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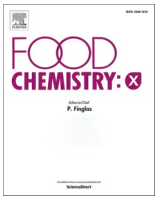

Revealing the differences in aroma of black tea under different drying methods based on GC–MS, GC-O

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ABSTRACT

Drying greatly affects the aroma of black tea. In this study, the differences in aroma of black tea under hot-air drying (HD), sun drying (SD), and pan-fired drying (PD) were investigated through quantitative descriptive analysis. Headspace solid-phase microextraction and solvent assisted flavor evaporation combined with gas chromatography–mass spectrometry and gas chromatography-olfactory were used to analyze the overall aroma profile of black tea. Aroma extract dilution analysis and odor activity values revealed that 15 aroma-active compounds led to differences in aroma, namely linalool, geraniol, phenylethyl alcohol, phenylacetaldehyde, (*Z*) -linalool oxide (furanoid), *β*-damascenone, dimethyl sulfide, methional, 2-methylbutanal, 3-methylbutanal, methyl salicylate, *β*-myrcene, hexanal, 1-octen-3-ol, and heptanal. Among them, geraniol, linalool, and methional significantly enhanced the floral and roasty aroma of HD, while hexanal enhanced the green aroma of SD. Finally, our results were validated through aroma recombination and addition experiments. This study provides a theoretical basis for improving the aroma of black tea.

1. Introduction

Black tea is the most popular type of fermented tea in the world, accounting for more than 78 % of global tea consumption because of its unique qualities and multiple health benefits (Li et al., 2022; Wang, Qin, et al., 2023; Wang, Shen, et al., 2022). Aroma as a key factor in measuring the quality of black tea, to a large extent, affects the selectivity and acceptability of tea drinkers (Lu et al., 2022). Black tea is produced in four important processes: withering, rolling, fermentation, and drying. Among them, drying, as the final processing step, plays a crucial role in the formation of the final aroma of black tea (Ma et al., 2024). Drying is a process of dehydration and passivation of enzymes, high temperature thermochemistry makes the volatile compounds significantly dissipated, on the other hand, the oxidative degradation, isomerization, and Maillard reactions generated by high temperature heating, finally forming the extremely harmonious and complex aroma of black tea (Chen et al., 2024; Wan, 2003).

Drying changes the categories and concentrations of volatile compounds in tea and thereby considerably affects the aroma of tea (Ye et al., 2023). In a previous study, Wang, Su, et al. (2022) studied the effects of various drying methods on the aged aroma of fermented Pu-erh tea (ripe tea) through GC–MS and odor activity values (OAV), which is defined as the ratio of the concentration of individual volatile compound to its odor threshold in water. The results indicated that dryer-drying at 60 °C increased the medicinal aroma of the tea and that sun and shade drying increasing the relative concentrations of volatile compounds. Zhang et al. (2023) studied the effect of Lu'an Gua Pian on tea aroma under different over-fired methods, and identified 12 aroma-active compounds through OAV. The results showed that pulley charcoal drying significantly enhanced the floral and fruity aroma. These existing studies have shown the importance of drying in the formation of aroma in different types of tea, and these experimental methods also provide valuable references for our subsequent experiments. The current research on black tea drying mainly focuses on the differences between black tea in traditional hot air drying and thermal radiation drying or hot roll drying, and also on the changes in black tea quality under different drying temperatures (90–140 °C) (Qu et al., 2019; Su et al., 2024; Ye et al., 2021). With the continuous expansion of the tea market, a new drying technology—sun drying has emerged, which has also led to differences in tea quality. Current studies on the quality of sun-dried

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black tea have only used headspace solid-phase microextraction (HS-SPME) and gas chromatography–mass spectrometry (GC–MS) to analyze it, and have not yet used systematic sensoryomics to decode the molecular characteristics of its aroma (Lv et al., 2016).

This study used quantitative descriptive analysis (QDA), HS-SPME, and solvent assisted flavor evaporation (SAFE) combined with GC–MS and GC-olfaction (GC-O) to investigate the aroma quality of black tea dried by traditional mainstream hot-air drying (HD) and pan-fired drying (PD) methods, as well as the emerging sun drying (SD) method. The aroma characteristics of the three drying methods were compared, and key aroma-active compounds were screened. In conclusion, this study elucidated the effects of different drying methods on the characteristics of black tea and provided a theoretical basis for improving the aroma quality.

2. Materials and methods

2.1. Tea samples collection

In March 2023, tea leaves (one bud and one or two leaves) were collected from large-leaf tea trees at Da Bai Tian tea industry Co., Ltd. in Fengqing County (Lincang City, Yunnan Province, China). After the leaves withered (temperature: 13–18 °C, humidity: 70 %–80 %, 12 h), they were rolled for 1 h (6CR- 65, Zhejiang Wuyi Zengrong Food Machinery Co., Ltd., Zhejiang, China), fermented in a fermentation room (temperature: 23–29 °C, humidity: 75 %–95 %, 8–9 h). The HD was dried in a louver dryer (6CHB-12, Zhejiang Wuyi Wanda Drying Equipment Manufacturing Co., Ltd., Zhejiang, China) with a crude fire at 110–120 °C for 10–15 min and a refire at 90–100 °C for 15–20 min. The SD was sun-dried at 20–34 °C, a light intensity of 82–150 Klux for 6 h and windless, with flipping once approximately every 30 min. The PD was dried using a band-forming machine (6CCB-80/12, Ya'an Chuangyu Machinery Co., Ltd., Sichuan, China) with a crude fire at 180–250 °C for 25 min and a refire at 90–95 °C for 30 min. Obtain the final tea sample with a water content <7 % according to T/CTSS 38–2021 Dianhong Gongfu Black Tea, and store it in a refrigerator (4 °C) until analysis is conducted.

2.2. Chemicals and materials

All chemical reagents and materials were displayed in the Table S1. For GC, the purity of all aroma-active compounds was greater than 95 %.

2.3. Tea brewing and QDA

Briefly, 3.0 g of each tea sample was weighed and subsequently brewed in 150 mL of purified boiling water. Following a 5-min period, tea infusion was filtered using 400-mesh gauze and transferred it into brown snuff bottles. Ten trained panelists (six women and four men, aged 22–30) evaluated the aroma of each tea infusion after SD, HD, and PD through QDA. All panelists were trained on perceiving aromas once a week for at least 2 months, with the training involving smelling objects with different aromas and describing the characteristics of these aromas. After they evaluated the aromatic characteristics of all tea samples, they selected the six characteristics that predominantly represented each tea infusion: floral, fruity, sweet, green grassy, minty, and roasty. Tea sensory evaluation, recombination experiments, and addition experiments were scored using a 5-point scale based on previous research (where 0 = absent; 1.0 = very weak; 2.0 = weak; 3.0 = moderate; 4.0 = strong; and 5.0 = very strong) (Yin et al., 2022). All experiments were conducted in a quiet environment with no external aromas. The mean values are presented as radar plots.

