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Identification of characteristic flavor compounds of *boletus edulis* from different regions based on by *E*-nose, HS-GC-IMS and HS-SPME-GC–MS

Weilan Li [a,b], Luxi Zi [a,b], Ningmeng Xu [a,b], Hao Yang [a,b], Shihao Dong [a,b], Fen Qin [a,b], Lei Guo [a,b,*]

[a] *The Key Laboratory of Forest Resources Conservation and Utilization in the Southwest Mountains of China Ministry of Education, Southwest Forestry University, Kunming, Yunnan, 650224, China*

[b] *College of Biological Science and Food Engineering, Southwest Forestry University, Kunming, Yunnan, 650224, China*

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Methional (PubChem CID: 18635)
1-Octen-3-one (PubChem CID:61346)
1-Dodecanol (PubChem CID: 8193)
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2-Undecanone (PubChem CID: 8163)
3-Phenyl-1-propanol (PubChem CID: 31234)

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ABSTRACT

In this study, *E*-nose, HS-GC-IMS, and HS-SPME-GC–MS technologies were used to evaluate the flavor characteristics of the pileus and stipe of *Boletus edulis* from eight origins. 23 key Volatile organic compounds (VOCs) with odor activity values (OAVs) > 1 were identified, and 19 aroma types have been identified in *Boletus edulis* at the same time. Vegetable and earthy were defined as the dominant aroma types for all pileus and stipe samples. Balsamic and musty were the main and characteristic aroma types for the pileus. The highest concentrations of VOCs in the pileus and stipe were originated from Chuxiong Prefecture and Aba Prefecture, respectively. 19 and 16 key VOCs were detected Chuxiong pileus and Aba stipe, respectively, and Methional was the decisive compound that influenced the vegetable aroma type. The results of this study could be helpful for flavor identification and application of pileus and stipe from *Boletus edulis.*

1. Introduction

Boletus edulis is a wild edible mushroom that attracts large groups of consumers with its high nutritional value and unique flavor (Liuzzi et al., 2023; Tan et al., 2022). Proteins, amino acids, and other nutrients are abundant in *Boletus edulis*, while physiological active functions such as hypoglycemia, anti-inflammation, and regulation of intestinal microorganisms are also part of its repertoire (Avram et al., 2023; Zanfirescu et al., 2024). In addition, a unique flavor of *Boletus edulis* is often one of the reasons why it is chosen by consumers (Huang et al., 2023). Previous studies have focused on the assessment of the nutritional value and bioactivity of chemical compounds from *Boletus edulis* (Popa et al., 2022; Yu et al., 2022). However, Differences in the composition of flavor compounds between the pileus and stipe of *Boletus edulis* have been no reports on these studies.

Flavor is an important indicator for evaluating the quality of *Boletus edulis*, and it is also one of the main factors affecting the market value of *Boletus edulis* deep-processed products. The overall flavor of *Boletus edulis* consists of the odor of the volatile organic compounds (VOCs), and variations in the amount and type of VOCs are responsible for the different intensity and type of aroma exhibited by *Boletus edulis* (Mourão et al., 2023). Up to now, the flavor profile of *Boletus edulis* have not been comprehensively identified and analyzed. Different geographical locations and environmental factors can lead to differentiated flavor characteristics of edible mushroom (Xu et al., 2024). Even in the same growing environment, there is a large difference in flavor characteristic between the pileus and stipe of edible mushroom. Li et al. (2019) analyzed the flavor differences of *Tricholoma matsutake* Singer and found that the characteristic difference of volatile compounds between the pileus and stipe was obviously.

A single detection technology cannot identify some key VOCs and therefore cannot reflect complete information about the flavor of the sample. At present, electronic nose (*E*-nose) is a popular smart sensing technology and applied in edible mushroom. It is equipped with

* Corresponding author at: The Key Laboratory of Forest Resources Conservation and Utilization in the Southwest Mountains of China Ministry of Education, Southwest Forestry University, Kunming, Yunnan 650224, China.

E-mail address: guolei@swfu.edu.cn (L. Guo).

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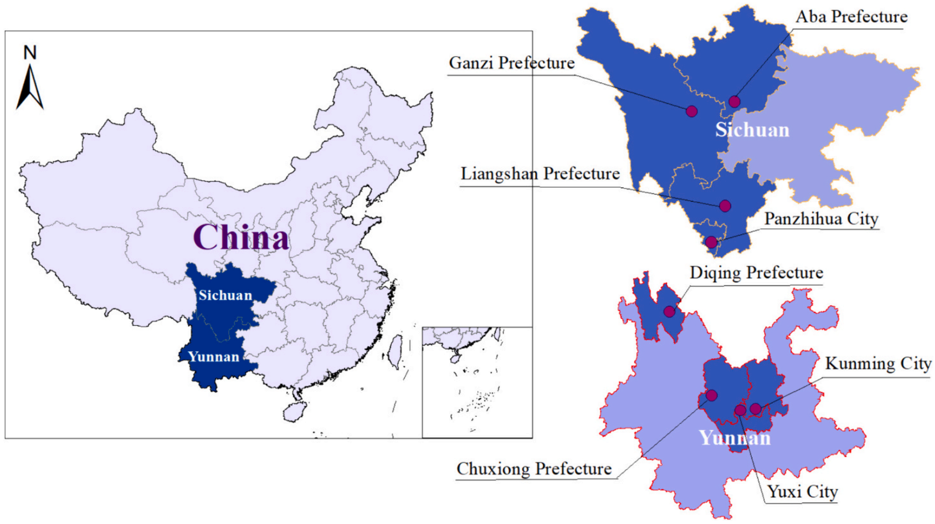

Fig. 1. The geographical distribution of the eight most representative Chinese *Boletus edulis*.

chemical sensors and pattern recognition systems that enable rapid detection of the overall flavor profile, which can partially eliminate the subjectivity of human sensory evaluation (Rong et al., 2023; Xiong et al., 2023). Headspace solid-phase micro-extraction gas chromatography–mass spectrometry (HS-SPME-GC–MS) technology can enable qualitative testing of flavor compounds, which have the safety of HS-SPME, the high accuracy and separation capacity of GC–MS (Liu et al., 2023; Xi et al., 2024). Headspace gas chromatography-ion mobility spectrometry (HS-GC-IMS) is a flavor detection technique that has emerged in recent years, which can realize visual analysis of flavor compounds (Xu, Zhang, et al., 2023; Yang et al., 2023). Some VOCs have not been identified because of the lack of a complete database for HS-GC-IMS (Xiao et al., 2022). But compared to HS-SPME-GC–MS, HS-GC-IMS has many advantages, such as high analytical efficiency, visualization of flavor substances, and low cost (Fan et al., 2023). Therefore, a combined HS-SPME-GC–MS and HS-GC-IMS approach for detecting VOCs may be an ideal strategy for more comprehensive information on volatiles. Chen, Qin, et al. (2021) characterized 32 and 28 volatile components in *shiitake* mushrooms at different drying stages by GC-IMS and GC–MS, respectively. 1-octene-3-ol and 3-octanone were the key flavor compounds and exude mushroom-like odor. In addition, in our previous study, 64 and 85 VOCs were identified in *Boletus edulis* with different drying methods by GC-IMS and GC–MS, respectively. Analysis of the relative odor activity values indicated 1-octen-3-ol was one of the key VOCs of *Boletus edulis* (Li, Yang, et al., 2024).

In this study, flavor profile of pileus and stipe in *Boletus edulis* originated from different origins was evaluated and analyzed by a combination of *E*-nose, HS-SPME-GC–MS, and HS-GC-IMS techniques. This study aimed to establish the flavor fingerprints of the pileus and stipe of *Boletus edulis*, and analyze the differences on characteristic flavor compound and aroma type of the pileus and stipe. The results of the study provide a certain scientific basis for the identification of VOCs and resource development and utilization of *Boletus edulis*.

