Discrimination of five brands of instant vermicelli seasonings by HS-SPME/GC–MS and electronic nose

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Abstract The flavor profile of five brands of instant vermicelli seasonings were identified by headspace solid-phase micro-extraction coupled with gas chromatography–mass spectrometry (HS-SPME/GC–MS) and electronic nose (e-nose). GC–MS showed that the volatile compounds of instant vermicelli seasonings were significantly different. Alkenes, alcohols, aldehydes and ketones were the major volatile compounds in instant vermicelli seasonings. The seasonings could be classified based on differences in volatile compounds. The overall volatiles profiles were also analyzed by e-nose. E-nose determination and GC–MS statistical analysis had similar results. The volatile compounds showed good correlation with e-nose sensors according to partial least square regression models. Both methods had good potential application in evaluating flavor quality and differentiating among instant vermicelli seasonings.

Keywords Instant vermicelli seasoning · Volatile compounds · Gas chromatography–mass spectrometry ·

Electronic nose · Principal component analysis · Partial least square regression

Introduction

Instant vermicelli has been widely consumed in China. Instant vermicelli refers to pre-packaged convenience foods made from potatoes, beans, and cereal starch—with or without added ingredients—processed into dry, no-cooking vermicelli and then flavored with seasonings. The seasoning is very important in people's daily diets because of its ability to flavor otherwise bland dishes. Add some seasoning according to different tastes, instant vermicelli will be delicious and fragrant. Seasoning is not only used to improve the flavor characteristics (Vázquez-Araújo et al. 2013), but can also increase caloric, protein and fat intakes (Best and Appleton 2011).

Nowadays, consumer awareness related to food quality has been raised in all aspects. Seasoning with uneven quality is spreading rapidly in the Chinese market. Its poor quality might harm the rights and health of the general public. Controlling and improving the quality of seasoning is becoming emergent. To a certain extent, brand represents the quality of products in the market. Instant vermicelli seasonings of different brands have different qualities (such as the chemical ingredients and flavor profile) (Liu and He 2008; Yang et al. 2011). Flavor is one of the most important indicators of the quality of seasoning. Volatile compounds play a crucial role in the typical flavor of seasoning.

Sensory evaluation and headspace solid-phase micro-extraction coupled with gas chromatography–mass spectrometry (HS-SPME/GC–MS) are traditionally used to analyze the flavor profile of seasoning. Sensory evaluation

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is performed by a group of panelists who have developed language and vocabulary to describe various seasoning aroma attributes (Tian et al. 2014). Sensory evaluation is expensive, time consuming and easily influenced by psychological and physiological factors of the assessors (Chen et al. 2011). HS-SPME/GC–MS is an advanced instrumental method that has been widely applied to qualitative and quantitative analysis for volatile compounds. It can simultaneously separate, identify and quantify multiple volatile compounds and provide specified information of seasoning flavor (Yang et al. 2018).

In the last few decades, the use of electronic nose (e-nose) is gaining popularity in odor analysis. E-nose is a reliable device which imitates human olfactory perception (Qiu and Wang 2017). E-nose has become an effective method for rapid, easy and accurate food odor analysis that is objective, highly automated and lower cost. E-nose can detect volatile compounds and provide integrated information of flavors to distinguish among samples (Tian et al. 2014). More recently, e-nose has been widely used for food quality control for broccoli (Chen et al. 2019), *Tremella aurantialba* (Dai et al. 2018), meat products (Wang et al. 2012), Chinese spirits (Liu et al. 2012), coffee (Dong et al. 2017), ginseng (Cui et al. 2017) and juices (Hong et al. 2014; Qiu et al. 2015; Wu et al. 2017). Quality evaluation of beef seasoning by GC–MS and e-nose (Tian et al. 2015), and discrimination of chicken seasonings and beef seasonings using e-nose and sensory evaluation have been previously reported (Tian et al. 2014).

The objectives of this work were to identify the volatile compounds of different brands of instant vermicelli seasonings by GC–MS and to investigate the feasibility of using an e-nose to discriminate seasoning samples of different brands by principal component analysis (PCA), linear discriminant analysis (LDA) and multilayer perceptron neural networks analysis (MLPN). The correlation between the volatile compounds and e-nose response signals was established using partial least squares regression (PLSR). This work may be helpful in providing some valuable information on evaluating and discriminating the quality of instant vermicelli seasoning to consumers.

