

Volatile Constituents of Japanese and Korean *Udo* (*Aralia cordata* Thunb.) and Butterbur (*Petasites japonicus* Miq.)

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Abstract: The aroma constituents from two kinds of wild vegetables collected respectively in Japan and Korea were investigated by gas chromatography (GC) and GC-mass spectrometry, of which one is *udo* (*Aralia cordata* Thunb.) and the other is butterbur (*Petasites japonicus* Miq.). The volatile constituents were extracted by a simultaneous distillation extractor devised by Likens and Nickerson. The steam distillation-extraction was carried out at atmospheric pressure, with diethyl ether as a solvent.

There were 45 and 114 compounds in Japanese and Korean *udo*, respectively. Among those compounds the major components were in common monoterpenes such as α -pinene, β -pinene and sabinene, amounting to 84 to 88% of relative peak area. It is presumed that these are deeply related to the flavor of *udo*. Beyond them, the following compounds were detected in Korean *udo*: 1-methyl-4-(1-methylethyl) benzene, 3-tridecen-1-yne, 2, 3, 4, 5, 6, 7-hexahydro-1H-inden-2-ol, octadecylo-4-methyl-6-(1-methylethyl)-3, 5-dioxo-4a(2H)-naphthalenecarboxyaldehyde.

The components detected by GC were at least 53 in Japanese butterbur and 85 in Korean one. There were great differences between the components of Japanese and Korean butterbur. Olefinic hydrocarbons such as 1-nonene and 2-undecene were largely shown in Japanese butterbur, while sesquiterpenes such as eremophilene, valencene, β -bisabolene, β -cadinene, α -caryophyllene and β -cubebene were more abundant in Korean one.

Introduction

Among wild vegetables, either *udo* or butterbur ("Fuki" in Japanese), is one of the most popularly edible plants, being distributed in Eastern Asia, especially in Japan, Korea and China. In these countries the stalk of *udo* is often used for green salad with dressing and that of butterbur is cooked. Most of Japanese and Korean have enjoyed eating the wild vegetables in the spring as their edible quality is the best during this season. *Udo* and butterbur give out their specific fragrance of mild intensity. There seems to be few papers on the volatile constituents of *udo*^{1,2)} and butterbur.³⁻⁵⁾ In the present paper we compared the volatile constituents of *udo* and butterbur grown in both Japan and Korea.

Materials and Methods

Materials Japanese *udo* (*Alalia cordata* Thunb.) and butterbur (*Petasites japonicus* Miq.)

were obtained from the Kochi Highland Agricultural Experiment Station in April to May, 1988. These vegetables were also collected in Korea at the same season: *udo* (*Araria continentalis* Kitagawa) and butterbur (the same species as Japanese one).

Extraction of volatile constituents Each immature stalk of *udo* and butterbur (300 g) was homogenized and then mixed with water (500ml). The volatile constituents were extracted with diethyl ether using a Likens and Nickerson's simultaneous steam distillation-extraction apparatus modified by FLATH and FORREY.⁶⁾ The extractions of volatile constituents from native *udo* and butterbur were independently carried out at Sawamura's and Kim's laboratories under the same conditions as mentioned by FLATH and FORREY.⁶⁾ The concentrates removed from ether were finally dried with anhydrous sodium sulfate.

