Determination and difference analysis of aroma compounds in An-Tea with different aging time

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Abstract. The differences of volatile substances of 4 kinds of An-tea samples, made in 2007 (high-aging sample, HAS), 2012 (middle-aging sample, MAS), 2017 (low-aging sample, LAS) and 2018 (control check, CK) were studied by headspace solid phase microextraction combined with gas chromatography-mass spectrometry. A total of 64 compounds, including 13 ketones, 12 aldehydes, 4 alcohols, 8 esters, 2 phenols, 13 hydrocarbon compounds, 7 nitrogenous compounds, 2 ethers and 3 heterocyclic compounds, were identified. Among them, there were 34 compounds in HAS, 41 compounds in MAS, 39 compounds in LAS, and 31 compounds in CK, indicating that different kinds and contents of volatile aroma components contribute significantly to the quality of tea, which leads to the unique aroma quality of tea samples with different storage time. Linalool and β-ionone are the common aroma components of 4 kinds of An-tea, and play an important role in the formation of An-teas’ aroma. In addition, with the extension of storage time, the relative content of alcohols in An-tea decreased gradually, while the relative content of esters increased, which is probably due to the esterification reaction between alcohols and acids.

1. Introduction
An-tea, a sort of dark tea, is a semi-fermented compressed tea which its refining process goes somewhat between that of black tea and green tea. Its manufacturing process is similar to that of Liuan tea so it is called An-tea, and it also called “Qingcha” or “Soft-branch tea”. An-tea originated from Qimen County, Huizhou Government. In the local document Qichang Records of the Ming Dynasty, there is record of “Soft-branch tea”, which is about 300 years away from now[1-3]. The buds and leaves of An-tea are plump and rich in nutrient substances. The color of manufactured tea is dark brown and lustrous, and the tea soup shows bright orange-red, pure flavour and aromatic scent. An-tea is not only an excellent drink, but also regarded as best medicine. It is often applied as messenger drugs in Lingnan traditional Chinese medicine prescription, being honored as “sacred tea” in Guangdong Province, China and Southeast Asia[4, 5].

Aroma components contribute to tea’s unique flavor. Therefore, the aroma is one of the key factors for judging the tea quality. By now, over 700 volatile aroma components have been separated and identified from the tea[6], falling into four categories[7-10]: terpene derivatives, aromatic derivatives, aliphatic derivatives, and the nitrogenous, oxygenic and sulfuric heterocyclic compounds. Among these
volatile aroma components (VACs), not all of them are essential to the quality of the tea, only a small portion of the volatile elements produces the unique flavor of a tea, so they are called the key aromas[6,11].

Xu et al[12] extracted VACs from the fermented Pu-erh tea and non-fermented Pu-erh tea using Headspace-solid phase microextraction (HS-SPME) and simultaneous distillation extraction. And VACs were analysed and identified by gas chromatography olfactory and gas chromatography-mass spectrometry (GC/MS). The results showed that main aroma components in fermented Pu-erh tea are floral fragrance (33.01%), stale odor (25.24%) and smell of wood (11.17%). What’s more, alcohols (floral fragrance), methoxy phenols (stale odor) and ketone compounds (smell of wood or floral fragrance) played a critical role in determining the unique flavor of fermented Pu-erh tea. Whereas the floral scent (38.62%) and fruity odor (16.55%) were main VACs for non-fermented Pu-erh tea, which were closely related to its odor characteristics and sensation.

