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Characterization of key odorants in 'Baimaocha' black teas from different regions

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ABSTRACT

'Baimmaocha' is a distinctive resource for production of high-quality black tea, and its processed black tea has unique aroma characteristics. 190 volatile compounds were identified by comprehensive two-dimensional gas chromatography–olfactometry-quadrupole-time-of-flight mass spectrometry(GC × GC-O-Q-TOMS), and among them 23 compounds were recognized as key odorants contributing to forming different aroma characteristics in 'Baimaocha' black teas of Rucheng, Renhua, and Lingyun (RCBT, RHBT, LYBT). The odor activity value coupled with GC-O showed that methyl salicylate (RCBT), geraniol (RHBT), *trans-β*-ionone and benzeneacetaldehyde (LYBT) might be the most definitive aroma compounds identified from their respective regions. Furthermore, PLS analysis revealed three odorants as significant contributors to floral characteristic, four odorants related to fruity attribute, four odorants linked to fresh attribute, and three odorants associated with roasted attribute. These results provide novel insights into sensory evaluation and chemical substances of 'Baimaocha' black tea and provide a theoretical basis for controlling and enhancement tea aroma quality.

1. Introduction

Tea is widely popular as a non-alcoholic beverage for its unique flavor and potential health benefits(Wang et al., 2022; Zhang et al., 2020). Based on the processing technology and quality characteristics, tea can be divided into six categories, of which production and consumption of black tea accounts for the main part (Zhang et al., 2019). In recent years, with the change in people's living standards and the improvement of tea quality requirements, high-grade and specialty black tea has been loved by consumers. 'Baimaocha' is known for its very abundant silver trichomes located on the underside of the leaves, mainly including Rucheng Baimaocha (*Camellia pubescens*), Renhua Baimaocha (*Camellia pubilimba*), and Lingyun Baimaocha (*Camellia pubilimba*), which has been identified as a distinctive resource for the production of high-quality black tea (Chen et al., 2010; Chen et al., 2023; Zhong et al., 2022; Meng et al., 2018; Yang et al., 2020). Studies have shown that tea trichomes contain some high content of volatiles, flavonols and amino acids compared to the trichome-removed leaves, which contribute to the formation of the aroma and flavor properties of tea (Li et al., 2020; Zhu et al., 2017; Wang et al., 2020). 'Baimaocha' black teas processed with these are full of golden hairs and exhibit unique flavor characteristics, which are widely welcomed by consumers. However, due to differences in the genetic basis of the tea plant, as well as factors such as climate, soil stress, and minerals, black teas from different regions have unique aroma characteristics(Kang et al., 2019; Wu et al., 2019; Zhou et al., 2022; Ge et al., 2024; Peng et al., 2021; Yun et al., 2021). Among them, 'Rucheng Baimaocha' black tea (RCBT) has a unique and robust floral flavor and is produced in Rucheng County of Hunan province. 'Renhua Baimaocha' black tea (RHBT) prepared in Renhua County of Guangdong province has a high and persistent rose fragrance (Wu et al., 2019; wang et al., 2012). Different from RCBT and RHBT, 'Lingyun Baimaocha' black tea (LYBT) is produced Lingyun

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County of Guangxi province and has an obvious sweet attribute (Yang et al., 2020; Yang et al., 2023). Although sensory evaluation can be used to distinguish the aroma types of 'Baimaocha' black tea, and some progress has been made in volatiles research. However, there have been few comprehensive studies and comparisons of the key aroma components that contribute to the typical flavors of 'Baimaocha' black tea.

As a comprehensive reflection of many complex volatile compounds, aroma, plays a significant role in assessing tea quality and influencing consumer preferences (Su et al., 2022). With different types of aromatic compounds and their content ratios, the aroma types and characteristic components of black tea from different origins have specific differences, resulting in the flavor of black tea with prominent regional characteristics(Wang et al., 2022). Previously, the flavor characteristics of black tea products were evaluated mainly through the traditional sensory evaluation, which required high experience and physical condition of the assessors (Zheng et al., 2023). With the continuous development of various instruments, more than 600 volatiles have been isolated and identified from black tea using gas chromatography-mass spectrometry (GC–MS). However, only a few of them are key aroma components and contribute to the overall aroma. The utilization of odor active value (OAV) in conjunction with gas chromatography–olfactometry (GC-O) is a good way to identify key odorants for aroma characterization of food products such as tea (Chen et al., 2020;Wang et al., 2020; Zheng et al., 2023). In addition, the recently developed comprehensive two-dimensional gas chromatography–olfactometry-quadrupole-time-of-flight mass spectrometry (GC × GC-O-Q-TOFMS) can obtain more comprehensive aroma profile and more accurate olfactory datas compared with GC–MS, which has been gradually applied to food flavor research(Kang et al., 2019; Wang et al., 2021; Yang et al., 2021).

Quantitative descriptive analysis (QDA) is utilized for sensory evaluation of food flavor. It provides flavor descriptions and intensity assessments to discern differences between samples, and is now widely used in tea flavor analysis(Zheng et al., 2023). It was reported that quality differences in Hunan Fu brick tea could be discerned by several key flavor attributes, including fermented, smoky, floral, sweet (fruity), and bitter (Li et al., 2019). Additionally, four different roasting degrees and three different tenderness of large-leaf yellow tea were distinguished by 16 aroma descriptors, respectively (Dai et al., 2022). Therefore, combining chemical information from instrumental analyses and sensory analyses is a effective approach to determine the differences in aroma substances among food products sourced from distinct regions. However, limited reports have been conducted to establish the correlation between aroma attributes and volatiles in black tea from different regions.

The study aimed to find differences in the aroma components and identify the key odorants of the aroma characteristics of 'Baimaocha' black tea from different regions through GC × GC-O-Q-TOFMS and sensory analysis. This study will contribute to a comprehensive understanding of the material basis of the aroma of 'Baimaocha' black tea from different regions and provide a theoretical basis for controlling and enhancing the aroma quality of 'Baimaocha' black tea.

2. Materials and methods

2.1. Chemicals and tea sample

Ethanol (chromatographic alcohol) was obtained from Sinopharm Chemical Reagents Ltd. (Shanghai, China). Ethyl decanoate (internal standard) and *n*-alkanes(C7-C25) were provided by Sigma-Aldrich Co., Ltd (St. Louis, Missouri, USA).

A total of 17 'Baimaocha' black tea samples, including 6 RCBT samples, 6 RHBT samples, and 5 LYBT samples, were obtained from local markets in China in this experimental (Table S1). These samples were processed in 2021 or 2022 from the fresh leaves of one bud and two leaves, and identified by the Tea Research Institute of Hunan Agricultural University. All tea samples were stored in their respective bags at a temperature of −20 °C in a refrigerator for subsequent analysis.

2.2. Sensory evaluation

Sensory evaluation experiments were performed under the approval of the Ethics Committee of Hunan Agricultural University(approval number, 2023–138, Changsha, Hunan, China). The sensory assessment was carried out by seven trained evaluators (three men and four women, aged 25–30 years, from Tea Research Institute of Hunan Agricultural University) in accordance with Chinese national standard (GB/T 23776–2018). Participants were given study details in writing and signed a consent form to join. A sample of 3 g of black tea was brewed in 150 mL of boiling water for 5 min, and the aroma quality of the tea was assessed by seven experts after pouring out the tea broth. A QDA with a 5-point hedonic scale (0 representing non-existen,3 medium intensity and 5 very strong) was used to assess the sensory attributes of tea. Based on the aroma attributes of samples, the evaluators selected 5 aroma common quality descriptors that most accurately captured the samples. The scores for sensory attributes were determined by calculating the the average scores from seven panelists.

2.3. Extraction and identification of volatile compounds

The extraction of volatile compounds in the black tea samples was carried out by headspace solid-phase microextraction (HS-SPME), and the extraction conditions were basically the same as those of our previous method with some slight adjustments (Chen et al., 2023). First, each sample was initially homogenized and ground to powder. 0.5 g of tea powder was weighed and placed into a 20 mL headspace vials. Then, a rotor was added, followed by 5.0 mL of boiling water and 10.0 μL of decanoic acid ethyl ester (8.63 mg/L) as an internal standard. The vials were immediately sealed and held for 10 min in a shaker at 80 ℃. The 85 μm carboxen/polydimethylsiloxane (CAR/PDMS) coating fiber (Sigma-Aldrich Trading Co., Ltd, Shanghai) was pushed into headspace of the sample and cubated for at 200 r/min and 80 ∘C for 30 min. After the extraction, the fiber head was inserted into the GC inlet for a 10 min desorption process 250 °C, after which data collection was performed. Each sample underwent three times.

