Characteristic GC-IMS fingerprints of volatile flavour compounds in Hunan flavour leisure-dried bean curds

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ABSTRACT: Volatile flavour compounds are essential components that affect the product's overall flavour in the Hunan flavour leisure-dried bean curd. This study analysed the characteristic volatile compounds of different leisure-dried bean curd brands using the combination of gas chromatography-ion mobility spectrometry (GC-IMS) and principal component analysis (PCA). The fingerprints of four flavour leisure-dried bean curd brands (A1, A2, A3 and A4) were constructed, wherein 98 monomers and dimers of specific volatile compounds were detected. The standard volatile flavour compounds of the four brands, mainly include aldehydes, ketones and alkenes. Brand A1 primarily consists of aldehydes, such as nonanal, butyraldehyde and octanal, unlike other brands. It accounts for 23% of all volatile compounds. Brands A2, A3 and A4 mainly comprise ketones, accounting for 32%, 30% and 23% of all the volatile compounds, respectively, with the most significant contribution to the flavour threshold. The PCA could identify the four brands and demonstrated that the content of flavour substances from different brands created their unique flavour. These findings suggest that differences in leisure-dried bean flavour depend on different brining processes. The establishment of flavour fingerprints of leisure bean products could be used for quality control, shelf life monitoring, origin traceability and brand identification.

KEYWORDS: gas chromatography-ion mobility spectrometry (GC-IMS), leisure-dried bean curd, volatile compounds, odour activity value, electronic nose, flavour compounds

INTRODUCTION

Soybean is an annual herb that belongs to the *Leguminosae* genus, which has a cultivation history of 5000 years in China. Soybean is rich in protein, carbohydrates, lipids, potassium, phosphorus, calcium, vitamins, isoflavones, saponins, sterols, phospholipids and other bioactive substances [1]. Therefore, soybean products promote intestinal health, prevent cancer, reduce cholesterol, glycemia and blood lipids, strengthen the brain, and improve intelligence [2]. Due to their high nutritional value and therapeutic properties, soybean products play a vital role in global food consumption [3]. Since the Zhou Dynasty in ancient China, many products have been derived from soybeans, such as Douchi and tofu, after thousands of years of cooking. Tofu was created by adding coagulants to soybean milk. Following dehydration and processing, tofu can be made into dried bean curd and then marinated into leisure-dried bean curd. This method prolongs the tofu's storage time and adds more flavour [4]. The processing industry of soybean products in China and the processing of leisure-dried bean curd have a long history. Leisure-dried bean curd has rich nutrition, good flavour and is easy to eat, digest and absorb. It is favoured by people and regarded as health-promoting leisure food [5]. However, many research works on leisure-dried bean curd mostly focussed on optimisation or new product research. Hence, little attention has been paid to detecting and comparing the flavour of leisure-dried bean curd brands.

The flavour is crucial for evaluating food sensory quality, which directly affects the choice of consumers and holds substantial economic value. Because the changes in food flavour substances are closely related to the alterations in their internal chemical composition and nutritional values [6, 7], the fingerprints of volatile compounds can encourage the development of the food industry and scientific research [8]. At present, the technologies for detecting food flavour components include the electronic nose, gas chromatography-mass spectrometry (GC-MS) and the recent gas chromatography-ion mobility chromatography (GC-IMS) [9]. GC-MS is the preferred volatile compounds analysis technology that is widely used in the food industry [10] and has high detection sensitivity [11]. Jiang et al [12] used electronic nose and GC-MS to analyse the volatile components in beer-containing *Cordyceps sinensis* extract. It is known that GC-MS cannot perform complex pre-processing and has a long detection time [13], while GC-IMS combines the high separation capacity of GC. Apparently, the fast response of GC-IMS became popular in food flavour analysis. It can investigate trace amounts and semi-volatile compounds in foods. Moreover, it has a fast detection speed, small sample demand and excellent reproducibility [14]. The IMS instrument generates a quick response and has a high sensitivity towards ions under normal pressure according to the mobility of neutral buffer gas in the electric field. It combines the

high-efficiency separation of gas chromatography with the advantage of trace rapid analysis of the ion mobility spectrum. The IMS instrument also obtains the three-dimensional spectra of retention time, drift time and signal strength after secondary separation [15, 16]. Recently, researchers have frequently used GC-IMS in food flavour detection. Yang et al [17] used GC-IMS to analyse the change in 47 volatile components of the jujube fruit during cold storage and determined that the volatile compounds were primarily alcohols, aldehydes esters, and ketones. They found that the characteristic volatile compound to distinguish fresh fruits was 3-pentanone. Pu et al [18] applied GC-IMS technology to analyse the volatile aroma compounds released from bread during chewing. They characterised the aroma perception signals of bread while not chewing and swallowing. GC-IMS can also be used to examine other food flavours, such as indica rice [19], tofu [20] and white apricot [21]. In this study, GC-IMS was used to determine the flavour of leisure-dried bean curds, which is highly significant in improving manufacturing process and product shelf life monitoring.

MATERIALS AND METHODS

Materials

The four brands (A1, A2, A3 and A4) of Hunan flavour leisure-dried bean curd were purchased from a local Walmart market in Shaoyang, China and stored at 4 °C. The A1, A2, A3, A4 brands were produced by Hunan Gongbing Food Co., Ltd., Hunan Manshifu Food Co., Ltd., Wugang Shuangquan Food Co., Ltd., and Hunan Xiangxiangzui Food Co., Ltd., respectively. All the chemical reagents used had chromatographic purity and were purchased from Shaoyang Keyi Chemical Glass Co., Ltd., (Shaoyang, China).