Chen et al[13] studied the volatile compounds of aging Pu-erh tea by HS-SPME and GC/MS and discovered that the main aroma components in aging Pu-erh tea are aromatic hydrocarbons and their derivatives as well as terpene compounds, followed by ester compounds and their derivatives. Yuan et al[14] extracted the VACs from 3 kinds of dark teas by distillation extraction, and the aroma compounds were analysed by GC/MS. The difference of VACs was compared and tea samples were judged for their sense. The results showed that there was apparent “arohid fluado-videver” in Fuzhuan tea, and the alkene-aldehyde compounds, such as (E, E)-2,4-hexadienal, was detected and played a positive role to odor. Cyan brick tea exuded a rich odor of Camphorwood, and that butylated hydroxytoluene and cedrol are vital components with special influence towards its odor. Dark brick tea contained little fragrance, probably due to the fact that most aroma, whose components are mainly such substances as phenol, alkane, alcohol and ester, is inverted or lost in the process of pile-fermentation. In general, the fresher the tea is, the higher the price is. But An-tea is different, which is valued for its “aging”. As long as it is well preserved, the more aging An-tea is, the more mellow it tastes. The saying is passed down only by word of mouth among tea lovers. However, there is few research on why An tea taste much better with longer aging time. Although the major kinds of teas have been studied extensively at home and abroad[15-19], An-tea, as a local characteristic tea of Qimen County, has rarely been studied. Therefore, aimed at different aging time, 4 An-tea samples were selected as objects. The changes of volatile components of different An-tea samples have been investigated using headspace solid-phase microextraction (HS-SPME) combined with gas chromatography mass spectrometry (GC/MS). This research established foundation to judge the quality and research the development and application of An-tea.

2. Materials and methods

2.1. Material
4 An-tea samples, collected from Jiangnanchun Tea Company in Anhui, China, were named high-aging sample (HAS, 2007), middle-aging sample (MAS, 2012), low-aging sample (LAS, 2017) and non-aging sample as control check (CK, 2018). All the solvents used in the experiment are commercially purchased analytical reagents and were purified by distillation prior to use. Table 1 lists the main equipments used in this experiment.

| Equipments                        | Type            | Manufacture                  |
|-----------------------------------|-----------------|------------------------------|
| Solid-phase microextraction fiber  | 75μm CAR/PDMS   | Supelco, America             |
| Gas chromatograph-mass            | 7890/5975C      | Agilent Technologies Co., Ltd|
| spectrometer                      |                 | Shimadzu Technology Co., Ltd |
| Electronic analytical balance     | AUY220          | Jintan Instrument manufacture|
| Thermostatic water bath           | HH-6            | Co., Ltd                     |
| Automatic ultra-pure water machine| Milli-Q         | Millipore, America           |

Table 1. The main equipments used in the experiment.
2.2. Procedure of the Experiment
About 4 g An-tea sample was placed in a special headspace bottle for solid phase microextraction, then 50 mL boiling ultra-pure water was added into the bottle. A manual injector with 75 μm CAR/PDMS solid phase microextraction fiber were slotted into a hole on the special headspace bottle. Subsequently, the sample was extracted in water bath at 60°C for 60 min to obtain the volatile components (VCs). After extraction, the fiber was inserted into the inject port of GC to desorpt 5 min at 250°C, then the VCs were analyzed with GC/MS. The GC/MS is equipped with a HP-5MS non-polar capillary column (30 m×0.25 mm × μm)[20]. The oven temperature was held at 40°C for 2 min, raised from 40°C to 85°C for 2 min at 5°C /min, and from 85°C to 110°C for 1 min at 2°C /min, and then from 110°C to 128°C for 1 min at 3°C /min, and from 128°C to 145°C for 2 min at 2°C /min, and then from 145°C to 230°C at 5°C /min, and finally held at 230°C for 8 min. Peak identification was based on the comparison of MS data and retention time with those of reference compounds and mass spectral library (NIST 08)[21-24]. Meanwhile, matching is greater than 90% as identified standard.

