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Volatile Profiling and Aroma Characterization of Commercial Mulberry Leaf Teas

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Abstract

Mulberry leaf tea (MLT) has gained increasing attention as a functional beverage; however, its aroma quality evaluation remains limited due to insufficient characterization of volatile profiles and aroma-active compounds. In this study, the volatile compositions of ten commercial MLT products were systematically investigated using headspace solid-phase microextraction coupled with gas chromatography–mass spectrometry (HS-SPME-GC-MS). A total of 123 volatile compounds were identified and classified into alcohols, aldehydes, ketones, esters, hydrocarbons, terpenoids, and other groups, with considerable variations observed among commercial products. Odor activity value (OAV) analysis revealed that 45 compounds exhibited OAV values ≥ 1, indicating their potential contribution to MLT aroma characteristics. Terpenoid and norisoprenoid compounds, including linalool, trans-β-ionone, trans-β-damascenone, and β-ionone, showed high odor activity and were associated with floral, fruity, and woody notes. Partial least squares discriminant analysis (PLS-DA) differentiated commercial MLT products, and five volatile compounds (limonene, *p*-cymene, eucalyptol, *trans*-β-ionone, and benzeneacetaldehyde) were identified as potential discriminative markers. This study provides comprehensive insights into the volatile profiles of commercial MLT products and identifies aroma-active compounds for aroma evaluation and product differentiation.

Keywords: mulberry leaf tea; volatile compounds; HS-SPME-GC-MS; odor activity value; PLS-DA; aroma-active markers

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1. Introduction

Mulberry leaf tea (MLT), derived from the leaves of *Morus alba* L., has been increasingly consumed as a functional herbal beverage due to its nutritional components and potential health-promoting properties [1]. Previous studies have demonstrated that mulberry leaves contain abundant bioactive compounds, including flavonoids, phenolic acids, alkaloids, and polysaccharides, which contribute to antioxidant, hypoglycemic, and metabolic regulatory activities [2–6]. With the growing consumer demand for natural and functional beverages, commercial MLT products have become increasingly diverse in terms of raw materials, manufacturing practices, and product characteristics [7].

Besides nutritional properties, aroma is an essential sensory attribute influencing consumer acceptance and overall quality perception of tea products [8]. The aroma char-

acteristics of tea are primarily determined by volatile compounds generated from plant metabolism [9,10], enzymatic reactions [11,12], thermal processing [13,14], and storage-related transformations [15]. In conventional tea products, aroma-active compounds such as terpenoids, alcohols, aldehydes, ketones, and esters have been widely investigated due to their significant contributions to floral, fruity, green, and roasted aroma characteristics [16–18]. However, compared with *Camellia sinensis* teas, the volatile composition and aroma-active components of mulberry leaf tea remain relatively unexplored.

Previous studies on MLT aroma have mainly focused on specific cultivars or experimentally prepared samples under controlled processing conditions [19,20]. Although these studies provide valuable information regarding volatile composition and aroma formation, they may not fully represent the chemical diversity of commercial products available to consumers. Commercial MLT products are influenced by multiple uncontrolled factors, including cultivar selection [21], geographical origin [22], harvesting stage, processing strategies [23], and storage conditions [24]. Therefore, systematic characterization of volatile profiles in commercial MLT products is important for understanding aroma diversity and identifying potential chemical indicators associated with product differentiation.

Gas chromatography–mass spectrometry (GC-MS), particularly combined with headspace solid-phase microextraction (HS-SPME), has been widely applied for comprehensive profiling of volatile compounds in tea [25,26] and plant-derived beverages [27,28]. However, the presence of numerous volatile compounds does not necessarily indicate their sensory relevance, because aroma perception depends largely on odor thresholds and relative concentrations. Therefore, odor activity value (OAV) analysis has been frequently employed to identify aroma-active compounds by evaluating the potential contribution of individual volatiles to overall aroma characteristics [29,30].

Furthermore, multivariate statistical approaches, including principal component analysis (PCA) and partial least squares discriminant analysis (PLS-DA), provide powerful tools for distinguishing complex food samples based on volatile fingerprints and identifying characteristic compounds responsible for sample differentiation [31,32]. Integrating sensory-related evaluation (OAV) with chemometric discrimination analysis may enable the identification of volatile compounds that are not only statistically different among products but also potentially relevant to aroma perception.

Therefore, the objective of this study was to comprehensively characterize the volatile profiles of commercial MLT products and identify aroma-active and discriminative volatile compounds. Ten commercial MLT samples were analyzed using HS-SPME-GC-MS, followed by OAV calculation, multivariate statistical analysis, and hierarchical clustering analysis. The findings provide insights into aroma diversity among commercial MLT products and propose potential chemical indicators for future aroma evaluation and quality characterization.

2. Materials and Methods

2.1. MLT Products and Sample Preparation

Ten commercial mulberry leaf tea (MLT) products were analyzed in this study. They were randomly selected from mainstream online shopping platforms and physical tea supermarkets nationwide based on market sales volume and consumer popularity, without preset artificial screening criteria, to represent mainstream commercially available finished mulberry leaf tea on the market. Detailed information on brand, manufacturer, origin, and product type is provided in Table S1 (Supplementary Material). All samples were uniformly purchased in October 2025 within the same production season via standardized official retail channels to guarantee a consistent procurement environment. Since the samples were purchased commercially, detailed information regarding cultivar, geographical origin, and

processing parameters was unavailable. Therefore, this study focused on characterizing aroma diversity among commercial products rather than establishing direct relationships between specific processing conditions and volatile formation. Prior to analysis, dried tea leaves were ground into fine powder, passed through a 40-mesh sieve, and stored in airtight brown glass bottles at −20 °C for no more than 14 days before further analysis [33].

