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# HS-SPME-GC-MS combined with relative odor activity value identify the key aroma components of flowery and fruity aroma in different types of GABA tea

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ABSTRACT

Anaerobic processing is a crucial factor influencing the formation of flavor quality in Gamma-aminobutyric acid (GABA) tea. In this study, headspace solid-phase microextraction combined with gas chromatography and mass spectrometry was employed to explore the flavor characteristics of different types of GABA tea. We utilized multivariate analyses to identify at least 146 volatile components (VOCs) across 12 functional groups in the GABA tea samples via principal component analysis (PCA). At least 40 differential VOCs were screened from the GABA tea samples via orthogonal partial least squares-discriminant analysis. Subsequently, a minimum of four VOCs were identified in the GABA tea samples via the Pearson correlation coefficient combined with relative odor activity values as potential markers for flowery and fruity aromas, clarifying the impact of the VOCs on these characteristics. The results of this study provide a theoretical basis for understanding the formation of flowery and fruity flavor characteristics in GABA tea.

## 1. Introduction

Yunnan Province, recognized as the origin of the world's tea plants, has a rich history of tea discovery and consumption that spans over a millennium (Zhang et al., 2020). With the ongoing development of China's tea industry, the consumer market has progressively expanded (Xu et al., 2016). In recent years, teas produced from Yunnan's unique large-leaf tea resources, such as baked green tea, kung fu black tea, and white tea, have garnered significant consumer favor due to their exceptional quality (Jiang, Su, & Fan, 2023). Current research on baked green tea, kung fu black tea, and white tea primarily examines the effects of cultivars and processing techniques on the flavor quality of these teas, which is essential for achieving consistent overall quality. For instance, the processing of baked green tea enhances the concentrations of certain aldehydes and ketones while decreasing the levels of some alcohols and hydrocarbons in the tea leaves. These modifications contribute to the flowery and fragrant aroma of the tea (Wang et al., 2016). Additionally, fresh baked green tea leaves significantly influence the aroma compounds and overall composition of the tea (Shao et al., 2022). In contrast, kung fu black tea is characterized by its sweet, flowery aroma, which results from the enrichment of linalool and its oxides as well as nerol. The enrichment of these compounds is impacted by fermentation conditions (Wang et al., 2022; Zhang et al., 2023) and tea varieties (Yang et al., 2020). The sun-dried flavor and fragrant aroma of Yunnan white tea are primarily attributed to the concentrations of alcohols and olefins, which result from the specific processing techniques employed for this tea (Huang et al., 2022). Collectively, these studies provide a crucial foundation for further research to develop high-quality and innovatively functional teas.

Gamma-aminobutyric acid (GABA) tea is characterized by elevated GABA levels. In 1987, Tsushida et al. discovered the method of GABA enrichment in tea and successfully developed GABA tea (GABA content >1.5 mg/g) (Tojiro & Toshinobu, 1987). Conventional tea typically exhibits low GABA levels; however, the application of nitrogen-filled

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anaerobic treatment has been shown to enhance the GABA concentration in tea (Norio, Katsuhiro, Toshihiro, & Hirokazu, 1988). The processes of glutamic acid decarboxylation and polyamine degradation primarily mediate GABA enrichment in tea (Ren et al., 2021). In recent years, GABA tea has attracted significant attention due to its notable health benefits and unique properties, including potential therapeutic effects on cardiovascular diseases and diabetes (Zhao et al., 2011). Previous studies on GABA tea have primarily focused on novel GABA enrichment methods (Wang, Tsai, Lin, & Ou, 2006) and enhancing the efficacy of GABA tea (Dai et al., 2020). Furthermore, the type and levels of ingredients in GABA tea can be significantly altered. This alteration primarily affects the precursor components and enzyme activities within known metabolic pathways (Liao et al., 2021). However, the influence of the processing method on the VOCs and flavor quality of GABA tea is still unknown. Our previous study demonstrated that a 6-h anaerobic treatment of Yunnan large-leaf tea varieties resulted in substantial GABA accumulation in the fresh leaves. Additionally, the processed GABA green, black, and white teas exhibited distinctive flavor profiles with notable research implications (Yang et al., 2013).

Aroma, a critical factor influencing tea flavor, significantly affects overall tea quality (Liu et al., 2021; Liu et al., 2021). The essence of tea aroma comprises a mixture of hundreds of volatile organic compounds (VOCs) at varying concentrations. These components primarily originate from biosynthetic pathways in fresh tea leaves and undergo transformations during post-processing, including their precursors, such as glycosides, carotenoids, fatty acids, and amino acids (Feng et al., 2019). To date, more than 700 VOCs have been identified and isolated from tea (Liu, Xu, Wen, et al., 2021; Liu, Xu, Wu, et al., 2021). However, due to the varying odor characteristics and thresholds of each compound, not all VOCs contribute directly to the quality of tea aroma. Only olfactory substances with low olfactory threshold values (OT) (Zheng et al., 2022) or high relative odor activity values (ROAVs) (Li et al., 2022) make significant contributions to the aroma quality of tea. Commonly employed methods for screening, identifying, and evaluating VOCs in various foods include headspace solid-phase microextraction (HS-SPME) technology (Zheng et al., 2023; Zheng et al., 2023), gas chromatography–mass spectrometry (GC–MS) technology (Meng et al., 2024), and sensory evaluation techniques (Deng et al., 2022).

In the present study, Yunnan large-leaf tea species were subjected to anaerobic processing to produce GABA green, black, and white teas. HS-SPME, combined with GC–MS, was employed for the qualitative and quantitative analyses of the VOCs in these teas. Sensory evaluation analysis, multivariate statistical methods, and ROAV analysis were employed to statistically analyze and assess the VOCs. This study aimed to (a) investigate the content and composition of VOCs in different GABA teas, (b) elucidate the impact of these VOCs on the aroma profiles of different GABA teas, and (c) identify key VOCs and potential characteristic marker components associated with the flowery and fruity aromas of GABA tea. Our findings could help address the existing knowledge gaps regarding the flavor quality of GABA tea and provide a theoretical foundation for optimizing its processing.

## 2. Material and methods

### 2.1. Tea samples

In July 2021, all samples (one bud and 2–3 leaves) from *Camellia sinensis* var. *assamica* (Yun Kang 10) were collected and processed at the Dalishu Tea Factory in Yunlong County, Dali. We prepared six types of tea samples: GABA baked green tea (GGT), conventional baked green tea (NGT), GABA Yunnan congou black tea (GBT), conventional Yunnan congou black tea (NBT), GABA white tea (GWT), and conventional white tea (NWT) (Fig. 1). Three replicates per group were analyzed.

Fresh GABA tea leaves were aired at room temperature (RT, 23 °C) for 2 h, and then placed in an $N_2$-filled chamber for anaerobic treatment for 6 h. Next, GABA green tea leaves were subjected to enzyme inactivation for 2 min, followed by rolling for 45 min and drying at 80 °C. GABA black tea leaves were withered indoors at RT for 6 h, rolled for 50 min, fermented at 35 °C for 6 h, and finally dried at 80 °C. GABA white tea leaves were withered indoors at RT for 10 h and then dried at 80 °C. Conventional tea samples (NGT, NBT, and NWT) were processed in the same manner as the GABA tea samples, except for the nitrogen-based anaerobic treatment.

### 2.2. Chemicals

N-hexane was purchased from Merck (Merck, Darmstadt, Germany). NaCl was obtained from China National Pharmaceutical Group Co., Ltd. (Yunnan, China). C7–C40 saturated alkanes and standards, used to determine linear retention indices (LRIs), were purchased from Sigma-Aldrich (Darmstadt, Germany). All chemicals were analytical grade.

### 2.3. Sensory panel evaluation of tea

Eleven trained reviewers (six females and five males; average age: 30 years; professional experience: 5–35 years) quantitatively evaluated

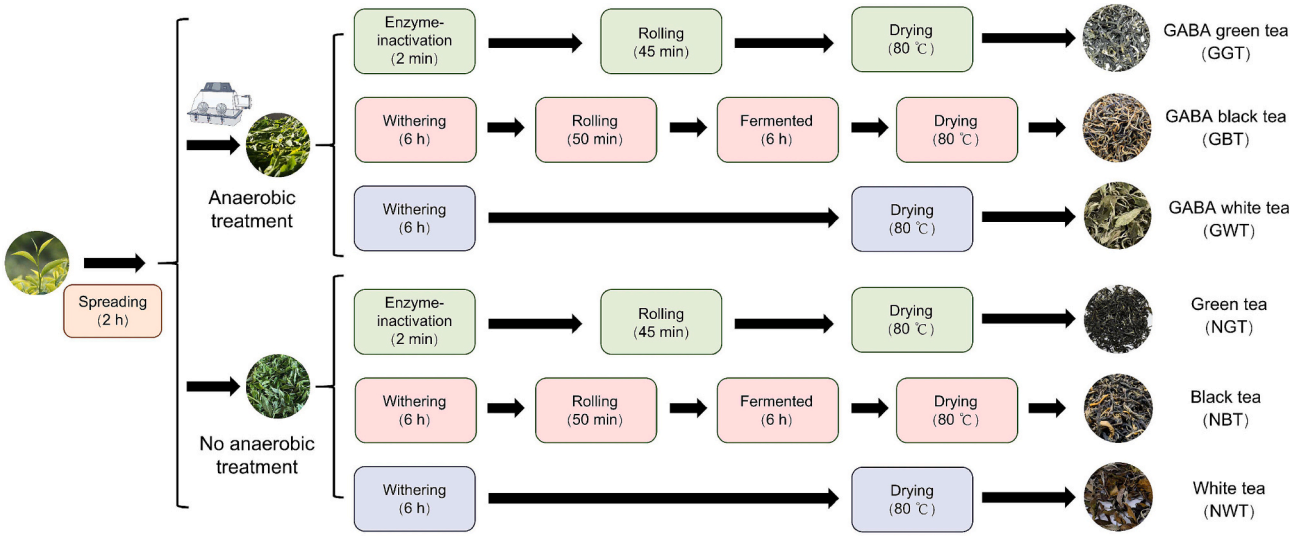


**Fig. 1.** The process of GABA tea and conventional tea.

the aroma, including intensity and attributes, of the tea samples following the Chinese national standard procedure (Su, He, Zhou, Li, & Zhou, 2022). All reviewers were recruited from the Tea College of Yunnan Agricultural University and possessed over three years of experience in sensory evaluation. Prior to the experiment, they underwent specialized training on the identification of tea aroma components (Wang et al., 2023).

The tea samples were presented in a randomized order to eliminate potential bias. Each tea sample, weighing 3 g, was brewed with 150 ml of boiling water for a duration of five minutes. Following this, the aroma was assessed and scored on a scale from zero to ten, where zero indicated no perceptible intensity and ten represented very high intensity. Aroma attributes evaluated included, but were not limited to, "fruity", "flowery", "sour fruity", "tender", "honey", "fragrant", "strong", "sour" and "high and intensive" (Wen et al., 2023). Finally, the collective opinions of the group members were summarized.

### 2.4. Sample preparation (HS-SPME conditions)

Materials were harvested, weighted, immediately frozen in liquid nitrogen, and stored at −80 °C until needed. Samples were ground to a powder in liquid nitrogen.

1 g of the powder was transferred immediately to a 20 ml head-space vial (Agilent, Palo Alto, CA, USA), adding 10 μL (50 μg/ml) internal standard solution (n-hexane, for relative quantification), containing NaCl saturated solution 1 ml, to inhibit any enzyme reaction. The vials were sealed using crimp-top caps with TFE-silicone headspace septa (Agilent). At the time of SPME analysis, each vial was placed in 100 °C for 5 min, then a 120 μm divinylbenzene/carboxen/polydimethylsilioxan fibre (Agilent) was exposed to the headspace of the sample for 15 min at 100 °C. Methods refer to Ma et al. (2023) and Ma et al. (2023).

### 2.5. GC–MS conditions

The detection conditions were determined according to previous experiments. After sampling, desorption of the VOCs from the fibre coating was carried out in the injection port of the GC apparatus (Model 8890; Agilent) at 250 °C for 5 min in the splitless mode. The identification and quantification of VOCs was carried out using an Agilent Model 8890 GC and a 5977B mass spectrometer (Agilent), equipped with a 30 m × 0.25 mm × 0.25 μm DB-5MS (5 % phenylpolymethylsiloxane) capillary column. Helium was used as the carrier gas at a linear velocity of 1.2 ml/min. The injector temperature was kept at 250 °C and the detector at 280 °C. The oven temperature was programmed from 40 °C (3.5 min), increasing at 10 °C/min to 100 °C, at 7 °C/min to 180 °C, at 25 °C/min to 280 °C, hold for 5 min. Mass spectra was recorded in electron impact (EI) ionisation mode at 70 eV. The quadrupole mass detector, ion source and transfer line temperatures were set, respectively, at 150, 230 and 280 °C. Mass spectra was scanned in the range *m/z* 50–500 amu at 1 s intervals. Identification of VOCs was achieved by comparing the mass spectra with the data system library (MWGC or NIST 2020) and linear retention index (RI, determined by n-alkane C5-C40), referencing RI information to increase qualitative accuracy and eliminating the interference of false positive substances. Methods refer to Ma, Gao, et al. (2023) and Ma, Sun, et al. (2023).

### 2.6. ROAV calculation

The following ROAV formula was developed based on the relative concentrations of various aroma components and their respective threshold values in water:

$$ROAV = (C_n/C_{max}) \times (T_{max}/T_n) \times 100$$

Here, $C_n$ represents the relative content of any VOC (μg/g), $C_{max}$ represents the component with the highest relative content (μg/g), $T_n$ represents the threshold of any VOC (μg/g), and $T_{max}$ represents the threshold of the component with the largest relative content (μg/g) (Wang et al., 2020). As evident from the formula, the greater the ROAV value of an aroma component, the greater its contribution to the overall aroma of the compound. The components with ROAV >1 were considered important contributors to the overall tea aroma, and the components with 0.1 < ROAV <1 were considered the modification of the overall tea aroma (Bi et al., 2022).

