ACTA SCIENTIFIC AGRICULTURE (ISSN: 2581-365X)

Volume 4 Issue 7 July 2020

Research Article

Chemical Variation of Essential Oils from Peels of *Citrus kinokuni* Tanaka, *C. reticulata* 'Dahongpao', and *C. reticulata* 'Zhang Shuensis'

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Received: May 25, 2020

Published: June 04, 2020

Abstract

The chemicals in essential oils from peels of three varieties of *Citrus reticulata* Blanco like *C. kinokuni* Tanaka (K), *C. reticulata* 'Dahongpao' (D) and *C. reticulata* 'zhang shuensis' (Z) were investigated by gas chromatography-mass spectrometry (GC-MS) and gas chromatography-flame ionization detector (GC-FID), respectively. A total of 97 compounds were quantified in which 80 components were identified and 17 unknown components were detected. The quantitation results of D and Z were profoundly different from previous studies due to the different treatment to peels. The different oil-types from K, D, and Z were high in β-myrcene, p-cymene, limonene, γ-terpinene, α-terpineol, thymol, (E, E)-α-farnesene, spathulenol and isospathulenol. The chemicals' fluctuation in oils of D and Z was more pronounced than that of K. Principal component analysis (PCA) revealed the peel of K, D and Z could be classified as three kinds according to their chemical changeability, respectively, consisting with the usage of these peels in Chinese medicine. Lastly, the oils' chemotypes of K, D, Z and other cultivars were analyzed, showing that the % variation of six characteristic components including limonene, γ-terpinene, methyl N-methylanthranilate, linalool, (E, E)-α-farnesene, and thymol, played an important role in discriminating oil-types from varieties of *C. reticulata*.

Keywords: *Citrus reticulata* Blanco; Citrus kinokuni Tanaka; *C. reticulata* 'Dahongpao'; *C. reticulata* 'Zhang Shuensis'; Citri Pericarpium Reticulatae and Citri Pericarpium Reticulatae Viride; Essential Oils; Chemical Changeability; Chemotype

Introduction

n The essential oils obtained from different varieties of *C. reticulata* (family Rutaceae, Genus *Citrus* L.), represent valuable samples in the world flavor market. In China, the peels collected in different growing stage from some varieties such as *C. reticulata* 'Chachi', *C. reticulata* 'Dahongpao' (D), *C. kinokuni* Tanaka (K), *C. reticulata* 'zhang shuensis' (Z), *C. erythrosa* Tanaka, *C. reticulata* 'Tangerina', etc., can be used as two kinds of Chinese materia medica (CMM) such as Citri Pericarpium Reticulatae (CPR) and Citri Pericarpium Reticulatae Viride (CPRV). The dried pericarps of ripe fruits (CPR) are usually collected from September to December; the dry pericarps of immature fruits (CPRV) can be subdivided into Fructus Citri Immaturus (FCI) and CPRV. The little fruits (FCI) are collected in May or June; the dry pericarps (CPRV) are usually collected in July or August [1-4]. As a result, the peels can be classified as three kinds like FCI, CPRV, and CPR. In the view of Chinese medicine, CPRV usually with green color is potent in action, especially for FCI, while CPR is soft in treatment. Oils are the main active components of CPRV and CPR. K, Z and D, are three varieties native to China, some literatures have reported related study on the oils' constituents of D [22-23,25], while few studies were reported on the chemicals in oils from peels of K and Z despite its abundant growth and recognized use [26].

Aim of the Study

The aim of this study was to investigate the chemical changeability and to analyze the chemotype of different oil-types from the peels of K, D, Z.

Experimental Study

Materials

The peels of K were collected each month from June to November in 2012 from the same plant growing in Nancheng town, Fuzhou city, Jiangxi province of China. The peels of D were gathered every month from July to December in 2013 from three adjacent plants located in Jianlong town of Bishan county in Chongqing municipality of China. The peels of Z were collected each month from June to November of 2012 from plants growing in Zengfang country of Sanhu town, Xingan city, Jiangxi province of China (Table 1).

Chemical standards and reagents

Hexane with high-performance liquid chromatography (HPLC) grade, n-nonane (98%) used as an internal standard (IS), linalool (98%+), and methyl N-methylanthranilate were purchased from Adamas Reagent Company, Limited (Ltd.). d-Limonene (96%) was purchased from Acros organics, USA. Thymol was purchased from Shanghai Titan Chemistry Company, Ltd., China. Carvacrol was purchased from Tokyo Chemical Industry, Japan. n-Alkane standard solution of C_{10}-C_{25} purchased from Dr. Ehrenstorfer incorporated company, Germany, was used to determine linear retention index (LRI). n-Nonane (IS) hexane solution (0.5 μg/μL) was used for internal standardization purposes.

Oils' extraction

Since all peels were dried, distillation was used to extract oils. The peels of K divided into about 0.4 cm × 0.4 cm sections weighed

No.	Collected time (Year/Month/Date)	The color of dried peels	Classification	Extraction rate (%)
K1	2012/06/15	Black and green	FCI	2.33
K2	2012/07/15	Black and green	FCI	3.00
K3	2012/08/15	Yellow with green	CPRV	1.17
K4	2012/09/15	Yellow with green	CPRV	1.25
K5	2012/10/15	Yellow	CPR	1.75
K6	2012/11/15	Yellow	CPR	2.14
D1	2013/07/03	Black and green	FCI	0.08
D2	2013/08/07	Black and green	FCI	0.08
D3	2013/09/13	Green	CPRV	0.08
D4	2013/10/08	Green with yellow	CPRV	0.04
D5	2013/11/10	Red	CPR	0.04
D6	2013/12/08	Red	CPR	0.03
Z1	2012/06/15	Green and yellow	CPRV	0.17
Z2	2012/07/15	Green and black	CPRV	0.04
Z3	2012/08/15	Yellow with green	CPRV	0.04
Z4	2012/09/15	Yellow with black	CPRV	0.04
Z5	2012/10/15	Black with red	CPR	0.05
Z6	2012/11/15	Red with green	CPR	0.05

Table 1: Information about peels' collection and their oil extraction rate.

Note: In the first column 'No.', the first character like K, D, and Z before corresponding number denotes *C. kinokuni* Tanaka, *C. reticulata* 'Dahongpao', and *C. reticulata* 'zhang shuensis', respectively. Extraction rate of oils was calculated by the volume of oils (mL) dividing the weight of corresponding peels (g) and then multiplying 100%.

between 30.0 - 40.0g and the peels of D and Z respectively smashed into powder weighed between 25.0 - 45.0g, were swollen with about 10 times volume (V) 250 - 450 mL of pure water in a 500 or 1000 mL round-bottomed flask, then soaked for 0.5h at 40°C. The oils were extracted by Clevenger-type apparatus for 3 - 4h, then prepared according to the procedure described in Chinese pharmacopoeia [4] and then stored in separate screw-capped vials in a refrigerator at 4°C until needed. The oils were diluted by n-hexane to 1: 10 (V/V) and then subjected to analyses by GC-FID and GC-MS, except for sample K2 by GC-MS and D6 by GC-FID and GC-MS, which were diluted as 1: 20.

GC-FID analysis

A 9790II GC (FULI instrument, Zhejiang province of China) matched with a FID was used for GC-FID analysis. An Agilent fused silica capillary column - DB-5 (30m × 0.25 mm i.d., 0.25 μm film thickness) was used. The oven temperature was programmed from 60°C (3-min hold) to 270°C at 3°C min^{-1} and then held for 2 minutes. The carrier gas was nitrogen at a constant flow of 1 mL min^{-1}. The injector and detector were maintained at 280°C and 290°C, respectively. The splitting ratio was 20:1, injection volume was 1 μL. The detection was done in triplicate for each sample.

GC-MS analysis

An Agilent 7890B GC (USA) matched with an Agilent 5977A mass spectrometer was used for GC-MS analysis. The capillary column and oven temperature program were the same as that of GC-FID. The carrier gas was helium at a constant flow of 1 mL min^{-1}. The injector and ion-source were maintained at 280°C and 200°C, respectively. Transfer line was set to 280°C, splitting ratio was 20:1, solvent delay was 3 minutes. Electron impact mass spectra were taken at 70 eV. Scan at 3.9 scans s^{-1} from m/z 30 to 400 amu. The injection volume was 1 μL. The detection was done in duplicate for each sample.

Component identification

The peaks in total ion chromatograms (TICs) obtained by GC-MS were identified by probability based matching (PBM) searching first. Since the overlapped or even embedded peaks extensively exist in the TICs, the identification result may be incorrect for these peaks. In such conditions, AMDIS (Automated Mass spectral Deconvolution and Identification System) compiled in the GC-MS Chemstation was used to deconvolve these peaks. After deconvolution, the purified mass spectra of these peaks were compared with the NIST (National Institute of Standards and Technology) 08 database or the mass spectra of standards, respectively. The LRIs were calculated relatively to the retention time of a homolog series of n-alkanes (C_{10}-C_{25}) and then compared with the LRIs of the corresponding chemicals provided by NIST 08 database, literatures or standards.

Quantitation of components

The quantitation data of each sample were obtained from the mean value of three chromatograms obtained from GC-FID. The peak normalization was used to calculate the relative area percentage of each peak. The quantitation of concentration (μg/μL) was based on the peak area and response factor (RF) of each component. The standards' RF relative to IS was calculated using the following equation:

$$RF = C_{abs\ sta} \bullet A_{abs\ IS} / (A_{abs\ sta} \bullet C_{abs\ IS})$$

Herein, $C_{abs\ sta}$ or $C_{abs\ IS}$ was the absolute concentration of standard or IS dissolved in n-hexane, $A_{abs\ sta}$ or $A_{abs\ IS}$ was the absolute peak area of standard and IS, respectively. In this study, the $C_{abs\ sta}$ such as d-limonene, linalool, thymol, and methyl N-methylanthranilate was 0.42, 0.44, 3.70, 0.55 μg/μL, respectively, the Cabs IS of n-nonane was 0.50 μg/μL.,

The RFs of d-limonene, linalool, thymol, and methyl N-methylanthranilate relative to n-nonane were calculated as 0.9 ± 0.0, 0.7 ± 0.0, 1.2 ± 0.0, 1.7 ± 0.0, respectively. The RFs of identified components in chromatograms were determined by their functional groups compared with that of standards. In this way, the RFs of hydrocarbon monoterpene (HM), hydrocarbon sesquiterpene (HS) and polycyclic aromatic hydrocarbon were the same as that of d-limonene, the RFs of phenol and its ether were the same as that of thymol, the RFs of other oxygenated compounds were the same as that of linalool, the RFs of compounds with nitrogen were equal to that of methyl N-methylanthranilate and the RFs of alkane and cycloalkane were equal to that of n-nonane [5].

Statistical analysis

The data in table 2 and 3 were analyzed by PCA using SPSS-23 (Statistical Product and Service Solutions).