2.4. Extraction of volatile compounds by HS-SPME

HS-SPME was selective for volatile compounds, depending on the type of fiber used, and it is more suitable for extracting high-volatility and low-molecular-weight compounds (Tan et al., 2019; Zhang et al., 2023). HS-SPME was based on the method proposed by Liu et al. (2023), and some modifications have been made. Briefly, tea infusions were brewed as outlined in Section 2.3 and were subsequently subjected to rapid cooling. Subsequently, each tea infusion of 10 mL was moved into a 20 mL spiral headspace flask, and then 10 μL of 10 mg/L ethyl decanoate was added as an internal standard, along with 3.0 g of NaCl. Finally, the headspace flask was kept in a water bath at 30 °C for 15 min, followed by extraction with an SPME needle for 30 min.

2.5. Extraction of volatile compounds by SAFE

SAFE was typically used to extract low-volatile and high-molecular-weight compounds from tea, and it retains the original characteristics of black tea (Tan et al., 2019; Zhang et al., 2023). SAFE was based on the method proposed by Huang et al. (2022), and some modifications have been made. Briefly, 7 μL of 1000 mg/L ethyl decanoate was incorporated as an internal standard to 150 mL of tea infusion and then processed through a SAFE device at 40 °C in a vacuum environment (10^{-3} Pa). Following a triple extraction of the distillate using 30 mL of distilled dichloromethane, it was dehydrated using anhydrous sodium sulfate and then condensed to 100 μL at 25 °C with a steady flow of nitrogen.

2.6. GC–MS analysis of volatile compounds

Volatile compounds subjected to SPME and SAFE were separated and identified using an 8890B—5977B GC–MS (Agilent, CA, USA) equipped with an HP-5MS capillary column (30 m × 0.25 mm × 0.25 μm) (Agilent, CA, USA), and helium as the carrier gas (purity >99.99 %). SPME analysis was conducted in splitless injection mode, and SAFE analysis was conducted with a 3-min solvent postponement and a 1 μL volume of injection. The GC oven was operated in Table S2. Operating in a positive electron-ion mode, the mass selective detector functioned within a mass scanning spectrum of *m/z* 30–350 at 70 eV. A sequence of C_6–C_{40} *n*-alkanes was used to derive the linear retention index.

2.7. Aroma-active compounds obtained using GC-O and aroma extract dilution analysis (AEDA)

GC-O analysis was conducted in accordance with a method proposed by Huang et al. (2022). Briefly, an 8890B—5977B GC–MS equipped with an olfactory detection port was employed to separate and identify volatile compounds in tea. The outlet of analytical column was split evenly, directing half of the gas into the mass spectrometer (250 °C) and the other half into the sniffing port (230 °C). As the carrier gas, helium was employed at a steady speed of 40 cm/s. The samples were injected using two methods into the gas chromatograph. For SPME, the tip of the SPME fiber was placed in the inlet port of the gas chromatograph and maintained in place for 5 min. For SAFE, 2 μL of the SAFE fraction was injected into the gas chromatograph's inlet. The samples were injected into the inlet of the gas chromatograph using a splitless mode. Conditions for the gas chromatograph's temperature and mass spectrometer matched those outlined in Section 2.6.

AEDA on HS-SPME and SAFE was carried out by three trained evaluators (one man and two women) to determine the intensity of the aroma and the effect of each compound. For HS-SPME, the aroma activity was diminished by altering the GC separation ratio (1:1, 3:1, 7:1, etc.) until the olfactory detection port revealed no scent; for SAFE, the distillates were progressively diluted with dichloromethane at a volume ratio of 1:1. For each series of dilutions, GC-O was carried out in varying concentrations, from high to low, until the olfactory detection port became odorless. Finally, a flavor dilution (FD) value was assigned to each identified aromatic compound.

2.8. Quantitative analysis of volatile compounds and calculation of OAVs and ACIs

Internal standard quantification: the concentration of ethyl decanoate were employed to quantitate the concentrations of volatile compounds without real standards. External standard quantification: a standard curve, derived from various established levels of standard compounds, was employed to quantitate the concentrations of aroma-active compounds. The calculation of OAVs was based on the proportion of each aroma-active compound's concentration in water relative to its specific threshold value. Aroma-active compounds with OAVs $\geq$1 were considered to enhance to the aroma characteristics of the tea (Wang et al., 2020).

Aroma characteristics impact (ACI): the ratio of the OAV of volatile compounds to the sum of the OAVs of all key volatile components (Chen et al., 2022).

2.9. Aroma recombination and addition experiments

A recombination experiment was conducted by Wei et al. (2024). In short, aroma-active compounds with OAVs $\geq$1 in HD, SD, and PD tea infusions were recombined in 25 mL of deionized water based on their quantitate concentrations. As described in Section 2.3, a control group (original tea infusion) was established and placed in stoppered 50 mL brown triangular bottles. Both aroma recombination and control samples were heated in a water bath at 40 °C. As indicated in Section 2.3, QDA was conducted on all samples to evaluate the intensity of each aroma.

An addition experiment was conducted as described by Huang et al. (2022). The addition experiment was performed by using the highest concentration of the key differential actives screened in the three black tea samples as the addition standard. The differential concentrations were added to two low content tea extracts to keep the concentration of key aroma active compounds at the same level. After standard aromatic compounds were added, QDA was conducted again on the three samples of tea infusions.

2.10. Statistical analysis of data

The principal component analysis (PCA) and hierarchical cluster analysis (HCA) were performed using SIMCA-P V14.1 (Umetrics, Umea, Sweden). The analysis of variance (ANOVA) utilized the least significant difference (LSD) test within IBM SPSS Statistics v.26.0 (IBM, Armonk, NY, USA). $p < 0.05$ was considered to indicate a significant difference. The visualization of the data was conducted through Origin 2022 (OriginLab, MA, USA). All tests were conducted thrice and the results were presented as mean ± standard deviation.

3. Results and discussions

3.1. Aromatic characteristics of black tea subjected to three drying methods

QDA provides a clearer description of the aroma characteristics of tea in terms of accuracy. In the present study, we conducted a sensory QDA on six aroma types of three tea samples, followed by analysis of variance on the results. As presented in Fig. 1, significant differences were observed in the floral ($p < 0.01$) and roasty ($p < 0.01$) and green ($p < 0.05$) aromas among three groups of samples. Specifically, the HD exhibited enhanced floral and roasty aromas, the SD exhibited enhanced green and weakest roasty aromas, and the PD exhibited less enhanced roasty and weakest mint-like aromas. There was no significant difference in the sweet and fruity aroma of black tea under the three drying methods. These results indicate that each drying method exerted unique effects on the intensity of tea aromas. The observed effects could stem from alterations in the concentrations of certain volatile compounds in black tea after drying resulting in varied aromatic characteristics (Wu et al., 2022). These results were validated using GC–MS in the following text.

