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Research Article

Chemical Composition and Aromatic Profiles of Essential Oil from *Rosa laevigata* by GC-MS/GC-O Analysis

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Abstract: Chemical composition and aroma of essential oil from the fruit of *Rosa laevigata* obtained by steam distillation was analyzed by Gas Chromatography-Mass Spectrumeter (GC-MS) and Gas Chromatography-Olfactometry (GC-O). Sixty seven compounds in the essential oil, accounting for 99.97%, were identified, in which palmitic acid (16.49%), cis-13-Octadecenoic acid (16.31%) and Linoleic acid (13.93%) were the main compounds. The overall aroma of the oil was comlex, smelled waxy, heavy refreshing and sweet and strong woody with a tiny tea. 24 aroma compounds were identified by the technique of GC-O with a posterior intensity method. The key active odor compound in this essential oil is myristic acid.

Keywords: Aromatic profile, chemical composition, essential oil, GC-MS, GC-O, *Rosa laevigata*

INTRODUCTION

Rosa laevigata Michx, belonging to the family of Rosaceae, is an evergreen climbing shrub which abundantly distributed in China. The fruit of this plant had a long history to be used for food and herbal medicines. Some studies have been reported that the fruit of *Rosa laevigata* has an influence on treating nocturnal emission, enuresis, frequent urination, vaginal discharge metrorrhagia, chronic diarrhea and other diseases (Zhang *et al*., 2004; Zou *et al*., 2006). The fruit was also used to make wine for a long time and this plant comes to be a remarkable fruit recently.

It is known that the fruit of *Rosa laevigata* contains abundant polysaccharides, saponins and flavonoids, which have strong abilities for eliminating active oxygen, anti-inflammatory and anti-tumor (Chen and Zhang, 2005; Wu *et al*., 2009; Xiao and Liu, 2006; Zhao *et al*., 2003). Moreover, it has been found that the total flavonoids from the fruit had hepatoprotective activity in mice treated by paracetamol (Dong *et al*., 2013; Liu *et al*., 2011). Natural essential oil has been widely and effectively applied in medicine, additive, cosmetics and so on. Therefore, it is necessary to study the essential oil from the fruit of *Rosa laevigata*.

Gas Chromatography-Olfactometry (GC-O) is a valuable technique for selection of odor active compounds from a complex mixture, (Grosch, 1993) which was developed by Fuller *et al*. (1964). Several methods have been developed to collect and process GC-O data and to estimate the sensory contribution of single odor active compound (Dravnieks and O'Donnell, 1971). One of the GC-O techniques is posterior intensity methods, which proposes the odor intensity measurement of a compound and its posterior scoring on a previously determined scale (Zellner *et al*., 2008). This method has been reported in some literatures (Van Ruth and O'Connor, 2001). It has been applied to evaluate volatile compounds of fruits (Casimir and Whitfield, 1978) and dairy products (Arora *et al*., 1995; Cadwallader and Howard, 1998). Nowadays, in order to identify the odor active volatile compounds more accurately, GC-O was always combined with GC-MS (Goodner *et al*., 2006; Marquez *et al*., 2013).

Aim of this study was to identify chemical compositions and main aromatic active compounds of the essential oil from fruit of *Rosa laevigata*.

MATERIALS AND METHODS

Plant materials: *Rosa laevigata* fruits were harvested from Zhejiang province of China, in September 2014. They were roughly processed in Anhui province of China (Tongling Hetian medicine Pieces Ltd), in October 2014. Those fruits were semi-crushed for oil extraction.