Materials and methods

Samples and chemicals

In this work, five brands of instant vermicelli seasonings with chicken and shiitake were purchased from the local supermarket in Shaanxi, China. Different brands were named as 1, 2, 3, 4 and 5, respectively. Each brand offers seasoning powder (P), seasoning mixture sauce (S) and the mixture of powder and sauce (M). The sample number

for each brand was 20 and a total of 100 samples were prepared for analysis. 7 g seasoning powder, 4 g seasoning mixture sauce and 11 g the mixture of powder and sauce were placed in three separate beakers. Then, 100 mL hot deionized water was added and stirred until all granules dissolved. The sample solutions were equilibrated for 10 min at 50 °C and then were analyzed by sensory evaluation, e-nose and GC–MS.

2-Octanol (internal standard, 99.5% purity) was purchased from Sigma-Aldrich Chemical Co. (St. Louis, MO). HPLC-grade methanol was obtained from a commercial source.

Sensory evaluation

Fifteen sensory assessors (ages 20–35, eight females and seven males) were chosen to evaluate the sensory attributes of instant vermicelli seasoning. Four sensory attributes, including color, taste, odor, and texture, were developed to describe the instant vermicelli seasoning flavor (Table S1). The sensory score standard was as follows: 1–2, very weak; 3–4, considerably weak; 5–6, neither weak nor strong; 7–8, considerably strong; and 9–10, very strong. Sensory evaluation of the mixture of powder and sauce was performed in triplicate in a sensory laboratory. Different seasoning samples were coded with three-digital codes in a random order to prevent bias. The evaluations were carried out at room temperature and one at a time, with 5 min wait times between samples. The evaluation of each assessor was recorded via questionnaire (Chen et al. 2018).

HS-SPME/GC–MS analysis

HS-SPME conditions

HS-SPME technology was employed to extract the volatile compounds of the instant vermicelli seasoning samples. An SPME fiber (Supelco, Bellefonte, PA, USA) coated with a polydimethylsiloxane-divinylbenzene (PDMS/DVB, 65 µm) film was used. A volume of 5 mL seasoning sample and 10 µL of 2-octanol (2.6 mg/L in methanol) were placed in a 20 mL headspace vial. The PDMS/DVB fiber was inserted into the headspace with heating (40 °C) and agitation (250 rpm) for 30 min. After extraction, it was put into the injection port of GC–MS (TRACE1310-ISQLT, Thermo Fisher Scientific) and desorbed at 250 °C for 3 min.

GC–MS analysis

A GC–MS apparatus equipped with a TG-WAXMS fused silica capillary column (30 m × 0.25 mm × 0.25 µm, Thermo Fisher Scientific, USA) was used to separate the

volatile compounds. Helium was used as the carrier gas at a flow rate of 1 mL/min in a splitless injection mode. The oven temperature was held at 40 °C for 2 min, raised to 230 °C at a rate of 4 °C/min and then held for 10 min. The MS fragmentation was conducted by electron impact (EI) source (electron energy, 70 eV; ionization temperature, 230 °C). The transfer line temperature was 230 °C. The mass acquisition range was 33–450 amu. All measurements were performed in triplicate.

E-nose analysis

A PEN3 portable E-nose from Win Muster Airsense Analytics Inc. (Schwerin, Germany) was used to analyze the volatile compounds. The instrument consists of a metal oxide gas sensor array, a gas sampling apparatus and a signal processing unit (Feng et al. 2011). For ensuring the stability of the data captured by the sensors, the e-nose was warmed up and calibrated with processed pure air before testing.

A 10 mL aliquot of the seasoning sample was placed in a 30 mL airtight glass vial and allowed to equilibrate for 30 min at room temperature. The e-nose parameters were as follows: chamber and injection flow rate of 400 mL/min, measurement time of 60 s, sample interval time of 1 s, flush time of 300 s, automatic zero adjustment time of 5 s. To ensure the accuracy of the data, all measurements were repeated ten times.