GC-IMS analysis of volatile compounds

Volatile compounds in different leisure-dried bean curd brands were analysed using a GC-IMS instrument Flavour Spec® (G.M.S., Germany). A standard curve was established using C4-C9 n-ketones as an external reference for calculating retention indices (RI), which were used for detecting volatility under the same chromatographic conditions. Volatile compounds were identified by comparing the RI and drift time (DT) of standard compounds in the National Institute of Standards and Technology (NIST) database and the GC-IMS database [10].

All samples were prepared as follows: 2.0 g of each brand were cut from the middle of the bean curd and placed in a 20-ml head-space glass bottle. The sample was incubated at 45 °C for 15 min under rotation at 500 rpm. Following incubation, 500 µl of head-space was injected with an air-tight syringe at 45 °C into the column at 60 °C. Each sample was tested thrice.

GC-IMS detection conditions: capillary column (MXT-5, 15 ml, 0.53 mm ID, 1 µm of FT equipped with an auto-sampler device (CTC, Switzerland)); the column temperature was set at 60 °C, the analysis time was 30 min; the carrier gas was N_2 (purity ⩾ 99.999%). The IMS temperature was set to 45 °C. Carrier gas flow programme: initial flow rate of 2 ml/min, first held for 2 min and then linearly increased to 10 ml/min within 10 min. Thereafter, it was linearly increased to 100 ml/min within 20 min. The drift gas flow speed was 150 ml/min.

Evaluation of odour activity value

The odour activity value (OAV) was used to evaluate the flavour contribution of each aroma component. When OAV > 1, it is defined as a key flavour component that directly affects the overall flavour. When 0.1 < OAV < 1, it is defined as a modifying flavour component that modifies the overall flavour. When OAV < 0.1, it is defined as a potential flavour component which exerts no significant impact on the overall flavour [22, 23].

Analysis of electronic nose

The INOSE electronic nose instrument (Shanghai Ruifen International Trade Co., Ltd.) was used to analyse the volatile components of different bean curd brands. The measurement was repeated thrice for each sample. The detection parameters: a sampling time of 120 s; the carrier gas flow rate of 0.5 l/min; and the waiting time of 10 s. The specific operations are as follows. The leisure-dried bean curd was cut into dices, mixed, and 5 g was weighed as the measurement sample, which was then placed in a 50-ml head-space sample bottle and kept at room temperature (25 °C) for 30 min. The sampling probe of INOSE extracted the volatile components from the head-space sample bottle for detection.

Statistical analysis

The qualitative and quantitative analysis software vocally supporting the instrument was applied to identify the compounds. The built-in NIST database and IMS (German G.M.S.-IMS) database were used to qualitatively analyse volatile substances. Laboratory Analytical Viewer (LAV) was used to plot the quantitative analytical spectra (3D spectrograms). The Reporter plug-in was applied to directly compare the differences in spectra between samples in the 2D top view. The Gallery Plot plug-in visually compared the differences in volatile organic compounds between fingerprint samples. The Gallery Plot plug-in was used to compare the fingerprint profiles. The Dynamic PCA plug-in was applied to perform dynamic PCA and phase identity analysis on the volatiles of the four leisure-dried bean curd brands. The Excel software was used to process the data. All determinations were made in triplicate, and all the data were presented as the mean ± SD. The data results were plotted by Origin software.

RESULTS AND DISCUSSION

GC-IMS spectra of different brands of Hunan flavour leisure-dried bean curds

The volatile compounds of four dried bean curd samples were analysed using GC-IMS to create a three-dimensional graphical infographic. The GC-IMS two-dimensional spectrum (top view) of the samples was determined after performing the dimensionality reduction process (Fig. 1A). The vertical coordinate of the graph is the retention time of GC, the horizontal coordinate is the ion migration time and the whole background of the spectrum is blue. The red vertical line at the horizontal coordinate 1.0 is the reactive ion peak (RIP peak). Each point on both sides of the RIP peak represents a volatile organic compound. The colour represents the concentration of the substance: white indicates a low concentration, and red indicates a high concentration. The darker the colour, the higher the concentration. The GC separation of volatile components in the dried bean curd can be achieved within 30 min, and the IMS unit performs a good separation. The volatile substances of different bean curd brands are more complex, and the types of compounds detected are approximately the same. However, the content of volatile components of different leisure dried bean curd brands is apparently different. The variability of their content exerts a decisive effect on the flavour of leisure-dried bean curd.

Comparison of GC-IMS two-dimensional maps of four different brands

To compare the differences in volatile components among the four dried bean curd samples, the topographic map of brand A1 was selected as a reference. The topographic map of the other three brands (A2, A3, A4) was deduced from brand A1's topographic map (Fig. 1B). Therefore, if the volatile compounds were consistent, the background after deduction was white. Meanwhile, a red background represents that the concentration of the substance in the sample was higher than the reference sample, and the blue background represents that the concentration was lower than the reference sample [24, 25]. In the topographic maps of the four dried-bean curd samples, the retention time of most of the signals was between 100 and 1200 s. A large number of signals could be identified in brand A4, which had the most volatile components and the highest concentration. The results demonstrated that the volatility of four different brands could be better separated by the GC-IMS technique. Different brands presented some differences in the GC-IMS characteristic spectral information. The content of some volatile components appeared to be higher or lower, showing more obvious differences. The volatile content of brands A2 and A3 were close to each other and exhibited a similar increase or decrease in the volatile content compared with brand A1. Among the four different brands, brand A4 had the highest signal intensity with the largest amount of volatile compounds.