### 2.7. Statistical analysis

SPSS26 was used for the analysis of variance (ANOVA) and the post-hoc test. Differences with $P < 0.05$ were considered statistically significant. Metware Cloud (https://www.metware.cn) was used for principal component analysis (PCA) and orthogonal partial least squares discriminant analysis (OPLS-DA). Origin 2022 were used for clustering heat map, radar map, volcano map, chord map, and other data analyses and mapping. For each analysis, three biological replicates were used, and the results were expressed as mean ± standard deviation (SD).

## 3. Results and discussion

### 3.1. Sensory evaluation of different types of GABA tea

The aroma characteristics of the NGT, NBT, and NWT samples significantly differed from those of the GGT, GBT, and GWT samples, respectively (Fig. 2).

As shown in Fig. 2A, NGT samples exhibited tender and stir-fried bean fragrances at higher intensities. In contrast, GGT exhibited flowery and fruity aroma characteristics, presenting a milder tender aroma and stir-fried bean aroma than NGT. Notably, the aroma of GGT was more intense and longer-lasting, contrary to findings from previous studies. For example, the GABA green tea produced from the Jinxuan (middle-leaf variety) exhibited the aroma of red dates (Zhen, 2012). This discrepancy might be attributed to variations in tea varieties and the processing parameters utilized in different studies. As shown in Fig. 2B, NBT exhibited a sweet, honey-scented, vibrant, and long-lasting aroma. However, GBT exhibited a strong flowery and honey fragrance, characterized by a more pronounced and enduring scent. This observation contrasted the findings from previous studies that reported a sweet aroma of GABA black tea derived from small-leaf varieties (Wu et al., 2017). This discrepancy might be attributed to the use of different tea cultivars in varying investigations. Fig. 2C illustrates that NWT presented a flowery, high-intensity aroma, whereas GWT exhibited more robust flowery and fruity notes, with a greater persistence in scent. This finding aligns with earlier studies documenting intense flowery and fruity aromas in GABA white tea (Li et al., 2023).

### 3.2. Analysis of VOCs in different types of GABA tea

Using HS-SPME-GC–MS the technology, we detected 147 VOCs in NGT and GGT, with NGT containing all 147 components and GGT containing 146 (Table 1). Furthermore, based on their composition, NGT and GGT were clustered separately, indicating that the data were representative (Zhou et al., 2022), with significantly different levels of VOCs in both tea samples (Fig. S1). The VOCs identified in NGT could be categorized into 13 functional groups, including 35 hydrocarbons, 26 terpenoids, 22 esters, 12 alcohols, 12 heterocyclic compounds, 10 ketones, 10 aromatics, 8 aldehydes, 4 phenols, 3 hydrocarbons, 3 acids, 1 nitrogenous compound, and 1 component in another functional group. In contrast, GGT contained only 12 functional groups of VOCs, lacking the nitrogenous compound and featuring only two hydrocarbons. However, the levels of VOCs in GGT were significantly higher than those in NGT (Fig. 3A). The five most abundant VOCs in GGT included indole; 9,12,15-octadecatrienoic acid (Z,Z,Z)-; linoleic acid ethyl ester;

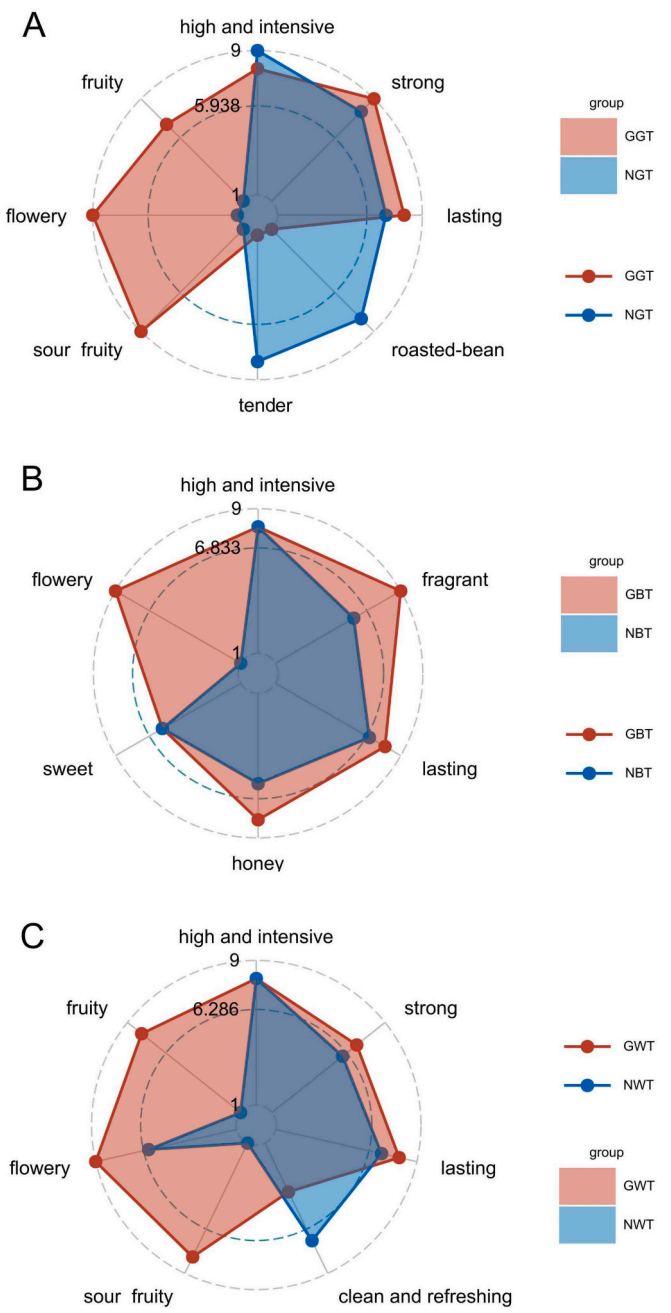


**Fig. 2.** Quantitative radar chart of aroma sensory evaluation results. (A) Aroma comparison results of green tea (NGT vs GGT). (B) Aroma comparison results of black tea (NBT vs GBT). (C) Aroma comparison results of white tea (NWT vs GWT).

ethanone, 1-(2-hydroxy-5-methylphenyl)-; and (Z,Z)-methyl ester, with concentrations of 5.95, 4.74, 4.44, 3.58, and 3.47 μg/g, respectively. The levels of these five VOCs in GGT were significantly higher than those in NGT (Fig. 3B).

Next, we detected 149 VOCs in NBT and GBT, with both containing the same set of 149 components (Table 1). Based on their composition, NBT and GBT could be clustered separately, indicating that the data were representative and that there were significantly different levels of VOCs in both tea samples (Fig. S2). The components in GBT were categorized into 13 functional groups, including 35 hydrocarbons, 26 terpenoids, 22 esters, 13 alcohols, 12 heterocyclic compounds, 11 ketones, 10 aromatics, 8 aldehydes, 4 phenols, 3 hydrocarbons, 3 acids, 1 nitrogenous compound, and 1 component belonging to another functional group. GBT exhibited significantly higher levels of VOCs than NBT (Fig. 3C). The five most abundant VOCs in GBT were decane, 5-methyl-; β-ocimene; diethyl (decyloxy)-borane; 5-methyl-1,2,5,6-tetrahydropyridin-2-one; and 2-(1,1-dimethylethyl)-6-(1-methylethyl) phenol, with concentrations of 9.78, 8.01, 7.95, 5.29, and 4.5 μg/g, respectively. GBT exhibited significantly higher levels of these VOCs than NGT (Fig. 3D).

Next, we detected 147 VOCs in both NWT and GWT, both containing the same set of components (Table 1). Based on their composition, NWT and GWT could be clustered separately, indicating that the data were representative and that the levels of VOCs in both tea samples were significantly different (Fig. S3). The components in GWT were categorized into 12 functional groups, including 34 hydrocarbons, 26 terpenoids, 22 esters, 13 alcohols, 12 heterocyclic compounds, 11 ketones, 10 aromatics, 8 aldehydes, 4 phenols, 3 hydrocarbons, 3 acids, and 1 nitrogenous compound. GWT exhibited significantly higher levels of VOCs than NWT (Fig. 3E). The five most abundant VOCs in GWT were (E)-2-hexenoic acid butyl ester; 4-methyl-5-hexen-2-ol; 2-heptanol; pentadecanal and heptanal, with concentrations of 8.14, 5.81, 4.76, 4.21, and 3.96 μg/g, respectively. The content of four VOCs (excluding pentadecanal) were significantly higher in GWT than in NWT (Fig. 3F).

To the best of our knowledge, our study was the first to assess the aroma components in GGT, GBT, and GWT processed from large-leaf species. Our findings indicated that GGT contains high levels of hydrocarbons, esters, and terpenoids, collectively accounting for 56.17 % of all VOCs in GGT. Furthermore, compared to NGT, GGT exhibited 5.5-, 2.6-, 1.9-, and 1.7-fold higher levels of acids, esters, aldehydes, and ketones, respectively, significantly contributing to its aroma profile. Similarly, previous studies have reported relatively higher levels of ketones, terpenoids, and heterocyclic compounds in baked green tea derived from large-leaf species, which enhance its fragrance and honey aroma (Wang et al., 2016). Additionally, GBT showed elevated levels of hydrocarbons, terpenoids, esters, and alcohols, accounting for 61.18 % of all VOCs in GBT. Compared to NBT, GBT exhibited 13.8- and 2.1-fold higher concentrations of other functional groups and nitrogenous compounds, potentially influencing its aroma profile. Consistent with our findings, a previous study reported higher acetophenone and ethyl acetate levels in black tea produced from large-leaf species, contributing to its strong, sweet, and fruity aroma (Zheng, Gan, et al., 2023; Zheng, Hu, et al., 2023). Finally, GWT exhibited high levels of hydrocarbons, terpenoids, esters, and alcohols, constituting 64 % of all VOCs in GWT. Moreover, compared to NWT, GWT contained 1.5- and 1.2-fold higher levels of alcohols and acids, respectively, potentially impacting its aroma profile. In agreement with our observations, earlier studies have also noted elevated levels of olefins and terpenes in white tea prepared from large-leaf species (Yan, Zhong, Lv, & Meng, 2019).

These differences might be attributed to two factors. On the one hand, anaerobic fermentation might promote the retention of metabolic precursors in GGT, such as indole and 9,12,15-octadecatrienoic acid (Z, Z,Z) (Yang et al., 2023). On the other hand, the withering process might accelerate enzymatic catalysis and the cleavage of aroma precursors such as glycosides, resulting in the elevation of terpenoids, ketones, and aldehydes in GABA teas (Fang, Liu, Xiao, Ma, & Huang, 2023; Ho, Zheng, & Li, 2015). Thus, the differences in the VOC levels between GBT and GWT were small. From the aroma characteristics and the proportion of functional groups, the GABA teas in this study were as significantly different from the control samples. In future studies, we will consider using absolute quantitative techniques to explore the quantitative influence of VOCs.

To assess whether the VOCs in different GABA teas were key contributors to their aromas, we also evaluated their odor activity values (OAVs) or ROAVs. We observed that hydrocarbons and terpenoids accounted for a major portion of the total VOC content in all three types of GABA teas, which might be attributed to their long-term anaerobic treatment (Wang et al., 2024). However, the three GABA teas differed in the relative proportions of each functional group. Therefore, we further