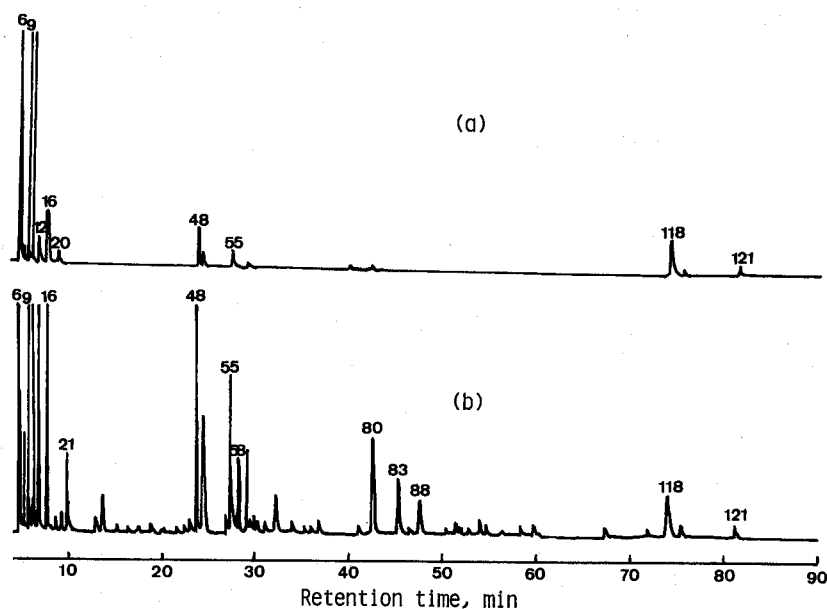
Fractionation of the concentrates The concentrates were fractionated by silica gel (Wako gel Q-23, 100-200 mesh) column (1 cm i. d. \times 25cm) chromatography to facilitate identification of the components. The eluent system was *n*-hexane followed by 90% *n*-hexane in ether.

GC and GC-MS conditions A Shimadzu GC-8 A gas chromatograph equipped with FID was used, and the analytical conditions were same as that in a previous paper.⁷⁾ The chromatographic data were treated with a Shimadzu C-RIB integrator. A GC-mass spectrometer used was a Shimadzu LKB-9000 system. The GC connected with the mass spectrometer was equipped with the same type of column as described above. The operation conditions for MS were as follows: separator temperature, 250°C; ion source temperature, 270°C; ionization energy, 70 eV. The other GC-MS, Shimadzu LKB-9020 DF, was also used for further qualitative analysis.

Results and Discussion

Volatile constituents of *Udo* Typical gas chromatograms on the volatile constituents of

Fig 1. Gas chromatograms of the volatile constituents from *udo*. (a), Japanese *udo*; (b), Korean *udo*.



Japanese and Korean *udo* are shown in Fig. 1 (a) and (b). As shown in these figures, greater number of peaks were detected from Korean *udo* than Japanese one, that is, 114 in the former and 45 in the latter. Their peak area ratios are shown in Table 1. Identification

Table 1. peak area percentages of Japanese and Korean *udo*

Peak No.	Retention time (min)	Peak area (%)		Compounds
		Japanese	Korean	
1	3.72	—	0.08	2-propanone
2	3.94	—	0.05	4-nonene, 2, 3, 3-trimethyl-, (E) -
3	4.09	—	0.03	
4	4.17	0.15	—	
5	4.24	—	0.02	
6	4.75	57.23	65.95	α -pinene
7	5.14	0.04	0.04	γ -terpinene
8	5.22	0.28	0.27	camphene
9	5.89	13.33	11.43	β -pinene
10	6.06	7.57	2.77	sabinene
11	6.58	—	0.24	3-carene
12	6.83	0.78	1.56	myrcene
13	6.91	0.02	—	
14	7.00	0.19	—	
15	7.26	0.18	0.02	
16	7.73	2.10	1.78	(+) -limonene
17	7.97	0.40	0.18	β -terpinene
18	8.61	0.79	0.06	cis- β -ocimene
19	8.99	0.42	0.04	4-carene
20	9.22	0.14	0.10	ocimene
21	9.76	0.09	0.41	benzene, 1-methyl-4-(1-methylethyl) -
22	10.17	0.16	0.05	terpinolene
23	10.49	—	0.02	α -pinene oxide
24	10.69	0.03	0.02	octanal
25	11.71	—	0.02	cyclopentane, 2-methyl-1-methylene-3-(1-methylethenyl)-
26	12.15	—	0.02	
27	12.56	—	0.02	
28	12.76	0.03	0.03	
29	12.94	0.12	0.11	5-undecen-1-ol, (Z)-
30	13.55	—	0.25	benzeneacetic acid, α -methoxy-
31	14.05	0.27	—	
32	14.82	0.04	0.03	3-undecyne
33	15.21	—	0.09	benzene, 1, 3-bis(1,1-dimethylethyl)-5-methyl-
34	16.42	—	0.04	
35	17.36	—	0.06	
36	17.59	0.06	0.05	α -cubebene
37	17.94	—	0.02	8-octadecynoic acid, methyl ester
38	18.88	—	0.16	1, 4-methano-1H-inden, octahydro-4-methyl-8-methylene-7-(1-methylethyl) -