2.4. GC × GC-O-Q-TOF-MS conditions

A setup consisting of an agilent GC × GC-Q-TOF-MS (Agilent, Santa Clara, CA, USA) instrument combined with an olfactory detection port (Sniffer 9100; Brechbühler, Schlieren, Switzerland) was used to detect volatile compounds in black tea. HP-5MS (37.69 m × 250 μm × 0.25 μm, Agilent Technologies, USA) and DB-17MS (2.89 m × 180 μm × 0.18 μm; Agilent Technologies, USA) were used as one-dimensional and two-dimensional column. High purity helium (99.99 %) was used at a flow rate of 1.1 mL/min. Both columns are placed in the same GC oven. The split ratio was 10:1. The GC column was initially set at 40 °C and maintained for 1 min. It was then ramped up to 180 °C at a rate of 4 °C/min, further increased to 250 °C at a rate of 20 °C/min, and finally maintained for 1 min. The solid-state modulator SSM1800 (J&X Technologies Co., Ltd, China) was used between the two columns to facilitate heating and cooling, operating with a modulation period of 4 s. The mass spectrometer parameters were as follow: 70 eV electron ionization energy with mass scan range from 45 to 500, quadrupole temperature of 150 ∘C, and ion source temperature of 200 ∘C.

In the 2D analysis mode, an additional helium flow was introduced from the first column, and this helium flow was mixed and split into two parts in a 1:1 ratio. One part of the sample was directed towards the ODP through a four-way microfluidic device, while the other portion passed through SSM and the second column into the mass spectrometer detector. This mode allowed for two-dimensional GC–MS analysis of samples, enabling olfactory testing of odor-active substance separated in the first dimension.

The GC-O analysis of aroma-active compounds was conducted with the participation of three panelists. All panelists are tea tasters with over 5 years of experience in sensory evaluation. Prior to the formal GC-O analysis, the team members underwent rigorous training over 90 h to familiarize themselves with various smells, different standards, and varying concentrations. During the olfactory assessment, team members documented the perceived odor information, which include odor descriptors, retention time, and intensity values. The intensities of the aroma-active substances were assessed on a scale of 1 to 5 by a panel of testers(1 was very weak, 3 was moderate, and 5 was extremely strong). The average aroma intensity value was computed based on the ratings provided by the corresponding panelists.

2.5. Identificate and quantitative analysis of volatile compounds

The data obtained from GC × GC-Q-TOFMS were processed and analyzed using Canvas software (version 5.2.0.25117). The volatiles compounds underwent qualitative processing by retention index (RI) and matching mass spectra in the standard NIST 20 library. RIs were calculated by *n*-alkane line(C7-C25) for each compound under the same chromatographic conditions. Based on our previous method with minor adjustments, volatiles with forward match > 700, reverse match > 800, RI deviation < 15 were retained(Chen et al., 2023). Quantification was calculated in ng/g by comparing their peak areas with peak area of ethyl caprate. According to the ratio of the concentrations of volatile compound in the tea sample to odor threshold value in the water, odor activity values of potential odorants were calculated. OTs for volatile components in the water were sourced from existing literature or website.

2.6. Statistical analysis

The analyses were repeated at least 3 times, and results were expressed as mean ± standard deviation (SD). Statistical significance of difference was determined using the Duncan's multiple tests of one-way ANOVA by SPSS22 (version 22.0, SPSS Inc., Chicago, IL, USA), and $p < 0.05$ was considered statistically significant. Principal component analysis (PCA), partial least squares–discriminant analysis (PLS–DA), hierarchical cluster analysis (HCA), and partial least squares analysis (PLS) were conducted using SIMCAP + 12.0 software (Umea, Sweden). Originpro (OriginLab Corporation, MA, USA) was employed for generating the spider plot, while TBtools (https://github.com/CJ-Chen/TBtools) utilized for creating the heatmaps.

3. Results and discussion

3.1. Sensory analysis

Representative appearance and infusion of 'Baimaocha' black tea from three regions were shown in Fig. 1A. It was very obvious that black tea of three regions were easily distinguished by their appearance and infusion. Then, sensory evaluation was used to select the aroma characteristics of black tea from three different regions. Five representative aroma attributes were recorded, including floral, fruity, sweet, roasted, and fresh, and the results were plotted in Fig. 1B. It was evident that similar distributions of aroma attribute intensity were observed in the RCBT and RHBT samples, while significant differences were observed in the RCBT/RHBT and LYBT samples. The differences may be due to the geographic proximity of RCBT and RHBT, which grow under similar climatic, environmental, and soil conditions, and are distant from the

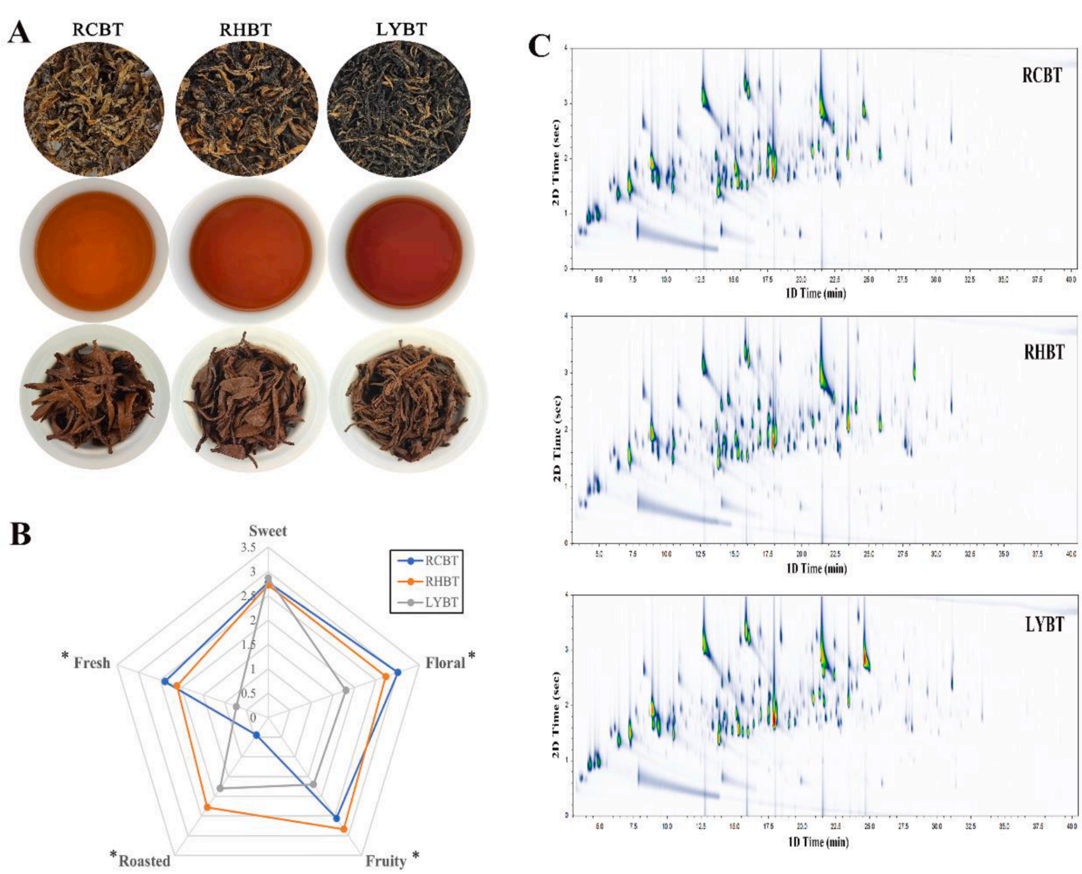

Fig. 1. Representative appearance and infusion of black tea from three regions(A); Spider plot of black tea aroma characters (B); The representative total ion chromatography (TIC) plots of black tea from three regions in 2D mode. (RCBT: 'Rucheng Baimaocha' black tea, RHBT: 'Renhua Baimaocha' black tea, LYBT: 'Lingyun Baimaocha' black tea).

origin of LYBT. Specifically, RCBT and RHBT samples both exhibited high intensity floral, fruity and sweet attributes. However, RHBT had a strong roasted flavor, which may be related to its higher temperature during drying. In addition, LYBT had a high sweet attribute and the other aroma attributes were of about the same intensity. Moreover, it was noteworthy that the fresh attribute intensity of RCBT and RHBT is significantly higher than that of LYBT.

