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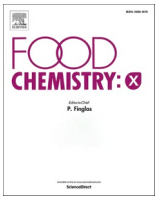

Characterization of key aroma in Huangjincha black tea processed from four different cultivars

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ABSTRACT

Huangjincha (HJC) is a cultivar rich in amino acids making it ideal for producing high-quality black tea. In this study, the aroma composition of HJC black tea from four different cultivars (HJC1, HJC2, HJC18, and HJC168) was analyzed by aroma sensory evaluation combined with volatile compound analysis. Among 271 identified volatiles, with 39 compounds as key odorants contributing to the diverse aromas of HJC black teas. The OAV and GC-O results indicated that geraniol and benzeneacetaldehyde (HJC1), 3-methylbutanal and 1-penten-3-ol (HJC2), (E, E)-2,4-hexadienal and damascenone (HJC18), as well as methyl salicylate and citral (HJC168) were the most significant aroma compounds. Furthermore, PLS analysis revealed four odorants contributed to floral characteristic, three were related to fruity attribute, four were associated with green attribute, three were connected to fresh attribute, three were linked to nutty profile, and three were tied to the sweet profile.

1. Introduction

Black tea is a non-alcoholic beverage that is one of six varieties of tea in China (S. Li et al., 2013). It is widely recognized for its distinct flavor, long history, cultural significance, and health benefits (H. Liu et al., 2021). Numerous factors impact the aroma characteristics of black tea such as variety, production region, processing methods (Kang et al., 2019). Typically, the richness of tea plant germplasm underlies the diversity of black tea flavor profiles (Qin et al., 2023). For instance, Dian Hong, produced from the Yunnan Big Leaf cultivar (C. Wang et al., 2017), is renowned for its unique caramel fragrance and 'mellow' flavor, making it highly favored by both the market and consumers. Keemun black tea made from the 'Qimen zhong' cultivar, boasts a unique 'Keemun aroma' with regional flavors, such as floral and honey (Huang et al., 2022). Huangjincha (HJC), which originates from Huangjin Village in Hulu Town, Baojing County, within Xiangxi Tujia and Miao Autonomous Prefecture of Hunan Province, is distinguished by its high amino acid content that significantly contributes to its unique flavor profile (Zhong et al., 2023). Abundant studies have demonstrated that amino acids serve as essential precursors in the formation of black tea aroma. Isoleucine undergoes enzymatic conversion to form 2-methylbutyraldehyde, which significantly contributes to the sweet aroma of black tea (Ho et al., 2015). Similarly, phenylalanine, catalyzed by deaminase or decarboxylase, produces phenylacetaldehyde, a key aroma-active compound in black tea (Kang et al., 2019). Therefore, HJC is recognized as an excellent raw material for producing high-grade black tea. However, only a few research studies have investigated the volatiles of HJC and reported that phenylethanol, geraniol, trans-2-nonenal, 2-methylbutyraldehyde, methyl salicylate, and phenylglyoxal contributed to the aroma of HJC black tea (Gong et al., 2022; Huang et al.,

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2020; Xie et al., 2022). Additionally, studies have demonstrated that the primary characteristic of the aroma in HJC black tea across different seasons is a sweet fragrance (Xie et al., 2022). Furthermore, no precise quantification experiments have been conducted, nor have recombination experiments been employed to verify the results. Thus, a comprehensive investigation into the compound basis of HJC black tea aroma remains lacking.

More than 700 volatile compounds have been identified in tea leaves and infusions to date (Zhai et al., 2022). Over the years, several methods have been developed to extract aroma components. Headspace solid-phase microextraction (HS-SPME), which relies on the adsorption and desorption of compounds on fused silica fibers coated with various polar or nonpolar adsorbents, is a selective, sensitive, rapid, and user-friendly method for collecting volatile substances (Kang et al., 2019). However, only a few volatile compounds contribute significantly to the overall flavor of tea (Yu et al., 2023). Therefore, it is crucial to identify key aroma-active compounds in tea through methods such as calculating the Odor Activity Value (OAV) (Chen et al., 2023) and Gas Chromatography-Olfactometry (GC-O) (Shi et al., 2021). Additionally, comprehensive two- dimensional gas chromatography-quadrupole-time-of- flight mass spectrometry (GC × GC-QTOFMS) has been commonly used for the analysis of volatile compounds in tea in recent years (Y. Yang et al., 2024). GC × GC-QTOFMS demonstrates superior separation capabilities, enhanced sensitivity, and increased peak capacity compared to GC–MS (Q. Wang et al., 2024).

In this study, the aroma differences among four different cultivars (HJC1, HJC2, HJC18, HJC168) of HJC black tea were investigated using GC × GC-QTOFMS combined with sensory evaluation and the GC-O method. The results will provide production guidelines and a theoretical basis for understanding the qualitative attributes of black tea from various HJC cultivars.

2. Materials and methods

2.1. Black tea sample preparation

The different cultivars of HJC used in this work were cultivated and managed in the Xiangxi District (Hunan Province, China). The HJC materials used in the experiment were four different cultivars, including HJC1, HJC2, HJC18 and HJC168. The samples were processed from the fresh leaves of one bud and one leaf in the spring of 2022.These HJC samples were according to the traditional HJC processing techniques, including withering, rolling, fermentation, and drying. Specifically, the fresh tea leaves were withered in the withering trough for 14 to 16 h. The withered tea leaves were moderately rolled in the rolling machine for three stages: the first light roll for 20 min, the second heavy roll for 20 min, and a final light roll for 20 min. To ensure complete fermentation, the tea leaves must be completely unblocked after rolling. Following this, the tea leaves are fermented at 30 °C with 95 % humidity for 4 to 5 h, and the leaves should be turned over every hour to ensure moderate fermentation. After fermentation, the leaves are initially dried in a chain dryer at 110 °C, then spread out to cool and regain moisture. Once softened and no longer prickly, the leaves are re-rolled for 5 min. Finally, the leaves are dried at 80 to 90 °C for 2 h and are subsequently aroma-enhanced at 100 to 110 °C for 10 min.

2.2. Sensory evaluation

The sensory evaluation of HJC black teas was conducted in accordance with the Chinese national standard (GB/T 23776–2018). In this process, three grams of each tea sample were placed in separate teacups, covered with lids, and infused with 150 ml of boiling water. After a 5-min brewing period, the tea infusion was subjected to sensory testing, where each assessor evaluated the fragrance in the teacup. Quantitative descriptive analysis (QDA) was employed to analyze the aromatic characteristics of the black tea (Chen et al., 2023). The intensity values and aroma descriptors of the samples were validated by seven assessors, consisting of three males and four females with an average age of 28 years. The aroma descriptors of the tea samples were attributed to floral, fruity, sweet, green, nutty and fresh. The intensity of each attribute was rated on a scale from 0 to 10, with higher scores indicating stronger intensity. Each sample was evaluated three times on different days to ensure consistency.