**Table 1**
Volatile components composition and relative contents.

| No | RI | components | Class I | CAS | Relative Content (μg/g) | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | NGT | GGT | NBT | GBT | NWT | GWT |
| 1 | 1477.9 | β-Ionone | Terpenoids | 217,482-81-0 | 1.16 ± 0.12 | 1.11 ± 0.04 | 1.15 ± 0.10 | 1.25 ± 0.07 | 1.10 ± 0.09 | 1.23 ± 0.08 |
| 2 | 1837.31 | Phytol, acetate | Alcohol | 1,000,375-01-4 | 1.13 ± 0.12 | 1.53 ± 0.09 | 1.33 ± 0.10 | 1.26 ± 0.11 | 0.52 ± 0.04 | 0.41 ± 0.01 |
| 3 | 1958.7 | Phthalic acid, butyl hept-4-yl ester | Ester | 1,000,356-78-4 | 0.48 ± 0.12 | 1.42 ± 0.04 | 0.59 ± 0.06 | 0.35 ± 0.02 | 1.10 ± 0.16 | 1.40 ± 0.08 |
| 4 | 2160.32 | Linoleic acid ethyl ester | Ester | 544-35-4 | 0.04 ± 0.00 | 4.44 ± 0.33 | 0.03 ± 0.00 | 0.05 ± 0.02 | 0.05 ± 0.00 | 0.03 ± 0.01 |
| 5 | 1946.9 | Isophytol | Terpenoids | 505-32-8 | 1.18 ± 0.44 | 1.88 ± 0.08 | 1.31 ± 0.12 | 1.17 ± 0.13 | 0.60 ± 0.03 | 0.65 ± 0.04 |
| 6 | 2113.79 | Phytol | Terpenoids | 150-86-7 | 1.42 ± 0.75 | 2.47 ± 0.29 | 1.50 ± 0.19 | 1.54 ± 0.37 | 0.52 ± 0.06 | 0.45 ± 0.03 |
| 7 | 1879.2 | 3,7,11,15-Tetramethyl-2-hexadecen-1-ol | Terpenoids | 102,608-53-7 | 1.12 ± 0.12 | 1.58 ± 0.09 | 1.30 ± 0.08 | 1.25 ± 0.11 | 0.54 ± 0.03 | 0.39 ± 0.00 |
| 8 | 2092.49 | 9,12-Octadecadienoic acid (Z,*Z*)-, methyl ester | Ester | 112-63-0 | 0.15 ± 0.05 | 3.47 ± 0.19 | 0.73 ± 0.13 | 0.41 ± 0.01 | 0.09 ± 0.02 | 0.09 ± 0.00 |
| 9 | 1990.7 | Hexadecanoic acid, ethyl ester | Ester | 628-97-7 | 0.02 ± 0.01 | 3.32 ± 0.23 | 0.06 ± 0.01 | 0.08 ± 0.02 | 0.11 ± 0.02 | 0.12 ± 0.03 |
| 10 | 1805.46 | Hexadecane, 2,6,10,14-tetramethyl- | Hydrocarbons | 638-36-8 | 1.06 ± 0.06 | 1.26 ± 0.17 | 1.04 ± 0.09 | 0.51 ± 0.03 | 2.18 ± 0.34 | 1.54 ± 0.09 |
| 11 | 1745.74 | Hexadecane, 2,6,11,15-tetramethyl- | Hydrocarbons | 504-44-9 | 1.15 ± 0.09 | 1.18 ± 0.12 | 0.98 ± 0.14 | 0.72 ± 0.01 | 1.73 ± 0.29 | 1.67 ± 0.10 |
| 12 | 2167.24 | 9,12,15-Octadecatrienoic Acid, (Z,Z,Z)- | Acid | 463-40-1 | 0.05 ± 0.03 | 4.74 ± 0.27 | 0.04 ± 0.01 | 0.05 ± 0.01 | 0.05 ± 0.01 | 0.06 ± 0.03 |
| 13 | 1998.17 | Sulfurous acid, 2-ethylhexyl hexyl ester | Ester | 1,000,309-20-2 | 0.90 ± 0.17 | 0.95 ± 0.16 | 0.73 ± 0.09 | 0.54 ± 0.11 | 1.78 ± 0.12 | 1.77 ± 0.14 |
| 14 | 1907.29 | 7,9-Di-tert-butyl-1-oxaspiro(4,5)deca-6,9-diene-2,8-dione | Heterocyclic compound | 82,304-66-3 | 1.32 ± 0.36 | 1.33 ± 0.21 | 1.16 ± 0.21 | 1.07 ± 0.34 | 1.65 ± 0.16 | 1.90 ± 0.29 |
| 15 | 1923.63 | Hexadecanoic acid, methyl ester | Ester | 112-39-0 | 0.37 ± 0.05 | 2.07 ± 0.15 | 1.54 ± 0.19 | 1.03 ± 0.05 | 0.17 ± 0.03 | 0.14 ± 0.02 |
| 16 | 1701.98 | Pentadecane, 2,6,10,14-tetramethyl- | Hydrocarbons | 1921-70-6 | 1.25 ± 0.02 | 1.31 ± 0.20 | 0.96 ± 0.14 | 0.78 ± 0.06 | 1.67 ± 0.27 | 1.15 ± 0.06 |
| 17 | 1842.46 | 2-Pentadecanone, 6,10,14-trimethyl- | Ketone | 502-69-2 | 0.49 ± 0.08 | 0.66 ± 0.04 | 0.42 ± 0.05 | 0.37 ± 0.01 | 1.36 ± 0.21 | 1.38 ± 0.10 |
| 18 | 2098.35 | (Z)-9,17-Octadecadienal | Aldehyde | 56,554-35-9 | 0.36 ± 0.12 | 2.93 ± 0.23 | 1.39 ± 0.24 | 0.84 ± 0.07 | 0.10 ± 0.03 | 0.07 ± 0.01 |
| 19 | 1560.48 | Farnesol, acetate | Ester | 1,000,352-67-2 | 0.51 ± 0.04 | 1.06 ± 0.12 | 2.47 ± 0.35 | 2.66 ± 0.14 | 0.89 ± 0.12 | 1.22 ± 0.08 |
| 20 | 1674.1 | Heptadecane, 2-methyl- | Hydrocarbons | 1560-89-0 | 1.04 ± 0.07 | 1.15 ± 0.15 | 1.04 ± 0.12 | 0.85 ± 0.05 | 1.45 ± 0.23 | 1.35 ± 0.05 |
| 21 | 1772.65 | Heptadecane, 3-methyl- | Hydrocarbons | 6418-44-6 | 1.17 ± 0.09 | 1.23 ± 0.13 | 0.90 ± 0.10 | 0.58 ± 0.01 | 1.60 ± 0.25 | 1.97 ± 0.13 |
| 22 | 1798.71 | Pentadecane, 2,6,10-trimethyl- | Hydrocarbons | 3892-00-0 | 0.96 ± 0.14 | 1.02 ± 0.11 | 0.84 ± 0.12 | 0.54 ± 0.04 | 1.97 ± 0.03 | 1.48 ± 0.10 |
| 23 | 1588.3 | 2-Oxobicyclo(3.2.2)nona-3,6-dien-1-yl benzoate | Ester | 73,830-87-2 | 0.40 ± 0.05 | 1.39 ± 0.13 | 1.89 ± 0.34 | 1.05 ± 0.03 | 1.31 ± 0.21 | 1.91 ± 0.16 |
| 24 | 1660.99 | Hexadecane, 4-methyl- | Hydrocarbons | 25,117-26-4 | 1.12 ± 0.10 | 1.21 ± 0.18 | 1.05 ± 0.12 | 0.87 ± 0.05 | 1.42 ± 0.04 | 1.30 ± 0.04 |
| 25 | 1666.84 | Hexadecane, 2-methyl- | Hydrocarbons | 1560-92-5 | 1.08 ± 0.08 | 1.22 ± 0.15 | 1.01 ± 0.16 | 0.76 ± 0.05 | 1.54 ± 0.04 | 1.30 ± 0.04 |
| 26 | 1698.5 | Heptadecane | Hydrocarbons | 629-78-7 | 1.01 ± 0.08 | 1.08 ± 0.12 | 1.01 ± 0.14 | 0.80 ± 0.03 | 1.46 ± 0.21 | 0.99 ± 0.03 |
| 27 | 1542.7 | 4-ethyl-Tetradecane | Hydrocarbons | 55,045-14-2 | 1.06 ± 0.00 | 1.16 ± 0.16 | 0.77 ± 0.05 | 0.83 ± 0.04 | 1.17 ± 0.05 | 1.00 ± 0.08 |
| 28 | 1598.46 | Hexadecane | Hydrocarbons | 544-76-3 | 1.17 ± 0.08 | 1.27 ± 0.17 | 1.05 ± 0.15 | 1.03 ± 0.05 | 1.31 ± 0.03 | 1.00 ± 0.07 |
| 29 | 1556.14 | Pentadecane, 4-methyl- | Hydrocarbons | 2801-87-8 | 1.16 ± 0.07 | 1.17 ± 0.14 | 0.92 ± 0.25 | 0.94 ± 0.07 | 1.19 ± 0.02 | 0.98 ± 0.12 |
| 30 | 1409.57 | Nonane, 2,2,4,4,6,8,8-heptamethyl- | Hydrocarbons | 4390-04-9 | 0.98 ± 0.07 | 1.02 ± 0.12 | 0.85 ± 0.03 | 1.09 ± 0.06 | 1.11 ± 0.04 | 0.77 ± 0.08 |
| 31 | 1569.34 | 3-methyl-Pentadecane | Hydrocarbons | 2882-96-4 | 1.13 ± 0.05 | 1.23 ± 0.15 | 0.88 ± 0.10 | 0.88 ± 0.07 | 1.21 ± 0.03 | 1.18 ± 0.06 |
| 32 | 1459.04 | 2,6,10-Trimethyltridecane | Hydrocarbons | 3891-99-4 | 0.58 ± 0.03 | 0.56 ± 0.07 | 0.36 ± 0.01 | 0.36 ± 0.02 | 0.33 ± 0.06 | 0.32 ± 0.02 |
| 33 | 999.1 | diethyl(decyloxy)-Borane | Others | 1,000,152-34-3 | 1.84 ± 1.56 | 2.14 ± 0.85 | 0.58 ± 0.18 | 7.95 ± 1.07 | – | – |
| 34 | 1716.2 | Pentadecanal | Aldehyde | 2765-11-9 | 0.62 ± 0.28 | 0.61 ± 0.07 | 1.29 ± 0.18 | 1.50 ± 0.16 | 4.39 ± 0.59 | 4.21 ± 0.79 |
| 35 | 1911.51 | Nerolidol 1 | Terpenoids | 1,000,285-43-5 | 1.11 ± 0.26 | 1.35 ± 0.04 | 0.75 ± 0.12 | 0.82 ± 0.04 | 1.37 ± 0.02 | 2.00 ± 0.11 |
| 36 | 1618.06 | (3R,3aS,6S,7R)-3,6,8,8-Tetramethyloctahydro-1H-3a,7-methanoazulen-6-ol | Alcohol | 19,903-73-2 | 0.89 ± 0.12 | 1.15 ± 0.03 | 1.23 ± 0.17 | 1.08 ± 0.07 | 2.22 ± 0.26 | 1.58 ± 0.07 |

(*continued on next page*)