2. Materials and methods

2.1. Sample preparation

Fresh *Boletus edulis* with similar freshness were collected from the main production areas in Yunnan and Sichuan Province, including Kunming (KM), Chuxiong (CX), Yuxi (YX), Diqing (DQ), Liangshan (LS), Panzhihua (PZH), Aba (AB), Ganzi (GZ) (Fig. 1). Details related to the collection locations of the eight samples are provided in the Supplementary Information (Table S1). All the fresh mushrooms were cleaned and washed, and then both pileus and stipe of the samples were cut into 5 mm slices before vacuum freeze-drying (YTLG-12 A, Shanghai Yetuo Technology Co., Ltd., Shanghai, China). After drying, the dried samples were stored at −80 °C for further analysis.

2.2. Regents and chemicals

The extraction fiber (DVB/CAR/PDMS, 50/30 μm) was purchased from Supelco (Bellefonte, PA, USA). All chemical reagents were analytical or chromatographic grade, and 1-Decanol was used as an internal standard and purchased from Tokyo Chemical Industry Co., Ltd. (Tokyo, Japan). n-hexane was procured from Guangdong Guanghua Sci-tech Co.,Ltd. (Guangdong, China). n-Ketones (C4-C9) and n-alkanes (C8-C40) were purchased from Sigma-Aldrich (St. Louis, MO, USA).

2.3. E-nose analysis

The E-nose (PEN3, WinMuster Airsense Analytics Inc. Schwerin, Germany) consists of a sampling system and 10 different metal oxide gas sensors, an intelligent pattern recognition and analysis system (Chen et al., 2020). Odors within the measurable range can be detected by the E-nose system. According to the method of Feng, Wang, et al. (2022) with slight modifications. Briefly, 0.5 g of the sample was weighed into a 20 mL headspace bottle and sealed. It was then left at room temperature for 30 min to enrich for volatiles. The samples were introduced by direct headspace aspiration at a flow rate of 400 mL/min, with a sampling time of 1 s for 100 s for each group.

2.4. HS-GC-IMS analysis

The VOCs produced by *Boletus edulis* samples in different regions were detected by HS-GC-IMS (flavorSpec®, g.a.s. Dortmund, Germany). According to the method of Yang et al. (2022), 1 g of sample was placed into a 20 mL headspace vial and sealed. The headspace vials were incubated at 60 °C for 20 min. After incubation, 500 μL of headspace gas was added to the injection port in non-separation mode using a 1 mL gas-tight syringe (Gerstel GmbH, Mühlheim, Germany). A polar capillary column (DB-WAX, 30 m × 0.32 mm × 0.25 μm, Agilent Company, USA) was used for separation. High purity nitrogen (purity ≥99.999%) was used as the carrier gas. Carrier gas flow rates were as follows: 2 mL/min for 2 min, 20 mL/min for 8 min, 100 mL/min for 10 min. The drift gas flow rate was set to a constant 150 mL/min. The identification of VOCs was identified based on the retention index (RI) and the drift time (DT) with the GC-IMS library.

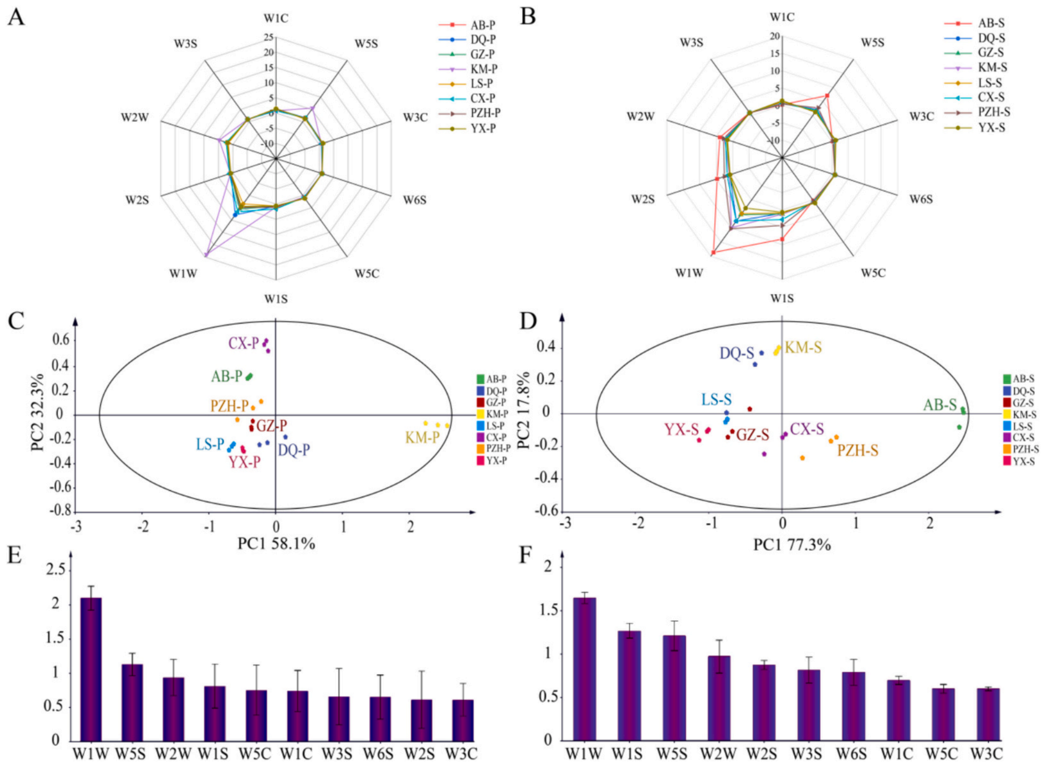

Fig. 2. E-nose response values of *Boletus edulis* from eight regions. Radar plot of pileus (A) and stipe (B). PCA score plot of pileus (C) and stipe (D). VIP plot of pileus (E) and stipe (F).

2.5. HS-SPME-GC–MS analysis

HS-SPME combined with GC–MS (7890B GC-5977B MSD) (Agilent Technologies Inc., Santa Clara, CA, USA) was used to analyze the VOCs of different samples. 0.5 g of sample was weighed into a headspace vial and 5.0 μL of 1-Decanol (0.1 mg/mL) was added as an internal standard. The mixture was equilibrated at 79 °C for 28 min. Then, the 50/30 μm DVB/CAR/PDMS fiber was inserted into the headspace vial to extract VOCs for 23 min. After extraction, VOCs were identified and quantified using GC–MS. GC–MS conditions were consistent with our previous study (Guo et al., 2023). Compounds with similarity >90% were retained by comparison with mass spectra from the NIST 2014 Standard Library. The retention indices (RIs) of the VOCs were calculated according to the linear formula for n-alkanes. The actual RIs were then compared with the theoretical RIs of the standard compounds in PubChem (https://pubchem.ncbi.nlm.nih.gov). The actual RIs were calculated as follows (Guo et al., 2021):

$$RI = 100 \times \left(n + \frac{T_i - T_n}{T_{n+1} - T_n}\right)$$

Where n represents the number of carbon atoms of n-alkanes; T_i is the retention time of VOC detected in the sample; T_n and T_{n+1} are the retention times of alkanes around target VOC ($T_n < T_i < T_{n+1}$).

The VOCs were analyzed by semi-quantitatively technology based on the linear relationship between peak areas and concentrations of the internal standard (1-Decanol, 0.1 mg/mL). The formula for quantitative calculation was as follows:

$$C_i = \frac{C_s \times A_i}{A_s}$$

Where C_i and C_s represent the concentrations of the target VOC and the internal standard, respectively; A_i and A_s were the peak areas of the compound and the internal standard respectively.

OAV analysis was used to assess the contribution of VOCs and the formula that follows was utilized:

$$OAV_i = \frac{C_i}{OT_i}$$

Where C_i and OT_i represent the concentration and odor threshold of the target VOC, respectively.

2.6. Statistical analysis

The experimental data were expressed as mean ± standard deviation. The ANOVA analysis of variance was performed by SPSS 27 (IBM, Armonk, NY, USA). ArcGIS software (http://www.esri.com) was used to draw the map. Radar charts and column stacking diagrams were plotted using Origin 2021 (Origin-Lab, Northampton, MA, USA). Cluster analysis heatmap was generated via https://www.chiplot.online/. Excel 2010 was used to draw sunburst and odor ring charts. Principal component analysis (PCA) and orthogonal partial least squares-discriminant analysis (OPLS-DA) were performed using SIMCA 14.1 software (Umetrics, Malmo, Sweden).