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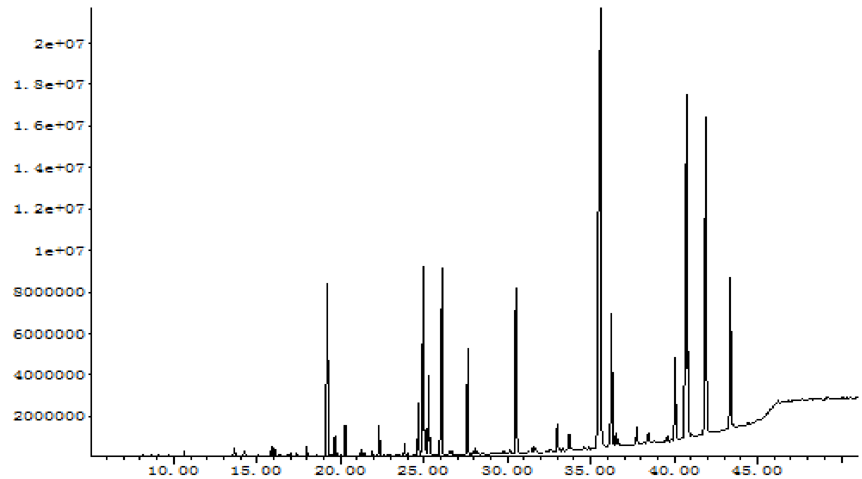

Fig. 1: Total ion chromatogram of the essential oil from *R. laevigata*

Extraction of the essential oil: An ultrasonic bath at 400 W was used to treat with 300 g of semi-crushed *Rosa laevigata* fruits for 30 min. Then the fruits were hydrodistilled for 3 h, using a modified Clevenger apparatus with 400 mL water to yield about 0.1% of essential oil. Anhydrous sodium sulphate (Na_2SO_4) was used to remove water from the oil. The treated oil was stored in a sealed brown glass vial at 4°C for the following use.

GC-MS analysis: Chemical compositions of the essential oil were analyzed by GC-MS (Agilent 7890-5975, Agilent Technologies Inc, USA). DM-WAX MS column (0.25 mm×30 m ID, 0.25 μm film thickness, Dikma Technologies Inc, USA) was used. The column temperature was programmed from 100 to 235°C at a 3°C/min first and then from 235 to 245°C at a 10°C/min, finally kept the final temperature for 10 min. The injector temperature was 250°C and flow rate of the carrier gas (He) was 0.8 mL/min. The essential oil was diluted with ethanol of 1:50. The volume of injected solution was 1 μL, splitless. Mass spectrometry analysis was performed in the Electron Impact (EI) ionization mode (70 eV) in the m/z range of 35-400 amu. The ion source temperature was 230°C and the interface line temperature was 280°C.

GC-O analysis: Aromatic profile of the essential oil was analyzed by GC-O instrument (Agilent 7890, Agilent Technologies Inc, USA). The column model, program temperature, injector temperature and other parameters were set the same as these in GC-MS analysis.

The essential oil was diluted with ethanol at 1:5 and 1:10 for injection, respectively. The aroma profiles, retention time and intension were recorded during the analysis process manually by three scent guiders.

Table 1: Five odor intensity notation

Intensity	The feeling sense of smell
0	Not detected
1	Weak, hardly recognizable odor
2	Clear and identify what kind of smell
3	Obviously but no intense odor
4	Ntense odor

Posterior intensity analysis: Posterior intensity method involves recording the odor intensity on a scale after a peak has eluted from the column (Van Ruth, 2001). The experienced experts measured and recorded the intensity of every aroma components by using five odor intensity notations, shown in Table 1. This posterior registration of the perceived intensity may cause a considerable variance between assessors (Zellner *et al*., 2008). However, this is a fast accurate method to find characteristics of aroma components from the essential oil.

Identification of compositions: Compositions of the oil were identified by direct comparison with authentic samples in some literatures (Adams, 2007; Miyazawa *et al*., 2007).

