Components of Essential Oil from Woods of *Prunus mume* Sieb. et Zucc.

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Abstract: The compositions of the essential oil from woods of *Prunus mume* Sieb. et Zucc. (ume), have been investigated by capillary GC and GC/MS. The oil was found to contain 97 components, representing 92.41% of the total oil. The main constituents were 6,10,14-trimethyl-2-pentadecanone (15.83%), α -acorenol (9.36%), (Z)- α -bisabolene (7.49%), benzaldehyde (3.87%), isopropyltiglate (3.84%), terpinen-4-ol (3.41%).

Key words: *Prunus mume* Sieb. et Zucc., essential oil, 6,10,14-trimethyl-2-pentadecanone, α -acorenol, (Z)- α -bisabolene, benzaldehyde

1 Introduction

Japanese apricot (*Prunus mume* Sieb. et Zucc., ume) is a deciduous tree of *Rosaceae* family. The tree have been carried to Japan from China in the end of 7th century. The fruits have a history of the used of tradition foods, umeboshi or pickled Japanese apricot, ume-liqueur, ume-juice, ume-jam, and as well as folk medicine for alleviating fever, cough and intestinal disorders in Japan. Therefore, there are a lot of reports of constitution from the fruits. From the fruits, some organic acids as acidity compounds have been isolated, such as malic acid, citric acid, oxalic acid, succinic acid (1-3). In addition, many volatile compounds have been identified (4-8). Chu-Chin Chen has reported 92 compounds identified from fruits and the main components were benzaldehyde, benzyl alcohol. Arikawa has reported 52 compounds identified from ume-liqueur by steam distillation and headspace. Ishida also reported 48 compounds were identified from umeboshi. On the other hand, the woods of *P. mume* are liked as a plant

for appreciation by the Japanese and have some flavonoids (9-11). However, there is no report of the essential oil from woods of *P. mume*. In this paper, essential oil from woods of *P. mume* was investigated using GC and GC/MS.

2 Experimental

2.1 Plant

The samples, woods of *P. mume*, were collected from Minabe-town (Wakayama, Japan) in July 2004.

2.2 Isolation of the Volatile Oil

Woods (15 kg) of *P. mume* were hydrodistilled with a Likens-Nickerson-type apparatus using diethyl ether to yield 0.015% of the yellowish green oil, which was dried over anhydrous sodium sulfate prior to analyses.

2.3 Gas Chromatography (GC)

GC was carried out using a Agilent Technologies 6890N, fitted with a flame ionization detector (FID) on

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a non-polar capillary column HP-5 (J&W Scientific; 30 m $\times$ 0.32 mm i.d., film thickness 0.25 μ m). The oven temperature was programmed from 40°C to 240°C at a rate of 4°C/min and held at 240°C for 5 min. The injector and detector temperatures were 240°C and 280°C. The flow rate of the carrier gas (He) was 1.8 mL/min.

2.4 Gas Chromatography-Mass Spectrometry (GC-MS)

The GC-MS analysis was carried out using a Hewlett Packard model 5890 GC with a Hewlett Packard 5972A MS. The capillary column was a nonpolar column DB-5MS (J&W Scientific; 30 m $\times$ 0.25 mm i.d., film thickness 0.25 μ m). The oven temperature was programmed from 60°C to 240°C at a rate of 2°C/min and held at 240°C for 5 min and the flow rates of carrier gas (He) was 0.679 mL/min. The injector and detector temperatures were 240 and 280°C respectively, with the actual temperature in the MS source reaching approximately 180°C and the ionization voltage 70 eV.

2.5 Identification of Constituents

The components of essential oils were identified by direct comparison of their mass spectral pattern and retention index (RI) with those published in the literature (12).

3 Result and Discussion

The yield of oil obtained by steam distillation from the woods of *P. mume* was 0.015% (w/w). The oil has a characteristic odor, which is a very woody with herbal and minty odor undertones. The oil was analyzed on GC and GC-MS, of which gas chromatogram was shown in **Fig. 1**. The percentage composition and retention index (RI) of the oil components were listed in **Table 1**. The oil was revealed the presence 97 components, representing 92.41% of the total oil. The main components in the oil were 6,10,14-trimethyl-2-pentadecanone (15.83%), α -acorenol (9.36%), (Z)- α -bisabolene (7.49%), benzaldehyde (3.87%), isopropyltiglate (3.84%), terpinen-4-ol (3.41%), α -cis-belg-amotene (2.91%), nonanal (2.28%) and heptadecane (1.84%). Classification of the essential oil was listed in **Table 2**. The oil included 5 monoterpenoids (3.98%), 30 sesquiterpenoids (35.30%), 1 diterpenoid (0.19%), 55 aliphatic miscellaneous (46.35%), 6 aroma compounds (6.55%). Its aroma compounds were less than some previous report of fruits oils and the woods oil contained rich chain compounds and mono-, sesqui-terpenoids. These components seemed to play an important role in the characteristic aroma of woods of *P. mume*.