2.2. HS-SPME of Volatiles from MLT

Volatile compounds were extracted using headspace solid-phase microextraction (HS-SPME) according to a previously reported method with minor modifications [34]. For each sample, 1.00 g of powdered MLT was accurately weighed into a 20 mL headspace vial. Then, 10 mL of boiling distilled water and 10 µL of ethyl decanoate solution (10 mg/L in methanol, internal standard) were added. The 1:10 solid-to-liquid ratio was designed to simulate the conventional daily brewing mode of mulberry leaf tea. Hot water hydration facilitates the release of bound volatile compounds within the tea matrix and reflects the actual sensory aroma perceived by consumers; a similar hydration strategy has been widely validated in volatile profiling studies of leaf tea products [35]. A procedural blank containing distilled water and an equal volume of internal standard solution was analyzed alongside all samples to eliminate background interference originating from the SPME fiber, glass vials and solvents, ruling out exogenous contamination of aromatic hydrocarbons such as toluene and xylene.

The vial was immediately sealed with a PTFE/silicone septum and equilibrated in a 60 °C water bath for 10 min. A 50/30 µm divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) SPME fiber (Supelco, Bellefonte, PA, USA) was exposed to the headspace at 60 °C for 30 min. Three fully independent extraction replicates were conducted for every MLT sample. Each replicate consisted of separate weighing, hydration, equilibration and headspace extraction procedures, rather than repeated injections from a single vial. All sample vials were randomized in the GC-MS analytical sequence to avoid systematic bias caused by differences in volatile concentration and gradual aging of the SPME fiber.

After extraction, the fiber was immediately inserted into the GC-MS injector for thermal desorption at 250 °C for 5 min. The fiber was conditioned prior to use according to the manufacturer's instructions.

2.3. GC-MS Analysis

Volatile compounds were analyzed on an Agilent 7890A gas chromatograph coupled to a 7000D mass spectrometer (Agilent Technologies, Santa Clara, CA, USA) [17]. Separation was performed on a DB-5 fused silica capillary column (30 m × 0.25 mm i.d., 0.25 µm film thickness; J&W Scientific, Folsom, CA, USA). Helium (≥99.999% purity) was used as the carrier gas at a constant flow rate of 1.0 mL/min. Injections were performed in splitless mode with the injector temperature maintained at 250 °C. The oven temperature program was as follows: 40 °C (held for 3 min), ramped to 120 °C at 4 °C/min, then to 240 °C at 6 °C/min (held for 12 min). Electron ionization (EI) was operated at 70 eV. The ion source and transfer line temperatures were 230 °C and 280 °C, respectively. Mass spectra were acquired in scan mode over the range of m/z 35–350.

Volatile compounds were tentatively identified by matching mass spectra with the NIST 17 library (match factor > 800) and by comparing calculated retention indices (RI) with literature values using *n*-alkanes (C8–C24) as standards. Identification was considered reliable when the calculated RI was within ±20 units of published values. All identified volatile compounds are listed in Table S2 (Supplementary Materials). Semi-quantification was performed using the single internal standard method (ethyl decanoate). It should

be noted that detector response factors vary across chemical classes, and concentrations are expressed as relative semi-quantitative concentrations (μg/kg on a dry weight basis) (Table S2, Supplementary Materials).

2.4. OAV Calculation

Odor activity values (OAVs) were calculated to evaluate the relative sensory contribution of individual volatile compounds using the formula: OAV = C/OT [36], where C represents the concentration of the volatile compound (μg/kg) determined by the internal standard method, and OT is its odor threshold concentration in water (μg/kg). Compounds with OAV ≥ 1 were identified as key aroma-active contributors to the overall aroma profile. Threshold values in water were compiled from published literature [37–40] and are summarized in Table S2 (Supplementary Materials). It should be noted that the odour thresholds reported in water may differ from those in tea infusion because of matrix effects arising from nonvolatile constituents and the complex aroma background of tea. Therefore, the OAVs calculated in this study were considered approximate indicators for comparative evaluation rather than direct measures of the actual sensory contribution of individual volatile compounds in tea infusion.

2.5. Statistical Analysis

For each commercial MLT product, three independent sample preparations were performed. Three separate portions of each powdered sample were independently weighed and subjected to the complete sample preparation and HS-SPME-GC-MS procedure. The commercial product was treated as the experimental unit, and the three independent preparations were used to characterize within-product variation. Data are expressed as mean ± standard deviation (SD). For one-way analysis of variance (ANOVA) followed by Duncan's multiple range test conducted using SPSS 26.0 (IBM Corp., Armonk, NY, USA), the average value calculated from three technical replicates of each product was adopted as a single statistical unit to assess significant differences among products ($p < 0.05$). Technical replicates were only used to evaluate analytical repeatability and reduce random detection error, and were not regarded as independent statistical individuals.

