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Investigation of the influence of different origins on the volatile components of chicory roots based on cluster analysis and OPLS-DA

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ABSTRACT: To investigate the influence of different origins on the volatile components of chicory roots, headspace solid phase microextraction-gas chromatography-mass spectrometry (HS-SPME-GC-MS) was employed to extract and analyze volatile constituents. The content of each component was determined and compared using an internal standard semi-quantitative method, and cluster analysis and orthogonal partial least squares discriminant analysis (OPLS-DA) were then applied to examine the differences in volatile components among chicory root samples from different origins. Results indicated that 95 volatile compounds were identified across chicory root samples from different origins, primarily comprising 20 aldehydes, 1 alcohol, 2 acids, 9 esters, 9 ketones, 30 hydrocarbons, 7 phenols, and 17 heterocyclic compounds, with aldehydes and hydrocarbons being the predominant classes. Significant differences were observed in the volatile compounds among chicory root samples from different origins. Among them, the volatile component content was highest in Xinjiang chicory root samples at 3120.98 μg/kg, while the lowest was found in Zhejiang chicory root samples at 1228.50 μg/kg. Based on OPLS-DA and cluster analysis, chicory roots from different origins could be effectively distinguished. Furthermore, according to the variable importance in projection (VIP values), 39 compounds, including 9-methylicosane, furfural, methyl palmitate, and 5-methylfurfural, were identified as key substances for distinguishing the geographical origins of chicory roots. This study reveals the differences in the composition and content distribution of volatile components in chicory roots from different origins, providing a scientific basis for further development of chicory root resources and origin tracing.

KEYWORDS: chicory root, geographical origin, HS-SPME-GC-MS, OPLS-DA, volatile components

INTRODUCTION

Chicory (*Cichorium intybus* L.), also referred to as coffee grass, is a perennial herb belonging to the *Cichorium* genus within the Asteraceae family, widely distributed across the globe [1, 2]. As a plant with both medicinal and edible uses, chicory's root is abundant in inulin, amino acids, vitamins, and other nutrients [3, 4], and it exhibits antioxidant, anti-inflammatory, lipid-lowering, and hypoglycemic properties [5, 6]. Furthermore, roasted chicory root develops a coffee-like aroma, making it suitable as a coffee substitute or additive, and it finds widespread application in the food additive industry. The aboveground parts of chicory are primarily consumed as vegetables or used as feed [7]. Current research on chicory, both domestically and internationally, predominantly focuses on the extraction of pharmacologically active ingredients and the assessment of their effects. For instance, Elgharabawy and colleagues [8] discovered that chicory extract may, to some extent, restore the antioxidant defense system of rat hearts by scavenging free radicals, thereby mitigating heart damage caused by PbO-NPs. Choudhary et al [9] reported that the methanol extract of chicory leaves possesses high antioxidant and antidiabetic activities. Additionally, a limited number of studies have reported on the impact of baking temperature [10] and extraction methods [11] on the volatile components of chicory root extract.

Headspace solid-phase microextraction coupled with gas chromatography-mass spectrometry (HS-SPME-GC-MS) enables the separation, identification, and qualitative and quantitative analysis of complex mixtures of volatile compounds [12]. As a primary method for the extraction and analysis of volatile components, it is widely used in food, plant, and environmental studies due to its short analysis time and low solvent consumption. In addition, electronic nose (E-nose) technology, often combined with GC-MS, has been successfully applied to characterize aroma profiles and volatile compounds in complex food matrices [13]. For plant matrices such as chicory root, which are rich in heat-sensitive and low-abundance aromatic compounds (e.g. terpenes, aldehydes, ketones, and furan derivatives), HS-SPME-GC-MS offers significant advantages over traditional steam distillation and simultaneous distillation-extraction methods. It enables the enrichment of volatile components under mild conditions, minimizing thermal degradation and the formation of artefacts, while requiring only small sample volumes and no complex purification procedures. Combined with the high separation capability

of gas chromatography and the accurate identification provided by mass spectrometry, HS-SPME-GC-MS provides reliable technical support for the comparative analysis of volatile compounds in samples from different geographical origins [14].

However, due to the influence of growth environments such as climatic factors and ecological conditions, the content and distribution of natural product extracts vary significantly across different origins. For example, Raffo et al [15] employed HS-SPME-GC-MS technology to investigate volatile organic compounds in wild fennel leaves and flowers. They found that fennel from Italy's east coast primarily contained limonene, whereas that from the west coast predominantly featured γ-terpinene, indicating significant differences in volatile organic compound composition between fennel from different geographical origins. Zhou et al [16] employed HS-GC-IMS to examine volatile compounds in goji berries from various cultivars and regions, observing notable differences in both the variety and concentration of these compounds. However, research on how geographical origin affects the volatile components of chicory remains limited. Therefore, this study employs chicory roots from different origins as raw materials and combines HS-SPME-GC-MS with cluster analysis and OPLS-DA to investigate variations in the composition and content of volatile compounds in chicory across origins. This work aims to provide a scientific foundation for developing region-specific flavor ingredients and establishing reliable methods for the geographical traceability of chicory.

MATERIALS AND METHODS

Experimental materials

Chicory roots from Guangxi, Yunnan, Zhejiang, Jilin, and Xinjiang were all harvested in October 2025. Specifically, the Xinjiang chicory roots were collected from the mountainous area of Urumqi Xinjiang Uygur Autonomous Region. The species was identified by the authors as *Cichorium intybus* L. based on its morphological characteristics according to *Flora Reipublicae Popularis Sinicae*, Vol. 80(1) [17]. Purified water was from Wahaha Group Co., Ltd., China and anhydrous ethanol (chromatography grade) was from Tianjin Damao Chemical Reagent, China.

Laboratory instruments

7890B/5977A Gas Chromatography-Mass Spectrometry System, Agilent Technologies, USA; Manual SPME Sampler, 50/30 μm DVB/CAR/PDMS Composite Extraction Head, Supelco, USA; Electronic Balance, Mettler-Toledo, USA; BGZ-240 Electric Hot Air Drying Oven, Shanghai Boxun Medical & Biological Instrument Co., Ltd., China; XL-20B Grinder, Guangzhou Xulong Machinery Equipment Co., Ltd., China.

Experimental methods

Pre-treatment of chicory root samples

Chicory root slices of good quality and free from pests or diseases were selected from various origins. After thoroughly cleaning to remove dust, the slices were baked in an oven at 150 °C for 1 h. They were then ground using a crusher, sieved through a 40-mesh screen, and the powder was stored in No. 5 resealable bags at low temperature.