3. Results and discussion

3.1. E-nose analysis

E-nose was used to characterize the flavor characteristics of *Boletus edulis* from different origins, and the flavor radar plots were shown in Fig. 2A and B. The response values of 10 metal oxide gas sensors to VOCs in *Boletus edulis* were visualized. As shown in Fig. 2A, KM pileus sample was clearly of greater abundance in pileus of all origins. But in stipe, AB stipe sample had the greater abundance (Fig. 2B). In addition, sensors W1W (sulfur-containing) and W5S (nitrogen oxides) responded significantly to compounds of both pileus and stipe. Notably, no significant response of W2S sensor was observed in all samples, which represented the absence of alcohols. Alcohols were the main volatile substances in edible mushrooms, which indicated that the *E*-nose is not fully sensitive

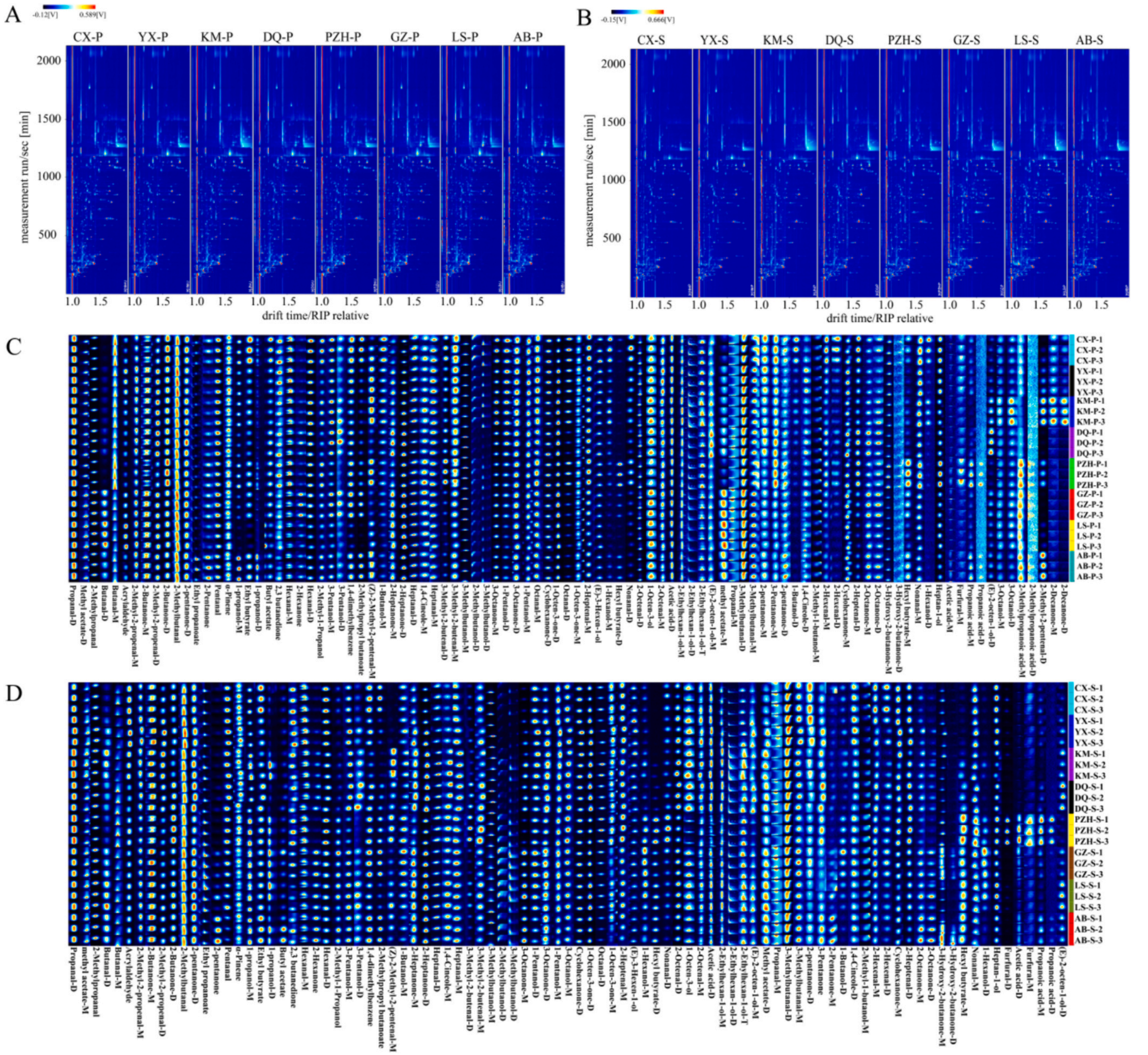

Fig. 3. Two-dimensional chromatogram results of VOCs in pileus (A) and stipe (B). Fingerprints of VOCs characterized in *Boletus edulis* of pileus (C) and stipe (D).

to the flavor substances of the *Boletus edulis* and there was only a part of flavor profile can be characterized. A similar finding was reported in the huajiao (Feng, Wang, et al., 2022).

Principal component analysis (PCA) is usually used to identify underlying patterns and relationships in the E-nose data to effectively assess similarities and differences between different samples (Xu, Wang, et al., 2023). As shown in Fig. 2C and D, the cumulative variance contribution rates of the pileus and stipe were 90.4% (PC1 58.1%, PC2 32.3%) and 95.1% (PC1 77.3%, PC2 17.8%) respectively, which included the whole flavor information elementarily. The samples of pileus and stipe from different regions were located in areas with an obvious boundary, indicating that PCA could significantly distinguish samples. The relative distances between KM and others in pileus, AB and other sample in stipe were far, respectively, which was consistent with the results depicted in radar chart.

The VIP value could filter the variables of variance that had important contributions to the model classification, and the variables with VIP values >1 were generally considered the main variance variables (Li, Wang, et al., 2024). As shown in Fig. 2E and F, the VIP values of the W1W and W5S sensors were >1 in pileus and stipe samples, which indicated that the W1W and W5S sensors contributed significantly to samples classification. In addition, the W1S (VIP > 1) sensor characterizing alkanes can be considered as a specific sensor for stipe samples.

3.2. Profile of VOCs in pileus and stipe from boletus edulis by HS-GC-IMS analysis

3.2.1. VOCs analysis

The top views of the HS-GC-IMS topographic maps were shown in Fig. 3A and B. The background of the HS-GC-IMS spectra was blue, and the red vertical line at abscissa 1.0 was reactive ion peak (RIP) after normalization. Each point on both sides of the RIP peak represented a VOC and the color reflected the concentration of the compound, and white color represents lower concentration and red means higher concentration (Feng, Sun, et al., 2022). Most signals occurred in the range of 100 to 1200 s RI and 1.0 to 2.0 ms DT. The peak intensities of all VOCs

Table 1
Identification of VOCs by HS-SPME-GC–MS.

