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GC-MS Analysis of Volatile Compounds the Peel and Pulp of *Citrus Medica* L. Var. *Sarcodactylis Swingle* (Foshou Fruit)

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***Abstract* – This study aimed to analyze and compare volatile compounds in Foshou fruit peel and pulp by gas chromatography-mass spectrometry. Foshou fruit contains 56 Hydrocarbons compounds, 33 Alcohols compounds, 11 Aldehydes compounds, 11 Esters compounds, 10 ketones compounds, 6 Acid compounds, 5 Phenols compounds, 4 Furans compounds, 3 Bases compounds, Amide compound, Halogen compound, Lactone compound, Nitrile compound, Oxide compound, and 9 other compounds. The results of this study showed that the Foshou fruit was rich in volatile compounds,"But the comparison of the peel and the pulp demonstrated the peel to be more rich in most of the volatile compounds".**

***Keywords* – Citrus Medica L. var. Sarcodactylis Swingle, Volatile Compounds, GC-MS.**

I. Introduction

Citron (*Citrus medica* L.) is known as one of the three fundamental ancestral species of Citrus genus with pummelo (*Citrus maxima* (Burm.) Merr.) and mandarin (*Citrus reticulata* Blanco) (Ramadugu et al., 2015). Foshou fruit (*Citrus medica* L. var. *sarcodactylis* Swingle) is classified under Rutaceae, which is broadly cultivated in the Oriental countries. Foshou fruit is usually consumed as functional fruit. Traditionally, Foshou fruit is used as a raw material in some Chinese medicine ingredients for treatment of several chronic diseases. To due to the presence of bioactive compounds in it (Wu, Li, Yang, Zhan, & Tu, 2013).

Volatile compounds of the plant are important secondary metabolites, that are widespread in higher plants (Cheng et al., 2007). It has been found that peels were contained higher concentrations of volatiles than the juice sacs in 108 samples of different variety of citrus fruits (Zhang et al., 2017).

Recently, many ways are devised to extract and identify active bio-compounds from different plant parts. Gas chromatography-mass spectrometry (GC–MS) is a useful and commonly used technique for aroma analysis such as odor/off-flavor of foods and volatile organic compounds. GC–MS technique is efficient for obtaining information about the structure of volatile compounds by matching the fragments of target compounds with the database available in the GC–MS system about the standard compounds (Song & Liu, 2018).

In food industry, the research interest in citrus volatile compounds is targeted to their optimization in order to utilize them as natural antimicrobials or their incorporation on edible film to treat fresh-cut vegetables and fruits. The addition of citrus volatile compounds to the food could enhance value and acceptability (Niedz, Moshonas, Peterson, Shapiro, & Shaw, 1997). This study aims to analyze and compare volatile compounds in peel and pulp of the Foshou fruit.

II. Materials and Methods

2.1. *Plant Materials*

Fresh Foshou fruits (*Citrus medica L. var. sarcodactylis*) were provided by Zhejiang golden hand Biological Technology Co., Ltd. from Jinhua, Zhejiang Province, China, in November 2018.

2.2. *Chemicals*

All chemicals and solvents used were of analytical grade and stored in ideal conditions according to the manufacturers specifications.

2.3. *Determination the Volatile Compounds*

The volatile compounds in the Foshou fruit peel and pulp were estimated using a GC–MS (Scion SQ 456-GC, Bruker, USA) with A DB-Wax column 30 m × 0.25 mm, 0.25 µm (J&W Scientific, Folsom, CA, USA). The Helium was used as mobile phase at 0.80 ml/min rate according to the method repeated by Sun, Zhang, Chen, Zheng, and Fang (2016).

The volatile compounds were identified by matching their mass spectra to standard compounds listed in MS libraries of Wiley, New York, USA and National Institute of Standards and Technology (NIST) Gaithersburg, Maryland, USA. Foshou fruit volatile compounds were classified by classification Volatile Compounds in Food 16.4 (VCF online, 2017, February 13). The relative contents of the volatile compounds were calculated by comparing the percentage of the peak areas.