Analysis of volatile components

HS-SPME Conditions: With reference to method [18] and slightly modified, 1.0 g of chicory powder was weighed and placed into a 65 ml headspace vial. Then, 2 μl of phenethyl acetate internal standard (0.1 μg/μl) was added. The vial was equilibrated at 80 °C for 30 min. A pre-conditioned SPME fiber (aged at 260 °C for 5 min) was inserted into the headspace vial and exposed for extraction at 80 °C for 30 min. Finally, the fiber was desorbed in the GC inlet for 4 min.

GC/MS Conditions: Chromatographic conditions: HP-5MS capillary column (60 m×0.25 mm×0.25 μm); Temperature program: Initial temperature: 50 °C (hold for 2 min), ramp at 3 °C/min to 260 °C, hold for 5 min; Inlet temperature: 260 °C; Carrier gas: High-purity helium; Flow rate: 1.0 ml/min; Splitless injection mode; Mass spectrometry conditions: Electron Impact (EI) ion source: 70 eV; Ion source temperature: 230 °C; Quadrupole temperature: 150 °C; Transfer line temperature: 260 °C; Full scan mode, mass range m/z 30–500.

Qualitative and quantitative methods: Qualitative analysis: The qualitative identification of volatile compounds was primarily carried out using MS mass spectrometry libraries and retention indices (RI). The MS's built-in data analysis software was used to open the GC-MS chromatogram; by comparing it with the NIST 17 standard library, only compounds with a match rate greater than 80% were retained. Furthermore, C_6–C_{20} n-alkane standards were analysed under the same chromatographic conditions to determine their retention times (retention time, RT) to calculate the retention index (RI) of the volatile compounds: $RI = 100 \times n + 100 \times (RT_x - RT_n)/(RT_{n+1} - RT_n)$, where n denotes the number of carbon atoms in a given n-alkane, x denotes the compound under analysis, and RT denotes the retention time of the substance, with RT_x lying between RT_n and RT_{n+1}.

Semi-quantitative analysis: A simple semi-quantitative analysis of the volatile compounds in the sample is performed using the internal standard method, with phenethyl acetate as the internal standard. The semi-quantitative calculation of the target compound is based on the amount of internal standard added and the peak area: $W_x = (C_S \times V_S)/(m \times A_s/A_x)$, where s denotes the internal standard compound phenethyl acetate, x

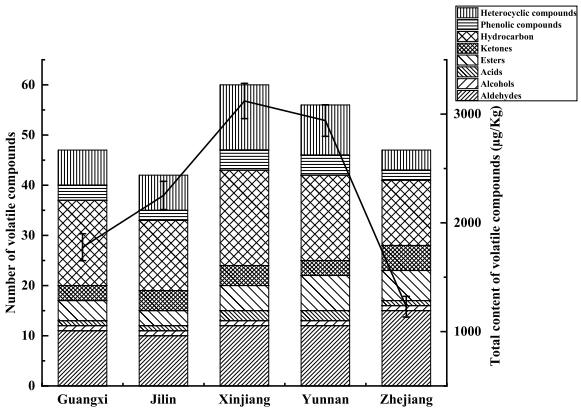

Fig. 1 Distribution map of quantity and total content of volatile compounds in chicory root samples from different habitats.

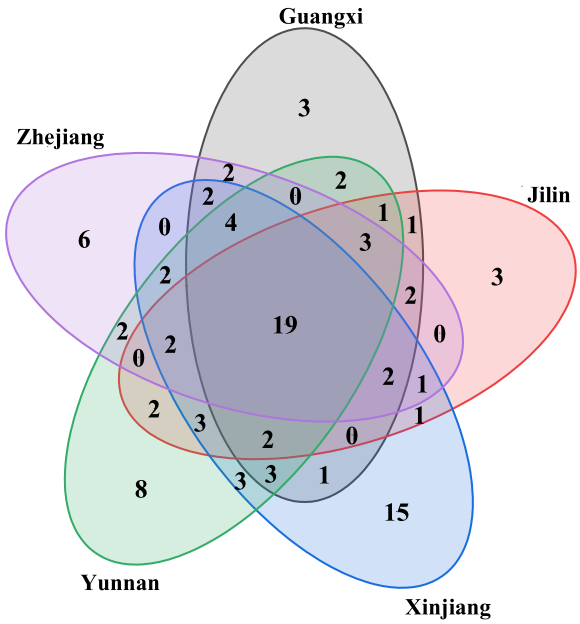

Fig. 2 Venn plot of volatile compounds in chicory root samples.

denotes the compound of interest, C and V represent the concentration (μg/μl) and volume (μl) of the internal standard, respectively, A represents the peak area, and m represents the mass (g) of the sample weighed. A response factor of 1.0 is assumed for all analytes. It should be emphasised that the reported values are semi-quantitative (relative concentrations) rather than absolute concentrations, as most identified volatile compounds do not have corresponding reference standards.

Data analysis

HS-SPME-GC-MS experiments were conducted in triplicate, with data expressed as mean ± standard deviation (Mean ± SD). IBM SPSS Statistics 27.0 software was used for one-way analysis of variance (ANOVA) and multiple comparisons ($p < 0.05$). Cluster heatmap analysis of quantitative results for volatile components in chicory roots from different origins was performed using Tbtools software. OPLS-DA analysis of quantitative data for volatile components in chicory roots from different origins was conducted using SIMCA 14.1 software. Bar charts and line graphs were generated using Origin 2021 software.

RESULTS AND DISCUSSION

Analysis of volatile components in chicory roots from different origins

The composition and content of volatile components in chicory roots from different origins are summarized in Table S1. A total of 95 volatile compounds were detected, including 20 aldehydes, 1 alcohol, 2 acids, 9 esters, 9 ketones, 30 hydrocarbons, 7 phenols, and 17 heterocyclic compounds. The distribution of differences in the number, total content, and content of various types of volatile compounds in chicory roots from different origins is shown in Fig. 1. Chicory roots from Xinjiang exhibited the highest diversity, with 60 volatile components detected. Samples from Yunnan, Guangxi, Zhejiang, and Jilin contained 56, 47, 47, and 42 components, respectively. In terms of total volatile content, the five origins ranked in the following descending order: Xinjiang > Yunnan > Jilin > Guangxi > Zhejiang. These results confirm that under identical pretreatment conditions, the composition and content of volatile compounds in chicory from different origins exhibit significant differences ($p < 0.05$). As shown in Fig. 2, 19 volatile compounds were shared among chicory roots from all five geographical origins, while each origin also possessed a number of unique volatile compounds. Xinjiang roots possessed the highest number of unique compounds (15), followed by Yunnan (8), Zhejiang (6), Guangxi (3), and Jilin (3). This indicates that the diversity of volatile compounds in chicory roots varies with origin, consistent with findings by Li et al [19].