3.2. Identification of volatile components in 'Baimaocha' black tea

The volatile compounds in 'Baimaocha' black tea samples from three regions were analyzed by GC × GC-Q-TOFMS, and representative chromatograms of black tea from each region were shown in Fig. 1C. Although the volatile compositions of black tea samples were similar, there were significant differences in some peaks, indicating that the volatility of the black tea samples from different regions varied greatly. 190 compounds were identified as common volatile compounds after comparison of MS spectrum and retention index (RI) among the collected 17 black tea samples (Table S2). These compounds can be categorized into 10 groups according to their chemical structure, including 44 aldehydes, 26 alcohols, 29 esters, 34 alkenes, 15 oxygen heterocyclic compounds, 21 ketones,10 aromatic hydrocarbons, 8 alkanes, 2 pyrrole derivatives, 1 acid. In addition, we explored the proportion of different types of volatile components in black tea from three regions based on the volatiles concentration. As shown in Fig. 2A-C, aldehydes, alcohols, esters, and ketones were predominant volatile components of 'Baimaocha' black tea, which account for more than 70 % of the identified substances. Previous studies have shown that aldehydes, alcohols and aldehydes may be the main contributors to the aroma formation of Chinese congou black tea, which is consistent with our current findings(Kang et al., 2019; Xiao et al., 2017). Notably, the content of aldehydes (average of 28.95 %) and alcohols (average of 25.35 %) was the highest in black tea in the three regions, followed by ketones, and alkenes. Aldehydes have the highest proportion of volatiles in most black teas, which primarily originate from lipid oxidation and strecker degradation(Ho et al., 2015; Zhai et al., 2022). Alcohols in tea, including straight and branched chain alcohols from Strecker's aldehyde reduction or the hydrolysis of glycoside precursors, enhance the variety of tea aroma and significantly impact the overall aroma quality of tea (Ho et al., 2015; Zhai et al., 2022). The ketone content was higher in RHBT(8.29 %) compared to RCBT(5.62 %) and LYBT(6.86 %), with 6-methyl-5-hepten-2-one, 2-heptanone, (*E*, *E*)-3,5-octadien-2-one, *trans*-β-ionone, (*Z*)-jasmone, α-ionone, acetophenone being the most abundant volatile compounds.

Furthermore, the top 10 volatile compounds and their corresponding proportion were shown in Fig. 3D-F. Methyl salicylate showed the most abundant proportion in RCBT(12.01 %) and RHBT(11.86 %), followed by linalool, and benzaldehyde. However, linalool (9.46 %) had the highest concentrations, followed by methyl salicylate (9.36 %) and benzaldehyde (7.01 %) in LYBT. Methyl salicylate is a characteristic component of black tea with a green and minty aroma, which may be associated with the stronger fresh attribute of RCBT and RHBT due to its highest concentration. Interestingly, it was also considered to be the key characteristic component in Ceylon black tea with a mint flavor (Kang et al., 2019). Linalool and geraniol are the most common among alcohols, and they were identified as key aroma-active compounds in the world's four most-known black teas, including Assam, Darjeeling, Keemun, and Ceylon black teas (Zhai et al., 2022; Kang et al., 2019). Benzaldehyde with nutty attribute was the third most abundant compound common to all three black tea origins, and it was recognized as the key aroma active compounds in Assam, Keemun, and Ceylon black tea(Kang et al., 2019). Meanwhile, some fatty acid derivatives including (*E*)-2-hexenal, hexanal, (*E*)-2-hexen-1-ol, have also been reported to be key odorants of black tea (Xiao et al., 2017). In addition, the content of anethole with sweet attribute was significantly higher in LYBT(5.73 %) than in RCBT(0.09 %) and RHBT(0.37 %), suggesting which have an important influence on the aroma formation of LYBT. In conclusion, the above results indicated that the proportion of dominant volatile compounds in the ''Baimaocha' black tea samples from three regions has a large difference.

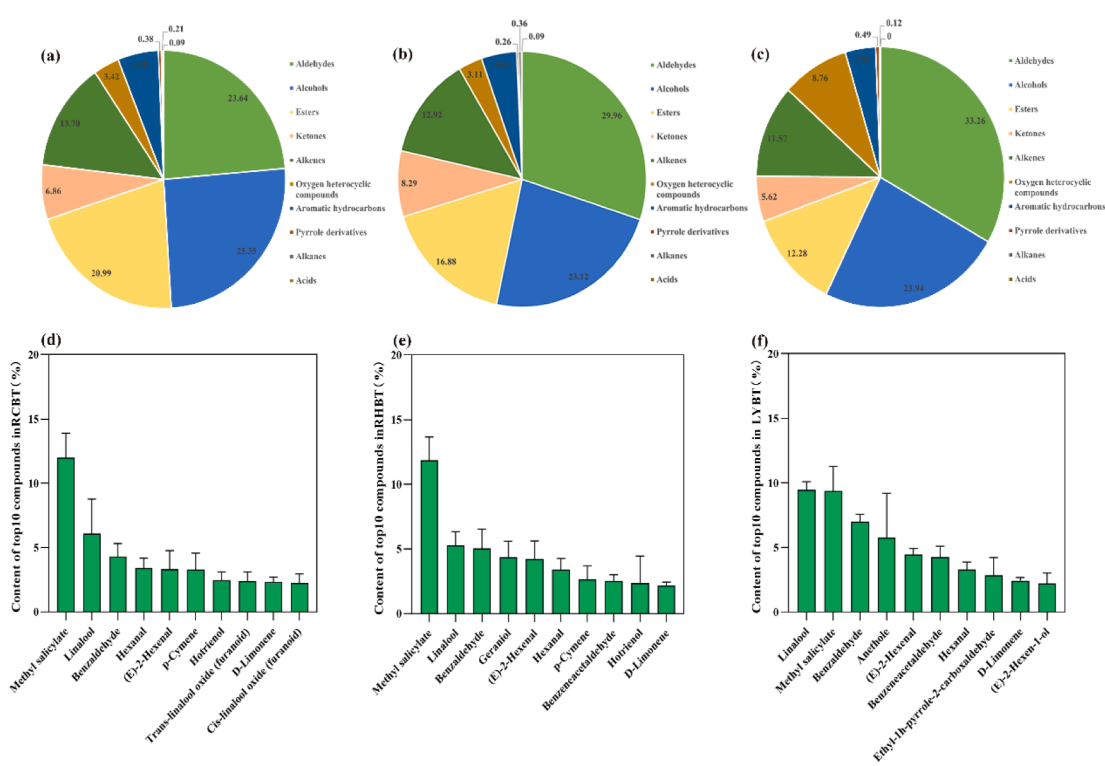

Fig. 2. The content distribution of different types of volatile compounds in 'Baimaocha' black tea(A-C); The contents of top10 compounds in 'Baimaocha' black tea of three regions (D-F).