III. Results and Discussions

Table 1 represented the volatile compounds profile of Foshou fruit peel and pulp, 153 compounds were detected in Foshou fruit as follows: Hydrocarbons, Alcohols, Aldehydes, Esters, ketones, Acid, Phenols, Furans, Bases, Amides, Halogens, Lactones, Nitriles, Oxides (56, 33, 11, 11, 10, 6, 5, 4, 3, 1, 1, 1, 1, 1) consecutively along with 9 others compounds. The peel contains 113 compounds, while the pulp contains 101 compounds. The percentage of volatile compounds ranged from 10.338 % to 0.023 % in the peel (Fig. 1).

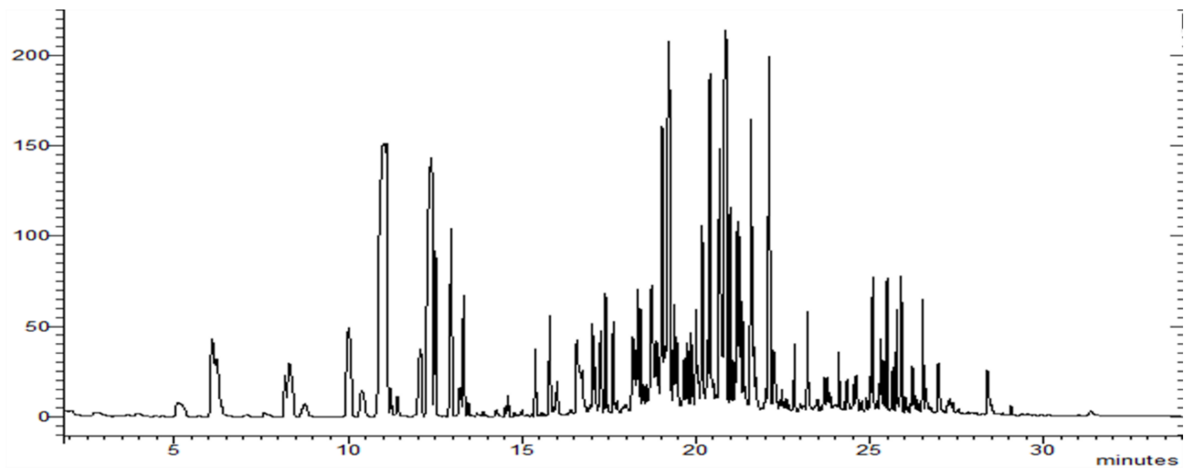

Fig. 1. Chromatograms the volatile compounds profile of the Foshou fruit peel from GC–MS analysis.

The percentage of volatile compounds in the pulp ranged from 16.482 % to 0.029 % (Fig.2). The peel monopolized 52 compounds. However, the pulp also monopolized 40 compounds while 61 compounds were shared between the Foshou fruit peel and pulp.

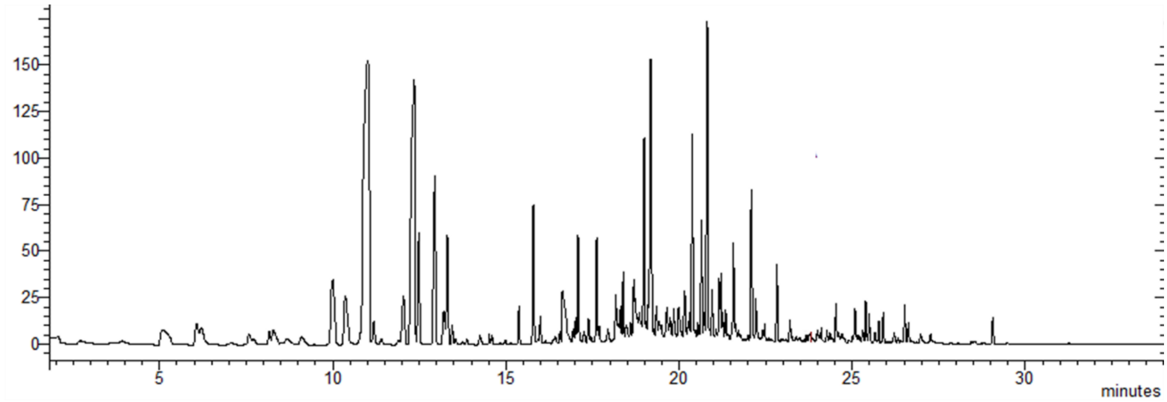

Fig. 2. Chromatograms the volatile compounds profile of the Foshou fruit pulp from GC–MS analysis.