Statistical analysis

Duncan's multiple test was applied to verify significant ($p < 0.05$) differences among the sensory evaluation data using Minitab 16.2. The identification of volatile compounds was performed by comparing their similarity index (SI), reverse similarity index (RSI) and mass spectra with the MS library of NIST (match degree $> 85\%$). PCA of volatile compounds was performed with Minitab 16.2. PCA and LDA of e-nose data were performed with Win Muster software of PEN3 E-nose. MLPN was performed using SPSS 20.0. PLSR was analyzed with Unscrambler software.

Results and discussion

Sensory evaluation

The statistical results for the sensory evaluation of five brands of instant vermicelli seasonings are shown in Table S4. The sensory attributes of taste, odor and texture were significantly different ($p < 0.05$) among all seasoning samples. The color attribute was not significantly different

among the seasoning samples. All samples were golden yellow and had shiitake and chicken taste and flavor. The sensory evaluation results indicated that 1M had the highest scores for color, taste and texture attributes; and 3M had the highest scores for odor attribute. 5M got the lowest scores in all attributes, and its overall flavor qualities were the lowest.

HS-SPME/GC–MS analysis

Volatile compounds of the instant vermicelli seasonings

Table S2, S3 and S4 shows the volatile compounds detected in the instant vermicelli seasoning powder, seasoning mixture sauce and the mixture of powder and sauce, respectively. A total of 68, 64 and 75 volatile compounds were detected. Five brands of instant vermicelli seasoning powder showed significantly different combinations and amounts of volatile compounds. Alkenes, alcohols, aldehydes and ketones were the major volatile compounds in different seasoning powders.

As shown in Table S2, alkenes were the largest volatile compounds in the seasoning powder. The compounds β -pinene, with a pine and turpentine aroma; D-limonene, with a lemon and orange aroma; caryophyllene, with a wood, spice and clove aroma; and 3-carene, with lemon and resin aroma, were the main and typical alkenes of seasoning powder. 1P had the highest concentrations of β -pinene (4.189 $\mu\text{g/L}$) and 3-carene (9.934 $\mu\text{g/L}$). 4P had the highest concentrations of D-limonene (10.615 $\mu\text{g/L}$) and caryophyllene (12.877 $\mu\text{g/L}$). 3-carene is the main component of monoterpene in essential oils and is widespread in many foods such as roasted foods and chewing gum. Tian et al. (2015) demonstrated that limonene was positively associated with beef seasoning aroma, while caryophyllene was negatively associated.

As shown in Table S3, 4S had the highest concentration of alkene compounds (0.744 $\mu\text{g/L}$), while the content of alkenes in 1S was the smallest (0.062 $\mu\text{g/L}$). β -sesquiphellandrene, α -curcumene and (E)- α -farnesene were important compounds of 4S. Caryophyllene was identified in 2S, 4S and 5S with 0.028 $\mu\text{g/L}$, 0.053 $\mu\text{g/L}$ and 0.261 $\mu\text{g/L}$, respectively. The content of alkenes (0.062–0.744 $\mu\text{g/L}$) in the seasoning mixture sauces were significantly lower than seasoning powder (8.068–48.410 $\mu\text{g/L}$).

As shown in Table S4, alkenes were the most abundant volatile compounds. 4M showed higher content of alkenes (43.151 $\mu\text{g/L}$) than other samples. β -pinene, 3-carene, o-cymene, caryophyllene and humulene were detected in all samples. 4M accounted for the higher concentration of 3-carene (9.427 $\mu\text{g/L}$) and caryophyllene (10.325 $\mu\text{g/L}$).

Among alcohols, linalool with floral aroma and α -terpineol with oil, anise and mint aroma, were identified as key compounds. They were identified in all samples. They accounted for the highest proportion of volatiles in 4P, with the values of 1.074 $\mu\text{g/L}$ and 0.202 $\mu\text{g/L}$. Linalool is a natural spice component and can be generated by the hydrolysis of glycosides, which causes the seasoning to deliver floral flavors (Xu et al. 2007). 1-octen-3-ol was in 3S and 3M at high concentrations (4.557 $\mu\text{g/L}$ and 5.278 $\mu\text{g/L}$, respectively). 1-octen-3-ol, an unsaturated alcohol derived from linoleic acid oxidation, was a main component of the overall flavor with a strong mushroom aroma (García-González et al. 2008). 1-octen-3-ol can also modify and enhance meat odor (Zhang et al. 2017). Other alcohols, due to their relative high odor thresholds, were usually not regarded as important contributors to the flavors.

