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Analysis of Volatile Components of Tieguanyin and Dongding Oolong Teas by Simultaneous Distillation Extraction Coupled with Gas Chromatography-Mass Spectrometry

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The difference of volatile component in Tieguanyin (from Fujian province) and Dongding oolong tea (from Taiwan province) in China was studied by simultaneous steam distillation extraction coupled with gas chromatography-mass spectrometry (GC-MS). A total of 84 volatile compounds were identified in these two kinds of oolong teas and mainly including alcohols, hydrocarbons, aldehydes, ketones and esters compounds, *etc*. Among them, alcohols are the most abundant components in Tieguanyin and Dongding oolong teas, also includes phytol, nerolidol, linalool, *etc*. In addition, nitrogens (*viz*., indole and pyrazines compounds) existed in higher concentration in Dongding oolong tea. The SDE/GC-MS is proven to be a suitable technique to extract and analyze the volatile components of oolong tea. In comparison, there were remarkable differences in flavor and aroma composition of the two types of oolong tea, were helpful for distinguishing these two oolong teas with similar appearance and quality characteristics.

Keywords: Oolong tea, Steam distillation extraction, Volatile component, GC-MS.

INTRODUCTION

Oolong tea is a semi-fermented tea goes through an intermediate process involving withering, rolled, brief oxidation and drying. Because of undergoing semi-fermentation process, tea leaves refer to natural reactions induced by oxidative enzymes. The fermentation degree of oolong tea is mostly in the range of 20-80 %[1,2], among Tieguanyin and Dongding oolong teas are the two typical oolong tea samples produced from Fujian and Taiwan province of China. And the fermentation degrees of them are approximately 20-40 %[3].

The original purpose of tea fermentation was to enhance the flavor of tea. Therefore, flavor is the most important element for tea evaluation[4]. In China, Tieguanyin and Dongding oolong teas are two kinds of famous oolong teas with slight fermentation degree. Because of the degree of fermentation is relatively close, from the point of sensory evaluation, these two kinds of oolong teas have similar appearance characteristics, taste and flavor characteristics. In fact, the difference of production areas, elevation and climate, processing techniques will result in different chemical compositions and eventually affect the quality and characteristics of the tea[5,6]. Whereas it is almost impossible to find out these differences by simply using sensory evaluation methods. To date, no study has been reported on chemical difference in Tieguanyin and Dongding oolong teas by using instrumental analysis methods. Therefore, it is necessary to study the volatile composition of tea with similar appearance characteristics and further find out the aroma differences between them and therefore distinguish them objectively.

Till now, many extraction techniques have been developed to analyze the volatile components in tea. These techniques mainly including supercritical fluid extraction, steam distillation, liquid-liquid extraction, accelerated solvent extraction and simultaneous distillation extraction (SDE)[7-9], *etc*. Due to the main advantages of simplifying experimental procedures, relatively low cost, saving organic solvents and high uptake, steam distillation extraction has been widely used for analyzing the volatile components of various foods[10,11].

In this study, we attempt to find out some volatile components in tea that could be used as an indicator for distinguishing oolong teas with similar appearance and flavor characteristics and we also hope to find some volatile compounds which may associate with producing area, processing technology and climate, *etc*. Our study will also provide a scientific role on the special flavor quality assessment of oolong teas.

EXPERIMENTAL

Tea samples: In this work, Tieguanyin collected from Fujian province and Dongding oolong tea collected from Taiwan province in China, these samples were produced in 2013.

Extraction of volatile compounds: The preparation of tea extract and chromatographic conditions for analyzing volatile compounds were developed and validated according to reported method[12-14]. Briefly, 20 g tea powder was weighed and extracted with 400 mL boiling water for 3 h by the Likens-Nickerson steam distillation extraction with 40 mL dichloroethane heated at 40 °C by using a water bath as the solvent. The dichloroethane was dehydrated with 5 g anhydrous sodium sulphate and then concentrated to 1 mL by using a rotary evaporator. Finally, the sample essential oils were stored at 4 °C for analysis.

GC-MS analysis: An Agilent 7890B GC sytem coupled with an Agilent 5977A mass selective detector (MSD) was used to perform the analysis (Agilent Technologies, Palo Alto, CA, USA). An Agilent HP-5MS capillary column (30 m × 0.25 mm inner diameter, 0.25 µm film thickness) was equipped and the helium (percentage purity > 99.999 %) flow rate was 1 mL/min. The injector temperature was 250 °C and 1 µL was injected with the injection port operated at a split ratio of 1:5. The GC oven temperature was held at 50 °C for 5 min and programmed from 50 to 210 °C at 3 °C/min, this temperature was held for 3 min and then increased to 230 °C at 15 °C/min. The mass spectrometer was operated in an electron-impact mode of 70 eV with a source temperature of 230 °C and a quadrupole set of 150 °C, the mass scan range was 30-500 atomic mass units (amu) and solvent delay time was 3 min.