The volatile compounds listed in Table S1, including aldehydes, ketones, acids, esters, hydrocarbons, and phenols, have been previously reported in studies on plant substrates such as chicory [11], red dates [20], green tea [21] and maize [22]. The identification of key aroma-active compounds, particularly pyrazines (e.g. 2,5-dimethylpyrazine, 2,6-dimethylpyrazine, 2-ethyl-6-methylpyrazine) and furans (e.g. furfural and 5-methylfurfural) is supported by previous studies on the volatile constituents of chicory roots [23]. For compounds lacking literature reports, identification was based on mass spectrometry library matching (NIST library, match > 80%) and retention indices.

As shown in Fig. 1, analysis of volatile compounds

in chicory roots from different origins reveals that aldehydes and hydrocarbons are the most abundant classes across all regions. Xinjiang chicory roots contained the highest number of these compounds at 31 types, accounting for 38.36% of the total content. Followed by Yunnan (29 types), Guangxi (28 types), Zhejiang (27 types), and Jilin (24 types), accounting for 46.85%, 47.33%, 50.19%, and 45.47% of the total content, respectively. This indicates that hydrocarbons and aldehydes dominate the volatile compounds in chicory roots from different origins. This finding is consistent with the results of Chen et al [18] on the volatile components of different parts of chicory. Although hydrocarbons (such as alkanes, alkenes, and terpenes) constitute the most abundant class of compounds detected, their contribution to the overall aroma of chicory root is likely limited due to their high odour thresholds. Consequently, the following discussion focuses primarily on aroma-active compounds, including pyrazines, furans, aldehydes, and ketones.

The content and variety of alcohols and acids in chicory root samples from different origins were relatively low. Among the alcohols, furfuryl alcohol was detected in all samples, with the highest concentration found in chicory roots from Jilin. This compound imparts a distinct caramel aroma and roasted sweetness to chicory [24]. Regarding acidic compounds, substances such as acetic acid and hexanoic acid exhibit sharp, unpleasant odors reminiscent of sweat which may negatively impact the overall flavor profile of chicory [25].

Nine ester compounds were detected, typically formed through esterification reactions between acids and alcohols [26]. Most of them were esters of long-chain fatty acids, including methyl palmitate and ethyl palmitate, with methyl palmitate exhibiting a faint oily aroma. Furthermore, dibutyl phthalate and diisobutyl phthalate were detected in some chicory root samples, indicating the possible presence of exogenous contamination. Phthalate esters are widely used as plasticisers [27] and are known environmental pollutants; they may have entered the samples during sample preparation or via laboratory consumables (such as headspace vial seals). However, as the levels of these compounds were low and they are not key aroma-active compounds, they do not affect the main conclusions of this study.

Ketone compounds play a crucial role in the aroma of roasted chicory root. A total of 9 ketones were identified across chicory root samples from the five different origins. Among them, 3,5-dihydroxy-6-methyl-2,3-dihydro-4H-pyran-4-one (DDMP) was the most abundant and was found to be a volatile aroma compound common to all five origins, contributing a toasty sweetness to the overall aroma profile [10]. DDMP is a key intermediate in the Maillard reaction and also serves as a precursor for numerous flavor compounds. It is generally formed through the Maillard reaction and degradation of Amadori compounds, with most studies suggesting its origin from hexose degradation [28]. Specifically, 3-phenylcyclopentane-1,2-dione was detected exclusively in Guangxi chicory root samples, contributing a roasted sweet aroma; 3-octen-2-one and 1-(6-methyl-2-pyrazinyl)-1-ethanone were identified only in Xinjiang chicory samples; while 5-Butyl-dihydro-4-methyl-2(3H)-furanone and β-damascene were detected exclusively in Zhejiang chicory root samples, exhibiting fruity aromas [29].

Additionally, the chicory root samples contained a relatively high variety of heterocyclic compounds, primarily pyrazines and furans. These compounds impart the characteristic roasted and nutty aromas to chicory root samples. Among them, 2,5-dimethylpyrazine and 2-pentylfuran, and 2-acetylpyrrole were detected in all samples, contributing caramel, almond, and sweet aromas. Furan and pyrrole compounds are primarily formed during the third stage of the Maillard reaction, contributing to the roasted flavor profile of baked chicory roots [24]. It is worth noting that, in addition to the Maillard reaction, pyrazine compounds can also be generated via microbial metabolic pathways; for example, Zhang et al [30] found that *Bacillus subtilis* can produce 2-ethyl-3,5-dimethylpyrazine and 2-ethyl-3,6-dimethylpyrazine from L-threonine and D-glucose. However, given that the chicory root samples in this study underwent high-temperature roasting and that chicory roots are rich in reducing sugars and free amino acids [31], and since the Maillard reaction is the primary pathway for pyrazine formation in heat-processed foods [32], and Baek et al [33] detected a large amount of pyrazine compounds in baked chicory roots, we therefore still attribute the formation of pyrazines primarily to the Maillard reaction.

There were significant differences in the volatile constituents of chicory roots from the five production areas, with the Xinjiang sample exhibiting the highest total volatile content and the greatest number of unique compounds. These differences can be linked mechanistically to the unique geographical and environmental conditions of each production area from the perspective of plant stress physiology [34]. The total content of volatile compounds in chicory roots from Xinjiang was 3120.98 μg/kg, significantly higher than that of the other four regions. This may be related to the environmental factors and climatic conditions of the region. Extensive research indicates that drought stress is a key factor stimulating the synthesis of plant volatile organic compounds. When responding to water deficiency, plants upregulate secondary metabolic pathways to synthesise large amounts of compounds such as pyrazines and phenolics, thereby enhancing antioxidant capacity and cell membrane stability [35]. Xinjiang has a typical temperate continental arid climate, characterised by low annual precipitation and high evaporation rates [36]. Furthermore, the total pyrazine content in the Xinjiang samples

(e.g., 2,5-dimethylpyrazine at 188.29 µg/kg, 2-ethyl-5-methylpyrazine 120.76 µg/kg) is significantly higher than that of other origins, which is highly consistent with its arid environment. In contrast, Zhejiang and Guangxi are situated in humid subtropical regions, characterised by abundant annual precipitation, a mild climate and high cloud cover, which attenuates the intensity of UV-B radiation. The lower levels of environmental stress in these regions result in reduced activation of stress-responsive secondary metabolic pathways, consistent with the findings of lower levels of pyrazines, phenolics, and total volatile compounds [37]. Although Yunnan is at a lower latitude, it has a high-altitude plateau climate with strong UV-B radiation; at the same time, it receives abundant precipitation due to the influence of the monsoon, creating a moderately stressful environment, and its volatile compound content is at a moderate level. Jilin is located in the northeastern temperate region, with cold winters and a short growing season, which may limit the accumulation of volatile compounds dependent on heat and light, whilst its fertile black soil supports a unique composition of precursors. Malik et al [38] demonstrated that chicory populations from different agroclimatic zones in the Kashmir Himalayas exhibit significant differences in phytochemical composition, and that altitude, soil chemistry, and growing conditions significantly influence metabolite diversity.