RESULTS AND DISCUSSION

The essential oil was obtained by steam distillation from the fruits of *Rosa laevigata* and achieved the yield of about 0.1%. In GC-MS, 67 compounds in the oil were detected, as shown in Fig. 1. 58 components were identified, as shown in Table 2, which took up about 99.97% of the total oil . In these compounds, long carbon chain acids predominated with about 51.69%, followed by esters (27.48%) and alcohol (1.40%). Some main compounds identified in the essential oil were palmitic acid (16.49%), cis-13-Octadecenoic acid (16.31%), linoleic acid (13.93%), ethyl linoleate (7.05%), ethyl palmitate (6.39%), ethyl oleate (6.39%),

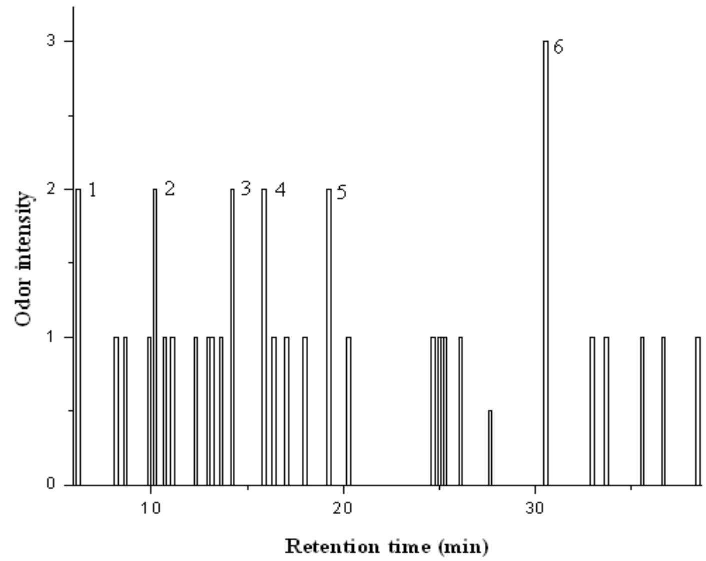

Fig. 2: Odor intensity of the compositions in the oil, 1: unkown; 2: unkown; 3: octanoic Acid; 4: 6, 10, 14-trimethyl-2-pentadecanone; 5: ethyl palmitates; 6: myristic acid

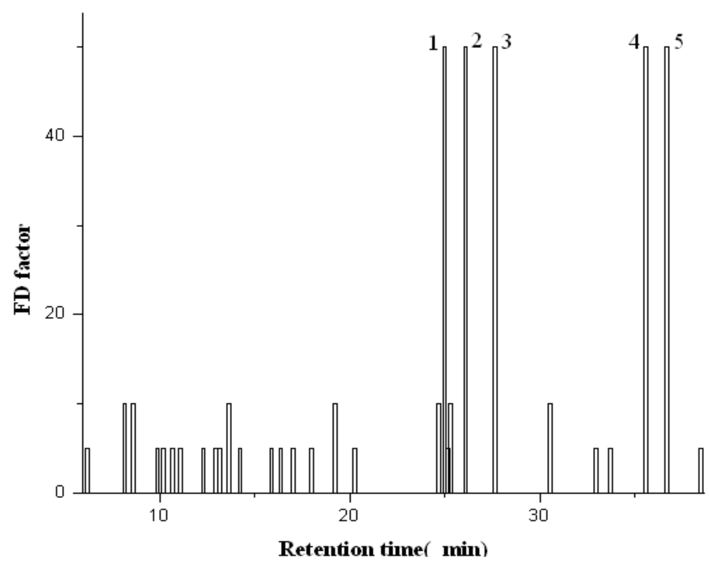

Fig. 3: FD factor of the compositions in the oil, 1: ethyl oleate; 2: ethyl linoleate; 3: ethyl linolenate; 4: palmitic acid; 5: 4, 8, 12, 16-tetramethyl heptadecan-4-olide

linolenic acid (6.04%), cis-9-Hexadecenoic acid (5.18%), ethyl linolenate (3.11%), octadecanoic acid (2.45%), cis-11-octadecenoic acid (2.20%), ethyl octadecanoate (1.40%), dodecanoic acid (1.14%), 1-acetoxyhexadecane (1.12%) and cetyl alcohol (1.01%).