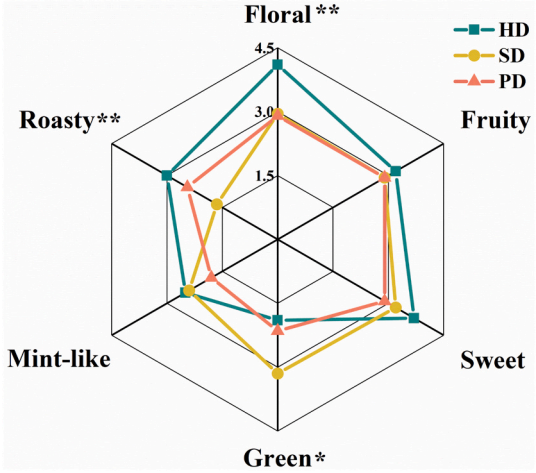

Fig. 1. QDA radar plot of aroma in black tea subjected to hot-air drying (HD), sun drying (SD), and pan-fired drying (PD). "*" significant difference ($p < 0.05$); "**", highly significant difference ($p < 0.01$).

3.2. Differences in volatile compounds profiles for each drying method

To thoroughly explain the differences observed in the aroma of black tea subjected to three drying methods, the study employed a combination of HS-SPME and SAFE to extract and analyze volatile compounds from tea. The volatile compounds extracted from the black tea subjected to HD, SD, and PD were preliminarily characterized using GC–MS and quantified against an internal standard (ethyl decanoate). The results indicate that the concentrations of all volatile compounds varied among the samples, and a total of 144 volatile compounds were identified in the three samples (Table S3). Specifically, 129, 125, and 128 volatile compounds were identified in the samples subjected to HD, SD, and PD, respectively, at relative concentrations of 3070.34, 2597.10, and 2347.55 μg/L, respectively (Fig. 2C). To enable explanation of the differences observed among the volatile compounds in black tea subjected to different drying methods, we conducted a multivariate statistical analysis (PCA and HCA) and analyzed the aforementioned volatile compounds. As presented in Fig. 2A, the first two principal components accounted for 49.3 % and 40.1 % of the total variance, respectively. The samples subjected to HD, SD, and PD were clustered into different quadrants and were well separated, indicating significant differences between them. As indicated in Fig. 2B, the samples were divided into three clusters, and the PD was further divided into separate clusters, suggesting a more pronounced disparity between the PD and other samples. These results indicate that HD and SD increased the concentrations of volatile compounds in the tea samples, a finding that is consistent with the results of an earlier research (Wang, Shen, et al., 2022). HD was dried briefly in a high-temperature enclosed environment of 110–120 °C, which minimizes the loss of volatile compounds as much as possible. Simultaneously, high-temperature heating may also facilitate the Maillard reaction, leading to the highest relative content of volatile compounds in HD. SD effectively preserved the aroma profile of its volatile compounds and also increased their relative content. The lowest concentration of volatile compounds in PD samples is likely due to the 180 °C temperature and open drying environment used in the PD process, which resulted in significant loss of volatile compounds.

Fig. 2D presents a Venn diagram of the types of volatile compounds detected in the three samples of black tea. In the three samples, 108 volatile compounds were identified in total. Four unique volatile compounds were identified in the HD, which give the tea samples a roasted

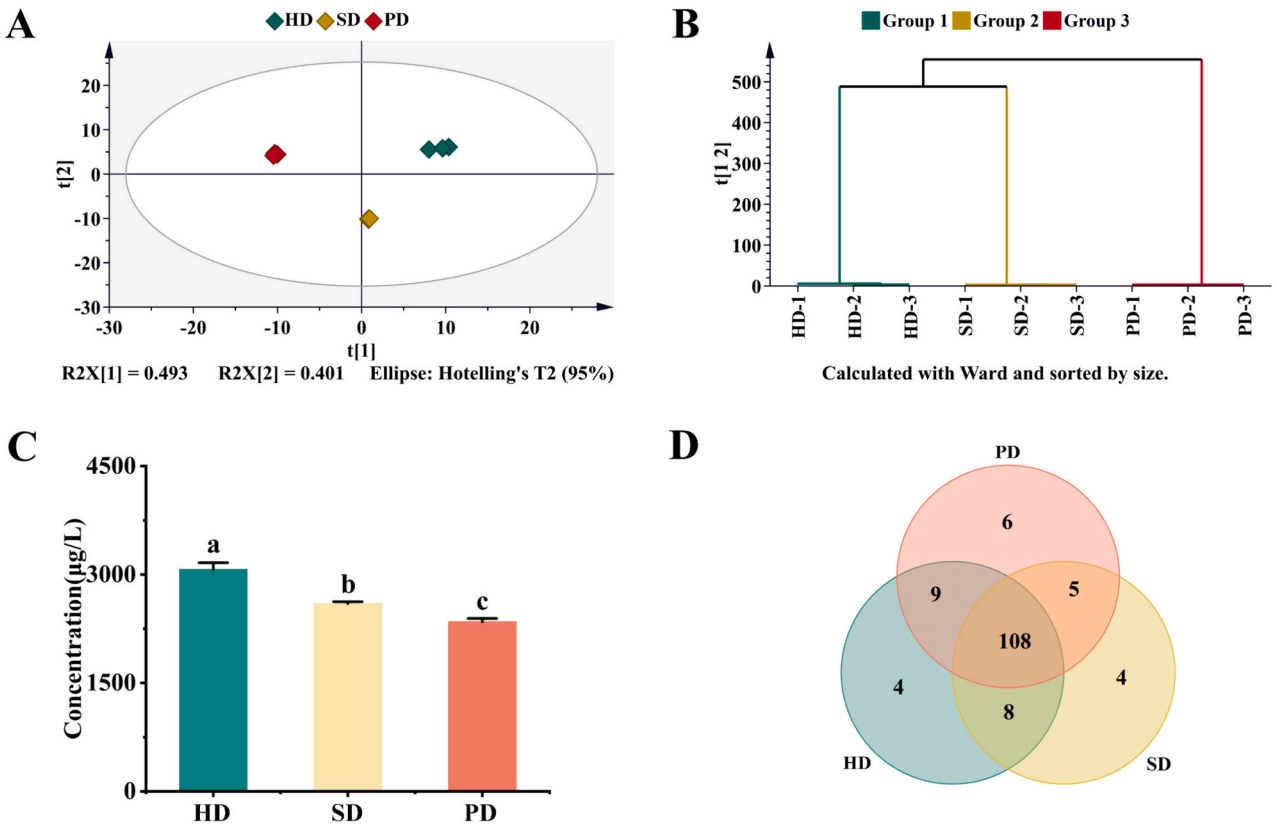

Fig. 2. Metabolic profile analysis: A. PCA results; B. HCA results; C. total relative concentrations of volatile compounds in three types of black tea; D. Venn diagram of the types of volatile compounds detected in black tea, with each number representing the number of volatile compounds in black tea.