The results of the current study showed a great dominance of Foshou fruit in terms of volatile compounds compared to the study conducted by Zhang et al. (2017) where they studied 108 samples representing seven species of citrus and showed that the volatile compounds in the citrus peels ranged from 30 to 65, while in juice they ranged from 6 to 41.

Many volatile compounds in Foshou fruit have biological effects according to previous studies, for example, Propanoic acid is generally used as an antifungal agent in food (Harrison, 1992). Caprylic acid is a medium-chain fatty acid reported effective in killing species of the bacterial pathogens, including *Campylobacter jejuni* (De Los Santos et al., 2009). Capric acid has anti-inflammatory and bactericidal activities against *P. acnes* (Huang et al., 2014).

Linalool has proved antimicrobial and insect-repellent properties (Beier et al., 2014). Farnesol generally has a wide variety of biological activities like block biofilm formation, inhibit the growth of many microorganisms, and exhibited an important role in resistance to oxidative stress (Wang, Liu, Zhang, & Niu, 2017). Phenoxyethanol is a well-established and widely used preservative with negligible long-term effects in topical applications (Puschmann, Herbig, & Müller-Goymann, 2018).

Tetramethylpyrazine has a strong anti-inflammatory activity, it was inhibited the increase the mRNA level of nuclear factor kappa B (NF-kB), and nuclear factor of activated T cells (NF-AT), and transcription factor-activated protein-1 (AP-1) induced by oxazolone (Chen, Chen, Zhu, Chen, & Lu, 2016). β-caryophyllene has beneficial effects, such as anti-spasmodic, anti-inflammation, inhibition of the hypersensitive immune reaction, and improvement of asthma. Moreover, β-caryophyllene shows antimicrobial activity against gram-positive and gram-negative bacteria (Yoo & Jwa, 2019). β-myrcene showed significant anti-catabolic and anti-inflammatory properties in vitro suggesting that it may be useful to halt or, at least, slow down cartilage destruction (Rufino et al., 2015).

Terpinolene has demonstrated anti-inflammatory properties, antioxidant activity andanticancer activity (de Christo Scherer et al., 2019). β-element has been proved to have anti-tumor activity in a broad range of solid tumors (Yu et al., 2017). β-ionone and its derivatives can show important pharmacological activities such as antifungal, anti-inflammatory, ant-leishmanial, and antibacterial activities(Ansari & Emami, 2016).

Carvacrol has several biological activities, which include antitumor, antimicrobial, antimutagenic, anticholinesterase and antigenotoxic activities (Guimarães et al., 2012). Carvacrol inhibits HepG2 cell growth by inducing apoptosis (Yin et al., 2012). Eugenol has anti-yeast, antibacterial, antioxidant, antifungal and insecticidal properties (Narayanan & Ramana, 2013).

Table 1. Comparison of volatile components content % in Foshou fruit peel and pulp identified by GC-MS.

