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Uncovering the differences in flavor volatiles of different colored foxtail millets based on gas chromatography-ion migration spectrometry and chemometrics

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ABSTRACT

The differences of volatile organic compounds in commercially available foxtail millets with different colors (black, green, white and yellow) were assayed through gas chromatography-ion migration spectrometry (GC-IMS) to explore their volatile flavor characteristics. Fifty-five volatile components were found in various colored foxtail millets, including 25 kinds of aldehydes (accounting for 39.19–48.69%), 10 ketones (25.36–32.37%), 15 alcohols (20.19–24.11%), 2 ethers (2.29–2.45%), 2 furans (1.49–2.95%) and 1 ester (0.27–0.39%). Aldehydes, alcohols and ketones were the chief volatiles in different colored foxtail millet, followed by furans, esters and ethers. These identified volatile flavor components in various colored foxtail millets obtained by GC-IMS could be well distinguished by principal components and cluster analysis. Meanwhile, a stable prediction model was fitted via partial least squares-discriminant analysis (PLS-DA), in which 17 kinds of differentially volatile components were screened out based on variable importance in projection (VIP>1). These findings might provide certain information for understanding the flavor traits of colored foxtail millets in future.

1. Introduction

Foxtail millet (*Setaria italica*) is a traditionally grown crop and can be adapted to cultivation in diverse environments all over the world, particularly in dry areas of India, China, the United States, and Nigeria (Zhang et al., 2021). Due to its advantages of storage resistance, drought resistance, and strong adaptability, together with abundant nutritional components, the output and yield of *Setaria italica* are increasing in the above-mentioned countries (He et al., 2015; Zhang et al., 2018). After processing to remove the husk, the obtained foxtail millets can be prepared for soups, steamed bread, and other domestic foods, which contain abundant protein, fat, carbohydrates, and micronutrients for the human body (Muthamilarasan et al., 2016; Yang et al., 2017; Bi et al., 2019). Meanwhile, foxtail millet can also be developed and is considered a healthy food source in traditional Chinese medicine, which has a variety of health-promoting functions, such as antioxidation, nourishing blood, promoting lactation, and improving human immunity (Sun et al., 2021; Chen et al., 2016; Xiang et al., 2019).

The color of foxtail millets is a major trait of appearance quality and is commonly used for evaluating their nutritional quality and grade (Li et al., 2021a). Generally, the majority of foxtail millets have a yellow color, but there are also various other colors (such as black, green, white, and gray) for sale in markets (Zhang et al., 2019; Jin et al., 2023; Li et al., 2021a). Volatile components are considered the most significant indicators for distinguishing their price and grade, which are closely related to their freshness and origin and can directly affect the

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Abbreviations: GC-IMS, gas chromatography-ion migration spectrometry; GC-MS, gas chromatograph-mass spectrometry; GC-O-MS, gas chromatograph olfactometry-mass spectrometry; GC-O, gas chromatograph-olfactometry; VOCs, volatile organic components; PCA, principal component analysis; PLS-DA, partial least squares-discriminant analysis; OPLS-DA, orthogonal partial least squares-discriminant analysis; VIP, variable importance in projection; RI, retention index; DT, drift time.

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preferences and purchasing desires of consumers (Zhang et al., 2018; Liu et al., 2017; Jin et al., 2022). Therefore, it is of great importance to investigate and compare the differences of volatile flavor substances of colored foxtail millets either raw or cooked form.

Currently, the detection of food volatile components has gained widespread attention in the food industry. Gas chromatograph-mass spectrometry (GC-MS), gas chromatograph-olfactometry-mass spectrometry (GC-O-MS), electronic nose, and gas chromatography-ion migration spectrometry (GC-IMS), have been employed to analyze the flavor components in various types of foods (Jin et al., 2023a; 2023b; 2023c; Li et al., 2019). Zhang et al. (2018) reported that the flavor compounds of different varieties of foxtail millets were affected by cooking time and pH based on GC-MS. Ye et al. (2022) used gas chromatograph-olfactometry (GC–O) to assess the differences in odor chemicals in millet huangjiu during different fermentation periods.

Compared with GC-MS, the GC-IMS technology showed great merits such as rapidness, high resolution, and visualization through simple sample preparation (Li et al., 2022a,b). Meanwhile, the differential volatile components of different samples can be easily screened through these technologies when combined with chemometrics such as principal component analysis (PCA), partial least squares-discriminant analysis (PLS-DA), orthogonal partial least squares-discriminant analysis (OPLS-DA), etc. (Li et al., 2022a,b; Contrerasdel et al., 2019). To date, the GC-IMS technology has been frequently employed to characterize volatile flavor components of various grain products (rice, foxtail millet, and quinoa, etc.) as affected by varieties, processing methods, and storage conditions. For instances, Song et al. (2021) used GC-IMS combined with PCA to assay differences and fingerprints in flavor organic chemicals of three different colored quinoas. Yang et al. (2021) further demonstrated the volatile flavor profiles of colored quinoas before and after cooking based on GC-IMS and chemometrics. Jin et al. (2022) also determined the differences in flavor volatiles of colored unpolished rice after cooking based on GC-IMS. These research work implied that the volatile flavor profiles of these colored grains (either raw or cooked) could be detected and discriminated by GC-IMS and multivariate statistical analysis.

Recently, several studies have reported differences in colored foxtail millets in cooking qualities (Liang et al., 2018), genetic variation in pigment accumulation (Yang et al., 2017), and metabolic basis (Li et al., 2021b). Volatile flavor is a important feature of foxtail millet that influences consumer preferences. Differently colored foxtail millets displayed versatile nutritional properties. Even though the cooking indices and volatile flavor components of several colored foxtail millets were reported via the GC-MS method (Zhang et al., 2018), there were still certain variations because of detection sensibility, pretreatment procedures, sample varieties, and methods. Recent studies have proved the differences in volatile flavor substances measured by GC-MS and GC-IMS, and both methods can complement of each other (Li et al., 2023; Jin et al., 2023a,b,c). Apart from both methodology studies, research trends point out that the key differential compounds could be screened out based on relative odor value, GC-O, and multivariate statistical analysis (PLS-DA or OPLS-DA), which could better understand the flavor profiles of different food products (Jin et al., 2023). However, identification, fingerprinting, and differential analysis of the whole volatile organic components in different colored foxtail millets by GC-IMS are rarely published.

Herein, differences in volatile organic compounds of different colored foxtail millets (black, green, white, and yellow) in the same geographic location were characterized by GC-IMS to visualize their volatile component fingerprints. Meanwhile, the differential volatile organic components were screened by PCA and PLS-DA, which might provide certain references for enriching the flavor characteristics of foxtail millets with different colors in the future.