No.	Volatile Compounds	Identification method	Actual RI	Theoretical RI	Concentration (μg/kg)		Aroma type
					pileus	stipe	
	Alcohols(16 kinds)						
1	1-Hexanol	MS,IMS, RI	869,1375*	867,1359*	0–7.52	0–5.73	herbal
2	Benzyl alcohol	MS, RI	1052	1052	0–175.64	0–179.9	floral
3	Phenylethyl alcohol	MS, RI	1111	1111	0–424.12	0–1155.92	floral
4	(*E*)-2-Octen-1-ol	MS,IMS, RI	1077,1455*	1067,1455*	0–578.43	0–479.47	green
5	1-Octen-3-ol	MS,IMS, RI	1014,1601*	1013,1603*	38.7–3532.22	32.23–2273.47	earthy
6	3-Octanol	MS,IMS, RI	1024,1498*	1013,1499*	0–1186.85	0–154.87	earthy
7	2-Ethylhexan-1-ol	MS,IMS, RI	1049,1444*	1035,1427*	225.52–2020.91	59.19–1088.24	citrus
8	1-Octanol	MS, RI	1079	1073	0–474.6	0–284.52	waxy
9	Cinnamyl alcohol	MS, RI	1304	1310	0–1096.93	0–615	balsamic
10	3-Phenyl-1-propanol	MS, RI	1198	1200	5.53–2079.33	1.56–1666.37	balsamic
11	Nonan-1-ol	MS, RI	1169	1171	0–18.78	0–22.89	floral
12	1,10-Decanediol	MS, RI	1514	1518	0–1080.28	0–995.1	NF
13	1-Dodecanol	MS, RI	1446	1464	138.95–643.51	165.01–734.71	waxy
14	(*E*)-Nerolidol	MS, RI	1584	1583	0–1465.36	0–172.48	floral
15	1-Hexadecanol	MS, RI	1889	1884	0–19.43	0–81.09	waxy
16	(9Z,12Z)-Octadeca-9,12-dien-1-ol	MS, RI	2035	2052	–	0–44.03	NF
	Aldehydes(8 kinds)						
17	Heptanal	MS,IMS, RI	899,1189*	899,1189*	0–23.3	2.27–11.93	green
18	Benzaldehyde	MS, RI	974	975	93.31–345.04	107.89–380.27	fruity
19	Phenylacetaldehyde	MS, RI	1058	1052	22.84–63.4	50.27–193.31	green
20	2-Octenal	MS,IMS, RI	1068,1430*	1063,1430*	0–5.96	0–17.39	fatty
21	2-Ethylhexanal	MS, RI	960	963	0–15.59	0–8.74	NF
22	Cinnamaldehyde	MS, RI	1266	1266	0–94.75	0–138.43	spicy
23	2-Phenyl-2-butenal	MS, RI	1274	1274	0–315.98	26.64–534.3	musty
24	Decanal	MS, RI	1215	1211	0–30.38	0–39.14	aldehydic
	Ketones(9 kinds)						
25	2-Heptanone	MS,IMS, RI	888,1195*	888,1198*	0–18.6	0–5.44	cheesy
26	Acetophenone	MS, RI	1073	1071	0–97.93	0–47.82	floral
27	1-Octen-3-one	MS,IMS, RI	983,1315*	983,1315*	0–20.59	0–69.01	earthy
28	3-Octanone	MS,IMS, RI	986,1266*	985,1266*	13.35–189.8	0–13.73	herbal
29	3-Decen-2-one	MS, RI	1236	1233	0–630.32	0–124.85	fatty
30	2-Decanone	MS,IMS, RI	1190,1479*	1191,1482*	0–243.65	0–47.08	floral
31	2-Undecanone	MS, RI	1272	1273	101.48–301.98	86.19–281.75	fruity
32	Geranylacetone	MS, RI	1432	1432	0–166.33	0–89.5	floral
33	6,10,14-Trimethylpentadecan-2-one	MS, RI	1855	1855	0–55.42	13.53–50.93	floral
	Esters(13 kinds)						
34	Methyl o-toluate	MS, RI	1175	1165	0–19.27	0–3.86	floral
35	Dimethyl phthalate	MS, RI	1460	1466	0–54.74	0–35.67	NF
36	2-Ethylhexyl acetate	MS, RI	1149	1149	0–8.49	–	earthy
37	Ethyl dodecanoate	MS, RI	1600	1597	0–26.83	0–206.71	waxy
38	2-Ethylhexyl salicylate	MS, RI	1815	1817	0–97.51	15.19–65.23	floral
39	Homosalate	MS, RI	1892	1904	0–34.72	0–33.78	NF
40	Dibutyl phthalate	MS, RI	1988	1970	17.74–53.6	15.21–136.35	NF
41	Diisobutyl phthalate	MS, RI	1884	1877	0–47.44	0–42.71	NF
42	Ethyl-hexadecenoate	MS, RI	1979	1978	–	0–82.34	NF
43	Ethyl palmitate	MS, RI	2010	1997	0–34.49	0–392.28	waxy
44	Ethyl Linoleate	MS, RI	2145	2144	0–165.96	0–817.25	fatty
45	Elaidic acid ethyl ester	MS, RI	2174	2174	–	0–133.59	NF
46	Bis(2-ethylhexyl) adipate	MS, RI	2399	2398	0–29.85	2.98–38.14	NF
	Acids(1 kind)						
47	2-Methylbutanoic acid	MS, RI	860	860	–	0–5.12	acidic
	Hydrocarbon(6 kinds)						
48	Styrene	MS, RI	886	886	0–37.09	ND	balsamic
49	m-Xylene	MS, RI	864	864	0–2.68	0–1.97	NF
50	3-Methyltridecane	MS, RI	1392	1377	0–60.17	0–103.24	NF
51	Undecylcyclopentane	MS, RI	1655	1656	–	0–22.59	NF
52	3-Methylpentadecane	MS, RI	1572	1571	0–22.29	0–119.85	NF
53	Undecylcyclohexane	MS, RI	1761	1760	0–27.13	0–22.31	NF
	Nitrogen-containing compounds(8 kinds)						
54	2-Methylpyrazine	MS, RI	818	826	12.08–60.69	0–4.53	nutty
55	2-Pyridinecarboxaldehyde	MS, RI	944	943	–	0–3.4	NF
56	2-Acetylpyrrole	MS, RI	1075	1063	0–238.28	0–238.29	musty
57	2,5-Dimethylpyrazin	MS, RI	912	912	46.34–346.93	0–27.25	chocolate
58	2-Ethenyl-6-methylpyrazine	MS, RI	1016	1016	0–53.03	0–21.71	NF
59	1-(6-Methyl-2-pyrazinyl)ethanone	MS, RI	1086	1095	31.86–142.34	ND	roasted
60	3-Ethyl-2,5-dimethylpyrazine	MS, RI	1083	1078	53.85–1439.55	0–235.33	nutty
61	2,3-Diethyl-5-methylpyrazine	MS, RI	1169	1171	0–94.13	ND	musty
	Sulfur-containing compounds(1 kind)						
62	Methional	MS, RI	904	904	19.09–191.7	43.88–693.74	vegetable
	Others(6 kinds)						
63	Furfural	MS, RI	830	835	0–9.57	0–220.99	bready

(*continued on next page*)

Table 1 (*continued*)

No.	Volatile Compounds	Identification method	Actual RI	Theoretical RI	Concentration (μg/kg)		Aroma type
					pileus	stipe	
64	2,3-Dihydro-3,5-dihydroxy-6-methyl-4 h-pyran-4-one	MS, RI	1144	1149	0–954.45	63.53–1363.77	NF
65	5-Methylfurfural	MS, RI	999	999	–	0–67.42	caramellic
66	2-Pentylfuran	MS, RI	1020	1003	–	0–22.77	fruity
67	(*Z*)-4-Hydroxy-6-dodecenoic acid lactone	MS, RI	1661	1661	0–10.54	0–28.17	fatty
68	2,4-Di-t-butylphenol	MS, RI	1513	1513	0–182.57	0–217.79	NF