3. Results and Analysis

3.1. Identification and Comparison of the Aroma Components among An-Teas with Different aging time
64 aroma compounds (ACs) were identified in the 4 An-teas with different aging time, including 13 ketones, 12 aldehydes, 4 alcohols, 8 esters, 2 phenols, 13 hydrocarbons, 7 nitrogenous compounds and 5 other compounds. The relative content (RC, %) of ACs with over 3% are listed Table 2.

| Compounds                          | RC (%) | Compounds                          | RC (%) | Compounds                          | RC (%) |
|------------------------------------|--------|------------------------------------|--------|------------------------------------|--------|
| 4-Amino-2-methylphenol             | 12.38  | β-Ionone                           | 13.30  | Benzaldehyde                       | 6.81   |
| 3,7-Dimethyl-1,5,7-octatriene-3-ol | 10.30  | Linalool                           | 8.80   | 1,3-Dimethoxybenzene               | 5.04   |
| Linalool                           | 9.38   | 3,7-Dimethyl-1,5,7-octatriene-3-ol | 6.86   | β-Ionone                           | 4.92   |
| Methyl 2-hydroxybenzoate           | 7.51   | Benzaldehyde                       | 5.04   | Linalool                           | 4.24   |
| β-Ionone                           | 6.37   | Geranylacetone                     | 4.82   | 2,7-Dimethyl-9H-carbazole          | 4.21   |
| Benzonitrile                       | 4.05   | (E)-4-(2,6,6-trimethylcyclohexa-1,3-dien-1-yl) but-3-en-2-one | 3.87 | 3,7-Dimethyl-1,5,7-octatriene-3-ol | 3.97   |
| 2-Methoxy-4-methylaniline          | 3.87   | β-cyclocitral                      | 3.59   | Dihydro-β-irisone                  | 3.24   |
| Benzaldehyde                       | 3.11   | Safranal                           | 3.02   |                                   |        |

31 compounds were identified in CK. The compounds whose RC are over 3% include 4-amino-2-methylphenol (12.38%), 3,7-dimethyl-1,5,7-octatriene-3-ol (10.30%), linalool (9.38%), methyl 2-hydroxybenzoate (7.51%), beta-Ionone (6.37%), benzonitrile (4.05%), 2-methoxy-4-methylaniline
(3.87%), benzaldehyde (3.11%). 39 compounds were identified in LAS. The compounds whose RC are over 3% include beta-ionone (13.30%), linalool (8.80%), 3,7-Dimethyl-1,5,7-octatriene-3-ol (6.86%), benzaldehyde (5.04%), geranylacetone (4.82%), (E)-4-(2,6,6-trimethylcyclohexa-1,3-dien-1-yl)but-3-en-2-one (3.87%), beta-cyclocitrail (3.59%), safranal (3.02%). 41 compounds were identified in MAS. The compounds whose RC are over 3% include benzaldehyde (6.81%), 1,3-dimethoxybenzene (5.04%), beta-ionone (4.92%), linalool (4.24%), 2,7-dimethyl-9H-carbazole (4.21%), 3,7-dimethyl-1,5,7-octatriene-3-ol (3.97%), dihydro-beta-irisone (3.24%). 34 compounds were identified in HAS. The compounds whose RC are over 3% include phenyl acetate (15.28%), methyl salicylate (6.88%), linalool (6.85%), beta-ionone (5.27%), dihydroactinidiolide (4.29%), dihydrobeta-irisone (3.40%). As shown in Figure 1, there was significant difference between the component and RC among 4 An-teas.

![Figure 1. Comparison of RC and number of identified components of 4 An-teas.](image)

The RC of ketones is 19.29%, 21.09%, 31.22% and 18.06% in HAS, MAS, LAS and CK, respectively. 4 Samples shared 5 common ketones, including heptan-2-one, 6-methylhept-5-en-2-one, alpha-ionone, beta-ionone and geranylacetone. Meanwhile, the RC of beta-ionone is highest for ketones in 4 samples.

The RC of aldehydes is 17.72%, 20.00%, 21.35% and 8.30%, respectively. 4 Samples shared 5 common aldehydes, including 3-methylbutanal, benzaldehyde, 2,4-heptadienal, nonanal and safranal. Compared with the other 3 samples, CK has the least amount of aldehyde compounds. As the storage year goes by, the relative contents of aldehyde compounds rises obviously. Among the aldehyde compounds, Benzaldehyde has the highest level in MAS, LAS and CK, while in HAS, 2,4-heptadienal (2.76%) is at the top, followed closely by benzaldehyde (2.65%). Benzaldehyde and safranal are the main aldehydes in An-teas.