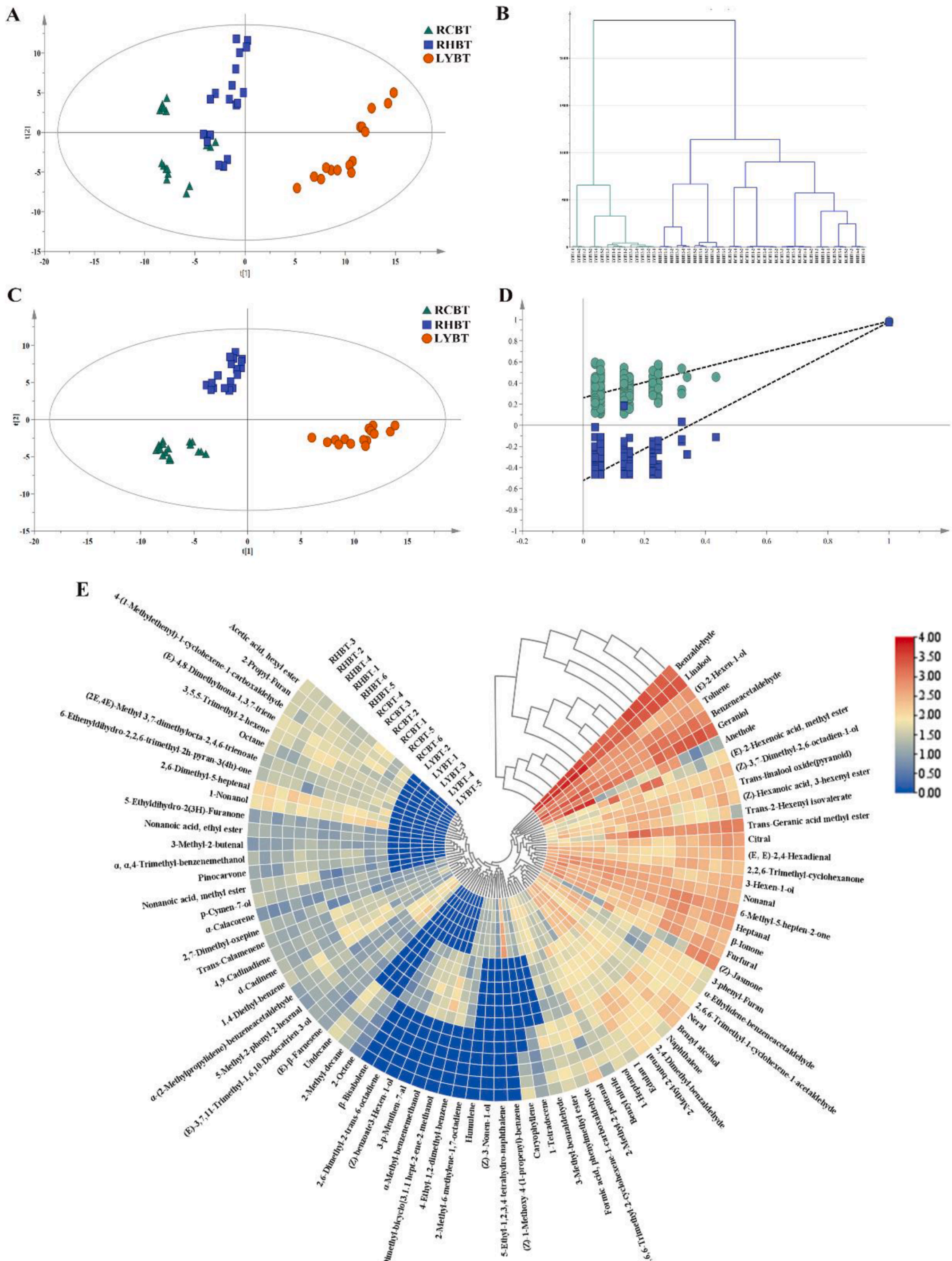

Fig. 3. PCA(A); HCA(B); PLS-DA(C); Validate model with 200 permutation tests(D); Heatmap of significantly differential volatile compounds(E).

3.3. Analysis of critical differential volatile compounds in black tea

To obtain a comprehensive understanding of the similarities and differences among 'Baimaocha' black tea in the three regions, we performed unsupervised PCA and HCA using the content of identified volatile compounds(Fig. 3A-B). PCA (PC1, 28.2 %; PC2, 14.9 %; R2X = 0.852) and HCA results showed a similar distributions in RCBT and RHBT samples, while LYBT and RCBT/RHBT samples are significantly segregated, indicating that the tea samples varied significantly in terms of their content levels of volatile compounds. These results are consistent with the above trends in aroma sensory analysis. To explore the differences in the distribution of volatile compounds in black teas of three different regions, PLS-DA was used to investigate the concentrations of 190 volatile compounds. As shown in Fig. 3C, the black tea from the three regions was clearly distinguished by the PLS-DA model (R2Y = 0.960, Q2 = 0.901), indicating significant differences in the distribution of the volatile compounds. The results of a validation test with 200 repeated calculations further showed the good reliability and predictive performance of model (Fig. 3D). 82 compounds were considered to be the critical differential volatile compounds of black teas from the three regions according to the variable importance in the projection(VIP) values being greater than 1.0 and *p*-value less than 0.05. The content of differential volatile compounds in black tea from three regions was visually displayed by heat map(Fig. 3E). It was evident that the contents of these critical volatile compounds have significant differences in 'Baimaocha' black teas from the three regions. Briefly, 26 critical volatile compounds showed the highest levels in the RCBT samples, while 24 and 32 differential metabolites showed the highest levels in RHBT and LYBT, respectively. Among them, higher levels of differential esters and alcohols may be contribute to the formation of the specific aroma of RCBT, including *trans*-geranic acid, methyl ester, (*E*)-2-hexenoic acid, methyl ester, (*Z*)-hexanoic acid, 3-hexenyl ester, 1-nonanol, and 1-heptanol (Hadi et al., 2013). In addition, higher levels of differential alcohols and ketones in RHBT samples, such as geraniol, (*Z*)-3,7-dimethyl-2,6-octadien-1-ol, 6-methyl-5-hepten-2-one, and (*Z*)-jasmone, could potentially influence the distinct aroma profile of RHBT. Similarly, the increased levels of distinct aldehyde and ketones compounds in LYBT, like benzaldehyde, benzeneacetaldehyde, nonanal, *trans*-*β*-ionone, may contribute to the formation of the specific aroma of LYBT. However, more verification is needed to confirm the role of these differential volatiles in the overall aroma of the tea samples.

3.4. Differential key odorants in black tea

As commonly recognized, the impact of volatile compounds on tea aroma is closely related to their concentration and odor threshold(Zheng et al., 2022). Interestingly, the aroma characteristics of teas are more significantly influenced by the presence of volatile compounds in low concentrations, rather than by those found in high concentrations (Chen et al., 2020; Plutowska & Wardencki, 2008). OAV analysis is widely employed as a fundamental method for assessing the contribution of aroma components, and volatile compounds with an OAV greater than 1 are commonly considered to be odorants (Xiao et al., 2022). In this study, 52 volatile compounds had an OAV greater than 1 and 25 substances had an OAV less than 1 (Table S3). Among them, there are 15 aroma compounds with OAV over 100 that are common to all three regions of 'Baimaocha' black tea, including *β*-damascenone, *trans*-*β*-ionone, α-ionone, (*Z*)-4-heptenal, (*E*)-2-nonenal, 2,4-nonadienal, linalool, octanal, nonanal, hexanal, benzeneacetaldehyde, 1-octen-3-ol, citral, geraniol, and methyl salicylate, suggesting that the above compounds have an important effect on the basic aroma formation of 'Baimaocha' black tea. Moreover, it is noteworthy that certain volatile compounds without OAV values may also affect the aroma formation in 'Baimaocha' black tea.

Combined with PLS-DA, a total of 23 compounds (VIP > 1, $p < 0.05$, OAV > 1) were identified as key odorants in the three regions of 'Baimaocha' black tea, including 8 aldehydes, 8 alcohols, 4 ketones, 2 aromatic hydrocarbons and 1 oxygen heterocyclic compound (Table 1). Among them, 3 aroma compounds were identified as distinguished odorants and had the highest content in the RCBT samples compared to the other two regions, including 1-heptanol, 1-nonanol, and *trans*-linalool oxide (pyranoid). Previous studies have shown that 1-heptanol with green and chemical attribute is an important aroma compound in Chinese Gongfu black tea(Xiao et al., 2017). 1-Nonanol with fresh and green attribute is an aroma-active compound in white tea, however it was detected only in RCBT and RHBT, suggesting that it may be an important contributor to the fresh attribute of RCBT and RHBT(Chen et al., 2020).

For RHBT samples, 9 contributing compounds were identified as critical odorants, including heptanal, 6-methyl-5-hepten-2-one, 2,2,6-trimethyl-cyclohexanone, 2,6-dimethyl-5-heptenal, myrtenol, neral, geraniol, citral, and (*Z*)-jasmone. The content of these compounds was highest in RHBT samples compared to the other two regions. It was reported that geraniol is the main contributor to the rose aroma of Keemun black tea and has an important influence on the aroma formation of black tea(Kang et al., 2019; Xiao et al., 2017). In our study, the content of geraniol in RHBT was notably higher compared to RCBT and LYBT, suggesting that geraniol may contribute to the characteristic rosy aroma of RHBT, which is in agreement with the findings of previous research (Wu et al., 2019). (*Z*)-Jasmone mainly exhibits floral aroma and is recognized as the aroma-active compound in Darjeeling and Ceylon black tea(Kang et al., 2019). In addition, heptanal, 2,2,6-trimethyl-cyclohexanone, neral and citral showed citrus or lemon odors, suggesting that they may contribute to the fruity attribute of RHBT. It was noteworthy that myrtenol with mint property and 2,6-dimethyl-5-heptenal with fruity and green were detected only in RHBT and RCBT, which may be related to its higher fresh attribute.

For LYBT samples, there were 11 contributing compounds had the highest contents compared to the other two regions and were identified as discriminatory odorants, including (*Z*)-3-hexen-1-ol, (*E*)-2-hexen-1-

Table 1
The key odorants among 'Baimaocha' black teas from three regions.