2.3. HS–SPME–GC*GC-QTOF–MS detection of volatile components

The extraction of volatile compounds was conducted using the headspace solid-phase microextraction (HS-SPME) method. A 20-ml headspace vial was filled with 0.5 g of black tea powder, followed by the addition of a rotor, 10.0 μl of ethyl caprate (10 ppm), and 5.0 ml of boiling water. The vial was immediately sealed and placed on a thermostatic oscillator equilibrated at 80 °C for 10 min. The sample headspace was then extracted for 30 min using a carboxen/polydimethylsiloxane (CAR/PDMS; 85 μm; 1.0 cm) coated fiber (Sigma-Aldrich Trading Co., Ltd., Shanghai). The volatile compounds were subsequently desorbed in a GC × GC-QTOFMS injector at 250 °C for 10 min. Volatile analysis was performed using an Agilent 7250 GC/Q-TOFMS. Chromatographic separation employed a DB-17MS column (2.89 m × 180 μm × 0.18 μm; Agilent Technologies) for the second dimension and an HP-5MS column (37.69 m × 250 μm; Agilent Technologies) for the first dimension. Helium (99.999 %) was used as the carrier gas at a constant flow rate of 1.0 ml/min, with the transmission line temperature maintained at 280 °C. A split ratio of 10:1 was applied. The column temperature was programmed to start at 40 °C, increase to 160 °C at a rate of 3.0 °C/min, then further rise to 250 °C at 20 °C/min, where it was held for 1 min, resulting in a total runtime of 46.5 min. The mass spectrometry (MS) parameters included an electron ionization (EI) source with an acquisition range of 45–500 *m/z*, an acquisition rate of 50 Hz, an ion source temperature of 230 °C, an ionization energy of 70 eV, and a filament emission current of 5.0 μA. A solid-state modulator (SSM1800; J & X Technologies, Shanghai, China) positioned between the two columns facilitated heating and cooling. The modulation time was 5.0 s, with the cold zone temperature set at 50 °C. One-dimensional to two-dimensional conversion was performed using an HV series modulation column (1.3 m × 0.25 mm; C5–C30; J & X Technologies, Shanghai, China).

2.4. Qualitative and quantitative analysis of volatiles

The GC × GC-QTOFMS data were processed using the CANVAS program (version 2.0.0.25117). A minimum signal-to-noise ratio of 10 was set as the threshold for peak identification. The mass spectrometric data of each chromatographic peak were compared against the 'mainlib,' 'replib,' and 'nist_ri' databases within the NIST 20 mass spectral library. Sample peak tables were then analyzed using the statistical comparison feature of the CANVAS software, with the penalty level set to 'very strong'. Retention index (RI) values were used to correct the matching results. Peaks meeting the criteria of an RI deviation <15, a positive match >700, and a reverse match >800 were considered valid (Chen et al., 2023). The relative concentration of volatiles was calculated as the ratio of the peak areas of the identified compounds to that of ethyl caprate.

2.5. Odor activity value (OAV) calculation

OAV of volatile compounds was determined by calculating the ratio of the relative concentration of each compound to its threshold for odor in the water.

2.6. Analysis of odor types and intensities of volatiles using GC-O

The extraction method for volatile compounds in GC-O analysis was

consistent with that of GC× GC-QTOTMS. The odor extract was divided equally between the olfactory detection port (ODP4, Gerstel, Germany) and the mass spectrometer. Both the GC-O injector and transfer line were set to a temperature of 250 °C, using high-purity nitrogen (99.99 %) as the carrier gas. Three trained assessors conducted the GC-O analysis, each undergoing two weeks of training. A 3-point intensity scale from 1 to 5 was used to evaluate aroma intensities, where "1" indicated weak, "3" moderate, and "5" strong. The sniffing results from the assessors were collected and analyzed. Odors detected by at least two assessors within the same time interval (time deviation <0.1 min) and having similar descriptions were selected. These selected odors were characterized by referencing the NIST spectral library and retention index, and their descriptions were validated through professional literature and websites.

2.7. Data analysis of volatile compounds

The analysis was repeated at least three times, and the results were expressed as mean ± standard deviation (SD). Statistical analyses were conducted using SPSS version 22.0 (SPSS Inc., Chicago, IL, USA) for Duncan's multiple range test in one-way ANOVA, with $p < 0.05$ considered statistically significant. Principal component analysis (PCA), orthogonal partial least squares-discriminant analysis (OPLS-DA), hierarchical cluster analysis (HCA), and partial least squares analysis (PLS) were performed using SIMCA-P+ 14.0 software. Spider plots were generated using OriginPro (Origin Lab Corporation, Northampton, MA, USA), and heat maps were created using TBtools (https://github.com/CJ-Chen/TBtools). Cytoscape was used to map the co-expression network of volatile compounds.

3. Results and discussion

3.1. Sensory profiles of HJC black tea derived from different cultivars

The appearance and infusion of the four varieties of HJC black tea are shown in Fig. 1A.As can be seen from the figure, it is challenging to distinguish them based on their appearance and infusion. To further analyze their sensory properties, a sensory evaluation was conducted to assess the aroma characteristics of the four varieties. According to the expert panel, the aroma profiles of HJC black tea were predominantly characterized by sweet, floral, and fruity notes. However, variations in intensity resulted in distinct sensory profiles that could be perceived and differentiated through human olfactory and gustatory senses.

QDA recorded six representative aroma attributes: floral, sweet, fruity, fresh, nutty and green. The results are shown in Fig. 1B. Samples HJC1 and HJC168 exhibited high-intensity floral and fruity aromas, HJC2 showed high-intensity nutty attributes, and HJC18 had high sweet attributes. Notably, HJC1 displayed a significantly higher intensity of

