Identification of the Different Aroma Compounds between Conventional and Freeze Dried Wuyi Rock Tea (Dangui) using Headspace Solid Phase Microextraction

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Wuyi rock tea, a kind of semi-fermented Oolong tea, is produced around Wuyi Mountain. Due to its unique aroma and taste, namely the aroma of flowers and the earthy taste of rocks (hence its name as Wuyi rock tea), it has become very popular in China. Its characteristics are associated with the manufacturing processes. During which many compounds are produced. Nonvolatile compounds contribute to the taste, while volatile compounds contribute to the aroma. In this study, volatile compounds from two samples using different drying methods were extracted using headspace-solid phase microextraction (HS-SPME) approach, and then analyzed, compared and identified with gas chromatography-mass spectrometry (GC-MS). Among the volatile compounds, *trans*-nerolidol, farnescene, hexyl hexanoate, indole and geraniol were identified, and found to have high content in both samples. The analytical results showed that freeze drying process is an alternative drying method, which is especially suitable for the production of highly scented Wuyi rock tea.

Keywords: Wuyi rock tea, aroma, headspace solid phase microextraction, freeze-drying

Introduction

Tea is one of the most popular beverages in the world. There are five major types of tea in China based on their processing methods: white, green, Oolong, black and Puer tea (Wang *et al.*, 2011). Wuyi rock tea is a special subcategory of Oolong tea growing around Wuyi Mountain, which is a UNESCO double World Heritage site, providing warm and moisture conditions, high elevation and a good ecological environment for the growth of this delightful Oolong tea. Wuyi rock tea is one of the most famous teas in China for its long history and unique characteristic. In addition, as a type of Oolong tea, it has potential health benefits against cardiovascular diseases and cancer as well as pharmaceutical activities, empirically recognized for aiding indigestion, lowering cholesterol levels and boosting metabolism (Chaturvedula and Prakash, 2011; de Mejia *et al.*, 2009; Dou *et al.*, 2008;

Kim *et al.*, 2011).

Wuyi rock tea is partly fermented and a moderate level of enzymatic oxidation is maintained during the manufacturing process, which is very complicated. Typically, the traditional production of Wuyi Rock tea involves 10 procedures interlocked with each other (Chen *et al.*, 2011; Dou *et al.*, 2007). The quality of Wuyi rock tea depends on the manufacturing procedures, which play an important role in the formation of the special taste and odour. Wuyi rock tea is generally produced from the buds and leaves of *Camellia sinensis* (Linnaeus) in the processes of picking and withering, fermentation, enzyme inactivation, rolling and drying (Chen *et al.*, 2012; Cho *et al.*, 2007). Conventional drying involves heating the tea leaves at a high enough temperature to maintain suitable water content after rolling. In contrast with conventional drying, vacuum freeze drying is a drying method which is performed at low temperature, and so it has been used for the long term preservation of heat sensitive food and other biological materials based on the phenomenon of subli-

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mation (Ceballos *et al.*, 2012). The freeze-drying process can preserve the properties of raw material such as shape, dimensions, appearance, taste, color, flavor, texture and biological activity. In the procedure, sample is frozen solid, and then exposed to a controlled temperature and reduced pressure (< 300 Pa) environment.

It has been well established that the tea flavor is principally determined by its chemical components, with volatile compounds contributing to the property of aroma and non-volatile compounds to the taste. Aroma is one of the most important factors which decides the properties and quality of each tea, especially Oolong tea and black tea (Cho *et al.*, 2007). However, little attention has been given on the aroma characterization of Wuyi rock tea. In the analysis of the volatile compounds in Wuyi rock tea, a mid extraction temperature would be helpful to maintain their chemical structure and composition. Headspace solid phase microextraction (HS-SPME) provides a much more simple process in comparison with the simultaneous distillation extraction (SDE) (Ravichandran and Parthiban, 1998; Wang *et al.*, 2008). Furthermore, in using HS-SPME, the volatile flavor compounds are isolated from the tea samples directly, which can avoid to some extent the effects of higher temperature in some ways.