Comparison and analysis of common volatile compounds in different brands of leisure-dried bean curd

The GC-IMS Library Search software was used to match and characterise the volatile compounds based on the GC retention time and IMS migration time of volatile organics in the samples. This software was also used to determine the volatile component monomers and dimerisation of some substances. The qualitative and quantitative analyses of the identified compounds were performed (Table S1), wherein 98 volatile compounds (including dimers) were identified from four dried-bean curd samples. The volatile compounds comprised of 26 aldehydes, 20 ketones, 12 alcohols, 11 alkenes, 6 esters, 6 pyrazines, 3 furans, 2 acids, 2 thiazoles, 1 ether and 9 others. Then, volatile compounds of each brand were compared (Fig. 2). The content of aldehydes in brand A1 was the highest, followed by ketones and alkenes. The content of thiazoles and acids was the lowest, accounting for 0.25% and 0.22% of the total volatile compounds, respectively. Among the volatile compounds of brand A2, the content of ketones was the highest, and the content of acids was the lowest, accounting for 0.19% of the total compounds. Brand A3 had the highest content of ketones and the lowest content of acids, accounting for only 0.11%. Brand A4 had the highest content of olefins, and the lowest content of thiazoles and acids, accounting for 0.41% and 0.21%, respectively. Ketones, aldehydes, and alkenes were more abundant, and acids were less abundant among the four different brands.

The contribution of aromatic substances to the overall flavour depends mainly on their content and threshold values, which can be characterised by OAV. The OAV distribution of compounds in the four dried bean curd samples is shown in Table 1. Aldehydes dominated in all four brands. Aldehydes were the key flavour components, followed by ketones, alkenes, alcohols and others, which constituted the prominent flavour of leisure-dried bean curd. Meanwhile, aldehydes, alcohols and alkenes simultaneously occupied the central position of modified flavour components in the leisure-dried bean curd. There were differences in the key flavour components of the four brands.

### Comparison and analysis of volatile components in four different brands

All peaks were selected for fingerprint comparison using the LAV software Gallery Plot plug-in to further clarify the specific differential volatile components among different brands. The results showed that the volatile components of the four different brands were quite different. Fig. 3 shows the visual information
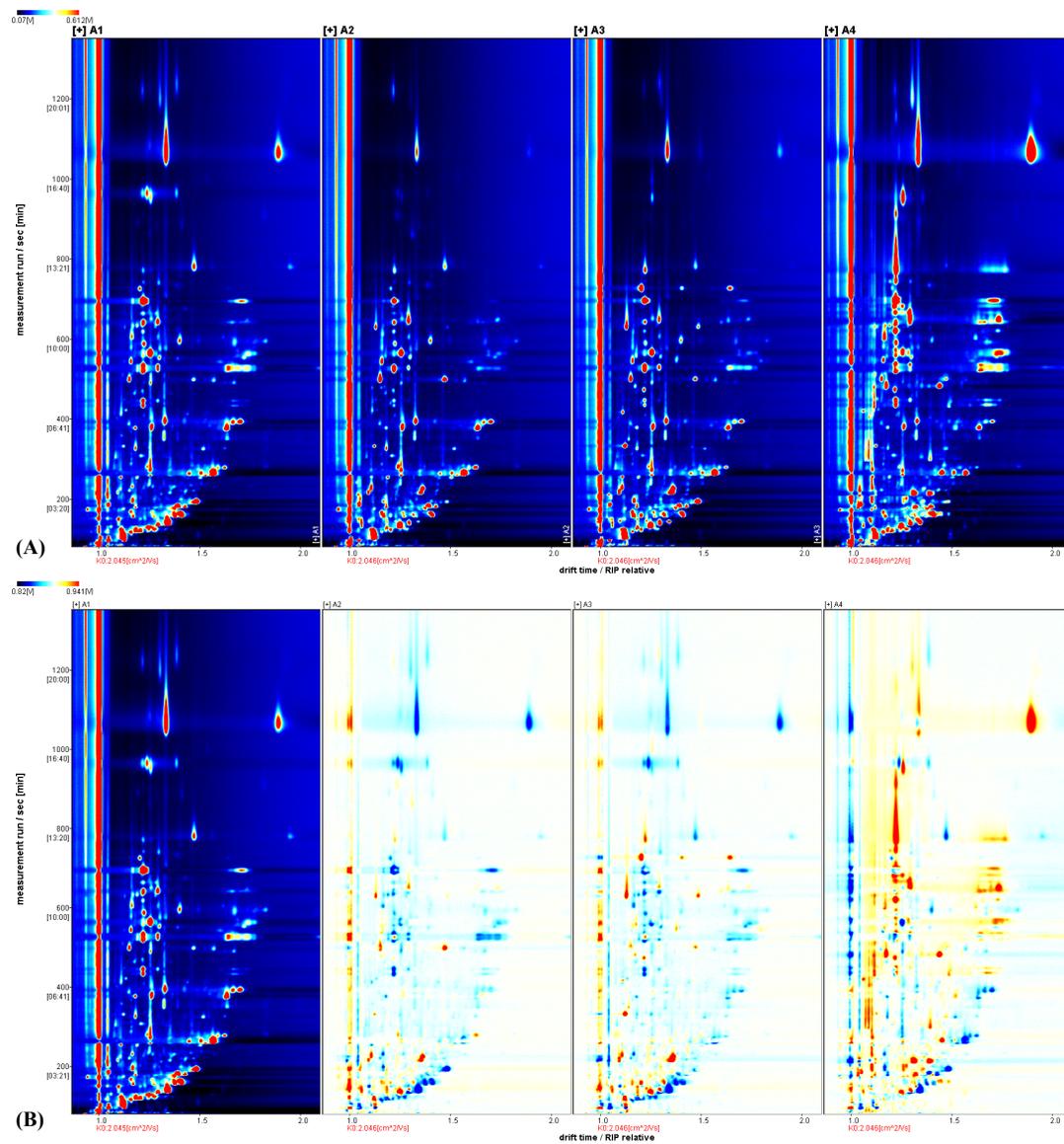
Fig. 1 GC-IMS two-dimensional spectra of volatile organic compounds of four dried bean curd samples (A); comparison of two-dimensional difference spectra of volatile organic compounds of the four bean curd samples (B).