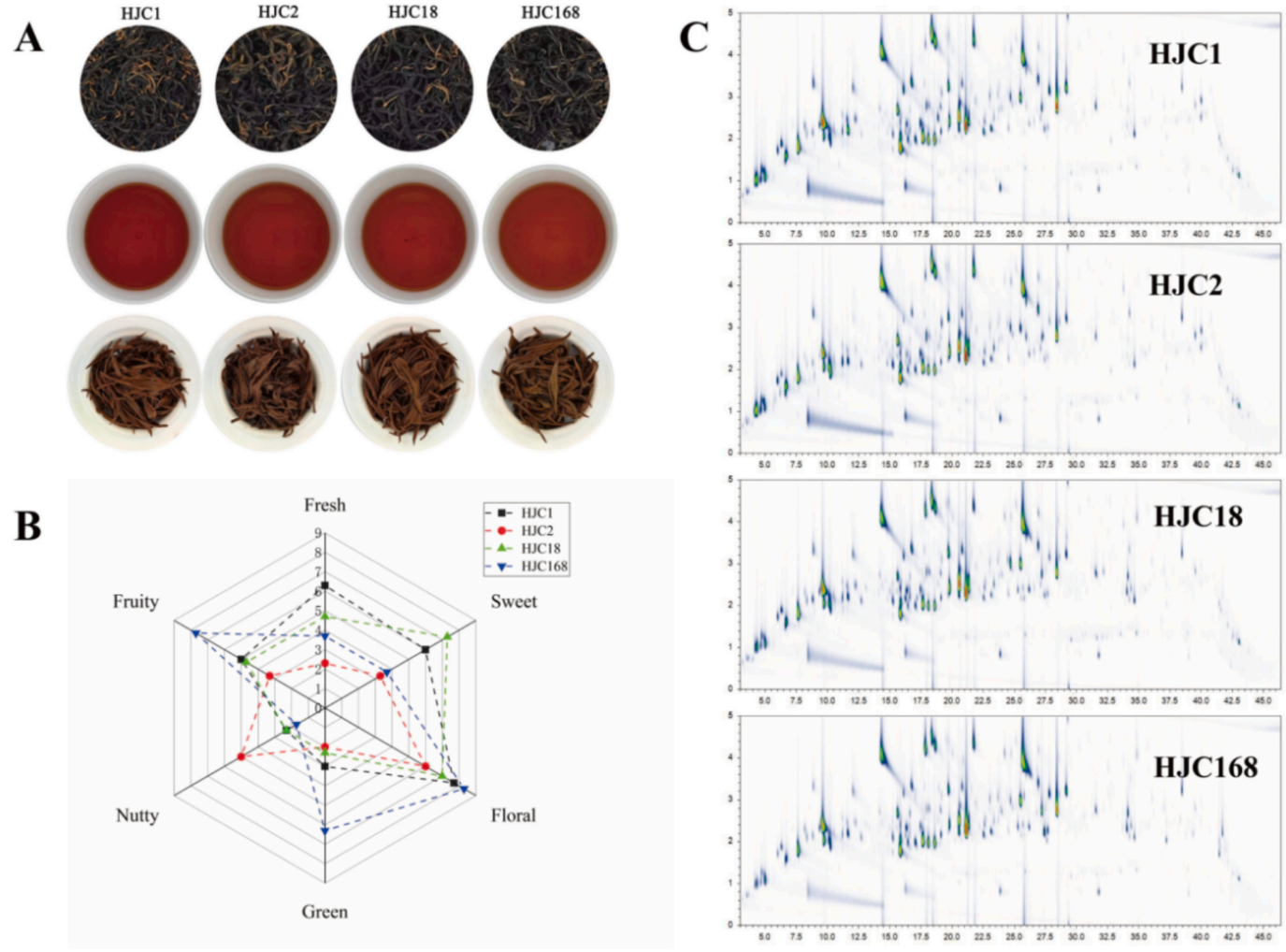

Fig. 1. Representative appearance and infusion of black tea from four cultivars(A); Spider plot of black tea aroma characters (B); The representative total ion chromatography (TIC) plots of black tea from four varieties in 2D mode (C). (HJC1: Huangjincha1 black tea, HJC2: Huangjincha2 black tea, HJC18: Huangjincha18 black tea, HJC168: Huangjincha1 black tea,)

the fresh attribute compared to the other three samples, and HJC168 had a prominent green attribute. These differences may be related to the tea cultivars.

3.2. Volatile compounds identified in HJC black tea using HS-SPME-GC–MS

The volatile components of various HJC black tea samples were analyzed using HS-SPME/GC × GC-QTOFMS and identified based on their mass spectra and retention index (RI) values (Table S1). The chromatograms of various black tea varieties are presented in Fig. 1C. While the volatile components of the black tea samples were compositionally similar, their concentrations varied significantly, highlighting substantial differences in volatility among the samples. A total of 271 volatile compounds were identified across the four samples, including 87 hydrocarbons, 54 aldehydes, 41 esters, 37 alcohols, 28 ketones, 14 heterocyclic compounds, 4 nitrogenous compounds, 3 sulfurous compounds, and 3 phenols. Specifically, 202, 190, 205, and 208 compounds were detected in HJC1, HJC2, HJC18, and HJC168, respectively.

The differences in the relative contents of various compounds in black tea samples from different cultivars of HJC can be shown in Fig. 2 AB. The major volatile compounds identified were alcohols (26.53–32.00 %), followed by aldehydes (29.42–38.83 %), hydrocarbons (12.54–17.82 %), and esters (7.14–15.81 %), collectively accounting for more than 80 % of the total volatile compounds in black tea. Previous studies have shown that alcohols and aldehydes are the primary aroma compounds in Hunan black tea (Yin et al., 2022), and contribute floral, sweet and fruity attributes. Aldehydes represent the highest proportion of volatiles in most black teas, mainly originating from lipid oxidation and Strecker degradation (Zhai et al., 2022), while the presence of other compounds contributes to a rich aroma hierarchy. Alcohols are prevalent in tea and can exist in both free form and as glycosides. The hydrolysis of glycoside-bound volatiles during black tea processing plays a crucial role in enhancing the aroma of tea (Ma et al., 2022).

In addition, the top 10 volatile compounds and their corresponding proportions are depicted in Fig. 2 D–G. Methyl salicylate exhibited the highest proportion in HJC18 (9.08 %) and HJC168 (10.60 %), followed by linalool and cyclohexanal. However, in HJC1, geraniol (7.36 %) was the most abundant, followed by cyclohexanal (6.78 %) and linalool (5.95 %). In HJC2, benzaldehyde (7.97 %) was the most abundant, followed by linalool (7.66 %) and methyl salicylate (5.81 %). Geraniol is one of the most abundant aroma compounds in black tea (Yu et al., 2023). It is a naturally occurring monoterpene alcohol that imparts a pleasant floral and fruity flavor to the tea leaves (Huang et al., 2022). The presence of geraniol in tea is influenced by various factors, including the specific type of tea leaves, processing techniques, growing conditions, and the time of year when the leaves are harvested. (E)-2-Hexenal, primarily produced in torn tea leaves, is notable for its pungent aroma, blending vegetable-like and green-fruit characteristics. It plays a prominent role in the aromatic profiles of black, white, and oolong teas and is classified as having a high aroma character impact, underscoring its importance in imparting distinctive and highly regarded olfactory qualities (Kunishima et al., 2016). Linalool is considered a key aroma compound in black tea, contributing floral and fruity notes, and it possesses a relatively low odor threshold of 3.8 μg/kg. Methyl salicylate, another characteristic component of black tea with a fresh, green aroma (Kang et al., 2019), was found in the highest concentrations in HJC18 (9.08 %) and HJC168 (10.60 %), potentially contributing to their fresh and invigorating qualities. Interestingly, methyl salicylate is also a key aromatic component in Ceylon black tea, known for its mint-like scent. Benzaldehyde, known for its nutty attributes, was most abundant in HJC2 and is a key aroma-active compound in Assam, Keemun, and Ceylon black teas (Kang et al., 2019). These results indicate significant differences in the volatile content among the HJC black tea varieties.

3.3. Volatile compound comparative analysis between varieties

Based on the overall analysis of volatile compounds, differential compounds were identified to elucidate the reasons for the aroma differences among the tea samples. A Venn plot was initially used to examine the differences in volatile compounds, revealing that 142 compounds were shared by both teas, while 15 were unique to HJC1, 13 to HJC2, 11 to HJC18, and 18 to HJC168 (Fig. 2C). To further explore the differences and similarities in the volatile components across the four HJC black tea cultivars, unsupervised principal component analysis (PCA) (Fig. 3A) and hierarchical cluster analysis (HCA) (Fig. 3B) were performed using the identified volatile compound data. The results of PCA (PC1: 45.8 %; PC2: 25.3 %; $R^2X = 0.97$) and HCA showed that samples HJC1, HJC2, HJC18, and HJC168 were distinctly separated

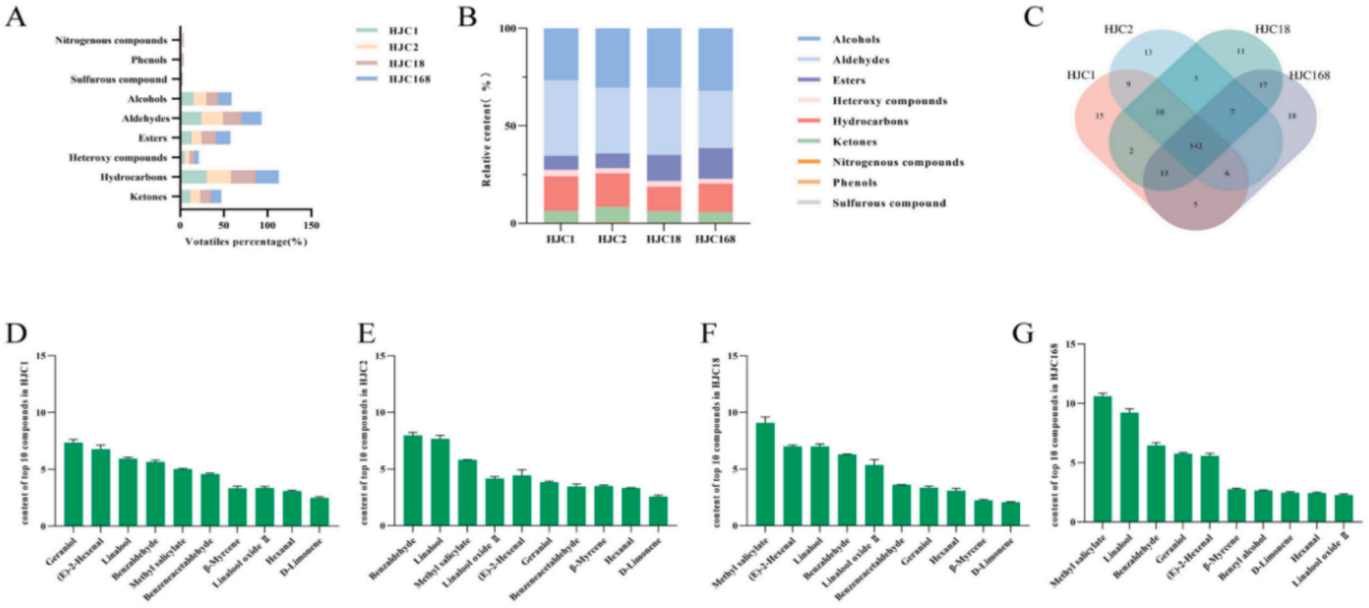

Fig. 2. The quantity distribution of different types of volatile compounds in HJC black tea(A); The content distribution of different types of volatile compounds in HJC black tea(B); Venn plot(C); The contents of top10 compounds in HJC black tea of four cultivars (D-G).