In this study, we investigated the volatile compounds in Wuyi rock tea under different dry protocols. HS-SPME accompanied with gas chromatography mass spectrometry (GC-MS) was employed to monitor the volatile compounds. By means of the sensory evaluation (Chen *et al.*, 2011; Mejia *et al.*, 2009), the quality of freeze dried and conventional dried Wuyi rock teas samples were compared and evaluated.

The main objectives of this research were to clarify the aroma compounds of Wuyi rock tea (Dangui), and to compare the difference in aroma and quality resulting from the two different drying methods.

Materials and Methods

Manufacture of Oolong tea The fresh tea leaves were supplied by the Wuyishan Qiming tea science research institute (Wuyi Mountain City, China). The tea samples were given a solar withering process for 1–2 h (sun or air drying to partially remove moisture at air temperature), followed by indoor cooling for 1–3 h, and then tossed and rolled (to bruise of tea leaf edges and create more contact surface for oxidation) for 10–30 min. The process was duplicated for five cycles, and was then followed by oxidation termination (about 5 min) and kneading (strand making before dehydration for about 5 min) processes. The final step was drying (heat treatment to dehydrate tea leaf) (Chen *et al.*, 2011). The conventional drying was carried out using drying machinery, and the drying temperature was kept from 80°C to 120°C for

about 0.5–2 h.

We also applied a novel, vacuum freeze-drying method (the earlier procedures were the same as in the conventional drying method), in order to compare with the conventional drying. The procedure for the vacuum freeze-drying method was as follows: a tea sample (0.1 kg) was distributed evenly over a stainless steel sample pan (18 cm diameter) and pre-frozen using an ultralow temperature refrigerator, then was put in a vacuum freeze drier (Shanghai Zhixin experimental apparatus technology Co. Ltd., model ZX-1C). In this drying process, the parameters were selected as: vacuum, 70 Pa; cold trap: –43°C; the temperature of hot plate, 40°C; time, 15 h.

Sample preparation Dry tea samples were prepared in our laboratory according to the above procedure. The tea leaves were then ground and passed through a filter with 20 mesh sieve size. For the preparation of the tea infusions, 0.25 g tea sample was placed into a headspace vial, and then 20 mL pure boiling water (Milli-Q, USA) was added. The vial was immediately sealed and placed in a hot water (80°C). After equilibrium was achieved (5 min), the prepared sample was ready for the next HS-SPME procedure.

HS-SPME procedure SPME of the volatiles released from the tea infusions was carried out using a commercial 50/30 µm poly divinylbenzene (DVB)/carboxen/ polydimethylsiloxane (PDMS) fiber (Supelco, Bellefonte, PA). Before application, the fiber was conditioned for 5 min in the GC injection port. The tea infusion was prepared following the description in the sample preparation. The SPME fiber was placed in the headspace vial to extract aroma compounds, and then the fiber was inserted into the GC injector to allow thermal desorption of the analytes.

In our study, the type of fiber coating was first selected and tested, and then the experimental conditions such as water bath temperature, extraction and desorption time, as well as the desorption temperature were optimized. The extraction conditions were then set as follows: 50/30 µm SPME head (DVB/CAR/PDMS) fiber, 20 min of extraction time, 80°C extraction temperature in the water bath, 3 min desorption time and 240°C desorption temperature. Three independent extractions were performed for each sample.

GC-MS analysis Volatile compounds from the SPME fiber were analyzed using a GC-MS (Shimadzu GCMS-QP2010 plus) equipped with a quadrupole mass analyzer. The analytical conditions were set as follows: separation column was 60 m × 0.32 mm i.d. 0.25 µm Rtx-5; the injection was operated in splitless mode (240°C, 3 min); the carrier gas was nitrogen at a constant flow of 1 mL/min. and the oven temperature was programmed as follows: 40°C for 2 min, 2°C/min to 180°C, and then 5°C/min to 200°C, 10°C/

min to 240°C, and hold for 5 min. Mass spectrometer detection was performed under electron ionization (EI) conditions as follows, and its conditions: ionization energy: 70 eV; mass scan range: 35 – 600 ; scan time: 0.45 s ; interscan delay: 0.05 s.

