



# Insight into aroma attributes change during the hot-air-drying process of white shrimp using GC-MS, E-Nose and sensory analysis

Di ZHANG<sup>1</sup> , Hong-Wu JI<sup>1,2,3,4,5\*</sup>, Gui-Xin LUO<sup>1</sup>, Hao CHEN<sup>1</sup>, Shu-Cheng LIU<sup>1,2,3,4,5</sup>, Wei-Jie MAO<sup>1,2,3,4,5</sup>

## Abstract

Aroma attributes are one of the most important criteria that affect the flavor quality of dried shrimp, but the dynamic changes of aroma attributes remain largely unknown during the drying process. The present study investigated aroma attributes change during the hot-air-drying process of shrimp using gas chromatography-mass spectrometry (GC-MS), electronic nose (E-nose) and sensory analysis. The potential correlations among volatile compounds, sensory attributes and E-nose data were analyzed by partial-least-squares regression (PLSR). Results showed that the aroma characteristic of shrimps changed significantly during processing. The odor in the fresh shrimp was very light, and the key aroma compounds mainly consisted of trimethylamine and three aldehydes. The aroma characteristics mainly consisted of roasted and meat-like odors had come into being gradually with the decrease of water activity ( $A_w$ ), and the aroma attributes were the most acceptable at about  $A_w$  0.274 (hot-air drying for 7 h). Four kinds of aroma-active compounds (pyrazines, amines, aldehydes and heterocyclic compounds) made important contributions to the formation of aroma characteristics. The PLSR result showed a good correlation between most variables of volatile compounds, E-nose data and sensory attributes.

**Keywords:** white shrimp; hot-air-drying; aroma attributes; sensory analysis; electronic nose; gas chromatography-mass spectrometry.

**Practical Application:** The current research about aroma attributes during the hot-air-drying process of shrimps provides a theoretical basis for the control of flavor and quality of dried shrimp.

## 1 Introduction

Over the past decades, white shrimp (*Penaeus vannamei*), a high yield economic fishery resource, is widely consumed because of its nutritional values and attractive flavor (Cheok et al., 2017; Kleekayai et al., 2016). Dried shrimp is highly appreciated by consumers for their distinctive aroma, which develops upon the heating process (Cheok et al., 2017; Chung et al., 2019; Mall & Schieberle, 2016, 2017; Zhang et al., 2020a). The drying process is the main step in the production of dried shrimp and also considered as an important step in the formation of the characteristic aroma (Souza & Bragagnolo, 2014; Tachihara et al., 2004; Zhang et al., 2020a). Shrimp aroma determines the individuality of dried shrimp products and is one of the most important criteria to evaluate the quality (Lu et al., 2011; Souza & Bragagnolo, 2014; Zhang et al., 2020a). It is well-known that fresh shrimp have little odor, generally showing a faint grassy, seawater-like odor. After the drying process, dried shrimp produce a characteristic shrimp aroma. N-containing heterocycles, trimethylamine, S-containing compounds and common carbonyl compounds were reported to make contributions to the formation of shrimp-like aroma (Mall & Schieberle, 2016, 2017; Okabe et al., 2019; Rochat et al., 2009; Zhang et al., 2020b). Up to now, most of the studies mainly focused on the composition of volatile compounds in

dried shrimp products, whereas the dynamic changes of aroma attributes during the drying process in dried shrimp products are still not well understood.

Currently, the sensory evaluation of shrimp aroma is still the main method in the shrimp industry. The methodology can be applied to describe various attributes of food samples by recording word descriptions and sensory intensities of trained assessors. Nevertheless, there are some deficiencies in sensory evaluation, e.g., human preference, time-consuming and variability (Calanche et al., 2019; Chen et al., 2019). Aroma components of shrimp products have also been traditionally analyzed using gas chromatography-mass spectrometry (GC-MS). Volatile compounds can be effectively identified to confirm the source of aroma attributes using GC-MS. However, analysis and interpretation of complex data are very time consuming and do not always lead in the correct direction due to the univariate methods and the inherent low selectivity of GC-MS (Gallegos et al., 2017). As a potential alternative to traditional techniques, electronic-nose (E-nose) technology is gaining popularity in the analysis of volatiles. In the last few decades, E-nose has experienced rapid development and played a tremendous role in many fields. The

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<sup>1</sup>College of Food Science and Technology, Guangdong Ocean University, Zhanjiang, China

<sup>2</sup>Guangdong Provincial Key Laboratory of Aquatic Product Processing and Safety, Zhanjiang, China