The RC of alcohols is 6.85%, 9.30%, 16.08% and 19.68%, respectively. In CK, 2 alcohols were identified, namely, linalool (9.38%) and 3,7-dimethyl-1,5,7-octatriene-3-ol (10.30%). In LAS, 3 alcohols were identified, which were linalool (8.80%), 3,7-Dimethyl-1,5,7-octatriene-3-ol (6.86%) and terpinen-4-ol (0.42%). 4 alcohols were identified in MAS, being linalool (4.24%), 3,7-dimethyl-1,5,7-octatriene-3-ol (3.97%), terpinen-4-ol (0.31%) and geraniol (0.78%), respectively. While in HAS, only 1 alcohol was found, that is, Linalool (8.61%). The result showed that, with the aging time being prolonged, the amount of alcohol compounds in An-tea tends to decline. Linalool was identified in all the 4 An-teas and its' RC was comparatively high. Therefore, it is speculated that linalool is the main alcohol compound in An-tea.

The RC of esters is 26.45%, 6.47%, 7.96% and 8.22%, respectively. Dihydroactinidiolide is the common compound in all An-teas. 2 ester compounds were identified in CK, between which the content of methyl salicylate (7.51%) is highest. And methyl salicylate was also found in HAS and LAS with high content, being 6.88% and 2.63%, respectively. In MAS, methyl salicylate wasn’t detected. 3 esters were identified in HAS, namely, phenyl acetate (15.28%), methyl salicylate (6.88%) and dihydroactinidiolide (4.29%), among which phenyl acetate was highest. But phenyl acetate wasn’t detected in the other 3 samples.

Few phenols were identified in 4 samples. No phenol was found in HAS and MAS. Only one was detected in LAS, being 4-amino-2-methylphenol (2.28%). And 4-amino-2-methyl-phenol and 2-methoxy-4-methylaniline were detected in CK, RCs of which were 12.38% and 3.87%. The RC of
hydrocarbons was 6.29%, 5.28%, 6.61% and 4.42%, respectively. Naphthalene was found in 4 samples. In CK, the main hydrocarbons were naphthalene (1.58%) and (+)-limonene (1.30%); Naphthalene (2.03%), D-Limonene (1.68%) and β-pinene (1.39%) occupy a dominant position in LAS. 4-isopropyl-1,1’-biphenyl (2.59%) and (+)-limonene (1.85%) being main in MAS. Limonene (2.55%) and naphthalene (1.35%) being main in HAS. It can be seen, naphthalene plays a leading role hydrocarbons.

The RC of nitrogen compounds is 1.48%, 7.16%, 1.26% and 4.69%, respectively. Two benzonitrile (4.05%) and 3, 5-dimethyl-9H-carbazole (0.64%) were detected in CK. 2-acetyl pyrrole (0.87%) and N-ethyl pyrrole (0.39%) were found in LAS. 2, 7-dimethyl-9H-carbazole (4.21%) and 3, 5-dimethyl-9H-carbazole (2.65%) were main nitrogen compounds in MAS. 2-acetyl pyrrole (0.59%), N-ethyl pyrrole (0.34%) and 1, 8-dimethyl-9H-carbazole (0.55%) are detected in HAS.

Some other chemical compounds, such as ethers and furans, were also found in the An-tea samples, but with relatively lower contents. Ethers, for example, take a percentage of 6.63 in MAS with 1,3-dimethoxybenzene taking the highest, up to 5.04%. However in LAS and CK no ethers were detected.

