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Analysis of volatile aroma compounds from five types of Fenghuang Dancong tea using headspace-solid phase microextraction combined with GC-MS and GC-olfactometry

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Abstract

The present work describes the relationship between volatile aroma components and flavours of Fenghuang Dancong tea. Volatile aroma components of five types of Fenghuang Dancong tea namely Baxian, Milanxiang, Yulanxiang, Guihuaxiang, and Yinhuaxiang were extracted by headspace solid-phase microextraction (HS-SPME), then analysed by gas chromatography-mass spectrometry (GC-MS) technique and gas chromatography olfactometry (GC-O) techniques. The GC-MS results showed that a total of 116 volatile components were detected, among which 21 (including alcohols, esters, olefins, aldehydes, ketones, and alkanes) were commonly detected in all types of tea. Based on GC-O analysis, 26 active ingredients that mainly contribute to grassy, sweet, floral, fruity, woody, and honey aromas were detected. Among these ingredients, four compounds including linalool oxide I, linalool, nerol, and neroli, which give floral, sweet, and honey aromas were abundant (with high aroma intensity) in all five types of tea. This suggests that these compounds are the main components contributing to the unique aroma of Fenghuang Dancong tea.

Introduction

Fenghuang Dancong tea is a type of oolong tea (*Camellia sinensis*) that has been planted in the Fenghuang Mountain in Chaozhou City, Eastern Guangdong Province, China for more than 700 years (Wu and Zhu, 2016). Fenghuang Dancong tea is famous for its unique fragrance, and according to sensory evaluation, it is divided into many types including Baxian, Milanxiang, Yulanxiang, Guihuaxiang, and Yinhuaxiang (Su *et al.,* 2016). Aroma is an important factor, which affects the flavour and price of the tea. At present, research on volatile aroma components of Fenghuang Dancong tea is rare and mainly carried out by gas chromatography-mass spectrometry (GC-MS), which provides rapid detection (Shi *et al.,* 2016). GC-MS is also highly efficient and can analyse volatile aroma components in tea, both qualitatively and quantitatively (Ma *et al.,* 2018). However, the aroma of tea not only depends on the content of volatile components, but also is affected by the odour and threshold of volatile components. Consequently, some active aroma components that contribute to the primary aroma of tea cannot be detected by GC-MS (Xu *et al.,* 2016). Gas chromatography-olfactometry (GC-O) is a technique that combines the separation ability of gas chromatography and human nose sensitivity of the olfactory test. The content of active aroma components in tea aroma can be determined by a sniffing test that is usually carried out by evaluators (Chen *et al.,* 2019). At present, GC-O is being applied to study the aroma components of various types of tea, such as green (Zhu *et al.,* 2018), pu-erh (Xu *et al.,* 2016), and black (Kang *et al.,* 2019) teas; however, it has not been applied on Fenghuang Dancong tea.

Extraction of volatile compounds is a critical step in the analysis of volatile components of tea. Many extraction methods for Fenghuang Dancong tea have been reported, and the simultaneous distillation (SDE) technique is the most popular among researchers. This technique can simultaneously separate and concentrate volatile compounds. However, in this technique, the sample is exposed to high temperature and humidity for a long period of time, thus causing its composition to change easily (Xu *et al.,* 2016). The headspace solid-phase microextraction (HS-SPME) technique allows the sample to be at a constant temperature in a closed container, in which, the sample is heated until

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its volatile components are volatilised. After the volatile components in the headspace bottle reach the gas-liquid (or gas-solid) phase equilibrium, they can be directly extracted and pumped into a GC-MS, in which, they are separated and detected (Dong *et al.,* 2019). The HS-SPME technique has many advantages including convenience, high sensitivity, and excellent selectivity and reproducibility (Ma *et al.,* 2017). It can accurately reflect the content of volatile components in the sample. This method has been applied to extract volatile components from different types of tea such as green (Tan *et al.,* 2019), white (Qi *et al.,* 2018), and pu-erh (Du *et al.,* 2019) teas. However, it has not been used to extract volatile components from Fenghuang Dancong tea.

In the present work, HS-SPME was employed to extract volatile components from five types of Fenghuang Dancong tea, and the components were then qualitatively and quantitatively analysed by GC-MS and GC-O. The difference between the compositions in the five different types of tea can provide a reference for the evaluation and development of Fenghuang Dancong tea in future studies.

Materials and methods

Sampling

A total of 50 Fenghuang Dancong tea samples produced in 2018 were obtained from reliable sources in Fenghuang County, Chaozhou City, Guangdong Province, China, and sorted to five types ($n = 10$, each) namely Baxian, Milanxiang, Yulanxiang, Guihuaxiang, and Yinhuaxiang. Three Fenghuang Dancong tea experts confirmed the identities of the tea samples, which were then ground, passed through a 60-mesh, and sealed for subsequent analysis.