<sup>3</sup>Guangdong Province Engineering Laboratory for Marine Biological Products, Zhanjiang, China

<sup>4</sup>Guangdong Provincial Engineering Technology Research Center of Marine Food, Zhanjiang, China

<sup>5</sup>Key Laboratory of Advanced Processing of Aquatic Product of Guangdong Higher Education Institution, Zhanjiang, China

\*Corresponding author: jihw62318@163.com

instrument is equipped with an array of metal oxide microbalance sensors, where each element responds to the sensed chemical (Chung et al., 2019; Feng et al., 2011; Chen et al., 2018). In recent years, combined applications of GC-MS, sensory evaluation and E-nose technology in shrimp products were widely reported by several studies (Kleekayai et al., 2016; Zhang et al., 2020b). However, it is still a challenge to match valuable information on the volatiles with sensory attributes.

Multivariate statistical tools, including partial-least-squares regression (PLSR) and principal component analysis (PCA), have been specifically designed for the visualization and analysis of complex sets in different samples (Granato et al., 2018; Zielinski et al., 2020). In many papers, multivariate statistical analysis was widely used to reveal the relationship between the chemical data and sensory attributes, and to identify those chemical components that have an important effect on the overall flavor (Miyazaki et al., 2012; Qin et al., 2013; Viljanen et al., 2014). Previous studies have reported the aroma characterization of shrimp products using sensory analysis, E-nose and GC-MS; however, only limited comprehensive studies have dealt with the correlation between sensory analysis and GC-MS analysis or E-nose data (Rochat et al., 2009; Zhang et al., 2020b). There is no report about the correlation of sensory attributes, GC-MS and E-nose data analysis regarding aroma attributes of dried shrimp products.

This study aimed to analyze aroma attributes change of white shrimp during the hot-air-drying process using sensory analysis, E-nose and GC-MS. The key active-aroma compounds were identified by odor activity value (OAV), and the potential correlations between volatile components, E-nose data and sensory attributes were analyzed by analysis of PLSR. These may provide information in-depth to enhance our understanding of the mechanisms on aroma formation during the hot-air drying process of shrimp.

## 2 Materials and methods

### 2.1 Materials

Raw fresh shrimp (*Penaeus vannamei*, 8-10 cm average length) were purchased at a local supermarket (Zhanjiang, China). Shrimp were covered with ice water to keep them alive, and transported to the laboratory within 1 h. The component of the raw shrimp was protein ( $17.56 \pm 0.47\%$ ), moisture ( $74.82 \pm 1.59\%$ ), fat ( $2.75 \pm 0.23\%$ ), sugar ( $0.87 \pm 0.06\%$ ) and ash ( $3.52 \pm 0.47\%$ ).

### 2.2 Preparation of shrimp samples

Fresh shrimp were killed using crushed ice. Raw shrimp (not peeling) were drained and placed in a hollow metal plate. Shrimp were dried using an Eyela NDO-710 electrothermostatic blast oven (Tokyo Rikakikai Co. Ltd., Tokyo, Japan). The samples were hot-air dried for 0, 1, 2, 3, 4, 5, 6, 7, 8 and 9 h at a constant relative humidity of ca. 20% and an air temperature of 85 °C. Shrimps were uniformly distributed in hollow metal plates ( $15 \text{ kg/m}^2$ ). Each shrimp was flipped every 30 min during the hot-air drying, to ensure both sides heat evenly. Each shrimp sample was frozen with liquid nitrogen and minced to a fine

powder. Water activity ( $A_w$ ) in shrimps at different drying time was determined at 25 °C using a Decagon Aqua Lab meter (Pullman, WA, USA) according to the method of Okpala (2015).

### 2.3 Sensory analysis

The sensory analysis was performed by the quantitative description analysis method. The sensory panel consisted of 11 experienced panelists from Guangdong Ocean University (Guangdong, China), who were well trained according to the ISO standard 8556:2012 (International Organization for Standardization, 2012). These panels showed accumulated sufficient experience and score accuracy for each aroma descriptor after training. A common description vocabulary was generated to characterize aroma attributes, and the characteristic descriptors of shrimp samples were quantified using six sensory descriptors (fishy, smoky/burnt, sweet, caramel, roasted/nutty and cooked-meat-like). The intensity scale was ranked on a scale from 0 (not perceivable) to 5 (strongly perceivable) in steps of 0.1 (Zhang et al., 2020a). The descriptors were compared with aqueous solutions of the following reference odorants (Zhang et al., 2018): fishy ((*Z*)-4-heptenal), smoky/burnt (2-methoxyphenol), cooked-meat-like (3-(methylthio)propionaldehyde), sweet (maltol), roasted/nutty (2,5-dimethylpyrazine) and caramel (4-hydroxy-2,5-dimethyl-3(2*H*)-furanone). For aroma profile analyses, five grams of each sample was weighed into a sealed bottle coded with three digit codes in a random order to prevent bias and equilibrated for 30 min in a water bath at 60 °C. The evaluation was carried out at room temperature and one at a time, with a 5 min wait between samples.