Figure 3 shows clearly that similar compounds tend to cluster together, mainly in the left quadrant. In addition, Qimen An-tea even contains 7 characteristic compounds, namely β-ionone, benzaldehyde, linalool, 3,7-dimethyl-1,5,7-octatriene-3-ol, methyl 2-hydroxybenzoate, phenyl acetate, and 4-amino-2-methylphenol. And phenylacetate is peculiar with a high content of 15.28% in HAS. While 4-amino-2-methylphenol and 3,7-Dimethyl-1,5,7-octatriene-3-ol were commonly found in newly-made teas, taking up to 12.38% and 10.30%, respectively. But 4-amino-2-methylphenol and 3,7-Dimethyl-1,5,7-octatriene-3-ol were gradually reduced to undetectable with aging time. It was inferred that these two compounds are the characteristic substances in non-aging or low-aging samples. β-ionone, linalool and benzaldehyde are the commonly compounds found in all the samples and the RC is 7.47%, 7.32% and

3.2. Principal Component Analysis

Principal component analysis (PCA) is the method applied in this study which simplifies data and reveals the relationship between the variables through dimension reduction[25,26]. As a multivariate analysis method, PCA has been widely used to identify volatile substances in tea and to construct quality evaluation model[27]. The PCA results of the 4 An-teas and the characteristic values of their principal components are shown in Table 3.

| Principal component | Eigenvalue | Contribution rate % | Cumulative contribution rate % |
|---------------------|------------|---------------------|--------------------------------|
| 1                   | 2.182      | 54.56               | 54.56                          |
| 2                   | 0.827      | 20.66               | 75.22                          |
| 3                   | 0.726      | 18.15               | 93.37                          |

As Table 3 exhibits, the contribution rate of Component 1, Component 2 and Component 3 is 54.56%, 20.66% and 18.15%, respectively, and the cumulative contribution rate reaches 93.37%. According to the extracting principal component, which contains over 85% information of PCA principle, it is sufficient to convince that the characteristics of the data can be explained by the first three principal components.

Component 1 mainly contains β-ionone, linalool, 3,7-dimethyl-1,5,7-octatriene-3-ol, benzaldehyde, methyl 2-hydroxybenzoate and 4-amino-2-methylphenol. Component 2 contains phenyl acetate, methyl 2-hydroxybenzoate, dihydroactinidiolide and limonene. Component 3 mainly contains benzaldehyde, 2,7-dimethyl-9H-carbazole, dihydro-β-irisone, 4-isopropyl-1,1’-biphenyl and 3,5-dimethyl-9H-carbazole. Therefore, it was inferred that such indexes as ketones, aldehydes, alcohols and esters can be used to evaluate the composition of volatile substances in Qimen An-tea.

Figure 2 was established by taking the eigenvectors as variables of the first and the second principal components among the 64 compounds. From the various positions of samplings in Figure 3, it can be seen clearly that similar compounds tend to cluster together, mainly in the left quadrant. In addition, Qimen An-tea even contains 7 characteristic compounds, namely β-ionone, benzaldehyde, linalool, 3,7-dimethyl-1,5,7-octatriene-3-ol, methyl 2-hydroxybenzoate, phenyl acetate and 4-amino-2-methylphenol. And phenylacetate is peculiar with a high content of 15.28% in HAS. While 4-amino-2-methylphenol and 3,7-Dimethyl-1,5,7-octatriene-3-ol were commonly found in newly-made teas, taking up to 12.38% and 10.30%, respectively. But 4-amino-2-methylphenol and 3,7-Dimethyl-1,5,7-octatriene-3-ol were gradually reduced to undetectable with aging time. It was inferred that these two compounds are the characteristic substances in non-aging or low-aging samples. β-ionone, linalool and benzaldehyde are the commonly compounds found in all the samples and the RC is 7.47%, 7.32% and
4.40%, respectively, indicating that these three substances are the main volatile substances in Qimen An-tea.