NO	Compounds	OTs (μg/kg)	OAV RCBT	RHBT	LYBT	Odor Characteristics	Concentration (ng/g) RCBT	RHBT	LYBT
1	Toluene	6	89.79	95.18	162.46	Sweet	538.74	571.08	974.77
2	(*E*)-2-Hexen-1-ol	359.3	1.61	0.98	3.51	Almond, green	579.44	353.36	1261.75
3	Heptanal	10	39.17	51.24	33.93	Citrus, green	391.65	512.42	339.29
4	(*E*, *E*)-2,4-Hexadienal	60	1.81	4.71	5.15	Citrus, green	108.85	282.43	308.81
5	Benzaldehyde	750.9	2.46	3.14	5.39	Sweet, almond	1845.14	2361.55	4047.82
6	6-Methyl-5-hepten-2-one	100	3.97	6.33	4.47	Roasted	397.22	632.69	447.05
7	Benzeneacetaldehyde	4	168.08	294.57	612.96	Floral, honey	672.32	1178.28	2451.82
8	2,6-Dimethyl-5-heptenal	16	3.73	7.45	0.00	Fruit, green	59.67	119.18	0.00
9	Linalool	6	439	400.36	907.19	Floral, fruity	2633.99	2402.18	5443.12
10	Nonanal	1	473.95	293.12	532.63	Floral, fruity	473.95	293.12	532.63
11	1-Nonanol	50	2.02	1.45	0.00	Fat, green	100.84	72.58	0.00
12	Trans-Linalool oxide(pyranoid)	3	76.08	68.89	24.15	Floral, sweet, green	228.25	206.68	72.46
13	Naphthalene	50	1.00	1.66	5.13	Pungent	49.95	82.91	256.71
14	Neral	100	0.65	1.49	0.84	Citrus, fruit, lemon	65.45	149.16	83.87
15	Geraniol	7.5	112.78	265.16	130.89	Floral, like rose	845.88	1988.69	981.7
16	Citral	1	178.55	397.97	155.76	Fruity, like lemon	178.55	397.97	155.76
17	Anethole	50	0.65	3.70	64.82	sweet	32.73	184.99	3241.04
18	(*Z*)-Jasmone	1.9	82.07	213.33	9.62	Fresh and floral, like jasmin	155.93	405.32	18.27
19	Trans-*β*-Ionone	0.007	27421.43	44048.57	60370.00	Floral, sweet	191.95	308.34	422.59
20	1-Heptanol	5.6	13.29	8.96	6.21	green	74.41	50.18	34.78
21	(*Z*)-3-Hexen-1-ol	70	4.93	3.10	7.37	grass, green	345.01	216.89	515.96
22	Myrtenol	7	11.08	12.29	0.00	mint	77.57	86.02	0.00
23	2,2,6-Trimethyl-cyclohexanone	100	1.47	2.58	2.33	citrus, honey	147.35	258.82	232.86

Note: OTs: Odor thresholds in water. The values were obtained from reported literature or relevant websites.

ol, (*E*,*E*)-2,4-hexadienal, benzaldehyde, benzeneacetaldehyde, linalool, nonanal, naphthalene, anethole, and *trans-β*-ionone. Among them, (*Z*)-3-hexen-1-ol and (*E*)-2-hexen-1-ol with green odor, which are converted from fatty acids, were identified as important aroma-active compounds in Chinese congou black tea (Ho et al., 2015; Xiao et al., 2017). Benzaldehyde is a compound with fruity attribute that is considered a key odorants of black tea, green tea and Fu brick tea(Xiao et al., 2017; Zheng et al., 2022; Zhu et al., 2018). Benzeneacetaldehyde, derived from phenylpropanoids, was identified as a major contributor to the honey sugar-like aroma in black tea(Yao et al., 2022). Linalool is an important terpene alcohol that has been shown to be a primary contributor to the floral and sweet aroma of black tea(Kang et al., 2019). Similarly, nonanal and *trans-β*-ionone are often present in black tea and produces a floral odor(Kang et al., 2019). In addition, naphthalene, characterized by a pungent odor and originating from long-chain hydrocarbons, was regarded as the key aroma compound in Fu brick tea (Zheng et al., 2022).

3.5. Aroma-active compounds in black tea by GC-O technique

GC-O is a technique that combines GC–MS and human olfaction to identify aroma-active compounds in complex mixtures and has been widely used in food flavor analysis(Song et al., 2018; Wang et al., 2020). It is generally accepted that the volatile compounds detected by GC-O experiments can be regarded as the aroma-active compounds of tea (Wang et al., 2020). To gain a more comprehensive understanding of the aroma active compounds and their specific odor attributes in 'Baimaocha' black tea, the volatile compounds of the tea samples were examined by GC-O combined with the GC × GC-Q-TOFMS technique, and one representative sample from each of the three regions of the black teas were chosen. As shown in Table 2, 35 key aroma active compounds were identified in the three tea samples, which mainly consisted of aldehydes, alcohols, ketones and esters.

In RCBT sample, the aroma intensity of linalool(3.33) was highest, which was perceived to be floral and sweet, and therefore is considered to be the main component of the aromatic quality of RCBT. It was reported that linalool is an important terpenoid alcohol present in a variety of plants and has been found to be a key tea aroma component (Yang et al., 2016). In addition, geraniol (floral and like rose, 3.00), benzeneacetaldehyde (floral and honey, 3.00), *β*-damascenone (floral, fruity and honey, 2.67), *trans-β*-ionone(floral and sweet, 2.67) of pleasant scents and hexanal (fresh and cut grass, 3.00), 1-octen-3-ol (fresh and mushroom-like, 3.00), 2-ethyl-5-methyl-furan(gassy, 2.67), methyl salicylate (fresh and wintergreen-like, 2.67), (*E*)-2-nonenal (green and cucumber-like, 2.67) of herbal-like scents also presented high aroma intensities. Among these compounds, linalool and geraniol are two important monoterpene alcohols that share the same aroma precursor, geranyl pyrophosphate (GPP), but exhibit different aroma profiles (Wang et al., 2020; Zhai et al., 2022). Moreover, it was reported that methyl salicylate has the highest concentration and exhibits very remarkably aroma intensity in Ceylon black tea, which is considered to be the main characteristic component of Ceylon black tea(Kang et al., 2019).

The higher aroma intensity and more aroma active compounds were detected in RHBT compared to RCBT and LYBT. In RHBT sample, benzeneacetaldehyde (floral and honey, 4.33) showed strongest aroma intensity, followed by geraniol (floral and like rose, 3.67), *trans-β*-ionone (floral and sweet, 3.67), neral (citrus, fruity and lemon, 3.33), *β*-damascenone (floral, fruity and honey, 3.00), 6-methyl-5-hepten-2-one (mushroom, pepper and rubber, 2.67), (*E*, *E*)-2,4-hexadienal (citrus, fat and green, 2.67), and heptanal (fat, green, and earth, 2.67). Among them, benzeneacetaldehyde with the highest aroma intensity was a

Table 2
Aroma-active compounds in 'Baimaocha' black teas identified by GC-O/MS.

NO.	Odorants	Odor descriptors	Aroma intensity			Identification
			RCBT	RHBT	LYBT	
1	Hexanal	fresh, cut grass	3.00	2.33	2.00	MS, RI
2	2-Ethyl-5-methyl-furan	gassy	2.67	2.00	2.33	MS, RI
3	1-Ethyl-1 h-pyrrole	roasted, chemical	–	1.00	2.00	MS, RI
4	p-Xylene	metal, sweet	2.00	1.33	1.33	MS, RI
5	(Z)-4-Heptenal	baked potatoes	3.00	2.67	2.67	MS, RI
6	(E, E)-2,4-Hexadienal	citrus, fat, green	1.67	2.67	2.33	MS, RI
7	Benzaldehyde	sweet, bitter almond	1.67	1.33	2.33	MS, RI
8	1-Octen-3-ol	fresh, mushroom-like	3.00	2.33	2.67	MS, RI
9	6-Methyl-5-hepten-2-one	mushroom, pepper	2.33	2.67	2.33	MS, RI
10	Benzeneacetaldehyde	floral, honey	3.00	4.33	4.67	MS, RI
11	Trans-linalool oxide (furanoid)	roasted, sweet	1.67	1.33	1.33	MS, RI
12	Benzoic acid, methyl ester	herb, prune	1.33	1.33	–	MS, RI
13	Linalool	floral, sweet	3.33	3.00	3.67	MS, RI
14	Nonanal	floral, fruity	3.33	2.33	3.33	MS, RI
15	Benzyl alcohol	floral, fruit	2.00	2.00	2.33	MS, RI
16	(R, S)-5-Ethyl-6-methyl-3E-hepten-2-one	fresh	1.33	1.00	1.00	MS, RI
17	(E, Z)-2,6-Nonadienal	fresh, cucumber-like	2.33	2.67	2.67	MS, RI
18	(E)-2-Nonenal	cucumber-like, green	2.67	2.00	1.33	MS, RI
19	(R)-4-Methyl-1-(1-methylethyl)-3-cyclohexen-1-ol	fresh, woody	3.00	2.33	2.33	MS, RI
20	Butanoic acid, hexyl ester	fruity, fresh	2.00	1.33	1.00	MS, RI
21	Methyl salicylate	fresh, wintergreen-like	2.67	2.00	2.33	MS, RI
22	3-Phenyl-furan	green	–	1.33	1.17	MS, RI
23	(Z)-3,7-Dimethyl-2,6-octadien-1-ol	floral, fruit, sweet	1.67	2.00	–	MS, RI
24	Neral	citrus, fruit, lemon	2.33	3.33	2.67	MS, RI
25	Geraniol	floral, like rose	3.00	3.67	2.33	MS, RI
26	Acetic acid, 2-phenylethyl ester	floral, honey	–	1.67	–	MS, RI
27	Citral	lemon	2.00	2.33	1.33	MS, RI
28	4-(1-Methylethenyl)-1-cyclohexene-1-carboxaldehyde	fat, green	–	2.00	1.50	MS, RI
29	Anethole	licorice, sweet	–	1.00	2.00	MS, RI
30	Edulan I	floral, rose	1.67	2.33	1.00	MS, RI
31	α-(2-Methylpropylidene)-benzeneacetaldehyde	cocoa	1.00	1.33	1.67	MS, RI
32	*β*-Damascenone	floral, fruit, honey	2.67	3.00	3.00	MS, RI
33	(Z)-Jasmone	floral, sweet	1.33	2.00	–	MS, RI
34	α-Ionone	floral	–	2.00	1.67	MS, RI
35	Trans-*β*-Ionone	floral, sweet	2.67	3.67	4.33	MS, RI