Table 1. (Continued)

Peak No	Retention time (min)	Peak area (%)		Compounds
		Japanese	Korean	
39	19.86	—	0.06	
40	20.16	—	0.05	
41	21.28	—	0.02	
42	21.67	0.06	0.04	
43	22.40	0.05	0.05	
44	22.59	—	0.03	(+)-longifolene
45	22.82	—	0.06	
46	23.15	—	0.11	naphthalene, decahydro-1, 6-bis(methylene)-4-(1-methylethyl)-, (4 α , 4a α , 8a α)-
47	23.56	0.08	—	
48	23.97	3.41	2.54	β -caryophyllene
49	24.36	1.15	0.82	terpinen-4-ol
50	25.06	0.11	0.02	bicyclo[7, 2, 0]undec-4-ene, 4, 11, 11-trimethyl-8-methylene-
51	25.47	—	0.05	bicycloelemene
52	25.76	—	0.03	bicyclo[3, 1, 1]hept-2-ene-2-carboxaldehyde, 6, 6-dimethyl-, (1S)-
53	26.97	—	0.13	bicyclo[3, 1, 1]heptane, 2, 6, 6-trimethyl-3-(2-propenyl)-, (1 α , 2 β , 3 α , 5 α)-
54	27.16	—	0.06	1H-benzocycloheptene, 2, 4a, 5, 6, 7, 8, 9, 9a-octahydro-3, 5, 5-trimethyl-9-methylene-, (4aS-cis)-
55	27.57	1.78	1.54	α -caryophyllene
56	28.02	—	0.16	cyclopentaneacetaldehyde, 2-formyl-3-methyl- α -methylene
57	28.14	0.02	—	
58	28.29	0.10	0.50	3-cyclohexene-1-methanol, α , α , 4-trimethyl-, (S)-
59	28.69	—	0.07	
60	29.32	0.39	0.61	α -terpineol
61	29.59	0.26	0.12	β -cubebene
62	30.02	0.12	0.16	naphthalene, 1, 2, 3, 5, 6, 7, 8, 8a-octahydro-1, 8a-dimethyl-7-(1-methylethenyl)-, 1S-(1 α , 7 α , 8a α)-
63	30.32	0.08	0.07	β -cadinene
64	30.54	—	0.02	6-octen-1-ol, 3, 7-dimethyl-
65	31.12	—	0.13	tricyclo[5, 4, 0, 02, 8]undec-9-ene, 2, 6, 6, 9-tetramethyl-
66	32.19	0.23	0.37	bicyclo[3, 1, 1]hept-2-ene-2-methanol, 6, 6-dimethyl-
67	33.36	—	0.02	
68	34.09	—	0.09	
69	34.27	—	0.02	
70	35.46	—	0.07	ethanone, 1-(1, 3a, 4, 5, 6, 7-hexahydro-4-hydroxy-3, 8-dimethyl-5-azulenyl)-
71	35.87	—	0.03	
72	36.36	—	0.07	(E)-carveol
73	37.02	—	0.11	benzene methanol, α , α , 4-trimethyl-
74	38.74	—	0.04	1-naphthalenol, 1, 2, 3, 4, 4a, 7, 8, 8a-octahydro-1, 6-dimethyl-4-(1-methylethyl)-, [1S-(1 α , 4 α , 4a β , 8a β)]-

Table 1. (Continued)