Table 1 OAV distribution of compounds of four dried bean curd samples.

Amount	A1		A2		A3		A4	
	OAV > 1	0.1 < OAV < 1	OAV > 1	0.1 < OAV < 1	OAV > 1	0.1 < OAV < 1	OAV > 1	0.1 < OAV < 1
Aldehydes	11	4	12	3	11	4	11	3
Alkenes	3	3	3	2	2	3	3	4
Ketones	6	1	5	3	6	2	5	3
Alcohols	3	4	3	4	3	3	4	3
Esters	2	0	1	1	2	0	1	1
Acids	0	0	0	0	0	0	0	1
Ether	1	0	1	0	1	0	1	0
Pyrazines	2	0	2	1	2	1	2	1
Thiazoles	1	0	1	0	1	0	1	0
Furans	2	0	2	0	2	0	2	0
Others	4	0	4	0	3	1	4	1
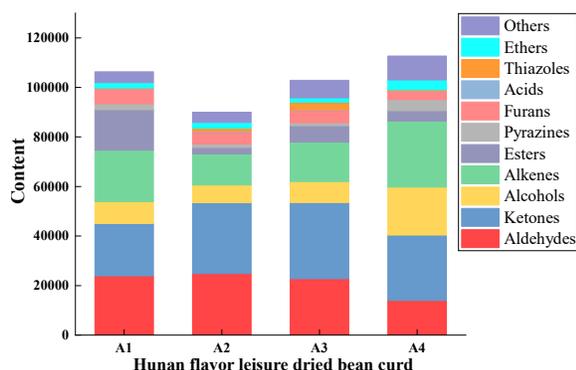
Fig. 2 Composition of volatile organic compounds of four dried bean curd samples.

related to the types and corresponding contents of volatile compounds in four different brands. Each row represented all signal peaks selected from one brand sample, and each column represented the signal peaks of the same volatile organic compound in four different brands. Some volatile compounds would create different forms of monomer (M) and dimer (D) due to a high concentration and other reasons, and corresponding migration peaks will appear [26].

Fig. 3 shows the content information of volatile compounds in four different brands in the form of fingerprint, which could more intuitively reflect the differences of volatile compounds. In total, 98 volatile compounds were detected by GC-IMS among four brands, and 7 substances were not identified because of the limitations in the GC-IMS spectrum library. Fig. 3 could be roughly divided into five regions (A, B, C, D and E). Region B contains common volatile compounds in the four brands. Regions A, C, D and E represent the unique volatile compounds of A1, A2, A3 and A4, respectively. Brand A1 had nine unique volatile compounds including 3-octanone, hexanoic acid, butanal, nonanal, (E)-hept-2-enal, citronellal, 1-menthol, and ethyl acetate. These compounds presented green cilantro and cucumber flavour. Brand A2 had nine unique volatile compounds: α-terpineol, oct-1-en-3-ol, benzaldehyde, 2-heptanone, 2-ethyl-3-methyl pyrazine, 2-hexanone, (E)-2-hexenal, butyl acetate and (E)-2-pentenal. These compounds presented lemon, cherry and sweet flavour. Brand A3 had 13 unique volatile compounds: acetone, pentanol-1-ol, 2-acetylfuran, 2-acetylthiazole, 2-furanmethanol, diallyl disulphide, and others. These compounds presented sweet, slightly green and burnt flavour. Brand A4 had 27 unique volatile compounds: linalool, 1,8-cineole, β-myrcene, thiosulfate, methylpyrazine, thiosulfate, etc. These compounds presented citrus and fruit flavour.

Principal component analysis of four brands of leisure-dried bean curd

PCA statistics were used to better present and distinguish the differences among the four different brands based on the determination of volatile components obtained from GC-IMS. The analysis results applied a dynamic PCA plug-in (Fig. 4). Fig. 4 demonstrates that the cumulative contribution rate of the first two principal components when the dimension reduction was 82%. After performing feature compression, the relatively complete information was retained, which could better represent the feature differences of the original variables. In the PCA analysis chart, a close distance between samples reflects a slight difference, and a far distance reflects a noticeable difference. Fig. 4 shows slight differences among the three parallel groups of the same sample. However, there were apparent differences in volatile organic compounds in different brands. Brand A2 and A3 were relatively close, and their volatile compounds were similar. Brands A1 and A4 were far away, indicating that the aroma characteristics of the two leisure-dried bean curd differed significantly from those of other brands. Therefore, the GC-IMS results combined with PCA analysis assisted in easily and quickly distinguishing different leisure dried bean curd brands. Jiang et al [27] used GC-IMS detection and PCA analysis to explore the significant differences in the quality inspection of volatile compounds in different quinoa. The PCA analysis obtained was consistent with the visual map of GC-IMS, and its research results were not different from the experimental results.