Headspace solid-phase microextraction

Volatile compounds were extracted by HS-SPME by using a PDMS/DVB fibre with a 65-μ m film thickness. Before extraction, the fibre in the GC injection port was pre-conditioned for 5 min. Next, 10 g of sample was weighed into a 150-mL vial, and infused with 30 mL of boiling distilled water. The vial was then covered with tetrafluoroethylene, and equilibrated at 60°C in a water bath for 5 min. After equilibration, the SPME fibre was exposed to the headspace vial for 60 min, and the sample temperature was maintained at 60°C. After extraction, the SPME fibre was introduced into the injector port of GC, and the analytes were allowed to thermally desorb for 3.5 min (Xu *et al.,* 2016).

Gas chromatography-mass spectrometry

The GC-MS analysis of the aroma compounds was carried out using an Agilent-6890 gas chromatograph coupled with an Agilent HP5973N mass spectrometer, equipped with an HP-5MS capillary column (30 m × 0.25 μm × 0.25 μm). Purified helium was used as the carrier gas at a constant flow rate of 1 mL/min. The oven temperature was programmed as follows: initial temperature of 60°C with a hold time of 5 min; increased to 120°C at a rate of 3°C/min with a hold time of 2 min; increased to 180°C at a rate of 5°C/min with a hold time of 2 min; and finally increased to 250°C at a rate of 10°C/min. The operating conditions for the mass spectrometer were set as follows: ion source temperature, 230°C; ionisation mode, electron impact (EI); electron energy, 70 eV; and mass scan range, 40 - 400 AMU (Ma *et al.,* 2018). Each volatile aroma compound was identified based on the data library of the National Institute of Standards and Technology (NIST98 MS), and by comparison of their retention time indices (RIs) with related references (Lin *et al.,* 2012). The RIs of aroma compounds were calculated according to Song *et al.* (2013). To obtain the RIs, a series of *n*-alkanes (C_8-C_{25}, Anpel, China) was used under the same GC conditions.

Relative proportions of the components were determined from normalised FID peak areas, and the relative content was calculated by Eq. 1:

Relative content (%) = (single constituent area/total area) × 100% (Eq. 1)

Gas chromatography-olfactometry

The GC-O analysis was performed on an Agilent-6890 equipped with a flame ionisation detector (FID) and a sniffing port (Sniffer 9000, Brechbühler, Switzerland). Samples were separated and evaluated with an HP-5MS capillary column (30 m × 0.25 mm, 0.25-μm film thickness). The operating conditions were programmed as follows: injector temperature, 250°C; detector temperature, 280°C; and carrier gas, nitrogen at a flow rate of 1.0 mL/min. The extracted aroma was desorbed in a splitless mode at a GC injector port for 3.5 min. The oven temperature was programmed as described in GC-MS. The post-column flow was split 1:1 using two identical deactivated columns (1.2 cm × 0.1 mm, i.d.); one was directed to the sniffing port, and the other directed to an FID. The temperature of the transfer line to the GC-O sniffing port was maintained at 250°C, and humidified air was added in the sniffing port at 50 mL/min to maintain olfactory sensitivity (Chen *et al.,* 2019).

Five assessors conducted the sniffing of the aroma extracted from Fenghuang Dancong tea. All assessors had at least three years of experience in sensory evaluation of Fenghuang Dancong tea, and received more than 30 h of training over two weeks on GC-O analysis. The aroma intensity was evaluated using a 4-point scale (0 to 4): 0 = none, 1 = slight, 2 = moderate, 3 = strong, and 4 = extreme intensity (Lv *et al.,* 2012). In the sniffing test, the assessors sniffed the extracted aroma compounds through a sniffing mask, then recorded the retention time, intensity value, and aroma descriptor. Each sample was sniffed twice by the same assessor, and the ten aroma intensity values obtained were averaged. An aroma that was detected by at least three assessors at similar intensity and retention time was considered to be an active aroma.

Results and discussion

GC/MS identification of the volatile components in five types of Fenghuang Dancong tea

The volatile components found in the five types of Fenghuang Dancong tea analysed by GC-MS are shown in Table 1. As can be seen, a total of 116 volatile aroma components were detected, including 64 in Baxian tea, 41 in Milanxiang tea, 58 in Guihuaxiang tea, 55 in Yulanxiang tea, and 75 in Yinhuaxiang tea. These volatile aromas included 24 types of alcohols, 23 esters, 18 alkenes, 15 aldehydes, 12 ketones, 9 hydrocarbons, and 15 types of other compounds; among which, a total of 21 types were found in all samples. Among these 21 types, the components that were found in comparatively higher contents were β-linalool, linalool oxide I, linalool oxide II, indole, nerolidol, phytane, and phytol. As shown in Table 1, Baxian tea contained high amounts of β-linalool, linalool oxide II, nerol oxide, indole, 2-methoxy-4-(1-propenyl)-phenol, and nerolidol. Milanxiang tea contained high amounts of linalool oxide II, β-linalool, nerol, indole, phytane, myristic acid, and methyl palmitoleate. Guihuaxiang tea contained high amounts of linalool oxide II, indole, myristic acid, linalool oxide I, indole, nerolidol, and octadecane. Yulanxiang tea contained high amounts of linalool oxide II, indole, myristic acid, phytane, phytol, methyl palmitoleate, and torreyol. Yinhuaxiang tea had high amounts of linalool oxide II, indole, 2-methoxy-4-(1-propenyl)-phenol, nerolidol, and octadecane. The contents of various components including alcohols, ketones, esters, aldehydes, alkanes, alkenes, and other components are shown in Figure 1.