2. Materials and methods

2.1. Materials and reagents

The different colored foxtail millets were used in the same geographic location (Qiqihar, China), and their variety names were Longjiang Huang (yellow-colored, with moisture content of 11.1 g/100 g, protein content of 9.31 g/100 g, fat content of 2.4 g/100 g, carbonhydrate content of 75.9 g/100 g, and ash content of 1.3 g/100 g), Longjiang Lv (green-colored, with moisture content of 10.8 g/100 g, protein content of 10.0 g/100 g, fat content of 4.8 g/100 g, carbonhydrate content of 72.2 g/100 g, and ash content of 2.2 g/100 g), Longjiang Bai (white-colored, with moisture content of 11.2 g/100 g, protein content of 7.68 g/100 g, fat content of 2.7 g/100 g, carbonhydrate content of 77.0 g/100 g, and ash content of 1.4 g/100 g), and Longjiang Hei (black-colored, with moisture content of 11.2 g/100 g, protein content of 10.4 g/100 g, fat content of 4.6 g/100 g, carbonhydrate content of 71.6 g/100 g, and ash content of 2.2 g/100 g), respectively. The four colored foxtail millets were purchased from Heilongjiang Yixing Rice Industry (Qiqihar, China) in mid-March 2022 as shown in Fig. 1S. The vacuum-packed samples were stored at 4 °C before use.

Analytical grade *n*-ketones (2-butanone, 2-pentanone, 2-hexanone, 2-heptanone, 2-octanone, and 2-nonanone, with purities above 99%) were bought from Sinopharm Chemical Reagents Co., Ltd (Shanghai, China).

2.2. Volatile organic component detection via GC-IMS

The volatile organic components (VOCs) of different colored foxtail millet were assayed through a GC-IMS method reported previously (Xu et al., 2023) with subtle modification. Briefly, the four foxtail millet samples were ground evenly into flour, precisely weighed (2.0 g) and poured into a 20 mL headspace container, respectively. After keeping them at 65 °C for 12 min, the headspace air (500 μL) were shotted and analyzed upon an MXT-5 pillar through a GC-IMS instrument (FlavourSpec®, Germany). The GC and IMS parameters were the same as our previous report (Jin et al., 2021). Every GC-IMS determination was performed in three separate runs. To prevent cross-contamination, the injector was depressed with force 30 s before each injection, as well as 5 min after each run. Several n-ketones was used as immigrant markers for calculating the retention index (RI) of each organic component. By contrasting RI and the drift time (DT) via the segment database of the GC-IMS device, VOCs were assayed by matching DT and RI to those of the immigrant marker chemicals. The relative ratio of flavor components was correlated with the peak singnal.

2.3. Statistical procedure

All data are reported as mean ± standard deviation of three individual runs. The qualitative procedure of VOCs was carried out by NIST 2014 and IMS databases. The data was also performed one-way analysis of variance and Duncan's multiple tests by SPSS 22.0 Software Package (SPSS, Inc., Chicago, IL, USA), and a level of $p < 0.05$ was considered as significant. The 3D, 2D spectra, and fingerprint map were obtained based on the built-in plug-ins. The relative proportion of various volatile components was estimated according to the peak volume normalization method and the corresponding histogram was drawn by Origin 8.5. Chemometrics were executed through SIMCA 14.1. Cluster analysis and heat-map were visualized through a web tool for visualizing clustering of multivariate data (https://biit.cs.ut.ee/clustvis/).

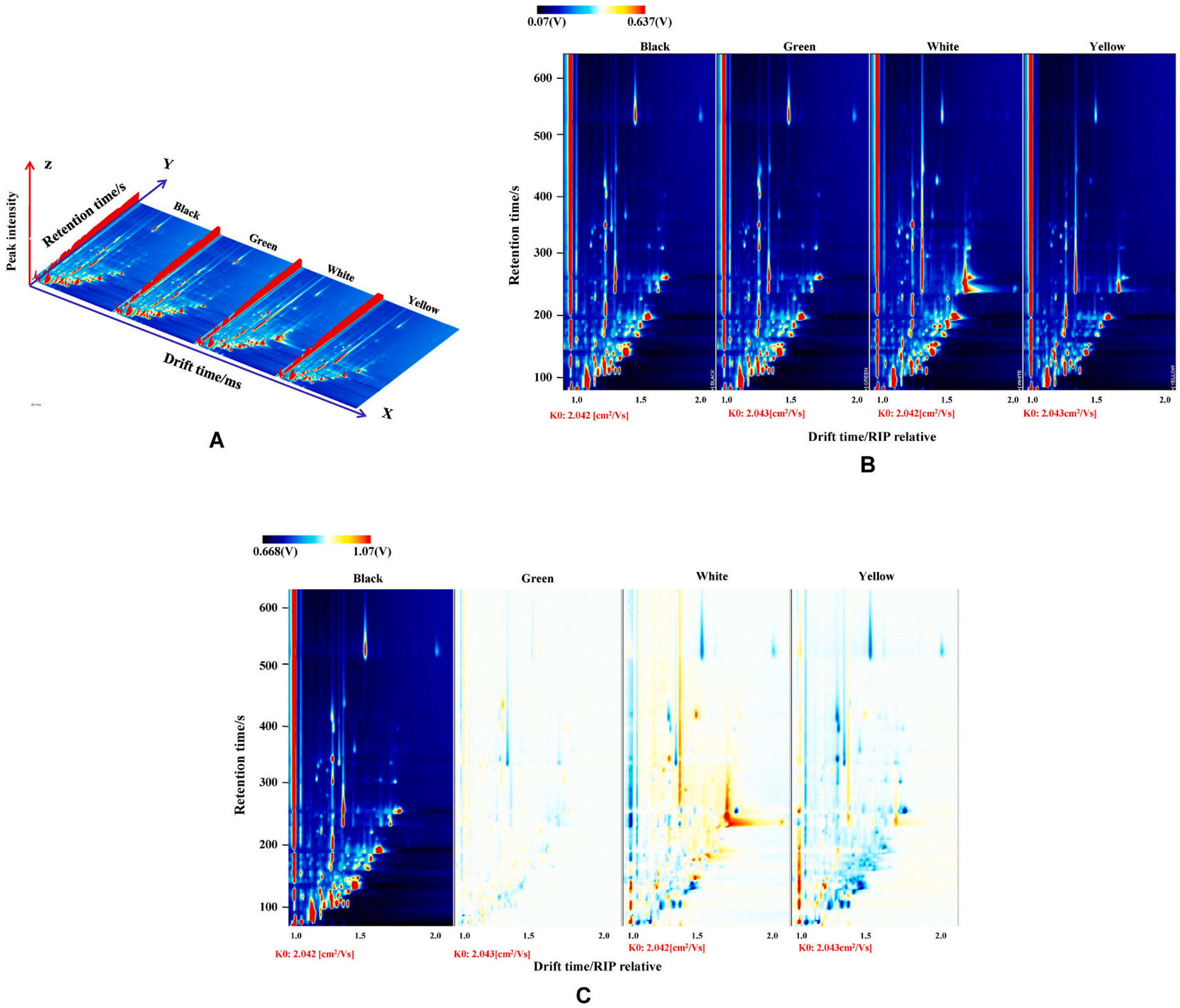

Fig. 1. 3D-topographic (A), airscape maps (B) and contrast maps (C) of GC-IMS in different colored foxtail millet (black, green, white and yellow). The red color indicates the signal intensity and each spot denotes one specific volatile component. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