In addition, the latest fingerprint analysis method, "nearest neighbour" fingerprint similarity, was applied in the study. The "nearest neighbour" function finds the "nearest neighbour" by calculating the Euclidean distance matrix between samples and then retrieves the minimum distance. The relatively close group measurement results compared with the farther groups were observed. As shown in Fig. 5, different colours represent different brands. Each sample was injected three times in parallel. The three boxes of the same colour represent the peak point of each parallel test. The grey line at the top represents the similarity of each sample. The closer the peak point, the darker the grey colour, indicating good reproducibility. The farther the grey colour, the lighter the colour, suggesting that the repeatability was worse. Fig. 5 demonstrates good repeatability of all dried bean curd samples. Brand A2 and A3 were relatively closer, and brands A3 and A4 were relatively further. This outcome indicates the difference and similarity of volatile compositions among brands. For instance, the volatile compound composition of brands A2 and A3 were relatively similar, which was consistent with the results from PCA analysis. It showed that the four different brands could be distinguished by volatile compounds. This study is
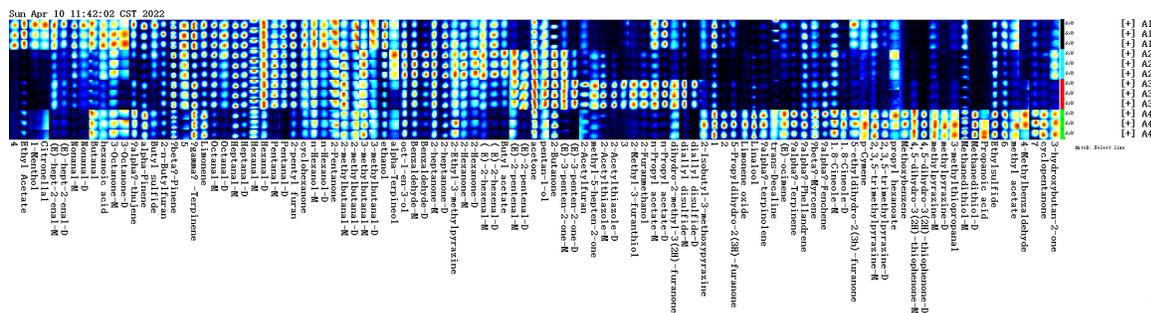
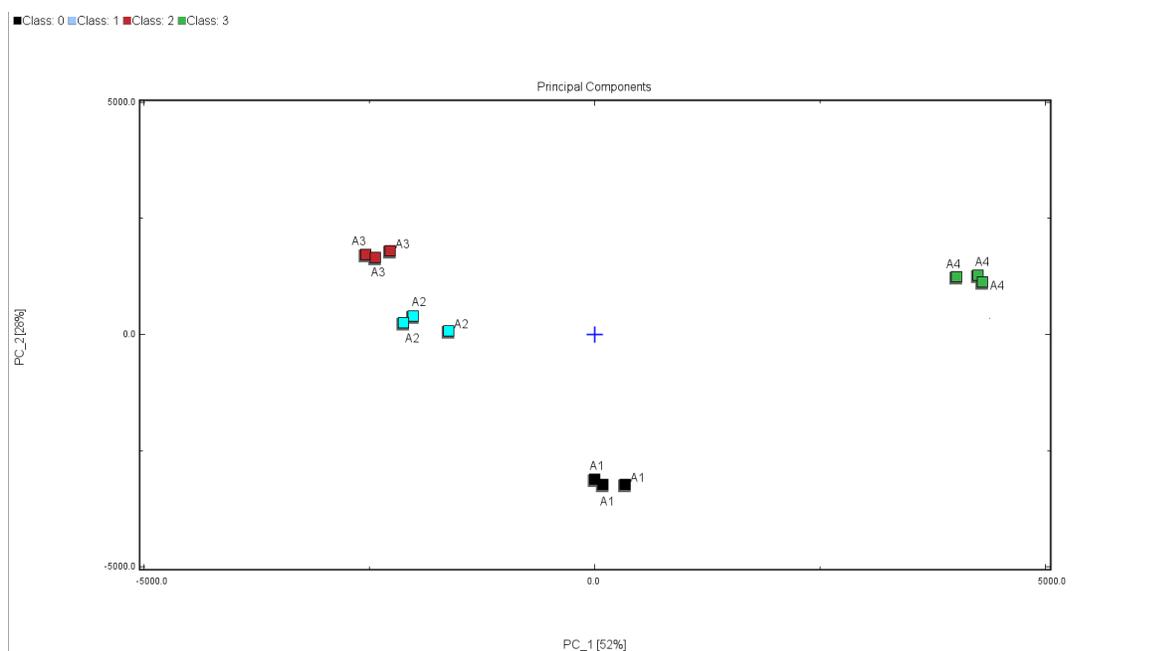**Fig. 3** GC-IMS fingerprints of four dried bean curd samples.

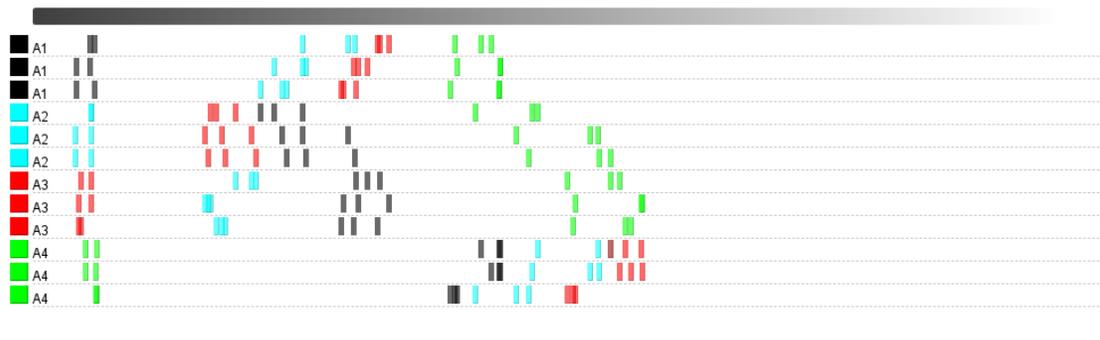**Fig. 4** PCA analysis of four dried bean curd samples.