and burnt aroma, namely 1H-pyrrole-2-carboxaldehide (burnt), 3-methyl-2-hexanone (bitter almond-like), 5-methyl-2-furancarboxaldehyde (sweet, bitter almond-like), and 3-methyl-2-butenal (sweet, nutty, almond), which are consistent with the results of QDA. Four unique volatile compounds were identified in the SD, namely α, α, 4-trimethyl-benzenemethanol (sweet, fruity), octanoic acid (vegetable, fatty), (*E*) -2-octen-1-ol (green, vegetable), and dihydroactinidolide (sweet peach). Among them, octanoic acid and (*E*) -2-octen-1-ol have a certain contribution to the green aroma of SD. Six unique volatile compounds were identified in the PD, of which five compounds had unpleasant aroma, namely (*E*) -2-hexenoic acid (musty), heptanoic acid (rancid, sweaty), 3-methyl-phenol (smoky, phenolic), benzothiazole (rubber-like, cabbage-like), and hexanoic acid, methyl ester (musty). The aroma types of volatile compounds were obtained on the website (http://www.perflavory.com/search.php).

3.3. Composition of aroma-active compounds in black tea samples subjected to HD, SD, and PD

Despite the identification of 144 volatile compounds, not all of them contribute to the aroma of black tea (Ma et al., 2021). To compare the odor and intensity of each aroma-active compound, GC-O was conducted in combination with AEDA for an analysis of the aromatic extracts of each tea sample.

GC-O analysis revealed 38 volatile compounds with an odor, which were divided into seven categories: 13 alcohols, 12 aldehydes, 4 esters, 2 heterocyclic compounds, 6 ketones, 2 acids, and 1 alkene (Table S4). These aroma-active compounds typically form as a result of the hydrolysis of glycosides and the degradation of carotenoids, lipids, and amino acids (Wang et al., 2017). Due to the low concentration of aromatic active compounds with FD $<$ 32, we were unable to accurately describe their aromatic characteristics (Huang et al., 2022). Therefore, 19 compounds with FD $\geq$ 32 were selected for further analysis (Table 1).

The aroma of black tea is primarily characterized by the presence of alcohols, which are its key volatile compounds (Ma et al., 2022). Among the alcohol aromatic compounds identified in the current study, six had an FD $\geq$ 32, namely linalool, geraniol, phenylethyl alcohol, (*Z*) -linalool oxide (furanoid), 1-octen-3-ol, and *endo*-borneol. Among the three samples, geraniol and linalool have the highest FD values, with geraniol having an FD value of 256 in HD and linalool having an FD value of 128 in SD and PD. Typically, monoterpenes (such as linalool and geraniol) and aromatic alcohols (like phenylethyl alcohol) result from the enzymatic hydrolysis of glycosidic bonds. Hydrolyzing glycosidic conjugates is crucial in regulating the aroma of black tea during processing. Linalool and geraniol are released from geranyl, a precursor of pyrophosphate, by geraniol synthase and linalool synthase, respectively. Linalool is characterized by a floral and woody aroma, and geraniol is characterized by a typical rose aroma (Li et al., 2024; Lin et al., 2022). Linalool oxide could result from the hydrolysis of glycosides, or it might originate from oxidation in the drying phase (Ma et al., 2022). Linalool, linalool oxide, geraniol, and phenylethyl alcohol are the main sources of black tea floral and fruity aromas, and are the main reasons for the more pronounced floral and fruity aromas in HD. In SD, 1-octen-3-ol and endo-borneol showed larger FD values. Biosynthesis of 1-octen-3-ol, responsible for the distinct mushroom and fish-like aroma in green and dark tea (Wen et al., 2023), involves oxidizing linoleic acid by lipoxygenase (LOX) or dioxygenase (DOX), and then hydroperoxide lyase cleaves the intermediate hydroperoxide (Tasaki et al., 2019). Endo-borneol is also a monoterpene alcohol, mainly producing earthy and moldy odors.

Six aromatic aldehydes had an FD $\geq$ 32, namely phenylacetaldehyde, hexanal, heptanal, methional, 2-methylbutanal, and 3-methylbutanal. 3-Methylbutanal and hexanal have the highest FD value of 64 in SD, while 2-methylbutanal, heptanal, and methanal have the highest FD value of 32 in HD. Phenylacetaldehyde has the same FD value in HD and

Table 1
The most odor-active volatiles (FD ≥ 32) in the aroma distillates of black tea infusions.

No.	RI[a]	Odorants[b]	FD[c]			Odor quality[d]	Identification basise[e]	Sources[g]
			HD[f]	SD[f]	PD[f]			
1	<600	Dimethyl sulfide	64	32	64	corn, sulfury	RI,MS,O,STD	SPME
2	629	3-Methyl- butanal	16	64	4	malty	RI,MS,O,STD	SPME
3	641	2-Methyl- butanal	32	16	4	malty	RI,MS,O,STD	SPME
4	801	Hexanal	32	64	16	green, grassy	RI,MS,O,STD	SAFE
5	903	Heptanal	32	16	4	fatty, nutty	RI,MS,O,STD	SAFE
6	907	Methional	32	8	16	cooked potato-like	RI,MS,O,STD	SAFE
7	981	1-Octen-3-ol	16	64	8	mushroom-like, green	RI,MS,O,STD	SAFE
8	988	6-Methyl-5-hepten-2-one	16	32	2	citrus, green, musty, lemongrass, apple	RI,MS,O,STD	SPME
9	993	*β*-Myrcene	16	32	2	sweet	RI,MS,O,STD	SPME
10	1074	(*Z*)-linalool oxide (furanoid)	64	8	32	floral	RI,MS,O,STD	SAFE
11	1051	Phenylacetaldehyde	32	32	16	floral, honey-like	RI,MS,O,STD	SAFE
12	1100	Linalool	128	64	128	citrus-like, floral	RI,MS,O,STD	SPME
13	1120	Phenylethyl alcohol	64	64	8	floral, honey-like	RI,MS,O,STD	SAFE
14	1168	*endo*-Borneol	16	32	8	earthy, moldy	RI,MS,O,STD	SAFE
15	1203	Methyl salicylate	64	64	4	mint-like	RI,MS,O,STD	SAFE
16	1263	Geraniol	256	128	128	rose-like, citrus-like	RI,MS,O,STD	SAFE
17	1388	*β*-Damascenone	128	128	128	cooked apple-like	RI,MS,O,STD	SPME
18	1483	(*E*)-*β*-Ionone	64	32	64	floral, violet-like	RI,MS,O,STD	SAFE
19	1538	Dihydroactinidolide	0	32	0	sweet, floral	RI,MS,O,STD	SPME

