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Research Article

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Study on the volatiles variability of *Citrus reticulata* 'zhang shuensis' peels collected in different time

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ABSTRACT

Citrus Reticulata 'Zhang Shuensis' peels were collected in three different time points from the same plant growing in China. The peel oils extracted by hydrodistillation were investigated by GC-MS to study the changeability of oil components and their contents. It could be seen that the monoterpenes content was increased while the oxygenated compounds amount was decreased with the delay of harvested time. A total of 32 compounds were identified representing 98.4%, 98.2%, 98.8%, respectively. The main compounds in oils were limonene (65.8%, 69.4%, and 72.5%), γ-terpinene (7.5%, 8.1%, and 8.4%), and linalool (11.0%, 4.1%, 3.9%). These oils could be classified as the chemotype of limonene/linalool or limonene.

Keywords: volatile oil variability; *Citrus Reticulata* 'Zhang Shuensis'; *Citrus reticulata* Blanco; chemotype; *Citri Reticulatae Pericarpium Viride; Citri Reticulatae Pericarpium*

INTRODUCTION

Citrus reticulata Blanco is a very complicated species which include many cultivars. In China, the main cultivars include *C. Reticulata* 'Dahongpao', *C. Reticulata* 'Chachi', *C. Reticulata* 'Zhang Shuensis', etc. [1-3]. The dried fruit peel of *C. reticulata* Blanco or its cultivars collected in different time can be used as two kinds of Chinese Materia Medica (CMM) such as Citri Reticulatae Pericarpium Viride (CRPV) and Citri Reticulatae Pericarpium (CRP) due to its ripe extent. The main bioactive constituents of CRPV and CRP consist of essential oil, flavonoid, and alkaloid [3]. This study first investigated the volatiles variability of *C. Reticulata* 'Zhang Shuensis' peels collected in different time, and its oil chemotype was first analyzed, too.

EXPERIMENTAL SECTION

Peels of *C. Reticulata* 'Zhang Shuensis' were collected from the same plant growing in Zengfang country of Sanhu Town, Jiangxi province of China, in 7-15, 9-15, 11-15 of 2012 year differently. The peels were dried in natural ventilation condition. A voucher specimen was deposited in the Department of TCM, Chongqing Medical University.

The dry peel was smashed and then distilled in a Clevenger-type apparatus for 4 h. The oil was dried over anhydrous sodium sulphate for 24 h at 4 ˚C and then stored in a screw-capped vial until needed. The oil was analyzed by an Agilent 7890A/5975C GC-MS equipped with a capillary column HP-5MS (5% phenylmethylsiloxane) (30 m × 0.25 mm, film thickness 0.25 μm). Column temperature, 60°C (3 minutes) to 250°C at 4°C/minute; split ratio, 1:20; injector and detector temperature, 230°C; carrier gas, He 1 mL/minute; injected volume, 1 μL of a 1/10 solution of the oil in hexane. MS scan condition: interface temperature, 250°C; source temperature, 230°C; ionization voltage 70 eV; Scan at 2.29 scans s^{-1} from *m/z* 30-350. Solvent delay was 3 min. Data analyses were based on a MSD Chem Station E. 02. 01. 1177. Components identification was based on the comparison of their mass spectrum with those

referred spectrum stored in NIST08 library, and the comparison of their linear retention indices (LRIs) determined by the retention time of a homolog series of *n*-alkanes (C_8–C_{20}) on the apolar column, with those referred from NIST08. The quantitative result was obtained by peak area normalization.

RESULTS AND DISCUSSION

The qualitative and quantitative results are shown in Table 1. Thirty-two components including 12 monoterpenes, 7 sesquiterpenes, and 11 oxygenated compounds were determined representing 98.4%, 98.2%, 98.8%, respectively. Monoterpenes (79.6%, 89.7%, 93.0%) represented the main compound family in all oils. Its major compounds were *D*-limonene (65.8%, 69.4%, and 72.5%) and *γ*-terpinene (7.5%, 8.1%, and 8.4%). Sesquiterpenes amount was so low and only represented 2.5%, 1.4%, 0.3%, individually. Oxygenated compounds content was 15.6%, 6.2%, 5.3% respectively. Linalool (11.0%, 4.1%, 3.9%) was the prominent compounds. It could be seen that the monoterpenes content was increased while the oxygenated compounds amount was decreased with the delay of harvested time.