Of all the volatile aroma components, β-linalool was the most abundant compound. This result is consistent with that described in many reports on aromas in oolong tea. Yang *et al.* (2013) reported that teas with different scents have different β-linalool levels. The content of this compound is related to the tea variety and production process. Lv *et al.* (2012) found high levels of β-linalool in many kinds of green and oolong tea, and in low levels in pu-erh tea. He proposed that the β-linalool content in tea is related to the fermentation conditions, and he speculated that β-linalool might undergo significant oxygenation during the post-fermentative process. In the present work, in all five tea samples, the levels of linalool oxide I, linalool oxide II, nerol, and nerolidol, which have floral and fruity aromas, were high, thereby having a great influence on aroma. Zhou *et al.* (2016) discovered that the ratios of β-linalool to nerolidol in different types of Fenghuang Dancong tea have a certain relationship. Tea with a higher ratio of β-linalool to nerolidol has a rich floral aroma, while that with a lower ratio has a fruity aroma. Nerolidol, which is rich in some of the Fenghuang Dancong teas, contributes to fruity aroma (Xiao *et al.,* 2017). In the present work, the nerolidol content in Yinhuaxiang tea was 15.43%, which is consistent with that reported by Ma *et al.* (2014), thus indicating that nerolidol plays an important contribution to the main aroma of Yinhuaxiang tea. Phytol has a faint scent of grass and a hint of pepper (Wang *et al.,* 2016), and it also contributes to the aroma of Fenghuang Dancong tea. In Yulanxiang tea, the phytol content was as high as 10.77%, thus suggesting that it is the key component contributing to the tea's aroma. Geraniol, which has a rose aroma, is also an important aroma compound in tea (Ma *et al.,* 2018). Although geraniol has a very low olfactory threshold with a low content of only

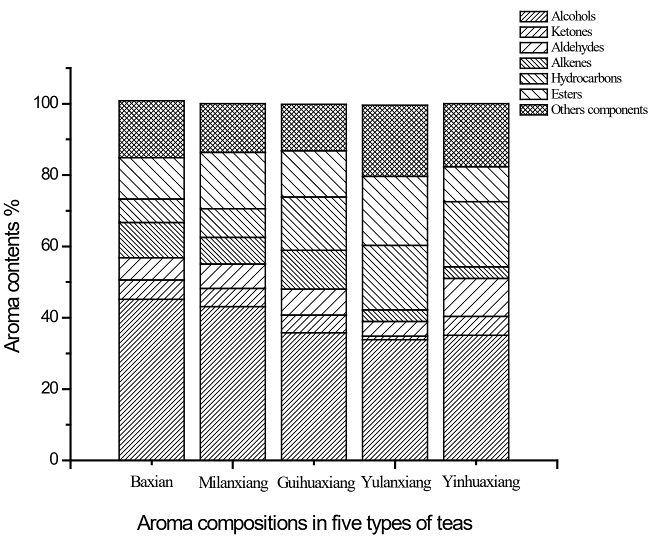

Figure 1. Comparison of the relative contents of different chemical classes of volatile compounds in Fenghuang Dancong tea.

Table 1. Volatile compounds of Fenghuang Dancong tea as detected by GC-MS.

No.	RI	Compound	Relative content (%)				
			Baxian (n = 10)	**Milanxiang (n = 10)**	**Guihuaxiang (n = 10)**	**Yulanxiang (n = 10)**	**Yinhuaxiang (n = 10)**
Alcohol							
1	1003	2-Ethyl-4-methyl-1-pentanol	-	2.19 ± 0.46	-	-	-
2	1006	*Trans*-2-decen-1-ol	-	-	-	0.29 ± 0.05	-
3	1030	Benzyl alcohol	0.97 ± 0.21	1.17 ± 0.38	-	0.94 ± 0.24	0.45 ± 0.08
4	1041	*Trans*-sdecen-1-ol 2	-	-	-	0.64 ± 0.11	0.30 ± 0.05
5	1064	Linalool oxide I	1.57 ± 0.36	1.67 ± 0.25	3.00 ± 0.53	0.43 ± 0.14	0.32 ± 0.01
6	1074	Linalool oxide II	4.03 ± 0.69	15.99 ± 2.17	12.24 ± 3.65	6.07 ± 0.94	6.71 ± 1.78
7	1102	β-Linalool	27.43 ± 3.95	2.61 ± 0.43	4.87 ± 0.17	1.58 ± 0.25	1.64 ± 0.30
8	1109	Hotrienol	-	-	0.46 ± 0.05	-	-
9	1115	Phenylethyl alcohol	-	-	-	0.48 ± 0.09	0.52 ± 0.17
10	1168	Epoxylinalool	0.44 ± 0.03	-	-	0.36 ± 0.02	-
11	1172	1-Nonanol	-	-	0.50 ± 0.03	0.31 ± 0.05	0.32 ± 0.01
12	1226	Nerol	0.79 ± 0.24	3.32 ± 0.08	2.45 ± 0.32	1.27 ± 0.33	1.39 ± 0.20
13	1229	(*Z*)-3-decenol	-	-	-	-	0.45 ± 0.09
14	1268	Nerol oxide	4.40 ± 0.62	2.90 ± 0.35	2.98 ± 0.05	1.26 ± 0.33	1.58 ± 0.20
15	1279	Geraniol	0.34 ± 0.05	0.73 ± 0.19	-	-	0.19 ± 0.03