Fig. 5 Fingerprint analysis of the "nearest neighbors" of four dried bean curd samples.
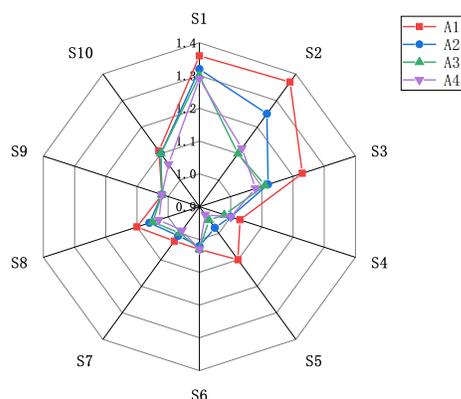
Fig. 6 Flavor radar map of four dried bean curd samples.

consistent with that reported by Luo et al [28] that the passion fruit with the same maturity had good repeatability, and there was a distance between passion fruits with different maturity.

Verification analysis based on electronic nose

Under the same conditions, the four dried bean curd samples were tested by an electronic nose. Fig. 6 showed the differences of response signals of four brands to 10 sensors of electronic nose. The response signals of sensors S2, S3, and S5 from brand A1 could be significantly distinguished from the other three brands, while the response signals of sensors S9 and S10 could not be separated from different brands, indicating the same findings as the GC-IMS measurement results. Yang et al [29] used an electronic nose to distinguish between genuine and fake brand leisure-dried bean curd, and the results were similar to the results of this study. The differences in the flavour of different brands of leisure-dried bean curd were determined by different manufacturing processes.

CONCLUSION

The volatile compounds of four brands of Hunan flavour leisure-dried bean curd were analysed and compared using GC-IMS. In total, 98 characteristic volatile compounds including 26 aldehydes, 20 ketones, 12 alcohols, 11 alkenes, 6 esters, 6 pyrazines, 3 furans, 2 acids, 2 thiazoles, 1 ether and 9 others were detected. The key and unique flavour components in the four brands were different based on the comparison of odour activity distribution. Each brand had its own unique characteristic volatile components, and its relative content could be used to identify specific samples. The content of nonanal, butyraldehyde, octanal, heptanaldehyde and hexanal was the highest in brand A1. Mushroom alcohol, benzaldehyde, 2-hexanone, 2-heptanone, butyl acetate had the highest content in

brand A2. Acetone, amyl alcohol, 2-acetylfuran, 2-acetylthiazole, and 2-furan methanol had the highest content in the brand A3. While brand A4 had the highest content of linalool, 1,8-cineole, β-laurene, acetoin, trimethyl pyrazine. These volatile compounds demonstrate different flavour. Flavour fingerprints are useful for origin traceability and product quality control.

Appendix A. Supplementary data

Supplementary data associated with this article can be found at http://dx.doi.org/10.2306/scienceasia1513-1874.2023.062.

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Appendix A. Supplementary data

Table S1 Volatile compounds of four dried bean curd samples.