The formation of aldehyde compounds was derived from the auto-oxidation of unsaturated fatty acids (Chen et al. 2018). Aldehydes were not only to contribute to the odor of the food, but also to react with other compounds to provide specific flavor through amino-carbonyl reactions (Adams et al. 2009; Shi et al. 2013). Hexanal and octanal were detected in all samples. Decanal, nonanal and heptanal were detected in most samples. These aldehydes were key flavor compounds in seasoning samples. Hexanal provides a grass, tallow and fat aroma at low concentrations and is positively associated with meat products. However, at high concentrations, it provides a stale odor which has a negative effect on products (Angelo et al. 1987). 3-methylbutanal with malt and almond aromas was identified in 1P (1.354 $\mu\text{g/L}$), 2S (0.987 $\mu\text{g/L}$), 1M (1.002 $\mu\text{g/L}$), 2M (0.981 $\mu\text{g/L}$) and 5M (0.287 $\mu\text{g/L}$). 3-methylbutanal was generated by Strecker degradation between α -dicarbonyls and amino acids (isoleucine and leucine) (Inoue et al. 2016). Inoue et al. (2016) showed that 3-methylbutanal was detected in raw soy miso, which increased through boiling. Soybean paste is one of the raw materials for producing 1S and 2S and, hence, may be the cause. Compared with seasoning powder and the mixture of powder and sauce, the content of aldehydes was relatively low in seasoning mixture sauce.

Carbonyl compounds were formed either through the reduction of unsaturated fatty acids or produced from amino acids. The partial degradation of amino acids results in the formation of higher alcohols. In the presence of oxygen, higher alcohols could be converted into aldehydes and ketones (Xiao et al. 2014). Ar-Turmerone and 6-methyl-5-hepten-2-one were the most abundant compound of ketones. The content of Ar-Turmerone and 6-methyl-5-hepten-2-one in the seasonings named 4 was much higher than other samples. 6-methyl-5-hepten-2-one has been reported to be responsible for sweet, fruity

flavors. 1-octen-3-one, with a mushroom-like flavor, was only identified in the seasonings named 3 (0.017–0.032 $\mu\text{g/L}$) and was formed from arachidonate 12-hydroperoxide via β -scission (Shi et al. 2013). The content of ketones in the seasonings named 4 (0.910–1.736 $\mu\text{g/L}$) was much higher than other samples.

Sulfur-containing compounds were detected mainly in the seasonings named 4 and 5 (4P, 4S, 4M, 5P, 5S, 5M). These compounds were mainly derived from the Maillard reaction between reducing sugars and amino acids, pyrolysis of amino acids, and degradation of thiamine (Xiao et al. 2014). Sulfur-containing compounds mostly provided an onion, metal, garlic, and grass aroma and were widely used in cooking, deodorization and flavoring because of their lower odor threshold.

Different manufacturers used different materials and production processes, which may cause the chemical ingredient contents and taste of instant vermicelli seasonings to differ among products. For example, the main ingredients on 1S packaged material were refined palm oil, fermented soy sauce, salt, spice, chicken extract, food additives (sodium glutamate, tert-Butylhydroquinone and edible essence), while the main ingredients of 3S were refined vegetable oil, chili, pepper, sesame and edible essence.

Discrimination of instant vermicelli seasoning flavor by PCA

In order to visualize the differences of volatile compounds between the instant vermicelli seasoning samples, PCA was performed based on all volatile compounds detected by GC–MS (Yao et al. 2015). Figure 1 showed the PCA plot of GC–MS results of instant vermicelli seasoning samples. For the seasoning powder, PC1 and PC2 explained 47.2% and 30.4% of the total variance, respectively. It can be seen in Fig. 1a that the five samples were well-separated from each other. The score on PC1 differentiated the 4P sample from the rest of the samples, and PC2 clearly discriminated 5P samples from the rest of the samples. The alcohols, ketones, ethers and sulfur-containing compounds were positively related to PC1 and associated with 4P. The aldehydes, acids and phenols were positively related to PC2 and associated with 1P, 2P and 3P. The esters and furans were negatively related to PC2 and associated with 5P.