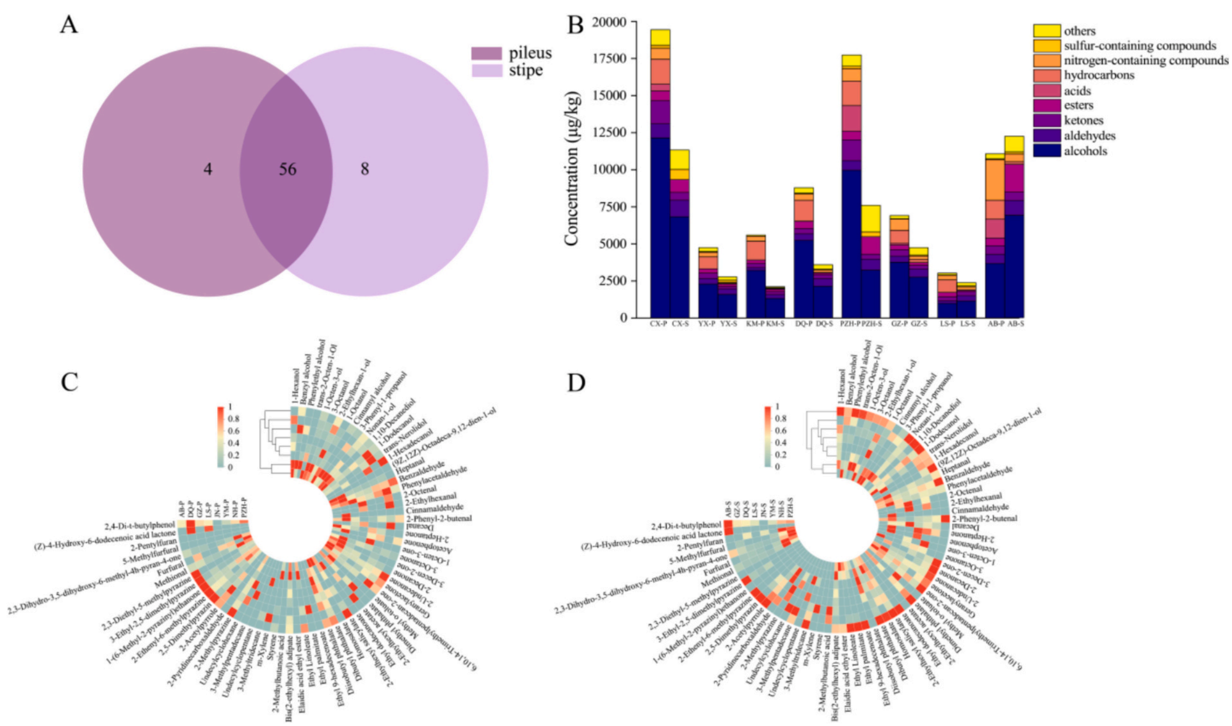

Fig. 4. Comparison of VOCs in pileus and stipe obtained by HS-SPME-GC–MS. (A) Venn diagram of the number of VOCs in pileus and stipe. (B) Bar chart of the content of each category of VOCs. Heatmaps of measured VOCs in pileus (C) and stipe (D).

were listed in Table S2. 56 and 53 VOCs were characterized in the pileus and stipe, respectively. Among them, all VOCs were common compounds between pileus and stipe, except 3-Octanol, 2-Decanone, 2-Methylpropanoic acid, which were detected only in pileus. Overall, 14 alcohols, 17 aldehydes, 12 ketones, 6 esters, 3 acids, 2 hydrocarbons and 2 others were characterized by HS-GC-IMS. It was noteworthy that 39 of these VOCs were available not only in the monomer (−M) form but also in the dimer (—D) form. In ionization region, the formation of dimers or polymers was related to the high proton affinity of the VOCs (Chen et al., 2023).

3.2.2. Fingerprint analysis

To further identify the differences of volatile profile among *Boletus edulis* from eight origins, all the VOCs identified in the HS-GC-IMS spectra were selected to generate volatile fingerprints using the Gallery Plot plug-in (Fig. 3C and D). Each row in the gallery plot revealed the entire signal peak of a grape sample, and each column showed the signal intensity of the same compound presented in different grape samples. As shown in Fig. 3C, it was evident that 3-Methylbutanal (M&D), 1-Octen-3-ol, Octanal-M, 3-Octanone-M, α-Pinene, 2-pentanone-D, 2-Methylbutanal, and Propanal-D were consistently present at high levels in all samples of pileus. In the volatile fingerprint of the stipe (Fig. 3D), 2-Butanone-M, 2-Methylbutanal, Propanal-D, and 3-methylbutanal-D had higher concentrations.

3.3. VOCs analysis of pileus and stipe from boletus edulis by HS-SPME-GC–MS

3.3.1. VOCs analysis

A total of 68 VOCs were identified by HS-SPME-GC–MS (Table 1), including 16 alcohols, 8 aldehydes, 9 ketones, 13 esters, 1 acid, 6 hydrocarbons, 8 nitrogen-containing compounds, 1 sulfur-containing compound and 6 others. As shown in Fig. 4A, there were 4 characteristic VOCs of the pileus, including 2-Ethylhexyl acetate (earthy, herbal odor), styrene (sweet, balsamic odor), 1-(6-Methyl-2-pyrazinyl) ethanone (roasted, coffee odor), and 2,3-Diethyl-5-methylpyrazine(musty, nut odor). Moreover, (9Z,12Z)-Octadeca-9, 12-dien-1-ol, Ethyl 9-hexadecenoate, Elaidic acid ethyl ester, 2-Methylbutanoic acid (acidic, fruity odor), Undecylcyclopentane, 2-Pyridinecarboxaldehyde, 5-Methylfurfural (sweet, caramellic odor), and 2-Pentylfuran (fruity, green odor) were detected only in the stipe, which indicated that there were significant variation in flavor characteristics between pileus and stipe of *Boletus edulis.*

The quantitative results of the VOCs in pileus and stipe were visualized and the results were shown in Fig. 4B. There were significant differences in the concentration of VOCs in the pileus and stipe of *Boletus edulis* from different origins. The highest sample concentration was found in CX pileus (16,087.64 μg/kg), followed by PZH pileus (12,907.53 μg/kg) and AB stipe (12,240.84 μg/kg). The lowest content was LS pileus (1683.81 μg/kg), which was only about 1/9 of CX pileus. It was worth mentioning that by comparing the difference in

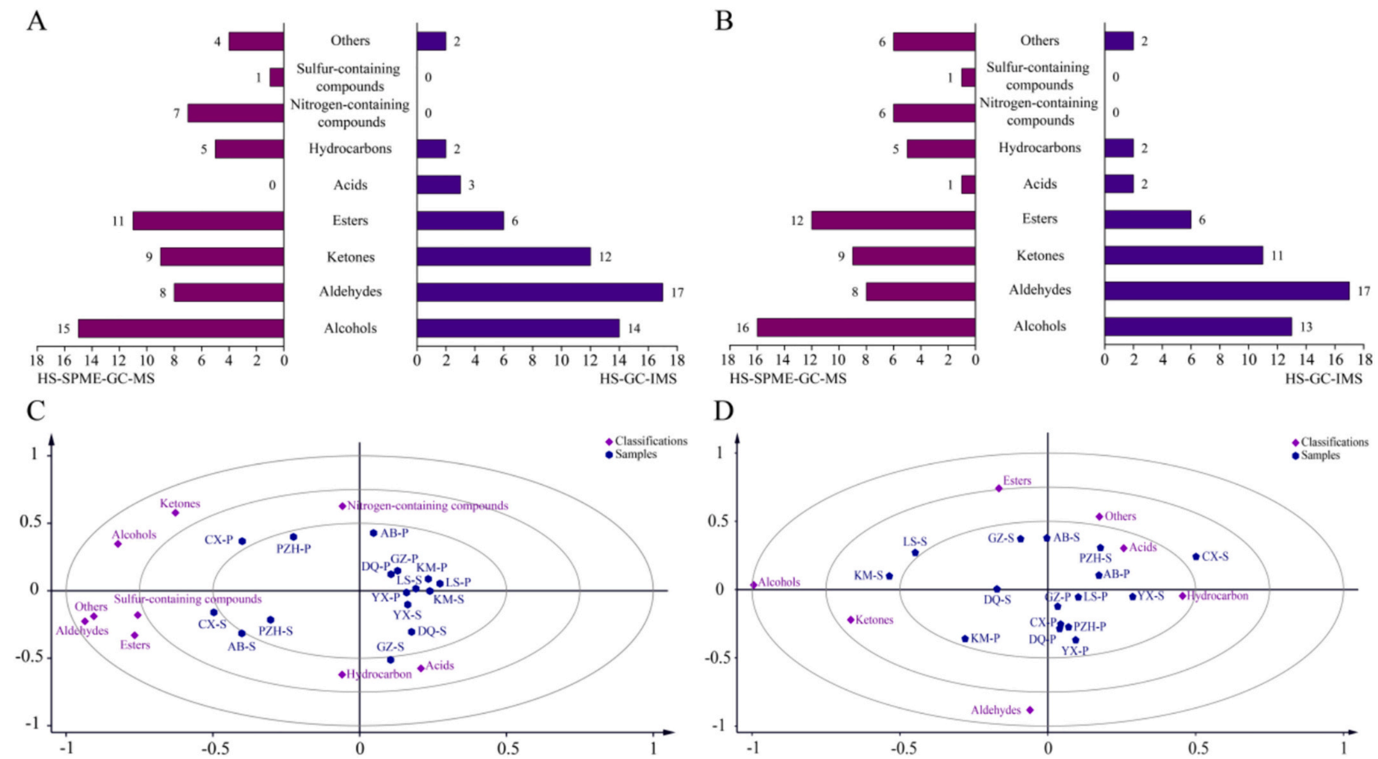

Fig. 5. Comparison of the results obtained by HS-GC-IMS and HS-SPME-GC–MS. Comparison of the number of each category of VOCs in pileus (A) and stipe (B). OPLS-DA biplots of HS-GC-IMS (C) and HS-SPME-GC–MS (D).

concentration between the pileus and stipe, it was found that the content of the pileus of *Boletus edulis* was significantly higher than that of the stipe, except for AB sample. This result showed that the pileus was more flavorful in most of the origins.