No.	LRI[a]	LRI[b]	Component	Area percentage ± SD (%)																	
				K1	K2	K3	K4	K5	K6	D1	D2	D3	D4	D5	D6	Z1	Z2	Z3	Z4	Z5	Z6
1	925	928*	α-Thujene	0.6 ± 0.0	0.3 ± 0.0	0.4 ± 0.1	0.3 ± 0.0	0.3 ± 0.0	0.3 ± 0.0	tr	nd	0.1 ± 0.0	tr	0.1 ± 0.0	0.1 ± 0.0	tr	0.1 ± 0.0	tr	0.1 ± 0.0	tr	0.1 ± 0.0
2	931	931*	**α**-Pinene	2.2 ± 0.1	1.8 ± 0.1	1.7 ± 0.2	1.4 ± 0.1	1.4 ± 0.1	1.5 ± 0.1	0.4 ± 0.1	0.1 ± 0.1	0.4 ± 0.2	0.6 ± 0.0	0.8 ± 0.1	0.6 ± 0.3	0.6 ± 0.3	0.8 ± 0.3	0.4 ± 0.1	0.8 ± 0.3	0.5 ± 0.4	0.5 ± 0.3
3	971	975*	Sabinene	0.2 ± 0.0	0.1 ± 0.0	0.3 ± 0.2	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	nd	nd	tr	nd	tr	tr	nd	nd	nd	nd	nd	nd
4	975	970*	**β**-Pinene	6.4 ± 0.2	2.4 ± 0.2	3.2 ± 0.3	2.3 ± 0.1	2.2 ± 0.2	2.1 ± 0.2	0.1 ± 0.0	nd	0.2 ± 0.1	0.2 ± 0.0	0.3 ± 0.0	0.7 ± 0.5	0.2 ± 0.2	0.3 ± 0.2	0.1 ± 0.0	0.1 ± 0.0	tr	0.2 ± 0.1
5	989	986 (6)	**β**-Myrcene	1.4 ± 0.0	1.9 ± 0.1	1.4 ± 0.1	1.6 ± 0.1	1.7 ± 0.2	1.8 ± 0.2	0.7 ± 0.1	0.4 ± 0.3	0.5 ± 0.3	1.1 ± 0.1	1.0 ± 0.1	0.8 ± 0.4	0.6 ± 0.3	0.9 ± 0.2	0.3 ± 0.1	0.1 ± 0.1	0.4 ± 0.4	0.6 ± 0.2
6	1004	1007*	**α**-Phellandrene	0.1 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	nd	0.1 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	tr	0.1 ± 0.0
7	1016	1017*	**α**-Terpinene	0.3 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	nd	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	nd	nd	nd	nd	nd	nd
8	1022	1020 (6)	*p*-Cymene.	22.9 ± 0.1	11.3 ± 0.1	12.3 ± 0.5	10.8 ± 0.2	8.0 ± 0.2	7.1 ± 0.0	0.8 ± 0.1	0.5 ± 0.6	1.5 ± 1.1	1.2 ± 0.1	0.5 ± 0.1	0.6 ± 0.2	1.1 ± 0.2	1.4 ± 0.2	0.9 ± 0.1	0.3 ± 0.1	0.3 ± 0.2	0.7 ± 0.2
9	1028	1020*	Limonene	45.6 ± 0.2	62.7 ± 0.6	59.3 ± 2.5	64.5 ± 1.1	69.1 ± 2.2	69.4 ± 0.1	21.3 ± 3.1	13.0 ± 14.5	17.4 ± 13.9	26.1 ± 2.4	50.8 ± 5.6	40.4 ± 18.3	22.9 ± 12.4	38.3 ± 5.9	7.8 ± 1.1	3.5 ± 1.0	28.5 ± 34.4	32.5 ± 15.5
10	1047	1038 (6)	**β**-*trans*-Ocimene	0.6 ± 0.0	0.3 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	nd	0.1 ± 0.0	tr	0.1 ± 0.0	0.1 ± 0.0	tr	0.1 ± 0.0	tr	tr	tr	tr
11	1055	1053*	γ-Terpinene	7.3 ± 0.1	7.8 ± 0.5	4.5 ± 0.2	5.0 ± 0.3	4.9 ± 0.4	5.2 ± 0.5	1.1 ± 0.1	0.9 ± 0.6	0.8 ± 0.5	1.1 ± 0.1	1.9 ± 0.2	1.2 ± 0.6	1.2 ± 0.7	2.1 ± 0.3	0.9 ± 0.1	0.6 ± 0.2	0.4 ± 0.3	1.7 ± 0.5
12	1086	1081*	*p*-Cymenene	0.1 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	tr	tr	tr	nd	nd	nd	nd	nd	nd	0.3 ± 0.1	0.3 ± 0.2	0.6 ± 0.1	0.5 ± 0.3	0.3 ± 0.2	0.3 ± 0.2
13	1086	1078*	Terpinolene	0.6 ± 0.0	0.5 ± 0.0	0.3 ± 0.0	0.3 ± 0.0	0.3 ± 0.0	0.3 ± 0.0	0.4 ± 0.1	0.2 ± 0.2	0.2 ± 0.1	0.6 ± 0.1	0.4 ± 0.0	0.3 ± 0.1	nd	0.1 ± 0.0	nd	nd	nd	0.1 ± 0.0
14	1097	1092 (6)	Linalool	3.3 ± 0.1	2.5 ± 1.2	5.2 ± 1.9	4.2 ± 0.6	2.8 ± 0.2	2.6 ± 0.1	0.3 ± 0.1	0.4 ± 0.4	0.7 ± 0.3	0.8 ± 0.1	0.2 ± 0.1	0.3 ± 0.1	0.8 ± 0.4	0.8 ± 0.2	0.6 ± 0.1	0.6 ± 0.2	1.4 ± 1.6	2.1 ± 2.3
15	1100	1090*	Hotrienol	tr	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.1	0.8 ± 0.4	3.7 ± 0.4	0.3 ± 0.0	0.4 ± 0.2	0.3 ± 0.1	0.4 ± 0.1	0.8 ± 0.1	0.8 ± 0.3	0.5 ± 0.3	0.5 ± 0.1
16	1109	1104*	1,3,8-*p*-Menthatriene	tr	0.1 ± 0.1	0.1 ± 0.0	tr	tr	tr	nd	nd	0.1 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	tr	0.1 ± 0.1	0.1 ± 0.1	nd	tr	0.1 ± 0.1

17	1116	1113*	*trans-p*-Mentha-2,8-dienol	tr	tr	0.2 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.0	0.6 ± 0.1	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.1	0.2 ± 0.0	0.1 ± 0.1	0.2 ± 0.0
18	1130	1134 (7)	*cis-p*-Mentha-2,8-dien-1-ol	tr	tr	0.2 ± 0.0	0.1 ± 0.0	tr	0.1 ± 0.0	nd	nd	0.1 ± 0.0	0.5 ± 0.2	tr	0.1 ± 0.0	tr	0.1 ± 0.0	0.1 ± 0.0	tr	0.1 ± 0.0	tr
19	1135	1138*	2-Cyclohexen-1-ol, 1-methyl-4-(1-methylethyl)-, trans-	0.1 ± 0.0	tr	0.1 ± 0.0	0.1 ± 0.0	tr	tr	nd	nd	nd	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.1	tr	0.1 ± 0.0	0.2 ± 0.0	tr	0.1 ± 0.0	0.1 ± 0.0
20	1140	1137 (6)	**β**-Terpineol	tr	0.1 ± 0.0	tr	tr	tr	tr	tr	0.1 ± 0.0	nd	nd	nd	0.1 ± 0.0	nd	nd	nd	nd	nd	nd
21	1161	1164 (6)	Borneol	0.1 ± 0.0	0.1 ± 0.0	tr	tr	tr	tr	0.1 ± 0.0	tr	tr	0.1 ± 0.0	tr	0.1 ± 0.0	nd	nd	nd	nd	nd	nd
22	1172		Unknown-1 (OM)	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	tr	tr	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
23	1174	1175 (6)	Terpinen-4-ol	1.0 ± 0.0	0.7 ± 0.1	0.7 ± 0.0	0.6 ± 0.1	0.6 ± 0.1	0.5 ± 0.0	0.1 ± 0.0	0.1 ± 0.1	0.2 ± 0.1	0.3 ± 0.0	0.1 ± 0.0	0.3 ± 0.1	0.3 ± 0.1	0.3 ± 0.0	0.3 ± 0.0	0.2 ± 0.1	0.2 ± 0.2	0.3 ± 0.1
24	1181	1172*	*p*-Cymen-8-ol	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.0	0.1 ± 0.1	0.2 ± 0.0	0.4 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	nd	nd	nd	nd	nd	nd
25	1183	1186*	Cyclohexanol, 2-methylene-5-(1-methylethenyl)-	tr	0.1 ± 0.0	0.3 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.3 ± 0.1	1.2 ± 0.1	0.1 ± 0.0	0.2 ± 0.1	0.1 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	0.1 ± 0.1	0.1 ± 0.0
26	1186	1185 (6)	α-Terpineol	0.9 ± 0.0	1.7 ± 0.2	1.0 ± 0.0	0.8 ± 0.1	0.7 ± 0.1	0.7 ± 0.1	0.6 ± 0.1	1.0 ± 0.2	0.4 ± 0.1	0.6 ± 0.1	0.2 ± 0.1	0.5 ± 0.2	0.6 ± 0.2	0.7 ± 0.1	0.7 ± 0.1	0.7 ± 0.2	0.3 ± 0.2	0.3 ± 0.1
27	1192	1183 (6)	Dihydrocarvone	tr	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	tr	tr	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.4 ± 0.1	tr	0.1 ± 0.1	nd	nd	nd	nd	nd	nd
28	1196	1192 (8)	*trans*-Isopiperitenol	tr	tr	0.2 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	tr	nd	0.2 ± 0.1	0.7 ± 0.1	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	0.2 ± 0.1	0.1 ± 0.0
29	1202	1193 (9)	Decanal	tr	tr	tr	tr	tr	tr	0.2 ± 0.0	nd	0.3 ± 0.2	0.7 ± 0.1	0.6 ± 0.1	0.6 ± 0.2	tr	0.1 ± 0.1	0.1 ± 0.1	tr	0.1 ± 0.0	0.1 ± 0.0
30	1214	1209 (6)	*trans*-Carveol	0.1 ± 0.0	0.1 ± 0.0	0.4 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.0	0.4 ± 0.3	0.6 ± 0.1	1.6 ± 0.2	0.5 ± 0.1	0.7 ± 0.3	0.6 ± 0.2	0.8 ± 0.1	0.9 ± 0.1	0.8 ± 0.2	0.8 ± 0.5	0.8 ± 0.2
31	1223		Unknown-2 (AM)	0.1 ± 0.0	0.1 ± 0.0	0.4 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.3 ± 0.1	0.3 ± 0.1	0.8 ± 0.3	1.7 ± 0.2	0.4 ± 0.0	0.3 ± 0.1	nd	nd	nd	nd	nd	nd
32	1225	1215 (6)	β-Citronellol	nd	nd	nd	nd	nd	nd	0.1 ± 0.0	0.1 ± 0.1	0.1 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	0.5 ± 0.2	nd	nd	nd	nd	nd	nd
33	1226	1222 (6)	*cis*-Carveol	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	tr	tr	tr	tr	tr	tr	tr	tr	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	tr
34	1231	1237 (10)	Thymol methyl ether	0.1 ± 0.0	tr	tr	tr	tr	tr	0.1 ± 0.0	tr	0.2 ± 0.2	0.2 ± 0.0	0.2 ± 0.0	0.1 ± 0.1	tr	tr	0.1 ± 0.1	tr	tr	0.1 ± 0.1
35	1237	1228 (6)	Carvone	tr	0.1 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	nd	0.1 ± 0.0	0.2 ± 0.0	nd	tr	nd	nd	nd	nd	nd	nd

36	1250	1243 (6)	*trans*-Geraniol	0.1 ± 0.0	0.1 ± 0.0	tr	0.1 ± 0.0	tr	tr	0.1 ± 0.0	0.2 ± 0.1	0.1 ± 0.1	0.2 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.1	0.2 ± 0.0	0.4 ± 0.1	0.2 ± 0.1	0.1 ± 0.1	0.1 ± 0.0
37	1268	1271 (11)	Perillaldehyde	tr	tr	tr	tr	tr	tr	0.1 ± 0.0	nd	0.1 ± 0.1	0.4 ± 0.1	nd	0.1 ± 0.0	nd	nd	nd	nd	nd	nd
38	1281		Unknown-3 (IT)	tr	tr	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.4 ± 0.2	0.3 ± 0.0	0.4 ± 0.0	0.2 ± 0.0	0.5 ± 0.2	0.3 ± 0.1	0.7 ± 0.1	0.2 ± 0.0	0.7 ± 0.3	0.2 ± 0.2	0.9 ± 0.3
39	1287	1290 (12)	Thymol	1.8 ± 0.0	0.6 ± 0.1	1.9 ± 0.1	1.4 ± 0.2	1.4 ± 0.2	1.3 ± 0.1	1.7 ± 0.3	1.8 ± 0.1	3.7 ± 0.9	4.9 ± 0.5	3.5 ± 0.5	4.2 ± 1.3	2.5 ± 2.0	2.5 ± 0.7	3.4 ± 0.6	6.0 ± 3.8	2.4 ± 1.8	1.6 ± 0.4
40	1292		Unknown-4 (IT)	tr	tr	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	tr	0.1 ± 0.0	0.2 ± 0.1	0.2 ± 0.1	0.2 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.4 ± 0.1	0.4 ± 0.0	0.2 ± 0.0	0.2 ± 0.1	tr
41	1297	1297 (6)	Carvacrol	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.0	0.3 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.1	0.2 ± 0.0	0.4 ± 0.0	0.6 ± 0.1	0.2 ± 0.0	0.4 ± 0.1	0.7 ± 0.6	0.5 ± 0.4	0.6 ± 0.1	1.8 ± 1.4	0.5 ± 0.5	0.1 ± 0.1
42	1306	1311 (13)	2-Methoxy-4-vinylphenol	tr	tr	tr	tr	tr	tr	0.3 ± 0.0	0.3 ± 0.2	0.4 ± 0.1	1.0 ± 0.1	0.6 ± 0.2	0.3 ± 0.1	0.3 ± 0.2	0.6 ± 0.0	0.5 ± 0.1	0.7 ± 0.3	0.4 ± 0.1	0.3 ± 0.2
43	1335	1334*	δ-Elemene	0.1 ± 0.0	0.1 ± 0.0	tr	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	tr	nd	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	tr	tr	tr	tr	nd	0.2 ± 0.0
44	1366	1374 (14)	*n*-Decanoic acid	tr	tr	tr	tr	tr	tr	0.2 ± 0.1	0.2 ± 0.0	tr	tr	0.1 ± 0.0	0.1 ± 0.0	nd	tr	tr	tr	nd	tr
45	1389	1387*	β-Elemene	0.1 ± 0.0	tr	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	nd	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	tr	0.4 ± 0.1
46	1430	1432 (15)	γ-Elemene	tr	tr	tr	tr	tr	tr	0.2 ± 0.1	0.2 ± 0.2	0.4 ± 0.0	0.2 ± 0.0	0.6 ± 0.1	0.4 ± 0.0	0.2 ± 0.0	0.4 ± 0.1	0.4 ± 0.1	0.5 ± 0.2	0.3 ± 0.1	0.8 ± 0.2
47	1478	1480*	Germacrene D	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	0.2 ± 0.1	0.1 ± 0.0	tr	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.1	tr	0.2 ± 0.1
48	1492	1500*	α-Selinene	0.1 ± 0.0	0.1 ± 0.0	tr	tr	tr	0.1 ± 0.0	0.2 ± 0.1	0.1 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.4 ± 0.1	0.2 ± 0.1	nd	nd	nd	nd	nd	nd
49	1507	1499*	(*E*, *E*)-α-Farnesene.	0.9 ± 0.0	1.0 ± 0.2	0.7 ± 0.1	0.7 ± 0.1	0.8 ± 0.1	1.1 ± 0.1	0.1 ± 0.0	nd	0.2 ± 0.0	0.1 ± 0.0	0.3 ± 0.0	0.1 ± 0.0	0.2 ± 0.2	0.2 ± 0.1	0.3 ± 0.1	0.2 ± 0.2	0.1 ± 0.0	0.3 ± 0.1
50	1520	1514*	δ-Cadinene	0.1 ± 0.0	tr	tr	tr	tr	tr	0.3 ± 00	0.2 ± 0.0	0.6 ± 0.2	0.4 ± 0.1	0.8 ± 0.1	0.4 ± 0.0	0.3 ± 0.1	0.7 ± 0.1	0.8 ± 0.1	1.3 ± 0.4	0.4 ± 0.2	1.1 ± 0.3
51	1545	1535*	Elemol	tr	tr	tr	tr	tr	tr	0.4 ± 0.1	0.3 ± 0.1	0.6 ± 0.1	0.3 ± 0.0	0.4 ± 0.0	0.4 ± 0.1	0.3 ± 0.0	0.3 ± 0.0	0.6 ± 0.1	0.3 ± 0.1	0.4 ± 0.2	0.7 ± 0.2
52	1553	1554*	Germacrene B	tr	nd	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	0.4 ± 0.1	0.4 ± 0.2	0.8 ± 0.1	0.8 ± 0.1	0.6 ± 0.4	0.6 ± 0.3
53	1564	1556*	Dodecanoic acid	tr	tr	tr	tr	tr	tr	0.8 ± 0.1	0.7 ± 0.3	1.6 ± 0.3	0.9 ± 0.4	0.8 ± 0.1	1.7 ± 0.8	tr	tr	0.2 ± 0.0	tr	tr	tr
54	1574	1569*	Spathulenol	0.4 ± 0.0	0.1 ± 0.0	0.4 ± 0.1	0.3 ± 0.0	0.3 ± 0.0	0.3 ± 0.0	1.4 ± 0.1	1.3 ± 0.4	2.4 ± 0.4	1.6 ± 0.2	1.2 ± 0.1	1.4 ± 0.2	1.6 ± 0.1	1.4 ± 0.1	2.9 ± 0.4	2.4 ± 0.6	1.6 ± 0.8	1.6 ± 0.3
55	1580	1578*	Globulol	tr	tr	tr	tr	tr	tr	0.5 ± 0.0	0.4 ± 0.0	0.5 ± 0.1	0.3 ± 0.0	0.3 ± 0.0	0.3 ± 0.1	0.3 ± 0.1	0.3 ± 0.0	0.6 ± 0.1	0.3 ± 0.3	0.3 ± 0.1	0.3 ± 0.0
56	1588		Unknown-***5*** (AS)	tr	tr	tr	tr	tr	tr	0.5 ± 0.1	0.4 ± 0.1	0.3 ± 0.1	0.3 ± 0.2	0.5 ± 0.0	0.4 ± 0.2	nd	nd	nd	nd	nd	nd