For the seasoning mixture sauce (Fig. 1b), the first two PCs explained 68.2% of the total variation. All five samples were separated from each other. The 1S and 2S samples were located in the positive space for PC2 and positively correlated to phenols and aldehydes. Ketones, alkenes and ethers were associated with 4S. Esters, furans

Fig. 1 PCA of GC–MS data of instant vermicelli seasoning samples (a seasoning powder, b seasoning mixture sauce, c the mixture of powder and sauce)

and sulfur-containing compounds contributed substantially to 5S. Alcohols and acids were abundant in 3S.

For the mixture of powder and sauce, the first two PCs accounted for 74.2% of the total variance with concentrations of 46.3% and 27.9%, respectively. The 5M sample was located in the first quadrant of the graph, whereas 1M and 3M samples were located in the third quadrant and 2M and 4M samples were located in the fourth quadrant. The loading vectors of sulfur-containing compounds, acids and esters had positive scores in the PC2 direction and greatly contributed to the 5M sample. The loading vectors of aldehydes and furans had negative scores in the PC1 direction and greatly contributed to the 1M and 3M samples. Alcohols were associated with 2M. Loading vectors of the remaining three compounds had positive scores in the PC1 direction and were associated with 4M.

Electronic nose analysis

E-nose response of the instant vermicelli seasonings

The typical e-nose responses of the instant vermicelli seasonings are presented in Fig. 2, where G/G_0 was used as the response value. Each curve shows the response values of a corresponding sensor with increasing time (Dong et al. 2019). All e-nose response curves had the same variation trend. The response values of sensors W1W, W2W and W5S increased to a maximum in the first 5 s, decreased rapidly in the interval of 5–35 s, and finally reached a stable equilibrium. The response values of W1S increased dramatically from 0 to 20 s. The response values of W3S, W2S and W6S increased slightly during the measurement. In contrast, the response values of W1C, W3C, and W5C decreased to a slight extent, where G/G_0 values were generally less than one (Li et al. 2017).

The maximum values of W1W and W2W for 1M were significantly higher than in 1P and 1S at 5 s. Thus, 1M may

Fig. 2 E-nose responses curve of 1P (a), 1S (b), 1M (c) and 2P (d)

produce more aromatics, terpenes and sulfur-containing compounds than 1P and 1S. A comparison of Fig. 2a and Fig. 2d indicated that 2P produced more methane compounds than 1P. Instant vermicelli seasoning powder, seasoning mixture sauce and the mixture of powder and sauce could not be differentiated by just observing the sensor signals. Therefore, it was necessary to further analyze using PCA and LDA.

PCA classification

The first two principal components (PCs, Fig. 3) accounted for 98.13% of the total variance of seasoning powder. For the seasoning mixture sauce, PC1 explained 93.42% of the total variation and PC2 explained 5.68% of the total variance. For the mixture of powder and sauce, the first two PCs accounted for 99.51% of the total variance with concentrations of 92.27% and 7.24%, respectively. The three accumulated contributions of PC1 and PC2 were over 90%,

implying that the first two PCs offered enough information to explain the total variance (Yu et al. 2009; Hong et al. 2015). All samples were well-separated. Five brands of instant vermicelli seasonings (seasoning powder, seasoning mixture sauce and the mixture of powder and sauce) could be distinguished using e-nose combined with PCA based on differences in aroma attributes.

LDA classification

The LDA (LDs, Fig. 4) showed a very clear discrimination among the various seasonings, and all groups were appropriately classified. The first two LDs accounted for 88.82% of the total variance of seasoning powder. For the seasoning mixture sauce, the variance of LD1 and LD2 accounted for 80.18% and 12.37%, respectively. For the mixture of powder and sauce, about 85.86% of the total variance was explained. LD1 explained 52.61% of the total variation and LD2 explained 33.25% of the total

Fig. 3 PCA of instant vermicelli seasoning powder (a), seasoning mixture sauce (b) and the mixture of powder and sauce (c) using e-nose

variance. The system had sufficient resolution to explain the aroma deviation of the instant vermicelli seasoning with different brands. The LDA analysis suggested that the e-nose could correctly distinguish the instant vermicelli seasonings of different brands.