Note: [a] Retention index (RI), calculated from the retention time of each compound with a homologous series of n-alkanes (C_6–C_{40}) separated using an HP-5MS capillary column. [b] Odorants identified in the three black tea infusions. [c] FD: Flavor dilution of odorants determined using the HP-5MS column. [d] Odor quality of each odorant at the sniffing port. [e] Methods of identification: MS, odorants were identified by mass spectrum; RI, retention indices; O, olfactometry; and Std, authentic standards. [f] Three processing methods for black tea: hot-air drying (HD), sun drying (SD), and pan-fired drying (PD). [g] Sources of identification.

SD, both of which are 32. Aldehydes have lower FD values in PD. Phenylacetaldehyde, methional, 2-methylbutanal, and 3-methylbutanal are primarily volatile aldehydes produced by the Strecker reaction of amino acids, which involves temperature and enzymatic catalysis (Zhang et al., 2023). Phenylacetaldehyde, 2-methylbutanal, and 3-methylbutanal are responsible for the floral and sweet aroma of tea, and methional is responsible for the roasty aroma of tea (Huang et al., 2022). Unsaturated fatty acids are precursors of aromatic compounds C_6 aldehydes and alcohols in the production process of black tea, and this reaction also depends on temperature and enzyme catalysis (Ma et al., 2022). Hexanal and heptanal derived from lipid degradation have FD ≥ 32. Hexanal has a green grassy odor, and heptanal has a fatty, herbal, and nutty odor (Zhu et al., 2018). These compounds together enrich the aroma of tea.

Three identified aromatic ketones had an FD ≥ 32, namely *β*-damascenone, (*E*) -*β*-ionone, and 6-methyl-5-hepten-2-one. The FD value of *β*-damascenone is 128 in three drying methods, which is produced by the degradation of neoxanthin and is a key sweetener for tea infusions (Wei et al., 2024). The FD value of (*E*) -*β*-ionone is highest at 64 in HD and PD, and 32 in SD. It primarily results from the oxidation and cleavage of carotenoids with enzymatic catalysis or thermal degradation, and it considerably contributes to the floral aroma of tea infusions (Wu et al., 2022). In addition, 6-methyl-5-hepten-2-one has the highest FD of 32 in SD, and its formation is due to the oxidation and degradation of unsaturated fatty acids, including linoleic acid and linolenic acid. In the present study, its FD value was higher in the SD, mainly catalyzed by enzyme activity in SD samples, which contributes to the green aroma of tea infusions (Wang et al., 2024).

Two identified aromatic esters had an FD ≥ 32, namely methyl salicylate and dihydroactinidolide. The FD value of methyl salicylate is 64 in both HD and SD, and the lowest in PD. Methyl salicylate, found everywhere in plants, results from the enzymatic degradation of phenylalanine, which results in the esterification of salicylic acid. Although heat treatment promotes this chemical reaction, excessive heat may induce ester cracking, resulting in the formation of carboxylic acids (Wang, Yu, et al., 2023), which may be a reason underlying the low concentration of methyl salicylate in the present study's PD infusions. Dihydroactinidindolide is only smelled in SD, with an FD value of 32. Prolonged calcination at high temperatures may cause further oxidation of (*E*) -*β*-ionone, resulting in *β*-carotene derivatives of dihydroactinidiolide (Ma et al., 2023), which primarily contribute to the development of a sweet peach aroma in SD.

One identified aromatic compound involving an alkene and a sulfide has an FD ≥ 32, namely *β*-myrcene and dimethyl sulfide. *β*-Myrcene, a volatile monoterpene with a highest FD value of 32 in SD. It is biosynthesized in the plastids through the methylerythritol phosphate pathway, presenting a sweet aroma (Yang et al., 2024). Dimethyl sulfide is formed by the decomposition of *S*-methylmethionine during tea processing and drying, mainly influenced by thermal effects (Lin et al., 2016), which may explain why, in current research, the concentration of dimethyl sulfide was higher in the tea samples subjected to HD and PD than in those subjected to SD.

3.4. Screening of key differential aroma-active compounds in black tea subjected to three drying methods

Because the FD value associated with AEDA is easily influenced by the shunt concentration, it is typically used as a preliminary method. Given that the concentrations of aroma-active compounds alone are insufficient to assess whether compounds substantially contribute to the aroma of tea, this study used an external standard curve to quantify aroma compounds absolutely (Table S5), and then screened aroma active compounds by calculating OAV ≥ 1, and evaluated the contribution level of aroma active compounds using ACI (Chen, Yang, et al., 2022). As a result, a total of 16 aroma-active compounds were screened, including linalool, geraniol, phenylethyl alcohol, phenylacetaldehyde, (*Z*) -linalool oxide (furanoid), (*E*) -*β*-ionone, *β*-damascenone, dimethyl sulfide, methional, 2-methylbutanal, 3-methylbutanal, methyl salicylate, *β*-myrcene, hexanal, 1-octen-3-ol, and heptanal (Table 2). The aroma-active compounds were subjected to significant difference analysis ($p < 0.05$) in the three samples, except for (*E*) -*β*-ionone, the other 15 aroma compounds had a significant impact on the differences among the three samples. Therefore, these 15 aromatic compounds were identified as differential characteristic compounds resulting from the different drying methods (Fig. 3).

Among the samples treated with HD, geraniol had the highest OAV of 598. Similar to geraniol and linalool and their oxides, phenylacetaldehyde and phenylethyl alcohol have a floral aroma. Among them, geraniol (31.11 %) and linalool (10.10 %) have the highest ACI values and are the main contributing compounds to the floral aroma of HD. The highest OAV value of *β*-damascenone in HD is 416, followed by PD of

Table 2
Concentrations, odor thresholds, and odor activity value of key aroma active substances in black tea.