The compounds were identified using retention index and mass spectral libraries Wiley 7 and National Institute of Standards and Technology (NIST) 08 library. Relative percentages were calculated from the Total Ion Current from the automated integrator.

Calculation of RIs In order to provide a more certain identification of the compounds, n-alkanes (C₈ – C₂₂, Sigma-Aldrich) were applied in the GC-MS. Retention indices were calculated using the n-alkanes as reference in the following expression:

$$RI(x) = 100 \times z + 100 \times \frac{RT(x) - RT(z)}{RT(z+1) - RT(z)}$$

Where RI(x) is the retention index of the unknown compounds x, z is the number of carbon atoms of the n-alkane eluted before the unknown compound x, z + 1 is the number eluted after the unknown compound x, RT(x), RT(z) and RT(z + 1) refer to the retention time of the unknown compound and the alkane before and after the unknown compounds x, respectively.

Sensory evaluation of tea samples The organoleptic quality of Oolong tea was assessed based on the appearance and the color of the tea leaves, the taste and aroma of the infusion and the features of infused young shoots (Wang *et al.*, 2010). In the sensory evaluation, the dry tea leaves were placed in a tray to evaluate their appearance. Five grams of tea leaves was put into a review cup and then infused with freshly boiled water for three times (each time 110 mL), the cup being filled with this water. The first infusing time selected was 1 min, the second time was 2 min, and the third time was 3 min. The each infusion was transferred into a tasting bowl. The sensory evaluation of the tea was estimated and scored by six professional tea tasters. The testers smelled the aroma, checked the taste, and finally, the infused tea leaves were put in a bowl with water, for their sensory evaluation. The grading system was the one commonly used to evaluate Wuyi rock tea quality in China. The system is based on a quality score (QS) of 100, 20% QS was awarded for the tea appearance score, 35% QS for the tea aroma score, 35% QS

for the tea taste score (TS) and 10% QS for the infused leaf score.

Results and Discussion

Results of sensory quality Generally, the tea grading is evaluated based on the four attributes: appearance (including the color, shape, regularity and uniformity of the tea leaf), taste and aroma of infusion, and the features of the infused leaves. A typical infusion of Oolong tea is golden with a fragrant flowery aroma, as well as a sweet and smoothing taste (Hui *et al.*, 2004). The sensory evaluation is shown in Table 1. The total scores (QS) combining the four attributes given by the tea-tasting panels were 75.5 and 86.3 for conventional and freeze dried samples, respectively. It was obvious that freeze drying tea had the higher score, its tea leaves showed a relatively lighter color, and the infusion presented a brighter color (Fig. 1).

Identification and quantification of volatile composition in Rock tea infusion The aroma constituents of various tea products are interesting research topics with potential commercial values and have been continually investigated. The first scientific studies were performed more than 70 years ago (Yamanishi and Kobayashi, 1999), leading to the identification of linalool, geraniol and

(Z)-3-hexenol in the volatile fractions of the tea leaf and beverage (Kuo *et al.*, 2011). In our study, the GC/MS

Fig. 1. Tea samples and tea infusions.

(a)conventional dried tea (b) vacuum freeze dried tea
(c) infusion of a normal dried tea (d) infusion of a freeze dried tea

Table 1. Sensory quality score of the Rock tea samples assessed by the testers (n = 6, mean ± S.D.)

Samples	Appearance score	Aroma score	Taste score	Infused leave score	QS
1	16.2 ± 1.3	26.6 ± 3.2	24.8 ± 1.3	7.9 ± 0.8	75.5 ± 1.9
2	17.6 ± 1.5	30.4 ± 2.7	29.7 ± 2.6	8.6 ± 1.4	86.3 ± 2.3

*1 conventional dried sample 2 freeze dried sample S.D. standard deviation.

chromatograms of the volatile compounds from conventional and freeze dried Wuyi rock tea samples revealed different profiles, but the highest peak of both is nerolidol (Fig. 2).