**Table 1** (*continued*)

| No | RI | components | Class I | CAS | Relative Content (μg/g) | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | NGT | GGT | NBT | GBT | NWT | GWT |
| 37 | 1664.61 | α-Cadinol | Terpenoids | 481-34-5 | 0.76 ± 0.12 | 1.53 ± 0.05 | 1.70 ± 0.23 | 1.94 ± 0.10 | 0.68 ± 0.09 | 1.00 ± 0.14 |
| 38 | 1586.64 | Diethyl Phthalate | Ester | 84-66-2 | 0.38 ± 0.06 | 0.52 ± 0.02 | 0.44 ± 0.05 | 0.35 ± 0.01 | 1.70 ± 0.01 | 1.57 ± 0.08 |
| 39 | 1572.66 | (3E,7*E*)-4,8,12-Trimethyltrideca-1,3,7,11-tetraene | Terpenoids | 62,235-06-7 | 1.48 ± 0.09 | 1.68 ± 0.27 | 1.43 ± 0.28 | 0.78 ± 0.06 | 0.18 ± 0.06 | 0.28 ± 0.03 |
| 40 | 1565.64 | (1R,4S,9aS)-1-Methyl-4-((*Z*)-pent-2-en-4-yn-1-yl)octahydro-1H-quinolizine | Heterocyclic compound | 151,805-13-9 | 1.23 ± 0.16 | 0.94 ± 0.02 | 1.48 ± 0.12 | 1.16 ± 0.05 | 1.13 ± 0.13 | 1.31 ± 0.06 |
| 41 | 1654.55 | 3-methyl-Tetradecane | Hydrocarbons | 18,435-22-8 | 1.15 ± 0.08 | 1.30 ± 0.16 | 0.99 ± 0.14 | 0.78 ± 0.05 | 1.48 ± 0.04 | 1.44 ± 0.11 |
| 42 | 1419.36 | Tetradecane, 4-methyl- | Hydrocarbons | 25,117-24-2 | 1.08 ± 0.07 | 1.03 ± 0.13 | 0.78 ± 0.01 | 0.89 ± 0.06 | 0.94 ± 0.02 | 0.68 ± 0.09 |
| 43 | 1498.34 | Pentadecane | Hydrocarbons | 629-62-9 | 1.11 ± 0.09 | 1.07 ± 0.14 | 0.96 ± 0.10 | 1.02 ± 0.03 | 1.08 ± 0.08 | 0.73 ± 0.03 |
| 44 | 1660.37 | 2,2′,5,5′-tetramethyl-1,1'-Biphenyl | Aromatics | 3075-84-1 | 0.82 ± 0.05 | 0.93 ± 0.09 | 0.81 ± 0.12 | 0.75 ± 0.01 | 1.76 ± 0.26 | 1.44 ± 0.11 |
| 45 | 1416.72 | 6-Methyl-6-(5-methylfuran-2-yl)heptan-2-one | Ketone | 50,464-95-4 | 0.65 ± 0.09 | 0.57 ± 0.05 | 1.40 ± 0.21 | 1.71 ± 0.12 | 1.49 ± 0.21 | 1.66 ± 0.10 |
| 46 | 1628.78 | 4′,6'-Dimethoxy-2′,3′-dimethylacetophenone | Ketone | 1,000,244-80-3 | 0.61 ± 0.09 | 0.53 ± 0.02 | 0.97 ± 0.18 | 0.60 ± 0.01 | 0.57 ± 0.09 | 0.94 ± 0.13 |
| 47 | 1504.35 | 2,4-Di-tert-butylphenol | Aromatics | 96-76-4 | 1.01 ± 0.09 | 1.02 ± 0.18 | 0.95 ± 0.25 | 0.82 ± 0.08 | 1.07 ± 0.36 | 1.02 ± 0.21 |
| 48 | 1167.75 | 2-(2-butoxyethoxy)-Ethanol,acetate | Ester | 124-17-4 | 1.20 ± 0.13 | 1.16 ± 0.06 | 1.03 ± 0.09 | 1.15 ± 0.09 | 1.06 ± 0.13 | 0.89 ± 0.14 |
| 49 | 1573.04 | 3-Hexen-1-ol benzoate | Ester | 1,000,132-06-6 | 0.95 ± 0.09 | 1.12 ± 0.12 | 1.93 ± 0.29 | 2.19 ± 0.11 | 0.36 ± 0.06 | 0.52 ± 0.05 |
| 50 | 1382.7 | Hexanoic acid, hexyl ester | Ester | 6378-65-0 | 0.32 ± 0.00 | 0.37 ± 0.05 | 2.81 ± 0.17 | 3.26 ± 0.23 | 0.65 ± 0.14 | 0.79 ± 0.06 |
| 51 | 1545.24 | α-Calacorene | Terpenoids | 21,391-99-1 | 1.07 ± 0.04 | 1.05 ± 0.13 | 1.40 ± 0.15 | 1.50 ± 0.05 | 0.50 ± 0.09 | 0.59 ± 0.04 |
| 52 | 1398.41 | Tetradecane | Hydrocarbons | 629-59-4 | 1.08 ± 0.03 | 1.18 ± 0.13 | 0.81 ± 0.03 | 0.97 ± 0.06 | 0.99 ± 0.11 | 0.79 ± 0.07 |
| 53 | 1319.7 | Dodecane, 4,6-dimethyl- | Hydrocarbons | 61,141-72-8 | 0.94 ± 0.17 | 1.09 ± 0.11 | 0.77 ± 0.07 | 0.91 ± 0.05 | 0.96 ± 0.02 | 0.63 ± 0.11 |
| 54 | 1493.53 | Tridecane, 2-methyl- | Hydrocarbons | 1560-96-9 | 0.39 ± 0.07 | 1.10 ± 0.05 | 1.99 ± 0.24 | 2.07 ± 0.07 | 1.95 ± 0.01 | 1.98 ± 0.15 |
| 55 | 1368.99 | 3,5-Dimethyldodecane | Hydrocarbons | 107,770-99-0 | 0.78 ± 0.05 | 1.12 ± 0.12 | 0.75 ± 0.00 | 0.89 ± 0.07 | 1.32 ± 0.17 | 1.02 ± 0.09 |
| 56 | 1377.24 | Hexanoic acid, 3-hexenyl ester, (Z)- | Ester | 31,501-11-8 | 0.41 ± 0.01 | 0.32 ± 0.04 | 2.68 ± 0.17 | 4.01 ± 0.28 | 0.12 ± 0.02 | 0.14 ± 0.01 |
| 57 | 1385.4 | (E)-Hexanoic acid, 2-hexenyl ester | Ester | 53,398-86-0 | 0.45 ± 0.01 | 0.47 ± 0.05 | 3.41 ± 0.27 | 2.58 ± 0.22 | 0.35 ± 0.06 | 0.56 ± 0.05 |
| 58 | 1445.93 | 5,9-Undecadien-2-one, 6,10-dimethyl-, (E)- | Ketone | 3796-70-1 | 0.98 ± 0.09 | 1.19 ± 0.10 | 0.79 ± 0.09 | 0.78 ± 0.09 | 1.02 ± 0.15 | 1.14 ± 0.06 |
| 59 | 1302.26 | Teaspirane | Terpenoids | 36,431-72-8 | 1.07 ± 0.13 | 1.30 ± 0.17 | 0.70 ± 0.04 | 0.92 ± 0.03 | 0.67 ± 0.13 | 0.65 ± 0.09 |
| 60 | 1853.02 | Caffeine | Heterocyclic compound | 58-08-2 | 1.26 ± 0.18 | 1.22 ± 0.01 | 1.18 ± 0.03 | 1.33 ± 0.12 | 1.24 ± 0.09 | 1.28 ± 0.07 |
| 61 | 1423.2 | α-Ionone | Terpenoids | 127-41-3 | 0.61 ± 0.05 | 0.67 ± 0.05 | 1.23 ± 0.12 | 1.47 ± 0.17 | 1.87 ± 0.06 | 2.03 ± 0.10 |
| 62 | 1337.69 | 5-Methyl-2,4-diisopropylphenol | Phenol | 40,625-96-5 | 0.70 ± 0.04 | 0.93 ± 0.09 | 0.65 ± 0.05 | 0.69 ± 0.07 | 0.36 ± 0.05 | 0.39 ± 0.01 |
| 63 | 1258.48 | 2-(1,1-Dimethylethyl)-6-(1-methylethyl) phenol | Phenol | 22,791-95-3 | 0.47 ± 0.03 | 0.35 ± 0.04 | 3.28 ± 0.17 | 4.50 ± 0.17 | 0.68 ± 0.11 | 0.84 ± 0.12 |
| 64 | 1380.04 | β-Damascenone | Terpenoids | 23,726-93-4 | 0.32 ± 0.03 | 0.62 ± 0.06 | 3.21 ± 0.18 | 3.67 ± 0.31 | 0.64 ± 0.09 | 0.94 ± 0.07 |
| 65 | 1407.67 | 4-(2,6,6-Trimethylcyclohexa-1,3-dienyl)but-3-en-2-one | Ketone | 1203-08-3 | 1.00 ± 0.01 | 0.79 ± 0.07 | 1.65 ± 0.20 | 1.51 ± 0.12 | 1.19 ± 0.17 | 1.44 ± 0.07 |
| 66 | 1591.17 | 1-Dodecanol | Alcohol | 112-53-8 | 1.33 ± 0.03 | 1.43 ± 0.19 | 1.16 ± 0.17 | 1.11 ± 0.12 | 1.23 ± 0.21 | 1.42 ± 0.07 |
| 67 | 1362.43 | Undecane, 2,9-dimethyl- | Hydrocarbons | 17,301-26-7 | 0.64 ± 0.06 | 0.82 ± 0.06 | 2.14 ± 0.46 | 1.99 ± 0.00 | 3.36 ± 0.05 | 3.03 ± 0.07 |
| 68 | 1162.73 | Nonane, 5-(2-methylpropyl)- | Hydrocarbons | 62,185-53-9 | 0.79 ± 0.28 | 1.00 ± 0.12 | 0.69 ± 0.18 | 0.99 ± 0.06 | 0.65 ± 0.02 | 0.57 ± 0.21 |
| 69 | 1260.38 | Decane, 3-ethyl-3-methyl- | Hydrocarbons | 17,312-66-2 | 0.89 ± 0.24 | 1.04 ± 0.12 | 0.71 ± 0.12 | 0.90 ± 0.01 | 0.58 ± 0.02 | 0.58 ± 0.19 |
| 70 | 1211.4 | Undecane, 4,6-dimethyl- | Hydrocarbons | 17,312-82-2 | 0.86 ± 0.25 | 1.03 ± 0.13 | 0.67 ± 0.13 | 0.91 ± 0.01 | 0.58 ± 0.01 | 0.58 ± 0.15 |
| 71 | 1329.38 | Undecane, 5,7-dimethyl- | Hydrocarbons | 17,312-83-3 | 0.94 ± 0.14 | 1.00 ± 0.12 | 0.70 ± 0.13 | 0.81 ± 0.11 | 0.82 ± 0.05 | 0.58 ± 0.09 |
| 72 | 1304.88 | Decane, 2,3,5-trimethyl- | Hydrocarbons | 62,238-11-3 | 0.92 ± 0.14 | 0.90 ± 0.03 | 0.78 ± 0.09 | 0.87 ± 0.23 | 0.75 ± 0.04 | 0.70 ± 0.11 |
| 73 | 1228.24 | cis-3-hexenyl ester | Ester | 35,852-46-1 | 0.31 ± 0.04 | 0.12 ± 0.02 | 2.51 ± 0.12 | 3.79 ± 0.21 | 0.43 ± 0.05 | 0.73 ± 0.09 |

(*continued on next page*)

**Table 1** (*continued*)

| No | RI | components | Class I | CAS | Relative Content (μg/g) | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | NGT | GGT | NBT | GBT | NWT | GWT |
| 74 | 1533.13 | 2(4H)-Benzofuranone, 5,6,7,7a-tetrahydro-4,4,7a-trimethyl-, (*R*)- | Ester | 17,092-92-1 | 0.47 ± 0.10 | 0.54 ± 0.02 | 0.86 ± 0.07 | 0.89 ± 0.05 | 2.20 ± 0.06 | 2.19 ± 0.13 |
| 75 | 1518.37 | 1,3-Benzenediol, 5-pentyl- | Phenol | 500-66-3 | 1.64 ± 0.23 | 2.02 ± 0.10 | 0.83 ± 0.12 | 0.49 ± 0.03 | 1.53 ± 0.18 | 1.74 ± 0.11 |
| 76 | 1807.58 | Aspirin | Aromatics | 50-78-2 | 1.38 ± 0.31 | 1.43 ± 0.13 | 1.41 ± 0.13 | 0.89 ± 0.05 | 3.10 ± 0.60 | 2.53 ± 0.12 |
| 77 | 1580.75 | Butyl benzoate | Ester | 136-60-7 | 0.54 ± 0.09 | 0.65 ± 0.01 | 2.24 ± 0.24 | 1.48 ± 0.03 | 1.63 ± 0.32 | 2.24 ± 0.22 |
| 78 | 1357.23 | 1, 1, 5-Trimethyl-1, 2-dihydronaphthalene | Terpenoids | 1,000,357-25-8 | 0.92 ± 0.02 | 0.67 ± 0.07 | 2.52 ± 0.18 | 2.27 ± 0.12 | 0.43 ± 0.07 | 0.44 ± 0.02 |
| 79 | 1284.27 | Ethyl 4-(ethyloxy)-2-oxobut-3-enoate | Ester | 1,000,305-38-2 | 0.18 ± 0.01 | 0.25 ± 0.03 | 2.99 ± 0.25 | 3.66 ± 0.34 | 0.80 ± 0.09 | 0.72 ± 0.04 |
| 80 | 1053.58 | Undecane, 5-methyl- | Hydrocarbons | 1632-70-8 | 0.79 ± 0.36 | 1.13 ± 0.13 | 0.81 ± 0.19 | 1.19 ± 0.01 | 0.77 ± 0.08 | 0.68 ± 0.15 |
| 81 | 1298.5 | Decane, 2,4-dimethyl- | Hydrocarbons | 2801-84-5 | 0.76 ± 0.05 | 0.87 ± 0.13 | 0.63 ± 0.05 | 0.87 ± 0.04 | 0.68 ± 0.04 | 0.52 ± 0.13 |
| 82 | 1070.91 | trans-Linalool oxide (furanoid) | Heterocyclic compound | 34,995-77-2 | 0.31 ± 0.02 | 0.25 ± 0.01 | 2.65 ± 0.07 | 2.77 ± 0.09 | 1.52 ± 0.15 | 1.67 ± 0.16 |
| 83 | 1437.17 | (*E*)-2-Hexenoic acid, butyl ester | Ester | 54,411-16-4 | 0.41 ± 0.08 | 0.32 ± 0.03 | 1.56 ± 0.18 | 1.53 ± 0.05 | 5.74 ± 0.27 | 8.14 ± 0.61 |
| 84 | 1175.18 | (3*R*,6*S*)-2,2,6-Trimethyl-6-vinyltetrahydro-2H-pyran-3-ol | Heterocyclic compound | 39,028-58-5 | 0.15 ± 0.01 | 0.22 ± 0.03 | 2.50 ± 0.14 | 2.85 ± 0.14 | 1.90 ± 0.25 | 1.96 ± 0.03 |
| 85 | 1491.4 | Jasmine lactone | Ketone | 25,524-95-2 | 0.49 ± 0.07 | 1.34 ± 0.08 | 2.00 ± 0.35 | 2.39 ± 0.15 | 2.51 ± 0.40 | 2.71 ± 0.23 |
| 86 | 1353.99 | 2,6-Octadienoic Acid, 3,7-dimethyl-, (E)- | Acid | 4698-08-2 | 0.31 ± 0.15 | 0.39 ± 0.05 | 4.44 ± 0.74 | 3.73 ± 0.43 | 1.49 ± 0.25 | 2.06 ± 0.24 |
| 87 | 1257.39 | 2,6,6-trimethyl-1-Cyclohexene-1-acetAldehyde | Aldehyde | 472-66-2 | 0.56 ± 0.03 | 0.64 ± 0.08 | 2.23 ± 0.12 | 2.54 ± 0.18 | 1.13 ± 0.15 | 1.10 ± 0.14 |
| 88 | 1593.22 | BenzAldehyde, 2,4-dihydroxy-3,6-dimethyl- | Aldehyde | 34,883-14-2 | 0.88 ± 0.20 | 1.19 ± 0.13 | 1.08 ± 0.18 | 1.19 ± 0.04 | 1.46 ± 0.25 | 1.14 ± 0.14 |
| 89 | 1394.17 | Cis-Jasmone | Ketone | 488-10-8 | 0.69 ± 0.12 | 1.76 ± 0.09 | 0.84 ± 0.13 | 1.07 ± 0.07 | 0.41 ± 0.05 | 0.40 ± 0.02 |
| 90 | 1573.33 | 2-(formyloxy)-1-phenyl-Ethanone | Ester | 55,153-12-3 | 0.67 ± 0.08 | 1.17 ± 0.11 | 2.47 ± 0.38 | 1.90 ± 0.06 | 0.69 ± 0.14 | 0.98 ± 0.09 |
| 91 | 1268.14 | Nonanoic Acid | Acid | 112-05-0 | 0.73 ± 0.13 | 0.98 ± 0.09 | 1.49 ± 0.34 | 1.23 ± 0.28 | 2.38 ± 0.50 | 2.51 ± 0.55 |
| 92 | 1011.55 | Decane, 5-methyl- | Hydrocarbons | 13,151-35-4 | 1.16 ± 0.21 | 1.11 ± 0.21 | 6.14 ± 1.10 | 9.78 ± 1.06 | – | – |
| 93 | 1205.23 | Decanal | Aldehyde | 112-31-2 | 0.51 ± 0.02 | 0.78 ± 0.08 | 1.78 ± 0.08 | 2.48 ± 0.15 | 1.68 ± 0.45 | 1.35 ± 0.26 |
| 94 | 1249.83 | Geraniol | Terpenoids | 106-24-1 | 0.44 ± 0.02 | 0.58 ± 0.11 | 1.43 ± 0.11 | 1.47 ± 0.11 | 1.17 ± 0.05 | 1.53 ± 0.01 |
| 95 | 1197.22 | L-α-Terpineol | Terpenoids | 10,482-56-1 | 0.98 ± 0.06 | 1.22 ± 0.12 | 0.76 ± 0.07 | 0.79 ± 0.04 | 0.62 ± 0.07 | 0.55 ± 0.03 |
| 96 | 1225.24 | 2,6-Octadien-1-ol, 3,7-dimethyl-, (*Z*)- | Alcohol | 106-25-2 | 0.93 ± 0.17 | 0.71 ± 0.04 | 1.17 ± 0.11 | 1.27 ± 0.11 | 1.00 ± 0.09 | 1.38 ± 0.05 |
| 97 | 1182.77 | L-4-terpineol | Terpenoids | 20,126-76-5 | 1.06 ± 0.07 | 0.80 ± 0.06 | 1.46 ± 0.01 | 1.73 ± 0.11 | 0.74 ± 0.08 | 0.67 ± 0.07 |
| 98 | 1101.31 | Linalool | Terpenoids | 78-70-6 | 0.43 ± 0.04 | 0.52 ± 0.02 | 1.29 ± 0.01 | 1.35 ± 0.05 | 1.23 ± 0.09 | 1.64 ± 0.15 |
| 99 | 1103.5 | 1,5,7-Octatrien-3-ol, 3,7-dimethyl- | Alcohol | 29,957-43-5 | 1.01 ± 0.07 | 0.75 ± 0.04 | 1.66 ± 0.13 | 1.47 ± 0.09 | 0.61 ± 0.07 | 0.73 ± 0.06 |
| 100 | 1220.95 | 2,6,6-trimethyl-1-Cyclohexene-1-carboxaldehyde | Terpenoids | 432-25-7 | 0.58 ± 0.04 | 0.64 ± 0.03 | 1.23 ± 0.04 | 1.29 ± 0.07 | 1.03 ± 0.00 | 1.13 ± 0.11 |
| 101 | 1267.57 | 2,6-Octadienal, 3,7-dimethyl-, (*E*)- | Terpenoids | 141-27-5 | 0.44 ± 0.03 | 0.39 ± 0.02 | 1.89 ± 0.23 | 1.94 ± 0.22 | 2.13 ± 0.12 | 2.49 ± 0.09 |
| 102 | 1194.38 | Methyl salicylate | Ester | 119-36-8 | 0.12 ± 0.03 | 0.06 ± 0.00 | 1.65 ± 0.11 | 1.70 ± 0.08 | 0.96 ± 0.03 | 1.25 ± 0.02 |
| 103 | 1345.34 | Methyl anthranilate | Ester | 134-20-3 | 1.56 ± 0.47 | 1.46 ± 0.05 | 1.19 ± 0.21 | 1.28 ± 0.08 | 1.26 ± 0.08 | 1.19 ± 0.01 |
| 104 | 1313.12 | Ethanone, 1-(2-hydroxy-5-methylphenyl)- | Ketone | 1450-72-2 | 1.13 ± 0.24 | 3.58 ± 0.06 | 0.75 ± 0.09 | 0.63 ± 0.06 | 1.23 ± 0.18 | 1.12 ± 0.08 |
| 105 | 1144.57 | 2-Methyl-7-*exo*-vinylbicyclo[4.2.0]oct-1(2)-ene | Hydrocarbons | 107,914-89-6 | 1.59 ± 0.11 | 1.59 ± 0.18 | 0.44 ± 0.02 | 0.53 ± 0.05 | 0.53 ± 0.04 | 0.91 ± 0.01 |
| 106 | 1280.53 | Benzene, pentamethyl- | Aromatics | 700-12-9 | 1.42 ± 0.04 | 1.20 ± 0.12 | 0.91 ± 0.02 | 1.40 ± 0.09 | 1.52 ± 0.23 | 1.14 ± 0.15 |
| 107 | 1287.43 | Anethole | Terpenoids | 104-46-1 | 0.82 ± 0.10 | 1.18 ± 0.08 | 0.87 ± 0.12 | 1.17 ± 0.11 | 1.45 ± 0.08 | 0.95 ± 0.05 |
| 108 | 1277.95 | 4-Formyl-3,5-dimethyl-1H-pyrrole-2-carbonitrile | Heterocyclic compound | 1,000,296-05-3 | 1.02 ± 0.31 | 1.15 ± 0.05 | 1.18 ± 0.27 | 1.24 ± 0.16 | 1.49 ± 0.20 | 1.64 ± 0.10 |
| 109 | 1148.7 | 4H-Pyran-4-one, 2,3-dihydro-3,5-dihydroxy-6-methyl- | Ketone | 28,564-83-2 | 0.00 ± 0.00 | 0.46 ± 0.40 | 2.71 ± 0.82 | 0.56 ± 0.18 | 0.20 ± 0.17 | 0.37 ± 0.25 |
| 110 | 1300.17 | Naphthalene, 2-methyl- | Aromatics | 91-57-6 | 1.54 ± 0.15 | 1.26 ± 0.09 | 0.86 ± 0.10 | 1.23 ± 0.10 | 1.80 ± 0.28 | 1.26 ± 0.08 |