No.	Compounds	CAS	Peel	Pulp
Acids				
1	propanoic acid (=propionic acid)	79-09-4	1.025	N.D.
2	nerolic acid	4613-38-1	0.666	N.D.
3	acetic acid	64-19-7	0.499	N.D.
4	octanoic acid (=caprylic acid)	124-07-2	0.257	0.082
5	2-methylpropanoic acid (=isobutyric acid)	79-31-2	0.135	N.D.
6	decanoic acid (=capric acid)	334-48-5	0.083	N.D.
Alcohols				
7	β-Fenchyl alcohol	470-08-6	N.D.	2.955
8	Geraniol	106-24-1	3.673	2.187
9	Nerol	106-25-2	2.535	1.328
10	trans Sabinene hydrate	17699-16-0	N.D.	1.453
11	2,3-butanediol (=2,3-butylene glycol)	513-85-9	N.D.	0.979
12	1,3-butanediol	24621-61-2	N.D.	0.973
13	cis-sabinene hydrate (=cis-4-thujanol)	15537-55-0	N.D.	0.849
14	Linalool	78-70-6	N.D.	0.73
15	(Z,E)-farnesol	3790-71-4	1.722	N.D.
16	β-citronellol	106-22-9	1.369	0.693
17	trans-sesquisabinenehydrate	145512-84-1	1.068	N.D.
18	α-bisabolol	515-69-5	0.990	0.359
19	trans Sabinene hydrate	17699-16-0	0.653	N.D.
20	p-8-menthene-1,2-diol	1946-00-5	0.408	N.D.
21	trans Sabinene hydrate	17699-16-0	0.353	N.D.
22	cis-p-2-menthen-1-ol	29803-81-4	N.D.	0.264
23	(E)-nerolidol	40716-66-3	0.305	0.222
24	1-dodecanol	112-53-8	N.D.	0.129
25	Nerolidol	7212-44-4	0.249	N.D.
26	2,6-dimethyl-1,7-octadiene-3,6-diol	51276-33-6	0.243	N.D.
27	cis-sesquisabinene hydrate	58319-05-4	0.191	0.053
28	2-decanol	1120-06-5	0.162	N.D.
29	γ-isogeraniol	13066-51-8	0.153	N.D.
30	trans-carveol (=trans-p-1(6),8-menthadien-2-ol)	1197-07-5	0.134	N.D.
31	p-8-menthene-1,2-diol	1946-00-5	0.122	N.D.
32	1,2-heptanediol	3710-31-4	0.105	N.D.

No.	Compounds	CAS	Peel	Pulp
33	benzyl alcohol (=benzenemethanol)	100-51-6	0.085	0.13
34	4-isopropylbenzyl alcohol	536-60-7	0.073	N.D.
35	cis-carveol (= cis-p-1(6),8-menthadien-2-ol)	1197-06-4	0.057	N.D.
36	trans-p-2-menthene-1,4-diol	40735-19-1	0.057	N.D.
37	1-heptanol	111-70-6	0.056	N.D.
38	2-phenoxyethanol	122-99-6	0.055	N.D.
39	α-cadinol	481-34-5	0.044	N.D.
Aldehydes				
40	Nonanal	124-19-6	0.816	1.997
41	Decanal (CAS)	112-31-2	0.691	1.380
42	(Z)-2-nonenal	60784-31-8	N.D	0.801
43	Hexanal (CAS)	66-25-1	N.D	0.511
44	nonanal (=pelargonaldehyde)	124-19-6	0.255	N.D.
45	3-(4-isopropylphenyl)-2-methylpropanal	103-95-7	0.162	0.365
46	octanal (=caprylaldehyde)	124-13-0	0.106	0.289
47	(E)-2-heptenal	18829-55-5	0.074	0.197
48	Neral	106-26-3	N.D	0.159
49	Lilial	80-54-6	0.046	0.145
50	Farnesal	19317-11-4	0.023	N.D.
Amides				
51	N,N-Bis(2-hydroxyethyl)dodecanamide	120-40-1	0.36	N.D.
Bases				
52	O-decylhydroxylamine	29812-79-1	N.D.	0.210
53	Tetramethylpyrazine	1124-11-4	0.798	0.190
54	1H-pyrrole-2-carboxaldehyde	1003-29-8	N.D.	0.162
Esters				
55	methyl dihydrojasmonate	24851-98-7	N.D.	0.424
56	1. 2-Isopropenyl-5-methyl-4-hexenal	6544-40-7	0.410	0.128
57	diethylene glycol monoethyl ether (=carbitol)	111-90-0	0.409	0.200
58	geranyl acetate	105-87-3	0.306	N.D.
59	methyl 2,2-dimethylpropanoate	598-98-1	0.156	0.170
60	diethyl phthalate	84-66-2	0.131	0.125
61	diethylene glycol monobutyl ether (=2-(2-butoxyethoxy)ethanol)	112-34-5	0.117	N.D.
62	geranyl butanoate	106-29-6	0.105	N.D.
63	diisobutyl phthalate	84-69-5	0.067	0.377
64	1-Octen-4-yne	24612-83-7	N.D.	0.114
65	neryl acetate	141-12-8	N.D.	0.082
Furans				
66	3,9-Epoxy-p-mentha-1,8(10)-diene	None	N.D.	0.378