### 2.4 Electronic-nose analysis

The E-nose analysis was performed according to the procedure described by Chen et al. (2018) with some modifications. A commercial PEN3 E-nose system (WinMuster Airsense Analytics Inc., Schwerin, Germany) was used to acquire data on the volatiles. Sensors of PEN3 E-nose respond to representative sensitive compounds (Melucci et al., 2016). Briefly, before detection, 4 g of each sample that came from the same specimen of GC-MS analysis was placed in a 25 mL glass bottle, then capped with a PTFE silicone stopper. After that, the headspace of the sample was equilibrated at room temperature (25 °C) for 20 min, which could avoid sensor drift caused by environmental changes. The measurement phase lasted for 60 s, and the interval for data collection was 1 s. In this work, only the stable values of sensors were used for further data analysis. Each test was performed for three samples and every sample was replicated at least five times until relatively stable results were obtained.

The measured data were analyzed using PCA with the WinMuster software of the E-nose system. Sensor response values obtained from the E-nose were preconditioned with the standard normal variate to eliminate signal drift (Zhu et al., 2019).

### 2.5 Volatile compounds extracted using solid-phase microextraction (SPME)

Extraction of volatile compounds was performed according to Zhang et al. (2020a). The SPME fiber coated with divinylbenzene/

carboxen/polydimethylsiloxane (DVB/CAR/PDMS, 50/30  $\mu\text{m}$ ) (Supelco, Bellefonte, PA, USA) was employed to extract volatile compounds in shrimp samples. The fiber was inserted into the headspace of a 25 mL glass vial that contained 2 g of sample and 2  $\mu\text{L}$  of methyl nonanoate (1.632 g/L in *n*-pentane). Samples were exposed to an SPME fiber with equilibration in a water bath at 65 °C for 40 min. After extraction, the fiber was desorbed at 240 °C for 4 min in the GC-MS injector in splitless mode.

## 2.6 GC-MS analysis

The GC-MS analysis was performed on a QP2010-Plus GC-MS instrument (Shimadzu, Tokyo, Japan). A DB-WAX capillary column (30 m  $\times$  0.25 mm  $\times$  0.25  $\mu\text{m}$ ; Agilent, Santa Clara, CA, USA) was employed. The initial oven temperature was 40 °C maintained for 3 min, then 40-100 °C at a rate of 5 °C/min, then 100-180 °C at 2 °C/min, then raised to 250 °C at 10 °C/min and held there for 5 min. The carrier gas was helium (99.999% purity) at a constant flow of 1.2 mL/min. The mass spectrometer had a mass range of *m/z* 30 to 500 at a scanning rate of 1.8 s<sup>-1</sup>. The electron ionization mode was used with an electron impact energy of 70 eV. Ion source temperature and interface temperature were set at 230 °C and 250 °C, respectively.

## 2.7 Identification and quantitative analysis of volatile compounds

The identification of volatile compounds was carried out by comparing the recorded mass spectra with the Wiley version 6.0 database (Wiley, Chichester, UK) and the NIST 2.0 MS libraries, retention index (RI) and comparing previous literature and published index data. The RIs were calculated from all of the volatile compounds using a C5-C25 *n*-alkanes series (Sigma-Aldrich Trading Co., Ltd., Shanghai, China), and the values were compared, when available, with values reported in the literature for similar chromatographic columns.

The internal standard (IS) method was used to quantify the volatile compounds. The mean value of triplicates was calculated using the following formula, odorant concentration = (compound peak area  $\times$  IS concentration)/IS peak area (Pu et al., 2019; Zhang et al., 2020b). The contribution of each odor to the overall fruit aroma was evaluated by the OAV, which was measured as the ratio of the concentration of each compound to its detection threshold in water. The threshold values were taken from information available according to Gemert (2011).