(*continued on next page*)

**Table 1** (*continued*)

| No | RI | components | Class I | CAS | Relative Content (μg/g) | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | NGT | GGT | NBT | GBT | NWT | GWT |
| 111 | 1016.79 | (+)-4-Carene | Terpenoids | 29,050-33-7 | 0.85 ± 0.16 | 0.70 ± 0.12 | 1.26 ± 0.05 | 1.44 ± 0.04 | 0.83 ± 0.05 | 1.10 ± 0.14 |
| 112 | 1029.13 | ᴅ-Limonene | Terpenoids | 5989-27-5 | 0.77 ± 0.14 | 0.88 ± 0.15 | 1.00 ± 0.04 | 1.20 ± 0.03 | 0.67 ± 0.02 | 0.75 ± 0.08 |
| 113 | 989.67 | β-Myrcene | Terpenoids | 123-35-3 | 0.31 ± 0.05 | 0.46 ± 0.08 | 1.64 ± 0.08 | 1.63 ± 0.05 | 1.09 ± 0.09 | 1.92 ± 0.14 |
| 114 | 1006.64 | (*S*)-(+)-α-Phellandrene | Terpenoids | 2243-33-6 | 0.67 ± 0.09 | 0.50 ± 0.06 | 1.51 ± 0.04 | 1.52 ± 0.06 | 1.05 ± 0.02 | 1.84 ± 0.01 |
| 115 | 1127.36 | 2,4,6-Octatriene, 3,4-dimethyl- | Hydrocarbons | 57,396-75-5 | 0.63 ± 0.12 | 0.44 ± 0.08 | 1.52 ± 0.09 | 1.63 ± 0.04 | 0.85 ± 0.02 | 1.41 ± 0.15 |
| 116 | 1058.03 | .gamma.-Terpinene | Terpenoids | 99-85-4 | 0.94 ± 0.21 | 0.87 ± 0.14 | 1.05 ± 0.07 | 1.36 ± 0.03 | 0.75 ± 0.10 | 0.84 ± 0.13 |
| 117 | 1045.17 | β-Ocimene | Terpenoids | 13,877-91-3 | 0.34 ± 0.04 | 0.24 ± 0.03 | 6.23 ± 0.35 | 8.01 ± 0.25 | 1.94 ± 0.32 | 2.10 ± 0.20 |
| 118 | 1120.46 | Benzene, 1,2,3,5-tetramethyl- | Aromatics | 527-53-7 | 1.10 ± 0.17 | 1.27 ± 0.12 | 0.92 ± 0.07 | 1.26 ± 0.05 | 1.10 ± 0.26 | 0.87 ± 0.15 |
| 119 | 1130.49 | 1,3,8-p-Menthatriene | Terpenoids | 18,368-95-1 | 0.94 ± 0.07 | 0.68 ± 0.12 | 1.44 ± 0.11 | 1.46 ± 0.08 | 0.47 ± 0.02 | 0.82 ± 0.07 |
| 120 | 1024.38 | Benzene, 1-methyl-3-(1-methylethyl)- | Aromatics | 535-77-3 | 0.88 ± 0.23 | 0.82 ± 0.10 | 1.13 ± 0.11 | 1.44 ± 0.01 | 0.87 ± 0.14 | 0.82 ± 0.13 |
| 121 | 1091 | Benzene, 1-methyl-3-(1-methylethenyl)- | Aromatics | 1124-20-5 | 0.95 ± 0.11 | 0.79 ± 0.12 | 1.19 ± 0.04 | 1.41 ± 0.02 | 0.57 ± 0.06 | 0.58 ± 0.05 |
| 122 | 1149.59 | Benzene, 4-ethenyl-1,2-dimethyl- | Aromatics | 27,831-13-6 | 1.18 ± 0.13 | 1.29 ± 0.10 | 0.86 ± 0.07 | 1.19 ± 0.04 | 1.27 ± 0.29 | 0.97 ± 0.18 |
| 123 | 1111.77 | Cyclohexanol, 2,6-dimethyl- | Alcohol | 5337-72-4 | 0.41 ± 0.03 | 0.50 ± 0.14 | 0.93 ± 0.04 | 1.20 ± 0.08 | 3.44 ± 0.38 | 3.70 ± 0.26 |
| 124 | 980.83 | 1-Octen-3-ol | Alcohol | 3391-86-4 | 0.27 ± 0.02 | 0.47 ± 0.04 | 0.30 ± 0.01 | 0.28 ± 0.02 | 0.45 ± 0.06 | 0.52 ± 0.15 |
| 125 | 1188.68 | Naphthalene | Aromatics | 91-20-3 | 1.46 ± 0.14 | 1.27 ± 0.08 | 0.82 ± 0.06 | 1.07 ± 0.10 | 1.60 ± 0.23 | 1.26 ± 0.07 |
| 126 | 975.03 | 3,5,5-trimethyl-2-Hexene | Hydrocarbons | 26,456-76-8 | 0.36 ± 0.05 | 0.65 ± 0.06 | 0.25 ± 0.01 | 0.20 ± 0.01 | 0.17 ± 0.04 | 0.38 ± 0.13 |
| 127 | 1012.98 | Benzyl chloride | Halogenated hydrocarbons | 100-44-7 | 0.61 ± 0.14 | 1.07 ± 0.17 | 1.89 ± 0.02 | 2.53 ± 0.09 | 0.66 ± 0.15 | 0.54 ± 0.15 |
| 128 | 1047.82 | 3-Formyl-4,5-dimethyl-pyrrole | Heterocyclic compound | 1,000,145-89-7 | 0.62 ± 0.10 | 0.87 ± 0.04 | 2.81 ± 0.27 | 2.15 ± 0.12 | 0.15 ± 0.02 | 0.15 ± 0.00 |
| 129 | 709 | Propane, 2-chloro-2-nitro- | Halogenated hydrocarbons | 594-71-8 | 0.84 ± 0.74 | 0.00 ± 0.00 | 1.25 ± 0.44 | 1.46 ± 0.26 | 0.47 ± 0.43 | 0.92 ± 0.05 |
| 130 | 841.47 | 1,3-Cyclopentadiene, 5,5-dimethyl-1-ethyl- | Hydrocarbons | 1,000,162-25-7 | 0.48 ± 0.11 | 0.53 ± 0.10 | 1.25 ± 0.18 | 2.02 ± 0.04 | 0.76 ± 0.07 | 0.79 ± 0.17 |
| 131 | 1114.93 | Phenylethyl Alcohol | Alcohol | 60-12-8 | 0.17 ± 0.03 | 0.18 ± 0.01 | 2.75 ± 0.35 | 2.95 ± 0.27 | 1.96 ± 0.18 | 3.03 ± 0.22 |
| 132 | 934 | Phenol, 3,5-dimethyl- | Phenol | 108-68-9 | 0.75 ± 0.11 | 1.21 ± 0.18 | 0.66 ± 0.05 | 0.72 ± 0.04 | 0.91 ± 0.48 | 0.90 ± 0.15 |
| 133 | 1219.63 | 2,3-dihydro-Benzofuran | Heterocyclic compound | 496-16-2 | 1.22 ± 0.23 | 1.34 ± 0.08 | 0.95 ± 0.03 | 0.77 ± 0.01 | 1.17 ± 0.14 | 1.12 ± 0.05 |
| 134 | 1295.1 | Indole | Heterocyclic compound | 120-72-9 | 0.47 ± 0.12 | 5.95 ± 0.16 | 0.35 ± 0.05 | 0.37 ± 0.03 | 0.27 ± 0.04 | 0.28 ± 0.02 |
| 135 | 902.75 | 2-Heptanol | Alcohol | 543-49-7 | 0.03 ± 0.00 | 0.03 ± 0.00 | 1.84 ± 0.14 | 2.01 ± 0.09 | 2.31 ± 0.37 | 4.76 ± 1.12 |
| 136 | 901.74 | Heptanal | Aldehyde | 111-71-7 | 0.16 ± 0.02 | 0.19 ± 0.04 | 1.97 ± 0.23 | 2.20 ± 0.11 | 2.36 ± 0.48 | 3.96 ± 0.90 |
| 137 | 888.82 | 4-Methyl-5-hexen-2-ol | Alcohol | 71,228-22-3 | – | – | 2.29 ± 0.11 | 2.13 ± 0.08 | 2.40 ± 0.42 | 5.81 ± 1.26 |
| 138 | 751.4 | Cyclobutanone, 2,2,3-trimethyl- | Ketone | 1449-49-6 | 0.78 ± 0.26 | 1.02 ± 0.27 | 0.99 ± 0.18 | 1.30 ± 0.04 | 0.76 ± 0.13 | 1.25 ± 0.42 |
| 139 | 1020.73 | 5-methyl-1,2,5,6-Tetrahydropyridin-2-one | Nitrogen compounds | 1,000,197-00-2 | 0.23 ± 0.20 | 0.00 ± 0.00 | 2.48 ± 0.21 | 5.29 ± 2.93 | 1.16 ± 1.05 | 1.32 ± 0.18 |
| 140 | 1036.04 | Benzyl Alcohol | Alcohol | 100-51-6 | 0.28 ± 0.04 | 0.27 ± 0.01 | 2.92 ± 0.34 | 2.87 ± 0.26 | 1.55 ± 0.16 | 2.31 ± 0.11 |
| 141 | 963.89 | BenzAldehyde | Aldehyde | 100-52-7 | 0.54 ± 0.08 | 0.44 ± 0.01 | 2.63 ± 0.04 | 3.29 ± 0.10 | 2.17 ± 0.29 | 2.27 ± 0.14 |
| 142 | 955.93 | Dihydro-3-(2H)-thiophenone | Heterocyclic compound | 1003-04-9 | 0.66 ± 0.08 | 0.58 ± 0.03 | 1.43 ± 0.14 | 1.34 ± 0.07 | 1.01 ± 0.15 | 0.99 ± 0.05 |
| 143 | 856.29 | 3-Hexen-1-ol, (E)- | Alcohol | 928-97-2 | 0.17 ± 0.01 | 0.11 ± 0.02 | 3.29 ± 0.08 | 3.74 ± 0.26 | 1.03 ± 0.15 | 1.98 ± 0.27 |
| 144 | 797.72 | Hexanal | Aldehyde | 66-25-1 | 0.15 ± 0.13 | 0.20 ± 0.02 | 2.66 ± 0.36 | 4.35 ± 0.12 | 3.36 ± 1.33 | 2.38 ± 0.72 |
| 145 | 797.57 | 3-Hexen-2-one | Ketone | 763-93-9 | 0.52 ± 0.12 | 0.53 ± 0.22 | 1.84 ± 0.23 | 2.79 ± 0.09 | 4.36 ± 4.30 | 2.00 ± 0.55 |
| 146 | 797.09 | 2-methoxy-Furan | Heterocyclic compound | 25,414-22-6 | 1.94 ± 0.21 | 1.58 ± 0.10 | 1.04 ± 0.09 | 0.95 ± 0.04 | 0.46 ± 0.07 | 0.53 ± 0.04 |
| 147 | 701.52 | Dimethylphosphinic fluoride | Halogenated hydrocarbons | 753-70-8 | 0.26 ± 0.02 | 0.29 ± 0.04 | 2.20 ± 0.57 | 3.91 ± 0.09 | 1.51 ± 0.13 | 1.67 ± 0.51 |