Principal component analysis (PCA) and partial least squares discriminant analysis (PLS-DA) were performed using SIMCA 14.1 software (Umetrics, Umea, Sweden). Prior to PCA and PLS-DA, quantitative data were normalized and subjected to Pareto scaling to minimize the dominance of high-abundance compounds.

Model robustness and anti-overfitting capabilities were evaluated using a 200-iteration permutation test, where the response variable (Y) was randomly permuted to construct distributions of R^2 and Q^2 under the null hypothesis. Data organization and visualization were carried out using Microsoft Excel 365 (Microsoft, Redmond, WA, USA) and Origin 2021 (OriginLab, Northampton, MA, USA).

3. Results and Discussion

3.1. Overview and Chemical Profiling of Volatiles in Commercial MLT

Volatile compounds from ten commercial MLT products were extracted using HS-SPME and analyzed by GC-MS, leading to the identification of 123 volatile compounds across all samples (Table S3, Figure 1a). These compounds were categorized into eight chemical classes: alkenes (27 compounds, 21.9%), alcohols (18, 14.6%), ketones (18, 14.6%), aromatics (16, 13.0%), esters (16, 13.0%), aldehydes (15, 12.2%), alkanes (8, 6.5%), and heterocyclic compounds (5, 4.1%). The 123 identified compounds represent a substantially broader chemical diversity than reported in previous single-process or single-cultivar MLT studies, such as those by Li et al. [41] and Yan et al. [13], which identified 63 and 50 volatile

compounds under specific drying or withering conditions, respectively. This expanded volatile space is primarily attributable to the diverse cultivars, processing technologies, and geographic origins represented across the commercial products analyzed here.

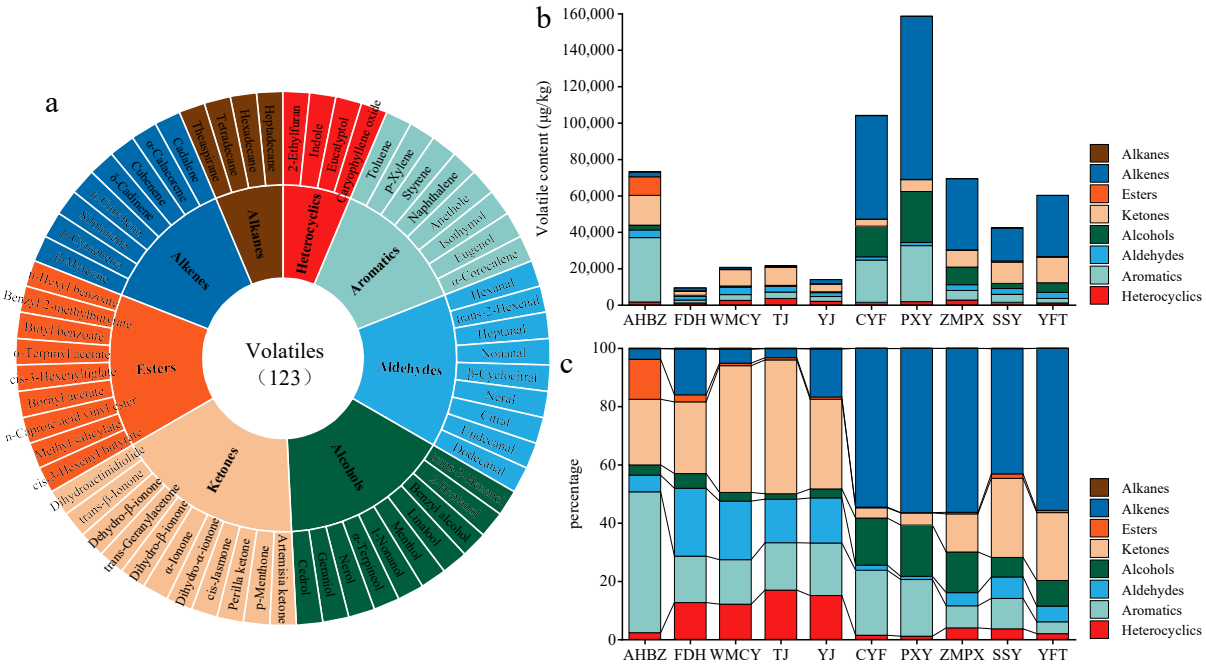


Figure 1. Volatile profiles of ten commercial mulberry leaf tea (MLT) products. (**a**) Chemical class distribution of the 123 identified volatile compounds across all samples (inner circle shows major classes); (**b**) total relative concentrations of volatile compounds (μg/kg, dry weight basis) by chemical class; (**c**) relative percentage contribution of each chemical class to the total volatiles in individual commercial products. Sample codes: AHBZ, FDH, WMCY, TJ, YJ, CYF, PXY, ZMPX, SSY, YFT (detailed information in Table S1).