## 2.8 Statistical analysis

Data analysis was performed using SPSS, version 19 software (SPSS Inc., Chicago, IL, USA). All experiments were performed three times, and mean values were reported. Differences between groups were declared significant at  $p < 0.05$  by ANOVA with Duncan's test.

The correlations between volatile compounds, E-nose data and aroma attributes during the hot-air-drying process of shrimp were analyzed using PLSR through the Unscrambler version 9.7 (CAMO ASA, Oslo, Norway). All variables, such as volatile compounds, E-nose data and sensory scores were centered and

standardized (1/Sdev) before applying PLS analyses and PLSR models were validated using full cross-validation.

## 3 Results and discussion

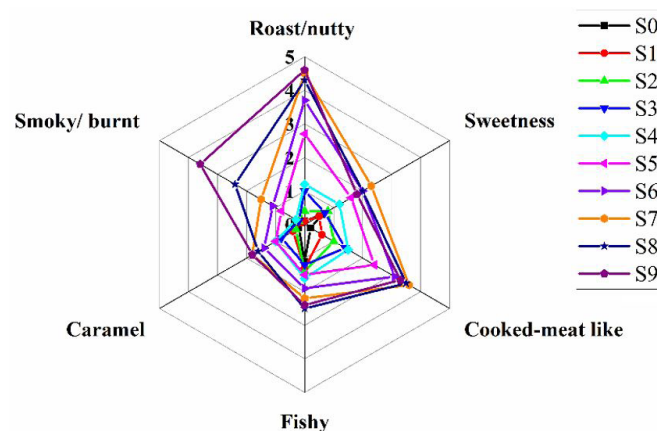
### 3.1 Sensory evaluation

Sensory evaluation is a reliable technology to directly reflect the characteristics and quality of food, and can translate color, odor, taste and texture into visual data (Calanche et al., 2019; Castilhos et al., 2019). A spider plot was created to observe the dynamic changes of aroma profile during the hot-air-drying process of shrimp (Figure 1).

Fresh shrimp had little odor. The score of fishy in S0 was the highest (1.1), followed by sweet (0.6), and scores of other aroma attributes were low. During the early period (0-2 h), there was no obvious change in sweet, fishy, caramel and smoky/burnt odors while scores of roast/nutty and cooked-meat-like odors increased slightly, which indicated that the overall aroma of shrimp changed little at the inception stage. During the hot-air drying for 2-7 h, scores of six aroma attributes increased significantly ( $p < 0.05$ ) compared with S0. In particular, scores of roast/nutty and cooked-meat-like odors were much stronger than other aroma attributes, which indicated that they were the main aroma characteristics of shrimp in the middle time of hot-air drying. During the late period (7-9 h), scores of most aroma attributes changed little. It was worth noting that the score of smoky/burnt odor in S8 and S9 increased significantly ( $p < 0.05$ ) compared with that of S7. The overall aromas of S8 and S9 due to the addition of smoky/burnt odor were unacceptable. The aroma attributes as a whole showed S7 were much better than the others.

### 3.2 Water activity

Water is an important medium for various chemical reactions, and most of the flavor precursors are water-soluble, so  $A_w$  played an important role in the formation of aroma compounds. Previous research had reported that the meat-like flavor components were mainly derived from Maillard reaction, and the water activity could qualitatively affect the kind and amount of major volatiles produced during heating, which made contributions to



**Figure 1.** Radar map of aroma profiles during the hot-air-drying process of shrimp. S0, S1, S2, S3, S4, S5, S6, S7, S8 and S9 represent hot-air-dried shrimp for 0 h, 1 h, 2 h, 3 h, 4 h, 5 h, 6 h, 7 h, 8 h and 9 h, respectively.

the formation of aroma characteristics (Hartman et al., 1984). Figure 2 showed that the  $A_w$  change in shrimps with the drying time. At the early process (0-2 h), the  $A_w$  of shrimps decreased slowly from 0.971 to 0.916. At the middle process (2-7 h), the  $A_w$  decreased rapidly from 0.916 to 0.274. At the late process (7-9 h), the  $A_w$  changed little (0.274-0.255). According to the result of sensory evaluation, the aroma attributes of shrimps at 7 h were considered as the most acceptable during drying process, meanwhile, the  $A_w$  0.274 at 7 h was appropriate for long time storage of dried shrimp. Therefore, the stage could be used as the optimal condition for the aroma formation.

