	−	0.07	0.08	−	−	−	−
4	1104	β-Pinene	1.51	1.49	1.46	1.29	9.81	11.14	0.90	1.11	0.30	0.59
5	1119	Sabinene	0.26	0.25	0.25	0.21	1.07	1.79	0.20	0.24	0.08	0.42
6	1147	δ-3-Carene	0.43	0.38	0.39	0.37	−	−	−	−	−	−
7	1165	β-Myrcene	2.57	2.49	2.43	2.43	1.35	1.80	2.90	2.53	16.62	2.97
8	1178	α-Terpinene	0.46	0.47	0.55	0.54	0.75	0.57	0.49	0.24	0.16	0.21
10	1200	Limonene	68.82	69.31	69.85	70.60	47.75	59.58	70.12	86.06	71.72	60.30
11	1206	β-Phellandrene	0.43	0.40	0.40	0.39	0.48	0.53	4.36	0.53	0.41	8.22
13	1238	(*Z*)-β-Ocimene	t[b]	t	t	t	0.05	0.09	−	0.08	−	t
14	1250	γ-Terpinene	15.48	15.71	15.42	14.85	15.54	12.43	10.59	0.17	4.28	5.12
15	1256	(*E*)-β-Ocimene	0.90	0.96	1.12	1.05	0.13	0.20	0.28	0.81	0.15	0.50
16	1274	*p*-Cymene	0.63	0.35	0.26	0.22	0.32	0.25	0.84	−	0.33	6.08
17	1288	α-Terpinolene	0.87	0.86	0.91	0.87	1.23	0.80	0.69	0.08	0.26	2.12
		Subtotal	94.95	95.26	95.52	95.27	80.63	92.14	93.63	92.70	95.25	88.59
		Monoterpene alcohols										
36	1551	Linalool	1.65	1.33	1.20	1.32	0.55	0.31	2.39	1.53	0.09	0.22
38	1572	(*Z*)-*p*-2-menthen-1-ol	−	−	−	−	0.24	0.14	t	−	−	t
43	1609	Terpinen-4-ol	0.17	0.11	0.08	0.09	0.84	0.76	0.25	0.08	−	0.11
45	1635	(*E*)-*p*-2-Menthen-1-ol	−	−	−	−	t	t	t	−	t	t
52	1706	α-Terpineol	0.23	0.13	0.10	0.11	1.20	0.60	0.41	0.32	0.17	0.11
64	1770	β-Cirtonellol	−	−	−	−	−	t	−	−	−	0.47
69	1804	Nerol	0.10	0.07	0.06	0.06	0.49	0.19	−	0.21	0.13	t
72	1854	Geraniol	t	t	−	t	0.11	0.16	t	0.21	t	−
		Subtotal	2.19	1.67	1.44	1.60	3.47	2.21	3.17	2.35	0.44	1.02
		Sesquiterpene hydrocarbons										
28	1475	δ-Elemene	t	t	t	t	0.21	−	0.11	−	−	0.50
31	1499	Copaene	0.22	0.22	0.23	0.20	−	0.09	0.09	−	−	0.32
34	1544	β-Cubebene	t	t	t	t	−	−	t	−	t	t
39	1582	α-Guaiene	−	−	−	−	−	−	−	−	−	0.11
40	1588	α-Bergamotene	−	t	−	t	0.99	0.29	−	−	t	−
41	1593	β-Elemene	0.08	0.07	0.07	0.07	0.16	−	0.07	−	0.17	1.93
42	1603	β-Caryophyllene	0.47	0.51	0.49	0.42	0.60	−	0.18	0.06	0.47	0.23
44	1616	β-Bisabolene	−	−	−	−	0.06	−	−	−	−	−
46	1643	γ-Elemene	−	−	−	−	t	−	−	−	−	0.15
48	1667	(*E*)-β-Farnesene	−	−	−	−	0.11	0.05	0.46	t	0.65	0.46
49	1680	α-Humulene	0.09	0.09	0.08	0.07	0.10	t	t	−	0.09	0.50
54	1720	Germacrene D	0.26	0.28	0.28	0.26	0.11	−	0.16	0.17	0.10	0.64
57	1732	α-Muurolene	−	−	−	−	−	−	t	−	−	0.41
59	1743	Bicyclogermacrene	−	−	−	−	−	−	0.67	−	0.09	−

Table 1 Continued.