No.	Compounds	Ots[a]	Concentration(μg/L)[b]			OAV[c]			ACI[d]		
			HD	SD	PD	HD	SD	PD	HD	SD	PD
1	Dimethyl sulfide	0.3[α]	20.66 ± 2.19[a]	7.91 ± 0.04[c]	13.13 ± 2.68[b]	68.87 ± 7.32[a]	26.35 ± 0.12[c]	43.78 ± 8.95[b]	3.58 %	1.49 %	3.08 %
2	*β*-Damascenone	0.006[β]	2.50 ± 0.02[a]	2.42 ± 0.02[c]	2.46 ± 0.01[b]	416.91 ± 2.77[a]	404.11 ± 3.67[c]	410.32 ± 2.37[b]	21.69 %	22.82 %	28.90 %
3	2-Methyl- butanal	1[α]	3.47 ± 0.26[b]	2.37 ± 0.07[c]	4.40 ± 0.53[a]	3.47 ± 0.26[b]	2.37 ± 0.07[c]	4.40 ± 0.53[a]	0.18 %	0.13 %	0.31 %
4	3-Methyl- butanal	1.1[α]	5.06 ± 0.43[a]	3.51 ± 0.03[b]	4.57 ± 0.55[a]	4.60 ± 0.39[a]	3.19 ± 0.03[b]	4.15 ± 0.50[a]	0.24 %	0.18 %	0.29 %
5	*β*-Myrcene	1.2[α]	20.34 ± 0.76[c]	39.68 ± 3.12[a]	35.47 ± 1.02[b]	16.95 ± 0.63[c]	33.07 ± 2.60[a]	29.56 ± 0.85[b]	0.88 %	1.87 %	2.08 %
OAV of compounds with sweet aroma											
6	1-Octen-3-ol	1.5[α]	9.56 ± 0.70[b]	24.78 ± 3.47[a]	4.81 ± 0.47[c]	6.37 ± 0.47[b]	16.52 ± 2.32[a]	3.20 ± 0.31[c]	0.33 %	0.93 %	0.23 %
7	Hexanal	5[α]	79.60 ± 10.17[b]	134.75 ± 13.86[a]	41.99 ± 2.20[c]	15.92 ± 2.03[b]	26.95 ± 2.77[a]	8.40 ± 0.44[c]	0.83 %	1.52 %	0.59 %
8	Methyl salicylate	40[α]	507.37 ± 75.51[a]	601.79 ± 41.53[a]	282.06 ± 22.62[b]	12.68 ± 1.89[a]	15.04 ± 1.04[a]	7.05 ± 0.57[b]	0.66 %	0.85 %	0.50 %
OAV of compounds with green aroma											
9	(*Z*)-linalool oxide (furanoid)	100[α]	165.24 ± 33.54[a]	150.76 ± 22.70[a]	70.93 ± 8.20[b]	1.65 ± 0.34[a]	1.51 ± 0.23[a]	0.71 ± 0.08[b]	0.09 %	0.09 %	0.05 %
10	Geraniol	1.1[α]	657.78 ± 116.99[a]	605.77 ± 103.32[a]	402.87 ± 22.51[b]	597.98 ± 106.35[a]	550.70 ± 93.93[a]	366.24 ± 20.47[b]	31.11 %	31.09 %	25.80 %
11	Phenylacetaldehyde	5.2[γ]	471.88 ± 65.50[a]	429.10 ± 86.21[a]	235.18 ± 30.55[b]	90.75 ± 12.60[a]	82.52 ± 16.58[a]	45.23 ± 5.87[b]	4.72 %	4.66 %	3.19 %
12	Linalool	2.7[β]	524.20 ± 38.95[a]	464.76 ± 71.56[ab]	375.71 ± 32.93[b]	194.15 ± 14.42[a]	172.13 ± 26.50[ab]	139.15 ± 12.20[b]	10.10 %	9.72 %	9.80 %
13	Phenylethyl alcohol	562.3[α]	556.04 ± 73.26[a]	452.74 ± 92.25[a]	258.64 ± 45.22[b]	0.99 ± 0.13[a]	0.81 ± 0.16[a]	0.46 ± 0.08[b]	0.05 %	0.05 %	0.03 %
14	(*E*)-*β*-Ionone	0.007[α]	2.99 ± 0.13[a]	3.00 ± 0.54[a]	2.39 ± 0.20[a]	427.15 ± 19.26[a]	429.08 ± 77.78[a]	341.57 ± 28.32[a]	22.22 %	24.23 %	24.06 %
OAV of compounds with floral aroma											
15	Heptanal	2.8[α]	12.23 ± 0.46[a]	8.55 ± 0.48[b]	1.73 ± 0.13[c]	4.37 ± 0.16[a]	3.05 ± 0.17[b]	0.62 ± 0.05[c]	0.23 %	0.17 %	0.04 %
16	Methional	0.45[α]	26.74 ± 1.86[a]	1.70 ± 0.17[c]	6.67 ± 0.95[b]	59.43 ± 4.13[a]	3.78 ± 0.37[c]	14.81 ± 2.12[b]	3.09 %	0.21 %	1.04 %
OAV of compounds with roasty aroma											

Note: [a] Odor thresholds (OTs) in water from the following sources: α (Gemert, 2011); β (Wei et al., 2024); γ (Zhang et al., 2023). [b] Mean values of triplicates with standard deviations (SDs). Three types of processing for black teas: hot-air drying (HD), sun drying (SD), and pan-fired drying (PD). [c] OAVs were calculated by dividing the concentrations by the respective odor thresholds. [d] ACIs were calculated by the ratio of the OAV of volatile compounds to the OAV of all key volatile components.

410, and the lowest in SD. Similar to dimethyl sulfide, 3-methylbutanal, 2-methylbutanal, and *β*-myrcene, *β*-damascenone is a compound that contributes to the sweetness of black tea, resulting in the most pronounced sweetness in HD, followed by sweetness in PD (Yang et al., 2024; Zhou et al., 2023). In addition, methional and heptanal are both compounds that exhibit a roasty aroma. Among them, methional has the highest ACI value content of 3.09 % and is the main compound that exhibits a roasty aroma in HD samples. The OAV of methional in HD is the highest at 59, followed by PD at 15, and the lowest in SD. This results in a distinct roasty aroma in HD and a weaker roasty in PD, which is consistent with the QDA results (Huang et al., 2022; Zhu et al., 2018).

In tea treated with SD, hexanal exhibited the highest OAV of 27, and both it and 1-octen-3-ol have a significant impact on the green aroma of tea (Rajkumar et al., 2017). Research indicates that the concentration of methyl salicylate is strongly associated with the fresh aroma of tea (Wang, Qin, et al., 2023). Among them, the ACI value of hexanal in SD is the highest at 1.52 %, which is the main contributor to the green aroma in SD.