phenylalanine volatile in tea, which may be key aroma compound contributing to the floral and sweet aroma in RHBT(Kang et al., 2019; Yang et al., 2013). In addition, geraniol is an important compound contributing to the floral attribute of RHBT, as its aroma intensity is much higher in RHBT than in other two regions. On the other hand, it was noteworthy that the extremely high content of benzaldehyde did not exhibit a obviously high aroma intensity (1.33) in RHBT, which may be related to its higher odor threshold. Notably, acetic acid, 2-phenylethyl ester with floral and honey odor, was only found in RHBT with a weak aroma intensity.

In LYBT sample, benzeneacetaldehyde (4.67) and *trans*-*β*-ionone (4.33) showed pleasant and remarkable intensities, followed by linalool (3.67), 1-octen-3-ol (2.67), heptanal (2.67) and neral (2.67). Among these compounds, it was interesting that benzeneacetaldehyde has the highest aromatic intensity in both RHBT and LYBT, which may be related to the proximity of their varietal genetic bases. In addition, *trans*-*β*-ionone originating from the oxidative degradation of *β*-carotene during tea drying, was considered as key odorants of LYBT due to its much higher aroma intensity than in RCBT and RHBT. Furthermore, other carotenoid derivatives, including *α*-ionone (floral, 1.67) and 6-methyl-5-hepten-2-one (mushroom, pepper, rubber, 2.33) were also considered to be key aroma-active compounds in LYBT. Moreover, 1-octen-3-ol formed by linoleic acid and 1-penten-3-one as precursors showed mushrooms odor and has been identified as a common odorant of Assam, Darjeeling, Keemun and Ceylon black tea(Ho et al., 2015; Kang et al., 2019).

3.6. The comparison of the results obtained by OAV and GC-O methods

Numerous volatiles have been found in various foods, and only a limited number of volatiles are considered as key aroma compounds that significantly influence the overall aroma profile (Song & Liu, 2018; Zhai et al., 2022). Although it was possible to distinguish the key aroma compounds of black tea from the above three regions by GC-O technique and OAV calculation. However, there were some unavoidable deviations in the results due to the subjectivity of GC-O analysis and inconsistent reporting of odor thresholds during OAV calculation (Chen et al., 2020; Kang et al., 2019; Wang et al., 2020). The utilization of GC-O and OAV can complement each other in the identification of key odorants, so it is essential to amalgamate and validate the findings from these two methods and to corroborate the identified compounds collectively.

The common compounds were identified by GC-O and OAV calculation in RCBT including heptanal, (*E*, *E*)-2,4-hexadienal, benzaldehyde, 6-methyl-5-hepten-2-one, benzeneacetaldehyde, linalool, nonanal, geraniol, citral, (*Z*)-jasmone, *trans*-*β*-ionone, which suggested them play a significant role in the aroma formation in RCBT. It was also noteworthy that methyl salicylate had obviously high aroma intensity and the highest concentration in RCBT, proving an important influence on aroma of RCBT.

Likewise, heptanal, (*E*, *E*)-2,4-hexadienal, benzaldehyde, 6-methyl-5-hepten-2-one, benzeneacetaldehyde, linalool, nonanal, neral, geraniol, citral, (*Z*)-jasmone, *trans*-*β*-ionone were the common compounds identified by the two methods in RHBT. Apparently, the OAV of geraniol in RHBT was much higher than that of the other tea samples, and thus it was considered to be one of the most distinctive odorant in RHBT, which was consistent with previous findings (Wu et al., 2019). In addition, heptanal, (*E*, *E*)-2,4-hexadienal, benzaldehyde, 6-methyl-5-hepten-2-one, benzeneacetaldehyde, linalool, nonanal, geraniol, citral, (*Z*)-jasmone, *trans*-*β*-ionone were common substances in LYBT. In particular, *trans*-*β*-ionone and benzeneacetaldehyde had significantly higher OAV values in LYBT than the other tea samples and might be the most definitive characteristic odorants.

3.7. Relationship between odorants and sensory attributes of black tea in three regions

To further explore deeper into the link between odorants (OAV > 1) and sensory attributes of 'Baimaocha' black tea, we performed a PLS model. This model featured two latent variables, explaining 45.0 % of the variance in the X-matrix (odorants) and 66.8 % of the variance in the Y-matrix (sensory attributes). As shown in the Fig. 4, the tea can be categorized into 3 groups according to different regions. The first component is mainly determined by the aroma descriptor, which displays the roasted and sweet attributes in the positive dimension and fresh, floral, and fruity in the negative dimension.

The qualities (floral, fruity, roasted, fresh) significantly associated with certain aroma compounds were all located in the region between the inner and outer ellipse, suggesting that they can be well explained by the model. Among them, the attribute of floral exhibited significant positive correlations with three compounds, namely *trans*-linalool oxide (pyranoid) (VC61), geraniol (VC67), and (*Z*)-jasmone(VC88). It suggested that the above three compounds may be the critical contributing compounds to the higher floral attribute of RCBT and RHBT than LYBT. The fruity attribute have a high positive correlation with four compounds, including 2,6-dimethyl-5-heptenal(VC20), neral(VC34), citral (VC36), and 1-methyl-4-(1-methylethenyl)-benzene(VC160). Previous studies have shown that aldehydes the majority of reported aldehydes contribute to the characteristic citrusy and green flavor of the tea broth (Zhai et al., 2022). The roasted attribute was positively correlated to (*Z*)-4-heptenal(VC10), (*E*, *E*)-2,4-heptadienal(VC16), and 6-methyl-5-hepten-2-one (VC75). Among them, (*E*, *E*)-2,4-heptadienal was reported to have roast potato properties in grilled beef and 6-methyl-5-hepten-2-one was considered as the key odorant of Chinese congou black tea (Frank et al., 2016; Xiao et al., 2017). The fresh attribute was positively correlated with (*E*)-2-nonenal(VC28), 1-heptanol(VC48), 1-nonanol(VC60), and myrtenol(VC65). It is widely acknowledged that some of the fatty acid derivatives in tea contribute to the fresh and green aroma(Wu et al., 2019; Ho et al., 2015).

4. Conclusion

In this study, the aroma components and key odorants in 'Baimaocha' black tea from three regions were comprehensively analyzed using HS-SPME combined with GC × GC-O-Q-TOMS. 190 volatile compounds were identified by GC × GC-Q-TOMS, among which aldehydes and alcohols were the main volatile components of black tea from the three regions. PCA and PLS-DA showed that the volatile components of black tea from the three regions could be significantly separated, with 82 differential volatile compounds as potential key aroma compounds. According to the OAV analysis, 23 compounds were recognized as critical odorants that contributing to the distinct aroma characteristics found in black tea from three regions, including heptanal, (*E*, *E*)-2,4-hexadienal, benzaldehyde, 6-methyl-5-hepten-2-one, phenylacetaldehyde, linalool, nonanal, geraniol, and citral. GC-O results showed that 29, 35, and 30 aroma-active compounds were identified in RCBT, RHBT, and LYBT, respectively. In addition, the comprehensive assessment of OAV combined with GC-O revealed that methyl salicylate (RCBT), geraniol (RHBT), *trans*-*β*-ionone and benzeneacetaldehyde (LYBT) might be the most definitive aroma compounds identified in the teas from their respective regions. Furthermore, *trans*-linalool oxide (pyranoid), geraniol, and (*Z*)-jasmone were recognized as significant contributors to floral attribute, 2,6-dimethyl-5-heptenal, neral, citral, and 1-methyl-4-(1-methylethenyl)-benzene were contributed to fruity attribute, (*E*)-2-nonenal, 1-nonanol, 1-heptanol, and 2,6-dimethyl-5-heptenal were contributed to fresh attribute, (Z)-4-heptenal, (*E*, *E*)-2,4-heptadienal, 6-methyl-5-hepten-2-one were contributed to roasted attribute by PLS analysis. These results help to deepen the understanding of the key odorants associated with 'Baimaocha' black tea from different regions, and provide a theoretical basis for controlling and