The total relative concentrations of volatile compounds exhibited a dramatic ~20-fold variation across the ten commercial products, ranging from 10,921 μg/kg in FDH to 213,045 μg/kg in PXY (Figure 1b). PXY possessed the highest total volatile abundance, followed by ZMPX (89,579 μg/kg) and YFT (63,981 μg/kg). Pairwise post hoc comparison revealed that AHBZ showed markedly lower total volatile levels than PXY, ZMPX, YFT and four other tested mulberry leaf tea samples ($p < 0.05$); no statistically significant difference was detected between AHBZ and FDH and WMCY. Collectively, FDH and WMCY exhibited the lowest overall volatile yields among all batches. As illustrated in Figure 1b, category distributions varied markedly among individual products. Alkenes predominated in most samples, particularly in PXY, ZMPX, and YFT, accounting for over 40% of their total relative concentrations of volatile compounds (Figure 1c). This enrichment was mainly driven by high concentrations of monoterpenes such as limonene and *p*-cymene. Notably, ANOVA indicated that the alkene concentration in PXY (118,467 μg/kg) was significantly higher than in all other products ($p < 0.001$). Ketones represented the second most abundant class in AHBZ, FDH, WMCY, TJ, and YJ, imparting floral and fruity characteristics. Alcohols (e.g., eucalyptol and benzyl alcohol) were particularly concentrated in PXY and ZMPX, while aldehydes were relatively uniformly distributed across all products.

Each commercial product exhibited a distinct chemical fingerprint. PXY was distinguished by elevated monoterpenes (limonene, *p*-cymene) and eucalyptol, contributing a fresh, citrus-like, and herbal aroma profile. ZMPX showed a balanced blend of alkenes and ketones, yielding fresh, grassy, and floral attributes. YFT was dominated by aromatic ketones and alkenes, favoring sweet and floral notes. In contrast, products like FDH and

WMCY presented lower overall volatile concentrations and fewer dominant compounds, implying a milder aroma intensity. Such substantial inter-product heterogeneity underscores the profound impact of unstandardized commercial processing—including fixation temperature, drying methods, and post-harvest fermentation—on volatile generation and retention [41].

The formation of these volatile compounds may involve multiple biochemical and processing-related pathways. Lipid-derived aldehydes and alcohols can originate from the oxidation and subsequent cleavage of fatty acids, whereas monoterpenes such as limonene, β-myrcene, and linalool are associated with terpenoid biosynthesis. C13-norisoprenoids, including β-ionone and β-damascenone, can arise from oxidative or enzymatic cleavage of carotenoids. During processing, tissue disruption, heating, and drying may promote precursor release, oxidation, and thermal transformation, thereby contributing to the final volatile profile of MLT.

3.2. Chemometric Discrimination and Differential Aroma Marker Identification

To identify volatile compounds responsible for differentiation among commercial MLT products, partial least squares discriminant analysis (PLS-DA) was conducted based on the volatile profiles. The PLS-DA score plot demonstrated clear separation among different commercial samples (Figure 2), indicating that volatile fingerprints contained sufficient information for discriminating MLT products.

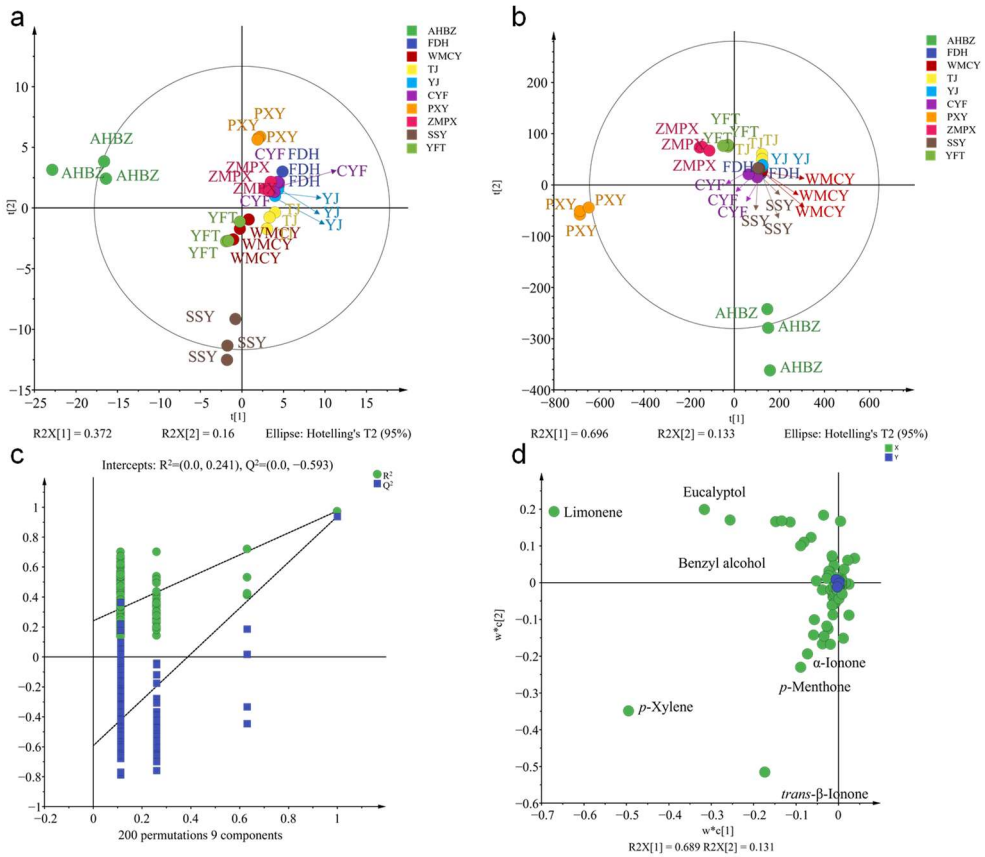


Figure 2. Multivariate statistical analysis of volatile profiles in ten commercial mulberry leaf tea (MLT) products. (**a**) PCA score plot (PC1 = 37.2%, PC2 = 16.0%); (**b**) PLS-DA score plot (R^2X = 0.696, R^2Y = 0.987, Q^2 = 0.956); (**c**) 200-permutation test validation (original R^2 = 0.241, Q^2 = −0.593 for permuted models); (**d**) loading plot of PLS-DA showing key differential volatile markers. Sample codes correspond to those described in Table S1.