16	1303	2-Butyl-1-octanol	0.35 ± 0.01	0.84 ± 0.38	0.47 ± 0.03	-	-
17	1372	1-Undecanol	0.43 ± 0.01	-	-	-	0.38 ± 0.01
18	1559	Nerolidol	2.59 ± 0.17	2.16 ± 0.33	4.12 ± 0.05	2.29 ± 0.47	15.43 ± 1.95
19	1604	Cedrol	0.38 ± 0.06	-	0.43 ± 0.06		0.30 ± 0.01
20	1628	(*Z*)-9-Hexadecen-1-ol	-	-	1.34 ± 0.03	0.44 ± 0.07	3.21 ± 0.52
21	1630	α-Cadinol	-	-	-	0.29 ± 0.04	0.16 ± 0.01
22	1711	2-Hexyl-1-decanol	0.32 ± 0.01	-	0.70 ± 0.24	-	0.86 ± 0.05
23	1850	Phytol	1.16 ± 0.23	5.61 ± 0.68	2.19 ± 0.16	10.77 ± 2.33	0.90 ± 0.18
24	1927	Torreyol	-	3.91 ± 0.56	-	6.43 ± 1.96	-
		Total	**45.20[a]**	**43.10[a]**	**35.75[b]**	**33.85[b]**	**35.11[b]**
Ketone							
1	859	2(1H)-Pyridinone	0.64 ± 0.12	-	-	-	-
2	875	γ-Butyrolactone	0.83 ± 0.29	1.38 ± 0.36	-	-	0.20 ± 0.04
3	938	Chlorobutyrophenone	0.64 ± 0.15	-	-	-	-
4	989	6-Methyle-5-hepten-2-one	-	-	-	-	0.25 ± 0.08
5	1038	2,2,6-Trimethylcyclohexanone	-	0.99 ± 0.25	-	-	-
6	1047	Isophorone	0.45 ± 0.04	0.64 ± 0.11	0.47 ± 0.19	0.25 ± 0.08	-
7	1385	*Cis*-Jasmone	1.39 ± 0.07	-	1.91 ± 0.13	-	1.85 ± 0.36
8	1422	Cyclopentadecanone	-	-	-	-	1.04 ± 0.09

9	1427	α-Ionone	0.60 ± 0.02	-	0.58 ± 0.03	0.27 ± 0.05	-
10	1486	β-Ionone	-	-	0.46 ± 0.14	0.48 ± 0.06	0.64 ± 0.11
11	1561	Chlorobutyrophenone	-	-	-	-	0.76 ± 0.13
12	1901	Farnesyl acetone	0.86 ± 0.05	2.13 ± 0.03	1.57 ± 0.09		0.56 ± 0.10
		Total	**5.41[a]**	**5.14[a]**	**4.99[a]**	**1.00[b]**	**5.30[a]**
Aldehyde							
1	807	Hexanal	2.39 ± 0.45	-	2.01 ± 0.18	0.79 ± 0.21	2.06 ± 0.35
2	872	Furfural	-	-	0.92 ± 0.27	-	-
3	884	*Trans*-2-Hexenal	0.59 ± 0.14	1.38 ± 0.33	-	-	0.67 ± 0.02
4	908	Heptaldehyde	-	-	-	-	0.18 ± 0.06
5	935	Benzaldehyde	-	-	-	-	0.26 ± 0.03
6	1010	Phenylacetaldehyde	0.28 ± 0.08	-	-	-	-
7	1027	Octanal	-	-	0.74 ± 0.25	-	-
8	1033	Benzeneacetaldehyde	-	-	-	-	1.50 ± 0.49
9	1105	Dodecanal	-	-	-	-	0.49 ± 0.08
10	1208	Decanal	1.34 ± 0.05	2.73 ± 0.66	1.80 ± 0.13	0.77 ± 0.25	2.55 ± 0.10
11	1205	β-Cyclocitral	1.60 ± 0.03	1.90 ± 0.38	1.40 ± 0.10	1.85 ± 0.22	1.52 ± 0.23
12	1399	2-Methylundecanal	-	-	-	0.32 ± 0.05	0.15 ± 0.01
13	1408	Dodecanal	-	-	-	-	0.57 ± 0.03