The compounds identified and the relative quantities of each compound, as calculated using the peak area, are listed in Table 2. Using the conventional drying method, 65 volatile compounds in the Wuyi rock tea sample could be identified and grouped into various classes of substances, including 15 alcohols, nine aldehydes, nine ketones, 12 esters, 13 hydrocarbons, an indole as well as other compounds. The main components were *trans*-nerolidol (34.57%), farnescene (12.18%), hexenyl hexanoate (7.36%), hexyl hexanoate (4.33%), 2-hexenyl butanoate (2.67%), indole (2.31%), 2-ethyl-1-hexanol (1.76%), caffeine (1.71%), geraniol (1.37%), pinanol (2.16%), 2,7-dimethyl-1,3,7-octatriene (1.08%), and these accounted for 71.50% of the total aroma content. While with the freeze dried sample extraction, 84 volatile compounds could be obtained and identified, including 22 alcohols, 16 aldehydes, 12 ketones, 18 esters, 12 hydrocarbons, an indole and miscellaneous compounds. The main components were *trans*-nerolidol (39.9%), farnescene (8.91%), 3-hexenyl hexanoate (7.0%), hexyl hexanoate (4.42%), 2-hexenyl hexanoate (3.39%), indole (3.62%), geraniol (3.03%), pinanol (1.76%), 2-phenylethyl benzoate (1.36%), 2-methyl-, 2-phenylethyl propionate (1.1%), phenylethyl alcohol (1.08%) and linalool (1.03%). These components represented 76.60% of the total aroma content.

Comparing the results of other studies, a total of 36 compounds were tentatively identified using agreement with their mass spectra in fresh Oolong tea (Kuo *et al.*, 2011), more than 70 volatile flavor compounds are detected in Dongding Oolong (Wang *et al.*, 2008); 52 compounds are identified from the brewed extract and 100 compounds from the SDE of Huang Chin Kuei, respectively. 49 compounds were identified from the brewed extract of Chan Pin Oolong (Kawakami *et al.*, 1995); and Wang has reported that nerolidol, indole, benzene acetaldehyde, linalool, linalool oxides, hexanal, benzyl nitrile, (*E*)-geraniol and 1-penten-3-ol are the prevailing volatile compounds detected in most of Oolong tea samples. These compounds together with methyl salicylate, methyl jasmonate, phenylethyl alcohol, benzyl alcohol, *cis*-jasmones and β -ionone are possibly the principal contributors to the fragrant flowery aroma in Oolong teas (Wang *et al.*, 2011). In addition, some aroma compounds were produced or increased during the manufacturing process, such as in the Taiwan Oolong tea, "Oriental Beauty", where the main volatile compounds increase during the manufacturing process (Cho *et al.*, 2007). The variety of aroma compounds of Wuyi rock tea mainly depends on its complex manufacturing process. Pripdeevech and Machan (2011)

Fig. 2. Total ion current chromatogram of Wuyi rock tea (Dangui). (a) conventional dried tea (b) vacuum freeze dried tea.

report that the major flavor volatiles are *trans*-nerolidol, *cis*-jasmones and geraniol in Chin Hsuan Oolong tea. According to these reports, Oolong teas have similar main aroma components, while Wuyi rock tea contains a great number of volatile compounds. As shown in Table 2, *trans*-nerolidol (No.105) is dominant in Wuyi rock tea (Dangui) and represents 34.5% and 39.9% of the total aroma constituents in the two samples. The freeze dried sample had a more complicated aroma pattern, and contains more varieties of aromatic alcohols, ester, ketone, aldehyde than those obtained from the conventional dried sample, while 2-furaldehyde could be found only in conventional dried tea samples. As Kato and Shibamoto (2001), report, 2-furaldehyde exhibited a toasted flavor known as a heat product from amino acids and sugars, and this could explain why 2-furaldehyde could not be found in freeze drying tea. Generally, hexanal (No. 6) and linalool (No. 39) give a pleasant greenish note to teas, as indicated in Table 2, hexanal existed in freeze dried samples whereas linalool occurred in both, and showed a relatively higher content in the freeze dried sample.

Using of t-test for paired samples (95% significant), there only several compounds, such as 2-Ethyl- 1-hexanol, (No. 24), 3-Hexenyl butanoate (No. 40), phenyl acetate (No. 49),

Table 2. The volatile compounds in Rock tea at different drying methods.