Unsupervised PCA was initially conducted to explore natural sample grouping and overall variance distribution (Figure 2a). The first two principal components explained 53.2% of the total variance (PC1 = 37.2%, PC2 = 16.0%). The moderate cumulative explained variance reflects the inherent high dimensionality of untargeted GC-MS metabolomics data, where the number of chemical variables significantly exceeds the sample size [42]. Nevertheless, the PCA score plot displayed distinct clustering among triplicates of the same product, alongside clear separation among different commercial brands. Specifically, AHBZ separated along the negative PC1 axis due to its unique abundance of perilla ketone and artemisia ketone, whereas PXY positioned at the far positive extreme of PC1, driven by its exceptional accumulation of monoterpenes and cyclic ether alcohols.

To maximize class separation and identify specific discriminatory markers, supervised PLS-DA was performed (Figure 2b). The model was validated via 7-fold cross-validation during construction, followed by a 200-times permutation test to rigorously exclude overfitting risk. The PLS-DA model achieved clear discrimination among all ten commercial products with high explanatory and predictive fit parameters ($R^2X = 0.696$, $R^2Y = 0.987$, and $Q^2 = 0.956$). PXY separated distinctly along the negative axis of the first predictive component, while AHBZ formed an isolated cluster in the lower-right quadrant, and the remaining products clustered symmetrically in the central domain with distinct boundaries. A 200-iteration permutation test yielded a Q^2 intercept of -0.593 (< 0) with all permuted R^2 and Q^2 values lower than the original model metrics (Figure 2c), confirming the absence of overfitting. PCA and PLS-DA were performed using the individual replicate measurements, with three replicate observations obtained for each of the ten commercial MLT products (30 observations in total). The replicates originated from separate sample preparations of the same commercial product and should therefore not be regarded as independent commercial samples. Accordingly, the clustering observed in the multivariate analyses was interpreted cautiously as an exploratory representation of similarities and differences among the commercial products.

Examination of the PLS-DA loading plot (Figure 2d) and Variable Importance in Projection (VIP > 1.0) scores revealed the primary chemical drivers responsible for product discrimination. Among these compounds, *trans*-β-ionone was particularly noteworthy because it showed both high aroma activity and strong discriminatory capacity. This suggests that *trans*-β-ionone may represent an important aroma-related marker linking sensory relevance with chemical differentiation [43]. Similarly, limonene and eucalyptol, although primarily identified as discriminative compounds, may contribute to differences in citrus-like and herbal aroma characteristics due to their relatively low odor thresholds [44].

3.3. Key Aroma-Active Compounds and Sensory Relevance via OAV Analysis

Because absolute chemical concentration does not directly dictate aroma perception, odor activity values (OAVs) were calculated to evaluate the sensory contributions of individual volatiles. A total of 45 compounds exhibited OAV ≥ 1 across the ten products (Table 1), with 7 compounds exceeding an OAV of 10, highlighting their dominant role in shaping the overall sensory character of MLT.

Table 1. Key aroma-active compounds (OAV ≥ 1) in ten commercial mulberry leaf tea (MLT) products.