No.	Compounds	CAS	Peel	Pulp
67	cis-linalool oxide	5989-33-3	0.041	0.366
68	2-(hydroxyethyl)-5-methylfuran	35942-94-0	N.D.	0.217
69	2-furancarboxaldehyde (CAS)	98-01-1	N.D.	0.176
Halogens				
70	1,3-dichlorobenzene	541-73-1	1.317	1.988
Hydrocarbons				
71	3-Methylene-bicyclo[3.2.1]oct-6-en-8-ol	None	10.338	16.482
72	γ-terpinene	99-85-4	N.D.	10.809
73	1,2-dimethylbenzene (=o-xylene)	95-47-6	7.047	N.D.
74	α-himachalene	3853-83-6	5.805	4.962
75	β-caryophyllene	87-44-5	4.942	5.519
76	trans-α-bergamotene	13474-59-4	N.D.	0.580
77	Bicyclogermacrene	24703-35-3	3.642	0.560
78	β-santalene	511-59-1	N.D.	0.501
79	β-copaene	18252-44-3	3.434	0.370
80	germacrene D	23986-74-5	N.D.	0.400
81	α-pinene	80-56-8	2.904	0.733
82	α-Thujene	2867-05-2	N.D.	0.710
83	Longifolene	475-20-7	2.483	N.D.
84	(1S)-β-pinene	18172-67-3	2.276	N.D.
85	Cymene	25155-15-1	2.246	3.900
86	α-bergamotene	17699-05-7	N.D.	3.064
87	β-myrcene	123-35-3	N.D.	2.659
88	β-ocimene	13877-91-3	N.D.	2.159
89	ethylbenzene (=α-methyltoluene)	100-41-4	1.814	N.D.
90	(E)-β-ocimene	3779-61-1	1.812	N.D.
91	β-pinene (=6,6-dimethyl-2 methylenebicyclo[3.1.1]heptane)	127-91-3	1.433	0.597
92	Bicyclo[8.1.0]undeca-2,6-diene, 3,7,11,11-tetramethyl-, (1S,2Z,6E,10S)-	None	1.381	0.349
93	p-1-menthene	5502-88-5	N.D.	0.348
94	(Z)-β-ocimene	3338-55-4	1.292	1.354
95	(+)- δ-cadinene	483-76-1	1.275	0.713
96	Terpinolene	586-62-9	1.135	1.963
97	Cyclohexene, 2-ethenyl-1,3,3-trimethyl-	5293-90-3	0.974	0.662
98	α-amorphene	20085-19-2	N.D.	0.313
99	α-copaene	3856-25-5	0.945	0.307
100	α-bisabolene	17627-44-0	N.D.	0.274
101	(+)-α-copaene	15917-91-6	0.936	1.451
102	(E)-β-farnesene	18794-84-8	0.890	0.622