No.	Compounds	RI ^a	Contenta ± S.E.(%)	Contentb ± S.E.(%)	Data of t-test
1	1,3-Octanediol	1035	0.22 ± 0.08		
2	3-Nitro-2-butanol	1054		0.01 ± 0.004	
3	Methyl 2-Methyl-2-propenoate	1073		0.33 ± 0.07	
4	Cyclobut-1-enylmethanol	1085		0.02 ± 0.005	
5	3-Methyl-1-butanol	1110		0.01 ± 0.003	
6	Hexanal	1120		0.23 ± 0.08	
7	1-Ethyl-pyrrole	1127	0.39 ± 0.09		
8	2-Furaldehyde	1135	0.67 ± 0.12		
9	2-Hexenal	1140		0.12 ± 0.04	
10	3-Hexen-1-ol	1153		0.06 ± 0.003	
11	2-Hepten-1-ol	1186		0.01 ± 0.002	
12	Heptanal	1223		0.10 ± 0.03	
13	Methyl 8- nonynoate	1267	0.03 ± 0.01		
14	3-Nonen-2-ol	1272		0.02 ± 0.004	
15	Benzaldehyde	1287	0.71 ± 0.09	0.53 ± 0.10	2.32
16	2-Furancarboxaldehyde,	1302	0.06 ± 0.01		
17	1-Octene	1316		0.02 ± 0.005	
18	6-Methyl-5-hepten-2-one	1329	0.26 ± 0.07	0.63 ± 0.21	-2.89
19	2,4-Nonadienal	1331		0.14 ± 0.005	
20	3-(1-Methylpropyl)- cyclohexene	1338	0.42 ± 0.09		
21	2,4-Heptadienal	1342	0.19 ± 0.08	0.74 ± 0.13	-6.2*
22	1,2-Bis(1-methylethenyl)-cyclobutane	1347	0.20 ± 0.05		
23	2,2,6-Trimethyl-cyclohexanone	1355	0.04 ± 0.05	0.04 ± 0.005	0.0
24	2-Ethyl- 1-hexanol	1362	1.76 ± 0.18	0.81 ± 0.21	5.50*
25	Benzeneacetaldehyde	1370	0.35 ± 0.08	0.55 ± 0.10	-2.7
26	Benzyl alcohol	1376	0.12 ± 0.04	0.16 ± 0.07	-0.8
27	2,2-Dimethyl-3-methylene-bicyclo[2.2.1] heptane	1384		0.03 ± 0.005	
28	1-Ethyl- 1H-pyrrole-2-carboxaldehyde	1397	0.78 ± 0.06		
29	4-Diazodamantanone	1405	0.03 ± 0.01		
30	3-Nonyl-2-ol	1413		0.04 ± 0.007	
31	cis-4-Decenal	1420		0.14 ± 0.04	
32	Phenyl carbamate	1438		0.01 ± 0.004	
33	3,5-Octadien-2-one	1447	0.09 ± 0.01	0.20 ± 0.05	-3.74
34	1-(1H-Pyrrol-2-yl)-ethanone	1470	0.55 ± 0.08		
35	1-Octanol	1487		0.15 ± 0.04	
36	3-Ethyl-2,5-dimethyl-pyrazine	1503	0.07 ± 0.01		
37	2-Furanmethanol	1530	0.15 ± 0.03	0.12 ± 0.04	1.04
38	Benzofuranone	1551		0.03 ± 0.005	
39	linalool	1554	0.84 ± 0.07	1.03 ± 0.16	-1.88
40	3-Hexenyl butanoate	1564	2.67 ± 0.17	0.40 ± 0.07	21.39*
41	3,3-Dimethyl- 1,5-heptadiene	1584		0.22 ± 0.08	
42	2-Ethenyl-1,1-dimethyl-3-methylene-cyclohexane	1591	0.27 ± 0.08		
43	Phenylethyl alcohol	1620	0.61 ± 0.08	1.08 ± 0.20	-3.71
44	1,3,5-Tris(methylene)- cycloheptane	1635		0.02 ± 0.001	
45	cis-3-Hexenyl iso-butyrate	1679		0.02 ± 0.005	
46	(R,S)-5-Ethyl-6-methyl-3E-hepten-2-one	1693	0.04 ± 0.01	0.04 ± 0.01	0.0
47	cis-10-Pentadecen-1-ol	1720		0.10 ± 0.03	
48	2-Tridecenal	1731	0.05 ± 0.006		
49	Phenylmethyl acetate	1739	0.03 ± 0.005	0.01 ± 0.002	6.43*
50	6-Ethenyltetrahydro-2,2,6-trimethyl-2H-pyran-3-ol	1748	0.10 ± 0.05	0.05 ± 0.008	1.71
51	Cyclopentacycloheptene	1754	0.10 ± 0.04		
52	2,7-dimethyl- 1,3,7-Octatriene	1766	1.08 ± 0.16		
53	Hexyl butanoate	1779	0.15 ± 0.05	0.19 ± 0.04	-1.08
54	2-Hydroxy-, methyl benzoate	1783	1.12 ± 0.11	0.86 ± 0.21	1.9
55	2,3,5,8-Tetramethyl-decane	1790	0.37 ± 0.07		
56	2,6,6-Trimethyl-1,3-cyclohexadiene-1-carboxaldehyde	1794		0.08 ± 0.02	
57	2,6-Dimethyl-3,7-octadiene-2,6-diol	1801		0.04 ± 0.008	