Clustering heatmap analysis of volatile components in chicory roots from different origins

Cluster heatmaps visualize the distribution of experimental data, serving to monitor data quality and concretely display variations. Fig. 3 displays the cluster heatmap of the 95 volatile compounds identified in chicory root samples from different geographical origins. In the heatmap, deeper blue shades correspond to lower compound concentrations, while deeper red shades indicate higher concentrations.

As shown in Fig. 3, chicory root samples from different origins can be grouped into three categories. Samples from Guangxi, Zhejiang, and Jilin form one group, indicating relatively minor differences in volatile components among these three origins. Within this cluster, the contents of aromatic constituents such as esters, hydrocarbons, phenols, and heterocyclic compounds was the lowest compared to chicory root samples from other origins. Furthermore, heptadecane and 2-methyl-trisilane were detected exclusively in these three samples; ketonals and other aromatic compounds exhibited relatively higher concentrations. Among them, 3-phenylcyclopentane-1,2-dione was detected exclusively in Guangxi chicory root samples, and 5-butyl-dihydro-4-methyl-2(3H)-furanone was identified solely in Zhejiang chicory root samples. The chicory root samples from the other two origins formed separate groups, indicating significant differences in volatile components between the Xinjiang and Yunnan chicory root samples, as well as between these two groups and the Guangxi, Zhejiang, and Jilin chicory root samples.

The Xinjiang chicory root sample exhibited the highest volatile component content, with relatively low levels of ketones and similar compounds. It contained the highest concentrations of hydrocarbons, phenols, and heterocyclic compounds—including (R)-terpene diene, eugenol, and 2,5-dimethylpyrazine—compared to samples from other regions. Additionally, it contained significant amounts of dodecane, 7-hexadecene, 3-methylheptadecane, 2,6, 10-trimethyldodecane, 6,6-diethyl octadecane, 2,6-dimethylphenol, 5-allyl-2-methoxyphenol, 2-acetyl-3-ethylpyrazine, 2-isopentyl-6-methylpyrazine, 2,5-dimethyl-3-(1-propenyl)pyrazine, and 3,5-dimethyl-2-propylpyrazine, these compounds were detected exclusively in Xinjiang chicory roots, significantly contributing to the overall aroma profile of chicory from this region. Yunnan chicory root samples showed higher levels of esters, ketones, hydrocarbons, phenols, and heterocyclic compounds. Among these, isovaleric acid isovalerate, di-sec-butyl phthalate, nonadecane, 1-undecene, (E)-2-methoxy-4-(1-allylphenol), 2-acetyl-3-methylpyrazine, and 2-methyl-6-(3-methylbutyl)pyrazine were exclusively detected in Yunnan chicory root samples.

OPLS-DA analysis of chicory roots from different origins

An OPLS-DA model was constructed based on the content of 95 volatile compounds in chicory roots from five different origins. With origin as the independent variable and the volatile compounds as dependent variables, this model effectively discriminated chicory root samples among the five origins. As illustrated in Fig. 4(a), the coefficient of determination for the independent variable (R_x^2) was 0.976. The closer R_x^2 approaches 1, the more stable the model. The dependent variable coefficient of determination (R_y^2) was 0.994; a higher R_y^2 indicates stronger model explanatory power. All sample confidence levels fell within the 95% confidence interval. The model prediction index (Q^2) was 0.988, with values above 0.5 indicating acceptable model fit [39]. Yunnan chicory root samples are located in the first quadrant, Xinjiang samples in the second quadrant, while samples from Guangxi, Jilin, and Zhejiang are clustered in the fourth quadrant. This spatial distribution indicates that, apart from minor variations in volatile profiles among Guangxi, Jilin, and Zhejiang origins, there are significant differences in the volatile compositions of chicory roots from different production regions.

As illustrated in Fig. 4(b), after 200 permutation tests, the permutation test yielded an R^2 value of 0.109 and a Q^2 value of −0.851. The intersection point of the Q^2 regression line with the vertical axis was below