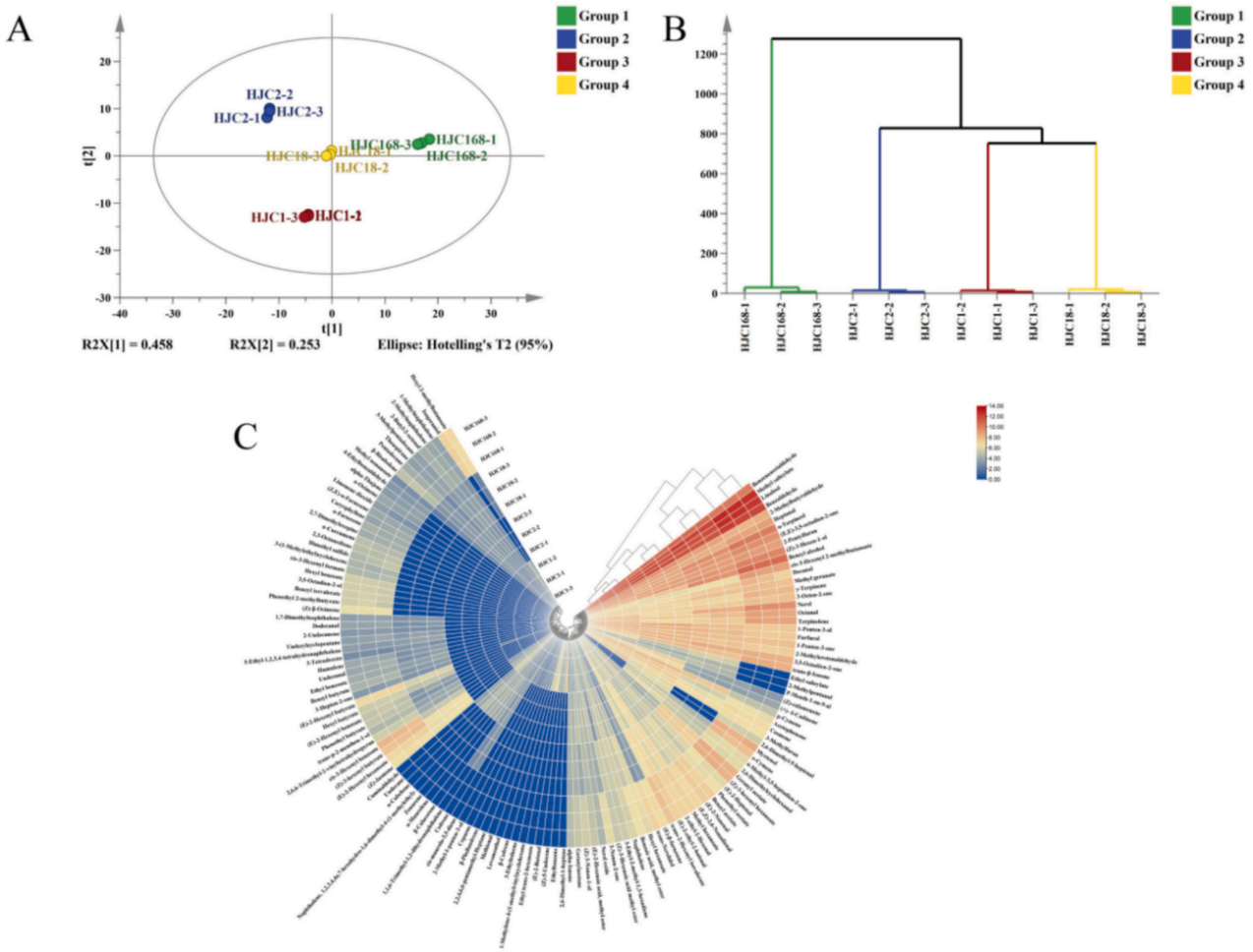

Fig. 3. PCA(A); HCA(B); Heatmap of significantly differential volatile compounds(C).

based on their volatile profiles, suggesting significant differences among them. Building on these findings, supervised orthogonal partial least squares discriminant analysis (OPLS-DA) was applied to further investigate the differences in aroma components. The OPLS-DA model analysis ($R^2Y = 0.981$, $Q^2 = 0.998$) confirmed significant differences in the distribution of volatile compounds among the various HJC black tea cultivars. Furthermore, a total of 136 distinct compounds were identified through significance testing ($p < 0.05$). These compounds likely contribute to the formation of distinct aroma profiles and help differentiate the HJC black tea cultivars. Specifically, 30 key volatile compounds were most abundant in HJC1, 13 in HJC2, and 6 and 87 differential metabolites were most abundant in HJC18 and HJC168, respectively. Notably, HJC1 is characterized by higher concentrations of hydrocarbons and aldehydes, including p-cymene, 2,6-dimethyl-1-heptene, and benzeneacetaldehyde, which likely contribute to its distinctive aroma. Similarly, hydrocarbons, aldehydes, and alcohols such as cosmene, (*Z*)-calamenene, (+)-δ-cadinene, 2-methylcrotonaldehyde, and 1-penten-3-ol found in HJC2 significantly enhance its unique aroma profile. The increased presence of aldehydes, ketones, esters, and heterocyclic compounds, such as 3,5-octadien-2-one, 3-ethyl-2-methyl-1,3-hexadiene, 2-pentylfuran, and (Z)-3-hexenoic acid methyl ester, may also contribute to the characteristic aroma of HJC18. The distinctive aroma of the HJC168 tea sample may be attributed to the presence of various volatile compounds, including esters, aldehydes, alcohols, and ketones such as methyl salicylate, cis-3-hexenyl 2-methylbutyrate, benzaldehyde, octanal, linalool, benzyl alcohol, (E, E)-3,5-octadien-2-one, and trans-β-ionone. Nevertheless, further research is needed to scientifically validate the specific roles of these volatile components in influencing the overall aroma of each tea sample.

3.4. Key aroma compounds identified by OAV in HJC black tea

The contribution of volatile compounds to the aroma characteristics of tea is closely related to their concentration levels and corresponding odor perception thresholds (Yin et al., 2022). Studies have shown that compounds with an OAV greater than 1 are considered to be aroma-active compounds (Chen et al., 2023). In this study, 105 volatile compounds exhibited an OAV exceeding 1, while 46 substances had an OAV below 1(Table S2). Among these, 36 aroma compounds with an OAV greater than 100 were shared by the four different varieties of HJC black tea. These compounds include hexanal, 3-methylbutanal, sulcatone, (E)-2-hexenal, 1-octen-3-ol, furfural, β-myrcene, benzaldehyde, D-limonene, decanal, ethyl hexanoate, α-ionone, damascenone, trans-β-ionone, acetophenone, octanal, heptanal, (E)-2-nonenal, 2-heptanone, (E)-2-heptenal, (Z)-4-heptenal, linalool, citral, α-terpineol, styrene, 2-methylbutyraldehyde, nonanal, benzeneacetaldehyde, α-pinene, geraniol, toluene, 1-penten-3-one, (E,Z)-2,6-nonadienal, (E)-2-pentenal, 1-heptanol and naphthalene. These compounds significantly contribute to the base aroma of HJC black tea.