### 3.3 E-nose analysis

#### *E-nose response to shrimp samples' aroma during the hot-air-drying process*

The aroma characteristics of shrimps during at different drying time were analyzed using E-nose equipped with ten sensors,

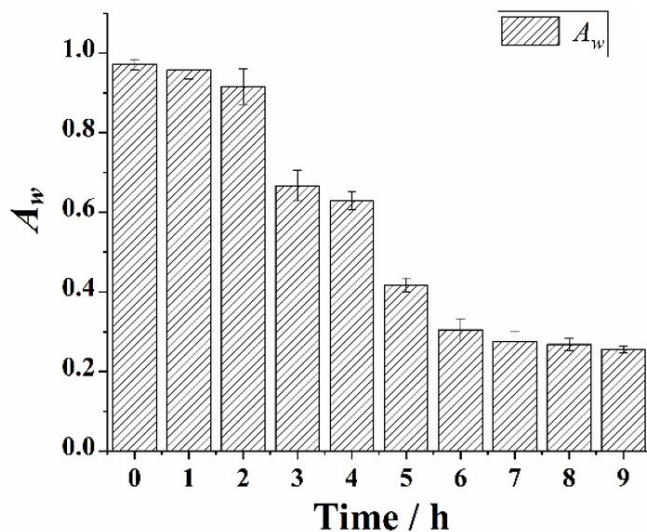


Figure 2. The  $A_w$  change in shrimps with the drying time.

which depended on not only the concentration of molecules in odors, but also what odor molecules consist of Chen et al. (2018). Figure 3a shows polar graphs of the responses of the sensors to the 10 shrimp samples during the hot-air-drying process. Fresh shrimp samples (S0) represented a low response on all sensors, which indicated that fresh shrimp had little odor. During the early process (0-2 h), most of the sensors in S1 and S2 changed little while sensor W1W and sensor W3C increased a bit compared with S0. Radar chart shapes changed as the hot-air drying proceeded (2-7 h), it was observed that responses of all sensors increased to different extents in S2 to S7, especially sensor W1W, sensor W2W, sensor W1S and sensor W2S. This indicated that an abundance of aroma compounds was produced in shrimp during the late stage of the hot-air-drying process. In addition, the radar chart shapes of S7, S8 and S9 were quite close, which suggested that these three samples might have similar aroma attributes during the late period (7-9 h).

#### *Classification of aroma attributes of shrimp during the hot-air-drying process using PCA*

Principal component analysis is a statistical technique for the reduction of input data dimension and is largely used for feature extraction. It captures the relevant information in a set of input data providing a lower dimension (Fernandes et al., 2019; Nascimento et al., 2020). For improved visualization of the data, PCA was performed to distinguish aroma attributes of 10 shrimp during the hot-air-drying process (Figure 3b). The contribution of the first two PCs (PC1 and PC2) reflects the completeness of the variable information based on PC1 and PC2. Most data points of shrimp samples (S0, S1 and S2) were distributed in the third quadrants, which indicated that aroma attributes of shrimp have not substantially changed at an early stage of hot-air drying. During the middle period (2-7 h), data points of shrimp samples were distributed in the first and second quadrants, which suggested that aroma attributes of shrimp were obviously different from fresh shrimp at this stage. In particular, data points of shrimp samples (S7, S8 and S9) were distributed in