Peak No	Retention time (min)	Peak area (%)		Compounds
		Japanese	Korean	
75	39.92	—	0.05	
76	40.30	0.30	—	
77	41.27	—	0.10	γ -cadinene
78	41.99	—	0.02	
79	42.39	—	0.07	1, 3, 6, 10-dodecatetraene, 3, 7, 11-trimethyl-, (Z, E)-
80	42.79	0.17	0.98	3-tridecen-1-yne, (Z) -
81	44.06	—	0.02	bicyclo [3, 1, 1] heptane, 6, 6-dimethyl-3-methylene-
82	44.39	—	0.04	hexadecanal
83	45.44	0.07	0.57	1 H-inden-2-ol, 2, 3, 4, 5, 6, 7-hexadehydro-
84	46.01	—	0.03	
85	46.61	—	0.07	cyclohexanol, 3-ethenyl-3-methyl-2-(1-methylethenyl)-6-(1-methylethyl) -
86	46.92	—	0.06	naphthalene, 1, 2, 3, 4, 4a, 7-hexahydro-dimethyl-4-(1-methylethyl) -
87	47.41	—	0.02	2-naphthalenemethanol, decahydro- α , α , 4a-trimethyl-8-methylene-, [2R-(2 α , 4a α , 8a β)] -
88	47.82	—	0.39	4a(2H)-naphthalenecarboxaldehyde, octahydro-4-methyl-6-(1-methylethyl)-3, 5-dioxo-, [4S-(4 α , 4a α , 6 α , 8a α)] -
89	49.69	—	0.02	1-naphthalenol, decahydro-4a-methyl-8-methylene-2-(1-methylethyl) -, acetate
90	50.02	—	0.03	γ -muurolene
91	50.87	—	0.06	androsta-4, 16-dien-3-one
92	51.54	—	0.04	azulene, 1, 2, 3, 4, 5, 6, 7, 8-octahydro-1, 4-dimethyl-7-(1-methylethylidene) -, (1S-cis) -
93	51.86	—	0.13	naphthalene, decahydro-1, 1, 4a-trimethyl-6-methylene-5-(3-methyl-2, 4-pentadienyl) -, [4aS-(4a α , 5 α , 8a β)]
94	52.22	—	0.07	1 H-cycloprop [e] azulene-4-ol, decahydro-1, 1, 4, 7-tetramethyl-, [1aR-(1a α , 4 α , 4a β , 7 β , 7a β , 7a α)] -
95	52.56	—	0.04	5-azulenemethanol, 1, 2, 3, 4, 5, 6, 7, 8-octahydro- α , α , 3, 8-tetramethyl-, acetate
96	53.37	—	0.15	
97	54.27	—	0.04	
98	54.49	0.03	0.13	6, 9-octadecadiynoic acid, methyl ester
99	54.82	—	0.02	
100	55.22	0.09	0.09	cyclohexanol, 3-ethenyl-3-methyl-2-(1-methylethenyl)-6-(1-methylethyl) -, acetate
101	56.62	—	0.03	
102	57.12	—	0.09	9-octadecyne
103	57.81	—	0.03	
104	58.86	—	0.18	1 H-inden-1-methanol, α -methyl-, acetate
105	59.62	—	0.02	
106	60.07	—	0.02	
107	60.51	—	0.14	10-pentadecen-5-yn-1-ol, (Z) -

Table 1. (Continued)

Peak No	Retention time (min)	Peak area (%)		Compounds
		Japanese	Korean	
108	61.01	—	0.04	
109	61.13	0.06	—	
110	61.71	—	0.02	
111	62.54	—	0.02	
112	64.61	—	0.02	
113	65.12	—	0.02	
114	66.36	—	0.03	
115	68.01	—	0.12	8, 11-octadecadiynoic acid, methyl ester
116	72.34	—	0.02	
117	72.62	—	0.09	1, 3, 6, 10-cyclotetradecatetraene, 3, 7, 11-trimethyl-14-(1-methylethyl)-, S-(E, Z, E, E)-
118	74.71	4.77	0.60	1, 5-hexadiene-3, 4-diol, 1, 6-diphenyl-
119	76.16	0.26	0.18	8-octadecynoic acid, methyl ester
120	78.54	—	0.04	
121	81.94	1.56	0.20	hexadecanoic acid
122	83.47	—	0.05	
123	88.07	0.21	—	
124	89.44	0.08	—	

of peaks was carried out by comparisons with the retention time from GC and the data from GC-MS of authentic compounds, and by referring to the spectral data base.^{8,9)} The common and major components in Japanese and Korean *udo* were monoterpenes such as α -pinene, β -pinene and sabinene, amounting to 84 to 88%. These major compounds would be also important components contributing to the characteristic odour of *udo*.¹⁾ This assumption would be supported from organoleptic evaluation that *udo* smells, in fact, like wood to be represented by pinenes. Among sesquiterpenes β -caryophyllene was considerably abundant in the *udo*. Bi-

Fig. 2. Mass spectrum of β -caryophyllene (bicyclo [7, 2, 0] undec-4-ene, 4, 11, 11-trimethyl-8-methylene-, [1R-(1R, 4E, 9S)]-).