Combined with OPLS-DA, a total of 53 compounds (VIP > 1, $p < 0.05$, OAV > 1) were identified as key odorants in four different cultivars of HJC black tea (Table 1). These compounds include 2 sulfurous compounds, 8 ketones, 11 hydrocarbons, 14 aldehydes and 7 alcohols (Fig. 4). The elevated concentrations of these aroma compounds likely play a crucial role in the formation of aroma characteristics among the various HJC black tea cultivars. Consequently, these differentiating

Table 1
The primary odorants found in four varieties of Huangjincha black tea.

NO	Compounds	OTs (μg/kg)	OAV				Odor Characteristics	Concentration (ng/g)			
			HJC1	HJC2	HJC18	HJC168		HJC1	HJC2	HJC18	HJC168
1	α-Ionone	0.0004	22,438.02	54,544.5	42,371.52	65,105.46	apple, floral, fruit, violet	8.98	21.82	16.95	26.04
2	Decanal	0.0026	25,335.55	40,919.99	65,197.41	81,532.94	fat, floral, fried, orange peel	65.87	106.39	169.51	211.99
3	o-Cymene	0.004	–	9932.34	5788.74	58,000.73	smoke, wood	–	39.73	23.15	232
4	trans-β-Ionone	0.007	18,673.35	34,490.96	29,225.35	50,564.84	cedarwood, dry, floral, raspberry, seaweed	130.71	241.44	204.58	353.95
5	Dimethyl sulfide	0.0075	–	–	–	3864.16	cabbage, organic, sulfur, wet earth	–	–	–	28.98
6	Furfural	0.008	36,760.71	36,359.84	31,170.29	21,180.68	almond, baked potatoes, bread, candy, floral	294.09	290.88	249.36	169.45
7	Acetophenone	0.01	15,980.18	15,893.2	4982.66	8321.48	almond, animal, flower, plastic	159.8	158.93	49.83	83.21
8	Octanal	0.015	14,700.79	10,989.72	14,537.36	29,153.99	citrus, fat, fruit, green, nut	220.51	164.85	218.06	437.31
9	(E)-2-Nonenal	0.025	2090.41	1255.29	2409.34	6282.93	cucumber, cut grass, fat, green	52.26	31.38	60.23	157.07
10	Ethylbenzene	0.026	888.62	–	–	–	strong	23.1	–	–	–
11	(E)-2-Heptenal	0.034	1510.8	1005.98	1762.34	2183.11	almond, fat, fruit, metal, soap	51.37	34.2	59.92	74.23
12	Methyl nonanoate	0.04	93.62	140.87	118.81	157.72	coconut, floral, fruit, sweet	3.74	5.63	4.75	6.31
13	Methional	0.06	106.57	–	–	–	cooked potato, earth, potato, roast, soy	6.39	–	–	–
14	(E)-2-Butenal	0.067	240.04	–	–	–	flower	16.08	–	–	–
15	Benzaldehyde	0.085	30,652.56	46,163.67	35,406.35	51,419.85	almond, berry, bitter, bitter almond, burnt sugar	2605.47	3923.91	3009.54	4370.69
16	3-Ethyltoluene	0.088	439.79	–	–	–	fragrant	38.7	–	–	–
17	(E,Z)-2,6-Nonadienal	0.2	247.15	173.36	253.11	557.67	cucumber, green, lettuce, melon, wax	49.43	34.67	50.62	111.53
18	Naphthalene	0.2	156.98	221.3	238.88	262.24	mothball, tar	31.4	44.26	47.78	52.45
19	Heptanal	0.26	2709.52	1710.81	1236.91	1745.78	citrus, dry fish, fat, green, nut	704.48	444.81	321.6	453.9
20	Benzoic acid, methyl ester	0.52	30.62	50.66	79.37	169.82	herb, lettuce, prune, sweet, violet	15.92	26.34	41.27	88.31
21	Ethyl benzoate	0.62	–	–	17.87	25.12	camomile, celery, fat, flower, fruit	–	–	11.08	15.58
22	Undecanal	0.7	–	–	12.09	21.22	citrus, fat, oil, pungent, sweet	–	–	8.46	14.86
23	α-Terpineol	0.86	670.6	434.7	856.92	1357.42	floral, fresh, lily, mint, oil	576.71	373.84	736.95	1167.38
24	1-Penten-3-one	1	284.67	240.14	223.19	213.9	green, herb, metal, mustard, pungent	284.67	240.14	223.19	213.9
25	2-Methylbutyraldehyde	1.3	626.86	532.53	349.8	232.44	almond, chocolate, cocoa, fermented, hazelnut	814.92	692.29	454.75	302.17
26	2-Methylpentanal	1.6	17.74	10.45	14.08	–	green	28.38	16.72	22.53	–
27	(Z)-Jasmone	1.9	8.19	–	47.56	50.5	floral	15.57	–	90.36	95.95
28	1,1,6-Trimethyl-1,2-dihydronaphthalene	2.5	1.99	3.05	2.45	–	licorice	4.97	7.61	6.13	–
29	2-Undecanone	3	–	–	2.2	2.87	fresh, green, orange, pineapple, rose	–	–	6.6	8.6
30	2-Methylnaphthalene	3	2.77	2.42	3.58	4.98	moss, mothball, pungent, root	8.3	7.26	10.75	14.95
31	Linalool	3.8	721.43	991.81	884.6	1642.23	bergamot, coriander, floral, flower, grape	2741.44	3768.9	3361.5	6240.48
32	Benzeneacetaldehyde	4	529.81	424.08	432.68	300.78	berry, floral, flower, geranium, honey	2119.26	1696.31	1730.73	1203.12
33	2-Pentylfuran	6	56.95	77.34	84.71	84.59	butter, floral, fruit, green, green bean	341.69	464.01	508.25	507.54
34	3-Octen-2-one	6.7	17.81	19.5	23.2	31.4	crushed bug, dull, green, nut, rose	119.31	130.64	155.46	210.35
35	Myrtenol	7	7.07	9.52	–	48.51	mint, cool	49.48	66.62	–	339.57
36	p-Cymene	7.2	37.43	35.93	24.75	4.66	citrus, fresh, gasoline, solvent, wood	269.52	258.68	178.19	33.56
37	1-Methylnaphthalene	7.5	0.95	0.88	1.19	1.6	antiseptic, hay, mothball, pleasant	7.13	6.62	8.89	11.96
38	Geranyl acetate	9	2.68	2.35	16.88	36.96	lavender, rose, sweet	24.08	21.17	151.91	332.65
39	Benzyl isovalerate	10	–	–	–	4.58	fruit	–	–	–	45.83
40	1-Penten-3-ol	10	31.02	37.71	22.54	16.67	burnt, fish, grass, green, meat	310.19	377.07	225.42	166.71

(*continued on next page*)

Table 1 (*continued*)