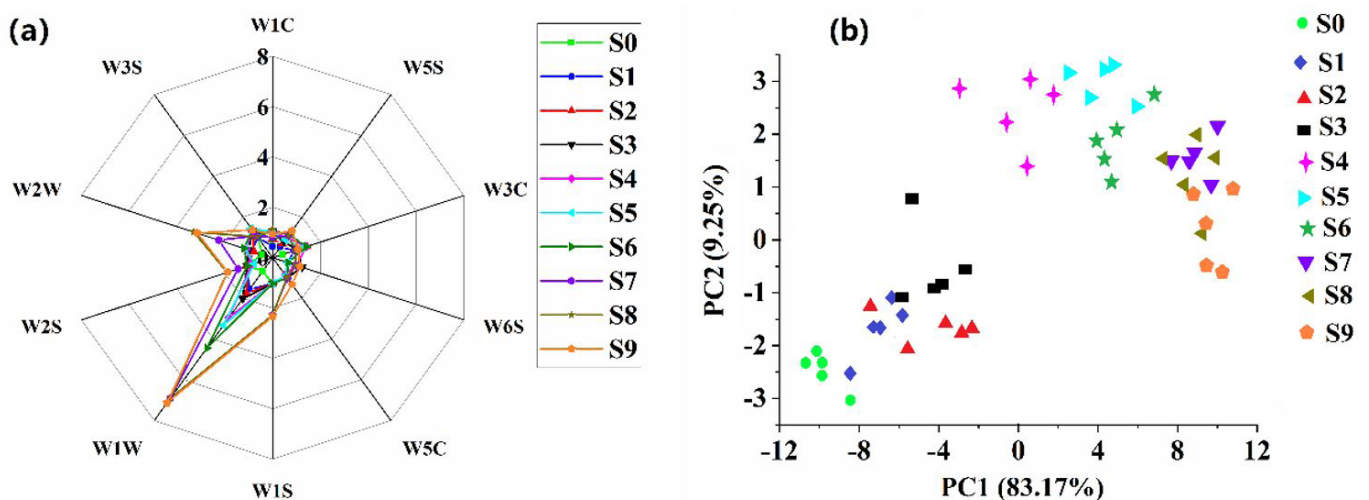


Figure 3. Radar map (a) and PCA analysis (b) of the E-nose during the hot-air-drying process of shrimp. S0, S1, S2, S3, S4, S5, S6, S7, S8 and S9 represent hot-air-dried shrimp for 0 h, 1 h, 2 h, 3 h, 4 h, 5 h, 6 h, 7 h, 8 h and 9 h, respectively.

a fairly small band, and these three samples might have similar aroma attributes. According to the GC-MS results, an abundance of aroma compounds was produced during the hot-air-drying process for 2-7 h, which may dramatically change the overall aroma of shrimp. E-nose analysis was consistent with the result of sensory evaluation.

### 3.4 GC-MS analysis

Shrimp collected during the hot-air-drying process were analyzed using GC-MS to illuminate the dynamic changes of volatile compounds. A total of 79 volatile compounds were identified and quantified, including two S-containing compounds, 15 pyrazines, 16 ketones, 17 hydrocarbons, three amines, 10 alcohols, three heterocyclic compounds, nine aldehydes and four esters (Table 1). As a whole, species and contents of volatile compounds varied greatly during the process. During the hot-air drying for 0-9 h, pyrazines, hydrocarbons and heterocyclic compounds were the three most variable compounds. In the fresh shrimp, hydrocarbons, ketones and aldehydes were the most abundant compounds, which accounted for 94.06%. During the hot-air drying for 0-2 h, most of the compounds in S1 and S2 were comparatively close to fresh shrimp while a few pyrazines were produced (lower than 10 ng/g), the content of ketones decreased significantly ( $p < 0.05$ ), and the content of amines increased significantly ( $p < 0.05$ ). During the hot-air drying for 2-7 h, the kind and number of volatile compounds in shrimp samples increased drastically from 1667.73 ng/g (S2) to 17891.31 ng/g (S9). At this stage, pyrazines, ketones, amines, aldehydes, S-containing compound and heterocyclic compounds increased rapidly. Most of these compounds have low thresholds, and they contributed to the aroma characteristics in hot-air-dried shrimp. At the late period (7-9 h), large amounts of S-containing compounds and heterocyclic compounds were detected, but the number of volatile compounds changed little.

OAVs of volatile compounds in the hot-air-dried shrimp were calculated to identify the contributors to the aroma profile. Volatile compounds with  $OAV \geq 1$  (based on the published odor thresholds determined in water) are identified as aroma-active compounds (AACs), and AACs in shrimps at different drying time were shown in Table 2. About 4, 4, 2, 5, 7, 11, 16, 16, 17 and 15 kinds of AACs were identified at 0 h-, 1 h-, 2 h-, 3 h-, 4 h-, 5 h-, 6 h-, 7 h-, 8 h- and 9 h-dried samples, respectively. Drying process of shrimps was divided into three periods according to the AACs changes, early period: 0-2 h, middle period: 2-7 h, and late period: 7-9 h.

#### Early period (0-2 h)

In fresh shrimp (S0), OAV summation was only 21.41, and trimethylamine, benzaldehyde, caproic aldehyde and amylaldehyde were the main AACs (accounting for 95.07%). Trimethylamine comprised 60.04% of the total OAVs in fresh shrimp, and the main source of the odors. During the hot-air drying for 0-2 h, OAV summation increased slightly, and the OAV summations of AACs in S1 and S2 were 43.31 and 93.45, respectively. However, the composition of AACs in the two samples changed little compared with S0, the main sources in AACs were mainly of

rapidly increasing trimethylamine (accounted for 74.31-92.61% of the total), with a few pyrazines (accounted for 7.45-20.24%).