Fig. 1 Gas Chromatogram of the Essential Oil from Woods of *Prunus mume*. HP-5 (30 m $\times$ 0.32 mm i.d., film thickness 0.25 μ m); flow rate of a carry gas (He), 1.8 mL/min; oven temperature, 40-240°C at 4°C/min and held at 240 °C for 5 min.

Table 1 Components of the Essential Oil from Woods of *Prunus mume*.

No.	RI	Compound	Peak area (%)	No.	RI	Compound	Peak area (%)
1	849	2-hexenone	0.10	59	1481	oxide calamenene	0.30
2	885	<i>p</i> -xylene	tr	60	1485	α -zigiberene	1.01
3	900	heptanal	tr	61	1488	β -selinene	tr
7	948	benzaldehyde	3.87	62	1492	α -muurolene	1.14
8	952	isopropyltiglate	3.84	63	1498	(<i>Z</i>)- α -bisabolene	7.49
9	965	1-octen-3-ol	tr	64	1500	pentadecane	0.32
10	969	6-methyl-5-hepten-2-one	0.31	65	1504	β -bisabolene	0.52
11	974	2-pentyl furan	1.13	66	1512	δ -cadinene	0.81
12	988	1-decene	tr	67	1517	γ -dehydro- <i>ar</i> -himachalene	0.13
13	996	(2 <i>E</i> , 4 <i>E</i>)-heptadienal	0.16	68	1519	(<i>Z</i>)-nerolidol	tr
14	1046	(2 <i>E</i>)-octenal	0.26	69	1526	italicene ether	0.40
15	1059	<i>cis</i> -sabinene hydrate	0.02	70	1536	(<i>E</i>)- α -bisabolene	0.22
16	1061	octanol	0.02	71	1546	elemicin	0.35
17	1094	linalool	tr	72	1566	3-methyl pentadecane	0.22
18	1097	(<i>E</i>)-6-methyl-3,5-heptadien-2-one	0.47	73	1572	caryophyllene oxide	0.40
19	1100	nonanal	2.28	74	1592	longiborneol	0.83
20	1140	4-keto-isophorone	0.49	75	1600	hexadecane	0.27
21	1157	(2 <i>E</i>)-nonenal	0.77	76	1626	α -acorenol	9.36
22	1171	nonanol	tr	78	1645	himachalol	0.75
23	1177	terpinen-4-ol	3.41	79	1647	α -bisabolol oxide B	0.33
24	1182	dill ether	0.57	81	1661	<i>neo</i> -intermedeol	1.25
25	1200	dodecane	tr	82	1667	intermedeol	0.91
26	1206	decanal	1.06	83	1671	2-methyl hexadecane	0.64
27	1214	(2 <i>E</i> , 4 <i>E</i>)-nonadienal	0.46	84	1677	3-methyl hexadecane	0.31
30	1238	carvenone	tr	85	1692	1-heptadecene	0.47
31	1248	<i>p</i> -anisaldehyde	0.98	86	1700	heptadecane	1.84
32	1257	decanol	0.69	87	1714	pentadecanal	0.31
33	1287	(2 <i>E</i> , 4 <i>Z</i>)-decadienal	0.74	89	1793	1-octadecene	0.63
34	1300	tridecane	0.33	90	1800	octadecane	0.69
35	1311	(2 <i>E</i> , 4 <i>E</i>)-decadienal	1.46	91	1843	6,10,14-trimethyl-2-pentadecanone	15.83
37	1338	α -longipinene	tr	92	1893	1-nonadecene	0.84
38	1344	eugenol	0.22	93	1900	nonadecane	1.36
39	1348	(2 <i>E</i>)-undecenal	0.37	94	1947	isophytol	0.19
40	1360	undecanol	0.51	95	1968	palmitic acid (C16)	tr
41	1367	protoillud-6-ene	tr	96	1994	1-eicosene	0.85
42	1373	3-methyl tridecane	tr	97	2000	eicosane	0.81
43	1400	tetradecane + β -longipinene	0.74	98	2094	1-heneicosene	0.61
44	1401	2-dodecanone	0.85	99	2100	heneicosane	1.12
45	1408	α - <i>cis</i> -belgamotene	2.91	100	2156	ethyl linoleate	0.56
46	1415	β -cedrene	tr	101	2194	1-docosene	0.27
47	1424	β -gurjunene	0.85	102	2200	docosane	0.34
48	1428	α - <i>trans</i> -bergamotene	1.11	103	2294	1-tricosene	0.23
51	1446	geranyl acetone	0.82	104	2300	tricosane	0.46
52	1454	2-methyl tetradecane	0.59	106	2395	1-tetracosene	tr
53	1456	α -acoradiene	0.92	107	2400	tetracosane	0.18
54	1461	3-methyl tetradecane	0.59	108	2500	pentacosane	tr
55	1466	β -chamigrene	0.71				
57	1473	(<i>E</i>)- β -ionone	0.54				
58	1477	<i>ar</i> -curcumene + β -chamigrene	1.70			total	92.41