![Figure 2. The scatter plot of PCA about 64 VCs.](image)

4. Discussion

64 aroma compounds were identified from 4 An-teas with different aging time. 34 compounds were found in HAS, 41 in MAS, 39 in LAS and 31 in CK. 14 aroma compounds can be found in each An-tea sample, mainly including ketones, aldehydes, alcohols, esters and so on. These common compounds mix with each other to produce an improved fragrance, giving Qimen An-tea characteristic aroma. Each of An-tea has its own characteristic aroma compounds. With aging years going, it is clear that the sweet flavor (3,7-Dimethyl-1,5,7-octatriene-3-ol, octane-2,3-dione and 2-Acetylfuran and so on) gradually transforms into fruity and woody flavor (dihydro-β-ionone, heptanal, Limonene and α-terpinene, and so on) in An-tea.

The aroma components are mostly involved in ketones, aldehydes, alcohols and phenols in CK, most of which are β-ionone (6.37%), linalool (9.38%), 3,7-Dimethyl-1,5,7-octatriene-3-ol (10.30%), methyl 2-hydroxybenzoate (7.51%) and 4-amino-2-methylphenol (12.38%), accounting for 45.94% of the total aroma. The aroma components are mostly involved in ketones, aldehydes and alcohols in LAS. The key aroma compounds are mainly determined by β-ionone (13.30%), geranylacetone (4.82%), benzaldehyde (5.04%), linalool (8.80%) and 3,7-dimethyl-1,5,7-octatriene-3-ol (6.86%), taking up 38.82% of the total aroma. The aroma components are mostly involved in ketones, aldehydes, alcohols and ethers in MAS. The aroma compounds are mainly β-ionone (4.92%), benzaldehyde (6.81%), linalool (4.24%) and 1,3-dimethoxybenzene (5.04%), accounting for 21.01% of the total aroma. The aroma components are mostly involved in ketones, aldehydes, alcohols and esters in HAS, taking a percentage of 43.98 of the total aroma. These aroma compounds are mostly β-ionone (5.27%), benzaldehyde (2.65%), 2,4-heptadienial (2.76%), linalool (6.85%), phenyl acetate (15.28%), methyl 2-hydroxybenzoate (6.88%) and dihydroactinidiolide (4.29%) and so on.

Linalool, with a flavor of flower and fruit, is found to be a common compound, taking a large amount of the content in 4 samples. The research[28-30] showed that linalool was the key aroma compounds of different tea. Benzaldehyde was of a smell of almond. The smell of violet and wood were granted for β-ionone and geranylacetone, having great influence on tea aroma[9]. The Study[31] showed that β-ionone, as volatile compound, was rich in Pu-erh tea. Geranylacetone was the main volatile comound in Fuzhuan tea[32]. Zheng et al[17] found that the pure aroma of Kangzhuan tea was mainly due to ketones and aldehydes, including (E)-geranylacetone and β-ionone. Di et al[33] found that (E, E)-2,4-heptadienal and benzaldehyde were the key aroma of An’ji Black tea. And (E, E)-2,4-heptadienial could adjust the tea’s sweet fragrance while benzaldehyde could intensify its floral aroma.

Compared with the other three tea samples, the 2018 An-tea has a relatively lower content of ketones. However, the content of ketones increased in the 2017 An-tea. This happens probably because alcohols in An-tea are oxidized to ketones after one year of storage. With the increase of storage time, the ketones continue to be oxidized, presenting the tendency of decrease. One can draw a conclusion that alcohols
in An-tea tends to decrease with the aging time prolonging. The 2007 An-tea owns the longest aging time. In this tea, the esters, such as phenyl acetate, methyl 2-hydroxybenzoate and dihydroactinidiolide, are in a relatively higher content. It may be due to a series of esterification reactions between acids and alcohols in the tea during the long storage, which is consistent with detected compounds. The PCA shows that β-ionone, benzaldehyde, linalool, 3,7-dimethyl-1,5,7-octatriene-3-ol, methyl 2-hydroxybenzoate, phenyl acetate and 4-amino-2-methylphenol are the volatile characteristic compounds, which can distinguish Qimen An-tea from other teas.

Tea aroma is caused by the different aroma compounds and their varied contents. Meanwhile, aroma is affected by many other factors, such as the type of tea tree, natural environment and the processing technology. Until now, An-tea is paid few attention, much less of its aroma components. Therefore it is significant to identify and compare the aroma compounds of An-tea with different aging time. Aimed at enriching the tea field and providing the theoretical basis for the further development and application of An-tea, it is expected to further researches related to An-tea.