Lota et al. [4] first discriminate the oil chemotype of different *C. reticulata* cultivars by the contents of limonene and *γ*-terpinene. Enlighted and based on the research outcome by Lota et al., the author first systematically analyzed the oil chemotype by the content of four characteristic compounds such as limonene, *γ*-terpinene (≥9.0%), linalool (≥4.0%), benzoic acid, 2-methylamino-methyl ester (≥1.0%) [5]. Thus, the oil in this study can be classified as the chemotype of limonene/linalool or limonene.

Tab. 1 Main Compositions in the volatile oils

Components	LRI	%		
		7-15	9-15	11-15
α-Thujene	926	tr	0.2	0.2
α-Pinene	933	0.6	2.4	3.1
2H-Pyran, 2-ethenyltetrahydro-2,6,6-trimethyl-	971	0.2	nd	nd
Sabinen	973	tr	tr	0.1
β-Pinene	976	0.3	1.9	2.0
β-Myrcene	991	0.8	0.8	0.8
Cosmene	1006	tr	0.2	nd
α-Terpinene	1018	0.1	tr	tr
p-Cymene	1026	4.0	6.6	5.8
D-Limonene	**1033**	**65.8**	**69.4**	**72.5**
***γ*-Terpinene**	**1059**	**7.5**	**8.1**	**8.4**
Terpinolene	1089	0.4	0.3	0.3
Linalool	**1101**	**11.0**	**4.1**	**3.9**
Nonanal	1105	0.2	nd	tr
1,3,8-*p*-Menthatriene	1113	0.1	tr	nd
cis-*p*-Mentha-2,8-dien-1-ol	1137	0.1	tr	tr
Bicyclo[3.1.0]hexan-2-ol, 2-methyl-5-(1-methylethyl)-, (1α, 2α, 5α)-	1177	0.5	0.3	0.3
α-Terpineol	1192	2.4	0.7	0.6
Decanal	1207	0.2	nd	tr
unknown	1229	0.5	0.2	nd
Methyl thymyl ether	1236	0.2	0.1	tr
Carvone	1246	0.1	tr	nd
Thymol	1294	0.7	0.3	0.2
δ-Elemene	1339	0.1	nd	nd
β-Elemene	1394	0.4	0.1	nd
Methyl methanthranilate	**1411**	**tr**	**0.7**	**0.3**
γ-Elemene	1436	0.3	0.2	tr
(Z)-*β*-Farnesene	1459	0.6	0.2	nd
Germacrene D	1484	0.5	0.4	nd
α-Farnesene	1511	0.5	0.4	0.3
δ-Cadinene	1526	0.1	0.1	nd
unknown	1631	0.2	0.7	0.2
Class of substance				
Total compounds[a]		32(98.4)	28(98.2)	22(98.8)
Monoterpene hydrocarbons		12(79.6)	12(89.7)	10(93.0)
Sesquiterpene hydrocarbons		7(2.5)	6(1.4)	2(0.3)
Total oxygenated compounds		11(15.6)	8(6.2)	9(5.3)
unknown		2(0.7)	2(0.9)	1(0.2)

tr, trace (<0.1%); nd, not detected.

[a] Number of compounds. The value in brackets is total percentage.

CONCLUSION

This study demonstrated that the monoterpenes content was increased while the oxygenated compounds amount was decreased with the delay of harvested time. Such volatiles changeability was nearly the same as previous research outcome to other cultivars' peel oil by the author and his colleagues [6-7]. The oil in this study can be classified as the chemotype of limonene/linalool or limonene relative to the standard proposed by the author, which was most like the chemotype of *C. reticulata* 'Dahongpao' peel oil [5].

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