Count	Compound	RI	RT (s)	Dt (au)	A1	A2	A3	A4
1	Nonanal-M	1100.6	781.274	1.47521	2003.60±190.22	1384.69±185.39	785.05±24.07	292.73±5.36
2	Nonanal-D	1100.6	781.274	1.95072	259.34±44.79	134.77±31.47	59.32±6.29	50.97±6.06
3	Octanal-M	1006.5	595.858	1.40430	1260.77±47.53	1161.60±122.56	1024.80±28.96	418.70±28.30
4	Benzaldehyde-M	959.0	499.828	1.15324	1247.72±9.33	2466.92±8.48	1786.27±16.02	1031.29±113.06
5	Benzaldehyde-D	959.2	500.281	1.47049	428.61±20.71	1839.84±100.10	854.55±20.37	496.32±102.70
6	(E)-hept-2-enal-D	954.5	491.210	1.67174	257.83±48.99	103.63±19.14	108.68±6.60	101.16±8.15
7	Heptanal-D	899.0	395.186	1.70326	1817.90±30.29	1461.07±88.98	1244.94±35.69	614.38±111.32
8	Heptanal-M	900.0	396.723	1.33396	2258.80±34.06	2161.28±31.00	2086.18±9.46	1516.04±90.39
9	Octanal-D	1006.6	596.024	1.82959	209.75±9.99	138.23±36.01	100.51±3.11	39.89±5.41
10	(E)-hept-2-enal-M	955.4	492.785	1.25945	410.57±21.82	169.16±17.62	267.39±8.47	72.13±8.93
11	3-methylthiopropanal	905.6	405.470	1.09195	23.86±1.28	19.64±2.80	31.20±3.70	129.98±15.20
12	(E)-2-hexenal-M	848.5	328.460	1.18795	426.95±22.16	486.64±27.55	295.68±20.51	139.15±16.48
13	Hexanal-M	791.3	267.010	1.25963	1096.80±10.24	1273.18±140.26	1322.05±20.22	1215.15±68.72
14	Hexanal-D	787.5	263.352	1.57163	5209.92±44.57	4913.70±161.20	5285.40±90.27	2446.20±72.47
15	Pentanal-D	691.0	182.700	1.43234	979.89±41.33	838.06±47.90	821.61±47.02	96.59±6.01
16	(E)-2-hexenal-D	847.3	327.037	1.52262	256.80±18.85	258.86±29.66	121.70±2.56	37.52±3.16
17	(E)-2-pentenal-M	747.4	226.270	1.11141	149.59±8.28	675.66±13.10	634.81±8.64	348.83±44.84
18	Pentanal-M	687.5	180.488	1.18412	439.76±12.15	557.94±25.48	537.41±8.94	86.44±0.41
19	(E)-2-pentenal-D	750.3	228.831	1.36528	266.37±6.54	1660.65±97.43	2087.78±38.65	241.76±49.32
20	2-methylbutanal-M	662.5	168.631	1.16276	412.85±4.68	558.22±5.12	601.98±7.65	488.28±17.79
21	3-methylbutanal-M	638.7	158.037	1.17142	433.23±8.53	527.67±15.97	490.97±9.34	557.42±30.80
22	2-methylbutanal-D	659.0	167.042	1.40212	2116.57±25.17	1199.96±55.04	1354.73±8.66	1912.32±39.56
23	3-methylbutanal-D	640.2	158.699	1.41708	997.07±10.73	567.91±29.21	594.32±25.12	774.13±27.84
24	Butanal	552.3	124.931	1.28165	615.66±15.22	258.92±3.02	253.79±5.60	603.79±22.55
25	Citronellal	1174.7	966.806	1.22014	374.67±94.04	51.93±12.72	32.79±2.87	208.84±44.12
26	4-Methylbenzaldehyde	1084.1	744.874	1.19416	14.51±0.73	13.36±2.00	22.02±5.40	68.22±24.56
27	5-Propyldihydro-2(3H)-furanone	1170.8	956.247	1.26123	2020.61±492.78	353.12±84.94	839.62±182.78	3815.37±796.19
28	2-heptanone-M	888.6	379.819	1.26406	2036.96±28.89	2365.24±25.48	2139.00±32.04	1646.36±91.69
29	2-heptanone-D	888.3	379.435	1.63731	3110.05±172.17	2947.26±161.95	2302.21±50.34	1555.58±234.74
30	methyl-5-hepten-2-one	987.8	559.474	1.17882	21.07±1.60	62.41±8.92	152.11±13.04	66.42±13.57
31	3-Octanone-M	986.1	555.903	1.30224	246.91±31.82	241.28±14.08	143.51±15.69	251.04±20.24
32	5-ethyldihydro-2(3h)-furanone	1028.3	634.460	1.19164	243.99±55.28	69.66±16.74	50.71±7.99	250.61±37.41
33	cyclohexanone	895.2	389.441	1.15163	154.53±3.94	148.32±5.99	118.69±5.17	99.54±5.29
34	4,5-dihydro-3(2H)-thiophenone-M	950.8	484.161	1.17631	33.71±5.92	72.20±3.94	207.21±11.95	1144.27±48.61
35	4,5-dihydro-3(2H)-thiophenone-D	949.9	482.308	1.43886	55.88±2.38	56.88±8.57	72.82±8.69	2264.66±163.54
36	2-Hexanone-M	781.2	257.254	1.19386	246.49±6.87	384.49±8.73	280.43±1.71	182.80±21.71
37	2-Pentanon	679.8	176.788	1.37612	413.69±6.71	435.23±15.96	481.75±11.45	340.03±48.69
38	3-Octanone-D	986.0	555.582	1.7238	126.15±10.31	77.96±11.70	29.95±1.76	173.97±28.14
39	2-Hexanone-D	777.0	253.162	1.50473	158.63±7.64	229.52±19.46	138.62±3.98	97.19±19.27
40	(E)-3-penten-2-one-M	737.5	217.946	1.09592	257.67±12.07	753.57±6.07	849.47±60.82	118.84±10.20
41	(E)-3-penten-2-one-D	736.3	216.986	1.35217	235.70±8.15	1495.35±47.31	3702.37±37.68	106.99±6.59
42	dihydro-2-methyl-3(2H)-furanone	804.8	280.375	1.07923	139.71±5.72	67.87±11.14	294.80±2.66	179.01±4.03
43	2-Butanone	577.9	133.936	1.25095	2584.14±73.39	6611.52±91.53	6257.12±20.42	4300.90±326.33
44	acetone	497.3	107.535	1.12425	8926.21±268.42	12018.66±70.18	12526.80±171.30	9183.47±339.70
45	cyclopentanone	799.2	274.717	1.10918	82.20±6.90	131.37±35.66	98.08±3.74	549.38±29.74
46	3-hydroxybutan-2-one	715.9	200.824	1.33490	42.86±4.96	69.17±2.32	25.62±3.68	82.66±8.54
47	(E)-ocimene	1045.7	667.006	1.21986	378.06±92.57	199.13±67.29	55.31±4.11	881.97±77.67
48	Lemons	1032.1	641.358	1.22122	1986.56±294.51	1347.45±264.38	1090.04±233.45	2118.90±101.02
49	α-Terpins	1020.4	620.075	1.22258	522.47±114.48	527.48±173.32	403.49±93.72	3164.44±593.21
50	α-Phellandrene	1004.1	591.698	1.22394	1080.38±76.87	756.46±71.56	654.39±61.95	1321.85±200.23
51	β-Myrcene	991.3	567.141	1.22258	1068.31±265.00	711.76±158.83	799.81±139.04	1842.68±152.46
52	β-Pinene	972.9	527.850	1.22258	5138.79±165.81	3982.18±702.56	4739.35±262.72	4511.05±204.30
53	α-Fenchene	945.6	474.371	1.21850	242.88±63.51	387.06±198.06	997.94±272.38	1187.16±397.88
54	α-Pinene	931.5	448.915	1.22131	1596.43±398.02	579.63±318.05	817.84±208.15	1851.85±368.26
55	α-thujene	923.2	434.544	1.22253	1387.70±370.54	376.91±210.75	657.09±178.32	1470.86±287.17

Note: M is monomer, D is dimer.

Table S1 Continue . . .