Data analysis: Identifications of the peaks were made by searching National Institute of Standards and Technology (NIST) 08. L MS library (a match quality of 85 % minimum was used as a criterion). The relative percentages of the detected peaks were obtained by peak-area normalization and all relative response factors being taken as one.

RESULTS AND DISCUSSION

Analysis of volatile compounds between tieguanyin and dongding oolong tea: In total ion chromatogram (TIC) of tested tea samples, the identification of peaks was carried out by NIST searching. A total of 84 aroma compounds were identified in two kinds of oolong teas and their TICs were presented in Fig. 1 and the information of these compounds are tabulated in Table-1.

A total of 62 volatile compounds were identified in Tieguanyin oolong tea and jointly represented of 98.58 % of the total extracts. These identified compounds mainly including phytol (50.04 %), nerolidol (11.22 %), dihydrojasmone lactone (6.41 %), indole (4.64 %), benzylcarboxaldehyde (2.64 %), phenylethyl alcohol (2.45 %), perhydrofarnesyl acetone (2.42 %), methyl jasmonate (1.93 %), dihydroactinidiolide (1.85 %) and linalool (1.29 %), *etc*.

A total of 75 volatile compounds were identified in Tieguanyin oolong tea and jointly represented of 95.60 % of the total extracts. These identified compounds mainly including indole (11.32 %), nerolidol (10.27 %), 3,7-dimethylocta-1,5,7-triene-3-ol (7.15 %), linalool (5.38 %), benzylcarboxaldehyde (4.54 %), β-ionone (3.26 %), dihydroactinidiolide (2.94 %), hexanal (2.79 %), perhydrofarnesyl acetone (2.16 %) and benzyl cyanide (2.05 %), *etc*.

TABLE-1
GC-MS ANALYSIS RESULTS OF AROMA COMPONENTS IN THE TWO KINDS OF OOLONG TEAS

No	Retention time	Compounds	Match degree (%)	Relative percentage content (%)[a]	
				Tieguanyin	Dongding oolong
1	3.526	1-Pentanol	86	0.09	0.41
2	3.591	*(Z)*-2-Penten-1-ol	93	0.13	0.80
3	4.232	Hexanal	90	0.50	2.79
4	4.428	2-Methyltetrahydrofuran-3-one	87	-	0.56
5	4.636	N-Ethylpyrrole	87	-	0.31
6	4.879	2-Methylpyrazine	89	-	0.46
7	5.206	Furfural	91	0.09	1.59
8	5.865	*trans*-2-Hexenal	94	0.20	0.46
9	5.965	Leaf alcohol	86	0.08	-
10	7.342	2-Heptanone	87	-	0.13
11	7.746	Heptanal	93	0.09	0.39
12	8.084	2,5-Dimethyl pyrazine	87	-	0.27
13	8.150	2-Acetylfuran	85	-	0.97
14	10.292	Benzaldehyde	95	0.24	1.10
15	10.542	5-Methyl furfural	90	-	0.40
16	11.343	1-Octen-3-ol	87	0.07	0.29
17	11.598	2,3-Octandione	85	0.13	-
18	11.723	Heptanoic acid	86	0.12	1.14
19	11.883	2-Pentylfuran	88	-	0.75
20	12.144	*(E,E)*-2,4-Heptadienal	94	0.10	0.16
21	12.310	2-Ethyl-5-methylpyrazine	91	-	0.19
22	12.465	Octanal	90	0.09	-
23	13.604	Dipentene	95	-	0.18
24	13.907	Benzyl alcohol	97	0.36	0.64
25	14.352	Benzylcarboxaldehyde	94	2.64	4.54
26	14.649	1-Ethylpyrrole-2-carboxaldehyde	90	-	1.45