NO	Compounds	OTs (μg/kg)	OAV				Odor Characteristics	Concentration (ng/g)			
			HJC1	HJC2	HJC18	HJC168		HJC1	HJC2	HJC18	HJC168
41	2,6-Dimethyl-5-heptenal	16	12.44	4.12	–	2.97	citrus, fruit, green, melon, sweet	199.01	65.9	–	47.48
42	Phenethyl acetate	20	2.18	2.43	2.04	3.87	floral, fruit, honey, rose, tobacco	43.57	48.54	40.86	77.34
43	Hexyl 2-methylbutanoate	22	0.45	–	0.21	4.98	strawberry	10.01	–	4.59	109.57
44	Benzyl acetate	30	0.81	1.33	2.14	4.66	boiled vegetable, fresh, fruit, honey, jasmine	24.45	39.77	64.22	139.94
45	(Z)-β-Ocimene	34	–	–	–	2.3	citrus, flower, herb, mold	–	–	–	78.08
46	Methyl salicylate	40	57.92	71.48	109.08	179.27	almond, caramel, fresh, medicine, mint	2316.77	2859.13	4363.09	7170.61
47	Terpinolene	41	4.98	2.65	4.08	11.32	pine, plastic, sweet	204.07	108.66	167.41	464.05
48	(Z)-3-Hexen-1-ol	70	4.27	7.37	8.75	13.13	bell pepper, grass, green, green leaf, herb	298.92	515.86	612.75	918.76
49	Methyl hexanoate	70	0.54	1.56	1.59	1.91	fresh, fruit, pineapple, sweet	37.79	109.37	111.53	133.72
50	Ethyl salicylate	84	1.37	0.37	0.6	–	flower, fruit, mint, sweet, wintergreen	114.68	31.26	50.28	–
51	(E,E)-3,5-octadien-2-one	100	5.1	3.88	5.91	7.1	almond, balsamic, dill, sweet, vanilla	510.14	387.79	591.34	709.88
52	Benzyl alcohol	100	1.12	1.8	7	17.93	almond, boiled cherries, chemical, floral, fruit	111.82	180.09	700.17	1792.93
53	Nerol	290	0.96	0.44	1.23	2.56	floral, fruit, rose, sweet	277.07	126.24	356.8	741.07

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

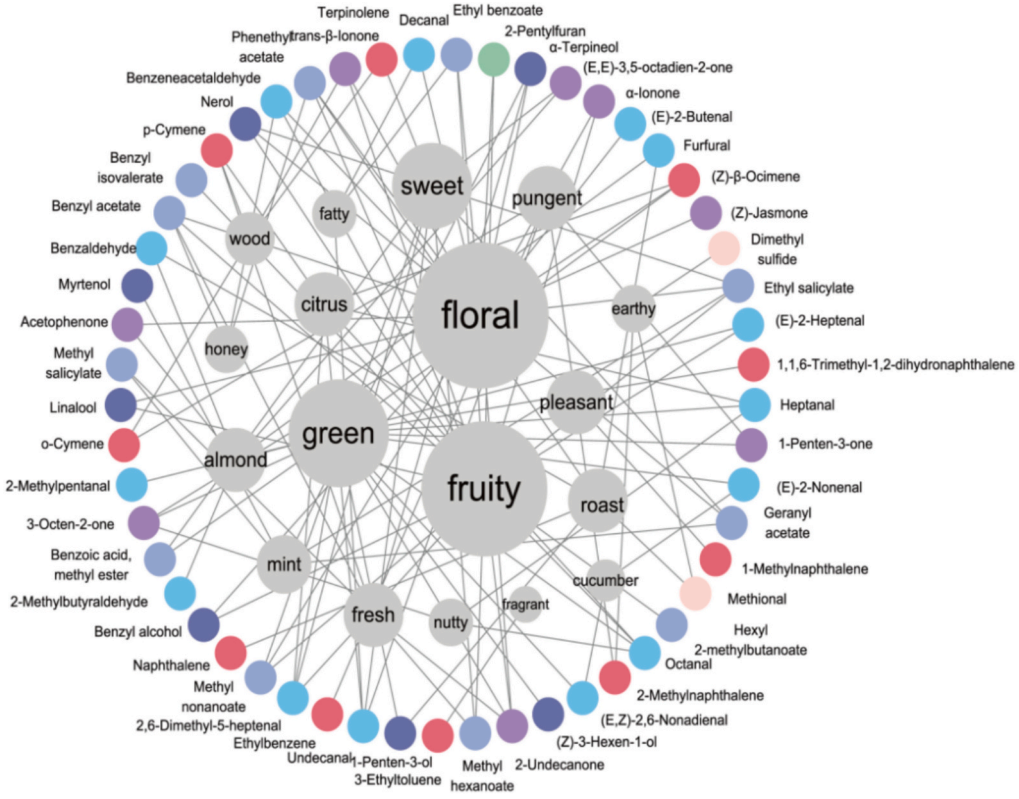

Fig. 4. The correlation network between sensory attributes and differential aroma-active compounds of black tea with different cultivars.

aroma compounds were considered key aroma contributors across different HJC black tea cultivars. Notably, 13 aroma compounds were identified as differentiating odorants with high levels in the HJC1 sample, including benzeneacetaldehyde, 2-methylbutyraldehyde, heptanal, furfural, 1-penten-3-one, ethyl salicylate, acetophenone, 2-methylpentanal, 2,6-dimethyl-5-heptenal, 3-ethyltoluene, ethylbenzene, (E)-2-butenal and methional. Previous studies have shown that benzeneacetaldehyde, known for its floral and fruity characteristics, is a critical aroma compound in the world's four most renowned black tea varieties (Kang et al., 2019). Our study revealed that the content of benzeneacetaldehyde in HJC1 was significantly higher compared to HJC2, HJC18, and HJC168, suggesting its potential contribution to the floral and fruity aroma profile of HJC1. 2-methylbutyraldehyde is the primary contributor to the aroma differences among black teas with varying degrees of fermentation (Y. Yang et al., 2024), imparting notes of almond, chocolate, and cocoa. Heptanal serves as a principal odorant

in chestnut (Zhu et al., 2018) and fresh green teas (Q. Wang et al., 2024), imparting fresh, green aroma attributes. The content of (E)-2-butenal decreased following the fixation process in Longjing Tea (Wang et al., 2020), and it exhibits floral aroma characteristics. Notably, (E)-2-butenal was exclusively detected in HJC1, suggesting its presence as a trace aroma-active compound that may contribute to the characteristic aroma profile of HJC1 black tea.

For the HJC2 sample, seven key odorants were identified—benzaldehyde, 1-penten-3-ol, p-cymene, phenethyl acetate, α-ionone, 1,1,6-trimethyl-1,2-dihydronaphthalene, and methyl nonanoate, which were found to be more abundant in the HJC2 sample compared to the other three samples. Benzaldehyde has been reported as a key aroma compound for distinguishing the aroma profiles of black teas from different regions within Guangdong (Liu et al., 2021), it also serves as an important marker for changes in the aroma quality of black tea during the withering process (H. Wang, Wang et al., 2022). P-cymene, known primarily for its citrus and fresh aroma characteristics, serves as a key aroma-active compound in Rucheng 'Baimaocha' (Chen et al., 2023). In addition, phenethyl acetate, α-ionone, 1,1,6-trimethyl-1,2-dihydronaphthalene and methyl nonanoate exhibited fruity odors, suggesting their potential contribution to the fruity characteristics of HJC2.

In the HJC18 sample, 13 contributing compounds exhibited the highest levels compared to the other three samples and were identified as distinguishing odorants. These compounds include (*Z*)-3-hexen-1-ol, 2-pentylfuran, (E,E)-3,5-octadien-2-one, α-terpineol, 3-octen-2-one, methyl hexanoate, decanal, naphthalene, (E)-2-heptenal, (Z)-jasmone, ethyl benzoate, undecanal, and 2-undecanone. Among these, (Z)-3-hexen-1-ol, derived from fatty acids and exhibiting a green odor, has been identified as an important aroma-active compound in Chinese Gongfu black tea (Ho et al., 2015). 2-pentylfuran, characterized by its fruity attributes, is recognized as a key odorant in green, oolong, and black teas (Guo et al., 2022; Kang et al., 2019). Decanal, a compound with floral characteristics, shows a significant increase in concentration when green tea broth is treated with β-glucosidase, thereby enhancing its floral attributes (T. Zhang et al., 2020).