14	1578	Tridecanal	-	-	-	-	0.24 ± 0.05
15	1593	(*Z*)-7-tetradecenal	-	0.80 ± 0.15	0.40 ± 0.01	0.39 ± 0.03	0.39 ± 0.02
		Total	**6.20**[b]	**6.81**[b]	**7.27**[b]	**4.12**[c]	**10.58**[a]
Alkene							
1	840	2,4,Dimethyl-1-heptene	-	-	0.58 ± 0.14	-	-
2	903	1,3,5,7-Cyclooctatetraene	0.49 ± 0.08	1.25 ± 0.21	1.28 ± 0.30	-	0.24 ± 0.01
3	905	Styrene	-	-	-	0.91 ± 0.23	-
4	927	α-Pinene	-	-	1.44 ± 0.29	-	-
5	1012	Myrcene	0.31 ± 0.02	-	-	-	-
6	1015	α-Terpinene	0.31 ± 0.13	-	0.48 ± 0.09	-	-
7	1024	Limonene	0.92 ± 0.19	0.73 ± 0.20	0.74 ± 0.31	-	-
8	1049	α-Ocimene	1.65 ± 0.18	1.59 ± 0.32	3.12 ± 0.45	0.61 ± 0.05	0.43 ± 0.02
9	1251	3,7,7-Trimethyl-bicyclo[4,1,0]hept-2-ene	4.54 ± 0.78	-	-	-	0.20 ± 0.02
10	1328	Germacrene	-	-	-		0.22 ± 0.01
11	1357	α-cubebene	0.76 ± 0.28	1.82 ± 0.05	1.01 ± 0.06	0.38 ± 0.05	0.47 ± 0.11
12	1388	Longifolene	-	1.36 ± 0.20	-	-	-
13	1391	α-Cedrene	-	-	0.88 ± 0.05	-	-
14	1510	α-Farnesene	-	-	-	-	0.19 ± 0.01
15	1525	δ-Cadinene	-	-	-	-	0.26 ± 0.01

16	1585	1-Hexadecene	0.44 ± 0.02	-	-	-	0.59 ± 0.12
17	1705	Cadalene	-	0.72 ± 0.26	0.43 ± 0.05	0.68 ± 0.12	0.26 ± 0.01
18	1777	1-Octadecene	0.48 ± 0.09	-	0.92 ± 0.15	0.61 ± 0.03	0.40 ± 0.05
		Total	**9.90[a]**	**7.47[b]**	**10.88[a]**	**3.19[c]**	**3.26[c]**
Hydrocarbon							
1	861	3-Ethyl-hexane				0.44 ± 0.05	-
2	1000	2,2,3,3-Tetramethylbutane	1.91 ± 0.35	-	2.58 ± 0.49	1.61 ± 0.27	0.80 ± 0.05
3	1069	Decane	0.51 ± 0.22	-	0.68 ± 0.29	0.63 ± 0.08	0.32 ± 0.0.13
4	1369	3-Methyltridecane	-	-	0.41 ± 0.08	-	0.79 ± 0.03
5	1590	Hexadecane	1.12 ± 0.08	-	-	-	0.41 ± 0.28
6	1699	Heptadecane	0.98 ± 0.36	-	-	-	-
7	1787	Octadecane	0.84 ± 0.35	1.86 ± 0.23	9.03 ± 1.73	1.90 ± 0.10	14.54 ± 3.68
8	1793	Phytane	1.22 ± 0.02	6.17 ± 0.41	2.28 ± 0.45	10.14 ± 2.03	1.43 ± 0.22
9	1938	Nonadecane	-	-	-	3.39 ± 0.23	-
		Total	**6.58[c]**	**8.03[c]**	**14.98[b]**	**18.11[a]**	**18.29[a]**
Ester							
1	880	Butylacetate	-	1.57 ± 0.09	-	-	0.29 ± 0.07
2	1036	Phenylmethyl ester	1.81 ± 0.28	1.22 ± 0.16	1.54 ± 0.33	0.68 ± 0.08	-
3	1045	Dodecalactone	-	-	-	0.63 ± 0.22	0.45 ± 0.04

4	1059	γ-Hexanolactone	0.27 ± 0.01	-	-		
5	1136	2-Acetylbutyrolactone	0.71 ± 0.12	-	-	-	-
6	1220	Hexyl pivalate	0.53 ± 0.05	-	0.70 ± 0.01	0.33 ± 0.02	0.30 ± 0.01
7	1235	*Cis*-3-hexenyl-2-methylbutanoate	-	-	-	-	0.18 ± 0.02
8	1273	Pentyl hexanoate	-	-	-	-	0.19 ± 0.02
9	1321	Hydroxyundecanolide	0.95 ± 0.20	-	-	1.01 ± 0.22	0.56 ± 0.06
10	1325	Hexyl hexanoate	-	1.37 ± 0.26	1.01 ± 0.17	-	-
11	1336	Methyl geranate	0.39 ± 0.02	-	-	-	0.33 ± 0.01
12	1383	*(Z)*-3-Hexenyl hexanoate	0.88 ± 0.09	-	0.86 ± 0.25	-	-
13	1393	*Trans*-2-hexenyl hexanoate	1.28 ± 0.18	0.82 ± 0.33	-	3.20 ± 0.12	2.88 ± 0.09
14	1413	β-Caryophyllene	0.33 ± 0.01	-	-	-	-
15	1563	Dihydroactinidiolide	0.52 ± 0.03	1.50 ± 0.08	0.69 ± 0.12	0.33 ± 0.01	0.45 ± 0.02
16	1575	2,2,4-Trimethyl-1,3-pentanediol diisobutyrate	0.42 ± 0.06	-	-	-	0.27 ± 0.01
17	1581	*Cis*-3-Hexenyl benzoate	-	-	-	0.53 ± 0.02	0.65 ± 0.11
18	1632	Methyl jasmonate	-	-	-	0.25 ± 0.01	-
19	1707	Methyl hexadecanoate	1.17 ± 0.09	1.66 ± 0.20	1.71 ± 0.02	1.11 ± 0.34	0.71 ± 0.25
20	1716	Methyl myristate	0.86 ± 0.14	2.61 ± 0.09	1.34 ± 0.26	2.32 ± 0.15	1.45 ± 0.06
21	1811	Isopropyl myristate	0.48 ± 0.08	-	0.49 ± 0.12	-	0.27 ± 0.03
22	1814	Methyl pentadecanoate	-	-	-	0.45 ± 0.06	-