#### Middle period (2-7 h)

The content and amount of AACs changed significantly during this period, and the OAV summation increased rapidly from 93.45 (S2) to 752.91 (S7). These changes were mainly attributed to the increase of pyrazines, amines, aldehydes, and heterocyclic compounds by more than 85% in the OAV increment.

It is reported that pyrazines are important aroma compounds in shrimp products subjected to drying or heating treatment (Neethling et al., 2016; Tachihara et al., 2004; Zhang et al., 2020a). Pyrazines are derived from the Maillard reaction, which commonly have popcorn, peanut, roasted, and meat-like odors. Pyrazines' OAV showed the fastest increase, and reached to (549.79) at hot-air drying for 7 h (S7), which were increased by 86.15 times compared with S2. 3-Ethyl-2,5-dimethylpyrazine, 2,5-dimethylpyrazine, 2,3,5-trimethylpyrazine and 2-methyl-3,5-diethylpyrazine were the highest OAV AACs in S7, which made important contributions to the formation of aroma attributes.

Amines are mainly produced from degradation of N-containing organic compounds (Gu et al., 2013; Fan et al., 2017). Trimethylamine was the only amine in AACs, which could be found in all samples. It is very common in seafood products and usually regarded as a reduction product of trimethylamine oxide. Trimethylamine contributes to fishy and seawater-like odors according to previous papers (Mall & Schieberle, 2016; Zhang et al., 2020b). Though OAVs of trimethylamine represented a substantial increase as the hot-air drying proceeded (2-7 h), the proportion in total constantly decreased from 74.30% to 22.26%.

Aldehydes are important volatile flavor compounds in aquatic products, and produced from the deamination of amino acids. OAVs of benzaldehyde and 3-(methylthio)propionaldehyde obviously increased as the hot-air drying proceeded, and OAV summation of the two compounds accounted for 6.44% of the total in S7. 3-(methylthio)propionaldehyde contributed to cooked-meat-like and onion odors, and benzaldehyde has an unpleasant almond odor.

Heterocyclic compounds are well-known as the common flavor components in the thermal treatment of meats and aquatic products. Furan compounds are important heterocyclic compounds and generally contribute to milk, cooked-meat-like, fat and roasted potato flavors (Shahidi, 1998; Zhang et al., 2019, 2020a). During the middle period, two heterocyclic compounds in AACs were identified as 2-pentylfuran and pyridine. Pyridine contributed to an unpleasant odor and had a negative effect on the aroma attributes. In general, the hydrocarbons, phenols, alcohols, acids and esters are generally considered to make little contribution because of their high odor thresholds.

#### Late period (7-9 h)

It is observed that compositions and contents of AACs during this period were close, OAV summations were 752.91 (S7), 844.59 (S8) and 896.95 (S9), and they had 12 AACs in common. Four volatile compounds (3-(methylthio)propionaldehyde, trimethylamine,