For sample HJC168, 20 differentiating odorants were found at the highest levels, including linalool, methyl salicylate, trans-β-ionone, benzyl alcohol, octanal, nerol, terpinolene, myrtenol, benzyl acetate, o-cymene, (E,Z)-2,6-nonadienal, (E)-2-nonenal, benzoic acid methyl ester, geranyl acetate, 2-methylnaphthalene, 1-methylnaphthalene, hexyl 2-methylbutanoate, dimethyl sulfide, (Z)-β-ocimene and benzyl isovalerate. Linalool is an important naturally occurring terpene alcohol and is the main contributing volatile compound to the floral and sweet aroma in black tea (Zhang, Sun, et al., 2023). In addition, methyl salicylate, a volatile organic compound with a wintergreen-mint odor, plays an important role in the formation of sweet floral aroma in tea and has been shown to be a major aroma component of Keemun black tea (Kang et al., 2019). Similarly, trans-β-ionone, benzyl alcohol, octanal and nerol are also common floral volatile compounds in black tea and contribute significantly to the floral attributes of HJC168 black tea (Zhang, Zhou, et al., 2023).

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

Gas Chromatography-Olfactometry (GC-O) technology combines the analytical power of gas chromatography with the discriminatory capabilities of human sensory olfaction, allowing for the precise identification and characterization of aroma-active compounds in complex mixtures. This technique has become widely adopted in flavor analysis, particularly for meat products, nuts, flavorings, fragrances, dairy items, tea, and various other food products (Mahmoud & Zhang, 2024). Currently, GC-O is widely recognized as an effective technique for screening aroma-active components in tea (Ma et al., 2022). The variations in the compound content among different HJC black tea samples were significant. However, these elevated concentrations did not consistently result in a notable impact on the overall aroma profile of tea. By utilizing GC-O analysis in conjunction with relevant odor database information (accessible at https://olfab.iiita.ac.in/olfactionbases), we conducted a thorough evaluation of the olfactory attributes associated with the compounds in the samples. Subsequently, 51 compounds with significant aroma activity in HJC black tea were identified and classified based on their unique odor characteristics. The outer circle visually represents the odors of various aroma-active compounds, while the inner circle distinguishes odor characteristics using color coding, as shown in Fig. 5. Notably, most compounds exhibited floral, fruity, and green odor characteristics. The identification results of 51 major aroma-active compounds across the four tea samples are presented in Table S3, primarily including aldehydes, alcohols, ketones, and esters.

In the HJC1 samples, geraniol exhibited the highest aroma intensity (4.67) with a floral scent, making it a principal contributor to the aroma quality of HJC1. Research has demonstrated that geraniol is a significant monoterpene alcohol in tea, with its accumulation varies markedly among different tea plant cultivars. In tea plants, geraniol primarily exists in the form of glycosides, which are catalyzed by endogenous glycoside hydrolases, leading to the release of free geraniol during processing (D. Wang et al., 2000). Additionally, the pleasant-smelling compounds benzeneacetaldehyde (floral, 4.33), linalool (floral, 4.33), trans-β-ionone (floral, 3.67), and sulcatone (mushroom, 4.33) also exhibit high aroma intensities. During the processing of tea, particularly fermentation and drying, phenylalanine undergoes decarboxylation and oxidative deamination, producing benzeneacetaldehyde (Guo et al., 2022). This compound considered critical for contributing to the floral and sweet aromas characteristic of HJC1 (Kang et al., 2019). Furthermore, trans-β-ionone, generated through the oxidative degradation of β-carotene during drying (Yin, Kong, et al., 2022), is identified as a key olfactory component in HJC1, owing to its significantly greater aromatic impact compared to HJC2, HJC18, and HJC168.

In the HJC2 sample, benzaldehyde (4.33), 3-methylbutanal (4.33), and 1-octen-3-ol exhibited the highest aroma intensities, followed closely by methyl salicylate (3.67), hexanal (3.67), and (Z)-4-heptenal (3.67). Benzaldehyde and 3-methylbutanal, both characterized by an almond-like odor, showed aroma intensities exceeding those of HJC1, HJC18, and HJC168, establishing them as the primary odorants in HJC2. 1-octen-3-ol is a critical aroma compound in Taiping Houkui (H. Zhou et al., 2022) and Longjing teas (Wang et al., 2020) and may contribute to the aged and woody aroma in the storage process of dark teas (Shen et al., 2023). Additionally, 1-penten-3-ol and 2-heptanone, both with a grassy odor (Q. Li et al., 2020), were found to have high aroma intensities in HJC2 and could be the key aroma compounds contributing to the green character of HJC2 black tea. Moreover, (Z)-4-heptenal, noted for its roasted aroma, is a prominent aroma-active compound in Keemun black tea (Huang et al., 2022).

Among the HJC18 samples, linalool (4.67), methyl salicylate (4.33), geraniol (4.33), benzeneacetaldehyde (4.33) and damascenone (4.33) exhibited high aroma intensities. Notably, linalool and geraniol, two important monoterpene alcohols in tea, share a common biosynthetic precursor, geranyl pyrophosphate (GPP), yet possess distinct aroma profiles (Wang et al., 2020). Furthermore, Additionally, damascenone is known for its strong odor and very low odor threshold (Kinoshita et al., 2010). It is a critical component of the honey-like aroma in Xinyang black tea (Yao et al., 2023) and contributes to the distinctive sweet aroma of Lichuan black tea (Qin et al., 2023), suggesting that it may play a significant role in the unique sweet aroma of HJC18 black tea.

HJC168 exhibited a higher aroma intensity and a greater number of aroma-active compounds compared to HJC1, HJC2, and HJC18. Specifically, within HJC168, methyl salicylate had the highest aroma intensity (4.67), followed by linalool (4.33), hexanal (4.33), α-ionone (4.33), citral (4.33), benzaldehyde (4.00), (E)-2-hexenal (3.67), β-myrcene (3.67), (E,Z)-2,6-nonadienal (3.67), α-terpineol (3.67), octanal (3.67), heptanal (3.67), (E)-2-nonenal (3.67), o-cymene (3.67) and styrene (3.67). Methyl salicylate, known for its characteristic

Fig. 5. Aroma-active compounds detected using a GC-O sniffing port. The inner circle shows the collection of the corresponding odor characteristics of the compounds in the outer circle, while the outermost displays the aroma compounds that GC-O smelled.

wintergreen and mint odors, is considered a primary component of the aroma profile of Ceylon black tea (Kang et al., 2019). Citral is essential for the sweet aroma of black tea, appropriate kneading can enhance the citral content in stripped green tea, thereby enhancing its floral and fruity attributes (Y. Yang et al., 2024). (E)-2-nonenal is a principal aroma component responsible for the chestnut-like aroma in green tea, imparting a nutty and slightly fatty aroma (Zhu et al., 2018). It is also a significant contributor to the aroma of jasmine tea (Zhang, Zhou, et al., 2023), influencing the aroma characteristics of beaded green tea (Wang et al., 2020). O-cymene is a typical constituent in fermented tea that provides a woody aroma attribute (X. Wang et al., 2023).