(*continued on next page*)

**Table 1** (*continued*)

| No | RI | components | Class I | CAS | Relative Content (μg/g) | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | NGT | GGT | NBT | GBT | NWT | GWT |
| 148 | 809.67 | 3-ethyl-1H-Pyrrole | Heterocyclic compound | 1551-16-2 | 1.19 ± 0.18 | 1.78 ± 0.14 | 0.80 ± 0.10 | 0.79 ± 0.06 | 0.12 ± 0.01 | 0.12 ± 0.02 |
| 149 | 769.54 | 1-Pentanol | Alcohol | 71-41-0 | 0.37 ± 0.06 | 0.49 ± 0.02 | 2.95 ± 0.05 | 2.39 ± 0.08 | 0.32 ± 0.06 | 0.43 ± 0.03 |

'–' indicates that it is not detected. Each tea sample was measured in parallel for 3 times, and all data were expressed as mean value ± SD.

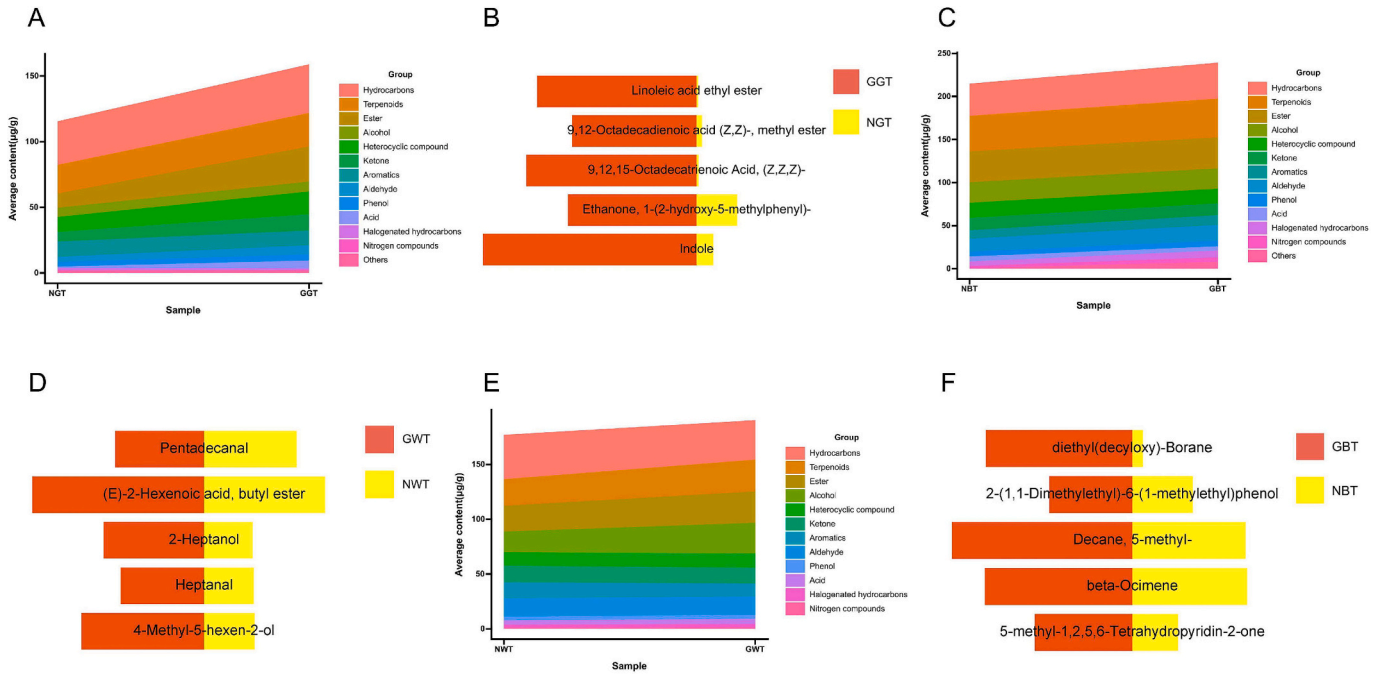


**Fig. 3.** (A) The proportion diagram of volatile components group content (NGT vs GGT). (B) The difference comparison diagram of the five volatile components with the highest content (NGT vs GGT). (C) The proportion diagram of volatile components group content (NBT vs GBT). (D) The difference comparison diagram of the five volatile components with the highest content (NBT vs GBT). (E) The proportion diagram of volatile components group content (NWT vs GWT). (F) The difference comparison diagram of the five volatile components with the highest content (NWT vs GWT).

employed PCA and ROAV to explore the different VOCs.

### 3.3. PCA and OPLS-DA analysis of the VOCs of different types of GABA tea

The PCA map effectively illustrates the abundance of VOCs in the samples. The closer the samples are on the map, the more similar they are (Chen et al., 2022; Chen et al., 2022). We observed that GGT, GBT, and GWT could be clustered separately, with a clear distinction among the groups (Fig. 4A, B, and C). Furthermore, we used OPLS-DA to distinguish and analyze the differential VOCs among the tea groups (Fig. 4D, E, and F) (Feng et al., 2023).

A total of 44 differential VOCs were identified by comparing the VOCs of NGT and GGT (VIP $> 1$, $p < 0.05$). Compared to NGT, GGT exhibited an upregulation of 34 VOCs, including linoleic acid ethyl ester, anethole, linalool, etc., and a downregulation of 10 VOCs, including (E)-3-hexen-1-ol, β-ocimene, trans-linalool oxide (furanoid), etc. (Fig. 4G). A total of 47 differential VOCs were identified between NBT and GBT (VIP $> 1$, $p < 0.05$). Compared to NBT, GBT exhibited an upregulation of 31 VOCs, including hexanoic acid, (Z)-3-hexenyl ester, teaspirane, etc., and a downregulation of 16 VOCs, including hexadecanoic acid methyl ester, aspirin, 1-pentanol, etc. (Fig. 4H). Finally, 40 differential VOCs were identified between NWT and GWT (VIP $> 1$, $p < 0.05$). Compared to NWT, GWT exhibited an upregulation of 25 VOCs, including linalool, geraniol, trans-nerolidol, etc., and a downregulation of 15 VOCs, including anethole, pentadecane, hexadecane, etc. (Fig. 4I).

Previous studies suggested that the VOCs in tea were primarily formed through four pathways: precursors from carotenoids, lipids, and glycosides, as well as the Maillard reaction pathway (Feng et al., 2019). Most characteristic VOCs in green tea arise from thermal degradation and lipid oxidation pathways (Guo, Ho, Schwab, & Wan, 2021). However, in the current study, GGT exhibited high relative levels of terpenoids and ketones. This finding might be attributed to the anaerobic treatment of tea, which enhances the accumulation of glycosides and carotenoids that act as precursors for VOCs (Wu et al., 2018). The processing methods for white and black teas also included the withering step. After enzymatic oxidation, the VOCs derived from carotenoids and glycosides significantly impacted the aromas of GWT and GBT (Chen, Hu, et al., 2022; Chen, Zhu, et al., 2022; Hao et al., 2023). Additionally, the enzymatic oxidation of lipids during the withering of white tea and the non-enzymatic oxidation of carotenoids under acidic conditions during the fermentation of black tea increased the levels of VOCs (Wu et al., 2022; Yao et al., 2023). Therefore, to explore the characteristic VOCs of GABA teas post-anaerobic processing, further investigation into the aroma contribution values of these VOCs is warranted.

### 3.4. Key active components underlying different types of aroma

The VOC compositions of GGT, GBT, and GWT were highly complex. PCA and OPLS-DA analyses revealed that not all VOCs significantly

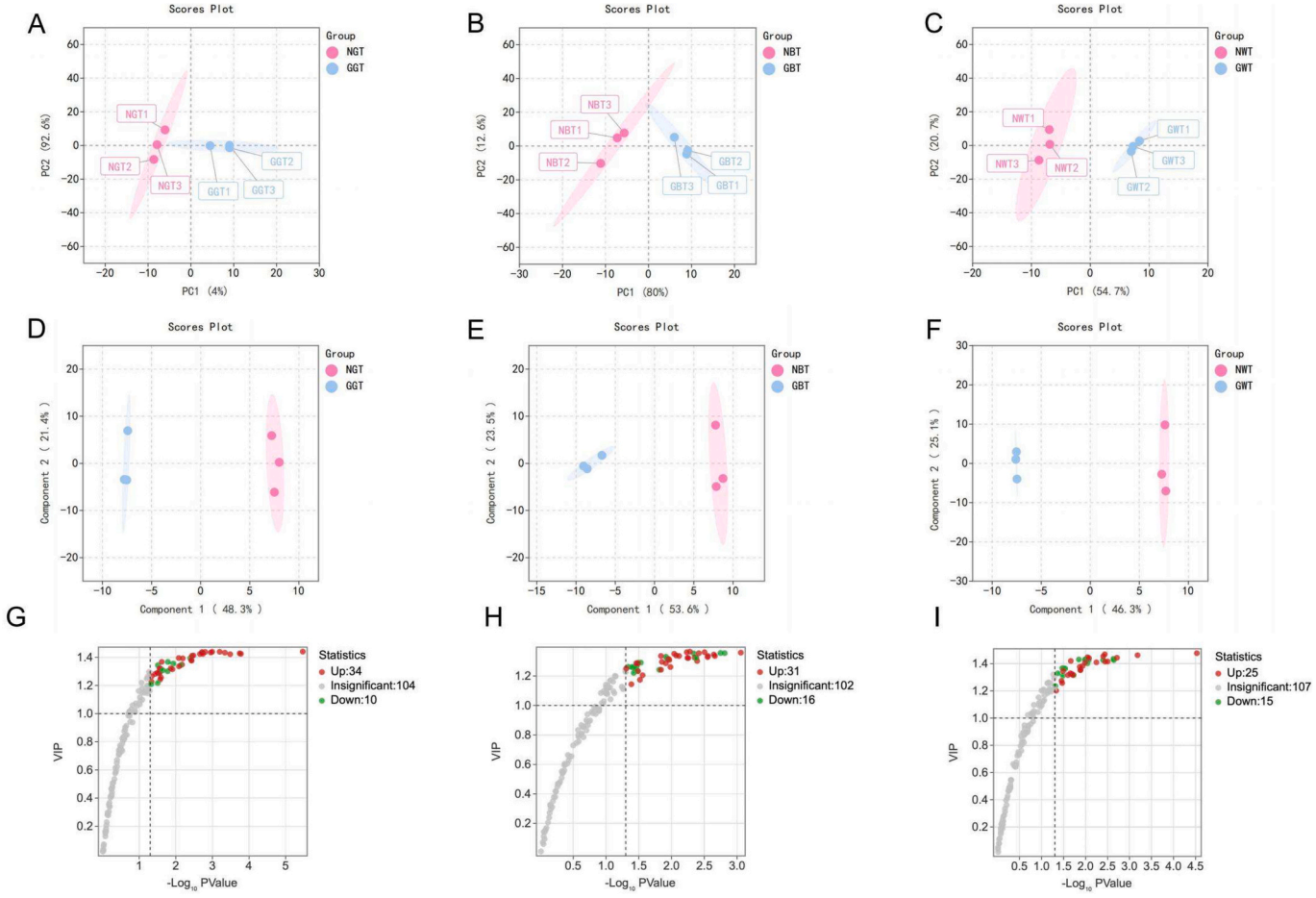


**Fig. 4.** (A) PCA model score scatter plot (NGT vs GGT). (B) PCA model score scatter plot (NBT vs GBT). (C) PCA model score scatter plot (NWT vs GWT). (D) Scatter plot of scores of OPLS-DA model (NGT vs GGT). (E) Scatter plot of scores of OPLS-DA model (NBT vs GBT). (F) Scatter plot of scores of OPLS-DA model (NWT vs GWT). (G) Volcano plot of differential components (NGT vs GGT). (H) Volcano plot of differential components (NBT vs GBT). (I) Volcano plot of differential components (NWT vs GWT).

impacted the tea aroma. For instance, some VOCs were identified as differential but contributed only minimally to the overall aroma due to their tastelessness or low OAV. Therefore, we assessed the key VOCs that substantially influenced the aromas of GGT, GBT, and GWT. Analysis of ROAVs and the overall aroma revealed 12, 16, and 16 key VOCs in GGT, GBT, and GWT, respectively (Table 2).