23	1872	*(Z)*-9-Tetradecen-1-ol acetate	-	-	-	1.00 ± 0.08	-
24	1895	Methyl palmitoleate	0.99 ± 0.36	5.08 ± 1.22	4.58 ± 2.35	7.54 ± 0.91	0.77 ± 0.13
		Total	**11.59^{c}**	**15.83^{b}**	**12.92^{bc}**	**19.38^{a}**	**9.75^{c}**
Other							
1	854	*Para*-xylene	-	-	0.49 ± 0.09	-	-
2	867	2-Aminopyridine	-	-	0.53 ± 0.03	-	-
3	898	Ethylbenzene	0.36 ± 0.02	-	-	-	0.15 ± 0.03
4	951	1,3,5-Trimethyl-1H-pyrazole	-	-	0.83 ± 0.32	-	-
5	956	Hexanoic acid	-	-	-	-	0.77 ± 0.19
6	973	*D*-Glucopyranoside	0.53 ± 0.26	-	0.66 ± 0.22	-	-
7	1043	3-Methoxy-benzenamine	2.16 ± 0.40	-	2.41 ± 0.36	-	-
8	1155	Benzyl cyanide	2.18 ± 0.13	0.80 ± 0.14	0.43 ± 0.10	0.62 ± 0.04	1.09 ± 0.39
9	1232	1-Methyl-1-phenylethylhydroperoxide	-	-	-	0.25 ± 0.01	-
10	1290	Indole	4.75 ± 0.52	4.59 ± 0.37	3.48 ± 0.26	10.15 ± 2.31	4.97 ± 0.89
11	1359	1,1,6-Trimethyl-1,2-dihydronaphthalene	-	-	0.96 ± 0.06	0.69 ± 0.15	-
12	1368	2-Methoxy-4-(1-propenyl)-pheno	4.30 ± 0.20	2.40 ± 0.18	-	1.35 ± 0.46	8.50 ± 0.55
13	1402	2,4-Bis(1,1-dimethylethyl)-pheno	0.35 ± 0.05	0.83 ± 0.15	0.82 ± 0.32	0.58 ± 0.10	1.04 ± 0.04
14	1772	Myristic acid	1.33 ± 0.13	5.02 ± 0.38	2.48 ± 0.11	6.34 ± 0.52	1.23 ± 0.30
		Total	**15.96^{bc}**	**13.64^{c}**	**13.09^{c}**	**19.98^{a}**	**17.75^{ab}**

Mean values with similar lowercase superscript do not differ significantly according to the Duncan test.

10 μg/kg, its aroma can be easily distinguished. Torreyol is a sesquiterpene alcohol, a steroidal compound that can be produced by the cytosolic mevalonate or pyruvate pathway in which a specific enzyme is required (Lin *et al.,* 2013a), or by high temperatures such as an isomer of β-linalool (Baldermann *et al.,* 2014). In the present work, a high proportion of torreyol was detected in Milanxiang (3.91%) and Yulanxiang (6.43%) teas, which is consistent with the results reported by Zhou *et al.* (2014).