3. Results and discussion

3.1. GC-IMS maps of various colored foxtail millets

The VOCs in different colored foxtail millet samples (yellow, green, white, and black) were detected by the GC-IMS method. As can be seen from Fig. 1A, a 3D topographic map was exported via the GC-IMS instrumental plug-in. The ion relative drift time, retention time, and peak intensity are represented on the *X*, *Y*, and *Z* axes, respectively. All volatile components were represented as spots on the spectrum, and the color indicated the signal intensity (Yang et al., 2021). For the sake of visually comparing differences in flavor components in various colored foxtail millets, a 2D-topographic plot was obtained in Fig. 1B. Meanwhile, the topographic subtraction plot for comparison was also acquired using black-colored foxtail millets as a reference (Fig. 1C). The volatile substances in four colors of foxtail millets were well distinguished by GC-IMS. The proportion of some flavor organic components in white-colored foxtail millets was more than that in black ones, while most of the volatile substances in yellow-colored foxtail millets were lower. The green-colored foxtail millets possessed volatile component profiles similar to those of black-colored foxtail millets. The relative discrimination for VOCs in four colors of foxtail millets differs in the GC-IMS characteristic spectrum probably caused by the differences in the contents of nutrients such as pigments, lipids, etc. (Tomar et al., 2022; Li et al., 2021a; Zhao et al., 2020).

3.2. Identification of volatile components in different colored foxtail millets

Six *n*-ketones (2-butanone, 2-pentanone, 2-hexanone, 2-heptanone, 2-octanone, and 2-nonanone)

were employed as immigrant references for obtaining the RI index of flavor components by comparing the values of RT and DT. Then, the qualitative analysis of flavor components was achieved by comparison of RI, RT, and DT on the NIST 2014 and GC-IMS libraries (Li et al., 2022a,b). A total of 55 volatile chemicals (containing monomers, and dimers) were identified from 70 signal peaks detected by GC-IMS in the four different colored foxtail millets, including 25 aldehydes, 15 alcohols, 10 ketones, 2 ethers, 2 furans, and 1 ester (Table 1). Table 1 also shows that the most abundant chemicals among colored foxtail millets were acetone (19.9–25.71%), nonanal (3.19–6.34%), ethanol (3.14–5.72%), 2-methylbutanal (2.83–4.56%), and 3-methylbutanal (2.64–4.21%). These volatile categories were similar to those in previous reports on foxtail millets (Zhang et al., 2018; Zhao et al., 2020; Ye et al., 2022), whereas the quantities and relative contents varied, probably owing to analytical methods, geographical origins, varieties, storage conditions, etc.

3.3. Fingerprint profile comparisons in different colored foxtail millets

A gallery fingerprint was generated by the built-in plug-in to visually

Table 1
Volatile compounds recognized from different colored foxtail millets.