27	14.690	3,7-Dimethyl-1,3,6-octatriene	93	0.09	-
28	14.922	γ- Hexalactone	85	0.17	0.47
29	15.462	Acetyl benzene	90	0.07	0.21
30	15.842	Linalool oxide I	90	0.31	1.54
31	16.216	2-Ethyl-3,5-dimethylpyrazine	90	-	0.44
32	16.625	Linalool oxide II	90	0.22	1.17
33	16.934	*(E,E)*-3,5-Octadien-2-one	87	-	0.16
34	17.243	Linalool	97	1.29	5.38
35	17.462	3,7-Dimethylocta-1,5,7-triene-3-ol	86	0.89	7.15
36	17.783	Phenylethyl alcohol	94	2.45	1.24
37	19.077	Benzyl cyanide	96	0.57	2.05
38	20.554	Linalool oxide III	90	0.23	0.67
39	20.774	Linalool oxide IV	90	0.52	1.79
40	21.480	1,3,5,8-Undeeatetraene	96	0.16	1.15
41	21.569	α-Terpineol	86	0.45	1.40
42	21.712	Methyl salicylate	98	-	0.25
43	21.854	α-Methylbenzyl acetate	95	-	0.19
44	21.985	Safranal	98	-	0.37
45	22.388	Caprinaldehyde	90	-	0.26
46	22.994	2-Methyl-3-aminopropionitrile	85	0.32	0.87
47	26.312	Indole	95	4.64	11.32
48	27.410	*(E,E)*-2,4-Decadienal	90	-	0.17
49	28.899	1,1,6-Trimethyl-1,2-dihydronaphthalene	90	-	0.24
50	29.107	3,7-Dimethyl-6-octadienoic acid	90	0.07	0.46
51	29.754	Decanoic acid	85	0.12	0.31
52	30.330	3-Hexenyl hexanoate	85	0.30	-
53	30.371	β-Damascenone	98	-	0.77
54	30.538	Hexyl hexanoate	86	0.09	0.23
55	30.977	Jasmone	98	0.22	1.20
56	32.194	α-Ionone	95	0.09	0.49
57	32.366	Coumarin	87	0.18	0.84
58	33.286	Geranylacetone	91	0.23	0.64
59	34.591	β-Ionone	86	0.72	3.26
60	34.900	Dihydrojasmone lactone	96	6.41	1.51
61	34.953	δ-Decanolide	86	-	0.29
62	35.221	Pentadecane	91	-	0.29
63	35.553	Farnesene	87	0.60	0.62
64	36.200	Dihydroactinidiolide	96	1.85	2.94
65	37.743	Nerolidol	91	11.22	10.27
66	39.031	α-Naphthalenol	90	0.21	0.72
67	39.102	Hexadecane	98	0.17	0.51
68	40.639	2-Phenethyl hexanoate	86	0.30	-
69	40.889	Methyl jasmonate	99	1.93	0.69
70	42.806	Heptadecane	97	0.21	0.59
71	43.031	2,6,10,14-Tetramethylpentadecane	96	0.37	0.97
72	43.583	Geranylgeraniol	85	0.23	-
73	44.972	Benzyl benzoate	97	0.22	-
74	45.121	Paranaphtalene	94	0.13	0.28
75	46.320	Octadecane	95	0.08	0.37
76	46.652	2,6,10,14- Tetramethyl hexadecene	91	0.26	0.61
77	47.619	Caffeine	97	0.28	-
78	47.851	Perhydrofarnesyl acetone	98	2.42	2.16
79	50.261	Farnesyl acetone	90	0.33	0.49
80	50.551	Methyl hexadecanoate	99	0.22	1.37
81	51.222	Isophytol	90	0.31	0.22
82	55.763	Methyl linoleate	99	0.52	0.85
83	55.959	Methyl linolenate	99	0.94	1.73
84	56.451	Phytol	91	50.04	1.45

a. Not detected

The degree of fermentation could cause a change of volatile flavor components **1**. As the result, there were considerable amounts of alcohols such as phytol, nerolidol, phenylethyl alcohol, linalool in the Tieguanyin oolong tea. There were higher amounts of 3,7-dimethylocta-1,5,7-triene-3-ol, indole, linalool, jasmone, β-ionone detected in the Dongding oolong tea, whereas the Tieguanyin produced by light fermentation processing showed significantly lower amounts of volatile components. In addition, 22 volatile components including 2,5-dimethyl pyrazine, 2-acetylfuran, 2-methyltetrahydrofuran-