No.	RI	Compounds	Hetsuka－daidai				lime	lemon	Yuzu	Kabusu	Kabosu	Sudachi
			Sep.	Oct.	Nov.	Dec.						
61	1750	α-Farnasene	–	–	–	–	0.24	–	–	–	–	0.90
63	1766	δ-Cadinene	0.24	0.25	0.24	0.23	–	–	0.12	t	0.33	0.26
65	1773	δ-Selinene	–	–	–	–	0.08	–	–	–	–	–
66	1775	β-Sesquiphellandrene	–	–	–	–	–	–	t	–	0.06	–
71	1843	Germacrene B	t	t	t	t	0.22	–	0.10	–	–	0.76
		Subtotal	1.48	1.56	1.50	1.38	2.92	0.47	2.11	0.32	2.01	7.21
		Aliphatic aldehydes										
18	1295	Octanal	–	–	t	t	t	0.06	–	0.24	0.16	0.97
23	1394	Nonanal	–	t	t	t	0.05	0.15	–	0.07	0.12	t
32	1502	Decanal	–	–	–	–	0.21	–	–	0.51	0.76	–
53	1713	Dodecanal	–	–	–	–	0.13	t	–	t	0.09	–
		Subtotal	–	t	t	t	0.43	0.25	–	0.86	1.13	1.00
		Aldehydes										
29	1483	Citronellal	t	t	t	0.05	t	0.06	–	0.20	–	0.07
50	1689	Neral	0.12	0.10	0.12	0.10	2.81	1.49	–	0.13	t	–
58	1739	Geranial	0.15	0.14	0.14	0.13	3.54	1.91	–	0.12	0.07	–
67	1789	Cuminaldehyde	–	–	–	–	t	t	–	–	–	0.07
68	1795	Perillaldehyde	t	–	–	t	0.07	t	–	0.07	–	t
		Subtotal	0.33	0.26	0.30	0.31	6.49	3.51	–	0.51	0.12	0.17
		Aliphatic alcohols										
19	1313	1-Hexanol	–	t	0.09	0.19	–	–	–	0.05	–	–
21	1347	(*Z*)-3-Hexen-1-ol	0.09	0.06	0.07	–	–	0.08	0.09	0.36	0.15	0.14
		Subtotal	0.09	0.11	0.15	0.19	–	0.08	0.09	0.42	0.15	0.14
		Sesquiterpene alcohols										
79	2047	(*E*)-Nerolidol	t	0.06	0.08	0.10	–	–	0.10	0.17	0.08	t
82	2095	Elemol	t	t	t	t	–	–	t	–	–	0.05
88	2190	γ-Eudesmol	0.05	0.05	t	t	0.07	–	–	–	0.05	0.05
90	2210	τ-Muurolol	–	t	t	–	–	–	t	–	–	–
93	2234	α-Bisabolol	–	–	–	–	0.13	t	–	–	–	–
94	2245	α-Eudesmol	–	–	–	–	–	–	–	–	–	t
95	2256	α-Cadinol	t	t	t	t	–	–	0.05	–	t	0.07
		Subtotal	0.15	0.22	0.22	0.21	0.20	t	0.20	0.17	0.17	0.24
		Diterpene alcohol										
115	2604	Phytol	t	0.06	0.06	t	0.07	0.06	–	0.09	0.07	0.05
		Subtotal	t	0.06	0.06	t	0.07	0.06	–	0.09	0.07	0.05
		Ketones and Phenols										
33	1524	Camphor	–	–	–	–	t	t	–	–	–	–
60	1746	Carvone	0.34	0.27	0.28	0.31	–	–	–	0.06	–	0.28

Table 1 Continued.