In GGT (Fig. 5A), only β-damascenone (fruity, flowery) had an ROAV of 100. The VOCs with 1 < ROAV <100 included linalool (woody, flowery, fruity, sweet), cis-jasmone (flowery, tender), 1-octen-3-ol (clean, fatty, mushroomy) and decanal (sweet-orange). The VOCs with 0 < ROAV <1 included 1,3,8-p-menthatriene (grassy, flowery), β-ocimene (woody, flowery), indole (flowery, fresh), jasmine lactone (sour-fruity), linoleic acid ethyl ester (fruity, flowery), phytol (fragrant), and anethole (licorice) (Table S1). Due to its significantly higher ROAV, we inferred that β-damascenone dominated the GGT aroma, imparting a flowery and fruity character.

In GBT (Fig. 5B), only decanal (sweet-orange) had an ROAV of 100. The VOCs with 10 < ROAV <100 included β-ocimene (woody, flowery), 2-methyl-naphthalene (licorice), benzaldehyde (nut, flowery, fruity) and cis-3-hexenyl valerate (clean and refreshing). The VOCs with 1 < ROAV <10 included D-limonene (flowery, lemony), naphthalene (fragrant), hexanal (fruity, honey), 1,2,3,5-tetramethylbenzene (camphoric), anethole (licorice), L-4-terpineol (woody, flowery) and hexanoic acid, 3-hexenyl ester (Z) (fruity). The VOCs with 0 < ROAV <1 included teaspirane (fruity, woody, sweet), gamma-terpinene (orange, lemony), 1-pentanol (fruity) and (*E*)-hexanoic acid, 2-hexenyl ester (fruity) (Table S1). GBT primarily exhibited flowery, fruity, sweet, honey, and rich aromas. Since it contained several components with ROAV >10, GBT demonstrated a wide variety of pleasant characteristic aromas.

In GWT (Fig. 5C), similar to GGT, only β-damascenone (fruity, flowery) had an ROAV of 100. The VOCs with 10 < ROAV <100 included only linalool (woody, flowery, fruity, sweet), while those with 1 < ROAV <10 included geraniol (flowery) and β-myrcene (fatty). The VOCs with 0 < ROAV <1 included phenylethyl alcohol (flowery), α-citral (lemony), 1,3,8-p-menthatriene (grassy, flowery), methyl salicylate (caramel, peppermint), nerol (fruity, flowery), (*S*)-(+)-α-phellandrene (lemony), (E)-hexanoic acid, 2-hexenyl ester (fruity), (E)-3-hexen-1-ol (clean and refreshing), benzyl alcohol (sweet, flowery), anethole (licorice), 3-hexen-1-ol benzoate (fragrant), and phytol (fragrant) (Table S1). GWT primarily exhibited flowery and fruity (sour fruity) aromas. These findings indicated that β-damascenone (ROAV = 100) significantly impacted the aroma of GWT.

In GGT (Fig. 5D), β-damascenone (ROAV = 100) was the primary contributor to the overall tea aroma, followed by linalool, cis-jasmone, 1-octen-3-ol, and decanal (ROAV = 7.35, 5.38, 5.06, and 3.33, respectively). This finding was consistent with previous research indicating that linalool significantly contributes to the aroma of baked green tea in Yunnan (Wang et al., 2016). To the best of our knowledge, this study was the first to report β-damascenone as a major contributor to the aroma of baked green tea, while cis-jasmone, 1-octen-3-ol, and decanal play important roles in enhancing the flowery and fruity aroma of GGT. In

**Table 2**
ROAV and aroma types of key aroma components in different types of GABA tea.

| Index | ROAV | | | VIP | | | Fold Change | | | Aroma type |
|---|---|---|---|---|---|---|---|---|---|---|
| | GGT | GBT | GWT | GGTvsNGT | GBTvsNBT | GWTvsNWT | GGTvsNGT | GBTvsNBT | GWTvsNWT | |
| Linoleic acid ethyl ester | 0.05 | – | – | 1.44 | – | – | 108.62 | – | – | Fruity, Flowery |
| Phytol | 0.05 | – | 0.01 | 1.35 | – | 1.43 | 1.41 | – | 0.72 | Fragrant |
| β-Damascenone | 100.00 | – | 100.00 | 1.39 | – | 1.35 | 1.95 | – | 1.47 | Fruity, Flowery |
| Jasmine lactone | 0.10 | – | – | 1.43 | – | – | 2.74 | – | – | Sour-fruity |
| Cis-Jasmone | 5.38 | – | – | 1.42 | – | – | 2.53 | – | – | Flowery,Tender |
| Decanal | 3.33 | 100.00 | – | 1.37 | 1.32 | – | 1.53 | 1.40 | – | Sweet-orange |
| Linalool | 7.35 | – | 15.28 | 1.27 | – | 1.32 | 1.21 | – | 1.33 | Woody, Flowery, Fruity, Sweet |
| Anethole | 0.03 | 2.35 | 0.01 | 1.34 | 1.17 | 1.44 | 1.45 | 1.35 | 0.65 | Licorice |
| 1-Octen-3-ol | 5.06 | – | – | 1.41 | – | – | 1.75 | – | – | Clean, Fatty, Mushroomy |
| Indole | 0.25 | – | – | 1.44 | – | – | 12.75 | – | – | Flowery, Fresh |
| β-Ocimene | 0.28 | 86.30 | – | 1.22 | 1.33 | – | 0.72 | 1.29 | – | Woody, Flowery |
| 1,3,8-p-Menthatriene | 0.37 | – | 0.29 | 1.21 | – | 1.44 | 0.72 | – | 1.75 | Grassy, Flowery |
| Hexanoic acid, 3-hexenyl ester, (Z)- | – | 1.03 | – | – | 1.32 | – | – | 1.50 | – | Fruity |
| Teaspirane | – | 0.92 | – | – | 1.34 | – | – | 1.31 | – | Fruity, Woody, Sweet |
| cis-3-hexenyl valerate | – | 12.71 | – | – | 1.35 | – | – | 1.51 | – | Clean and refreshing |
| L-4-terpineol | – | 1.74 | – | – | 1.25 | – | – | 1.19 | – | Woody, Flowery |
| Naphthalene, 2-methyl- | – | 30.99 | – | – | 1.26 | – | – | 1.44 | – | Licorice |
| D-Limonene | – | 7.15 | – | – | 1.34 | – | – | 1.20 | – | Flowery, Lemony |
| .gamma.-Terpinene | – | 0.13 | – | – | 1.34 | – | – | 1.29 | – | Orange, Lemony |
| Benzene, 1,2,3,5-tetramethyl- | – | 4.15 | – | – | 1.32 | – | – | 1.37 | – | Camphoric |
| Naphthalene | – | 4.30 | – | – | 1.20 | – | – | 1.31 | – | Fragrant |
| BenzAldehyde | – | 22.09 | – | – | 1.36 | – | – | 1.25 | – | Nut, Flowery, Fruity |
| Hexanal | – | 4.18 | – | – | 1.34 | – | – | 1.64 | – | Fruity, Honey |
| (E)-Hexanoic acid, 2-hexenyl ester | – | 0.08 | 0.07 | – | 1.24 | 1.35 | – | 0.76 | 1.60 | Fruity |
| 1-Pentanol | – | 0.10 | – | – | 1.36 | – | – | 0.81 | – | Fruity |
| 3-Hexen-1-ol benzoate | – | – | 0.01 | – | – | 1.26 | – | – | 1.42 | Fragrant |
| Geraniol | – | – | 2.85 | – | – | 1.45 | – | – | 1.31 | Flowery |
| Nerol | – | – | 0.24 | – | – | 1.41 | – | – | 1.38 | Fruity, Flowery |
| α-citral | – | – | 0.87 | – | – | 1.32 | – | – | 1.17 | Lemony |
| Methyl salicylate | – | – | 0.29 | – | – | 1.46 | – | – | 1.30 | Caramel, Peppermint |
| β-Myrcene | – | – | 1.62 | – | – | 1.44 | – | – | 1.76 | Fatty |
| (S)-(+)-α-Phellandrene | – | – | 0.13 | – | – | 1.48 | – | – | 1.75 | Lemony |
| Phenylethyl Alcohol | – | – | 0.94 | – | – | 1.42 | – | – | 1.55 | Flowery |
| Benzyl Alcohol | – | – | 0.01 | – | – | 1.41 | – | – | 1.49 | Sweet, Flowery |
| 3-Hexen-1-ol, (E)- | – | – | 0.02 | – | – | 1.38 | – | – | 1.93 | Clean and refreshing |

'–' indicates that it is not detected. Each tea sample was measured in parallel for 3 times, and all data were expressed as mean value.

addition, due to the consumption of certain amino acid components (such as arginine) by polyamine degradation during anaerobic treatment, the heterocyclic compounds with toasty aroma do not have sufficient reaction precursors during the drying process, which is one of the reasons why the compounds with flowery and fruity aroma characteristics contribute relatively more prominently to the overall aroma (Yang et al., 2023). In GBT, decanal (ROAV = 100) was the predominant contributor to the overall tea aroma, followed by β-ocimene; 2-methylnaphthalene; benzaldehyde; cis-3-hexenyl valerate; D-limonene; naphthalene; hexanal; 1,2,3,5-tetramethylbenzene; anethole; L-4-terpineol; and hexanoic acid, 3-hexenyl ester, (*Z*)- (ROAV = 86.30, 30.99, 22.09, 12.71, 7.15, 4.30, 4.18, 4.15, 2.35, 1.74, and 1.03, respectively). In previous studies, GC-MS and OAV analyses showed that furfuryl alcohol and related alcohols contributed most prominently to the aroma of large-leaf black tea, which was contrary to our findings. Moreover, the present study identified the significant contributions of decanal, β-ocimene, benzaldehyde, and other components to the overall tea aroma. Decanal is synthesized via the esterification and hydrolysis of fatty acid components. In the GABA enrichment pathway, the content of fatty acid components positively correlates with the GABA levels. It aligns with the previous research on Keemun black tea (Niu et al., 2022), which reported that aldehydes and terpenes predominantly contributed to flowery, fruity, and fragrant aroma in black tea. In GWT, β-damascenone (ROAV = 100) was the primary contributor to the overall tea aroma, followed by linalool, geraniol, and β-myrcene (ROAV = 15.28, 2.855, and 1.62, respectively). This finding was consistent with previous studies reporting substantial contributions of linalool and geraniol to the aroma of GABA white tea (Li et al., 2023). However, contrary to these studies, we identified β-damascenone as the major contributor to the aroma of GWT. This discrepancy might be attributed to differences in processing parameters and tea varieties used across studies (Li et al., 2023).

The present study revealed, for the first time, the contribution of β-damascenone to the aroma of GABA green and white teas and the contribution of decanal to the aroma of GABA black tea. In the present study, the contributions of different VOCs to the flowery and fruity aroma characteristics of various GABA teas were determined using ROAV. The discrepancies between the findings of previous studies and the present work warrant further validation of the dynamic changes in VOCs during processing and the repeatability of the related assessment approaches.

### 3.5. Potential marker components underlying different GABA tea aroma

By determining the correlations among the VOCs by ROAVs, the contribution of characteristic VOCs to the overall tea aroma can be quantified, which serves as one of the screening criteria for potential marker components (Romeo, Mora, Noguera, & Vázquez, 2023). The Pearson product-moment correlation coefficient (PPMCC) was employed to validate the roles of characteristic VOCs. We found that

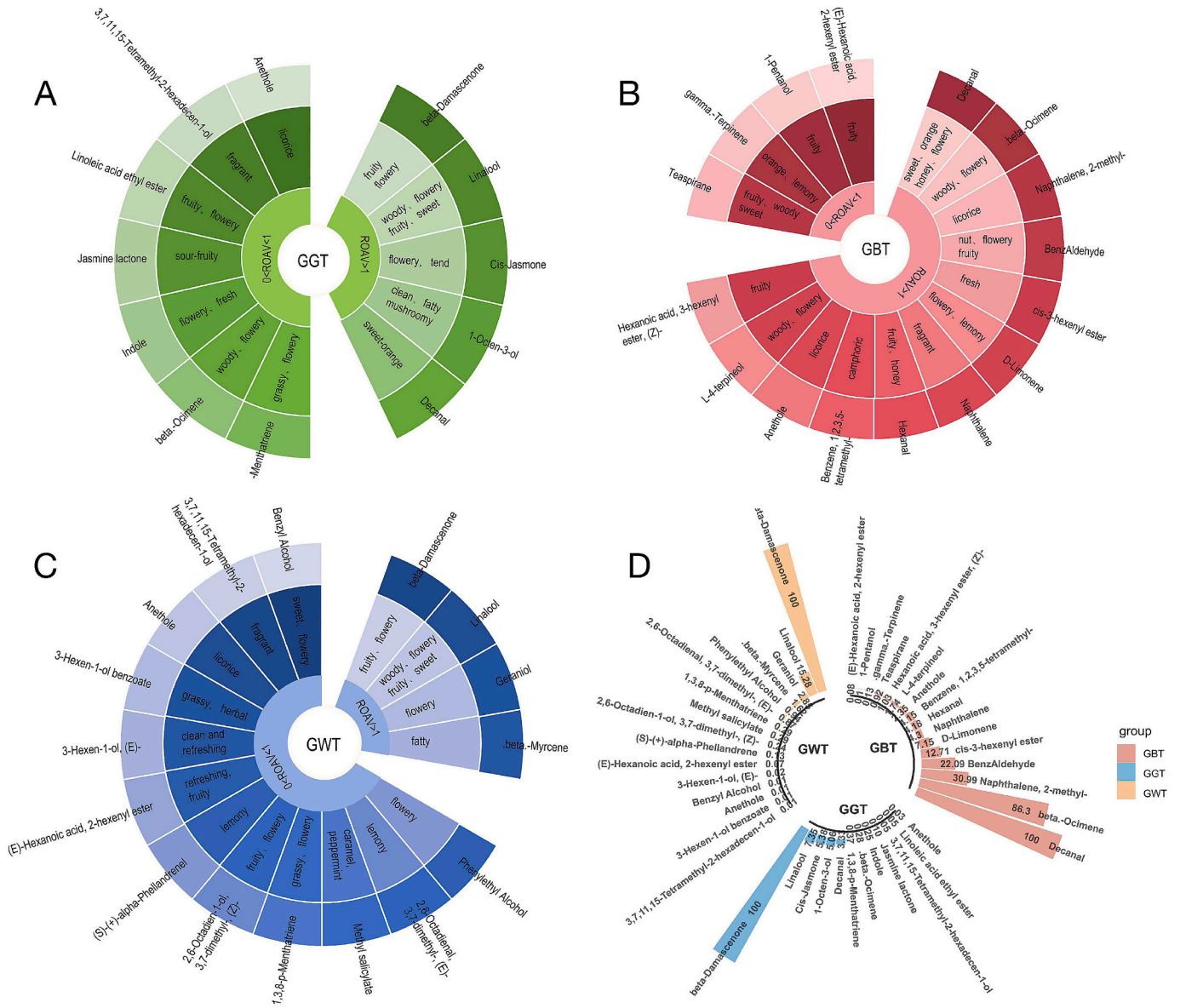


**Fig. 5.** (A) Discrimination of aroma types and contribution of volatile components based on ROAV (GGT). (B) Discrimination of aroma types and contribution of volatile components based on ROAV (GBT). (C) Discrimination of aroma types and contribution of volatile components based on ROAV (GWT). (D) The proportion of aroma contribution of volatile components in different types of GABA teas.

their effects on aroma types were markedly different, effectively corroborating the screening results of OPLS-DA (Fig. 6). We used the criteria of VIP $>$ 1, ROAV $>$1, $p < 0.05$, and PPMCC $>$0 to identify potential markers for the aromas of GGT, GBT, and GWT (Fig. 7).