3.6. The comparison of the key aroma compounds in HJC black tea obtained by OAV and GC-O methods

A substantial number of volatile compounds have been identified in tea science research. However, only a subset of these volatiles has been recognized as key aroma-active components that significantly influence the overall aroma profile of tea (Zhai et al., 2022). While GC-O combined with OAV has been utilized to identify these key aroma-active compounds in four varieties of HJC black tea, the GC-O method carries a degree of subjectivity due to inter-individual variability among assessors (P. Yang et al., 2021). Furthermore, OAV calculations, which quantify the contribution of compounds to the overall aroma based on their concentration relative to their odor thresholds, can be influenced by experimental conditions, limiting the accuracy and reproducibility of results (Li et al., 2013). Given the complementary nature of GC-O and OAV techniques in aroma analysis, it is essential to integrate both methods and further validate and refine their findings through additional experimental designs and technical approaches.

The compounds identified in HJC1 via GC-O and OAV analysis included geraniol, linalool, sulcatone, benzeneacetaldehyde, nonanal, trans-β-ionone, methyl salicylate, hexanal, α-ionone, benzaldehyde, (E)-2-hexenal, and β-myrcene, indicating that these compounds play a

significant role in HJC1 aroma formation. Notably, benzaldehyde had a notably high aroma intensity and the highest concentration in HJC1, suggesting that it exerts a major influence on the aroma of HJC1. Similarly, the common compounds identified in HJC2 using both methods included benzaldehyde, 3-methylbutanal, 1-octen-3-ol, methyl salicylate, hexanal, (Z)-4-heptenal and 1-penten-3-ol. The OAV of 3-methylbutanal in HJC2 was significantly higher than in other tea samples, making it one of the most characteristic odorants in HJC2. Additionally, linalool, methyl salicylate, geraniol, benzeneacetaldehyde, damascenone, (E, E)-2,4-hexadienal, 3-methylbutanal, hexanal, sulcatone, and α-ionone were common in HJC18, where damascenone and (E, E)-2,4-hexadienal exhibited significantly higher aroma intensities compared to other tea samples. Likewise, methyl salicylate, linalool, hexanal, α-ionone, citral, benzaldehyde, (E)-2-hexenal, β-myrcene, (E, Z)-2,6-nonadienal, α-terpineol, octanal, (E)-2-nonenal, heptanal, o-cymene and styrene were identified in HJC168 by both GC-O and OAV, with methyl salicylate showing the highest aroma intensity and concentration, making it the key aroma-active compound in HJC168.

3.7. Relationship between odorants and sensory attributes in four varieties of black tea

To investigate the relationship between aroma-active substances (OAV $>$ 1) and sensory attributes, we performed a PLS model. This model encompasses two latent variables, accounting for 94.80 % of the variance in the x-matrix (sensory attributes) and 84.90 % of the variance in the y-matrix (aroma-active compounds). As illustrated in Fig.S1, the different HJC varieties were distributed across four quadrants: HJC1 was positioned in the positive region of PC2 and the negative region of PC1; HJC2 was situated in the negative region of PC2 and the positive region of PC1; HJC18 was found in the positive regions of both PC1 and PC2; and HJC168 was located in the negative regions of both PC1 and PC2. This distribution clearly indicates that the tea samples can be categorized into four distinct groups.

The qualities significantly associated with certain aroma compounds—floral, green, fresh, sweet, fruity, and nutty—were all located within the region between the inner and outer ellipses, indicating that they can be well explained by the model. Among these, floral attributes were significantly positively correlated with nonanal (VC71), (E, Z)-2,4-heptadienal (VC44), (E)-2-octenal (VC60), and (E)-2-hexenal (VC16), suggesting that these four compounds may be key contributors to the higher floral attribute of HJC168 compared to the other three HJC black teas. Nonanal, in particular, appears to be a key odorant confirming the floral aroma of green tea (Q. Wang et al., 2024). Fruity attributes were highly positively correlated with terpinolene (VC65), nerol (VC88) and α-terpineol (VC79), with α-terpineol exhibiting an upward trend during the storage process of lemon-flavored hard tea beverages (He et al., 2018). The green property was positively correlated with α-phellandrene (VC47), (*E*)-2-nonenal (VC75), (E, Z)-2,6-nonadienal (VC74), and octanal (VC46). The fresh property was positively correlated with (E)-2-pentenal (VC9), heptanal (VC27), and p-1,3,8-menthatriene (VC73). It is known that certain fatty acid derivatives in tea contribute to the fresh and green aromas (Yang et al., 2024). Sweet attributes were positively correlated with benzeneacetaldehyde (VC57), 2,6-dimethyl-5-heptenal (VC59), and 1-penten-3-one (VC5). Notably, benzeneacetaldehyde is a key aroma substance in Jinmudan black tea (Yang et al., 2024). Nutty attributes were positively correlated with 3-methylbutanal (VC3), linalool oxide I (VC64), and octane (VC12). 3-methylbutanal is a common aroma substance in barley tea (Tatsu et al., 2020) and a key contributor to the almond aroma in black tea (Kang et al., 2019).

4. Conclusion

In this study, HS-SPME combined with comprehensive GC × GC-Q-TOFMS was employed to analyze the aroma components and key odorants of four cultivars of HJC black tea. A total of 271 volatile compounds were identified by GC × GC-Q-TOFMS, with aldehydes and alcohols comprising the primary volatile components of the different varieties of HJC black tea. PCA and OPLS-DA revealed that the volatile components of the four varieties of HJC black tea could be distinctly separated, and 39 differential volatile compounds were identified as potential key aroma compounds. Through OAV analysis, 53 key aroma substances were identified from the four HJC varieties, including 3-methylbutanal, sulcatone, (E)-2-hexenal, 1-octen-3-ol, benzaldehyde, damascenone, trans-β-ionone, linalool, citral, benzeneacetaldehyde, geraniol, 1-penten-3-one, (E, Z)-2,6-nonadienal and others. GC-O results indicated that HJC1, HJC2, HJC18, and HJC168 contained 39, 42, 44, and 50 aroma actives, respectively. Furthermore, a comprehensive evaluation combining OAV and GC-O suggested that geraniol and benzeneacetaldehyde (HJC1), 3-methylbutanal and 1-penten-3-ol (HJC2), (E, E)-2,4-hexadienal and damascenone (HJC18), methyl salicylate and citral (HJC168) were likely the most clearly identified aroma compounds in each variety. By partial least squares (PLS) analysis, nonanal, (E, Z)-2,4-heptadienal, (E)-2-octenal, and (E)-2-hexenal were recognized as important contributors to floral attributes. Terpinolene, nerol, α-terpineol, and (E)-β-ocimene were identified as significant contributors to fruity attributes. α-phellandrene, (E)-2-nonenal, (E,Z)-2,6-nonadienal, and octanal were considered key contributors to green attributes. (E)-2-pentenal, heptanal, and p-1,3,8-menthatriene were deemed important contributors to fresh attributes. Benzeneacetaldehyde, 2-methylbutyraldehyde, and 1-penten-3-one were noted as key contributors to sweet attributes. Finally, 3-methylbutanal, linalool oxide I, and octane were identified as significant contributors to nutty attributes. These results enhance our understanding of the key odorants related to different cultivars of HJC black tea and provide a theoretical foundation for controlling and improving the aroma quality of tea.

CRediT authorship contribution statement

Ronggang Jiang: Writing – original draft, Visualization, Software, Formal analysis, Data curation. **Jian Ouyang:** Software, Methodology, Investigation, Data curation. **Hongyu Chen:** Writing – review & editing, Formal analysis. **Xinyi Zhang:** Software, Methodology, Formal analysis. **Hao Xu:** Methodology, Formal analysis. **Kuofei Wang:** Supervision, Methodology, Data curation. **Yun Peng:** Resources, Formal analysis. **Jinhua Chen:** Supervision, Software, Methodology, Investigation. **Zhonghua Liu:** Supervision, Methodology, Funding acquisition, Conceptualization.

Declaration of competing interest

We declare that we have no commercial or financial interest that represents a conflict of interest in connection with the work submitted.

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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.2025.102426.

Data availability

Data will be made available on request.

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