57	1607		Unknown-***6*** (AS)	tr	tr	tr	tr	tr	tr	0.9 ± 0.3	0.5 ± 0.2	1.0 ± 0.1	0.4 ± 0.1	0.4 ± 0.0	0.4 ± 0.2	1.3 ± 0.1	0.9 ± 0.1	2.4 ± 0.4	2.5 ± 0.8	1.4 ± 0.6	1.0 ± 0.2
58	1614	1624*	Selina-6-en-4-ol	tr	tr	tr	tr	tr	tr	0.4 ± 0.1	0.3 ± 0.0	0.2 ± 0.1	0.1 ± 0.0	0.3 ± 0.1	0.3 ± 0.1	0.2 ± 0.0	0.2 ± 0.1	0.5 ± 0.3	0.3 ± 0.3	0.2 ± 0.2	0.1 ± 0.0
59	1621	1628*	.tau.-Cadinol	tr	tr	nd	tr	tr	tr	0.3 ± 0.1	0.2 ± 0.1	0.4 ± 0.2	0.1 ± 0.0	0.3 ± 0.0	0.3 ± 0.1	0.1 ± 0.0	tr	0.2 ± 0.2	0.2 ± 0.0	tr	0.1 ± 0.1
60	1626	1630*	Isospathulenol	0.2 ± 0.0	0.1 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	5.0 ± 0.4	4.7 ± 1.3	9.0 ± 2.7	4.2 ± 0.5	3.3 ± 0.1	4.0 ± 1.1	7.6 ± 0.2	4.8 ± 0.4	11.3 ± 1.2	10.1 ± 2.7	7.0 ± 3.4	6.5 ± 1.3
61	1628	1627*	γ-Eudesmol	tr	tr	tr	0.1 ± 0.0	tr	0.1 ± 0.0	0.8 ± 0.1	0.9 ± 0.1	0.8 ± 0.3	0.5 ± 0.0	0.8 ± 0.0	0.7 ± 0.2	0.6 ± 0.0	0.5 ± 0.0	0.9 ± 0.1	0.9 ± 0.3	0.6 ± 0.3	1.0 ± 0.2
62	1638	1628*	.tau.-Muurolol	tr	tr	tr	tr	tr	tr	0.7 ± 0.2	0.6 ± 0.1	0.7 ± 0.2	0.4 ± 0.1	0.6 ± 0.1	0.6 ± 0.2	nd	nd	nd	nd	nd	nd
63	1643	1646*	Torreyol	tr	tr	tr	tr	tr	tr	0.8 ± 0.1	0.7 ± 0.1	1.1 ± 0.5	0.5 ± 0.0	0.3 ± 0.0	0.5 ± 0.1	0.9 ± 0.1	0.6 ± 0.0	1.6 ± 0.2	1.5 ± 0.4	0.9 ± 0.4	0.8 ± 0.1
64	1646	1644*	β-Eudesmol	tr	tr	tr	tr	tr	0.1 ± 0.0	2.0 ± 0.1	1.8 ± 0.2	3.1 ± 1.1	1.1 ± 0.1	1.4 ± 0.0	1.6 ± 0.6	2.5 ± 0.1	1.5 ± 0.1	4.1 ± 0.4	3.6 ± 1.0	2.4 ± 1.2	3.0 ± 0.6
65	1650	1641*	α-Cadinol	0.1 ± 0.0	tr	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	2.4 ± 0.2	2.0 ± 0.1	2.8 ± 0.8	1.2 ± 0.0	1.4 ± 0.1	1.6 ± 0.6	2.3 ± 0.2	1.4 ± 0.1	3.4 ± 0.4	3.2 ± 0.9	2.5 ± 1.1	2.4 ± 0.4
66	1667		Unknown-***7*** (AS)	tr	tr	nd	nd	nd	nd	0.4 ± 0.1	0.4 ± 0.0	0.6 ± 0.2	0.2 ± 0.1	0.2 ± 0.0	0.3 ± 0.1	nd	nd	nd	nd	nd	nd
67	1675		Unknown-8 (AS)	tr	tr	tr	tr	tr	tr	0.3 ± 0.0	0.3 ± 0.0	0.6 ± 0.3	0.2 ± 0.0	0.1 ± 0.0	0.2 ± 0.1	0.8 ± 0.2	0.5 ± 0.1	1.3 ± 0.3	1.6 ± 0.6	0.7 ± 0.4	0.6 ± 0.1
68	1681		Unknown-9 (AS)	nd	nd	nd	nd	nd	nd	1.5 ± 0.1	1.5 ± 0.3	2.5 ± 1.6	0.9 ± 0.1	0.3 ± 0.0	0.6 ± 0.3	2.4 ± 0.3	1.1 ± 0.1	4.0 ± 0.5	3.6 ± 1.1	2.5 ± 1.3	1.2 ± 0.3
69	1691	1681*	Juniper camphor	tr	tr	tr	tr	tr	tr	0.4 ± 0.1	0.4 ± 0.1	0.3 ± 0.1	0.1 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	0.2 ± 0.1	0.3 ± 0.2	0.2 ± 0.2	0.4 ± 0.1
70	1703		Unknown-10 (AS)	nd	nd	nd	nd	nd	nd	0.8 ± 0.3	0.9 ± 0.1	0.9 ± 0.4	0.5 ± 0.1	0.2 ± 0.0	0.3 ± 0.1	nd	nd	nd	nd	nd	nd
71	1712		Unknown-11 (AS)	tr	tr	tr	tr	tr	tr	0.6 ± 0.1	0.6 ± 0.0	1.0 ± 0.3	0.5 ± 0.2	0.2 ± 0	0.4 ± 0.2	1.0 ± 0.2	0.5 ± 0.1	1.3 ± 0.2	1.5 ± 0.5	0.9 ± 0.5	0.9 ± 0.4
72	1715	1714*	6-Isopropenyl-4, 8a-dimethyl-1, 2, 3, 5, 6, 7, 8, 8a-octahydro-naphthalen-2-ol	tr	tr	tr	tr	tr	nd	0.5 ± 0.1	0.5 ± 0.0	1.0 ± 0.5	0.4 ± 0.0	0.2 ± 0.1	0.2 ± 0.1	1.4 ± 0.5	0.6 ± 0.2	1.8 ± 0.7	1.8 ± 0.9	1.2 ± 0.9	0.8 ± 0.3

73	1715		Unknown-12 (AS)	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	0.9 ± 0.1	0.3 ± 0.0	1.3 ± 0.2	1.4 ± 0.5	0.8 ± 0.5	0.4 ± 0.1
74	1749	1758 (10)	**α**-Sinensal	tr	tr	tr	tr	tr	tr	2.0 ± 0.1	1.4 ± 0.8	0.7 ± 0.1	1.0 ± 0.0	0.7 ± 0.0	1.1 ± 0.6	0.8 ± 0.6	0.4 ± 0.2	0.6 ± 0.2	0.3 ± 0.0	0.7 ± 0.5	0.2 ± 0.0
75	1759	1754*	7R, 8R-8-Hydroxy-4-isopropylidene-7-methylbicyclo[5. 3. 1] undec-1-ene	tr	tr	tr	0.1 ± 0.0	tr	tr	0.4 ± 0.0	0.6 ± 0.2	0.7 ± 0.2	0.6 ± 0.2	0.6 ± 0.5	0.2 ± 0.1	0.7 ± 0.1	0.3 ± 0.1	0.8 ± 0.0	1.3 ± 0.5	0.7 ± 0.4	0.5 ± 0.1
76	1842	1838*	2-Pentadecanone, 6, 10, 14-trimethyl-	tr	tr	tr	tr	tr	tr	0.5 ± 0.0	0.5 ± 0.3	0.4 ± 0.1	0.5 ± 0.1	0.2 ± 0.0	0.2 ± 0.2	0.1 ± 0.0	tr	0.1 ± 0.1	0.1 ± 0.0	tr	tr
77	1858	1857 (16)	Pentadecanoic acid	nd	tr	tr	tr	tr	tr	0.7 ± 0.2	1.0 ± 0.6	tr	0.2 ± 0.1	tr	0.1 ± 0.1	nd	nd	nd	nd	nd	nd
78	1923	1927 (17)	Hexadecanoic acid, methyl ester	nd	nd	nd	nd	nd	nd	0.5 ± 0.1	0.5 ± 0.2	0.3 ± 0.1	0.3 ± 0.0	0.1 ± 0.0	0.2 ± 0.1	0.3 ± 0.1	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.1	0.2 ± 0.2	0.3 ± 0.0
79	1945	1938*	Isophytol	nd	nd	nd	nd	nd	nd	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	tr	tr	tr	nd	nd	nd	nd	nd	nd
80	1960		Unknown-13 (OS)	tr	tr	nd	tr	tr	tr	nd	nd	0.7 ± 0.5	tr	tr	nd	nd	nd	nd	nd	nd	nd
81	1982	1972*	*n*-Hexadecanoic acid	tr	tr	tr	0.1 ± 0.1	0.2 ± 0.0	0.1 ± 0.0	20.1 ± 3.3	26.8 ± 12.4	8.3 ± 4.4	9.3 ± 2.9	5.6 ± 3.9	8.2 ± 8.5	13.4 ± 7.9	7.0 ± 3.3	8.8 ± 3.5	6.3 ± 0.8	10.5 ± 10.5	2.0 ± 1.7
82	1992	1992 (18)	Hexadecanoic acid, ethyl ester	tr	tr	tr	tr	tr	tr	0.2 ± 0.0	0.2 ± 0.1	0.1 ± 0.1	0.2 ± 0.1	0.2 ± 0.1	0.2 ± 0.1	nd	tr	nd	nd	nd	0.1 ± 0.1
83	2089	2087*	Linoleic acid, methyl ester	tr	tr	tr	tr	tr	tr	0.3 ± 0.1	0.2 ± 0.1	0.1 ± 0.1	0.1 ± 0.1	0.1 ± 0.1	0.2 ± 0.2	0.1 ± 0.0	tr	0.1 ± 0.1	0.1 ± 0.0	tr	0.1 ± 0.0
84	2095	2098*	Linolenic acid, methyl ester	tr	tr	tr	tr	tr	tr	0.4 ± 0.1	0.3 ± 0.1	0.2 ± 0.1	0.2 ± 0.0	0.1 ± 0.1	0.1 ± 0.1	0.3 ± 0.3	tr	0.1 ± 0.0	tr	0.2 ± 0.0	tr
85	2109	2104*	Phytol	nd	nd	nd	nd	nd	nd	0.3 ± 0.1	0.2 ± 0.1	0.6 ± 0.0	0.2 ± 0.1	0.2 ± 0.1	0.1 ± 0.2	0.5 ± 0.3	0.1 ± 0.0	0.2 ± 0.1	0.2 ± 0.1	0.4 ± 0.4	tr
86	2124	2133*	Octadecanoic acid, methyl ester	nd	nd	nd	nd	nd	tr	0.1 ± 0.1	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.1	tr	0.1 ± 0.1	0.4 ± 0.4	0.3 ± 0.3	0.2 ± 0.2	0.1 ± 0.0	0.1 ± 0.1	0.1 ± 0.0
87	2136	2131 (16)	Linoleic acid	nd	tr	tr	tr	tr	tr	2.5 ± 1.3	4.9 ± 3.1	0.5 ± 0.2	0.5 ± 0.3	0.7 ± 0.9	0.9 ± 1.2	1.1 ± 1.0	0.5 ± 0.3	0.5 ± 0.3	0.1 ± 0.1	0.2 ± 0.3	0.1 ± 0.0
88	2141	2134 (19)	Linolenic acid	tr	nd	tr	nd	nd	tr	1.5 ± 0.7	2.6 ± 1.6	0.1 ± 0.0	0.4 ± 0.2	0.6 ± 0.8	0.6 ± 0.8	0.8 ± 0.5	0.3 ± 0.1	0.3 ± 0.1	0.4 ± 0.5	0.5 ± 0.3	0.3 ± 0.2