No.	Odorants	OT(μg/kg)	Odor Quality	AHBZ	FDH	WMCY	TJ	YJ	CYF	PXY	ZMPX	SSY	YFT
1	*trans*-β-Ionone	0.007	woody, floral, berry	422,031.87	132,158.58	494,831.01	1,144,192.83	691,301.42	204,544.62	514,533.13	832,533.78	574,753.85	1,414,708.83
2	*trans*-β-Damascenone	0.002	fruity, rose	2505.76	818.90	393.93	1869.74	3826.93	4010.82	1570.20	1530.87	4411.20	2305.07
3	Eugenol	0.71	spicy, woody	1131.60	15.96	31.32	4.79	7.44	2.26	3.69	12.16	9.39	7.16
4	Linalool	0.22	floral, woody, fruity	774.09	235.88	292.69	326.55	263.50	117.37	441.82	315.83	799.09	1539.96
5	Nonanal	1.1	floral, fatty	215.26	89.31	440.24	149.60	120.50	88.11	100.20	116.70	94.08	106.49
6	Eucalyptol	1.1	herbal, medicinal	158.11	127.31	62.72	40.10	12.55	511.35	19,048.25	7404.85	202.52	2490.95
7	β-Myrcene	1.2	Spicy, fatty	134.51	114.68	84.99	98.27	54.41	101.61	9718.76	1872.55	221.72	301.50
8	α-Ionone	3.78	woody, floral, fruity	133.41	16.50	81.79	327.73	147.35	42.26	135.08	236.50	203.09	378.25
9	2,6-*(E,Z)*-Nonadienal	0.8	green, fatty, violet	106.31	22.31	93.78	74.90	52.50	16.55	63.02	259.85	39.71	769.02
10	2-Pentylfuran	5.8	fruity, green	92.00	173.55	116.10	483.83	181.80	101.97	183.48	455.93	203.83	161.91
11	*p*-Cymene	5.01	citrus, woody	91.26	155.76	13.36	33.45	26.43	831.96	11,356.02	3137.37	478.36	3580.30
12	Dihydro-β-ionone	1	woody, orris	77.44	1.68	7.98	16.86	9.28	3.22	6.61	68.67	6.39	29.05
13	β-Cyclocitral	3	herba, fruity	74.49	184.99	115.43	362.85	282.44	118.61	95.98	310.44	202.80	368.81
14	Heptanal	2.8	fatty, green, herbal	58.11	102.37	88.61	146.51	112.95	98.41	18.37	85.68	48.85	45.05
15	Naphthalene	6	N.F.	37.76	8.38	22.54	11.67	8.01	11.50	24.83	12.46	399.64	47.81
16	Benzeneacetaldehyde	6.3	green, sweet, floral	24.51	31.78	8.57	30.34	11.32	20.02	7308.82	27.70	12.85	14.75
17	2-Methylnaphthalene	3	sweet, floral, woody	20.32	2.00	7.67	6.07	3.36	3.14	2.97	2.59	21.46	10.13
18	Dodecanal	0.13	soapy, citrus, floral	16.88	6.80	36.56	14.60	11.60	7.04	12.79	21.69	25.20	12.76
19	Toluene	527	sweet	16.82	2.66	1.73	3.04	3.55	4.55	2.28	3.45	3.17	1.84
20	Theaspirane	0.5	herbal, woody, spicy	13.43	0.72	2.19	1.15	2.14	1.59	5.43	1.59	2.97	14.33
21	*trans*-Geranylacetone	60	green, fruity, woody	13.27	2.09	13.57	8.36	2.88	2.07	8.72	5.21	32.16	13.45
22	Decanal	3	sweet, citrus, floral	12.97	11.12	34.21	13.62	20.43	10.83	13.90	13.13	9.02	12.38
23	*p*-Xylene	1000	N.F.	9.41	0.19	0.32	1.99	0.50	0.45	0.30	1.76	0.57	0.24
24	2-Undecanone	5.5	fruity, fatty, floral	6.94	0.83	2.69	1.31	0.57	0.55	1.28	3.73	3.60	3.54
25	1-Methylnaphthalene	7.5	medicinal	5.62	0.56	2.20	1.96	1.02	0.95	1.05	0.85	5.37	2.54
26	Methyl salicylate	40	green, mint	5.20	6.93	1.29	1.04	1.55	0.60	0.74	1.27	0.85	1.04
27	*trans*-2-Hexenal	88.7	green, banana, fatty	4.19	0.36	7.67	0.74	1.39	0.08	0.24	0.60	0.71	0.63
28	Hexanal	65	green, fatty, fruity	3.80	14.91	8.09	15.15	7.88	10.34	3.06	18.97	8.24	5.02
29	Indole	11	floral	3.50	1.26	0.32	0.46	0.71	0.39	1.63	0.69	0.77	0.48
30	Dibenzofuran	3.3	N.F.	3.22	0.99	2.48	1.03	0.96	0.75	0.65	0.87	2.29	1.11
31	6-Methyl-5-heptene-2-one	68	citrus, green, apple	2.45	2.89	3.69	4.33	2.88	2.83	4.11	4.31	9.75	3.18
32	1-Nonanol	45.5	fatty, floral	1.78	0.26	1.14	0.27	0.29	0.22	0.11	0.22	0.32	1.61
33	Citral	40	lemon, sweet	1.73	0.17	0.70	0.16	0.09	0.10	0.23	0.21	0.56	0.16
34	Limonene	200	citrus, herbal	1.58	4.76	2.14	1.40	0.16	22.34	188.47	167.34	7.48	85.99
35	Caryophyllene	64	sweet, woody, spicy	1.28	0.01	0.06	0.03	0.02	0.05	0.17	0.02	0.03	0.15
36	2-Heptanol	65.235	floral, fruity, green	0.69	0.57	0.57	0.70	0.78	0.53	0.08	0.48	0.25	0.35
37	Carvone	27	minty, licorice	0.64	0.03	0.06	0.04	0.02	0.05	4.18	0.24	0.16	0.87
38	Neral	53	sweet, citral, lemon	0.62	0.03	0.09	0.04	0.03	0.02	0.13	0.03	0.10	0.11
39	*(E,E)*-3,5-Octadien-2-one	100	fruity, grassy	0.62	3.47	0.81	8.62	2.68	0.93	6.09	14.07	5.81	3.46
40	*p*-Cymenene	85	spicy, clove-like	0.42	0.29	0.22	0.43	0.41	0.77	6.61	1.79	1.28	1.59
41	*trans*-3-Hexenol	110	green, floral, oily,	0.35	1.53	0.76	1.53	0.70	0.96	0.33	1.84	0.79	0.53
42	β-Pinene	140	woody, green	0.24	0.95	0.69	0.81	0.41	0.62	69.63	13.93	1.84	1.62
43	Benzyl alcohol	2546.21	floral	0.07	0.05	0.02	0.03	0.02	0.15	6.64	2.12	0.06	0.69
44	Terpinolene	200	woody, sweet, citrus	0.06	0.04	0.02	0.02	0.03	0.06	8.19	1.00	0.18	0.59
45	Geraniol	1.1	sweet, floral, fruity	0.00	3.08	4.34	7.88	7.17	3.82	4.00	8.02	4.94	12.56