MLPN predicted modeling with e-nose data

MLPN predicted modeling was used to classify different seasoning samples. MLPN is a supervised learning model and suited for nonlinear systems. It is a classical feed-forward multilayer network. A typical MLPN structure involves an input layer, several hidden layers and an output layer. MLPN enables the output of neurons to classify input patterns by training the weights.

The samples were randomly divided into a training group of 70 samples and a test group of 30 samples. The input layer and output layer were set as 10 and 5, respectively. The number of the hidden layer is determined by SPSS software. The activation function of the hidden layer is a hyperbolic tangent function, and the activation function of the output layer is a Softmax function. The identification

results of the training and test group are shown in Table 1. The identification rates were both 100% in the training and test group. In other words, all samples were identified correctly according to their brands. Effectively, the MLPN classified samples into their correct treatment groups with excellent precision. Wang et al. (2019) identified adulterants in mutton by e-nose accompanied with MLPN predictive modeling and produced similar results. The MLPN results demonstrate that e-nose is an effective and reliable tool for distinguishing among mutton samples adulterated with different ratios of duck.

Correlation between GC–MS and e-nose

We explored the correlation between GC–MS data and e-nose response values with PLSR (Fig. 5). The PLSR modeling (Fig. 5a) between the e-nose response values and the volatile compounds of instant vermicelli seasoning powder provided a two-factor model that explained 98% of the variance in the X matrix (e-nose response values) and 94% of the variance in the Y matrix (volatile compounds). The shorter the distance between a compound and the

Fig. 4 LDA of instant vermicelli seasoning powder (a), seasoning mixture sauce (b) and the mixture of powder and sauce (c) using e-nose

Table 1 Results of MLPN models of e-nose

Models	Identification results of models (%)	
	Train group	Test group
Seasoning powder	100	100
Seasoning mixture sauce	100	100
The mixture of powder and sauce	100	100

sensor response, the higher the correlation of the compound with the sensor response (Long et al. 2019). W1S, W2S and W5S were closest to acids, indicating that these sensor responses had a high positive correlation with acids. W1C, W3C and W5C were closest to sulfur-containing compounds, indicating that the change of these sensor responses was greatly affected by sulfur-containing compounds.

Figure 5b shows the correlation between the e-nose response values and the volatile compounds of seasoning mixture sauce. The PLSR explained 100% of the variance in the X matrix and 83% of the variance in the Y matrix. W1S, W2S, W3S, W5S, W6S, W1W and W2W sensors were sensitive to alcohols, ethers and acids.

Figure 5c shows the correlation between the e-nose response values and the volatile compounds of the mixture of powder and sauce. The PLSR explained 100% of the

variance in the X matrix and 31% of the variance in the Y matrix. W5S, W1W and W2W sensors were positively correlated with alcohols. The W1C sensor was associated with esters, sulfur-containing compounds and acids. The W1C sensor was associated with alkenes.

Conclusion

A total of 68, 64 and 75 volatile compounds were identified through GC–MS in the instant vermicelli seasoning powder, seasoning mixture sauce and the mixture of powder and sauce, respectively. The content and type of volatile compounds in each sample category were significantly different. Flavor attributes could be differentiated via PCA of the GC–MS results. E-nose successfully discriminated

Fig. 5 Partial least squares regression (PLSR) correlations of volatile compounds and the e-nose sensor responses (**a** seasoning powder, **b** seasoning mixture sauce, **c** the mixture of powder and sauce)

among five brands of seasonings, and MLPN predictive modeling demonstrated the reliability of e-nose. PLSR was successful in detecting positive and negative relationships between e-nose sensors and volatile compounds. We conclude that combining GC–MS and e-nose with multivariate statistical analysis is a useful technique for evaluating and distinguishing variation among instant vermicelli seasonings.

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Compliance with ethical standards

Conflict of interest All authors declare that they have no conflict of interest.

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