58	Decanal	1813	0.25 ± 0.08	0.20 ± 0.06	0.87
59	2,6,6-Trimethyl-1-cyclohexene-1-carboxaldehyde	1824	0.36 ± 0.14	0.32 ± 0.10	0.40
60	<i>cis</i> -3-Hexenyl-.alpha.-methylbutyrate	1831	0.71 ± 0.06	0.78 ± 0.24	-0.49
61	Hexyl- 2-methylbutyrate	1835		0.31 ± 0.09	
62	Hexyl 2-methyl-butanoate	1843	0.25 ± 0.08		
63	1-Ethoxy-4-methyl-2-pentene	1847	0.18 ± 0.07	0.16 ± 0.04	0.43
64	Geraniol	1860	1.37 ± 0.16	3.03 ± 0.21	-10.89*
65	<i>cis</i> -2-Decenal	1863		0.07 ± 0.012	
66	2-Isopropenyl-5-methyl-4-hexenal	1870		0.12 ± 0.04	
67	1-Dodecene	1873		0.04 ± 0.007	
68	3-Dodecen-1-yne	1880		0.07 ± 0.012	
69	<i>cis</i> -3-Hexenyl isovalerate	1882		0.04 ± 0.007	
70	Octyl hexanoate	1891		0.10 ± 0.02	
71	1,2-Dimethyl-3-(1-methylethenyl)-cyclopentanol,	1901	0.39 ± 0.12		
72	2-Phenylethyl benzoate	1910	0.66 ± 0.14	1.36 ± 0.31	-3.56
73	Indole	1913	2.31 ± 0.69	3.62 ± 0.10	-3.25
74	3-isobutyl-Cyclohexene	1915		0.10 ± 0.03	
75	3,9-Dodecadiyne	1920	0.33 ± 0.14		
76	8-Methyl- 1-undecene	1922		0.08 ± 0.02	
77	Gamma. Dodecalactone	1932	0.16 ± 0.04		
78	Dihydro-5-pentyl-2(3H)-furanone	1934		0.14 ± 0.05	
79	2-Butyl- 2-octenal	1937		0.05 ± 0.007	
80	3-Hexenyl hexanoate	1941	7.36 ± 1.20	7.00 ± 1.60	0.31
81	Hexenyl hexanoate	1943	4.33 ± 1.02	4.42 ± 0.71	-0.12
82	2-Hexenyl hexanoate	1950	2.69 ± 0.55	3.39 ± 0.73	-0.32
83	3-Methyl-4-undecene	1952		0.05 ± 0.01	
84	Tetradecane	1953	0.19 ± 0.06		
85	3-Methyl-2-(2-pentenyl)-2-cyclopenten-1-one	1956	0.31 ± 0.11	0.52 ± 0.16	-1.87
86	Undecanal	1960		0.01 ± 0.003	
87	3-Acetylmethyl-beta.-pinene,	1962	0.23 ± 0.07		
88	Cedrene	1967		0.04 ± 0.007	
89	2-Methyl-4-(2,6,6-trimethylcyclohex-1-enyl)but-2-en-1-ol	1968	0.14 ± 0.06		
90	4-(2,6,6-Trimethyl-2-cyclohexen-1-yl)-3-buten-2-one	1973	0.14 ± 0.04	0.19 ± 0.05	-1.35
91	3-Hexenyl lactate	1976		0.08 ± 0.03	
92	5-Phenyl-2,4,6(1H,3H,5H)-pyrimidinetrione	1978		0.10 ± 0.03	
93	2-Methyl-, 2-phenylethyl propionate	1981	0.82 ± 0.01	1.10 ± 0.28	-1.61
94	6,10-Dimethyl-5,9-undecadien-2-one	1982	0.31 ± 0.08	1.06 ± 0.30	-4.18*
95	6,11-Dimethyl-2,6,10-dodecatrien-1-ol	1986	0.08 ± 0.11		
96	4-Methyl-1-(1-methylethyl)-bicyclo[3.1.0] hexan-3-ol	1993	0.06 ± 0.11		
97	4-(2,6,6-Trimethylcyclohexa-1,3-dienyl)but-3-en-2-one	1997	0.04 ± 0.01		