89	2146		Unknown-14 (AD)	nd	tr	tr	tr	tr	tr	0.5 ± 0.0	0.3 ± 0.2	0.3 ± 0.2	0.3 ± 0.1	0.1 ± 0.0	0.2 ± 0.2	nd	nd	nd	nd	nd	nd
90	2159		Unknown-15 (OD)	nd	nd	nd	nd	nd	nd	0.2 ± 0.0	0.1 ± 0.1	0.2 ± 0.1	0.4 ± 0.1	0.1 ± 0.0	0.1 ± 0.1	nd	nd	nd	nd	nd	nd
91	2163	2166*	Linolenic acid, ethyl ester	tr	tr	tr	tr	nd	tr	0.1 ± 0.0	0.1 ± 0.0	tr	nd	tr	tr	nd	nd	nd	nd	nd	nd
92	2173	2182 (20)	Hexadecana-mide	nd	tr	tr	tr	tr	tr	0.1 ± 0.0	0.1 ± 0.1	0.2 ± 0.1	0.2 ± 0.1	0.1 ± 0.1	0.3 ± 0.4	nd	nd	nd	nd	nd	nd
93	2192		Unknown-16 (AD)	nd	nd	nd	nd	nd	nd	1.0 ± 0.1	0.5 ± 0.4	0.5 ± 0.2	0.6 ± 0.1	0.2 ± 0.1	0.4 ± 0.4	nd	nd	tr	nd	nd	nd
94	2261		Unknown-17 (Al)	tr	tr	tr	nd	tr	tr	0.5 ± 0.3	0.5 ± 0.6	0.5 ± 0.7	0.4 ± 0.4	0.2 ± 0.2	tr	nd	0.8 ± 0.0	0.4 ± 0.0	0.8 ± 0.0	1.3 ± 0.0	0.8 ± 0.0
95	2297	2300*	Tricosane	tr	tr	tr	tr	tr	tr	0.5 ± 0.1	0.5 ± 0.3	0.1 ± 0.1	0.2 ± 0.2	0.1 ± 0.2	tr	tr	0.1 ± 0.0	tr	1.0 ± 0.0	0.5 ± 0.8	0.1 ± 0.1
96	2351	2375 (21)	Oleamide	nd	nd	nd	nd	nd	nd	0.5 ± 0.1	0.8 ± 01	0.6 ± 0.0	0.4 ± 0.1	tr	nd	0.7 ± 0.3	0.4 ± 0.4	0.3 ± 0.1	0.8 ± 0.1	0.7 ± 0.6	0.8 ± 0.2
97	2497	2500*	Pentacosane	tr	tr	tr	tr	tr	tr	0.2 ± 0.1	0.2 ± 0.3	nd	tr	tr	tr	nd	tr	nd	0.4 ± 0.0	nd	nd
			AM (17)	5.9 ± 0.1	5.8 ± 0.8	9.0 ± 1.9	6.9 ± 0.4	4.9 ± 0.4	4.7 ± 0.3	2.3 ± 0.4	3.1 ± 1.1	4.7 ± 1.4	12.7 ± 1.5	2.4 ± 0.3	4.0 ± 1.7	3.2 ± 1.3	4.0 ± 0.4	4.6 ± 0.5	3.9 ± 0.5	4.0 ± 2.2	4.5 ± 1.7
			HS (8)	1.4 ± 0.0	1.3 ± 0.2	0.9 ± 0.1	1.0 ± 0.1	1.1 ± 0.1	1.5 ± 0.1	1.0 ± 0.2	0.6 ± 0.3	1.8 ± 0.1	1.2 ± 0.1	2.7 ± 0.4	1.4 ± 0.1	1.2 ± 0.3	2.1 ± 0.1	2.5 ± 0.3	3.1 ± 0.5	1.4 ± 0.7	3.5 ± 0.9
			Ester (6)	nd	nd	nd	nd	nd	nd	1.6 ± 0.3	1.4 ± 0.5	0.8 ± 0.3	0.9 ± 0.2	0.5 ± 0.2	0.8 ± 0.7	1.1 ± 0.3	0.5 ± 0.3	0.5 ± 0.2	0.3 ± 0.2	0.5 ± 0.5	0.5 ± 0.2
			Aldehyde & ketone (7)	0.1 ± 0.0	0.3 ± 0.0	0.4 ± 0.0	0.3 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	3.0 ± 0.2	2.0 ± 1.1	1.7 ± 0.3	3.2 ± 0.3	1.5 ± 0.0	2.1 ± 0.4	0.9 ± 0.7	0.5 ± 0.3	0.8 ± 0.2	0.4 ± 0.5	0.8 ± 0.6	0.4 ± 0.5
			Phenol (5)	1.9 ± 0.0	0.7 ± 0.1	2.3 ± 0.1	1.9 ± 0.2	1.8 ± 0.2	1.6 ± 0.1	2.4 ± 0.4	2.9 ± 0.5	5.0 ± 0.8	7.1 ± 0.5	4.6 ± 0.7	5.5 ± 1.6	3.9 ± 2.7	4.7 ± 1.3	5.1 ± 0.7	9.3 ± 4.8	3.7 ± 2.8	2.9 ± 0.8
			Unknown (17)	0.2 ± 0.0	0.2 ± 0.0	0.6 ± 0.0	0.4 ± 0.0	0.4 ± 0.0	0.4 ± 0.0	7.7 ± 0.4	6.9 ± 0.6	10.4 ± 2.6	7.0 ± 0.7	3.2 ± 0.3	4.1 ± 1.4	6.9 ± 0.7	5.1 ± 1.0	11.2 ± 1.0	12.3 ± 2.4	8.0 ± 4.0	5.7 ± 1.5

Table 2: Chemical constituents by area percentage ± standard deviation (SD) (%) in peels' oils from K, D, and Z.

Note: Components are categorized as unknown if their exact chemical structure couldn't be elucidated by MS, but sometimes its structure type could be.

Abbreviations AM, AS, AD, OM, OS, OD, IT, and Al refer to alcohol of monoterpene, alcohol of sesquiterpene, alcohol of diterpene, oxygenated monoterpene but not AM, oxygenated sesquiterpene but not AS, oxygenated diterpene but not AD, isomer of thymol, and alkane, respectively. The numbers in brackets denote the sum of components belonging to such class. LRI^a means the value referred to the DB-5 column used in this experiment, LRI^b means the value of apolar or semi-polar column were provided by NIST 08 database or reported in previous literatures. Symbol * represents the LRI is referred to NIST 08 database, the number in bracket after the value of LRI^b represents corresponding reference showing the value. tr = trace, which is recorded when the value is less than 0.05, nd = not detected. The same for following tables.

No.	Component	Concentration ± SD (μg/μL)																	
		Kc1	Kc2	Kc3	Kc4	Kc5	Kc6	Dc1	Dc2	Dc3	Dc4	Dc5	Dc6	Zc1	Zc2	Zc3	Zc4	Zc5	Zc6
1	α-Thujene	4.4 ± 0.0	2.1 ± 0.0	2.6 ± 0.5	1.9 ± 0.0	1.7 ± 0.0	1.8 ± 0.0	tr	nd	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	tr	0.1 ± 0.1	0.1 ± 0.1	0.1 ± 0.1	tr	0.2 ± 0.2
2	**α**-Pinene	16.1 ± 0.1	12.6 ± 0.2	11.7 ± 0.8	9.8 ± 0.2	9.0 ± 0.2	9.7 ± 0.2	0.7 ± 0.1	0.2 ± 0.2	0.7 ± 0.4	0.9 ± 0.1	1.2 ± 0.0	1.1 ± 0.4	0.6 ± 0.3	0.8 ± 0.2	0.9 ± 0.4	0.6 ± 0.4	0.7 ± 0.4	0.6 ± 0.3
3	Sabinene	1.4 ± 0.1	0.5 ± 0.0	2.5 ± 1.5	1.3 ± 0.0	1.1 ± 0.2	1.4 ± 0.0	nd	nd	0.1 ± 0.1	nd	tr	0.1 ± 0.0	nd	nd	nd	nd	nd	nd
4	**β**-Pinene	47.6 ± 0.3	17.2 ± 0.4	21.4 ± 1.5	15.8 ± 0.2	14.9 ± 0.3	14.2 ± 0.3	0.1 ± 0.0	nd	0.3 ± 0.2	0.4 ± 0.0	0.4 ± 0.0	1.7 ± 1.6	0.1 ± 0.1	0.3 ± 0.1	0.2 ± 0.1	tr	0.2 ± 0.3	0.2 ± 0.1
5	**β**-Myrcene	10.4 ± 0.1	13.1 ± 0.2	9.8 ± 0.7	11.0 ± 0.1	10.9 ± 0.2	11.7 ± 0.3	1.2 ± 0.0	0.5 ± 0.5	0.8 ± 0.5	1.6 ± 0.1	1.5 ± 0.0	1.5 ± 0.4	0.5 ± 0.3	0.8 ± 0.1	0.7 ± 0.3	0.1 ± 0.0	1.6 ± 2.1	0.9 ± 0.5
6	**α**-Phellandrene	0.7 ± 0.0	1.3 ± 0.0	0.7 ± 0.1	0.5 ± 0.0	0.4 ± 0.0	0.4 ± 0.0	0.1 ± 0.0	nd	0.1 ± 0.0	0.3 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.1	0.1 ± 0.0	0.4 ± 0.0	tr	0.1 ± 0.1	0.1 ± 0.0
7	**α**-Terpinene	2.5 ± 0.3	1.6 ± 0.0	1.3 ± 0.1	0.9 ± 0.0	1.0 ± 0.0	1.0 ± 0.0	0.1 ± 0.0	nd	0.1 ± 0.1	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.1	nd	nd	nd	nd	nd	nd
8	*p*-Cymene.	169.4 ± 6.0	80.0 ± 3.7	84.1 ± 3.8	75.9 ± 3.6	53.1 ± 4.9	47.6 ± 3.8	1.5 ± 0.0	0.7 ± 0.8	3.3 ± 3.0	1.8 ± 0.1	0.8 ± 0.0	1.1 ± 0.2	1.0 ± 0.2	1.3 ± 0.1	1.9 ± 0.2	0.3 ± 0.0	0.5 ± 0.2	0.9 ± 0.1
9	Limonene	337.5 ± 11.9	442.9 ± 20.5	403.7 ± 18.4	453.3 ± 21.4	461.5 ± 42.3	463.9 ± 37.5	37.4 ± 0.3	18.5 ± 19.5	31.1 ± 22.4	37.7 ± 0.9	77.9 ± 1.1	73.4 ± 22.4	19.8 ± 10.5	35.4 ± 3.5	17.5 ± 1.9	2.8 ± 0.3	121.7 ± 166.8	53.0 ± 40.0
10	**β**-*trans*-Ocimene	4.4 ± 0.0	2.3 ± 0.1	0.9 ± 0.1	0.8 ± 0.0	1.0 ± 0.0	1.0 ± 0.0	0.1 ± 0.0	nd	0.1 ± 0.1	tr	0.1 ± 0.0	0.1 ± 0.0	tr	0.1 ± 0.0	tr	tr	tr	tr
11	**γ**-Terpinene	53.8 ± 0.7	54.7 ± 1.4	30.5 ± 0.4	34.8 ± 0.4	32.5 ± 0.6	34.4 ± 0.8	2.0 ± 0.0	1.3 ± 0.8	1.4 ± 0.8	1.6 ± 0.0	2.8 ± 0.0	2.3 ± 0.7	1.1 ± 0.5	1.9 ± 0.2	2.0 ± 0.3	0.5 ± 0.1	0.6 ± 0.3	2.3 ± 0.2
12	*p*-Cymenene	0.9 ± 0.0	1.3 ± 0.0	0.4 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	nd	nd	nd	nd	nd	nd	0.2 ± 0.1	0.3 ± 0.2	1.3 ± 0.2	0.4 ± 0.2	0.4 ± 0.3	0.5 ± 0.2
13	Terpinolene	4.7 ± 0.0	3.4 ± 0.1	2.1 ± 0.1	2.0 ± 0.0	2.0 ± 0.0	2.0 ± 0.0	0.6 ± 0.0	0.3 ± 0.2	0.4 ± 0.1	0.8 ± 0.0	0.6 ± 0.0	0.5 ± 0.1	nd	0.1 ± 0.1	nd	nd	nd	0.1 ± 0.1
14	Linalool	20.3 ± 1.4	15.0 ± 7.9	29.9 ± 11.9	24.6 ± 4.0	15.8 ± 1.2	14.7 ± 0.7	0.4 ± 0.1	0.4 ± 0.5	1.1 ± 0.4	1.0 ± 0.0	0.3 ± 0.0	0.5 ± 0.1	0.6 ± 0.3	0.6 ± 0.1	1.1 ± 0.2	0.4 ± 0.1	4.9 ± 6.7	3.3 ± 4.1
15	Hotrienol	0.2 ± 0.0	0.3 ± 0.0	0.8 ± 0.0	0.6 ± 0.0	0.5 ± 0.0	0.5 ± 0.0	0.2 ± 0.0	0.2 ± 0.2	1.2 ± 0.6	4.4 ± 0.1	0.3 ± 0.0	0.7 ± 0.2	0.2 ± 0.1	0.3 ± 0.0	1.4 ± 0.2	0.5 ± 0.1	0.6 ± 0.3	0.5 ± 0.0
16	1,3,8-*p*-Menthatriene	0.2 ± 0.0	0.2 ± 0.0	0.4 ± 0.0	0.3 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	nd	nd	0.1 ± 0.1	0.3 ± 0.0	0.1 ± 0.0	0.2 ± 0.0	tr	0.1 ± 0.1	0.3 ± 0.2	nd	tr	0.1 ± 0.1
17	*trans*-*p*-Mentha-2,8-dienol	0.2 ± 0.0	0.1 ± 0.0	1.3 ± 0.0	0.6 ± 0.0	0.5 ± 0.0	0.5 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.3 ± 0.1	0.7 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	tr	0.3 ± 0.2	0.1 ± 0.0	0.2 ± 0.1	0.2 ± 0.1
18	*cis*-*p*-Mentha-2,8-dien-1-ol	0.1 ± 0.0	0.1 ± 0.0	1.0 ± 0.1	0.4 ± 0.0	0.2 ± 0.0	0.3 ± 0.0	nd	nd	0.2 ± 0.1	0.6 ± 0.1	tr	0.1 ± 0.0	tr	0.1 ± 0.0	0.1 ± 0.1	tr	0.1 ± 0.1	tr
19	2-Cyclohexen-1-ol, 1-methyl-4-(1-methylethyl)-, *trans*-	0.4 ± 0.0	0.2 ± 0.0	0.5 ± 0.0	0.3 ± 0.0	0.3 ± 0.0	0.3 ± 0.0	nd	nd	nd	0.2 ± 0.0	0.1 ± 0.0	0.2 ± 0.1	tr	0.1 ± 0.1	0.4 ± 0.0	tr	tr	0.1 ± 0.0
20	**β**-Terpineol	0.1 ± 0.0	0.7 ± 0.1	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	0.2 ± 0.0	nd	nd	nd	0.1 ± 0.0	nd	nd	nd	nd	nd	nd
21	Borneol	0.4 ± 0.0	0.5 ± 0.1	0.3 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	tr	tr	0.1 ± 0.0	tr	0.1 ± 0.1	nd	nd	nd	nd	nd	nd