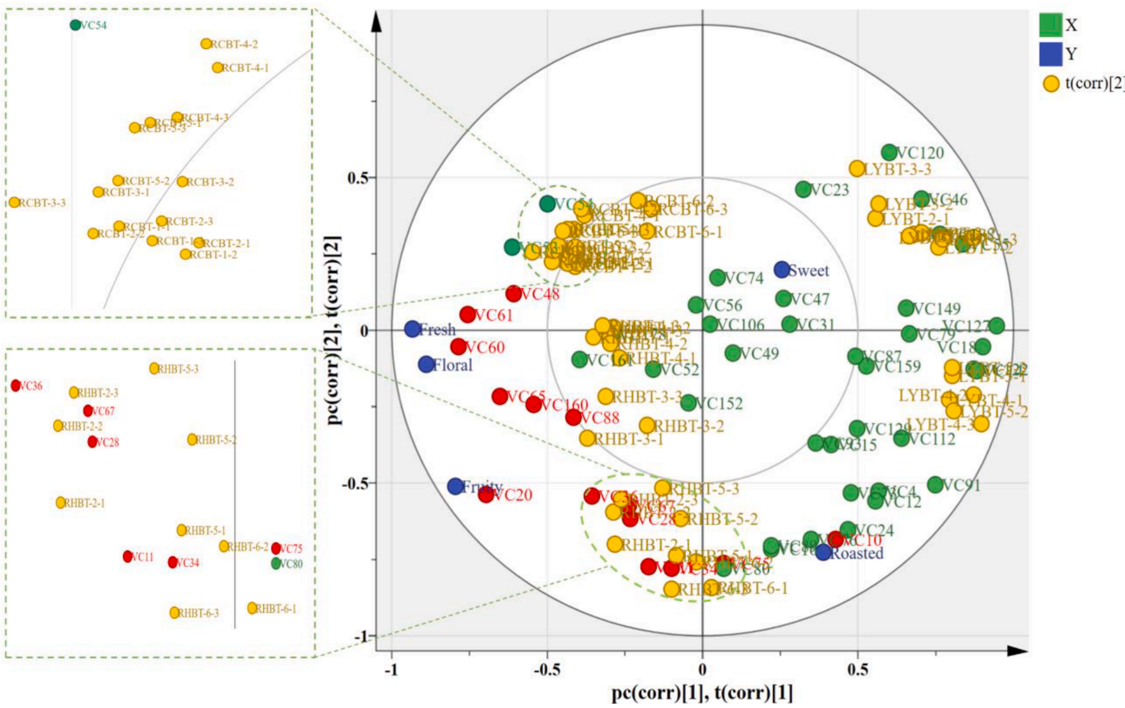

Fig. 4. PLS plots for the tea samples, the sensory attributes and aroma-active compounds (OAV > 1). VC10: (*Z*)-4-heptena; VC16: (*E*)-2-hexen-1-ol; VC20: 5-heptenal; VC28: (*E*)-2-nonenal; VC34: neral; VC36: citral; VC48: 1-heptanol; VC60: 1-nonanol; VC61: *trans*-linalool oxide (pyranoid); VC65: myrtenol; VC67: geraniol; VC75: 6-methyl-5-hepten-2-one; VC88: (*Z*)-jasmone; VC160: 2,6-dimethyl- 1-methyl-4-(1-methylethenyl)-benzene.

enhancement of tea aroma quality.

CRediT authorship contribution statement

Jian Ouyang: Writing – original draft, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Ronggang Jiang:** Investigation, Formal analysis, Data curation. **Hongyu Chen:** Investigation, Formal analysis. **Qi Liu:** Methodology, Data curation. **Xiaoqin Yi:** Methodology, Investigation. **Shuai Wen:** Investigation, Data curation. **Fangfang Huang:** Investigation, Data curation. **Xinyi Zhang:** Formal analysis, Data curation. **Juan Li:** Investigation, Formal analysis. **Haitao Wen:** Methodology, Data curation. **Ligui Xiong:** Writing – review & editing, Investigation, Data curation. **Zhonghua Liu:** Writing – review & editing, Project administration, Data curation, Conceptualization. **Jianan Huang:** Writing – review & editing, Supervision, Project administration, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fochx.2024.101303.

References

Chen, D., Li, J. X., Zhuo, M., Li, D. C., Wu, H. L., Zhao, C. Y., Yan, C. Y., Huang, H. L., Sun, S. L., & Xie, H. S. (2010). Selection and breeding of a new highly aromatic black (white) tea cultivar danxia2. *Guangdong Agricultural Science, 37*(11). https://doi.org/10.16768/j.issn.1004-874x.2010.11.039

Chen, Q. C., Zhu, Y., Yan, H., Chen, M., Xie, D.-C., Wang, M.-Q., Ni, D.-J., & Lin, Z. (2020). Identification of aroma composition and key odorants contributing to aroma characteristics of white teas. *Molecules, 25*(24), 6050. https://doi.org/10.3390/molecules25246050

Chen, H., Zhang, X., Jiang, R., Ouyang, J., Liu, Q., Li, J., Wen, H., Li, Q., Chen, J., Xiong, L., Huang, J., & Liu, Z. (2023). Characterization of aroma differences on three drying treatments in rucheng baimao (Camellia pubescens) white tea. *LWT-Food Science and Technology, 179*, Article 114659. https://doi.org/10.1016/j.lwt.2023.114659

Dai, Q. Y., An, Q., Zheng, F. L., Xiao, M. X., Chen, Y. Q., Zhang, Y., & Qiu, T. (2022). Analysis of aroma sensory characteristics of and preference for large-leaf yellow tea using quantitative descriptive analysis and check-all-that-apply. *Journal of Food Science, 43*(21), 23–33. https://doi.org/10.7506/spkx1002-6630-20210827-354

Frank, D., Ball, A., Hughes, J., Krishnamurthy, R., Piyasiri, U., Stark, J., Watkins, P., & Warner, R. (2016). Sensory and flavor chemistry Characteristics of australian beef: Influence of Intramuscular fat, feed, and breed. *Journal of Agricultural and Food Chemistry, 64*(21), 4299–4311. https://doi.org/10.1021/acs.jafc.6b00160

Ge, Y., Wang, L., Huang, Y., Jia, L., & Wang, J. (2024). Characteristic flavor compounds in Guizhou green tea and the environmental factors influencing their formation: Investigation using stable isotopes, electronic nose, and headspace-gas chromatography ion migration spectrometry. *LWT-Food Science and Technology, 196*, Article 115887. https://doi.org/10.1016/j.lwt.2024.115887

Hadi, M., Zhang, F. J., Wu, F. F., Zhou, C. H., & Tao, J. (2013). Advances in fruit aroma volatile Research. *Molecules, 18*(7), 8200–8229. https://doi.org/10.3390/molecules18078200

Ho, C. T., Zheng, X., & Li, S. (2015). Tea aroma formation. *Food Science and Human Wellness, 4*(1), 9–27. https://doi.org/10.1016/j.fshw.2015.04.001

Kang, S., Yan, H., Zhu, Y., Liu, X., Lv, H. P., Zhang, Y., Dai, W. D., Guo, L., Tan, J. F., Peng, Q.-H., & Lin, Z. (2019). Identification and quantification of key odorants in the world's four most famous black teas. *Food Research International, 121*, 73–83. https://doi.org/10.1016/j.foodres.2019.03.009

Li, H. H., Luo, L. Y., Wang, J., Fu, D. H., & Zeng, L. (2019). Lexicon development and quantitative descriptive analysis of Hunan fuzhuan brick tea infusion. *Food Research International, 120*, 275–284. https://doi.org/10.1016/j.foodres.2019.02.047

Li, P., Xu, Y., Zhang, Y., Fu, J., Yu, S., Guo, H., Chen, Z., Chen, C., Yang, X., Wang, S., & Zhao, J. (2020). Metabolite profiling and transcriptome analysis revealed the chemical contributions of tea trichomes to tea flavors and tea plant defenses. *Journal of Agricultural and Food Chemistry, 68*(41), 11389–11401. https://doi.org/10.1021/acs.jafc.0c04075

Meng, X. H., Zhu, H.-T., Yan, H., Wang, D., Yang, C.-R., & Zhang, Y.-J. (2018). C-8 N-Ethyl-2-pyrrolidinone-substituted Flavan-3-ols from the leaves of *Camellia sinensis var .Pubilimba*. *Journal of Agricultural and Food Chemistry, 66*(27), 7150–7155. https://doi.org/10.1021/acs.jafc.8b02066