No	Chemicals	Retention index	Retention time/s	Drift time/ms	Relative amount/%				Odor description
					Black	Green	White	Yellow	
1	Nonanal-M	1110.7	510.121	1.47566	6.34 ± 0.04^{a}	6.29 ± 0.12^{a}	3.19 ± 0.25^{c}	3.58 ± 0.25^{b}	fat, citrus, green
2	Nonanal-D	1111	510.537	1.94842	0.99 ± 0.03^{a}	0.97 ± 0.01^{a}	0.30 ± 0.05^{b}	0.33 ± 0.04^{b}	fat, citrus, green
3	(*E*)-2-Octenal	1055.2	430.374	1.33125	0.95 ± 0.06^{c}	0.87 ± 0.01^{d}	1.70 ± 0.02^{a}	1.10 ± 0.03^{b}	cucumber, banana, green
4	Benzene Acetaldehyde	1039.7	408.021	1.25375	0.68 ± 0.03^{a}	0.71 ± 0.02^{a}	0.32 ± 0.01^{b}	0.21 ± 0.01^{c}	sweet, chocolate
5	Octanal	1005.7	359.126	1.40452	0.79 ± 0.05^{a}	0.73 ± 0.01^{b}	0.64 ± 0.03^{c}	0.49 ± 0.02^{d}	fat, soap, lemon, green
6	2-Pentyl furan	994.8	344.457	1.25516	2.73 ± 0.1^{b}	2.45 ± 0.01^{c}	4.47 ± 0.03^{a}	1.37 ± 0.03^{d}	green, earthy,waxy
7	(E)-Hept-2-enal-M	953.4	309.183	1.25939	3.27 ± 0.04^{b}	3.82 ± 0.01^{a}	2.80 ± 0.03^{c}	2.31 ± 0.03^{d}	green, fatty, fruity
8	Oct-1-en-3-ol	982.9	334.329	1.16217	0.28 ± 0.02^{c}	0.34 ± 0.01^{b}	0.47 ± 0.01^{a}	0.29 ± 0.02^{c}	mushroom, earthy
9	Benzaldehyde	959.2	314.072	1.15231	0.49 ± 0.02^{b}	0.52 ± 0.02^{a}	0.39 ± 0.01^{d}	0.42 ± 0.01^{c}	almond, burnt sugar
10	Methyl-5-hepten-2-one	991.9	342.013	1.18049	0.13 ± 0.01^{b}	0.14 ± 0.01^{b}	0.17 ± 0.01^{a}	0.12 ± 0.01^{b}	citrus
11	5-Methyl-2-hepten-4-one	973.9	326.645	1.21994	0.24 ± 0.03^{c}	0.21 ± 0.00^{d}	0.56 ± 0.01^{a}	0.47 ± 0.01^{b}	nutty
12	(*E*)-Hept-2-enal-D	954.7	310.265	1.67334	0.61 ± 0.02^{b}	0.81 ± 0.01^{a}	0.82 ± 0.02^{a}	0.27 ± 0.02^{c}	green, fatty, fruity
13	3-Hepten-2-one	929.6	288.858	1.22136	0.14 ± 0.03^{b}	$0.13{\pm}0^{b}$	$0.24{\pm}0^{a}$	0.25 ± 0.01^{a}	creamy, coconut, cheesy
14	Heptanal-D	900.7	264.197	1.69947	2.79 ± 0.05^{b}	3.05 ± 0.02^{a}	1.07 ± 0.02^{c}	0.7 ± 0.02^{d}	fat, citrus, rancid
15	Heptanal-M	901.6	264.971	1.33283	3.19 ± 0.08^{b}	3.21 ± 0.05^{b}	3.03 ± 0.04^{c}	4.04 ± 0.05^{a}	fat, citrus, rancid
16	n-Hexanol-M	870.1	245.1	1.32753	3.13 ± 0.16^{b}	3.04 ± 0.01^{b}	2.52 ± 0.10^{c}	5.13 ± 0.05^{a}	resin, flower, green
17	n-Hexanol-D	870.1	245.1	1.63991	1.77 ± 0.14^{c}	1.49 ± 0.01^{d}	2.88 ± 0.06^{b}	3.50 ± 0.06^{a}	resin, flower, green
18	2-Heptanone-M	891.3	256.455	1.25871	0.73 ± 0.03^{b}	0.79 ± 0.01^{a}	0.41 ± 0.00^{d}	0.5 ± 0.02^{c}	soap
19	2-Heptanone-D	893.2	257.745	1.63461	0.94 ± 0.14^{c}	0.82 ± 0.02^{c}	3.36 ± 0.10^{a}	1.62 ± 0.09^{b}	soap
20	2-n-Butylfuran	892.2	256.971	1.18326	0.22 ± 0.01^{b}	0.18 ± 0.00^{c}	0.32 ± 0.00^{a}	0.12 ± 0.00^{d}	fruity. sweet, spicy
21	(*E*)-2-Hexenal-M	848.5	233.488	1.18326	0.96 ± 0.03^{c}	1.12 ± 0.01^{a}	1.04 ± 0.00^{b}	0.94 ± 0.02^{c}	green, leaf
22	(*E*)-2-Hexenal-D	848	233.23	1.51681	0.6 ± 0.01^{c}	0.75 ± 0.00^{b}	0.96 ± 0.02^{a}	0.32 ± 0.02^{d}	green, leaf
23	2-Methyl-2-pentenal	849.9	234.262	1.15679	0.17 ± 0.01^{a}	0.16 ± 0.01^{a}	0.11 ± 0.01^{b}	0.09 ± 0.01^{c}	green, fruity, alliaceous
24	Hexanal-M	793.6	204.069	1.25474	2.97 ± 0.09^{c}	$3.01 \pm 0.02b^{c}$	3.11 ± 0.02^{b}	4.38 ± 0.09^{a}	grass, tallow, fat
25	Hexanal-D	792.1	203.295	1.56314	8.45 ± 0.03^{b}	8.45 ± 0.03^{b}	8.46 ± 0.02^{b}	8.73 ± 0.23^{a}	grass, tallow, fat
26	Pentan-1-ol-M	767.3	192.198	1.25606	1.56 ± 0.05^{b}	1.49 ± 0.02^{c}	$1.53 \pm 0.03b^{c}$	2.42 ± 0.04^{a}	pungent, fermented
27	Pentan-1-ol-D	767.3	192.198	1.51152	1.43 ± 0.03^{b}	1.51 ± 0.05^{b}	1.79 ± 0.16^{a}	1.68 ± 0.03^{a}	pungent, fermented
28	3-Methyl-2-butenal-M	780	197.359	1.09061	0.17 ± 0.01^{b}	0.18 ± 0.01^{b}	0.09 ± 0.01^{c}	0.20 ± 0.00^{a}	sweet, pungent, nut
29	3-Methyl-2-butenal-D	780	197.359	1.3593	0.32 ± 0.01^{a}	0.33 ± 0.01^{a}	0.13 ± 0.00^{b}	0.14 ± 0.02^{b}	sweet, pungent, nut
30	(*E*)-2-Pentenal-M	750.1	185.231	1.10649	0.23 ± 0.01^{d}	0.28 ± 0.01^{c}	0.33 ± 0.01^{a}	0.30 ± 0.00^{b}	pungent, green
31	(*E*)-2-Pentenal-D	748.2	184.456	1.3593	0.22 ± 0.01^{c}	0.35 ± 0.00^{b}	0.37 ± 0.01^{a}	0.13 ± 0.00^{d}	pungent, green
32	3-Methylbutan-1-ol-M	729.7	176.973	1.24812	0.83 ± 0.02^{c}	0.84 ± 0.01^{c}	0.91 ± 0.02^{b}	1.19 ± 0.07^{a}	fermented
33	3-Methylbutan-1-ol-D	731	177.489	1.49166	1.41 ± 0.04^{b}	1.40 ± 0.02^{b}	1.48 ± 0.02^{a}	1.31 ± 0.04^{c}	fermented
34	2-Methylbutan-1-ol	735.5	179.295	1.23224	0.06 ± 0.00^{b}	0.06 ± 0.01^{b}	0.08 ± 0.00^{a}	0.08 ± 0.00^{a}	fatty, whiskey, leathery
35	2-Butoxyethanol	899.9	263.514	1.20723	0.05 ± 0.00^{b}	0.04 ± 0.00^{c}	0.11 ± 0.01^{a}	0.04 ± 01^{c}	fragrance
36	Pentanal-M	696.3	163.422	1.18659	0.79 ± 0.01^{c}	0.79 ± 0.01^{c}	1.06 ± 0.03^{b}	1.47 ± 0.03^{a}	almond, malt, pungent
37	Pentanal-D	695.6	163.136	1.42642	1.26 ± 0.13^{b}	1.27 ± 0.13^{b}	2.81 ± 0.01^{a}	1.03 ± 0.03^{c}	almond, malt, pungent
38	2-Methylbutanal-M	666.4	154.294	1.16236	0.36 ± 0.02^{d}	0.41 ± 0.02^{c}	0.66 ± 0.02^{b}	1.14 ± 0.01^{a}	cocoa, almond
39	2-Methylbutanal-D	666.4	154.294	1.40052	4.56 ± 0.16^{a}	4.21 ± 0.01^{b}	2.83 ± 0.02^{d}	3.20 ± 0.01^{c}	cocoa, almond
40	3-Methylbutanal	642	147.734	1.41222	4.21 ± 0.12^{a}	4.14 ± 0.01^{a}	2.64 ± 0.01^{b}	2.71 ± 0.02^{b}	malt
41	1-Propanethiol-M	617.7	141.174	1.17239	0.59 ± 0.04^{c}	0.63 ± 0.01^{c}	0.7 ± 0.03^{b}	0.83 ± 0.01^{a}	alliaceous
42	1-Propanethiol-D	621.4	142.172	1.36458	1.35 ± 0.04^{a}	1.26 ± 0.03^{b}	0.91 ± 0.02^{c}	0.79 ± 0.02^{d}	alliaceous
43	2-Butanol	599.2	136.183	1.14648	0.11 ± 0.00^{c}	0.10 ± 0.00^{c}	0.13 ± 0.01^{b}	0.28 ± 0.01^{a}	wine
44	2-Butanone-M	586.5	132.76	1.06125	0.41 ± 0.01^{c}	0.36 ± 0.01^{d}	0.48 ± 0.02^{a}	0.44 ± 0.02^{b}	fruity, camphor
45	2-Butanone-D	589.7	133.616	1.2501	2.64 ± 0.03^{b}	2.20 ± 0.01^{d}	3.57 ± 0.02^{a}	2.41 ± 0.06^{c}	fruity, camphor
46	Tert-butylmethylether	550.6	123.062	1.3579	1.62 ± 0.01^{b}	1.63 ± 0.12^{b}	1.94 ± 0.10^{a}	1.95 ± 0.08^{a}	minty
47	1-Propanol	540	120.21	1.25177	2.28 ± 0.07^{d}	2.45 ± 0.02^{c}	2.95 ± 0.05^{b}	3.32 ± 0.04^{a}	alcohol, pungent
48	Ethanol	450.1	95.984	1.04755	5.64 ± 0.17^{a}	5.72 ± 0.04^{a}	3.56 ± 0.07^{b}	3.14 ± 0.10^{c}	sweet
49	Acetone	486.5	105.793	1.12061	19.97 ± 0.25^{c}	19.9 ± 0.10^{c}	22.69 ± 0.13^{b}	25.77 ± 0.11^{a}	apple, pear
50	Butanal	551.8	123.388	1.28328	2.38 ± 0.05^{a}	2.30 ± 0.03^{b}	1.26 ± 0.03^{c}	0.96 ± 0.02^{a}	pungent, green
51	2,3-butanedione	583.5	131.952	1.1854	0.60 ± 0.04^{b}	0.61 ± 0.01^{b}	0.44 ± 0.00^{c}	0.69 ± 0.03^{a}	sweet, creamy, milky
52	Ethyl Acetate	611.8	139.582	1.34048	0.29 ± 0.01^{c}	0.27 ± 0.01^{d}	0.34 ± 0.00^{b}	0.39 ± 0.01^{a}	pineapple
53	2-Pentanone	688	160.136	1.37012	0.16 ± 0.01^{c}	0.2 ± 0.01^{b}	0.27 ± 0.01^{a}	0.09 ± 0.01^{d}	sweet, wine banana
54	Pent-1-en-3-ol	682.8	158.734	1.34462	0.24 ± 0.01^{b}	0.32 ± 0.01^{a}	0.17 ± 0.01^{c}	0.10 ± 0.01^{d}	green
55	Ethylsulfide	714	170.568	1.04548	0.67 ± 0.09^{a}	0.72 ± 0.02^{a}	0.39 ± 0.03^{c}	0.51 ± 0.05^{b}	–