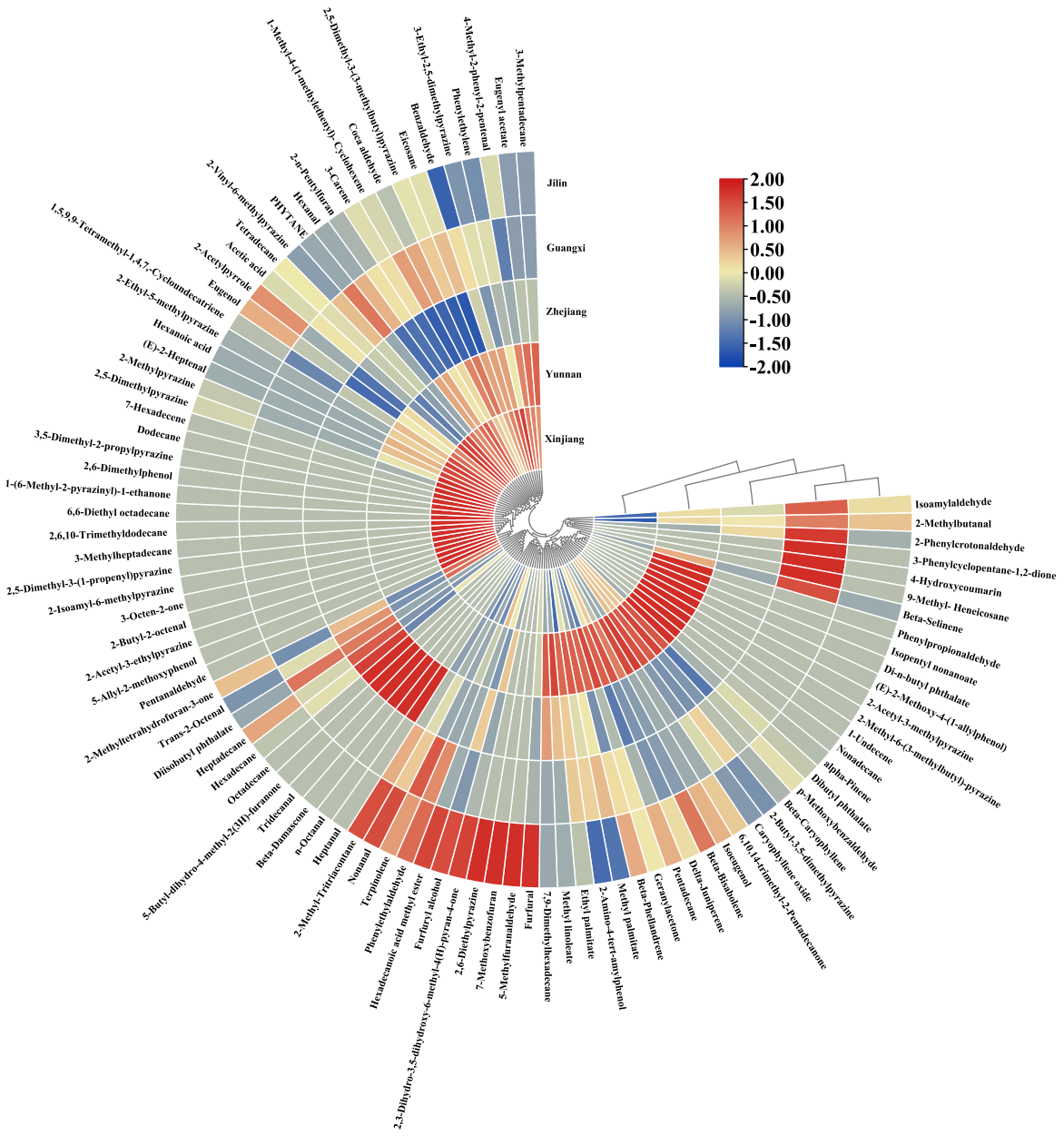

Fig. 3 Cluster heat map of volatile components in chicory root samples from different origins.

zero, and both the R^2 and Q^2 values at the far right exceeded the predicted values on the left. This indicates that the model exhibits no overfitting, validates the model's effectiveness, and suggests that the results can be utilised for origin identification analysis of chicory root aroma.

Fig. 4(c) presents the variable importance in projection VIP values. Variables with VIP > 1.0 are generally considered significant contributors to the classification process. A higher VIP value indicates a greater degree of differentiation between chicory root samples and a more pronounced contribution to distinguishing samples from different origins [40]. The experiment identified 39 distinct volatile compounds from chicory root samples sourced from various regions, including 9-methyl-heneicosane, 4-hydroxycoumarin, 3-phenylcyclopentane-1, 2-dione, 2-phenylcrotonaldehyde, β-selinene, furfural, hexadecanoic acid methyl ester, 5-methylfuranaldehyde, 7-methoxybenzofuran, 2,6-diethylpyrazine, terpinolene, furfural alcohol, 2,3-dihydro-3,5-dihydroxy-6-methyl-4(H)-pyran-5-

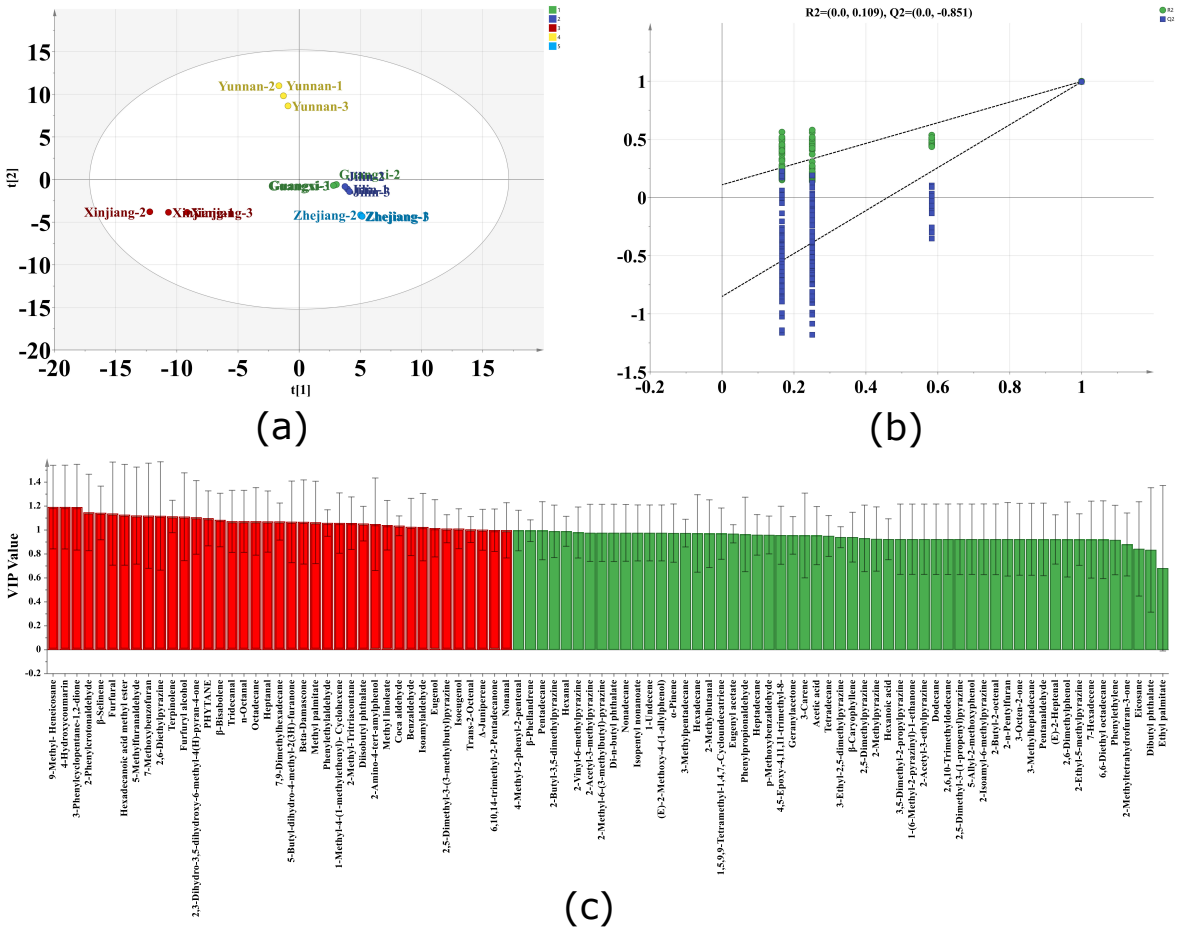

Fig. 4 OPLS-DA score plot (a), permutation test plot (b), and VIP plot (c) of volatile components in chicory roots from different origins.

one, phytane, β-bisabolene, tridecanal, n-octanal, octadecane, heptanal, 7,9-dimethylhexadecane, 5-butyldihydro-4-methyl-2(3H)-furanone, β-damascenone, methyl palmitate, phenylacetaldehyde, 1-methyl-4-(1-methylethenyl)-cyclohexene, 2-methyltrisilane, diisobutyl phthalate, 2-amino-4-tert-pentylphenol, methyl linoleate, coca-aldehyde, benzaldehyde, isovaleraldehyde, eugenol, 2,5-dimethyl-3-(3-methylbutyl)pyrazine, isoeugenol, trans-2-octenal, Δ-juniperene, 6,10,14-trimethyl-2-pentadecanone, and nonanal. These compounds may be employed to distinguish the provenance of chicory root.