Identification method: MS, identification based on NIST 2014 mass spectral database. IMS, identification based on GC-IMS database. RI, identification based on RI. Theoretical RI: the values were from PubChem database. Actual RI: RI values identified by GC–MS were calculated refereed to the RI of n-alkanes under the same conditions on HP-50 MS column. RI values identified by GC-IMS were calculated using the RI of the series of ketones on DB-WAX capillary column. *: actual RI and theoretical RI of the compounds obtained by HS-GC-IMS. -: not detected. NF: not found. Aroma type: the aroma type were from https://www.perflavory.com/.

Alcohols are the main VOCs in edible mushrooms, which are mainly formed by the oxidation of polyunsaturated fatty acids (Deng et al., 2023). Alcohols was accounted for 43.69% (AB) to 74.69% (DQ) of the total concentration in the pileus samples，and 42.51% (PZH) to 62.06% (KM) in the stipe samples. Among them, eight-carbon compounds are considered as the key volatile compounds in edible mushroom (Combet et al., 2006). Eight-carbon compounds that have been detected in this study mainly included Phenylethyl alcohol (sweet, floral odor), (*E*)-2-Octen-1-ol (green, citrus odor), 1-Octen-3-ol (mushroom, earthy odor), 3-Octanol (earthy, mushroom odor), 2-Ethylhexan-1-ol (citrus, fresh odor), and 1-Octanol (waxy, green odor). 1-Octen-3-ol, a typical eight-carbon compound in edible mushrooms, has been identified as a potential quality indicator for edible mushrooms (Liu et al., 2021). It is primarily a product of oxidation and cleavage through linoleic acids and linolenic acids in the presence of a range of enzymes (Maggi et al., 2010).

Aldehydes, which are products of lipid oxidation and could provide aroma characteristics of green, fruity and fatty to samples (Li, Wang, et al., 2024). The total content of aldehydes in the pileus samples was ranged from 143.13 μg/kg (LS) to 829.45 μg/kg (CX). While 257.95 (KM) to 1115.73 (CX) was the content range of aldehydes in the stipe. A total of 8 aldehydes were detected, namely Benzaldehyde (fatty, green odor), Phenylacetaldehyde (almond, fruity odor), 2-Octenal (fatty, green odor), 2-Ethylhexanal, Cinnamaldehyde (sweet, spicy odor), 2-Phenyl-2-butenal (musty, floral odor), and Decanal (sweet, aldehydic odor). Commonly, high concentration of aldehydes would result in strong nutty and fatty aroma while turn to green and pleasant odor with the decreased concentration (Feng, Sun, et al., 2022).

The thermal oxidation degradation of unsaturated fatty acids or amino acids is an important pathway for ketone generation (Zhou et al., 2023). Most ketones have a lower threshold, resulting in a greater impact on the flavor of the *Boletus edulis*. Overall, the percentage content of ketones in the pileus samples ranged from 5.57% (DQ) to 17.14% (LS), while in the stipe samples were ranged from 4.36% (PZH) to 11.78% (LS). The large differences in content suggested that ketones were one of the main reasons for flavor differences in different samples. It is worth noting that the threshold of 1-Octen-3-one (earthy, mushroom, vegetable odor) is extremely low and has a greater impact on the overall flavor. It was detected only in DQ stipe (69.01 μg/kg), KM stipe (32.72 μg/kg), and LS pileus (20.59 μg/kg).

Typically, esters are formed from the esterification of alcohols with acids (Chen, Li, et al., 2021). Floral, earthy, waxy and fatty are the main flavor characteristics of esters. The content of esters in the pileus samples was ranged from 60.93 μg/kg (DQ) to 405.95 μg/kg (CX), which accounted for 0.98% to 6.95% of the total flavor compounds in the pileus samples. However, the content of esters in the stipe ranged from 90.02 μg/kg to 1872.52 μg/kg, accounting for 2.94% to 15.72% of the total content of the stipe samples. It can be seen that it was also an important factor in the difference in flavor of *Boletus edulis* from different origins. Among them, the common VOCs of the stipe samples include 2-Ethylhexyl salicylate (orchid, sweet, balsamic odor) and Bis (2-ethylhexyl) adipate. Dibutyl phthalate (faint odor) was a compound common to all samples in the pileus and stipe.

Hydrocarbons were primarily formed by homolysis of alkoxy radicals in fatty acids (Xi et al., 2024). They typically have higher thresholds and make up the overall flavor of the sample by working together with

Table 2
The key VOCs identified in all regions.

Volatile Compounds	Odor description	Aroma type	Odor thresholds (μg/kg)	OAV	
				pileus	stipe
1-Hexanol	Pungent, etherial, oil, fruity, alcoholic	herbal	200	0–0.04	0–0.03
Benzyl alcohol	Sweet, floral, fruity	floral	5500	0–0.03	0–0.03
Phenylethyl alcohol	Sweet, floral, fresh	floral	45	0–9.42	0–25.69
1-Octen-3-ol	Mushroom, earthy, green, oily	earthy	1	38.7–3532.22	32.23–2273.47
3-Octanol	Earthy, mushroom, dairy, musty	earthy	100	0–11.87	0–1.55
2-Ethylhexan-1-ol	Citrus, fresh, floral, oily, sweet	citrus	80	2.82–25.26	0.74–13.6
1-Octanol	Waxy, green, citrus	waxy	54	0–8.79	0–5.27
3-Phenyl-1-propanol	Spicy, cinnamon, fruity, floral	balsamic	1.5	3.69–1386.22	1.04–1110.91
Nonan-1-ol	Fresh, clean, fatty, floral	floral	2	0–9.39	0–11.45
1-Dodecanol	Earthy, soapy, waxy, fatty, honey	waxy	66	2.11–9.75	2.5–11.13
(*E*)-Nerolidol	Floral, green, citrus, woody	floral	300	0–4.88	0–0.57
Heptanal	Fresh, aldehydic, fatty, green	green	31	0–0.75	0.07–0.38
Benzaldehyde	Almond, fruity, powdery, nutty	fruity	300	0.31–1.15	0.36–1.27
Phenylacetaldehyde	Honey, floral, rose, sweet	green	9	2.54–7.04	5.59–21.48
2-Octenal	Fresh, cucumber, fatty, green	fatty	61	0–0.1	0–0.29
Cinnamaldehyde	Sweet, spicy, candy	spicy	500	0–0.19	0–0.28
Decanal	Sweet, aldehydic, orange	aldehydic	5	0–6.08	0–7.83
2-Heptanone	Cheesy, fruity, ketonic, green	cheesy	680	0–0.03	0–0.01
Acetophenone	Sweet, cherry, marzipan	floral	3000	0–0.03	0–0.02
1-Octen-3-one	Earthy, metallic, mushroom	earthy	0.005	0–4118.75	0–13,802.7
3-Octanone	Musty, mushroom, ketonic	herbal	1000	0.01–0.19	0–0.01
2-Decanone	Orange, floral, fatty, peach	floral	9	0–27.07	0–5.23
2-Undecanone	Waxy, fruity, ketonic, fatty	fruity	10	10.15–30.2	8.62–28.17
Geranylacetone	Fresh, green, fruity	floral	10	0–16.63	0–8.95
Ethyl dodecanoate	Sweet, waxy, soapy	waxy	330	0–0.08	0–0.63
Ethyl palmitate	Waxy, fruity, creamy, milky	waxy	1500	0–0.02	0–0.26
Ethyl Linoleate	Mild, fatty, fruity, oily	fatty	4000	0–0.04	0–0.2
2-Methylbutanoic acid	Acidic, fruity, dirty, cheesy	acidic	740	–	0–0.01
Styrene	Sweet, balsamic, floral	balsamic	22	0–1.69	–
2-Methylpyrazine	Nutty, brown, nut	nutty	1000	0.01–0.06	–
2,5-Dimethylpyrazin	Nutty, roasted, potato	chocolate	20	2.32–17.35	0–1.36
3-Ethyl-2,5-dimethylpyrazine	Potato, cocoa, roasted, nutty	nutty	79	0.68–18.22	0–2.98
2,3-Diethyl-5-methylpyrazine	Musty, nut, skin, earthy	musty	0.05	0–1882.61	–
Methional	Musty, potato, tomato, earthy	vegetable	0.04	477.25–4792.49	1097.02–17,343.58
Furfural	Sweet, brown, woody, bready	bready	800	0–0.01	0–0.28
5-Methylfurfural	Sweet, caramellic, bready, brown, coffee	caramellic	6000	–	0–0.01
2-Pentylfuran	Fruity, green, earthy	fruity	4.8	–	0–4.74
(*Z*)-4-Hydroxy-6-dodecenoic acid lactone	Sweet, fatty, waxy, dairy, creamy, fruity	fatty	0.1	0–105.39	0–281.71