RI: Retention index on HP-5 column; tr: trace<0.01%.

Table 2 Classification of the Components of Woods Oil from *Prunus mume*.

Component	Peak area (%)
<i>Aliphatic</i>	
Monoterpenoid	
alcohols	3.41
ketones	tr
miscellaneous	0.57
Sesquiterpenoid	
hydrocarbons	20.25
alcohols	13.42
ketones	0.54
miscellaneous	1.09
Diterpenoids	
alcohols	0.19
Miscellaneous	
hydrocarbons	13.97
alcohols	1.22
aldehydes	7.88
ketones	18.88
acids	tr
esters	0.56
miscellaneous	3.84
<i>Aromatic</i>	
alcohol	0.22
aldehyde	4.85
hydrocarbon	tr
miscellaneous	1.48
<i>unknown</i>	7.59
tr: trace < 0.03%.	

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References

1. Y. CHUDA, H. ONO, M. OHNISHI-KAMEYAMA, K. MATSUMOTO, T. NAGATA and Y. KIKUCHI, Mumefural, Citric Acid Derivative Improving Blood Fluidity from Fruit-Juice Concentrate of Japanese Apricot (*Prunus mume* Sieb. et Zucc), *J. Agric. Food Chem.*, Vol. **47**, 828-831 (1999).
2. W. TAKATSUJI, S. IKEMOTO, H. SAKAGUCHI and H. MINAMI, Development of a New Type of Umeshu Using Immobilized Growing Yeast Cells, *Nippon Jozo Kyokaiishi*, Vol. **87**, 533-537 (1992).
3. M. MIYAZAWA, T. YAMADA and H. UTSUNOMIYA, Suppressive Effect of the SOS-inducing Activity of Chemical Mutagen by Citric Acid Esters from *Prunus mume* Sieb. Et Zucc. Using the *Salmonella typhimurium* TA1535/PSK1002 UMU Test, *Nat. Prod. Res.*, Vol. **17**, 319-323 (2003).
4. H. KAMEOKA and C. KITAGAWA, The Constituents of the Fruits of *Prunus Mume* Sieb. et Zucc., *Nippon Nogei Kagaku Kaishi*, Vol. **50**, 389-393 (1976).
5. C. YUKAWA, Y. IMAYOSHI and H. IWABUCHI, Volatile Components of Japanese Apricot (*ume*, *Prunus mume* Sieb. et Zucc.) (I), *Foods Food Ingredients J.*, Vol. **166**, 76-79 (1995).
6. C.C. CHEN, M.C. KUO, S.E. LIU and C.M. WU, Volatile Components of Salted and Pickled Prunes (*Prunus mume* Sieb. et Zucc.), *J. Agric. Food Chem.*, Vol. **34**, 140-144 (1986).
7. T. ARIKAWA, S. OHSHIMA and H. TAKAGAKI, Aroma Substances in Umeshu (Japanese apricot liqueur) and Their Changes during Storage, *Nippon Kaseigaku Kaishi*, Vol. **48**, 295-301 (1997).
8. M. ISHIDA, Volatile Components of Umeboshi, *Koryo*, Vol. **211**, 139-156 (2001).
9. M. HASEGAWA, Flavonoids of Various *Prunus* Species. X. Wood Constituents of *Prunus tomentosa*, *Shokubutsugaku Zasshi*, Vol. **82**, 458-461 (1969).
10. M. HASEGAWA, Flavonoids of Various *Prunus* Species. IX. Two New Flavonoid Glycosides from the Wood of *Prunus mume*, *Shokubutsugaku Zasshi*, Vol. **82**, 148-154 (1969).
11. M. HASEGAWA, Flavonoids of Various *Prunus* Species. VIII. Flavonoids in the Wood of *P. mume*, *J. Org. Chem.*, Vol. **24**, 408-409 (1959).
12. R.P. ADAMS, Identification of Essential Oil Components by Gas Chromatography/ Quadrupole Mass Spectroscopy., Allured Publ. Corp., Carol stream, IL, USA (2001).