CONCLUSION

In this study, HS-SPME-GC-MS combined with OPLS-DA and cluster heatmap analysis was applied to compare the volatile profiles of chicory root samples from different geographical origins. The Xinjiang sample contained the highest number of volatile compounds (60 in total), while the Jilin sample contained the fewest (42 in total). Significant variations were observed in both the variety and concentrations of volatile compounds among samples from different regions. Nineteen volatile compounds were common to all samples, whereas Xinjiang, Yunnan, Jilin, Guangxi, and Zhejiang chicory roots exhibited 15, 8, 6, 5, and 5 unique compounds, respectively. Aldehydes and hydrocarbons were identified as the predominant volatile classes in chicory roots across all origins. Ketones were confirmed to be key contributors to the baked aroma of chicory root, with 2,3-dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one exhibiting the highest content across chicory root samples from different origins. Cluster heatmap analysis grouped chicory roots from Zhejiang, Guangxi, and Jilin into one cluster, indicating a high similarity in their volatile compositions. This was further supported by OPLS-DA, which confirmed only minor differences among these three origins, while significant compositional variations distinguished them from samples of other origins. Based on VIP values, 39 compounds were selected as key markers for origin discrimination, including 9-methyl-heneicosene, furfural, 5-

methylfuranaldehyde, and 2,3-dihydro-3,5-dihydroxy-6-methyl-4(H)-pyran-5-one. This study provides scientific support for chicory origin tracing and further development of chicory root resources.

Appendix A. Supplementary data

Supplementary data associated with this article can be found at https://dx.doi.org/10.2306/scienceasia1513-1874.2026.074.

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Appendix A. Supplementary data

Table S1 Comparative analysis of volatile components and content (μg/kg) of chicory root samples from different habitats.

Code	Compound	RI	Guangxi	Jilin	Xinjiang	Yunnan	Zhejiang	Qualitative methods
1	Isoamylaldehyde	648	7.70 ± 0.10^{c}	4.60 ± 0.20^{b}	—	4.59 ± 0.34^{b}	3.74 ± 0.54^{a}	MS, RI
2	Pentanaldehyde	692	—	—	5.42 ± 0.54	—	—	MS, RI
3	Hexanal	701	9.04 ± 1.59^{c}	5.60 ± 1.13^{b}	10.06 ± 1.17^{d}	9.33 ± 1.92^{c}	3.57 ± 0.12^{a}	MS, RI
4	Furfural	762	42.65 ± 3.71^{b}	147.80 ± 11.20^{d}	60.24 ± 4.04^{c}	57.38 ± 5.17^{c}	36.85 ± 2.63^{a}	MS, RI
5	Heptanal	867	—	—	—	—	1.33 ± 0.05	MS, RI
6	Phenylpropionaldehyde	1171	—	—	—	30.83 ± 2.71	—	MS, RI
7	Benzaldehyde	948	20.91 ± 2.63^{b}	—	30.75 ± 2.37^{d}	27.67 ± 3.34^{c}	16.11 ± 1.21^{a}	MS, RI
8	Phenylethylaldehyde	1043	72.54 ± 5.27^{d}	76.89 ± 10.25^{d}	44.84 ± 3.29^{a}	55.22 ± 4.10^{c}	49.44 ± 5.76^{b}	MS, RI
9	Nonanal	1102	72.85 ± 4.28^{d}	87.01 ± 7.10^{e}	55.80 ± 4.09^{a}	61.90 ± 6.98^{b}	67.82 ± 4.69^{c}	MS, RI
10	Tridecanal	1417	—	—	—	—	19.68 ± 1.91	MS, RI
11	n-Octanal	989	—	—	—	—	19.57 ± 1.92	MS, RI
12	Coca aldehyde	1412	49.03 ± 3.10^{d}	31.72 ± 4.64^{b}	44.39 ± 2.23^{c}	54.61 ± 5.10^{e}	14.00 ± 1.13^{a}	MS, RI
13	5-Methylfuranaldehyde	964	—	50.16 ± 5.08	—	—	—	MS, RI
14	2-Methylbutanal	658	11.87 ± 1.06^{d}	9.17 ± 2.04^{c}	—	8.22 ± 1.76^{b}	7.46 ± 0.75^{a}	MS, RI
15	4-Methyl-2-phenyl-2-pentenal	1391	—	25.51 ± 2.89^{b}	54.44 ± 3.87^{c}	52.69 ± 6.32^{c}	13.25 ± 1.89^{a}	MS, RI
16	p-Methoxybenzaldehyde	1284	21.39 ± 1.75^{b}	26.50 ± 3.07^{c}	33.63 ± 2.11^{e}	51.03 ± 6.11^{d}	5.84 ± 0.66^{a}	MS, RI
17	2-Phenylcrotonaldehyde	1290	12.81 ± 1.12^{b}	—	—	—	4.61 ± 0.50^{a}	MS, RI
18	Trans-2-Octenal	1056	13.35 ± 1.10^{a}	—	33.52 ± 2.12^{c}	—	28.05 ± 2.09^{b}	MS, RI
19	(E)-2-Heptenal	949	—	—	7.95 ± 1.11^{b}	3.84 ± 0.39^{a}	—	MS, RI
20	2-Butyl-2-octenal	1385	—	—	74.83 ± 5.13	—	—	MS, RI
	Aldehydes Subtotal		334.14 ± 25.71^{a}	464.96 ± 22.98^{b}	455.87 ± 21.65^{b}	417.31 ± 28.46^{b}	291.32 ± 47.60^{a}	
21	Furfuryl alcohol	830	0.69 ± 0.08^{a}	50.02 ± 3.27^{d}	20.91 ± 4.45^{b}	0.58 ± 0.06^{a}	25.81 ± 1.88^{c}	MS, RI
22	Acetic acid	605	37.32 ± 2.94^{d}	30.33 ± 3.23^{c}	82.27 ± 5.42^{e}	7.31 ± 0.32^{a}	17.13 ± 1.50^{b}	MS, RI
23	Hexanoic acid	973	—	—	34.39 ± 3.73^{b}	18.77 ± 1.69^{a}	—	MS, RI
	Subtotal for acids		37.32 ± 2.94^{c}	30.33 ± 3.23^{b}	116.66 ± 7.15^{d}	26.08 ± 0.24^{b}	17.13 ± 1.50^{a}	
24	Eugenyl acetate	1443	—	—	32.53 ± 3.25^{b}	36.45 ± 3.71^{c}	7.91 ± 0.70^{a}	MS, RI
25	Isopentyl nonanoate	1492	—	—	—	29.87 ± 1.99	—	MS, RI
26	Methyl palmitate	1867	28.46 ± 2.10^{c}	—	13.01 ± 1.68^{a}	41.53 ± 2.70^{d}	21.95 ± 1.57^{b}	MS, RI
27	Ethyl palmitate	1900	9.06 ± 1.60^{ab}	8.10 ± 1.20^{ab}	6.79 ± 0.77^{a}	10.18 ± 1.93^{b}	9.02 ± 1.67^{ab}	MS, RI
28	Methyl linoleate	2009	—	—	—	15.81 ± 2.08^{b}	7.65 ± 1.31^{a}	MS, RI
29	Hexadecanoic acid methyl ester	1868	—	20.86 ± 2.79^{b}	10.28 ± 1.71^{a}	—	—	MS, RI
30	Dibutyl phthalate	1895	4.57 ± 1.05^{a}	4.26 ± 1.00^{a}	3.86 ± 0.92^{a}	7.33 ± 1.30^{b}	3.91 ± 0.94^{a}	MS, RI
31	Diisobutyl phthalate	1846	15.06 ± 1.92^{a}	—	—	—	14.75 ± 1.91^{a}	MS, RI
32	Di-n-butyl phthalate	1746	—	—	—	26.50 ± 2.45	—	MS, RI
	Subtotal for Esters		57.15 ± 6.67^{b}	33.22 ± 4.99^{a}	66.47 ± 8.33^{b}	167.67 ± 12.76^{c}	65.19 ± 8.10^{b}	