other compounds. Hydrocarbons accounted for 0.09% (PZH) to 1.81% (YX) of the total VOCs concentration in pileus samples. While the hydrocarbons proportion in the total concentration of VOCs in the stipe sample ranges from 0 to 4.25%. No hydrocarbons were detected in the CX stipe and PZH stipe samples.

Nitrogen-containing compounds mainly include pyrazine and pyridine compounds. *Boletus edulis* was given nutty, chocolate and roasted flavors through nitrogen-containing compounds. Its content in the pileus ranged from 245.45 μg/kg (LS) to 2304.43 μg/kg (AB), and in the stipe ranged from 0 to 522.57 μg/kg (AB). Similar to hydrocarbons, nitrogen-containing compounds were not detected in the CX stipe and PZH stipe samples. It was not difficult to find that a higher content of nitrogen-containing compounds were detected in the pileus. The common compounds in the pileus samples include 2-Methylpyrazine (nutty, brown, nut odor), 2,5-Dimethylpyrazin (nutty, roasted, potato odor), 1-(6-Methyl-2-pyrazinyl) ethanone (roasted, coffee, cocoa odor), and 3-Ethyl-2,5-dimethylpyrazine (potato, cocoa, roasted odor).

Sulfur-containing compounds are considered to be the most important compounds in shiitake mushrooms according to previous report (Hiraide et al., 2010). It is also widely found in other edible mushrooms. Methional (musty, potato, tomato, earthy odor), with a lower threshold, which was the only sulfur-containing compound identified in this study. Its content was ranged from 19.09 μg/kg (LS) to 191.70 μg/kg (CX) in all pileus samples and from 43.88 μg/kg (KM) to 693.74 μg/kg (CX) in stipe samples, respectively. It was obvious that the stipe samples were rich in Methional.

2-Methylbutanoic acid (acidic, fruity, cheesy odor) was the only one type of acids in our study and was only detected in the DQ stipe and GZ stipe. It might be due to high polarity and low content in our samples, making it difficult to separate and detect them effectively on the HP-5 MS column (Li, Wang, et al., 2024). Other compounds mainly include some heterocycles, such as Furfural (sweet, bready odor), 2,3-Dihydro-3,5-dihydroxy-6-methyl-4 h-pyran-4-one, 5-Methylfurfural (sweet, caramellic odor), 2-Pentylfuran (fruity, green odor), (*Z*)-4-Hydroxy-6-dodecenoic acid lactone (sweet, fatty odor), 2,4-Di-t-butylphenol.

3.3.2. *Cluster analysis*

In order to further analyze the overall flavor differences of *Boletus edulis* from different origins, cluster analysis was performed using the content of VOCs as indicators, and the results were displayed in the heatmaps (Fig. 4C and D). The CX and PZH samples were clustered together in the heatmaps of the pileus and stipe. Interestingly, the same situation was also reflected in the YX and KM samples. It indicated that similar VOCs were appeared in them. In general, all pileus samples were clustered into a large group, with the exception of CX and PZH samples. While the heatmap of stipe samples showed that CX and PZH were clustered together, and GZ, DQ, LS, KM and YX were clustered together.

3.4. *Comparison of abilities of HS-GC-IMS and HS-SPME-GC–MS to identify VOCs of boletus edulis*

As shown in Fig. 5A and 5B, 60 and 56 VOCs were detected in the pileus of *Boletus edulis* by HS-SPME-GC–MS and HS-GC-IMS, respectively. 64 and 63 compounds were detected in the stipe, respectively. In the pileus and stipe samples, all species were detected in greater quantities by HS-SPME-GC–MS, except for acids, ketones and aldehydes,