22	Unknown-1 (OM)	0.5 ± 0.0	0.3 ± 0.0	0.6 ± 0.0	0.4 ± 0.0	0.2 ± 0.0	0.3 ± 0.0	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
23	Terpinen-4-ol	6.5 ± 0.0	4.2 ± 0.3	4.0 ± 0.1	3.4 ± 0.2	3.1 ± 0.1	2.6 ± 0.1	0.1 ± 0.0	0.2 ± 0.1	0.2 ± 0.1	0.4 ± 0.0	0.2 ± 0.0	0.4 ± 0.1	0.2 ± 0.1	0.3 ± 0.0	0.6 ± 0.1	0.2 ± 0.1	0.3 ± 0.2	0.3 ± 0.1
24	*p*-Cymen-8-ol	0.7 ± 0.0	0.5 ± 0.0	0.7 ± 0.0	0.4 ± 0.0	0.3 ± 0.0	0.3 ± 0.0	0.3 ± 0.0	0.1 ± 0.1	0.2 ± 0.0	0.5 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	nd	nd	nd	nd	nd	nd
25	Cyclohexanol, 2-methylene-5-(1-methylethenyl)-	0.1 ± 0.0	0.5 ± 0.0	1.8 ± 0.1	0.8 ± 0.1	0.5 ± 0.0	0.7 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.5 ± 0.2	1.4 ± 0.0	0.2 ± 0.0	0.4 ± 0.1	0.1 ± 0.0	0.1 ± 0.0	0.5 ± 0.1	0.1 ± 0.1	0.2 ± 0.1	0.1 ± 0.0
26	α-Terpineol	5.4 ± 0.0	10.0 ± 0.8	5.7 ± 0.3	4.8 ± 0.3	4.0 ± 0.1	3.6 ± 0.1	0.8 ± 0.0	1.3 ± 0.4	0.5 ± 0.0	0.7 ± 0.0	0.3 ± 0.1	0.8 ± 0.2	0.4 ± 0.1	0.5 ± 0.0	1.3 ± 0.2	0.5 ± 0.1	0.4 ± 0.2	0.3 ± 0.0
27	Dihydrocarvone	0.3 ± 0.0	0.3 ± 0.0	0.6 ± 0.0	0.3 ± 0.0	0.3 ± 0.0	0.3 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.1 ± 0.1	0.5 ± 0.0	tr	0.2 ± 0.1	nd	nd	nd	nd	nd	nd
28	*trans*-Isopiperitenol	0.2 ± 0.0	0.2 ± 0.0	1.2 ± 0.1	0.5 ± 0.0	0.4 ± 0.0	0.5 ± 0.0	tr	nd	0.3 ± 0.1	0.9 ± 0.0	0.1 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.3 ± 0.1	tr	0.2 ± 0.2	0.1 ± 0.1
29	Decanal	0.1 ± 0.0	tr	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.0	nd	0.4 ± 0.2	0.9 ± 0.0	0.7 ± 0.0	0.9 ± 0.2	tr	0.1 ± 0.0	0.3 ± 0.2	tr	tr	0.1 ± 0.1
30	*trans*-Carveol	0.6 ± 0.0	0.7 ± 0.1	2.1 ± 0.1	0.9 ± 0.1	0.7 ± 0.0	0.8 ± 0.0	0.3 ± 0.0	0.4 ± 0.3	0.9 ± 0.1	1.9 ± 0.1	0.6 ± 0.0	1.0 ± 0.2	0.4 ± 0.1	0.6 ± 0.0	1.7 ± 0.3	0.6 ± 0.1	1.0 ± 0.4	0.9 ± 0.1
31	Unknown-2 (AM)	0.3 ± 0.0	0.8 ± 0.1	2.1 ± 0.1	1.3 ± 0.1	1.0 ± 0.0	1.1 ± 0.0	0.7 ± 0.0	0.7 ± 0.2	2.0 ± 0.7	3.3 ± 0.0	0.8 ± 0.0	0.8 ± 0.2	nd	nd	nd	nd	nd	nd
32	β-Citronellol	nd	nd	nd	nd	nd	nd	0.1 ± 0.1	0.1 ± 0.1	0.2 ± 0.0	0.3 ± 0.0	0.1 ± 0.0	0.8 ± 0.2	nd	nd	nd	nd	nd	nd
33	*cis*-Carveol	0.3 ± 0.0	0.4 ± 0.0	0.7 ± 0.0	0.3 ± 0.0	0.3 ± 0.0	0.3 ± 0.0	nd	nd	nd	nd	nd	nd	0.1 ± 0.1	0.1 ± 0.1	0.2 ± 0.2	0.1 ± 0.1	0.1 ± 0.1	0.1 ± 0.1
34	Thymol methyl ether	0.8 ± 0.0	0.3 ± 0.0	0.5 ± 0.0	0.3 ± 0.0	0.3 ± 0.0	0.3 ± 0.0	0.3 ± 0.0	tr	0.5 ± 0.3	0.4 ± 0.0	0.5 ± 0.0	0.3 ± 0.1	tr	tr	0.3 ± 0.3	tr	0.1 ± 0.2	0.3 ± 0.1
35	Carvone	0.3 ± 0.0	0.8 ± 0.1	1.0 ± 0.1	0.5 ± 0.0	0.3 ± 0.0	0.4 ± 0.0	0.1 ± 0.0	nd	0.1 ± 0.0	0.2 ± 0.0	nd	0.1 ± 0.0	nd	nd	nd	nd	nd	nd
36	*trans*-Geraniol	0.5 ± 0.0	0.5 ± 0.1	0.3 ± 0.0	0.3 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.1	0.2 ± 0.1	0.2 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.1	0.2 ± 0.0	0.8 ± 0.1	0.1 ± 0.0	0.1 ± 0.1	0.1 ± 0.0
37	Perillaldehyde	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.1	0.1 ± 0.1	0.1 ± 0.1	0.1 ± 0.1	0.1 ± 0.0	nd	0.2 ± 0.1	0.5 ± 0.0	nd	0.1 ± 0.0	nd	nd	nd	nd	nd	nd
38	Unknown-3 (IT)	0.5 ± 0.0	0.3 ± 0.0	0.8 ± 0.1	0.9 ± 0.1	0.7 ± 0.0	0.6 ± 0.0	0.2 ± 0.0	0.8 ± 0.3	0.6 ± 0.1	0.8 ± 0.0	0.4 ± 0.0	1.2 ± 0.1	0.4 ± 0.1	0.9 ± 0.1	0.7 ± 0.1	0.7 ± 0.1	0.5 ± 0.3	1.5 ± 0.1
39	Thymol	17.4 ± 0.4	5.8 ± 0.6	17.0 ± 1.4	12.8 ± 1.3	12.4 ± 0.4	11.3 ± 0.3	4.0 ± 0.3	3.7 ± 0.2	9.2 ± 3.5	9.2 ± 0.0	7.1 ± 0.5	10.5 ± 1.1	3.1 ± 2.6	3.1 ± 1.3	10.2 ± 1.4	7.2 ± 5.5	4.1 ± 1.5	2.8 ± 0.2
40	Unknown-4 (IT)	0.4 ± 0.0	0.2 ± 0.0	0.5 ± 0.1	0.6 ± 0.0	0.5 ± 0.1	0.4 ± 0.0	0.2 ± 0.0	0.4 ± 0.1	0.4 ± 0.1	0.3 ± 0.0	0.2 ± 0.0	0.2 ± 0.1	0.1 ± 0.1	0.5 ± 0.2	1.1 ± 0.3	0.3 ± 0.2	0.4 ± 0.2	0.1 ± 0.1
41	Carvacrol	1.2 ± 0.1	0.6 ± 0.1	2.0 ± 0.2	2.4 ± 0.2	1.7 ± 0.0	1.5 ± 0.0	0.6 ± 0.1	0.5 ± 0.1	1.0 ± 0.3	1.2 ± 0.3	0.5 ± 0.0	0.9 ± 0.1	0.8 ± 0.8	0.7 ± 0.6	1.7 ± 0.3	2.1 ± 1.9	0.8 ± 0.4	0.2 ± 0.2
42	2-Methoxy-4-vinyl-phenol	0.2 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.1	0.2 ± 0.0	0.1 ± 0.0	0.6 ± 0.1	0.6 ± 0.3	0.9 ± 0.1	1.9 ± 0.1	1.1 ± 0.3	0.8 ± 0.0	0.4 ± 0.2	0.7 ± 0.1	1.6 ± 0.6	0.7 ± 0.2	0.9 ± 0.4	0.4 ± 0.1
43	δ-Elemene	0.4 ± 0.0	0.4 ± 0.0	0.3 ± 0.0	0.4 ± 0.1	0.4 ± 0.0	0.5 ± 0.0	0.1 ± 0.0	nd	0.1 ± 0.1	0.1 ± 0.0	0.3 ± 0.0	0.2 ± 0.0	tr	tr	tr	tr	nd	0.2 ± 0.0
44	*n*-Decanoic acid	tr	tr	tr	tr	tr	tr	0.4 ± 0.2	0.3 ± 0	tr	tr	0.1 ± 0.0	0.1 ± 0.0	nd	tr	tr	tr	nd	tr