M and D suffixed after the chemicals indicated monomer and dimer, respectively. Different letters in the same row denote significant difference($p < 0.05$).odor description from: http://www.thegoodscentscompany.com/search2.html.

display the variances of volatile components in four different colors of foxtail millets with 3 parallels (Fig. 2). The volatile chemicals are represented in columns, while samples are represented in rows. Higher contents of volatile components were detected when the red areas occupied larger areas (Chen et al., 2021; Jin et al., 2022). As demonstrated in Fig. 2, the contents of characteristic flavor components in black-colored foxtail millets and green-colored foxtail millets such as 2-methyl-2-pentenal, benzene acetaldehyde, butanal, octanal, nonanal, 3-methylbutanal, heptanal, 1-propanethiol, ethyl sulfide, 3-methyl-2-butenal, ethanol, 2,3-butanedione, benzaldehyde, 2-heptanone, acetone, 1-propanol and tert-butylmethylether, were relatively higher and showed minor differences. Most of aldehydes in raw grains or cooked cereals might be produced by linoleic acid oxidation during sample heating process, but could contribute differently to aroma properties of cereals (Fan et al., 2021; Jin et al., 2023a,b,c). The characteristic volatile components in white-colored foxtail millets were quite different from those in black-colored foxtail millets and green-colored foxtail millets, as the contents of (*E*)-2-hept-2-enal, pentanal, 2-butanone, tert-butyl-methylether, 2-methylbutan-1-ol, 3-hepten-2-one, 2-pentyl furan, 2-pentanone, and pentan-1-ol were relatively higher. Ketones (2-butanone, 3-hepten-2-one and 2-pentanone) and alcohols (2-methylbutan-1-ol, pentan-1-ol) could also come from the oxidative dissociation of fatty acids, and their threshold values were greater than that of aldehydes (Zhang et al., 2018; Yang et al., 2021). Compared with the other three colored foxtail millets, there were fewer characteristic volatile components in yellow-colored foxtail millets, including 2-butanol, 2-methylbutan-1-ol, hexanol, 1-propanol, acetone, pentanal, pentan-1-ol, and 3-methylbutan-1-ol. The characteristic flavor substances of different colored foxtail millets, which may have a greater contribution to their overall flavor differences were well characterized by the gallery fingerprint. A similar gallery fingerprint of several quinoas with different colors through the GC-IMS technique was also reported by Song et al. (2021).

To better illustrate the variations in colored foxtail millets, the peak volumes of various organic components on the fingerprint were normalized to obtain their relative proportions of volatile components (Fig. 2S). The results indicated that the four kinds of foxtail millets were composed of aldehydes, ketones, alcohols, esters, ethers, and furans, accounting for 39.19–48.69%, 25.36–32.37%, 20.19–24.11%, 2.29–2.45%, 1.49–2.95% and 0.27–0.39%, respectively. The prevailing volatile categories in different colored foxtail millets were aldehydes, ketones, and alcohols, respectively. The proportion of aldehydes were the highest in black and green-colored foxtail millets, reaching 47.78% and 48.69%, respectively, while the proportion of ketones were greater in white and yellow-colored foxtail millets, at 32.19% and 32.36%, respectively.

Aldehydes have been noted as major flavor components in grains, which are chiefly manufactured by fatty acids oxidation during the storage and processing processes (Liu et al., 2012; Wang et al., 2014; Liu et al., 2017). At low concentrations, aldehydes smell like grass and fruit, but have an old and off-odor smell at high concentrations. The most abundant aldehyde in colored foxtail millets was nonanal, which possesses the characteristic odors of fat, citrus, and grass, followed by 2-methylbutanal, which smells like cocoa and almond, and 3-methylbutanal, which smells like malt (Fig. 2S and Table 1). Ketones and alcohols chiefly originate from the oxidative dissociation of lipids, and their threshold values were greater than that of aldehydes, possessing a certain floral and fruity fragrance. The most abundant ketones and alcohols in colored foxtail millets were acetone and ethanol (Table 1), respectively. Esters were primarily the commodities after chemical reactions between acids and alcohols, which could enhance the whole volatile profiles of various grains (Zhang et al., 2018; Bi et al., 2019; Jin et al., 2022).