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Among these, the C13-norisoprenoids *trans*-β-ionone and *trans*-β-damascenone displayed exceptionally low odor detection thresholds in water (0.007 μg/kg and 0.002 μg/kg, respectively), yielding extraordinary OAV values across all commercial products (maximum OAV of 1.41×10^6 for *trans*-β-ionone in YFT; 4.41×10^3 for *trans*-β-damascenone in YJ). These two compounds emerged as universal core aroma contributors in MLT, imparting characteristic woody, floral, rose-like, and berry notes. These C13-norisoprenoids are typically derived from the oxidative or enzymatic cleavage of carotenoids (e.g., β-carotene and lutein) during heat-intensive processing steps such as fixation and drying [19,20]. Their consistent presence across all products confirms their fundamental contribution to the baseline aroma of MLT, aligning with findings in true teas (*Camellia sinensis*) and herbal infusions [45,46].

The occurrence of extremely high numerical OAVs (e.g., $>10^5$) requires careful scientific interpretation.

In addition to core norisoprenoids, several other key aroma-active compounds contributed to product-specific flavor nuances. Eugenol (OAV: 2–1132), linalool (OAV: 117–1540), α-ionone (OAV: 17–378), and nonanal (OAV: 88–440) provided essential spicy, floral, woody, green, and citrus attributes. Eucalyptol exhibited remarkably high OAVs in PXY (19,048) and ZMPX (7405), delivering a distinct fresh and herbal note. Geraniol (OAV $\geq$ 1 in 9 products) and 2-methylnaphthalene (OAV $\geq$ 1 in 7 products) acted as important secondary aroma modifiers. Notably, compounds present at high concentrations with moderate thresholds (e.g., limonene at 37,694 μg/kg in PXY, OAV = 188; *p*-cymene at 56,894 μg/kg in PXY, OAV = 11,356) contributed significantly to aroma profile differentiation, whereas abundant non-aroma-active compounds with high thresholds (e.g., toluene at 8865 μg/kg in AHBZ, OAV = 17) played minimal sensory roles. This observation underscores the necessity of combining quantitative profiling with OAV assessment for meaningful flavor evaluation.

The high estimated odor activity of several compounds may be related to their specific precursor pathways. In particular, β-ionone and β-damascenone are carotenoid-derived C13-norisoprenoids, whereas linalool and other oxygenated monoterpenes are associated with terpenoid metabolism and subsequent processing-related transformations. Lipid oxidation may also contribute to the formation of highly odor-active aldehydes such as hexanal and nonanal. These pathways may partly explain the important role of these compounds in the aroma profiles of MLT.

3.4. Distribution Patterns of Aroma-Active Compounds Revealed by Heatmap Analysis

To further investigate the variation of aroma-active compounds among commercial MLT products, hierarchical clustering heatmap analysis was performed based on the relative abundance of 45 compounds with OAV values $\geq$ 1 (Figure 3). Euclidean distance was adopted as the distance metric, and Ward's minimum variance method was used as the linkage criterion. Hierarchical clustering was conducted simultaneously on both samples (row clusters on the top) and aroma-active volatile compounds (column clusters on the left); prior to clustering, Z-score normalization was applied to the dataset to eliminate the interference caused by large concentration disparities among volatile components.