No.	Compounds	CAS	Peel	Pulp
103	cis-Bicyclogermacradiene	67650-90-2	0.870	N.D.
104	α-terpinene	99-86-5	0.710	2.026
105	β-ylangene	20479-06-5	0.706	N.D.
106	γ-muurolene	30021-74-0	0.698	N.D.
107	cis-β-copaene	41530-63-6	0.675	N.D.
108	epi-β-santalene	25532-78-9	0.644	N.D.
109	α-muurolene	31983-22-9	0.600	0.249
110	cis-α-bisabolene	29837-07-8	0.587	0.943
111	(1S)-β-pinene	18172-67-3	0.508	0.296
112	neo-allo-Ocimene	7216-56-0	0.485	0.526
113	epi-β-santalene	25532-78-9	N.D.	0.179
114	1-isopropenyl-4-methylbenzene	1195-32-0	N.D.	0.161
115	Tetradecane	629-59-4	N.D.	0.085
116	trans-β-Farnesene	28973-97-9	0.482	N.D.
117	epi-β-santalene	25532-78-9	0.460	N.D.
118	(-)-β-elemene	515-13-9	0.425	0.035
119	Aromadendrene	489-39-4	0.397	0.663
120	sabinene (=4(10)-thujene)	3387-41-5	0.300	N.D.
121	β-phellandrene	555-10-2	0.184	0.378
122	trans-β-copaene	41530-64-7	0.147	0.259
123	Hexadecane	544-76-3	0.122	0.326
124	Dodecane	112-40-3	0.056	N.D.
125	viridiflorene (=ledene)	21747-46-6	0.040	0.039
126	α-ylangene (=ylangene)	14912-44-8	0.024	0.067
Ketones				
127	2,3-butanedione (=diacetyl)	431-03-8	1.676	1.279
128	(E)-β-ionone	79-77-6	0.705	0.223
129	ionone (unkn.str.)	8013-90-9	0.568	0.175
130	α-ionone	127-41-3	0.406	N.D.
131	3-hydroxy-2-butanone (Acetoin)	513-86-0	0.393	0.855
132	Geranyl acetone	3796-70-1	0.391	0.567
133	1. 3-Buten-2-ol,4-(2,6,6-trimethyl-1-cyclohexen-1-yl)-	472-80-0	0.375	N.D.
134	2-dodecanone	6175-49-1	0.206	0.234
135	6-methyl-5-hepten-2-one	110-93-0	0.173	0.162
136	(4S)-(+)-carvone	2244-16-8	N.D.	0.029
Lactones				
137	γ-butyrolactone	96-48-0	N.D.	0.322
Nitriles				
138	1-Decen-4-yne, 2-nitro-	80255-22-7	0.207	N.D.

No.	Compounds	CAS	Peel	Pulp
Oxides				
139	β-caryophyllene oxide	1139-30-6	N.D.	0.428
Phenols				
140	2,6-di-tert-butyl-4-methylphenol	128-37-0	N.D.	0.907
141	carvacrol (=5-isopropyl-2-methylphenol)	499-75-2	0.696	0.261
142	B.H.T.	128-37-0	0.43	0.274
143	Eugenol	97-53-0	0.423	0.538
144	BHT-OH	10396-80-2	N.D.	0.038
Others				
145	trans,cis,trans-Bicyclo[4.4.0]decane, 2,9-dimethyl	129967-66-4	0.757	N.D.
146	trans-β-Farnesene	28973-97-9	N.D.	0.304
147	2,5-Methano-1H-inden-7(4H)-one, hexahydro-	27567-85-7	N.D.	0.153
148	1-(7-Hydroxy-1,6,6-trimethyl-10-oxatricyclo[5.2.1.0(2,4)]dec-9-yl)ethanone	None	0.128	N.D.
149	2-Cyclohexen-1-one, 3,5-dimethyl-	1123-09-7	0.12	N.D.
150	1,7-Heptanediol	629-30-1	0.083	N.D.
151	2,6-Dimethyl-3(E),5(E),7-octatriene-2-ol	None	0.080	N.D.
152	Allyl 2-ethylbutyrate	7493-69-8	0.076	N.D.
153	Dodecamethylcyclohexasiloxane	540-97-6	0.066	0.121
		Total	100	100

Abbreviations: CAS, Chemical Abstracts Service Number; N.D., not detected.

IV. Conclusions

This study was conducted to obtain comprehensive data on the composition of Foshou fruit peel and pulp in terms of volatile compounds. Remarkably, Foshou fruit showed a high content of volatile aromatic compounds especially in peel, which contained relatively much higher content of volatile aromatic compounds as compared to the rest of the citrus. Depending on the results of this study, we can conclude that Foshou fruit is rich in volatile compounds, which may have potential use not only in food but other industries as well.

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