A previous study employed GC-MS to assay the odor chemicals in 13 commercially available foxtail millet samples, and 52 volatiles were detected, containing 19 aldehydes (62.88–81.6%), 5 alcohols (1.06–6.73%), 10 ketones (3.07–6.56%), 9 hydrocarbons (4.33–11.59%), 6 benzene derivatives (1.14–3.92%), and 3 others (6.63–15.26%) (Liu et al., 2017). These kinds of volatile components were similar to those in the present study, yet the number and the proportion of various kinds of volatiles were greatly different, probably due to the variations in detection methods and foxtail millet varieties (Bi et al., 2019; Zhang et al., 2018; Li et al., 2022a,b). A large number of studies have proven that the GC-IMS is more effective than the common SPME-GC-MS, particularly for detecting trace volatile substances (Li et al., 2022a,b; Li et al., 2023), so the two complementary technologies can work together to provide the whole flavor profiles in foods. As it is still difficult to determine which foxtail millets possess better flavor in present results, more work seem indispensable to determine the key volatile flavor components and odor relative value of colored foxtail millets by GC-MS, GC-O, and sensory evaluation in the future.

3.4. Similarity comparison of volatile components via PCA and cluster

PCA and cluster models were used to analyze the difficult-to-find and complex variables and distinguish differences in volatile components in different sorghum cultivars (Fan et al., 2021), and colored unpolished rice (Jin et al., 2022). Currently, the whole GC-IMS spectrum data of the four colored foxtail millets were performed via PCA and cluster comparison, and the results were demonstrated in Fig. 3. It presented that

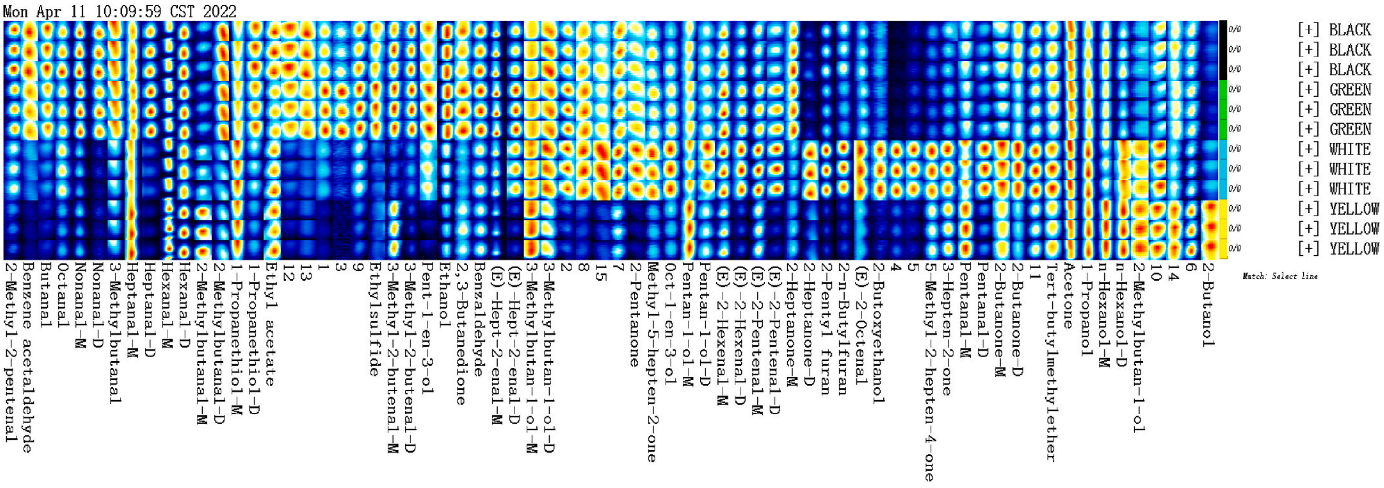

Fig. 2. Fingerprint of flavor organic chemicals in four colored foxtail millets.

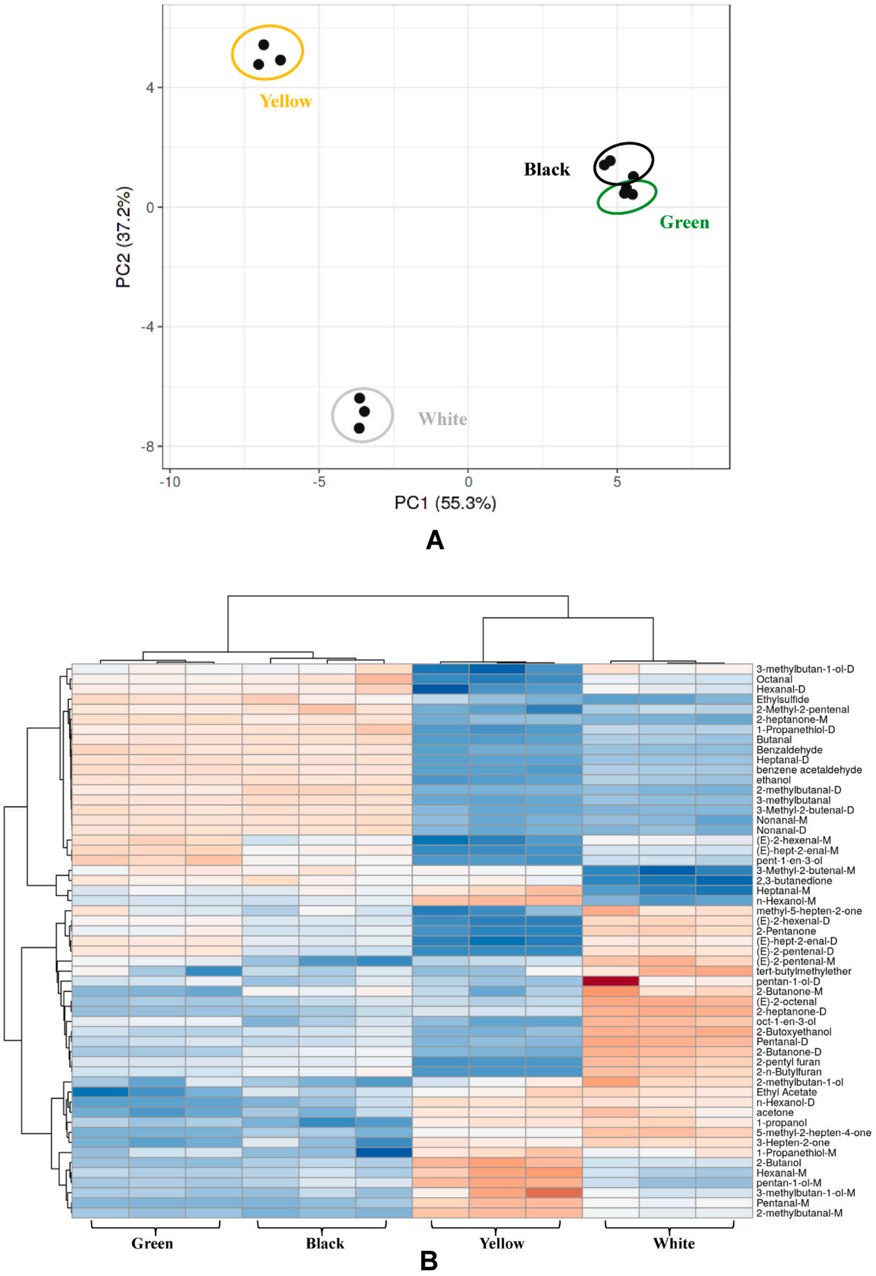

Fig. 3. Comparison of volatile components in different colored foxtail millet by PCA score plot (A) and clustering heatmap (B).