98	<i>trans</i> -2-Pinanol	2006	2.16 ± 0.31	1.76 ± 0.32	1.55
99	alpha.-Farnesene	2008	12.18 ± 1.48	8.91 ± 1.06	3.11
100	4-(2,2-Dimethyl-6-methylenecyclohexyl) butanal	2011	0.45 ± 0.06		
101	2,6,11,15-Tetramethyl-hexadecane	2021	0.20 ± 0.05		
102	4-Methyl-, phenylmethyl pentanoate	2029		0.04 ± 0.001	
103	2,10-Dimethyl-9-undecenal	2032	0.12 ± 0.04		
104	Cyclopropanemethanol	2036		0.36 ± 0.07	
105	<i>trans</i> -Nerolidol	2041	34.57 ± 1.91	39.90 ± 2.64	-2.83
106	3-Hexenyl benzoate	2046	1.19 ± 0.10	0.56 ± 0.12	6.98*
107	3-Methyl-3-(4-methyl-3-pentenyl)-oxiranemethanol	2013		0.31 ± 0.07	
108	Hexyl benzoate	2025		0.38 ± 0.08	
109	3-Benzoyl-2-t-butyl-4-vinyl-oxazolidin-5-one	2033		0.26 ± 0.06	
110	Cedrol	2053		0.14 ± 0.05	
111	3,7-Dihydro-1,3,7-trimethyl-1H-purine-2,6-dione	2071		0.71 ± 0.22	
112	Caffein	2094	1.71 ± 0.23		
113	bis(2-Methylpropyl) 1,2-Benzenedicarboxylate	2103		0.32 ± 0.07	
114	Methyl hexadecanoate	2109		0.23 ± 0.06	

Each sample was analyzed using 3 same samples. ± is the S.E. for %content, and * indicates significant differences; RI is the Retention Index.

Geraniol (No. 64), 3-Hexenyl benzoate (No. 106) were significantly different, and, on the whole, there was little difference between the two drying methods.

Conclusion

In this study, the volatile compounds in conventional and freeze dried Wuyi rock teas were detected and identified. More than 80 volatile flavor compounds were found in Dangui dried using the freeze drying method.

All the tea samples contained nerolidol as the most abundant aroma constituent, but only a relatively small proportion of them showed significant differences in composition between the conventional and freeze dried teas. The results from sensory evaluation indicated that the tea sample prepared using the freeze drying method presented more natural, fresh and characteristic flavor. These results suggested that the tea processing method is a very important factor in the manufacture of Wuyi rock tea with a good aroma quality, and that the vacuum freeze drying technique is a useful method to enhance the quality of Wuyi rock tea, which could be used in the mass production of a highly scented tea.

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