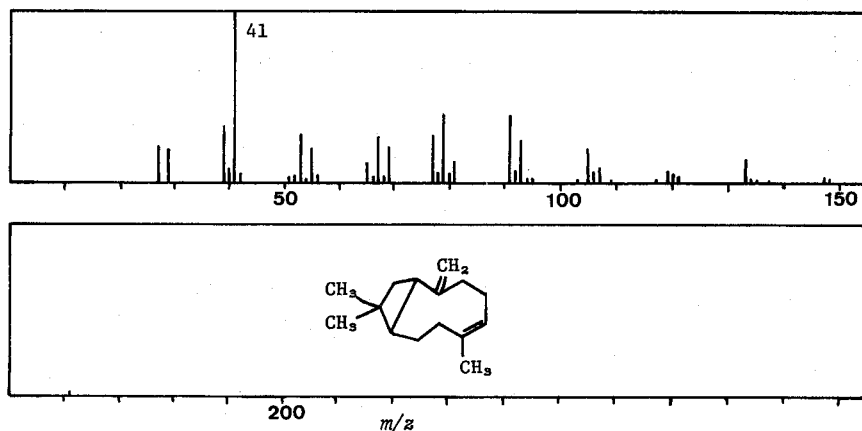
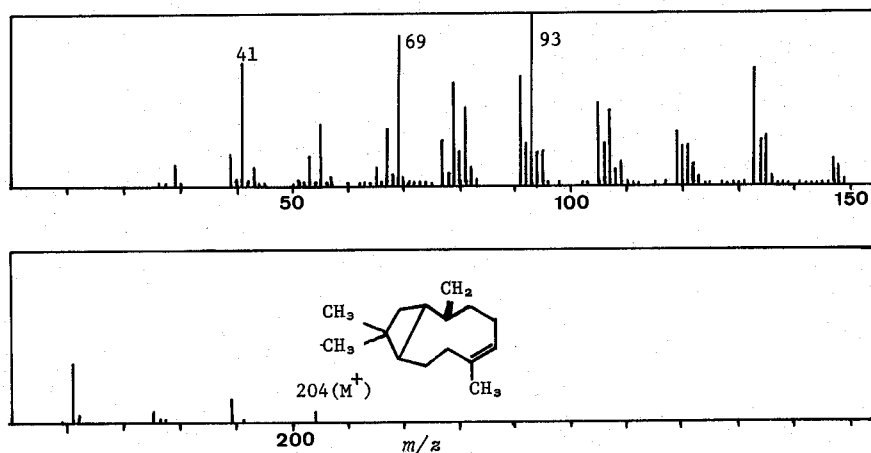


Fig. 3. Mass spectrum of bicyclo [7 , 2 , 0] undec-4-ene-4, 11, 11-trimethyl-8-methylene.



cyclo [7 , 2 , 0] undec-4-ene-4, 11, 11-trimethyl-8-methylene (peak No. 50), an isomer of β -caryophyllene, was also identified by GC-MS. These mass spectra are shown in Fig. 2 and 3. Terpinen-4-ol was noticed among minor oxygenated compound. As shown in Fig. 1, several peaks as higher-boiling point compounds appeared in the gas chromatogram of Korean *udo*. The compounds specific to Korean *udo* were as follows: 1-methyl-4-(1-methylethyl) benzene (peak No. 21); 3-tridecen-1-yne (No. 80); 2, 3, 4, 5, 6, 7-hexahydro 1 H-inden-2-ol (No. 83); octahydro-4-methyl-6-(1-methylethyl)-3, 5-dioxo-4 a (2 H)-naphthalenecarboxaldehyde (No. 88). Both *Aralia cordata* grown in Japan and *Aralia continentalis*

Fig. 4. Gas chromatograms of volatile constituents from butterburs.
(a), Japanese butterbur; (b), Korean butterbur.