Table S1 Continued ...

Code	Compound	RI	Guangxi	Jilin	Xinjiang	Yunnan	Zhejiang	Qualitative methods
33	6,10,14-trimethyl-2-Pentadecanone	1828	—	10.21 ± 1.64^{a}	—	23.29 ± 2.67^{b}	—	MS, RI
34	Beta-Damascone	1436	—	—	—	—	7.75 ± 1.02	MS, RI
35	3-Octen-2-one	1038	—	—	9.92 ± 1.36	—	—	MS, RI
36	Geranylacetone	1326	37.16 ± 1.80^{b}	36.61 ± 5.95^{b}	—	78.30 ± 7.33^{c}	19.77 ± 1.86^{a}	MS, RI
37	2-Methyltetrahydrofuran-3-one	714	—	1.15 ± 0.23^{a}	1.77 ± 0.32^{a}	—	1.17 ± 0.09^{a}	MS, RI
38	3-Phenylcyclopentane-1,2-dione	1391	32.33 ± 2.30	—	—	—	—	MS, RI
39	1-(6-Methyl-2-pyrazinyl)-1-ethanone	1126	—	—	55.54 ± 5.09	—	—	MS, RI
40	2,3-Dihydro-3,5-dihydroxy-6-methyl-4(H)-pyran-4-one	1284	230.98 ± 6.68^{b}	550.89 ± 18.07^{d}	208.33 ± 17.63^{ab}	367.08 ± 15.15^{c}	196.89 ± 14.43^{a}	MS, RI
41	5-Butyl-dihydro-4-methyl-2(3H)-furanone	1342	—	—	—	—	17.35 ± 2.10	MS, RI
	Ketones subtotal		300.47 ± 10.78^{b}	598.86 ± 21.49^{d}	275.56 ± 24.64^{ab}	468.67 ± 25.15^{c}	242.93 ± 20.20^{a}	
42	Eugenol	1375	163.45 ± 4.51^{b}	216.92 ± 13.34^{d}	248.55 ± 13.97^{e}	186.36 ± 6.80^{c}	105.02 ± 7.83^{a}	MS, RI
43	Isoeugenol	1477	—	28.04 ± 5.48^{a}	—	50.62 ± 5.70^{b}	—	MS, RI
44	4-Hydroxycoumarin	1656	41.82 ± 3.81	—	—	—	—	MS, RI
45	2,6-Dimethylphenol	1220	—	—	28.32 ± 4.91	—	—	MS, RI
46	5-Allyl-2-methoxyphenol	1375	—	—	218.64 ± 17.32	—	—	MS, RI
47	2-Amino-4-tert-amylphenol	1689	75.33 ± 3.66^{b}	—	61.13 ± 8.36^{a}	123.71 ± 9.54^{c}	58.70 ± 4.54^{a}	MS, RI
48	(E)-2-Methoxy-4-(1-allylphenol)	1476	—	—	—	56.77 ± 3.46	—	MS, RI
	Phenols subtotal		280.60 ± 9.98^{b}	244.96 ± 18.82^{b}	556.64 ± 44.56^{c}	417.46 ± 11.90^{d}	153.72 ± 12.37^{a}	
49	2-Methylpyrazine	758	—	6.31 ± 1.25^{a}	47.41 ± 3.95^{c}	12.51 ± 1.74^{b}	—	MS, RI
50	2,5-Dimethylpyrazine	880	20.76 ± 1.28^{a}	42.85 ± 2.41^{b}	188.29 ± 10.07^{c}	15.31 ± 1.95^{a}	23.34 ± 2.07^{a}	MS, RI
51	2,6-Diethylpyrazine	1079	—	21.51 ± 3.17	—	—	—	MS, RI
52	3-Ethyl-2,5-dimethylpyrazine	1078	16.77 ± 0.25^{a}	—	42.63 ± 3.33^{c}	31.44 ± 4.19^{b}	—	MS, RI
53	2-Ethyl-5-methylpyrazine	991	—	—	120.76 ± 9.47^{b}	49.33 ± 6.93^{a}	—	MS, RI
54	2-Vinyl-6-methylpyrazine	1011	5.82 ± 0.54^{b}	—	10.54 ± 2.02^{c}	—	2.56 ± 1.02^{a}	MS, RI
55	2-Acetyl-3-methylpyrazine	1129	—	—	—	23.72 ± 2.15	—	MS, RI
56	2-Acetyl-3-ethylpyrazine	1172	—	—	45.62 ± 4.01	—	—	MS, RI
57	2-Butyl-3,5-dimethylpyrazine	1326	48.37 ± 1.64^{a}	—	53.26 ± 6.37^{a}	90.08 ± 7.81^{b}	—	MS, RI
58	2-Isoamyl-6-methylpyrazine	1260	—	—	77.70 ± 8.82	—	—	MS, RI
59	2,5-Dimethyl-3-(3-methylbutyl)pyrazine	1326	68.42 ± 3.54^{b}	54.53 ± 5.45^{a}	65.24 ± 4.02^{b}	95.62 ± 7.01^{c}	—	MS, RI
60	2,5-Dimethyl-3-(1-propenyl)pyrazine	1236	—	—	35.63 ± 4.00	—	—	MS, RI
61	3,5-Dimethyl-2-propylpyrazine	1339	—	—	54.17 ± 5.39	—	—	MS, RI
62	2-Methyl-6-(3-methylbutyl)-pyrazine	1260	—	—	—	40.60 ± 3.66	—	MS, RI
63	2-n-Pentylfuran	976	33.03 ± 1.93^{bc}	27.51 ± 3.54^{b}	41.66 ± 4.59^{e}	37.30 ± 3.97^{cd}	20.86 ± 2.10^{a}	MS, RI
64	7-Methoxybenzofuran	1351	—	15.61 ± 1.95	—	—	—	MS, RI

Table S1 Continued ...