Indole is an aroma component found at a high level in all five tea samples. At a high concentration, indole exhibits a pungent odour, and at a low concentration, a subtle floral aroma (Lin *et al.,* 2013b). Indole is often detected in oolong tea, and contributes to a certain aroma (Zeng *et al.,* 2016). Dihydroactinidiolide has sweet and milky aromas (Li *et al.,* 2017), and is an essential source of sweetness and milky aroma of Fenghuang Dancong tea. Some ester components such as methyl palmitoleate and methyl mstate are dehydrated and condensed from higher fatty acids and lower alcohols, and as they have no substantial odour, they have no significant impact on tea aroma (Zhang *et al.,* 2019). Hexanal has a grass scent, providing a fresh and elegant taste to tea (Sheibani *et al.,* 2016). Among the five tea samples, Baxian, Guihuaxiang, and Yinhuaxiang teas had the highest hexanal contents of 2.39, 2.01, and 2.06%, respectively. Thus, it is evident that hexanal has a greater impact on the aroma of these types of tea as compared to others. Monoterpenes and sesquiterpenoids such as α-terpinene, myrcene, limonene, ocimene, α-farnesene, δ-jussonene, and longene also greatly contribute to tea aroma. Previous studies have shown that monoterpenes and sesquiterpenoids have sweet, floral, or woody aromas (Jang *et al.,* 2010), and their different compositions and contents may contribute to different aroma characteristics of Fenghuang Dancong teas. For example, limonene, which was detected in Baxian, Milanxiang, and Guihuaxiang teas, gives a fresh lemon flavour (Nie *et al.,* 2019). According to previous reports, limonene is also often detected in green tea, and considered as an important aroma component in green tea; the higher the level of green tea, the higher the amount of limonene (Baldermann *et al.,* 2014). α-terpinene, which also has a lemony aroma (Qin *et al.,* 2013), was detected in Baxian and Guihuaxiang teas. α-farnesene and δ-jussonene, which provide a light floral aroma (Lin *et al.,* 2013a), were detected in Yinhuaxiang tea. Ocimene has a sweet aroma, and was found at high levels in Guihuaxiang, Baxian, and Milanxiang teas, thus providing a sweet aroma. Myrcene, which also has a sweet aroma (Kim *et al.,* 2018), was detected in Baxian tea. α-cubebene is a very stable sesquiterpene olefin that is often detected in green tea (Li *et al.,* 2017). α-cubebene has a pleasant scent that is irritating and somewhat bitter (Li *et al.,* 2017), and was detected in all five tea samples. Several ketones such as α-ionone, β-ionone, *cis*-jasmone, and isophorone, which can contribute to some aromas (Yang *et al.,* 2013), were also detected in all five types of tea. α-ionone, which has a floral aroma together with violet and woody notes and also a little fruity aroma (Joshi and Gulati, 2015), was detected in Baxian, Guihuaxiang, and Yulanxiang teas at low levels. β-ionone, a carotenoid-derived compound with a sweet and fragrant aroma, has a low odour perception threshold (Liang *et al.,* 2005), thus contributing significantly to tea aroma. *Cis*-jasmone has jasmine-like aroma (Pripdeevech and Machan, 2011), and is one of the most important ingredients in jasmine oil. It was found at high levels in Baxian, Guihuaxiang, and Yinhuaxiang teas. Isophorone has a minty aroma, and as reported by Wu and Zhu (2016), has been detected in pu-erh tea. In the present work, isophorone was detected in all types of tea, except for Yinhuaxiang tea.

In the present work, 26 aroma compounds were identified by GC-O (Table 2). These included grass, sweet, floral, fruit, wood, and honey aromas. Among these aromas, the intensity of four aroma compounds (linalool oxide I, β-linalool, nerol, and nerolidol) had values of greater than 2 in all five types of tea, indicating that these compounds made high contributions to the Fenghuang Dancong tea aromas. There were nine aroma compounds with aroma intensities of greater than 2 in Baxian tea; among which were hexanal, ethylbenzene, α-terpinene, linalool oxide I, β-linalool, geraniol, nerol, indole, and nerolidol. There were nine compounds with aroma intensities greater than 2 in Milanxiang tea namely linalool oxide I, linalool oxide II, β-linalool, geraniol, decanal, nerol, dihydroactinidiolide, nerolidol, and phytol. Ten compounds with aroma intensities greater than 2 were detected in Guihuaxiang tea namely hexanal, α-terpinene, linalool oxide I, linalool oxide II, β-linalool, hotrienol, isophorone, nerol, β-ionone, and nerolidol. Seven compounds with aroma intensities greater than 2 were found in Yulanxiang tea namely hexanal, linalool oxide I, β-linalool, nerol, (*Z*)-hexenyl-3-hexanoate, nerolidol, and phytol. Eight compounds with aroma intensities greater than 2 were detected in Yinhuaxiang tea namely hexanal, ethylbenzene, heptaldehyde, linalool oxide I, β-linalool, geraniol, nerol, and nerolidol.

Linalool oxide I, β-linalool, nerol, and

Table 2. Aroma-active compounds of Fenghuang Dancong tea as detected by GC-MS and GC-O.