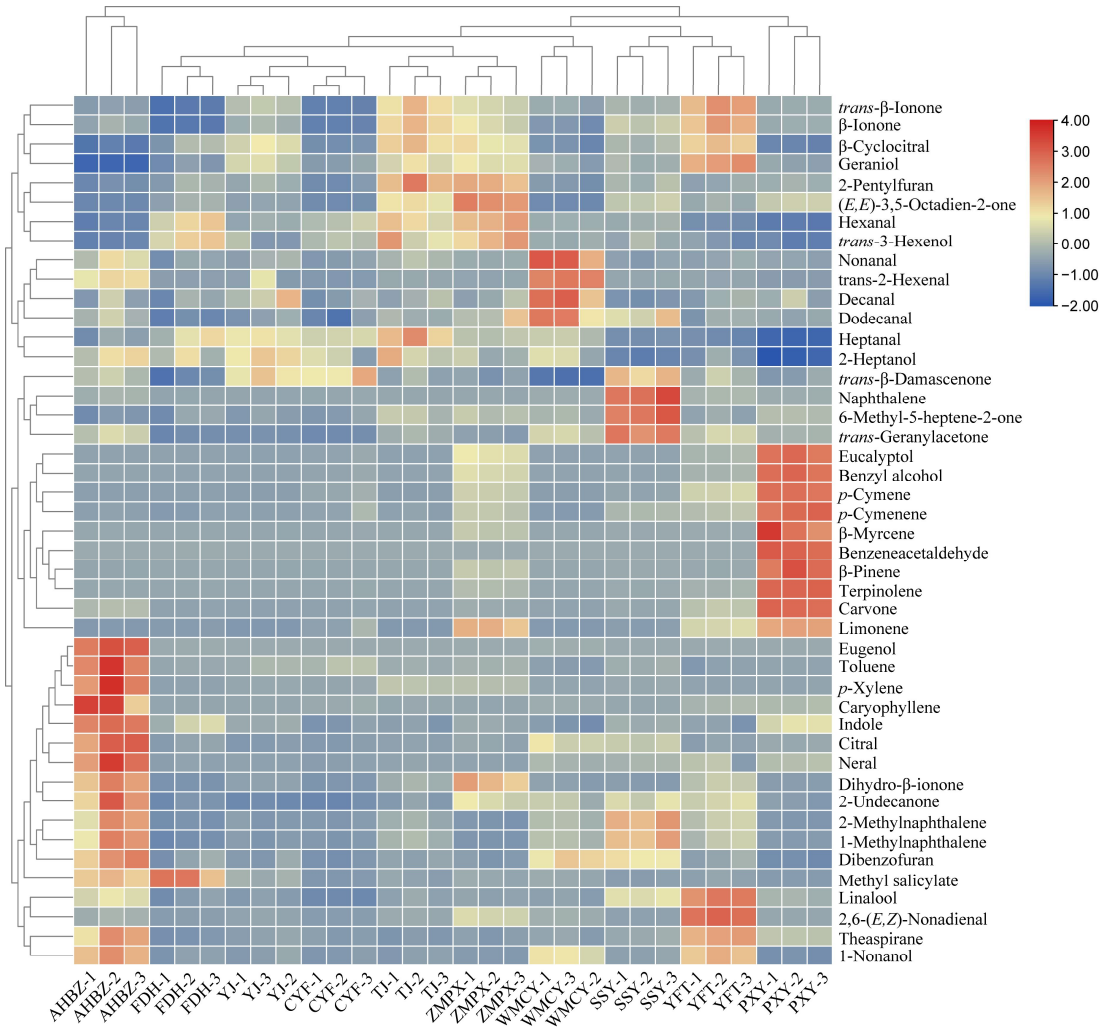


Figure 3. Hierarchical clustering heatmap of aroma-active compounds in ten commercial mulberry leaf tea (MLT) products. The heatmap was constructed using Z-score normalized (autoscaled) relative abundance data for 45 volatile compounds with OAVs ≥ 1. Red and blue denote higher and lower normalized abundance following Z-score transformation before plotting. Sample codes refer to Table S1.

The heatmap revealed distinct distribution patterns of aroma-active compounds among the ten samples, confirming the considerable heterogeneity observed from the overall volatile profiling analysis. Samples were clustered according to their aroma-active compound profiles, suggesting that differences in aroma perception among commercial products were associated with coordinated variations of multiple aroma-related compounds.

Several samples exhibited relatively higher levels of monoterpenoid compounds, including eucalyptol, limonene, *p*-cymene, and α-pinene. These compounds are commonly associated with herbal, citrus-like, and fresh aroma characteristics. In contrast, some products showed increased abundance of aldehyde compounds, including nonanal, decanal, and *trans*-2-hexenal, which may contribute to green and fatty aroma impressions.

Norisoprenoid compounds, including *trans*-β-ionone, displayed variable distribution patterns among commercial products. Considering their high odor activity, differences in their abundance may contribute to variations in floral and woody aroma characteristics among samples.

The heatmap analysis further demonstrated that aroma differentiation among commercial MLT products was associated with complex changes in multiple aroma-active compounds rather than differences in only a few individual volatiles. This finding high-

lights the importance of considering aroma-active compound patterns when evaluating commercial tea products.

4. Limitations of the Study

Although this study provides comprehensive insights into the volatile characteristics of commercial mulberry leaf teas, several limitations should be acknowledged. First, the commercial samples analyzed in this study were collected from different suppliers, and detailed information regarding cultivar, geographical origin, harvesting conditions, processing parameters, and storage history was not fully available. Therefore, the observed volatile differences cannot be directly attributed to a specific production factor.

Second, aroma contribution was evaluated based on OAV calculations derived from literature-reported odor thresholds. Although OAV analysis is widely applied for screening aroma-active compounds, the actual sensory impact of individual compounds may be affected by matrix effects and interactions among volatile components. Further validation using sensory evaluation and gas chromatography–olfactometry (GC-O) analysis is required.

Third, the number of commercial samples analyzed was limited, and future studies involving larger sample populations and controlled processing experiments are necessary to validate the identified aroma markers and establish more robust aroma evaluation models for commercial mulberry leaf teas.

5. Conclusions

This study systematically characterized the volatile profiles and key aroma-active compounds across ten commercial mulberry leaf tea (MLT) products using HS-SPME-GC-MS, OAV analysis, and multivariate statistics. A total of 123volatiles were identified, revealing substantial market heterogeneity with total volatile concentrations varying by nearly 20-fold (10,921–213,045 μg/kg). While *trans*-β-ionone and *trans*-β-damascenone served as universal core aroma contributors, PLS-DA modeling successfully identified limonene, *p*-cymene, eucalyptol, *trans*-β-ionone, and benzeneacetaldehyde as key differential markers for product discrimination.

These findings provide essential chemical fingerprints for quality assessment, authentication, and flavor standardization of commercial MLT. Future research should focus on GC-O sensory validation, aroma reconstitution assays, and controlled processing trials to further advance quality control protocols for the herbal tea industry.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/beverages12090100/s1, Table S1. Information of Mulberry Leaf Tea used in this study; Table S2. Identification of volatile compounds in MLT and their odor thresholds; Table S3. Relative contents of volatiles in MLT of different varieties (μg/kg, on dry weight).

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