45	β-Elemene	0.6 ± 0.0	0.3 ± 0.0	0.4 ± 0.1	0.4 ± 0.1	0.4 ± 0.0	0.5 ± 0.0	0.1 ± 0.0	nd	0.2 ± 0.1	0.1 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.0	0.3 ± 0.1	0.1 ± 0.1	tr	0.6 ± 0.1
46	γ-Elemene	0.2 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.3 ± 0.1	0.3 ± 0.3	0.7 ± 0.2	0.3 ± 0.0	1.0 ± 0.1	0.7 ± 0.2	0.2 ± 0.0	0.4 ± 0.1	0.9 ± 0.2	0.4 ± 0.1	0.4 ± 0.1	1.0 ± 0.0
47	Germacrene D	0.7 ± 0.0	0.4 ± 0.0	0.5 ± 0.1	0.5 ± 0.1	0.5 ± 0.0	0.7 ± 0.0	0.2 ± 0.0	0.1 ± 0.1	0.4 ± 0.1	0.1 ± 0.1	0.3 ± 0.1	0.3 ± 0.1	tr	0.1 ± 0.1	0.3 ± 0.1	0.1 ± 0.1	tr	0.2 ± 0.2
48	**α**-Selinene	0.5 ± 0.0	0.5 ± 0.0	0.3 ± 0.1	0.3 ± 0.0	0.4 ± 0.0	0.5 ± 0.0	0.4 ± 0.1	0.1 ± 0.0	0.4 ± 0.2	0.2 ± 0.0	0.6 ± 0.0	0.3 ± 0.1	nd	nd	nd	nd	nd	nd
49	(*E*, *E*)-**α**-Farnesene	6.8 ± 0.3	6.7 ± 0.7	4.7 ± 1.0	4.9 ± 0.8	5.0 ± 0.3	7.1 ± 0.3	0.2 ± 0.0	nd	0.3 ± 0.1	0.2 ± 0.0	0.4 ± 0.0	0.2 ± 0.0	0.2 ± 0.2	0.2 ± 0.0	0.7 ± 0.4	0.2 ± 0.2	0.2 ± 0.2	0.4 ± 0.1
50	**δ**-Cadinene	0.5 ± 0.0	0.3 ± 0.0	0.2 ± 0.1	0.3 ± 0.0	0.3 ± 0.0	0.3 ± 0.0	0.6 ± 0.1	0.4 ± 0.1	1.1 ± 0.2	0.5 ± 0.0	1.2 ± 0.1	0.9 ± 0.3	0.3 ± 0.0	0.6 ± 0.1	1.8 ± 0.4	1.0 ± 0.2	0.7 ± 0.2	1.5 ± 0.1
51	Elemol	tr	tr	tr	tr	tr	tr	0.5 ± 0.1	0.4 ± 0.1	0.9 ± 0.3	0.3 ± 0.0	0.5 ± 0.0	0.8 ± 0.3	0.2 ± 0.0	0.3 ± 0.0	1.2 ± 0.3	0.2 ± 0.2	0.4 ± 0.0	0.7 ± 0.0
52	Germacrene B	0.1 ± 0.0	nd	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	tr	tr	tr	tr	tr	tr	0.3 ± 0.1	0.4 ± 0.2	1.8 ± 0.3	0.7 ± 0.2	0.7 ± 0.2	0.8 ± 0.4
53	Dodecanoic acid	tr	tr	tr	tr	tr	tr	1.1 ± 0.3	0.9 ± 0.5	2.5 ± 1.1	1.1 ± 0.5	1.0 ± 0.3	3.4 ± 2.5	tr	tr	0.3 ± 0.1	tr	tr	tr
54	Spathulenol	2.7 ± 0.1	0.6 ± 0.1	2.5 ± 0.6	1.6 ± 0.2	1.4 ± 0.1	1.5 ± 0.1	2.0 ± 0.1	1.6 ± 0.4	3.7 ± 1.0	1.9 ± 0.2	1.5 ± 0.0	2.6 ± 1.1	1.2 ± 0.2	1.1 ± 0.1	5.6 ± 1.3	1.7 ± 0.5	1.9 ± 0.3	1.8 ± 0.2
55	Globulol	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.7 ± 0.0	0.6 ± 0.1	0.7 ± 0.2	0.3 ± 0.0	0.4 ± 0.0	0.6 ± 0.3	0.2 ± 0.0	0.2 ± 0.0	1.2 ± 0.4	0.3 ± 0.3	0.4 ± 0.1	0.4 ± 0.1
56	Unknown-5 (AS)	tr	tr	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.7 ± 0.1	0.6 ± 0.1	0.4 ± 0.0	0.4 ± 0.2	0.6 ± 0.1	0.8 ± 0.6	nd	nd	nd	nd	nd	nd
57	Unknown-6 (AS)	0.2 ± 0.0	tr	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	1.3 ± 0.6	0.6 ± 0.2	1.6 ± 0.6	0.5 ± 0.2	0.5 ± 0.0	0.8 ± 0.5	1.0 ± 0.1	0.7 ± 0.0	4.7 ± 1.4	1.7 ± 0.6	1.6 ± 0.4	1.2 ± 0.2
58	Selina-6-en-4-ol	tr	tr	tr	tr	tr	tr	0.6 ± 0.1	0.4 ± 0.1	0.3 ± 0.1	0.2 ± 0.0	0.4 ± 0.1	0.4 ± 0.1	0.2 ± 0.1	0.1 ± 0.0	1.1 ± 0.7	0.2 ± 0.3	0.3 ± 0.2	0.1 ± 0.1
59	*.tau.*-Cadinol	tr	tr	nd	tr	tr	tr	0.4 ± 0.1	0.2 ± 0.1	0.6 ± 0.4	0.2 ± 0.0	0.3 ± 0.1	0.6 ± 0.3	0.1 ± 0.1	tr	0.4 ± 0.4	0.1 ± 0.2	0.1 ± 0.1	0.1 ± 0.1
60	Isospathulenol	1.1 ± 0.1	0.3 ± 0.1	0.9 ± 0.3	1.4 ± 0.2	1.2 ± 0.1	1.3 ± 0.1	7.4 ± 0.6	6.1 ± 1.1	13.3 ± 3.7	5.1 ± 0.4	4.2 ± 0.3	7.5 ± 4.2	5.8 ± 0.8	3.7 ± 0.4	21.9 ± 5.0	7.1 ± 2.5	8.0 ± 1.4	7.5 ± 0.8
61	γ-Eudesmol	tr	tr	tr	0.1 ± 0.2	0.1 ± 0.0	0.1 ± 0.0	1.2 ± 0.1	1.2 ± 0.3	1.3 ± 0.8	0.6 ± 0.0	1.0 ± 0.1	1.3 ± 0.8	0.5 ± 0.1	0.4 ± 0.0	1.8 ± 0.5	0.7 ± 0.3	0.7 ± 0.2	1.2 ± 0.2
62	*.tau.*-Muurolol	0.2 ± 0.0	tr	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	1.0 ± 0.1	0.8 ± 0.1	1.1 ± 0.4	0.5 ± 0.0	0.8 ± 0.0	1.2 ± 0.8	nd	nd	nd	nd	nd	nd
63	Torreyol	tr	tr	tr	tr	tr	tr	1.2 ± 0.1	1.0 ± 0.1	1.6 ± 0.6	0.6 ± 0.0	0.4 ± 0.0	0.9 ± 0.4	0.7 ± 0.1	0.5 ± 0.0	3.1 ± 0.8	1.1 ± 0.4	1.1 ± 0.2	0.9 ± 0.1
64	**β**-Eudesmol	0.1 ± 0.0	tr	tr	0.3 ± 0.1	0.2 ± 0.0	0.3 ± 0.1	2.9 ± 0.2	2.4 ± 0.2	4.5 ± 1.4	1.4 ± 0.1	1.8 ± 0.2	3.1 ± 1.9	1.9 ± 0.3	1.2 ± 0.1	7.9 ± 1.8	2.5 ± 1.0	2.7 ± 0.6	3.4 ± 0.4
65	**α**-Cadinol	0.4 ± 0.0	0.2 ± 0.0	0.4 ± 0.1	0.7 ± 0.2	0.4 ± 0.0	0.6 ± 0.2	3.5 ± 0.4	2.7 ± 0.4	4.2 ± 1.3	1.4 ± 0.2	1.9 ± 0.1	3.1 ± 1.9	1.8 ± 0.3	1.1 ± 0.1	6.7 ± 1.6	2.2 ± 0.9	2.9 ± 0.6	2.8 ± 0.3
66	Unknown-7 (AS)	0.2 ± 0.0	tr	nd	nd	nd	nd	0.6 ± 0.1	0.5 ± 0.0	0.9 ± 0.5	0.3 ± 0.1	0.2 ± 0.0	0.6 ± 0.4	nd	nd	nd	nd	nd	nd
67	Unknown-8 (AS)	tr	tr	tr	tr	tr	tr	0.5 ± 0.0	0.4 ± 0.0	0.8 ± 0.4	0.3 ± 0.0	0.1 ± 0.0	0.3 ± 0.1	0.6 ± 0.2	0.4 ± 0.0	2.5 ± 0.9	1.0 ± 0.4	0.9 ± 0.3	0.7 ± 0.1

68	Unknown-9 (AS)	nd	nd	nd	nd	nd	nd	2.2 ± 0.2	1.9 ± 0.3	3.5 ± 1.7	1.2 ± 0.2	0.4 ± 0.1	1.1 ± 0.8	1.8 ± 0.1	0.8 ± 0.1	7.6 ± 2.0	2.6 ± 1.1	3.0 ± 0.7	1.3 ± 0.2
69	Juniper camphor	tr	tr	tr	tr	tr	tr	0.5 ± 0.1	0.6 ± 0.2	0.5 ± 0.3	0.2 ± 0.0	0.2 ± 0.1	0.3 ± 0.2	0.2 ± 0.0	0.1 ± 0.0	0.5 ± 0.2	0.2 ± 0.2	0.2 ± 0.2	0.4 ± 0.1
70	Unknown-10 (AS)	nd	nd	nd	nd	nd	nd	1.2 ± 0.3	1.1 ± 0.0	1.3 ± 0.3	0.6 ± 0.2	0.3 ± 0.0	0.6 ± 0.5	nd	nd	nd	nd	nd	nd
71	Unknown-11 (AS)	tr	tr	tr	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.9 ± 0.0	0.7 ± 0.0	1.6 ± 0.7	0.6 ± 0.2	0.3 ± 0.0	0.7 ± 0.4	0.8 ± 0.1	0.4 ± 0.1	2.5 ± 0.8	1.0 ± 0.4	1.0 ± 0.4	1.0 ± 0.3
72	6-Isopropenyl-4, 8a-dimethyl-1, 2, 3, 5, 6, 7, 8, 8a-octahydro-naphthalen-2-ol	0.2 ± 0.0	tr	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	nd	0.7 ± 0.1	0.7 ± 0.0	1.4 ± 0.7	0.5 ± 0.1	0.2 ± 0.1	0.5 ± 0.4	1.1 ± 0.4	0.5 ± 0.1	3.8 ± 1.8	1.2 ± 0.8	1.6 ± 0.8	1.0 ± 0.2
73	Unknown-12 (AS)	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	tr	tr	tr	tr	tr	tr	0.7 ± 0.1	0.3 ± 0.0	2.6 ± 0.7	1.0 ± 0.4	1.0 ± 0.3	0.5 ± 0.0
74	**α**-Sinensal	tr	tr	tr	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	3.0 ± 0.3	1.9 ± 1.2	1.2 ± 0.6	1.3 ± 0.1	0.9 ± 0.1	2.1 ± 1.7	0.7 ± 0.5	0.3 ± 0.3	1.1 ± 0.4	0.2 ± 0.3	0.6 ± 0.6	0.3 ± 0.4
75	7R, 8R-8-Hydroxy-4-isopropylidene-7-methylbicyclo[5. 3. 1]undec-1-ene	tr	tr	0.2 ± 0.0	0.3 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.5 ± 0.1	0.8 ± 0.3	1.0 ± 0.4	0.8 ± 0.3	0.9 ± 0.7	0.5 ± 0.3	0.5 ± 0.1	0.3 ± 0.1	1.6 ± 0.3	0.8 ± 0.1	0.8 ± 0.1	0.5 ± 0.1
76	2-Pentadecanone, 6, 10, 14-trimethyl-	tr	tr	tr	tr	tr	tr	0.8 ± 0.2	0.7 ± 0.4	0.6 ± 0.4	0.6 ± 0.1	0.2 ± 0.1	0.4 ± 0.5	0.1 ± 0.1	tr	0.1 ± 0.1	0.1 ± 0.1	tr	0.1 ± 0.1
77	Pentadecanoic acid	nd	tr	tr	tr	tr	tr	1.1 ± 0.5	1.3 ± 0.9	tr	0.2 ± 0.1	0.1 ± 0.1	0.2 ± 0.2	nd	nd	nd	nd	nd	nd
78	Hexadecanoic acid, methyl ester	nd	nd	nd	nd	nd	nd	0.7 ± 0.2	0.7 ± 0.2	0.5 ± 0.4	0.3 ± 0.1	0.1 ± 0.0	0.4 ± 0.3	0.2 ± 0.1	0.1 ± 0.1	0.2 ± 0.2	0.1 ± 0.1	0.2 ± 0.2	0.3 ± 0.0
79	Isophytol	nd	nd	nd	nd	nd	nd	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.1	0.1 ± 0.0	tr	tr	nd	nd	nd	nd	nd	nd
80	Unknown-13 (OS)	tr	tr	nd	tr	tr	tr	nd	nd	0.8 ± 0.6	tr	tr	nd	nd	nd	nd	nd	nd	nd
81	*n*-Hexadecanoic acid	0.1 ± 0.1	0.1 ± 0.1	0.3 ± 0.2	0.7 ± 0.3	0.9 ± 0.2	0.6 ± 0.2	31.2 ± 9.9	37.2 ± 19.0	13.1 ± 8.1	11.7 ± 4.3	7.9 ± 6.5	18.4 ± 21.7	10.5 ± 7.2	5.6 ± 2.8	17.0 ± 8.3	4.5 ± 1.4	9.8 ± 9.3	2.3 ± 2.0
82	Hexadecanoic acid, ethyl ester	tr	tr	tr	tr	tr	0.2 ± 0.0	0.3 ± 0.0	0.3 ± 0.1	0.2 ± 0.1	0.2 ± 0.1	0.2 ± 0.1	0.4 ± 0.4	nd	tr	nd	nd	nd	0.1 ± 0.1
83	Linoleic acid, methyl ester	0.2 ± 0.0	tr	tr	0.2 ± 0.0	tr	0.2 ± 0.0	0.4 ± 0.2	0.3 ± 0.2	0.2 ± 0.2	0.1 ± 0.1	0.1 ± 0.1	0.4 ± 0.4	0.1 ± 0.1	tr	0.2 ± 0.2	tr	tr	0.1 ± 0.1
84	Linolenic acid, methyl ester	0.2 ± 0.0	tr	tr	0.2 ± 0.0	tr	0.2 ± 0.0	0.5 ± 0.2	0.4 ± 0.2	0.3 ± 0.2	0.2 ± 0.1	0.1 ± 0.1	0.3 ± 0.3	0.2 ± 0.2	tr	0.1 ± 0.2	tr	0.1 ± 0.2	tr
85	Phytol	nd	nd	nd	nd	nd	nd	0.4 ± 0.3	0.3 ± 0.1	0.9 ± 0.3	0.3 ± 0.1	0.3 ± 0.2	0.3 ± 0.4	0.3 ± 0.2	0.1 ± 0.1	0.5 ± 0.5	0.2 ± 0.1	0.5 ± 0.4	tr
86	Octadecanoic acid, methyl ester	nd	nd	nd	nd	nd	tr	0.2 ± 0.1	0.2 ± 0.0	0.2 ± 0.1	0.1 ± 0.2	tr	0.2 ± 0.2	0.3 ± 0.3	0.2 ± 0.3	0.4 ± 0.4	0.1 ± 0.1	0.1 ± 0.1	0.1 ± 0.0
87	Linoleic acid	nd	tr	tr	tr	tr	tr	4.1 ± 2.7	6.9 ± 4.4	0.7 ± 0.3	0.7 ± 0.4	1.0 ± 1.4	2.1 ± 2.9	0.9 ± 0.9	0.4 ± 0.3	0.9 ± 0.9	0.1 ± 0.1	0.2 ± 0.3	0.1 ± 0.0
88	Linolenic acid	tr	nd	tr	nd	nd	tr	2.4 ± 1.2	3.6 ± 2.3	0.1 ± 0.0	0.5 ± 0.3	0.8 ± 1.2	1.4 ± 1.9	0.6 ± 0.5	0.3 ± 0.2	0.6 ± 0.5	0.4 ± 0.4	0.4 ± 0.2	0.3 ± 0.3