No.	RI	Compound	Intensity				
			Baxian (*n* = 10)	Milanxiang (*n* = 10)	Guihuaxiang (*n* = 10)	Yulanxiang (*n* = 10)	Yinhuaxiang (*n* = 10)
Alcohol							
1	1061	Linalool oxide I	2.15 ± 0.32	2.64 ± 0.17	3.08 ± 0.24	2.39 ± 0.36	2.44 ± 0.18
2	1076	Linalool oxide II	1.36 ± 0.03	2.22 ± 0.15	2.28 ± 0.04	1.45 ± 0.15	1.17 ± 0.08
3	1105	β-Linalool	3.10 ± 0.39	2.19 ± 0.32	3.92 ± 0.14	3.54 ± 0.21	2.35 ± 0.12
4	1110	Hotrienol	-	-	2.23 ± 0.05	-	-
5	1225	Nerol	3.77 ± 0.29	2.45 ± 0.19	3.23 ± 0.25	2.05 ± 0.10	3.17 ± 0.46
6	1281	Geraniol	2.41 ± 0.16	3.27 ± 0.23	-	-	2.30 ± 0.05
7	1560	Nerolidol	2.37 ± 0.09	2.11 ± 0.11	3.33 ± 0.15	2.02 ± 0.21	3.85 ± 0.46
8	1852	Phytol	1.05 ± 0.08	2.48 ± 0.21	1.25 ± 0.07	2.03 ± 0.15	1.39 ± 0.08
Ketone							
1	1049	Isophorone	1.32 ± 0.25	1.85 ± 0.08	2.76 ± 0.15	1.24 ± 0.06	-
2	1382	*Cis*-Jasmone	1.11 ± 0.05	-	1.52 ± 0.19	-	1.64 ± 0.16
3	1430	*α*-Ionone	1.42 ± 0.08	-	1.68 ± 0.12	1.04 ± 0.20	-
4	1485	*β*-Ionone	-	-	2.46 ± 0.18	1.52 ± 0.09	1.12 ± 0.06
Aldehyde							
1	809	Hexanal	2.23 ± 0.09	-	2.13 ± 0.05	3.85 ± 0.12	3.01 ± 0.08
2	910	Heptaldehyde	-	-	-	-	2.15 ± 0.23
3	940	Benzaldehyde	1.47 ± 0.08	-	-	-	1.58 ± 0.08
4	1026	Octanal	-	-	1.17 ± 0.05	-	-
5	1211	Decanal	1.42 ± 0.07	2.16 ± 0.17	1.18 ± 0.08	1.03 ± 0.28	1.63 ± 0.12
Alkene							
1	931	α-Pinene	-	-	1.22 ± 0.20	-	-
2	1020	α-terpinene	2.00 ± 0.13	-	3.36 ± 0.25	-	-
3	1513	α-Farnesene	-	-	-	-	1.39 ± 0.11
Ester							
1	883	Butylacetate	-	1.65 ± 0.09	-	-	1.24 ± 0.10
2	1325	*(Z)*-Hexenyl-3-hexanoate	1.08 ± 0.07	-	-	2.96 ± 0.25	1.87 ± 0.22
3	1565	Dihydroactinidiolide	1.32 ± 0.14	2.64 ± 0.17	1.19 ± 0.11	1.26 ± 0.08	1.04 ± 0.06
4	1631	Methyl jasmonate	-	-	-	1.55 ± 0.18	-

nerolidol were all detected at high concentrations by GC-MS and also by GC-O analysis, and had aroma intensities greater than 2. These compounds have pleasant floral, honey-scented, and fruity aromas (Ma *et al.,* 2018) thus causing Fenghuang Dancong tea to have significant floral and fruity aromas. Nerolidol has a long-lasting fruity aroma, and is one of the aromas that are resistant to brewing (Ma *et al.,* 2014). Both dihydroactinidiolide (a sweet and milky aroma) and phytol (a grassy taste) were detected in all five teasamples, thus indicating that they are important aroma components in Fenghuang Dancong tea. The floral and sweet aromas of decanal and light floral-scent of indole were also detected in all five types of tea. These compounds contribute to the floral and sweet aroma characteristics of Fenghuang Dancong tea. Hexanal was detected in nearly all types of tea except for Milanxiang tea, and its aroma intensity was greater than 2 which contributes to a strong grassy aroma of tea (Xu *et al.,* 2016). Geraniol has a rose aroma (Xiao *et al.,* 2017) with a low threshold, and was detected in Baxian, Milanxiang, and Yinhuaxiang teas with an aroma intensity of higher than 2, thus indicating that it plays a vital role in the aroma of these types of teas. Hotrienol has been reported to be a unique component of Guihuaxiang tea (Sheibani et al., 2016). It is found in baked tea, and its amount increases significantly with baking time. It provides remarkable flower and fruity aromas (Schuh and Schieberle, 2006), thus having a positive effect on the aroma quality of Guihuaxiang tea. Based on the GC-O results, hotrienol was detected only in Guihuaxiang tea at an aroma intensity of 2, thus suggesting that it may be the component that contributes to the tea's unique aroma. The fruity α-terpinene was detected in Baxian and Guihuaxiang teas with intensities between 2 and 3. α-terpinene is often detected in various types of teas, and generally has higher aroma intensity, for example, in pu-erh and Zhengshan small black tea, its intensity can reach a value of 3 (Lv *et al.,* 2012).

GC-MS is an essential technique for analysing the aroma of Fenghuang Dancong tea. However, some of the detected volatile components are odourless, and some have thresholds so high that they cannot be detected. Therefore, not all aroma volatile components that were detected contribute to the aroma of Fenghuang Dancong tea. GC-O, which combines the separation by GC and the sniffing by trained evaluators, was applied to determine the aroma compounds and to identify the active aroma components (Chen et al., 2019). The combination of GC-MS and GC-O can effectively separate and identify active aroma components, as well as their categories and strengths which play an important role in the overall aroma of tea.

Conclusion

In the present work, HS-SPME was used to extract volatile components from five types of Fenghuang Dancong tea. One hundred and sixteen components (including alcohols, esters, and olefins) were identified by GC-MS, among which, 21 components were commonly found. Among these 21 components, those that were found in higher levels including *β*-linalool, linalool oxide I, linalool oxide II, indole, nerolidol, phytane, and phytol. Combined with GC-O analysis, 26 species of aroma compounds including grassy, sweet, floral, and fruity aromas were identified, and those that had aroma intensities of greater than 2 were linalool oxide I, β-linalool, nerol, and nerolidol. This indicates that these four aroma compounds contribute greatly to the overall aroma of Fenghuang Dancong tea.

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