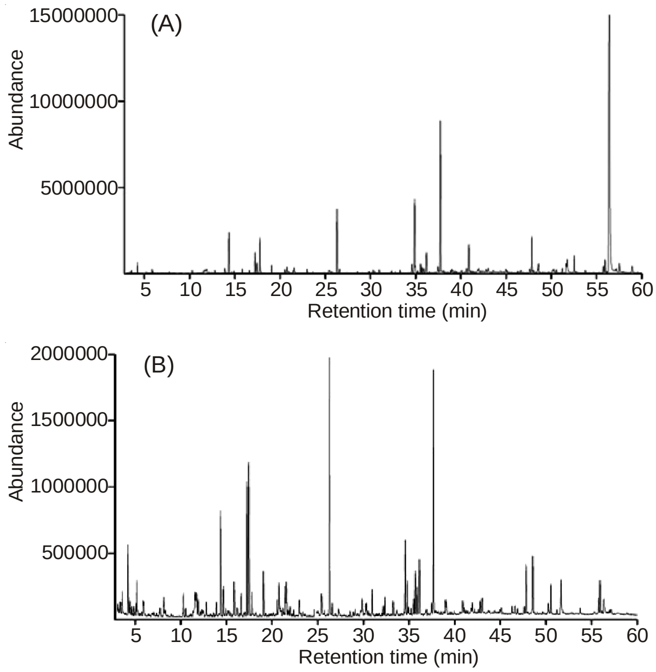

Fig. 1. GC-MS TIC of aroma components in the two kind of oolong teas (A: Tieguanyin oolong tea, B: Dongding oolong tea)

3-one, N-ethylpyrrole, 5-methyl furfural, 2-pentylfuran and methyl salicylate, *etc.* were only detected in the Dongding oolong tea. In all, the differences between the oolong teas might be resulted from different varieties, geographical regions and processing method, *etc*.

Analysis of chemical differences of aroma compounds in Tieguanyin and Dongding oolong teas: The aroma compounds comparison result in two different oolong teas was shown in Fig. 2. As can be seen in Fig. 2, there were great differences in the content of aroma components between the tested samples. The alcohols were the most abundant aroma components in the Tieguanyin oolong tea (69.10 %), while only 35.14 % was identified in the Dongding oolong tea. Meanwhile, the content of nitrogens in Dongding oolong tea (17.36 %) was higher than in the Tieguanyin oolong tea (5.81 %). The total contents of lactone in Tieguanyin oolong tea (8.43 %) were greater than those in Dongding oolong tea (5.21 %). The content of esters in the Tieguanyin oolong tea and the Dongding oolong tea was close and were identified 4.52 and 5.31 %, respectively. In addition, the result showed that the hydrocarbons, aldehydes and ketones existed at the lower contents in the Tieguanyin oolong tea than those in the Dongding oolong tea. The content of acids and furans were relatively lower in the two tested tea samples.

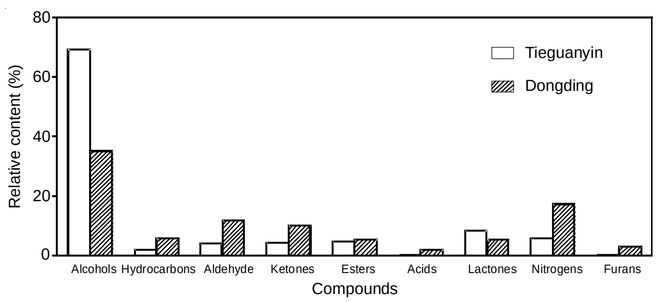

Fig. 3 Comparison of aroma components in two kinds of oolong tea

Although Tieguanyin and Dongding oolong tea have similar shape and taste characteristics, the results showed that these two kinds of tea existed notable differences on the content and the composition of main volatile components. This shows a certain degree of advantages of modern instrumental analysis and the defective of sensory evaluation. It is well known that presence of the innumerable compounds in tea and their multidimensional contribution in determining the final quality of tea[15], so it is remarkably important to understand of the chemical information existed in tea. In the further work, more samples and the combination with multivariate statistical methods will be applied to further explore the difference between these two kinds of oolong teas.

Conclusion

In this work, steam distillation extraction coupled with GC-MS was developed for the determination of the aroma components of Tieguanyin and Dongding oolong tea. By using the developed method, 84 volatile components were detected in the two tested oolong tea samples. The alcohols existed at the higher contents in the Tieguanyin oolong tea (69.10 %) than in the Dongding oolong tea (35.14 %). Whereas the content of nitrogens in Dongding oolong tea was nearly three folds than that in the Tieguanyin oolong tea. The hydrocarbons, aldehydes and ketones existed at the higher contents in the Dongding oolong tea than those in the Tieguanyin oolong tea.

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