89	Unknown-14 (AD)	nd	tr	tr	tr	tr	tr	0.7 ± 0.2	0.4 ± 0.3	0.6 ± 0.6	0.4 ± 0.1	0.1 ± 0.1	0.4 ± 0.4	nd	nd	nd	nd	nd	nd
90	Unknown-15 (OD)	nd	nd	nd	nd	nd	nd	0.2 ± 0.0	0.2 ± 0.1	0.3 ± 0.2	0.5 ± 0.1	0.1 ± 0.0	0.2 ± 0.2	nd	nd	nd	nd	nd	nd
91	Linolenic acid, ethyl ester	tr	tr	tr	tr	nd	tr	0.1 ± 0.0	0.2 ± 0.1	0.1 ± 0.1	nd	tr	0.1 ± 0.1	nd	nd	nd	nd	nd	nd
92	Hexadecanamide	nd	tr	tr	tr	tr	tr	0.4 ± 0.1	0.3 ± 0.3	0.8 ± 0.6	0.6 ± 0.3	0.2 ± 0.3	1.7 ± 2.4	nd	nd	nd	nd	nd	nd
93	Unknown-16 (AD)	nd	nd	nd	nd	nd	nd	1.6 ± 0.4	0.7 ± 0.5	0.8 ± 0.5	0.8 ± 0.2	0.2 ± 0.1	0.8 ± 1.0	nd	nd	tr	nd	nd	nd
94	Unknown-17 (Al)	tr	tr	tr	nd	tr	tr	0.9 ± 0.6	1.0 ± 1.1	1.4 ± 1.9	0.6 ± 0.8	0.4 ± 0.5	tr	nd	0.8 ± 1.2	0.8 ± 1.1	0.8 ± 1.2	1.1 ± 1.6	1.1 ± 1.6
95	Tricosane	0.2 ± 0.0	tr	tr	tr	tr	tr	1.0 ± 0.3	0.9 ± 0.7	0.1 ± 0.2	0.4 ± 0.4	0.3 ± 0.3	0.1 ± 0.1	tr	tr	tr	1.0 ± 1.4	0.4 ± 0.6	0.1 ± 0.2
96	Oleamide	nd	nd	nd	nd	nd	nd	1.9 ± 0.6	2.4 ± 0.1	2.0 ± 0.7	1.0 ± 0.4	0.1 ± 0.2	nd	1.1 ± 0.4	0.8 ± 0.7	1.2 ± 1.1	1.2 ± 0.2	1.8 ± 1.1	2.0 ± 0.4
97	Pentacosane	tr	tr	tr	tr	tr	tr	0.4 ± 0.3	0.5 ± 0.6	tr	tr	tr	tr	nd	tr	nd	0.2 ± 0.3	nd	nd
	AM (17)	36.3 ± 1.5	34.7 ± 6.4	52.6 ± 12.1	39.6 ± 3.0	28.1 ± 1.0	26.7 ± 0.4	3.5 ± 0.1	4.0 ± 1.1	7.7 ± 2.1	16.6 ± 0.2	3.4 ± 0.1	6.5 ± 1.6	2.4 ± 0.9	3.1 ± 0.2	8.8 ± 1.1	2.7 ± 0.4	8.1 ± 7.9	6.0 ± 4.1
	HS (8)	9.8 ± 0.4	8.7 ± 0.8	6.6 ± 1.2	7.1 ± 1.0	7.3 ± 0.4	9.9 ± 0.3	1.7 ± 0.3	0.9 ± 0.3	3.3 ± 0.8	1.5 ± 0.0	3.9 ± 0.3	2.7 ± 0.6	1.1 ± 0.1	2.0 ± 0.3	6.2 ± 1.4	2.7 ± 0.5	2.0 ± 0.5	4.9 ± 0.2
	Ester (6)	0.4 ± 0.1	nd	nd	0.4 ± 0.0	nd	0.6 ± 0.0	2.3 ± 0.8	2.0 ± 0.8	1.5 ± 0.9	1.0 ± 0.3	0.6 ± 0.4	1.6 ± 1.8	0.8 ± 0.2	0.5 ± 0.3	1.4 ± 0.4	0.4 ± 0.2	0.4 ± 0.5	0.7 ± 0.3
	Aldehyde & ketone (7)	1.3 ± 0.0	1.5 ± 0.1	2.4 ± 0.2	1.6 ± 0.1	1.2 ± 0.1	1.4 ± 0.1	4.4 ± 0.5	2.8 ± 1.6	2.6 ± 1.1	4.0 ± 0.2	1.8 ± 0.2	3.7 ± 1.9	0.8 ± 0.6	0.4 ± 0.3	1.6 ± 0.3	0.3 ± 0.4	0.6 ± 0.5	0.4 ± 0.6
	Phenol (5)	19.7 ± 0.5	7.0 ± 0.7	20.4 ± 1.7	16.9 ± 1.6	15.5 ± 0.4	13.9 ± 0.3	5.6 ± 0.4	5.9 ± 0.4	12.1 ± 3.9	13.5 ± 0.4	9.3 ± 0.7	13.6 ± 1.4	4.7 ± 3.7	5.9 ± 2.3	15.2 ± 2.3	10.8 ± 7.2	6.6 ± 2.4	5.1 ± 0.1
	Unknown (17)	0.7 ± 0.1	1.6 ± 0.1	4.4 ± 0.3	3.7 ± 0.2	3.0 ± 0.0	3.0 ± 0.0	12.1 ± 1.2	9.9 ± 1.2	17.0 ± 5.7	10.5 ± 1.6	4.5 ± 1.0	8.7 ± 4.7	5.3 ± 0.7	4.7 ± 1.6	22.5 ± 4.7	9.1 ± 2.5	9.4 ± 0.4	7.3 ± 1.5

Table 3: Chemical constituents by concentration ± SD (μg/μL) in peels' oils from K, D, and Z.

Note: The c in Kc, Dc, and Zc is the simplification of concentration. The same for following tables.

Results and Discussion

Altogether, 97 compounds were quantitated in which 80 components were identified and 17 components with unknown structures were detected. With regards to their relative area ratio or concentration, β-myrcene, p-cymene, limonene, γ-terpinene, α-terpineol, thymol, spathulenol, and isospathulenol were found with relatively high levels in K, D, and Z, respectively.

The quantitation results of D and Z differed greatly from that reported in former researches [22-26]. Meanwhile, the total chemicals' concentration (μg/μL) was significantly less compared with that of K for both D and Z. The unexpected findings should be related to the extremely low extraction rate of D and Z (from 0.03% to 0.17%), which should affect the accuracy of quantitation. The extraction rate was greatly lower than that reported in former researches [22-27,29-31], which should be due to the peels for samples of D and Z were smashed into powder and stored for approximate 6 months prior to extraction. In this situation, the oil sacs were destroyed being ground, and some chemicals in the oils, usually with high quantities, while with relatively low boiling point like limonene, γ-terpinene, p-cymene, β-myrcene, β-pinene, linalool, etc., were easily vapored, and resulting in the relatively high quantitation for chemicals with high boiling point like some com-

pounds belonging to alcohol sesquiterpene (AS), n-hexadecanoic acid, etc.

K

Altogether, 88 compounds were detected. The content of a notable sesquiterpene as (E, E)-α-farnesene was relatively high compared with that of other varieties, its value can be from 0.7 to 1.1 while the total content of HS including 8 compounds was from 0.9 to 1.5 in area % making it account for 64.3 - 77.8% of the HS, showing that it should be an characteristic component to distinguish K from other varieties [15,27]. The finding is consistent with previous report which demonstrated that the HS fraction is highly specific for different species of *Citrus*, its total content by % can be from 0.1 to 0.4 for mandarin and can be extraordinarily high from 3.6 to 9.0 for Lime. (E, E)-α-farnesene was also a highlight identity in some varieties of mandarin accounting for 12.9 - 46.8% of total HS, however, its quantity can hardly beyond 0.2 in % [15]. Linalool, thymol and α-terpineol, were prominent monoterpenoid including oxygen atom. Spathulenol and isospathulenol were prominent AS, which % levels were relatively high compared with that of other varieties showing that these two compounds should be the potential characteristic identities. Compared with other varieties, the content of limonene was less while the content of p-cymene was higher [15,22-27,29-31].

D

Altogether, 95 compounds were detected. 11 compounds including limonene, γ-terpinene, thymol, spathulenol, isospathulenol, β-eudesmol, α-cadinol, α-sinensal, n-hexadecanoic acid, unknown-9 belonging to AS and linoleic acid were found with relatively high value in % or concentration. Limonene's quantity related to % or concentration both reached maximum for sample D5 classified as CPR. Five of them belonging to AS like spathulenol, isospathulenol, β-eudesmol, α-cadinol and unknown-9 attained the maximum value in % or concentration respectively for sample D3 categorized as CPRV, and isospathulenol was the highlight one among them. Therefore, the total amount in % or concentration of AS was relatively high and its maximum value in % or concentration was also found in sample D3. The amount in % or concentration of α-sinensal reached its maximum for sample D1 categorized as FCI and the amount in % or concentration of n-hexadecanoic acid and linoleic acid attained its maximum in sample D2 categorized to FCI, respectively. A high level both in % and concentration of n-hexadecanoic acid should be noted. As a result, total % value of oxygenated compounds was much higher than that reported by former studies on D [22-25,27-28].

Z

Altogether, 74 compounds were detected. 7 among them like limonene, thymol, isospathulenol, β-eudesmol, α-cadinol, unknown-9, and n-hexadecanoic acid were found with relatively high quantitation. Limonene's quantity reached maximum for Z2 classified as CPRV by % and for Z5 categorized as CPR by concentration, the minimum value was presented in Z4 classified as CPRV by % and concentration. Thymol's qutitation attained maximum for Z4 categorized as CPRV in % and for Z3 categorized as CPRV in concentration and reached minimum for Z6 classified as CPR by % and concentration. The content of isospathulenol, β-eudesmol, α-cadinol, and unknown-9, all belonging to AS, in which isospathulenol was highlighted, attained the maximum for Z3 categorized to CPRV and reached the minimum for Z2 classified as CPRV, respectively. Therefore, the total amount of AS attained its maximum value in Z3 and minimum value in Z2. The % amount of n-hexadecanoic acid was greatly higher than expected, its maximum value was found in Z1 categorized to CPRV, and the minimum value was present in Z6 classified as CPR. The concentration of n-hexadecanoic acid reached the maximum in Z3 belonging to CPRV and attained the minimum in Z6. As a result, total % of oxygenated compounds was much higher than that found in former studies [22-28], reaching the maximum in Z3 and the minimum in Z2 categorized to CPRV. At the same time, total concentration of oxygenated compounds reached the maximum in Z3 and attained the minimum in Z2.

Comparison of K, D, and Z

It can be seen that the chemical changeability of D and Z was drastically different from that of K in % or concentration, particularly for components like limonene, spathulenol, isospathulenol, n-hexadecanoic acid, and linoleic acid. In general, the % of limonene in D and Z was less than that of K. The % of total oxygenated compounds (TOC) in D and Z was significantly higher than that observed in K, and the % of AS or n-hexadecanoic acid in D and Z was also greatly more than that in K. Although, the concentration of total compounds in D and Z was greatly less than that in K, respectively, the concentration of AS or n-hexadecanoic acid in D and Z was also greatly more than that in K.

It is noteworthy that the intermediate growing stage such as August or September is an important change point for K, D, and Z.

PCA result

The PCA result for the peel oils of K, D, and Z by relative % or concentration of 97 components demonstrated that the peels of K, D, and Z all can be classified into three categories in generally, which was consistent with the traditional usage of these peels in Chinese medicine on the whole (Figure 3). For D, class I included D2, II included D1, D3, and D4, and III included D5, and D6. For K, class I included K1, II included K3, K4, and III included K5, K6, and K2. For Z, class I included Z3 and Z4, II included Z1, and III included Z5, Z6, and Z2. It can also be seen that the chemical variation of D and Z was more profound than that of K.

Figure 1: The Oils' TIC of D2 (a), K2 (b), and Z2 (c). The identified compounds with corresponding numbers denoted in this figure are the same as those with the identical numbers in table 2, respectively.

Figure 2: The chemical changeability by area percentage (A) and concentration (B) of K, D, and Z. Abbreviations HM and TOC refer to hydrocarbon monoterpene and total oxygenated compounds, respectively.