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References
[1] Zhan, S.L. (2017) Exploring the origin of An-tea. Journal of Tea Business, 39: 36-37.
[2] Han, A.L., Li, L.X., Xu, X.Q. (2016) Quality and chemical com-position analysis of An-tea. Journal of Tea Business, 38: 79-83.
[3] Geng, Q.M. (2014) Technology and quality characteristics of traditional historic An-Tea. Anhui Agricultural Science Bulletin, 20: 169-170.
[4] Huang, J.Q., Wang, Y.J. (2009) Study on the quality and chemical constituents of An-tea. China Tea Processing, 02: 29-31.
[5] Shu, Q.L. (1991) A brief introduction to "An-tea". Agricultural Archaeology, 02: 186-188.
[6] Wang, M.Q., Zhu, Y., Zhang, Y., Shi, J., Lin, Z., Lv, H.P. (Food Science), 2019. Recent Research on the Key Aroma Compounds of Volatile Compounds in Tea. http://kns.cnki.net/kcms/detail/11.2206.TS.20181213.1450.076.html.
[7] Shi, L.T. Jiang, H.Y., Zhang, J.Y., Wang, W.W., Su, W. (2018) Progress on components and detection technology of tea aroma. Science and Technology of Food Industry, 39: 347-351.
[8] Wang, Z.N. (1988) In: ZeNong,W.(Eds.), Tea Biochemistry. Agriculture Press, Beijing. 157-161.
[9] Wan, X.C. (2003) Tea Biochemistry. China Agriculture Press, Beijing.
[10] Lin, J. (2013) Analysis of aromatic profile and application on product quality authentification of tea (Camellia sinensis). Hangzhou: Zhejiang University.
[11] Mao, S.H., Yue, C.N., Xiao, L., Tong, H.R. (2018) Key aroma components of tea and their contribution to tea sensory quality. Health Medicine Research and Practice, 15: 4-9, 2.
[12] Xu, Y.Q., Wang, C., Li, C.W., Liu, S.H., Zhang, C.X., Li, L.W., Jiang, D.H. (2016) Characterization of aroma-active compounds of Pu-erh tea by headspace solid-phase microextraction (HS-SPME) and simultaneous distillation-extraction (SDE) coupled with GC-Olfactometry and GC-MS. Food Analytical Methods, 9: 1188-1198.
[13] Chen, M.C., Chen, Z., Shi, H., Liu, B., Pan, Z.Z., Zhu, Y.J. (2014) Analysis on characteristic flavor components of aged Pu-erh tea. Journal of Tea Science, 34: 45-54.
[14] Yuan, S.S., Bai, Z., Huang, Y.H., Lai, X.F., Wu, C.L., Zhao, W.F. (2014) Analysis of aroma components in three kinds of dark tea. Food Science, 35: 252-256.
[15] Wu, Y.S., Lv, S.D., Wang, C., Gao, X.M., Li, J.B., Meng Q.X. (2016) Comparative analysis of volatiles difference of Yunnan sun-dried Pu-erh green tea from different tea mountains: Jingmai and Wuliang mountain by chemical fingerprint similarity combined with principal component analysis and cluster analysis. Chemistry Central Journal, 10: 11.
[16] Shi, J., Wang, L., Ma, C.Y., Lv, H.P., Chen, Z.M., Lin, Z. (2014) Aroma changes of black tea prepared from methyl jasmonate treated tea plants. Journal of Zhejiang University-SCIENCE B (Biomedicine & Biotechnology), 15: 313-321.

[17] Zheng, P.C., Liu, P.P., Wang, S.P., Teng, J., Feng, L., Gong, Z.M. (2018) Comparative analysis of the aroma components in five kinds of dark tea. Science and Technology of Food Industry, 39: 82-86,143.