Count	Compound	RI	RT (s)	Dt (au)	A1	A2	A3	A4
56	α-terpinolene	1084.3	745.471	1.22681	204.47±42.70	149.05±44.47	162.44±30.89	1378.65±207.56
57	γ-Terpinene	1062.1	699.203	1.22122	7155.28±228.69	3587.59±755.18	5553.79±648.87	6930.36±151.86
58	1-Menthol	1174.3	965.949	1.24052	1094.89±172.50	180.37±55.98	57.43±5.35	203.51±25.54
59	α-Terpineol	1257.2	1226.288	1.21741	121.62±12.46	150.97±12.95	59.30±23.28	88.65±23.97
60	linalool	1099.3	778.311	1.22681	816.87±69.27	1507.11±324.65	2571.45±364.81	11007.92±1268.09
61	oct-1-en-3-ol	981.2	545.182	1.16303	1003.12±155.07	1497.91±81.25	1075.22±103.29	834.03±100.17
62	n-Hexanol-D	867.4	351.792	1.63979	207.62±18.85	125.30±12.64	119.55±4.94	116.59±19.56
63	n-Hexanol-M	868.3	352.944	1.32344	500.43±31.59	413.26±13.60	404.97±4.31	357.66±25.02
64	2-Methyl-3-furanthiol	865.6	349.487	1.14452	24.96±3.03	23.20±1.81	187.83±1.43	23.66±4.69
65	2-Furanmethanol	852.4	333.095	1.12719	224.60±41.61	463.54±103.89	2041.99±89.36	333.46±48.33
66	Methanedithiol-D	734.0	215.053	1.39361	73.06±5.97	91.37±6.94	172.64±17.35	3016.22±83.51
67	Methanedithiol-M	734.3	215.309	1.04385	1491.84±14.29	700.76±6.46	448.90±5.08	1788.91±76.91
68	pentan-1-ol	762.2	239.396	1.25205	239.54±5.19	319.40±8.03	386.30±15.39	216.77±20.53
69	ethanol	462.5	97.829	1.05068	2983.42±155.83	1655.75±38.54	984.17±9.73	1345.70±107.80
70	propyl hexanoate	1079.6	735.349	1.39126	71.38±11.93	117.93±26.81	123.30±23.14	131.89±35.94
71	n-Propyl acetate-D	706.1	193.482	1.48540	4339.68±200.94	496.95±36.18	3977.82±64.55	582.27±109.24
72	Butyl acetate	804.3	279.822	1.62785	602.98±76.26	1086.08±72.44	478.28±34.95	144.52±20.91
73	n-Propyl acetate-M	711.0	197.136	1.16862	862.50±28.59	414.57±11.11	938.37±54.70	208.80±32.63
74	Ethyl Acetate	604.8	144.133	1.34307	10233.05±48.54	354.91±16.17	1024.22±34.00	2994.99±251.86
75	methyl acetate	535.7	119.395	1.03866	262.35±8.60	113.74±0.81	59.94±0.47	181.85±24.17
76	2-Isobutyl-3-methoxypyrazine	1251.0	1204.355	1.30692	670.06±73.44	242.99±100.27	398.42±12.89	1193.07±275.80
77	2,3,5-trimethylpyrazine-M	1012.7	606.546	1.16654	1044.59±121.41	602.57±54.44	400.94±5.09	1660.13±124.32
78	2-Ethyl-3-methylpyrazine	996.7	579.274	1.16257	164.84±25.36	198.30±7.34	150.09±3.70	175.59±25.87
79	2,3,5-trimethylpyrazine-D	1011.5	604.401	1.61854	117.70±15.22	62.30±15.41	59.41±2.67	312.12±56.66
80	methylpyrazine-D	825.7	302.375	1.39728	35.06±2.72	32.29±6.89	28.95±2.37	405.65±46.29
81	methylpyrazine-M	831.1	308.426	1.08658	337.90±13.61	214.05±8.60	207.10±24.73	707.36±52.95
82	2-Acetylfuran	911.3	414.780	1.11370	445.87±29.43	329.34±32.49	752.41±55.73	
83	2-n-Butylfuran	887.8	378.667	1.18624	558.99±85.03	349.60±13.70	401.49±25.98	302.34±26.54
84	2-pentyl furan	990.4	565.229	1.26088	4992.80±904.27	4324.28±250.57	4129.18±381.20	2242.81±345.14
85	hexanoic acid	995.9	577.479	1.30453	188.40±15.98	145.22±8.41	83.28±5.48	136.47±5.52
86	Propanoic acid	710.7	196.857	1.26266	42.47±4.56	27.48±0.27	29.55±2.90	243.81±75.19
87	2-Acetylthiazole-M	1025.2	628.730	1.13210	203.93±12.49	1072.51±87.90	2374.53±135.02	379.24±129.69
88	2-Acetylthiazole-D	1026.2	630.610	1.48612	60.55±6.20	141.20±10.24	647.00±55.50	86.56±28.94
89	Ethylsulfide	696.5	186.526	1.04747	2030.28±133.88	2324.96±143.17	1770.29±111.97	3882.58±136.09
90	Limonene oxide	1154.6	912.588	1.22623	121.68±11.36	195.81±41.80	130.23±22.32	1250.30±242.56
91	trans-Decaline	1053.6	682.286	1.21850	528.11±95.95	172.80±56.06	164.06±47.60	921.61±34.23
92	Butyl sulfide	1082.2	740.845	1.29863	334.86±75.53	226.33±51.88	73.92±10.82	198.22±33.10
93	1.8-Cineole-M	1032.8	642.640	1.29480	1937.36±336.43	2494.76±519.68	1712.43±229.66	4047.88±96.38
94	1.8-Cineole-D	1035.8	648.343	1.73844	215.74±45.62	388.99±147.10	286.97±43.31	2112.79±340.32
95	p-Cymene	1028.5	634.786	1.17041	161.27±6.72	90.26±10.07	60.85±2.78	228.59±34.83
96	Methoxybenzene	914.9	420.560	1.06277	76.76±5.49	100.00±9.16	108.93±4.85	286.62±32.05
97	diallyl disulfide-M	1076.4	728.687	1.20813	893.30±297.10	397.06±137.46	3142.21±196.93	492.48±69.64
98	diallyl disulfide-D	1075.3	726.361	1.64330	99.37±48.58	57.16±4.38	1378.42±406.33	53.08±2.50

Note: M is monomer, D is dimer.