3.5. Analysis of aroma recombination and addition experiments

Aroma recombination plays a crucial role in analyzing and verifying the accuracy of qualitative and quantitative results of major aromatic compounds (Xiao et al., 2022). Volatile compounds identified through GC-O and OAV calculation are usually considered key aromatic compounds. In accordance with the quantitative results, 16 key aromatic compounds were added to deionized water at their calculated concentrations. Subsequently, QDA was employed to identify the sensory variances between the recombinant sample and control sample to confirm the accuracy of the recombination results. As indicated in Fig. 4A–4C, although the aroma properties of these samples are slightly different from the corresponding black tea aroma properties, their overall aroma characteristics have good similarity with the corresponding black tea ($p > 0.05$), suggesting that the aroma of these black teas has been successfully simulated. Furthermore, these differences may be attributed to the influence of non-volatile compounds on the aroma of tea or the synergistic effect between flavor compounds (Chen, Liu, et al., 2022).

To further verify the differences between the three samples, aroma addition experiments were conducted on the samples. If the highest concentration of 15 aroma components in three tea infusion is taken as the benchmark, adding the difference in aroma compound concentration in the two tea samples with low content can make the aroma characteristics of these three tea soups converge. To verify this hypothesis, we added the missing concentrations of the aromatic compounds from the three samples to the tea infusions to equalize the concentrations of the 15 aroma-active compounds and compare them with those in the original tea infusions for QDA. The results indicate that after the missing concentrations were added, the aromatic characteristics of the three tea samples were highly similar, with no significant difference noted

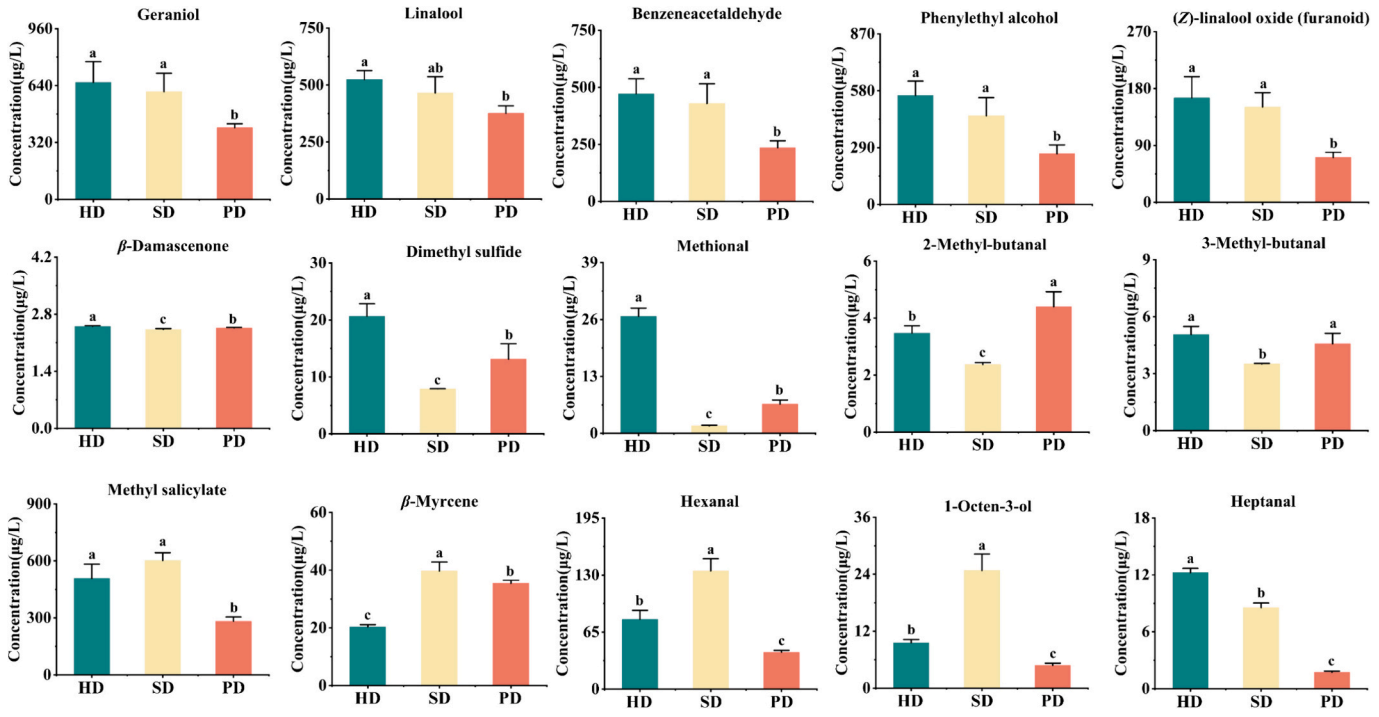

Fig. 3. Concentrations of key differential compounds in black tea subjected to hot-air drying (HD), sun drying (SD), and pan-fired drying (PD). Different letters indicate significant differences at the 0.05 level.

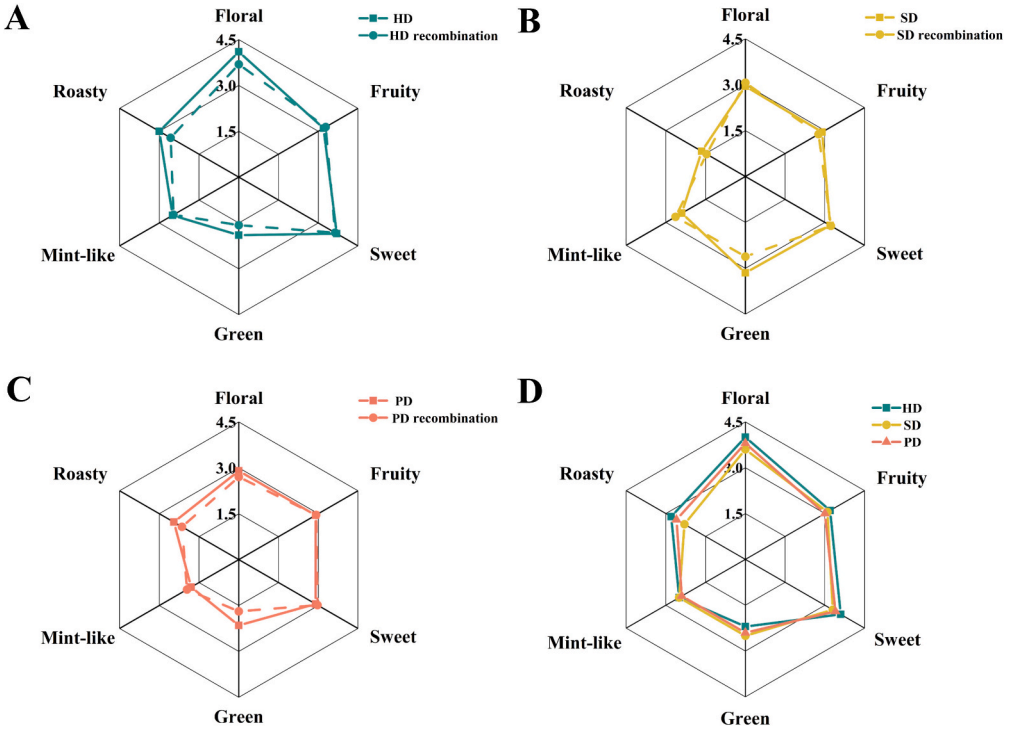

Fig. 4. A, B, C. QDA comparison of aroma characteristics of hot-air drying (HD), sun drying (SD), and pan-fired drying (PD) and recombinant tea infusion for black tea. There was no statistically significant difference among the three groups. D. QDA radar plots of black tea subjected to HD, SD, and PD with added differential aroma concentrations revealing no statistically significant difference among the groups.

between them ($p > 0.05$, Fig. 4D). These results confirm that the 15 aforementioned aromatic compounds served as key differential compounds in the three tea samples.