The overall aroma of the oil was complex, smelled waxy, heavy refreshing and sweet and strong woody with a tiny tea. 24 aroma compounds in essential oil were identified by three scent guiders, as shown in Table 1. In these aromatic compounds, myristic acid had a strong refreshing and heavy burnt aroma accompanying with fishy smell. Therefore, it played an important role in the characteristic aroma of the essential oil. Using the value of Dilution Factor (FD) and Odor intensity, two histograms were obtained, as shown in Fig. 2 and 3, respectively. In Fig. 2, the odor intensity of myristic acid is the biggest of all the compounds. Octanoic acid, 6, 10, 14-trimethyl-2-pentadecanone, ethyl palmitates and two unknown compounds also have a relatively large odor intensity. All these compounds are the main ingredients for the odor of this essential oil. In Fig. 3, five compounds, ethyl oleate, ethyl linoleate, ethyl linolenate, palmitic acid and 4, 8, 12, 16-tetramethyl heptadecan-4-olide, whose FD factor reached 50 were considered to be the major impact for the odor of the essential oil. Structures of the five main aromatic compositions have been shown in Fig. 4.

Table 2: Chemical compositions and aromatic profiles of the essential oil from *R. laevigata*

No.	Retention time/min	Compound	Percentage/ %	FD	Odor intensity	Descriptor
1	6.21			5	2	Green odor
2	8.17	2,4-decadienal	0.09	10	1	Refreshing and light woody odor
3	8.64	ethyl laurate	0.03	10	1	Herbaceous, light taste and woody odor
4	9.12	hexanoic acid	0.07			
5	9.89			5	1	Tiny fruity and sweet odor
6	10.19			5	2	Fruity and sweet odor
7	10.69			5	1	Refreshing and sweet odor
8	11.10	citronellyl isobutyrate	0.01	5	1	Refreshing sweet and fruity odor
9	12.30			5	1	Fruity and burnt sweet odor
10	12.96	phenol	0.05	5	1	Refreshing sweet and green odor
11	13.17	3-methyl-5-propylnonane	0.07	5	1	Honey
12	13.64	ethyl tetradecanoate	0.30	10	1	Green odor
13	14.23	octanoic acid	0.11	5	2	Fruity and sweet odor
14	15.89	6,10,14-trimethyl-2-pentadecanone	0.42	5	2	Burnt sweet odor
15	16.37	ethyl pentadecanoate	0.07	5	1	Refreshing sweet and citrusy odor
16	17.02	nonanoic acid	0.11	5	1	Refreshing, similar tea and herbal odor
17	17.98	methyl palmitate	0.13	5	1	Fruity and green odor
18	19.23	ethyl palmitate	6.39	10	2	Refreshing and sweet odor
19	19.68	ethyl 9-hexadecenoate	0.59			
20	19.82	n-decanoic acid	0.07			
21	20.29	1-acetoxyhexadecane	1.12	5	1	Sweet odor
22	20.68	1,2-Hexadecene epoxide	0.05			
23	21.41	heptadecane	0.12			
24	21.90	ethyl heptadecanoate	0.06			
25	22.31	cetyl alcohol	1.01			
26	22.60	undecanoic acid	0.02			
27	22.86	(Z)-11-hexadecenol	0.05			
28	23.02	1,14-tetradecanediol	0.02			
29	23.32	1,11-dodecadiene	0.02			
30	23.49	methyl stearate	0.04			
31	23.84	methyl 11-octadecenoate	0.32			
32	24.66	ethyl octadecanoate	1.40	10	1	Tiny sweet odor
33	24.99	ethyl oleate	6.39	50	1	Refreshing and woody odor
34	25.14	elaidic acid ethyl ester	0.68	5	1	Engine oil odor
35	25.29	dodecanoic acid	1.14	10	1	Tiny engine oil odor
36	26.09	ethyl linoleate	7.05	50	1	Refreshing and sweet odor
37	26.58	nonadecane	0.12			
38	27.65	ethyl linolenate	3.11	50	0.5	Tiny gum odor
39	27.95	tridecanoic acid	0.06			
40	28.09	oleyl alcohol	0.21			
41	28.25	(Z)-11-hexadecenol	0.03			
42	28.50	phytol	0.05			
43	29.79	19-methyl-arachidic acid methyl ester	0.10			
44	30.15	9-Hexacosene	0.06			
45	30.26	Heptaethylene-glycol (6CI,7CI,8CI)	0.03			
46	30.51	myristic acid	2.54	10	3	Refreshing, heavy burnt odor with fishy smell
47	31.54	hexacosane	0.11			
48	31.65	polyethylene glycol octadecyl ether	0.17			
49	32.97	pentadecanoic acid	0.45	5	1	Sweet odor
50	33.33	5-dodecyldihydro-2(3H)-furanone	0.07			
51	33.69	oxacyclotetradecane-2,11-dione, 13-methyl-	0.79	5	1	Burnt sweet odor
52	35.57	Palmitic acid	16.49	50	1	Sour odor
53	36.25	cis-9-hexadecenoic acid	5.18			
54	36.50	cis-11-hexadecenoic acid	0.43			
55	36.66	4,8,12,16-tetramethyl heptadecan-4-olide	0.10	50	1	Refreshing and sweet odor
56	37.41	1-octadecyne	0.05			
57	37.76	heptadecanoic acid	0.34			
58	38.46	cis-10-Heptadecenoic acid	0.55	5	1	Woody
59	40.06	octadecanoic acid	2.45			
60	40.74	cis-13-octadecenoic acid	16.31			
61	40.83	cis-11-octadecenoic acid	2.20			
62	41.90	linoleic acid	13.93			
63	43.36	linolenic acid	6.04			