Peng, Y., Li, G., Liu, X. Y., Lv, C. Y., & Xiong, C. Y. (2021). SPME/GC-MS analysis of aroma quality of black tea from different producing areas. *Science and Technology of Food Industry, 42*(9), 237–244. https://doi.org/10.13386/j.issn1002-0306.2020060025

Plutowska, B., & Wardencki, W. (2008). Application of gas chromatography–olfactometry (GC–O) in analysis and quality assessment of alcoholic beverages – a review. *Food Chemistry, 107*(1), 449–463. https://doi.org/10.1016/j.foodchem.2007.08.058

Song, H., & Liu, J. (2018). GC-O-MS technique and its applications in food flavor analysis. *Food Research International, 114*, 187–198. https://doi.org/10.1016/j.foodres.2018.07.037

Su, D., He, J.-J., Zhou, Y.-Z., Li, Y.-L., & Zhou, H.-J. (2022). Aroma effects of key volatile compounds in keemun black tea at different grades: HS-SPME-GC-MS, sensory evaluation, and chemometrics. *Food Chemistry, 373*, Article 131587. https://doi.org/10.1016/j.foodchem.2021.131587

Wang, M. Q., Ma, W.-J., Shi, J., Zhu, Y., Lin, Z., & Lv, H.-P. (2020). Characterization of the key aroma compounds in Longjing tea using stir bar sorptive extraction (SBSE) combined with gas chromatography-mass spectrometry (GC–MS), gas chromatography-olfactometry (GC-O), odor activity value (OAV), and aroma recombination. *Food Research International, 130*, Article 108908. https://doi.org/10.1016/j.foodres.2019.108908

Wang, X., Guo, M., Song, H., Meng, Q., & Guan, X. (2021). Characterization of key odor-active compounds in commercial high-salt liquid-state soy sauce by switchable GC/GC × GC–olfactometry–MS and sensory evaluation. *Food Chemistry, 342*, Article 128224. https://doi.org/10.1016/j.foodchem.2020.128224

Wang, Q., Qin, D., Huang, G., Jiang, X., Fang, K., Wang, Q., Ni, E., Li, B., Pan, C., Li, H., Chen, D., & Wu, H. (2022). Identification and characterization of the key volatile flavor compounds in black teas from distinct regions worldwide. *Journal of Food Science, 87*(8), 3433–3446. https://doi.org/10.1111/1750-3841.16248

Wu, H., Huang, W., Chen, Z., Chen, Z., Shi, J., Kong, Q., Sun, S., Jiang, X., Chen, D., & Yan, S. (2019). GC–MS-based metabolomic study reveals dynamic changes of chemical compositions during black tea processing. *Food Research International, 120*, 330–338. https://doi.org/10.1016/j.foodres.2019.02.039

Xiao, Z., Wang, H., Niu, Y., Liu, Q., Zhu, J., Chen, H., & Ma, N. (2017). Characterization of aroma compositions in different chinese congou black teas using GC-MS and GC-O combined with partial least squares regression. *Flavour and Fragrance Journal, 32*(4), 265–276. https://doi.org/10.1002/ffj.3378

Xiao, Y., Huang, Y., Chen, Y., Xiao, L., Zhang, X., Yang, C., Li, Z., Zhu, M., Liu, Z., & Wang, Y. (2022). Discrimination and characterization of the volatile profiles of five fu brick teas from different manufacturing regions by using HS–SPME/GC–MS and HS–GC–IMS. *Current Research in Food Science, 5*, 1788–1807. https://doi.org/10.1016/j.crfs.2022.09.024

Yang, Z., Baldermann, S., & Watanabe, N. (2013). Recent studies of the volatile compounds in tea. *Food Research International, 53*(2), 585–599. https://doi.org/10.1016/j.foodres.2013.02.011

Yang, T., Zhu, Y., Shao, C.-Y., Zhang, Y., Shi, J., Lv, H.-P., & Lin, Z. (2016). Enantiomeric analysis of linalool in teas using headspace solid-phase microextraction with chiral gas chromatography. *Industrial Crops and Products, 83*, 17–23. https://doi.org/10.1016/j.indcrop.2015.12.025

Yang, J., Li, Z., Lu, J., He, W., Feng, H., Liu, H., Xu, D., Liang, G., & Luo, L. (2020). Analysis and discrimination of aroma compounds of guire 2 black tea treated with different drying methods. *Science and Technology of Food Industry, 41*(16), 67–73+87. https://doi.org/10.13386/j.issn1002-0306.2020.16.012

Yang, F., Liu, Y., Wang, B., Song, H., & Zou, T. (2021). Screening of the volatile compounds in fresh and thermally treated watermelon juice via headspace-gas chromatography-ion mobility spectrometry and comprehensive two-dimensional gas chromatography-olfactory-mass spectrometry analysis. *LWT-Food Science and Technology, 137*, Article 110478. https://doi.org/10.1016/j.lwt.2020.110478

Yang, J., Liang, G., Li, Z., Liang, X., & Chen, Y. (2023). Analysis of aroma characteristics of fermented lingyun baihao tea based on odor activity value. *Journal of Food Science, 44*(14), 336–343. https://doi.org/10.7506/spkx1002-6630-20220923-254

Yun, J., Cui, C., Zhang, S., Zhu, J., Peng, C., Cai, H., Yang, X., & Hou, R. (2021). Use of headspace GC/MS combined with chemometric analysis to identify the geographic origins of black tea. *Food Chemistry, 360*, Article 130033. https://doi.org/10.1016/j.foodchem.2021.130033

Zhai, X., Zhang, L., Granvogl, M., Ho, C., & Wan, X. (2022). Flavor of tea (Camellia sinensis): A review on odorants and analytical techniques. *Comprehensive Reviews in Food Science and Food Safety, 21*(5), 3867–3909. https://doi.org/10.1111/1541-4337.12999

Zhang, L., Ho, C., Zhou, J., Santos, J. S., Armstrong, L., & Granato, D. (2019). Chemistry and biological activities of processed Camellia sinensis teas: A comprehensive review. *Comprehensive Reviews in Food Science and Food Safety, 18*(5), 1474–1495. https://doi.org/10.1111/1541-4337.12479

Zhang, L., Cao, Q. Q., Granato, D., Xu, Y.-Q., & Ho, C.-T. (2020). Association between chemistry and taste of tea: A review. *Trends in Food Science & Technology, 101*, 139–149. https://doi.org/10.1016/j.tifs.2020.05.015

Zheng, X. X., Hong, X., Jin, Y. L., Wang, C., Liu, Z. H., Huang, J. A., & Li, Q. (2022). Characterization of key aroma compounds and relationship between aroma compounds and sensory attributes in different aroma types of fu brick tea. *Food Chemistry: X, 13*, Article 100248. https://doi.org/10.1016/j.fochx.2022.100248

Zheng, F., Gan, S., Zhao, X., Chen, Y., Zhang, Y., Qiu, T., Zheng, P., Zhai, X., & Dai, Q. (2023). Unraveling the chemosensory attributes of Chinese black teas from different regions using GC-IMS combined with sensory analysis. *LWT-Food Science and Technology, 184*, Article 114988. https://doi.org/10.1016/j.lwt.2023.114988

Zheng, X., Hu, T., Xie, H., Ou, X., Huang, J., Wang, C., Liu, Z., & Li, Q. (2023). Characterization of the key odor-active compounds in different aroma types of Fu brick tea using HS-SPME/GC-MSO combined with sensory-directed flavor analysis. *Food Chemistry, 426*, Article 136527. https://doi.org/10.1016/j.foodchem.2023.136527

Zhong, X. G., Huang, H. S., Li, N., & Su, B. W. (2022). Effect of withering and fermentation technology on the quality of "Rucheng Baimaocha" processed black tea. *Food and Fermentation Industries, 48*(4), 137–144. https://doi.org/10.13995/j.cnki.11-1802/ts.027873

Zhou, B., Chen, Y., Zeng, L., Cui, Y., Li, J., Tang, H., Liu, J., & Tang, J. (2022). Soil nutrient deficiency decreases the postharvest quality-related metabolite contents of tea (Camellia sinensis (L.) kuntze) leaves. *Food Chemistry, 377*, Article 132003. https://doi.org/10.1016/j.foodchem.2021.132003

Zhu, M., Li, N., Zhao, M., Yu, W., & Wu, J.-L. (2017). Metabolomic profiling delineate taste qualities of tea leaf pubescence. *Food Research International, 94*, 36–44. https://doi.org/10.1016/j.foodres.2017.01.026

Zhu, Y., Lv, H. P., Shao, C. Y., Kang, S., Zhang, Y., Guo, L., Dai, W.-D., Tan, J.-F., Peng, Q.-H., & Lin, Z. (2018). Identification of key odorants responsible for chestnut-like aroma quality of green teas. *Food Research International, 108*, 74–82. https://doi.org/10.1016/j.foodres.2018.03.026