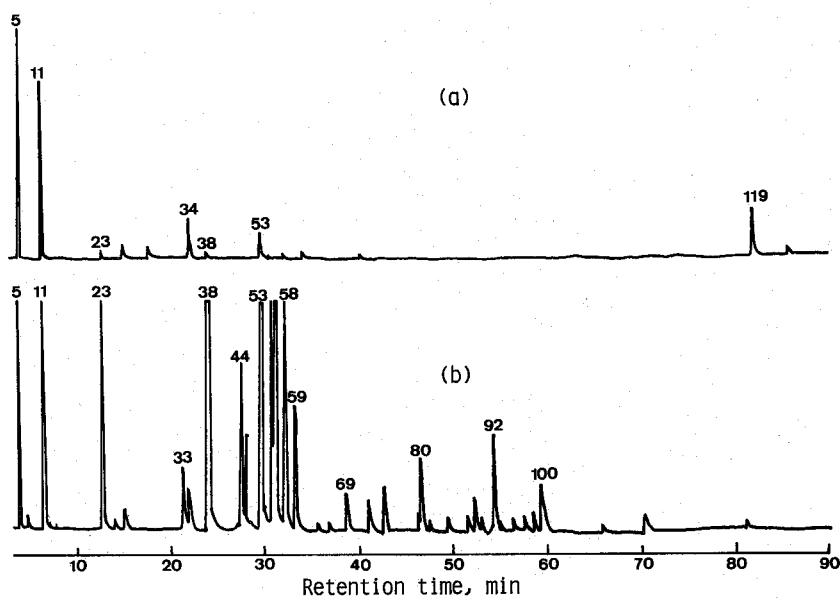


Table 2. Peak area percentages of Japanese and Korean butterbur

Peak No.	Retention time (min)	Peak area (%)		Compounds
		Japanese	Korean	
1	3.04	—	0.23	1-butanol
2	3.38	—	0.04	
3	3.64	0.31	0.06	
4	3.83	0.86	0.05	
5	3.93	41.56	0.96	1-nonene
6	4.16	0.08	—	
7	4.81	0.24	0.04	1-decene
8	5.01	0.06	—	
9	5.46	0.17	—	
10	5.75	0.04	—	
11	6.44	11.66	3.19	2-undecene
12	6.86	0.03	—	
13	6.97	0.05	—	
14	7.06	—	0.03	
15	7.17	0.29	0.04	
16	7.49	0.32	—	
17	7.75	0.21	0.04	
18	8.34	0.77	—	
19	8.69	—	0.08	bicyclo [2, 2, 1] heptane, 2-butylidene-
20	8.98	—	0.05	1-dodecene
21	9.66	0.05	—	
22	9.86	0.08	—	
23	12.66	0.37	4.76	tridecene
24	12.87	0.76	—	1-hexanol
25	14.07	—	0.20	3-hexen-1-ol, (Z)-
26	14.23	0.65	—	
27	15.03	—	0.29	2-hexen-1-ol, (Z)-
28	15.21	2.13	—	
29	17.41	0.16	0.09	α -cubebene
30	17.96	1.47	—	
31	18.95	—	0.10	
32	20.22	—	0.06	
33	21.24	—	1.20	β -cubebene
34	21.43	0.52	—	
35	21.92	—	0.39	linalool
36	22.17	5.16	—	
37	22.94	—	0.09	
38	24.23	0.90	23.58	eremophilene
39	25.16	—	0.12	
40	25.37	0.16	—	
41	25.83	—	0.11	naphthalene, 1, 2, 3, 5, 6, 8a-hexahydro-4, 7-dimethyl-1-(1-methylethyl)-, (1 <i>S</i> -cis)
42	26.23	—	0.14	

Table 2. (Continued)