We identified five potential markers in GGT: β-damascenone, linalool, cis-jasmone, 1-octen-3-ol, and decanal. Linalool and cis-jasmone have previously been reported as potential markers of the aroma of baked green tea (Yu et al., 2023). In GBT, we identified 12 potential markers, including decanal, β-ocimene, naphthalene, 2-methyl-naphthalene, benzaldehyde, cis-3-hexenyl valerate, D-limonene, hexanal, 1,2,3,5-tetramethylbenzene, anethole, L-4-terpineol, hexanoic acid, and (Z)-3-hexenyl ester. Among these, benzaldehyde has previously been reported as a potential marker of the aroma of Yunnan congou black tea (Ma et al., 2022). Finally, we identified four potential aroma markers in GWT: β-damascenone, linalool, geraniol, and β-myrcene. Among these, β-damascenone, linalool, and geraniol have previously been shown as potential aroma markers of Yunnan white tea (Ma, Gao, et al., 2023; Ma, Sun, et al., 2023).

To the best of our knowledge, this study was the first to assess different types of GABA teas processed from fresh leaves of large-leaf tea plants, exhibiting characteristic flowery and fruity aromas. Furthermore, potential marker components contributing to these aromas were identified. Among these, terpenoids were deemed major contributors to the overall tea aroma due to their low OTs, diverse varieties, and high relative contents, accentuating the flowery and fruity characteristics of GABA teas. Previous studies have indicated that the catalytic enzymes and reaction pathways involved in terpenoid synthesis are complex, particularly the single-enzyme catalytic processes (Sun et al., 2022; Xu et al., 2021). These findings underscore the importance of maintaining the quality of tea products rich in terpenoids. In the present study, the primary contributors to the aromas of GGT (β-damascenone and linalool), GBT (β-ocimene, D-limonene, anethole, and L-4-terpineol), and GWT (β-damascenone, linalool, geraniol, and β-myrcene) were terpenoids. Additionally, the contribution of terpenoids to the aromas of GABA teas increased to varying degrees following anaerobic processing. Under anaerobic conditions, the end product of glycolysis, pyruvate, cannot undergo further oxidation, leading to the accumulation of glyceraldehyde triphosphate, an intermediate product that enhances the

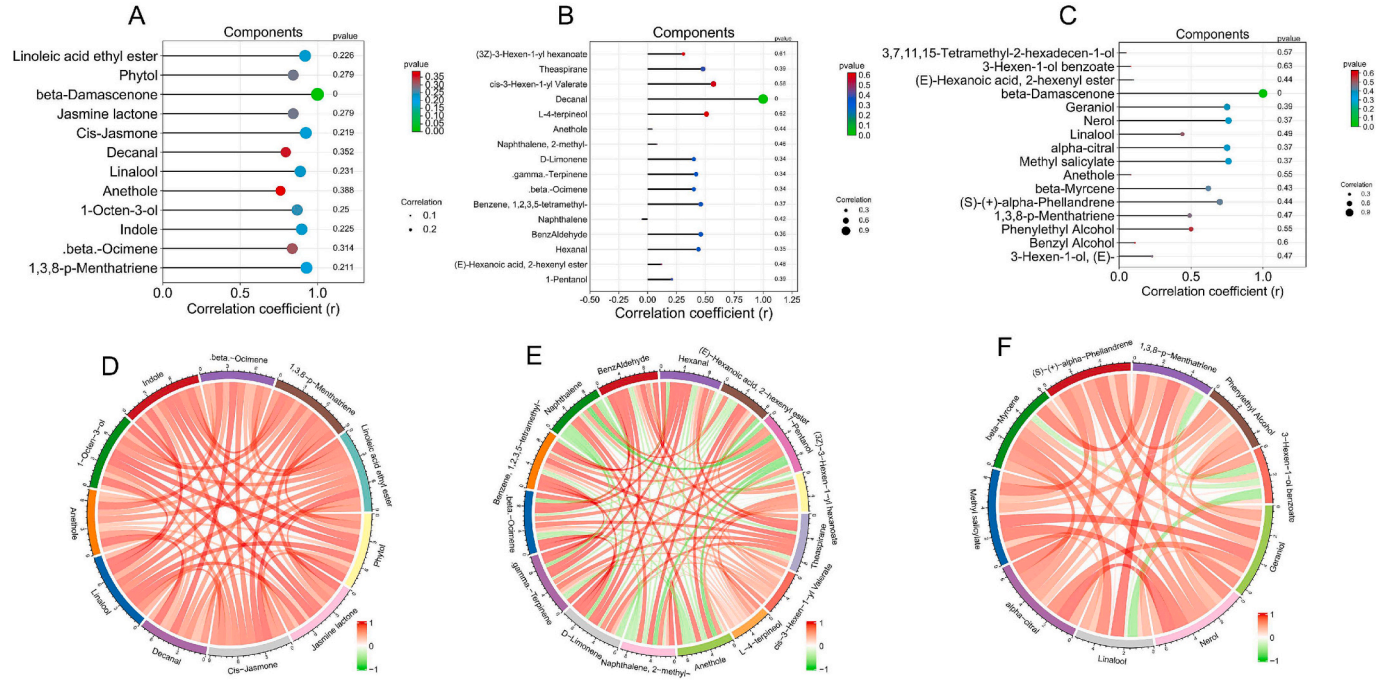


**Fig. 6.** Correlation analysis of characteristic aroma based on Pearson product moment correlation coefficient. (A) Pearson correlation coefficient diagram (GGT). (B) Pearson correlation coefficient diagram (GBT). (C) Pearson correlation coefficient diagram (GWT). (D) Correlation chord diagram (GGT). (E) Correlation chord diagram (GBT). (F) Correlation chord diagram (GWT).

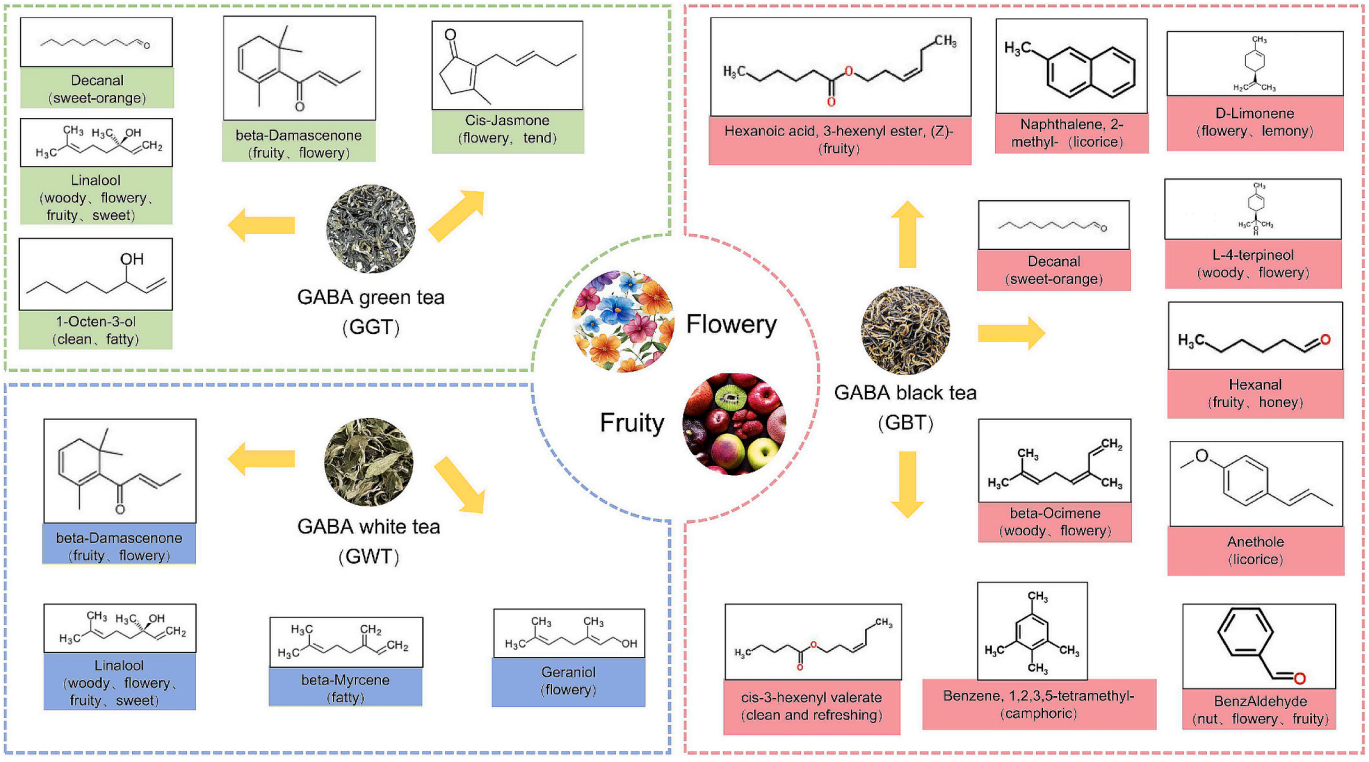


**Fig. 7.** Association diagram of potential marker volatile components and aroma types of different types of GABA teas.

enrichment of geranyl diphosphate, the synthetic precursor of terpenoids (Chen et al., 2020). Therefore, anaerobic treatment might augment the variety and levels of terpenoids in GABA teas. Furthermore, in the anaerobic environment, autooxidation might facilitate the enrichment of components such as cis-jasmone, 1-octen-3-ol, decanal, benzaldehyde, cis-3-hexenyl valerate, hexanal, hexanoic acid, and (Z)-3-hexenyl ester, which act as lipid precursors (Wang et al., 2021).

## 4. Conclusion

In this study, we identified the potential characteristic aroma components of different types of GABA tea. The aromas of GGT, GBT, and

GWT were unique, exhibiting more flowery and fruity notes than their conventional tea counterparts, as assessed by sensory evaluation. Furthermore, using HS-SPME-GC–MS and subsequent statistical analysis, 146, 149, and 147 VOCs and 44, 47, and 40 differential VOCs were detected in GGT, GBT, and GWT, respectively. Finally, 5, 12, and 4 potential marker VOCs were screened from GGT, GBT, and GWT, respectively, based on differential VOCs combined with ROAV analysis. By analyzing these potential characteristic markers, we determined that the aromas of different types of GABA tea were characterized by flowery and fruity notes. The identified potential markers included β-damascenone, linalool, cis-jasmone, and decanal for GGT; decanal, β-ocimene, benzaldehyde, D-limonene, hexanal, L-4-terpineol, hexanoic acid, and 3-hexenyl ester for GBT; and β-damascenone, linalool, and geraniol for GWT. Our study provided a theoretical basis for understanding the formation of flowery and fruity aromas in GABA teas and elucidated their characteristic VOCs, which can contribute to improving the quality of GABA teas. In future studies, response surface experiments can be designed to further optimize the processing parameters, providing a theoretical foundation for exploring quality optimization methods and ensuring the stability of tea product quality. Additionally, the dynamic change mechanisms and coordination of the characteristic VOCs associated with flowery and fruity aromas during GABA tea processing remain unclear and warrant further investigation.

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## Ethical statements

The experimental scheme involving sensory evaluation is in line with Chinese national law. In the course of the implementation of the study, no human body, animal and violation of law, morality or the 'Declaration of Helsinki 'were involved. And all participants have written consent.

## CRediT authorship contribution statement

**Chenyang Ma:** Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Conceptualization. **Qingyi Wang:** Investigation, Visualization, Writing – review & editing. **Di Tian:** Visualization, Validation, Data curation. **Wenxia Yuan:** Methodology, Writing – review & editing. **Xuan Tang:** Methodology, Formal analysis. **Xiujuan Deng:** Formal analysis, Writing – review & editing. **Yapeng Liu:** Investigation, Software. **Chang Gao:** Resources, Formal analysis. **Guofu Fan:** Software, Resources. **Xue Xiao:** Software, Formal analysis. **Baijuan Wang:** Funding acquisition, Validation, Visualization, Writing – review & editing. **Yali Li:** Writing – review & editing, Writing – original draft, Visualization, Conceptualization. **Hongjie Zhou:** Writing – review & editing, 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 the work reported in this paper.

## Data availability

The data that has been used is confidential.

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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.2024.101965.

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