No.	RI	Compounds	Hetsuka－daidai Sep.	Oct.	Nov.	Dec.	lime	lemon	Yuzu	Kabusu	Kabosu	Sudachi
89	2197	Thymol	–	–	–	–	–	–	0.33	–	–	–
92	2225	Carvacrol	t	–	–	–	–	–	–	–	–	t
112	2542	Nootkatone	t	t	t	0.11	t	–	–	–	–	–
		Subtotal	0.39	0.32	0.33	0.42	t	t	0.33	0.06	–	0.32
		Esters and Oxides										
9	1190	2,3-Dehydro-1,8-cineole	–	–	–	–	t	–	–	–	–	–
12	1208	1,8-Cineole	–	–	–	–	0.63	–	–	–	–	–
26	1450	(*E*)-Linalool oxide	t	–	–	–	–	–	–	–	–	–
27	1462	(*Z*)-Limonene oxide	–	–	–	–	–	–	–	–	–	t
30	1486	(*Z*)-Linalool oxide	–	–	–	–	–	–	t	–	–	–
37	1558	Linalyl acetate	–	–	–	–	–	–	–	0.84	t	–
47	1656	Citronellyl acetate	t	t	0.05	t	0.08	t	t	0.13	t	0.07
56	1730	Neryl acetate	0.11	0.12	0.12	0.12	4.02	0.77	–	0.25	0.24	–
62	1760	Geranyl acetate	–	–	–	–	0.36	0.22	–	0.44	0.06	–
75	1913	Perillyl acetate	–	–	–	–	–	–	–	0.06	–	–
77	2004	Caryophyllene oxide	–	–	–	–	–	–	–	–	–	t
80	2063	α-Humulene epoxide Ⅱ	–	–	–	–	–	–	–	–	–	t
		Subtotal	0.17	0.16	0.18	0.17	5.12	1.02	t	1.71	0.38	0.16
		Not Identified	0.22	0.38	0.27	0.36	0.63	0.22	0.42	0.81	0.29	1.10
		Total (%)	100.00	100.01	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00

[a] SD (±) were insignificant and excluded from the Table to avoid congestion. All compounds were identified by Kovats index (RI = retention index) and GC-MS library search.
[b] Detected < 0.05 %.
[c] Not detected.

of Yuzu, with differences arising from a slightly lower content of β-phellandrene and higher concentrations of β-pinene and γ-terpinene in Hetsuka-daidai as compared to Yuzu.

3.2 Monthly changes in the composition of Hetsuka-daidai oil

The relative compositional changes of the peel oil of Hetsuka-daidai between September and December are presented in Table 1. There was little to no change detected in monoterpene hydrocarbon content over this period (94.95% vs 95.52%). There seems to be a very slight increase in limonene (68.82% vs 70.60%) which is the main fragrance of citrus fruit. Due to an increase of limonene, which is the main component of the oil, the proportion of most other compounds appears to decrease. α-pinene (1.94% vs 1.83%), β-pinene (1.51% vs 1.29%), β-myrcene (2.57% vs 2.43%), γ-terpinene (15.48% vs 14.85%) and *p*-cymene (0.63% vs 0.22%) were notably decreased. On the other hand, α-terpinene and β-ocimene increased from 0.46% to 0.54% and from 0.90% to 1.05%, respectively. Among monoterpene alcohols, the proportion of linalool decreased appreciably from 1.65% to 1.32%. The proportion of sesquiterpene hydrocarbons decreased from 1.48% to 1.38%.

4 Conclusion

The volatile components of the essential oils of Hetsuka-daidai peel were investigated and compared with those of six other major sour citrus. Forty-five compounds were identified and quantified, accounting for 99.62% of the total peel oil. The fragrance and composition of Hetsuka-daidai and Tahiti lime oils were notably distinct, unlike what has been reported officially. In particular, β-pinene, neral, geranial, neryl acetate, and terpineols were much less abundant in Hetsuka-daidai. Amongst these sour citrus, the compositional pattern of the essential oils of Hetsuka-daidai was most similar to that of Yuzu. It is possible that Hetsuka-daidai could be used as an alternative ingredient in ponzu (Japanese salad dressing), Yuzu-Kosho (literally Yuzu and pepper; Japanese spicy sauce) and Yuzu tea (Korean popular tea). The changes in the compounds in the Hetsuka-daidai oil from September to December were investigated. The percentage of limonene, α-terpinene, and β-ocimene increased, and α-pinene, β-pinene, β-myrcene, γ-terpinene, *p*-cymene and linalool decreased during this period. The slow and weak changes in the composition of Hetsuka-daidai oil realized the very long harvesting and usable time. This work provides that essential first knowledge and will hopefully lead to further investigations and applications of this valued product.

Acknowledgment

We are grateful to Kazuki Kajiwara, NPO Rikuno-Takarajima Kishira, and the National Agriculture and Food Research Organization for providing samples. We thank Tsuyoshi Sawada and Kousei Migita (Division of Instrumental Analysis, Research Support Center, Kagoshima University) for technical support. This work was supported by Grants-in-Aid for Scientific Research (No.18K06732) from the Ministry of Education, Culture, Sports, Science and Technology, Japan, and by the Cooperative Research Program of the "Network Joint Research Centre for Materials and Devices" (No. 20181302 and 20191349).

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