Peak No.	Retention time (min)	Peak area (%)		Compounds
		Japanese	Korean	
43	27.31	—	0.12	α -caryophyllene
44	27.64	—	2.97	
45	27.86	0.28	—	
46	27.98	0.07	—	
47	28.16	—	2.31	
48	28.32	0.09	—	valencene
49	28.47	0.28	—	
50	28.74	—	0.23	
51	29.43	—	0.04	
52	29.67	0.88	—	
53	29.91	5.02	16.72	cyclopropano [d] naphthalene, 1, 1a, 4, 4a, 5, 6, 7, 8-octahydro-2, 4a, 8, 8-tetramethyl-, [1aS- (1a α , 4a β , 8aR)]-
54	30.18	—	0.10	
55	30.66	—	0.19	
56	31.06	0.27	5.02	
57	31.46	—	15.24	
58	32.43	1.26	8.52	β -bisabolene
59	33.43	—	1.91	β -cadinene
60	34.01	—	0.03	α -bisabolene
61	34.19	—	0.03	β -selinene
62	34.50	0.93	—	
63	35.31	—	0.03	
64	35.98	—	0.10	
65	37.06	—	0.12	
66	37.29	—	0.03	γ -cadinene
67	37.81	0.94	—	
68	38.43	—	0.08	
69	38.83	—	0.57	
70	39.13	0.11	—	
71	39.59	—	0.09	hydrazine, (phenylmethyl)-
72	40.04	—	0.12	
73	40.66	0.84	—	
74	41.36	—	0.40	
75	42.18	—	0.09	
76	42.46	—	0.06	(Z,E) - β -farnesene
77	42.84	—	0.75	
78	45.53	—	0.14	
79	46.44	—	0.16	
80	46.66	—	0.89	
81	46.99	—	0.31	nerolidol
82	47.29	0.25	0.03	α -muurolene
83	47.89	—	0.18	α -ylangene

Table 2. (Continued)

Peak No.	Retention time (min)	Peak area (%)		Compounds
		Japanese	Korean	
84	49.74	0.15	0.16	1H-cycloprop [e] azulene-4-ol, decahydro-1, 1, 4, 7-tetramethyl-, [1aR- (1a α , 4 β , 4a β , 7 β , 7a β , 7b α)]-
85	50.29	—	0.04	
86	50.68	—	0.15	
87	51.29	—	0.04	
88	51.93	—	0.24	
89	52.63	—	0.54	1H-3a, 7-methanoazulen-5-ol, octahydro-3, 8, 8-trimethyl-6-methylene-
90	53.21	—	0.26	1-naphthalenol, 1, 2, 3, 4, 4a, 7, 8, 8a-octahydro-1, 6-dimethyl-4-(1-methylethyl)-, [1S- (1 α , 4 α , 4a β , 8a β)]-
91	53.64	0.30	—	
92	54.58	—	1.25	tricyclo [5, 1, 0, 02, 4] octane-5-carboxylic acid, 3, 3, 8, 8-tetramethyl-, methyl ester
93	54.96	0.34	—	
94	55.28	—	0.09	
95	56.61	—	0.28	
96	57.09	—	0.05	
97	57.88	—	0.23	furan, 3-(4, 8-dimethyl-3, 7-nonadienyl)-, (E)-
98	58.29	0.30	—	
99	58.84	—	0.40	6, 9-octadecadiyonic acid, methyl ester
100	59.64	—	0.77	1, 6, 10-dodecatriene-3-ol, 3, 7, 11-trimethyl-
101	60.36	—	0.01	
102	60.86	—	0.03	
103	61.73	—	0.03	
104	62.34	—	0.04	
105	63.06	—	0.10	
106	63.69	0.25	—	
107	66.44	—	0.19	
108	67.14	—	0.08	
109	67.54	0.22	—	
110	67.64	—	0.07	
111	70.74	—	0.31	9-eicosyne
112	71.08	1.14	0.06	
113	73.63	0.10	—	
114	73.81	—	0.07	
115	76.81	—	0.05	
116	78.04	0.27	0.10	
117	78.54	0.12	—	
118	81.86	—	0.21	
119	82.44	13.15	—	
120	86.38	2.71	—	
121	87.98	0.36	0.02	

grown in Korea have been regarded as same species, but the present data show great differences of the composition of minor volatile components between Japanese and Korean *udo*. It is obscure whether these differences simply result from varied growing conditions or both species are not exactly identical.