Figure 3: The PCA scatterplot of peel oils from K, D, and Z. "—" stand for peel oils of K, "▽" stand for peel oils of D, "|" stand for peel oils of Z.

Component	Characteristic ion peaks (M/W, %)	Deduced formula
Unknown-1 (OM)	69 (100), 84 (70), 91 (24), 109 (22), 137 (16), 124 (16), 119 (15), 110 (12), 134 (9.5), 152 (4.9), 153 (0.6)	$C_{10}H_{16}O$
Unknown-2 (AM)	109 (100), 55 (59), 67 (57), 91 (55), 119 (46), 105 (26), 134 (24), 123 (19), 137 (14), 152 (3.2), 153 (0.4)	$C_{10}H_{16}O$
Unknow-3 (IT)	135 (100), 150 (35), 91 (16), 115 (15), 107 (7.8), 77 (6.7), 117 (6.2), 105 (4.9), 116 (3.8), 151 (3.6), 121 (2.8)	$C_{10}H_{14}O$
Unknown-4 (IT)	135 (100), 150 (30), 91 (15), 107 (7.3), 115 (7.3), 77 (7.0), 105 (4.7), 117 (4.3), 151 (3.2), 121 (2.8), 116 (1.6)	$C_{10}H_{14}O$
Unknown-5 (AS)	161 (100), 105 (89), 91 (56), 204 (40), 147 (33), 133 (31), 189 (30), 177 (12), 205 (7.8), 222 (1.3), 206 (1.2)	$C_{15}H_{26}O$
Unknown-6 (AS)	105 (100), 91 (98), 131 (97), 159 (85), 177 (29), 187 (23), 205 (16), 202 (9.0), 192 (5.7), 220 (5.4), 221 (0.9)	$C_{15}H_{24}O$
Unknown-7 (AS)	205 (100), 93 (94), 159 (46), 147 (35), 146 (35), 162 (23), 206 (15), 177 (16), 187 (18), 202 (11), 220 (3.9)	$C_{15}H_{24}O$
Unknown-8 (AS)	159 (100), 177 (44), 220 (27), 145 (21), 187 (19), 149 (15), 202 (15), 192 (9.0), 205 (9.0), 221 (4.6), 222 (0.4)	$C_{15}H_{24}O$
Unknown-9 (AS)	159 (100), 109 (86), 177 (28), 220 (26), 145 (22), 149 (18), 202 (16), 187 (14), 205 (6.8), 221 (4.0),222 (0.3)	$C_{15}H_{24}O$
Unknown-10 (AS)	133 (100), 131 (66), 145 (66), 159 (47), 187 (38), 161 (36), 220 (23), 202 (19), 205 (5.3), 221 (3.6), 222 (0.4)	$C_{15}H_{24}O$
Unknown-11 (AS)	91 (100), 93 (96), 105 (96), 159 (67), 205 (49), 202 (46), 220 (41), 173 (19), 177 (17), 221 (6.9), 222 (0.7)	$C_{15}H_{24}O$
Unknown-12 (AS)	93 (100), 159 (68), 220 (52), 187 (43), 205 (30), 164 (30), 177 (26), 202 (19), 191 (17), 221 (8.3), 222 (0.8)	$C_{15}H_{24}O$
Unknown-13 (OS)	55 (100), 95 (91), 139 (86), 266 (35), 181 (34), 195 (11), 223 (7.0), 267 (6.3), 205 (3.8), 235 (2.7), 237 (2.6)	$C_{16}H_{26}O_3$
Unknown-14 (AD)	69 (100), 136 (47), 286 (37), 243 (20), 271 (13), 268 (10), 287 (8.3), 225 (7.9), 257 (4.1), 253 (3.8), 288 (0.9)	$C_{20}H_{30}O$
Unknown-15 (OD)	300 (100), 107 (81), 79 (41), 301 (22), 135 (25), 203 (15), 243 (8.9), 257 (7.1), 302 (2.7), 254 (1.3), 272 (1.1)	$C_{20}H_{28}O_2$
Unknown-16 (AD)	107 (100), 69 (43), 243 (30), 136 (17), 286 (14), 271 (6.2), 268 (4.1), 287 (3.0), 257 (2.8), 253 (2.2), 288 (0.4)	$C_{20}H_{30}O$
Unknown-17 (Al)	57 (100), 71 (74), 43 (64), 85 (58), 99 (22), 127 (9.1), 281 (8.9), 197 (2.6), 309 (2.5), 310 (0.7), 267 (0.7)	$C_{24}H_{50}$

Table 4: The characteristic ion peaks of unknown compound.

The oils' chemotype

Lota., *et al*. [32] and Fanciullino., *et al.* [33] differentiated the chemotype of oils in peels of the cultivars of *C. reticulata* by the contents of limonene and γ-terpinene, which is demonstrated in table 5. In fact, nearly all oils consist of limonene as a primary component while its relative % can be ranged from 52.2% to 96.2% or even more, and most oils comprise γ-terpinene as the second most abundant component which content can be ranged from trace to 36.7% or even more. In addition to these two important components, other prominent components such as methyl N-methylanthranilate, linalool, and thymol should be considered, too [15,22-33]. As for methyl N-methylanthranilate, it is not detected or the content is trace in many varieties, whilst its content can be more than 1.0% in a few varieties like *C. reticulata* 'Chachi' [24,25,32-33]. As for linalool, its relative content can be ranged from 0 to 4.7% or even more, which in D is higher compared with that in other varieties [25]. As for thymol, it is usually detected with its

The number of cultivars			Collection place	Collected time	Peel status	Chemotype		Characterized compounds（relative area percentage detected by GC-FID					
								Limonene	γ-Terpinene	Linalool	Thymol	(*E*, *E*)-α-Farnesene	Methyl *N*-methylanthranilate
41	34	12	The Station de Recherches Agronomiques of the INRA-CIRAD in San Ghjulianu (Corsica, France)	1996/12-1997/04	Peel of fresh fruit [32]	Limonene	γ-Terpinene I	91.6-96.2	tr-0.5	0.1-2.5	nd	tr-0.1	nd
		22					γ-Terpinene II	83.8-91.4	3.3-6.0	0-2.5	0-0.1	tr-0.1	nd
	7	6				Limonene/γ-Terpinene 52.2		69.5-81.3	11.2-20.1	0-0.7	0-0.5	tr-0.1	0-0.4
		1						36.7	0.3	0.0	tr-0.1	1.1	
31	29			2002/01-2002/03	Peel of fresh fruit [33]	Limonene		86.0-95.7	0-5.8	0-1.6	0-0.1	0-0.7	0-0.4
	2					Limonene/γ-Terpinene		69.4, 62.5	17.1, 23.3	0.1, 1.4	0, 0.1	0.2, 0	0.7, 0

Table 5: The standards to classify the chemotype of essential oils from peels of *C. reticulata* reported by Lota., *et al*. [32] and Fanciullino, *et al.* [33].

isomers such as carvacrol, 3-methyl-4-isopropylphenol, etc., and its content is higher than the other isomers at most time [34]. The method to classify the chemotype was established by the author previously [35].

Taking account for the great variation of these characteristic components, there should be a more exact standard to classify the chemotype. In view of the important status of (E, E)-α-farnesene in this study, which content is ranged from 0.7% to 1.1% in K, while its content is not more than 0.2% in many other varieties [5,15,22-28,30-33], it should be included into the system to classify the chemotype. In present, the exact standard is listed in table 6 with two criteria for each component. Through analyzing the data in this study and reported in literatures [27,29-31,36-40], this system can effectively discriminate lots of peel oils from different varieties of *C. reticulata*, which can be seen in table 7.

Component	Standard (by the relative area percentage detected by GC-FID)	Chemotype classification
Limonene	≥ 85.0	Limonene I
	85.0 > ≥ 70.0	Limonene II
γ-Terpinene	≥ 12.0	γ-Terpinene I
	12.0 > ≥ 6.0	γ-Terpinene II
Methyl *N*-methylanthranilate	≥ 0.8	methyl *N*-methylanthranilate I
	0.8 > ≥ 0.4	methyl *N*-methylanthranilate II
Linalool	≥ 2.5	Linalool I
	2.5 > ≥ 1.5	Linalool II
(*E*, *E*)-α-Farnesene	≥ 0.8	(*E*, *E*)-α-Farnesene I
	0.8 > ≥ 0.3	(*E*, *E*)-α-Farnesene II
Thymol	≥ 1.2	Thymol I
	1.2 > ≥ 0.5	Thymol II

Table 6: The standards used for chemotype classification.

Cultivar	Collected place	Collected time	Classification of peel	Extraction method	Characterized compounds (relative % detected by GC-FID)					
					Limonene	γ-Terpinene	Linalool	Thymol	(*E*, *E*)-α-Farnesene	Methyl *N*-methylanthranilate
Ponkan	Kochi Fruit Experimental Station (Kochi, Japan)	2002/12	Fresh peel of mature fruits [36]	Cold-Pressed	80.3 II	4.7	0.6	tr	tr or nd	nd
Mandarin	the plantation, situated 30 km north of Bucaramanga (Santander, Colombia).	1993/12, 1994/02, and 1994/04	Completely green (I), intermediate maturity stage (II) and fully ripe fruits (III) [30]	Steam distillation[a]; cold pressing[b]	83.5- 84.3[a] II; 89.3-90.1[b] I	3.6- 4.3[a]; 3.4- 3.9[b]	nd[a]; nd[b]	nd[a]; nd[b]	nd[a]; nd[b]	nd[a]; nd[b]
C. reticulata	the vicinity of Obafemi Awolowo University (OAU, Nigeria)	-	Peels of unripe tangerine fruits [29]	Hydrodistillation	67.0	10.8 II	4.7 I	nd	0.2	nd
Kara	the Boarstal farm of the Kenya Prisons Department, Shimo la Tewa, Mombasa District, Coast Province.	-	Peel of fruits at maturity [31]	Cold-pressing	90.7 I	2.7	0.2	nd	nd	nd
Sabine, a local cultivar in Kenya					87.1 I	1.5	0.3	0.1	nd	nd
Mandarin from Burundi	Bujumbura, the capital of Burundi, cultivated in the surrounding areas.	2004/05	Fruit at its best harvest maturity [37]	Cold-pressing	84.8 II	5.4	0.7	tr	nd	nd
Vietnamese tangerine	the H`ang Da market in Hanoi; produced in H`atˆay province in the Northern Delta.	-	Fresh and of the best commercial quality [38]	Hand pressing	95.1 I	nd	0.2	nd	nd	nd
Santra	the Research Station of the Faculty of Agriculture, Banha University, Egypt	2014/01	[39]	Hydrodistillation	79.6 II	6.5 II	1.0	nd	nd	nd
Khasi mandarin	the farmers' fields of Tinsukia district of Assam in India	2008–2010	I: green II: turning, and III: full ripe (40)	hydrodistillation	89.0-90.5 I	4.1-4.9	0.2-0.6	-	-	-

Table 7: The relative area percentage of six characteristic components in different oil-types.

Note: The a or b in superscript denotes the data is related with steam distillation or cold pressing, respectively. The symbol I, and II after the number denotes it can be classified into that chemotype. The "-" denotes the value or the information is not reported by the literature.

By this standard, all oil-types of K can't be classified as the chemotype of Limonene and Methyl N-methylanthranilate. Furthermore, all oil-types can be classified as Linalool I, (E, E)-α-Farnesene, in which all belong to I except for K3 and K4 belonging to II, and Thymol, in which all belonging to I except K2 belongs to II. Only K1 and K2 can be classified as γ-Terpinene II. Based on the unexpected data of D and Z, all oil-types of them can't be classified as the chemotype of limonene, γ-terpinene, and methyl N-methylanthranilate, while can be classified as Thymol I. Only Z6 can be classified as Linalool II. Besides, D5, Z3, and Z6 can be classified as (E, E)-α-Farnesene II.

Conclusion

The chemical variability of peel oils from D and Z was significantly different from the chemical variability of peel oils from K and the chemical fluctuation of D and Z was much greater than that of K over time. In general, the peel of K, D, or Z can be classified into three kinds according to their chemical changeability of volatile oils, respectively, which is consistent with the traditional usage of peels from *C. reticulata* in China. The quantitation outcome of components in the peel oils of D and Z was substantially different from the data reported in previous studies [15,22-27,29-33]. The standards to classify the chemotype of peel oils from different varieties of *C. reticulata* established by the content variation of six characteristic components such as limonene, γ-terpinene, linalool, thymol, methyl N-methylanthranilate, and (E, E)-α-farnesene have important meaning to differentiate those oils. The data in this study demonstrated that (E, E)-α-farnesene is an important component for differentiating the peel oils of K from that of other varieties.

Moreover, due to the unexpected findings of D and Z, some components with high boiling point, which were seldom or never detected in previously report to the best of authors' knowledge, such as three diterpenoids - unknown-14 (AD), unknown-15 (OD) and unknown-16 (AD), oleamide, linoleic acid, linolenic acid, were detected and highlighted owing to their relative high % values in this study.

Acknowledgements

The authors are highly appreciative of the support from the scientific and technological research program of Chongqing Municipal Education Commission (Grant No. CY140304, KJ1500228) and Chongqing Municipal Health and Family planning Commission (No. zy201602104). The authors are grateful to the assistance of colleague Lianju Ma, undergraduates Jing Wu, Pinjian Zhao, and all employees for their efforts and supports for this research. Jamie de Seymour has proofread the manuscript.

Disclosure

There is no conflict of interest.

Funding Information

The scientific and technological research program of Chongqing Municipal Education Commission. Grant Number: no. CY140304, KJ1500228. Chongqing Municipal Health and Family planning Commission. Grant Number: no. zy201602104.

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