Code	Compound	RI	Guangxi	Jilin	Xinjiang	Yunnan	Zhejiang	Qualitative methods
65	2-Acetylpyrrole	1063	66.25 ± 3.00^{a}	102.32 ± 8.63^{bc}	104.61 ± 13.64^{c}	86.76 ± 4.89^{b}	50.26 ± 5.47^{a}	MS, RI
	Heterocyclic Subtotal		259.42 ± 12.18^{b}	270.64 ± 42.86^{b}	887.52 ± 79.68^{d}	482.67 ± 44.30^{c}	97.02 ± 10.06^{a}	
66	α-Pinene	909	1.83 ± 0.20^{a}	—	—	66.43 ± 6.42^{b}	—	MS, RI
67	Phenylethylene	873	2.92 ± 0.18^{b}	—	8.47 ± 1.89^{c}	3.48 ± 1.37^{b}	1.61 ± 0.51^{ab}	MS, RI
68	3-Carene	1010	5.38 ± 0.18^{a}	4.57 ± 1.11^{a}	9.52 ± 2.33^{b}	5.25 ± 1.28^{a}	—	MS, RI
69	1-Methyl-4-(1-methylethenyl)-Cyclohexene	1030	96.05 ± 7.32^{c}	62.01 ± 4.84^{b}	102.10 ± 16.53^{c}	75.86 ± 4.16^{b}	10.95 ± 1.41^{a}	MS, RI
70	Terpinolene	1090	22.78 ± 1.85^{b}	16.11 ± 2.15^{a}	—	—	—	MS, RI
71	β-Phellandrene	1414	38.58 ± 1.44^{a}	53.46 ± 3.80^{b}	—	74.65 ± 4.98^{c}	—	MS, RI
72	β-Caryophyllene	1452	72.10 ± 3.18^{b}	68.52 ± 4.36^{ab}	99.07 ± 14.45^{c}	138.59 ± 9.67^{d}	56.02 ± 4.23^{a}	MS, RI
73	β-Bisabolene	1528	—	71.19 ± 8.44^{b}	16.91 ± 1.78^{a}	69.80 ± 5.43^{b}	—	MS, RI
74	Δ-Juniperene	1550	—	37.67 ± 3.20^{a}	43.04 ± 6.52^{a}	79.61 ± 7.24^{b}	—	MS, RI
75	Caryophyllene oxide	1625	15.38 ± 1.01^{a}	—	28.01 ± 4.68^{b}	49.16 ± 6.53^{c}	—	MS, RI
76	β-Selinene	1521	48.66 ± 1.32^{b}	—	—	29.52 ± 3.74^{a}	—	MS, RI
77	1,5,9,9-Tetramethyl-1,4,7,-Cycloundecatriene	1487	—	23.79 ± 1.84^{a}	90.55 ± 9.70^{c}	54.75 ± 4.88^{b}	25.69 ± 1.89^{a}	MS, RI
78	PHYTANE	1711	20.74 ± 1.59^{a}	—	20.61 ± 2.02^{a}	—	—	MS, RI
79	Eicosane	1728	45.63 ± 18.27^{b}	39.71 ± 5.55^{b}	48.01 ± 6.21^{b}	48.33 ± 5.30^{b}	21.66 ± 1.88^{a}	MS, RI
80	Pentadecane	1749	19.52 ± 1.47^{b}	31.98 ± 3.98^{c}	25.46 ± 3.17^{b}	39.64 ± 5.61^{d}	12.20 ± 1.76^{a}	MS, RI
81	Tetradecane	1403	26.17 ± 1.84^{b}	29.93 ± 2.76^{b}	65.85 ± 4.00^{c}	—	21.50 ± 1.82^{a}	MS, RI
82	Hexadecane	1604	15.87 ± 1.49^{b}	11.04 ± 1.83^{b}	16.90 ± 1.66^{b}	—	41.99 ± 4.37^{a}	MS, RI
83	Heptadecane	1807	11.81 ± 0.55^{a}	25.73 ± 3.26^{b}	—	—	36.79 ± 2.42^{c}	MS, RI
84	Dodecane	1198	—	—	17.20 ± 1.79	—	—	MS, RI
85	Octadecane	1773	—	—	—	—	33.50 ± 1.76	MS, RI
86	7-Hexadecene	1592	—	—	23.16 ± 4.40	—	—	MS, RI
87	Nonadecane	1604	—	—	—	21.42 ± 1.84	—	MS, RI
88	1-Undecene	1162	—	—	—	71.92 ± 4.42	—	MS, RI
89	3-Methylheptadecane	1776	—	—	18.99 ± 2.64	—	—	MS, RI
90	3-Methylpentadecane	1576	—	—	65.40 ± 5.45^{b}	82.82 ± 6.74^{c}	16.59 ± 1.37^{a}	MS, RI
91	2,6,10-Trimethyldodecane	1550	—	—	19.00 ± 2.03	—	—	MS, RI
92	7,9-Dimethylhexadecane	1339	—	—	—	49.73 ± 5.53^{b}	34.56 ± 1.70^{a}	MS, RI
93	9-Methyl- Heneicosane	1542	11.87 ± 0.46	—	—	—	—	MS, RI
94	2-Methyl-Tritriacontane	1576	51.40 ± 2.56^{b}	83.39 ± 6.08^{c}	—	—	12.31 ± 1.34^{a}	MS, RI
95	6,6-Diethyl octadecane	1497	—	—	23.10 ± 4.53	—	—	MS, RI
	Hydrocarbons Subtotal		506.69 ± 13.36^{b}	559.31 ± 53.20^{b}	741.35 ± 95.78^{c}	960.96 ± 85.14^{d}	325.37 ± 26.46^{a}	

"—" indicates that the compound was not detected. Different lowercase letters within the same row denote significant differences ($p<0.05$).