Volatile constituents of butterbur The gas chromatograms for Japanese and Korean butterbur are shown in Fig. 4 (a) and (b), and the peak area percents are listed in Table 2. Fifty three peaks were detected in the essential oil from Japanese butterbur, and their major components were as follows: 1-nonene (41.5%), peak 119 (13.1%), 2-undecene (11.6%), peak 36 (5.1%) and valencene (5.0%). Olefinic hydrocarbons were characteristically predominant in the essential oils of Japanese butterbur. Major components of Korean butterbur, in contrast to the Japanese one, were sesquiterpenes such as eremophilene (23.5%), valencene (16.7%), β -bisabolene (15.2%), β -cadinene (8.52%), α -caryophyllene (2.97%) and β -cubebene (1.20%), amounting to about 70%. Terpinen-4-ol and α -terpineol were noticed among them; however as a whole, there were few oxygenated compounds such as alcohols and aldehydes in the butterbur of both countries. Consequently hydrocarbons would mainly contribute to the characteristic flavor of butterbur. NAYA *et al.*^{3,4)} have isolated some kinds of fukinolides from the flower stalks of wild butterbur. In the present study higher concentration of eremophilene, which is an isomer of valencene and supposed to be a precursor for the genesis of fukinolide and its homologues, was found in the stalks of Korean butterbur. Japanese butterbur, in contrast to the Korean one, contained only less than 0.1% of eremophilene. The mass spectra of valencene and eremophilene are shown in Fig 5 and 6. It is assumed that these differences of compositions between Japanese and Korean vegetables have resulted from their growing circumstances, even if they are of identical species.

Fig. 5. Mass spectrum of valencene (naphthalene, 1, 2, 3, 5, 6, 7, 8, 8a-octahydro-1, 8a-dimethyl-7-(1-methylethenyl), [1*R*, (1 *α* , 7 *β* , 8 *α*)]-

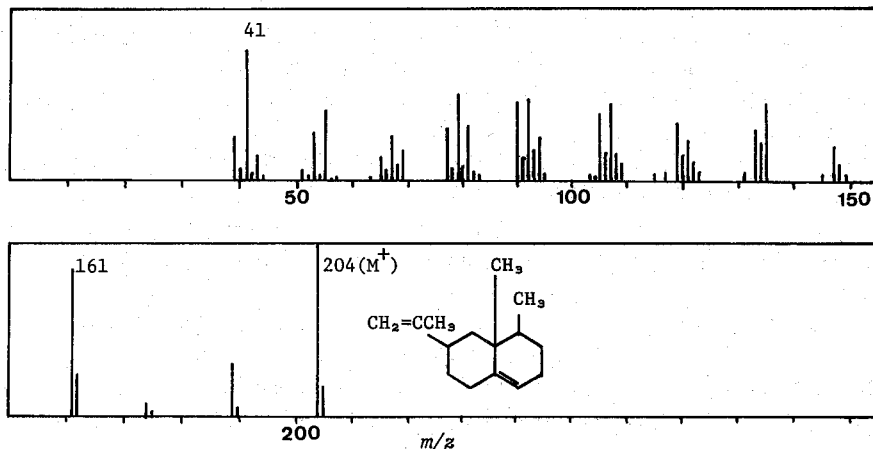
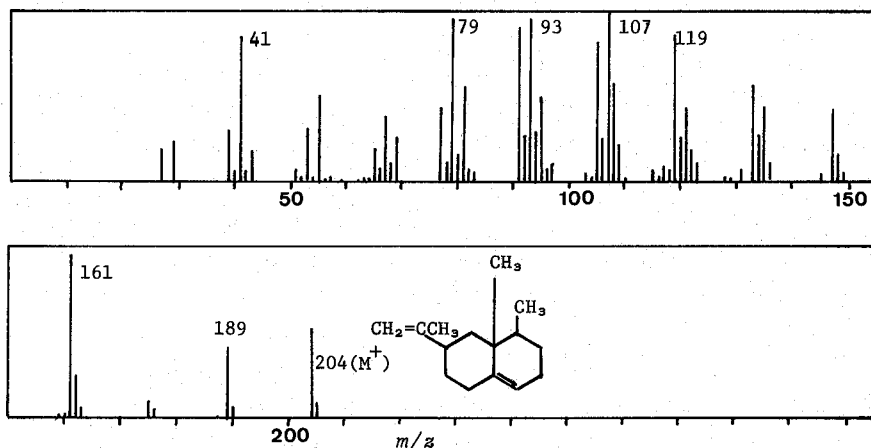


Fig. 6. Mass spectrum of eremophilene (naphthalene, 1, 2, 3, 5, 6, 7, 8, 8a-octahydro-1, 8a-dimethyl-7-(1-methylethenyl), [1S(1 α , 7 β , 8a α)]-



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