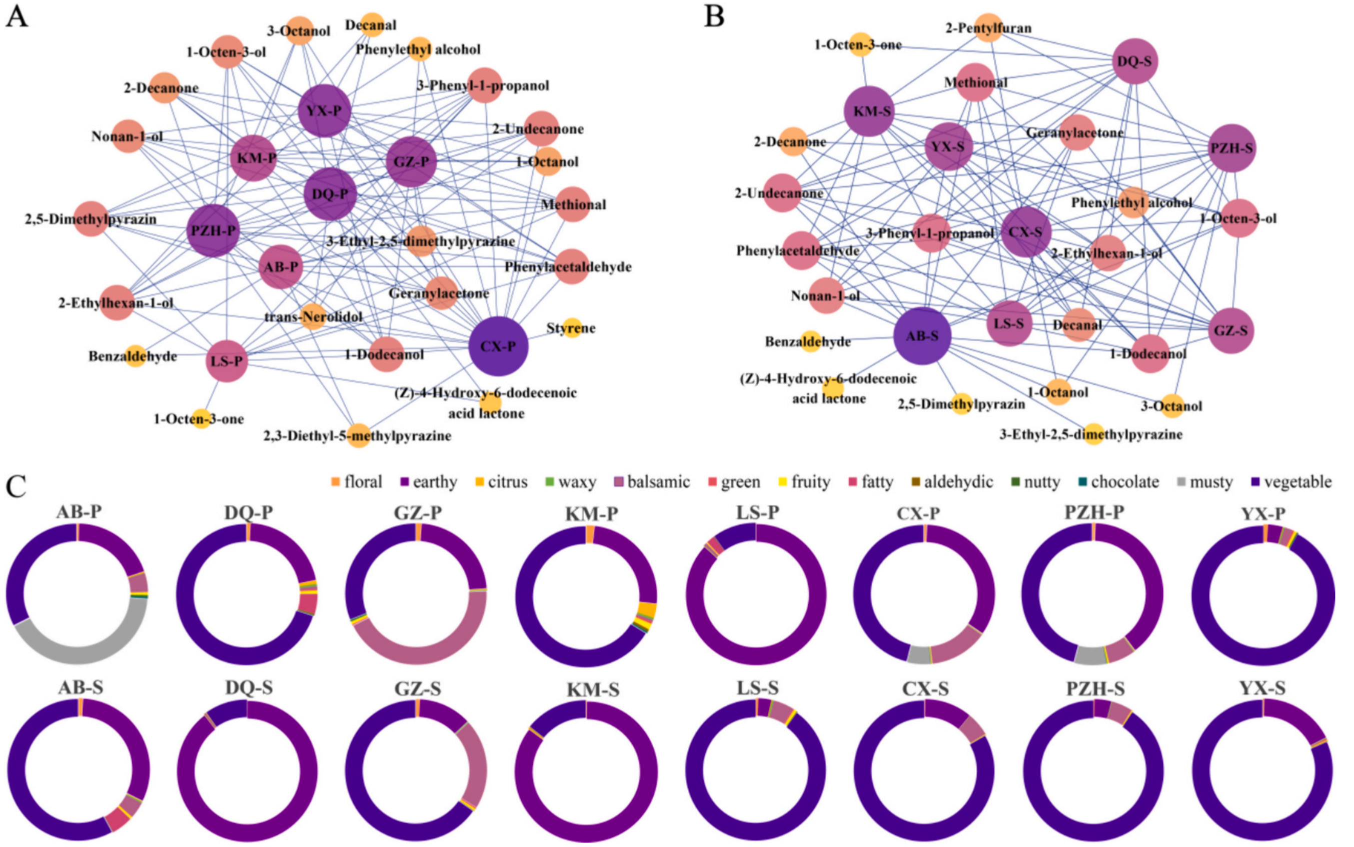

Fig. 6. Network analysis plot of correlation between regions and key VOCs (OAV > 1) of pileus (A) and stipe (B). (C) Odor ring charts of aroma types of pileus and stipe.

which were detected in greater quantities by the HS-GC-IMS. This indicated that acids, ketones and aldehydes were more easily detected by HS-GC-IMS. While alcohols, esters, hydrocarbons, sulfur-containing compounds and nitrogen-containing compounds were more easily detected by HS-SPME-GC–MS. Particular attention should be paid to nitrogen-containing compounds and sulfur-containing compounds, which can only be detected by HS-SPME-GC–MS. Thus, combining the two techniques allows for a more comprehensive detection of VOCs in *Boletus edulis*.

As shown in Fig. 5C, in the results detected by HS-SPME-GC–MS, all categories except acids were clustered on the left side of the biplot. PZH pileus, PZH stipe, CX pileus, CX stipe and AB stipe samples were also clustered on the left side, suggesting a correlation between them and these categories. And the correlation between indicators clustered in the same quadrant was even stronger. Specifically, the first quadrant had no category indicators, but samples AB pileus, DQ pileus, GZ pileus, KM pileus, LS pileus and LS stipe were clustered, indicating that they had similar flavor compositional characteristics. CX pileus and PZH pileus samples and the three categories of alcohols, ketones and nitrogen-containing compounds were clustered in the second quadrant. The third quadrant showed a cluster of CX stipe, PZH stipe , AB stipe samples and five categories, such as esters, hydrocarbons, aldehydes, others and sulfur-containing compounds. The fourth quadrant gathered YX stipe, DQ stipe and GZ stipe samples and acids.

Esters, ketones, aldehydes and alcohols have a significant impact on flavor. As shown in Fig. 5B, esters and alcohols were located in the second quadrant, and nearby samples mainly included the stipe samples of GZ, LS and KM. Ketones and aldehydes were located in the third quadrant, which indicated that the correlation between KM pileus and them was better. In addition, the proximity of pileus samples of GZ, LS, CX, PZH, DQ and YX indicates that they have similar aroma. It is worth noting that there was significant difference in the correlation between the category indexes and samples by use of the two detecting techniques, which suggested that the two techniques have different sensitivities to different kinds of VOCs.

3.5. Key VOCs analysis

The flavor contribution of a compound to a sample is determined by both its content and odor threshold. The ratio of concentration to threshold (OAV), is used to evaluate the contribution of a single VOC to the overall flavor. Table 2 listed the aroma types and odor descriptions of VOCs with OAV > 0.01. Among them, a total of 23 compounds with OAV > 1 can be regarded as key compounds.

The distribution of the 23 key VOCs in each sample were shown in Fig. 6A and B. As shown in Fig. 6A, CX (19), DQ (16), PZH (16) and YX (16) were detected in the largest number of key VOCs in the pileus samples. Among them, 1-Octen-3-ol, 2-Ethylhexan-1-ol, 3-Phenyl-1-propanol, 1-Dodecanol, Phenylacetaldehyde, 2-Undecanone, 2,5-Dimethylpyrazin, Methional were detected in all eight origins. In the stipe samples (Fig. 6B), the largest number of key VOCs was contained in AB (16), followed by KM (13) and CX (13). 1-Octen-3-ol, 3-Phenyl-1-propanol, 1-Dodecanol, Phenylacetaldehyde, 2-Undecanone and Methional were the common flavor compounds in all stipe samples include. It is worth mentioning that 1-Octen-3-ol, 1-Dodecanol, Phenylacetaldehyde, 2-Undecanone, Methional and 3-Phenyl-1-propanol were present in all samples and can be considered as the key characteristic VOCs in *Boletus edulis*.

The odor description were from https://www.perflavory.com/. The odor thresholds were obtained from Van Gemert, 2011 and the medium was water.

3.6. Aroma characteristic analysis

Each VOC has its own unique flavor and aroma type. The aromas produced by the different VOCs combine to create the unique flavor of *Boletus edulis*. A total of 19 aroma types were definition, and all of them were made up the rich and varied aroma of *Boletus edulis*. 13 aroma types with large cumulative of OAVs were selected as indicators to generate odor ring charts (Fig. 6C), which were used to observe the flavor profiles of the pileus and stipe of *Boletus edulis* from different origins.

As shown in Fig. 6C, there were large differences in aroma types reflected between pileus and stipe originated from the same origin. The main aroma types included earthy, balsamic, musty, and vegetable. Among them, vegetable and earthy aroma types were reflected in both pileus and stipe. The balsamic and musty aroma types were unique to the pileus. Both pileus and stipe samples from CX, PZH and YX showed vegetable aroma types, while all of them showed inconsistent aroma types with other samples. In fact, vegetable aroma served as the main aroma type and Methional was the decisive VOC. In addition, 1-Octen-3-ol and 1-Octen-3-one were considered to be the VOCs that played a major role in the earthy aroma type.

4. Conclusion

E-nose technique provided a better description of the overall flavor profile of the pileus and stipe of Boletus edulis, but did not identify the characteristic flavor compounds well, while the deficiency were supplemented by HS-GC-IMS and HS-SPME-GC–MS. The results of the E-nose analysis showed high abundance of nitrogen-containing compounds and sulfur-containing compounds in both pileus and stipe of *Boletus edulis*. Alcohols and aldehydes were the main VOCs in the pileus and stipe according to the results of HS-GC-IMS and HS-SPME-GC–MS analyses. The main aroma types presented in the both pileus and stipe samples was presented as vegetable (DQ pileus, KM pileus, CX pileus, CX stipe, PZH pileus, PZH stipe, YX pileus, YX stipe, AB stipe, GZ stipe and LS stipe) and earthy (LS pileus, DQ stipe, KM stipe), while balsamic (GZ pileus) and musty (AB pileus) aroma types were only present in the pileus. Methional, 1-Octen-3-ol and 1-Octen-3-one were considered to be the main compounds affecting aroma type. Current research on the flavor of *Boletus edulis* mainly focuses on the preservation and processing technology. However, the VOCs and its aroma characteristic of *Boletus edulis* originated from different regions even were not been studied. This research provided a certain scientific basis for the identification of the flavor compounds of *Boletus edulis*, and a comprehensive understanding for the flavor characteristic of the pileus and stipe from *Boletus edulis*. In addition, combining the effects of the climate condition, ecological environment and other factors on flavor of *Boletus edulis*, which may be meaningful to comprehend the intrinsic flavor differences of *Boletus edulis* originated from different regions.

CRediT authorship contribution statement

Weilan Li: Writing – original draft, Investigation, Formal analysis. **Luxi Zi:** Writing – review & editing, Visualization, Formal analysis, Data curation. **Ningmeng Xu:** Methodology, Investigation. **Hao Yang:** Visualization, Methodology, Investigation. **Shihao Dong:** Formal analysis, Data curation. **Fen Qin:** Software, Data curation. **Lei Guo:** Writing – review & editing, Supervision, Software, Resources, Project administration.

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

Data will be made available on request.

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Appendix A. Supplementary data

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