3.6. Potential reasons for the differences in aroma among the three samples

The three drying methods result in differences in the aroma of black tea due to differences in the conductive medium, temperature, and

duration of tea heating. The synthesis of geraniol, linalool, and methional is mainly affected by high temperature (Fig. 5A-5B). According to previous research, HD can precisely control temperature through contact heat transfer and thermal convection heat transfer, and the high temperature of 110–120 °C for crude fires can lead to increased leaf temperature and water evaporation (Zhu et al., 2022). It also causes the aroma of tea to diffuse from the inner layer to the surface of the tea leaf, resulting in the loss of low-boiling-point compounds, which are responsible for the green aroma of tea, and thereby enhancing the overall floral and fruity aroma of black tea (Lv et al., 2016). Overall, HD involves an even distribution of heat and is associated with a balanced formation of aromatic compounds. The tea samples processed by PD were dried on a machine at a higher temperature (180 °C) for about 25 min. The tea leaves were periodically compressed by a pressure rod in the metal slot of the straightening machine, and due to the open drying process, a large amount of volatile compounds were lost. Tea samples subjected to SD mainly use the light energy of the sun and the heat energy of the ground to remove water from the tea leaves after fermentation. Due to insufficient exposure of tea to heat, enzyme activity cannot be stopped in a timely manner, creating conditions for the production of low boiling compounds in tea. SD involves a suitable temperature, increases lipoxygenase activity, and facilitates the oxidation of fatty acids into hexanal with a green aroma (Fig. 5C) (Huang et al., 2022). By comparison, SD does not yield high-boiling-point compounds; instead, it results in a green aroma, which is unfavorable in black tea. Therefore, by comparing the three drying methods, it was found that HD is more conducive to the accumulation of aromatic components in black tea.

4. Conclusion

This study revealed that three different drying methods had a significant impact on the aroma differences of black tea. SD showed obvious green aroma, HD showed obvious floral and roasty aromas, and PD has a slightly strong roasty aroma. After multiple factor statistical screening, 15 key aroma-active compounds were identified as the reasons for the differences in aroma among the three tea samples: linalool, geraniol, phenylethyl alcohol, phenylacetaldehyde, (*Z*) -linalool oxide (furanoid), *β*-damascenone, dimethyl sulfide, methional, 2-methylbutanal, 3-methylbutanal, methyl salicylate, *β*-myrcene, hexanal, 1-octen-3-ol, and heptanal. ACI analysis showed that geraniol, linalool, hexanal, and methional are the main reasons for the differences in aroma of black tea among the three drying methods. Finally, the accuracy of our results was verified through aroma recombination and addition experiments. This study scientifically analyzed the effects of three drying methods on black tea aroma, providing a theoretical basis for improving black tea aroma.

Ethical statements

Participants gave informed consent via the statement "I am aware that my responses are confidential, and I agree to participate in this sensory evaluation" where an affirmative reply was required to enter the sensory evaluation. They were able to withdraw from the sensory evaluation at any time without giving a reason. The tea products evaluated were safe for consumption.

Human sensory ethical inspection

Ethical permission, to conduct a human sensory study, was granted by our institution. The appropriate protocols for protecting the rights and privacy of all participants were utilized during the execution of the research, such as no coercion to participate, full disclosure of study requirements and risks, written or verbal consent of participants, no release of participant data without their knowledge, and ability to withdraw from the study at any time.

CRediT authorship contribution statement

Mingxia Lu: Writing – original draft, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Caiyan Sheng:** Software, Methodology, Formal analysis. **Han Ke:** Software, Methodology, Formal analysis. **Tiehan Li:** Software, Resources, Methodology. **Qiuyan Liu:** Methodology, Formal analysis, Conceptualization. **Jixin Zhang:** Visualization, Supervision, Investigation. **Luqing Li:** Writing – review & editing, Supervision, Investigation. **Yujie Wang:** Writing – review & editing, Visualization, Supervision. **Jingming Ning:** Resources, Project administration, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence

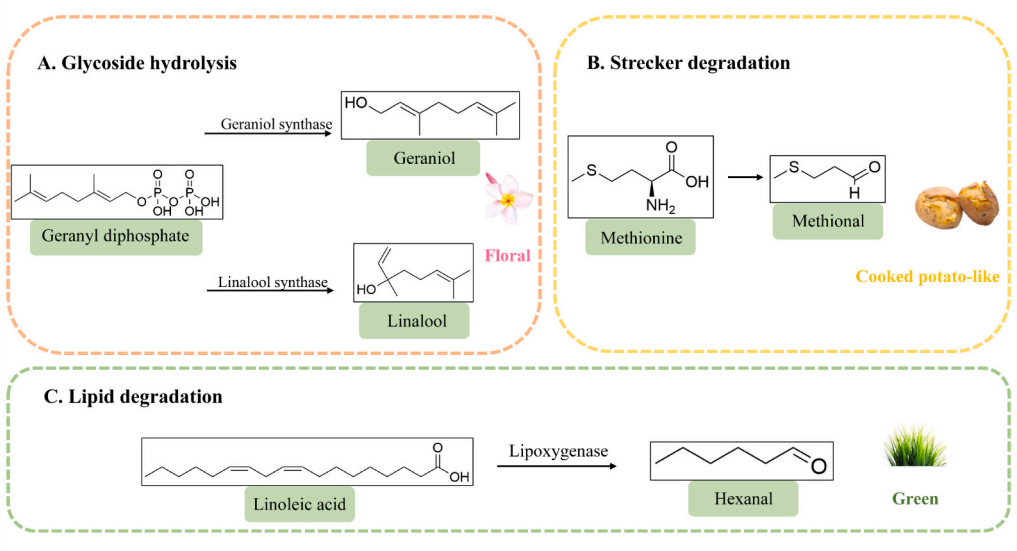

Fig. 5. The sources of key differential aroma compounds in black tea.

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.2024.101782.

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