[18] Peng, W., Wang, L., Qian, Y., Chen, T., Dai, B., Feng, B., Wang, B.J. (2017) Discrimination of unfermented Pu’er Tea aroma of different years based on electronic nose. Agricultural Research, 6: 436-442.

[19] Ma, L.J., Qiao, Y., Du, L.P., Li, Y.F., Huang, S.Y., Liu, F., Xiao, D.G. (2017) Evaluation and optimization of a superior extraction method for the characterization of the volatile profile of Black tea by HS-SPME/GC-MS. Food Analytical Methods, 10: 2481-2489.

[20] Gan, Z.T., Yao, T., She, X.S., Zou, Z.L., Wang, S.Q., Tang, M., Han, Y.F (2019) Fractional extraction and identification of the chemical components from Cordyceps cicadae product. Mycosystema, 38: 1149-1164

[21] Liu, P.P., Zheng, P.C., Gong, Z.M., Wang, S.P., Teng, J., Gao, S.W., Wang, X.P., Ye, F., Zheng, L. (2017) Analysis of aroma components in Qingzhuan dark tea. Food Science, 38: 164-170.

[22] Lv, S., Wu, Y., Li, C., Xu, Y., Liu, L., Meng, Q. (2014) Comparative analysis of Pu-erh and Fuzhuan teas by fully automatic headspace solid-phase microextraction coupled with gas chromatography–mass spectrometry and chemometric methods. Journal of Agricultural and Food Chemistry, 62: 1810–1818.

[23] Lv, H. P., Zhong, Q. S., Lin, Z., Wang, L., Tan, J. F., Guo, L. (2012) Aroma characterisation of Pu-erh tea using headspace-solid phase microextraction combined with GC/MS and GC–olfactometry. Food Chemistry, 130: 1074–1081.

[24] Lin, J., Dai, Y., Guo, Y., Xu, H., Wang, X. (2012) Volatile profile analysis and quality prediction of Longjing tea (Camellia sinensis) by HS-SPME/GC-MS. Journal of Zhejiang University-SCIENCE B (Biomedicine & Biotechnology), 13: 972–980.

[25] Liu, Z. (2011) SPSS Statistical Analysis and Application. Publishing House of Electronics Industry, Beijing.

[26] Guo, Q., Wu, W., Massart, D.L., Boucon, C., De Jong, S. (2002) Feature selection in principal component analysis of analytical data. Chemometrics and Intelligent Laboratory Systems, 61: 123-132.

[27] Zhang, L., Liu, W.J., Liu, T.F., Dong, M.H., Yu, Z.F. (2018) Modeling for quality evaluation of Dongting Biluochun tea based on principal component analysis. Food Research and Development, 39: 15-22.

[28] Zhao, C.R. (2010) Studies on the characteristic aroma components of Qimen black tea. Hefei: Anhui Agricultural University.

[29] Liu, L., Tong, H.R. (2010) The analysis on characteristic flavor components of Pu-erh tea. Chongqing: Southwest University.

[30] Chen, M.C., Liu, X.G., Zhu, Y.J., Pan, Z.Z., Zhang, H.F., Liu, B. (2016) Determination of aroma components of aged Pu-erh tea with different storage years based on solid phase microextraction-gas chromatography-mass spectrometry. Journal of Food Safety and Quality. 7:2396-2414.

[31] Liang, Y.R., Zhang, L.Y., Lu, J.J. (2005) A study on chemical estimation of Pu-erh tea quality. Sci Food Agric, 85: 381-390.

[32] Yan, H.F., Wang, M.L., Bai, X.Z., Zhu, S.H., Dai, H., Li, Y.J. (2014) Analysis of aroma composition in Hunan Fuzhuan tea by solid-phase microextraction combined with Gas Chromatography-Time of Flight-Mass spectrometry. Food Science, 35:176-180.

[33] Di, D.R. (2014) Analysis of physicochemical composition of “Anji black tea” and identification its aroma-active components. Zhejiang: Zhejiang A & F University.