PC1 and PC2 explained by 55.3% and 37.2% of the variance, respectively, with a cumulative contribution rate of 92.5% (Fig. 3A), basically covering the majority of all sample characteristic information. The volatile components of foxtail millets with the same color were relatively clustered together, in which black-colored foxtail millets and green-colored foxtail millets were closer, and were far away from white- and yellow-colored foxtail millets. These findings further confirmed the differences in volatile components in different colored foxtail millets. Fig. 3B shows the clustering heatmap of four colored foxtail millets. Generally, the four colored foxtail millets were clustered into three kinds, namely black- and green-colored foxtail millets (higher similarity), white-colored foxtail millets, and yellow-colored foxtail millets, respectively, which was in agreement with the PCA score plot (Fig. 3A). Thus, the volatile organic compounds of the four colored foxtail millets can be well distinguished by PCA and cluster analysis. Previous work also showed the discrimination effect of flavor volatiles of colored millet porridges based on GC-IMS and PCA (Jin et al., 2023). Another similar results of three quinoas with disparate kernel colors via GC-IMS-based volatile profiling and chemometrics was also reported by Song et al. (2021).

3.5. Multivariate statistical analysis by PLS-DA with cross-validation

PLS-DA is an effective discriminant statistical mining procedure which can establish the interaction model between variable signals and sample classification (Wang, Rogers, Li, Yang, Chen and Zhou, 2019a). R^2X and R^2Y denote the explanation percentage of the fitting equation of X and Y matrices, respectively, whereas Q^2 exhibits the forecast capacity of the fitting equation. Two coefficients of R^2 and Q^2 exceeding 0.5, and closer to 1.0 were considered as accurate results (Dou et al., 2022). In the present model (Fig. 4), most of the findings about the flavor compounds within different colored foxtail millets were covered by the fitting equation with forecast capacity Q^2 (cum) = 0.967, goodness-of-fit parameter R^2X(cum) = 0.961, and explanatory ability R^2Y(cum) =

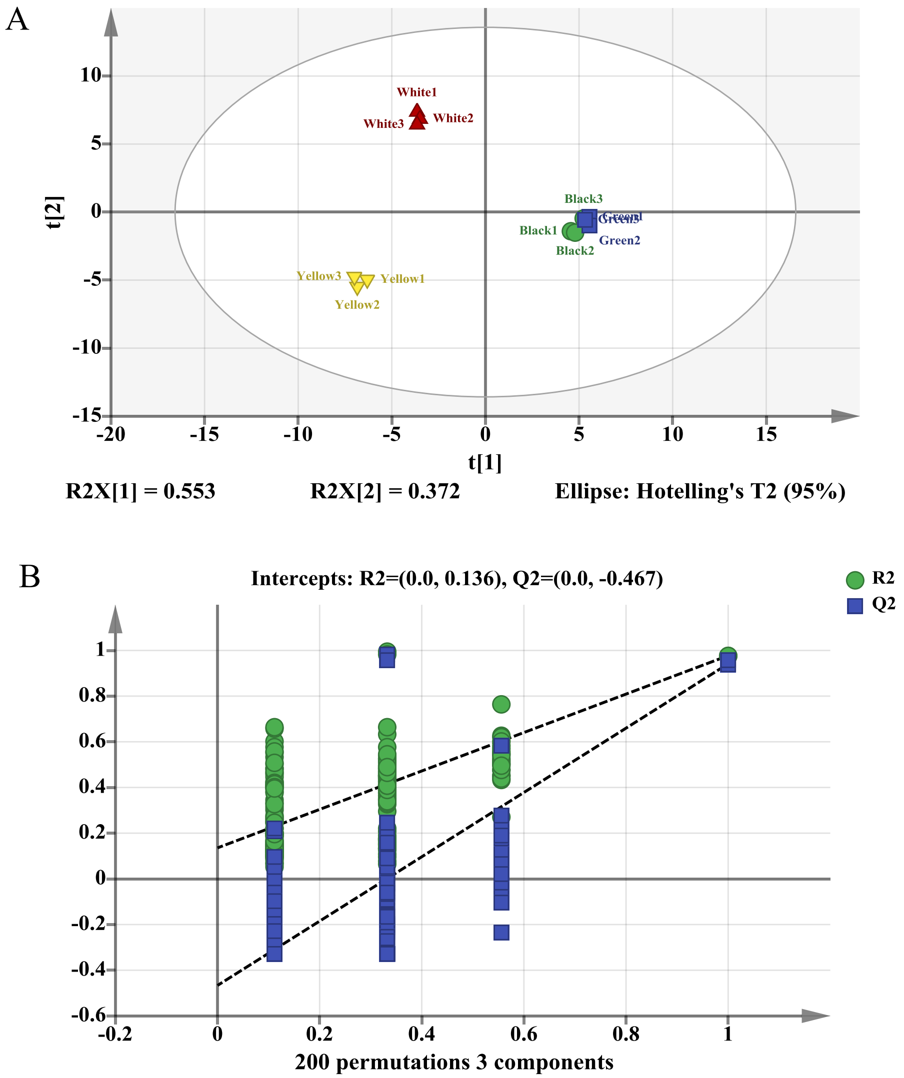

Fig. 4. PLS-DA analysis (A) and cross-validation by a permutation test (B) of volatile components in foxtail millet with different colors. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

0.987. Most colored foxtail millet samples could be well distinguished on the PLS-DA scatter plot (Fig. 4A), and the classification effect was similar with the PCA scatter plot (Fig. 3A). To avoid over-fitting, the reliability of the PLS-DA model was verified by a displacement test. The replacement test results are shown in Fig. 4B. After 200 cross validations, the regression line of model Q^2 crosses the abscissa, and the intercept value is negative (−0.467). All replacement tests R^2 and Q^2 are lower than the original values, indicating that the model is not over-fitted, and the constructed PLS-DA model is stable and reliable. Yang et al. (2021) also obtained similar OPLS-DA model of volatile profiles of several colored quinoas before and after cooking based on the GC-IMS profiling.