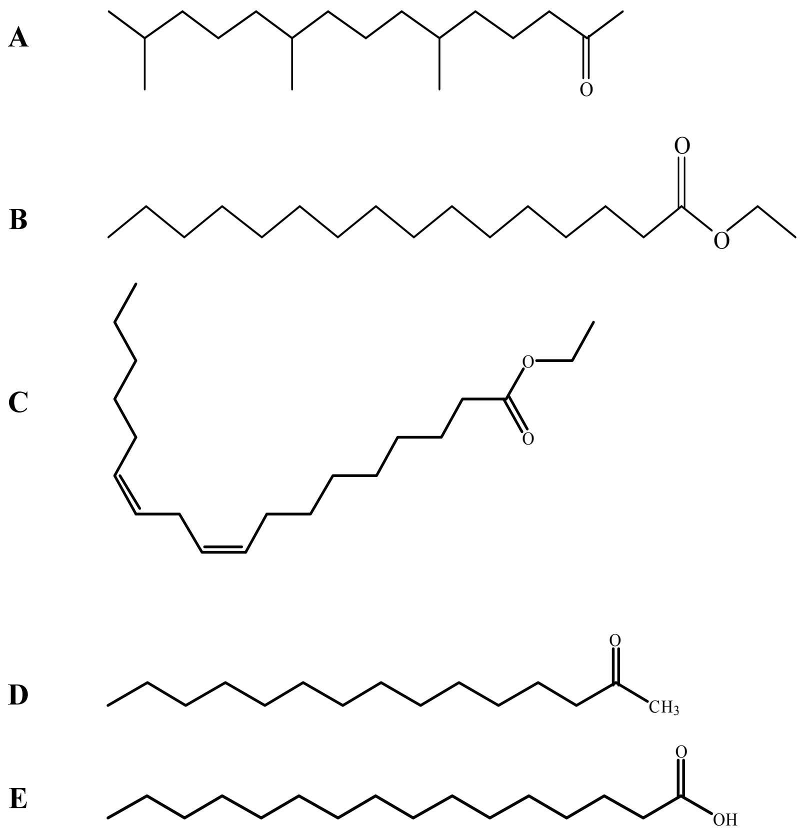

Fig. 4: Structures of five aromatic compositions; A: 6, 10, 14-trimethyl-2-pentadecanone; B: ethyl palmitate; C: ethyl linoleate; D: myristic acid; E: Palmitic acid

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