3.6. *Screening of differential volatile organic components in different colored foxtail millets*

After visualization of 55 volatile components in different colored foxtail millets by the GC-IMS fingerprint, the impact of each component for classification was quantified according to VIP (variable importance in projection) calculated by the construction of a reliable PLS-DA simulation. Then, the volatile chemicals with VIP value > 1 were strung out as the differential marker components in different samples (Zhang et al., 2022; Li et al., 2022a,b). There were 17 volatile organic compounds identified from the different colored foxtail millets (VIP value > 1), including 2-butanone-M, (*E*)-2-pentenal-M, (*E*)-2-pentenal-D, 2-butanone-D, (*E*)-2-hexenal-M, ethyl acetate, 2-n-butylfuran, 1-propanethiol, oct-1-en-3-ol, pent-1-en-3-ol, (*E*)-hept-2-enal-D, octanal, (*E*)-2-hexenal-D, 2-pentyl furan, 1-propanol, 2-butoxyethanol, and 2-pentanone (Fig. 5A). 2-butanone-M showed the highest VIP value (1.76), followed by (*E*)-2-pentenal, 2-butanone-D, (*E*)-2-hexenal-M, and ethyl acetate. 2-butanone-M, the highest relative proportion in white-colored foxtail millets, has a smell of fruity and camphor, and previous research showed that it might be produced by lipid oxidation (Yang et al., 2021). (*E*)-2-pentenal and (*E*)-2-hexenal may be released from the fatty acid oxidation, giving foxtail millets with the smell of green, fatty, and fruity (Table 1). Ethyl acetate has a smell of pineapple, abundant in yellow- and white-colored foxtail millets, which might be produced through chemical reactions between acids and alcohols in cereals (Bi et al., 2019). Li et al. (2021a) reported that 18 marker flavor volatiles were screened out from different colored foxtail millets (not the same geographic location) based on simultaneous distillation extraction-gas chromatography mass spectrometry and multivariate statistical analysis (PLS-DA and VIP above 1), these marker volatiles are different from the present results probably owing to raw materials,

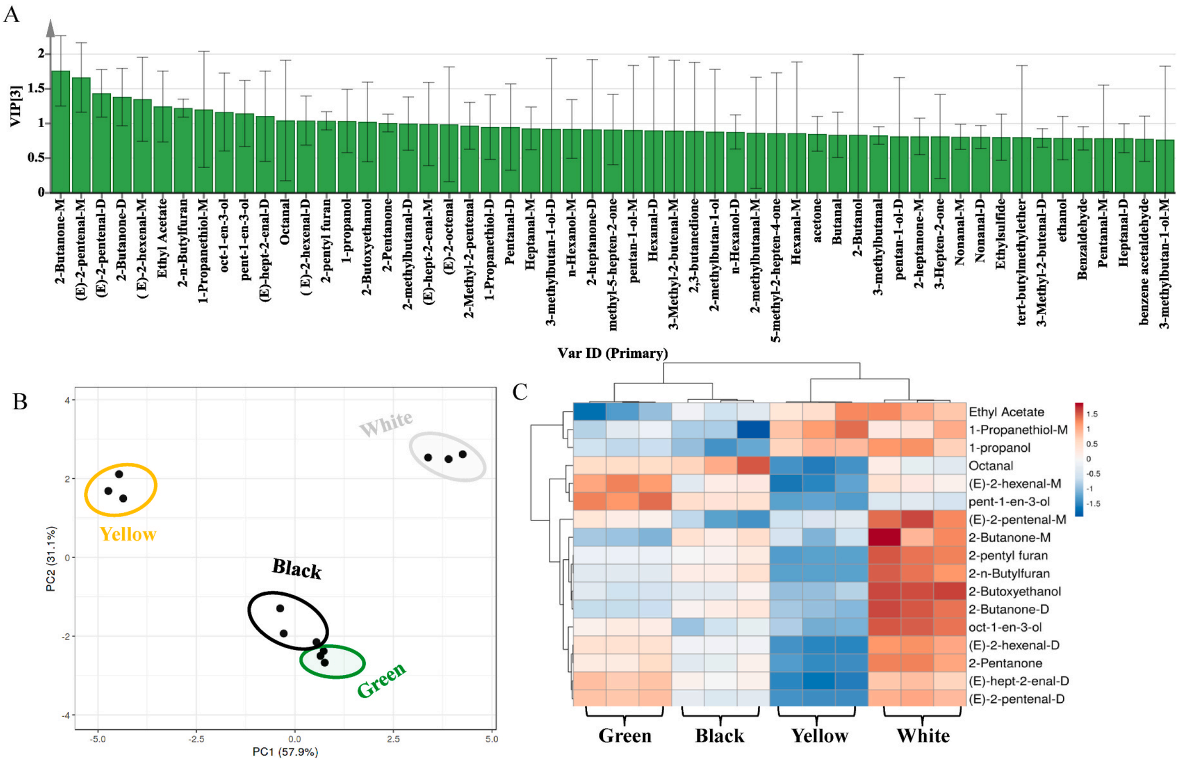

Fig. 5. Screening of differential volatile components in different colored foxtail millet (A) VIP value. (B) PCA score plot. (C) clustering heatmap.

detection methods and conditions.

PCA and heatmap clustering analysis were also performed based on the 17 differential volatile compounds (Fig. 5B and C). Most of the differences in samples can be discriminated by PCA with a sum ratio of 89% (the first two components were 57.9% and 31.1%, respectively). The results of heatmap clustering also showed that the 17 kinds of labeled volatile components in different colored foxtail millets could better classify the differences in samples. Wang et al. (2019b) also found that the differential marked volatile components screened by the OPLS-DA combined with VIP>1 could well distinguish two honey species. Another similar study on screening differential marker volatile compounds (OPLS-DA combined with VIP>1) from pigmented rice after puffing was also reported (Jin et al., 2023), whereas these studies lack the verification tests of the marker compounds. Although the differences in different colored foxtail millets can be well distinguished by these 17 marker volatiles combined with PCA and cluster analysis, future work should be performed to verify the feasibility and accuracy of these marker volatiles in practical products, together with their aroma characteristics though GC-O data.

4. Conclusions

To sum up, a total of 55 volatile organic chemicals were detected in four different colored foxtail millets by GC-IMS, including 25 aldehydes, 10 ketones, 15 alcohols, 2 ethers, 2 furans and 1 ester. The prevailing volatile components in different colored foxtail millets were aldehydes, ketones and alcohols, respectively. The differences in volatile components in different colored foxtail millets might be well recognized via GC-IMS data together with PCA and heatmap clustering analysis. A better stability prediction model was established through PLS-DA, and 17 differential volatile components were screened out as volatile markers for distinguishment of the four colored foxtail millets. Based on GC-IMS technology, the fingerprint of volatile organic compounds in four colored foxtail millet was established, which might provide certain reference for enriching the flavor, and quality characteristics of colored foxtail millets in the future.

CRediT authorship contribution statement

Wengang Jin: Investigation, Methodology, Visualization, Project administration, Funding acquisition. **Wenqiang Cai:** Methodology, Data curation, Visualization. **Shibo Zhao:** Methodology, Data curation, Visualization. **Ruichang Gao:** Writing – review & editing. **Pengfei Jiang:** Supervision, Resources, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financialinterestsor 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

Supplementary data to this article can be found online at https://doi.org/10.1016/j.crfs.2023.100585.

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