**Table 1.** Concentration of volatile compounds quantified during the hot-air-drying process of shrimp.

Compounds	Identification	RI	Concentration(ng/g)									
			S0	S1	S2	S3	S4	S5	S6	S7	S8	S9
Pyrazines (15)												
3-Ethyl-2,5-dimethylpyrazine	RI, MS	1437	ND	ND	ND	2.41i	45.62e	8.64f	156.87d	297.61b	303.85a	293.74c
2,5-Dimethylpyrazine	RI, MS	1317	ND	2.58i	4.7h	20.344g	66.848e	27.424f	95.736d	149.26c	176.952a	166.768b
2,3,5-Trimethylpyrazine	RI, MS	1397	ND	ND	ND	276.18g	1051.38e	1528.51d	677.16f	2880.91b	3130.38a	2741.31c
2-Methyl-3,5-diethylpyrazine	RI, MS	1484	ND	ND	ND	ND	76.88e	ND	412.26b	324.31d	364.27c	505.85a
2,3-Dimethylpyrazine	RI, MS	1343	ND	ND	ND	ND	33.71d	ND	762.09b	420.28c	ND	1158.28a
2,3,5,6-Tetramethylpyrazine	RI, MS	1468	ND	ND	ND	ND	192.62e	ND	666.12c	396.13d	948.65b	1387.81a
2,6-Dimethyl pyrazine	RI, MS	1337	ND	ND	ND	166.55c	ND	496.07a	ND	170.16b	ND	ND
2-Ethyl-6-methylpyrazine	RI, MS	1379	ND	ND	ND	ND	ND	ND	302.43b	235.24c	684.89a	180.29d
2-Isoamyl-6-methylpyrazine	MS	1264	ND	ND	ND	450.32d	830.54b	830.54b	630.28c	270.33e	ND	1350.45a
Methylpyrazine	RI, MS	1330	ND	ND	ND	315.22d	ND	ND	840.18a	630.45b	220.51e	346.54c
Ethyl pyrazine	RI, MS	1385	ND	ND	ND	45.41c	ND	ND	77.63c	87.52b	157.85a	33.19d
Vinyl pyrazine	MS	2190	ND	ND	ND	ND	ND	ND	15.27c	48.97c	176.85a	115.48b
2-Ethyl-5-methylpyrazine	RI, MS	1385	ND	ND	ND	ND	ND	ND	118.79a	ND	ND	89.07b
3,6-diethyl-2-methylpyrazine	RI, MS	2190	ND	ND	ND	ND	ND	ND	ND	ND	164.62	ND
2-Vinyl-6-methylpyrazine	MS	2097	ND	ND	ND	ND	ND	ND	ND	78.89b	ND	121.37a
Hydrocarbons (17)												
Tridecane	RI, MS	1098	124.09g	152.83f	354.41b	81.23h	538.40a	205.65c	167.22e	20.46i	ND	174.8d
Dodecane	MS	1198	76.34g	277.86c	163.82e	77.85f	ND	ND	235.41d	26.44h	426.85a	341.27b
Undecane	RI, MS	1098	21.43g	66.08d	48.54e	ND	12.35h	111.59a	86.59c	89.54b	28.18f	ND
Tetradecane	RI, MS	1396	215.62a	ND	121.86b	ND	ND	ND	ND	63.48d	ND	89.54c
Pentadecane	RI, MS	1496	35.18e	ND	ND	ND	41.09c	ND	212.35b	335.97a	13.42f	37.29d
2,6,10-Trimethyl-tetradecane	RI, MS	1549	178.49b	188.31a	ND	ND	ND	65.58d	ND	124.78c	ND	ND
Hexadecane	RI, MS	1596	ND	ND	ND	312.54a	33.24d	ND	ND	ND	67.59c	225.78b
2,6,10-Trimethyl-pentadecane	RI, MS	1644	ND	ND	ND	ND	ND	39.71b	107.25a	13.27c	9.18d	ND
2-Methyl-hexadecane	RI, MS	1660	356.19a	87.89d	55.27f	176.59b	54.76fg	ND	ND	78.41e	ND	129.96c
Heptadecane	RI, MS	1696	ND	ND	129.35a	123.21b	ND	ND	ND	74.03c	ND	ND
2,6,10,14-Tetramethyl-pentadecane	RI, MS	1701	56.75d	251.63a	ND	33.45f	78.69c	128.85b	45.32e	ND	ND	ND
2-Methyl-heptadecane	RI, MS	1760	ND	ND	ND	26.76c	ND	ND	ND	53.32b	232.16a	19.97d
Octadecane	RI, MS	1796	88.29c	ND	ND	ND	256.79a	ND	ND	95.86b	ND	87.59cd
2,6,10,14-Tetramethyl-hexadecane	RI, MS	1805	ND	ND	43.23f	135.68a	ND	46.39e	77.28d	85.39c	133.18b	ND
Nonadecane	RI, MS	1895	ND	331.25a	ND	ND	18.48d	ND	ND	ND	78.25b	43.26c
Eicosane	RI, MS	1998	115.54a	ND	ND	ND	ND	ND	95.26b	ND	ND	ND
Heneicosane	RI, MS	2097	ND	ND	189.89c	ND	ND	328.14a	ND	236.68b	ND	ND

Different letters indicate significant differences ( $p < 0.05$ ) in the same row. MS, mass spectrum; RI, retention index; ND, not detected. S0, S1, S2, S3, S4, S5, S6, S7, S8 and S9 represent hot-air-dried shrimp for 0 h, 1 h, 2 h, 3 h, 4 h, 5 h, 6 h, 7 h, 8 h and 9 h, respectively.

Table 1. Continued...

Compounds	Identification	RI	Concentration(ng/g)									
			S0	S1	S2	S3	S4	S5	S6	S7	S8	S9
Ketones (16)												
2-Octanone	RI, MS	1063	ND	32.76d	ND	ND	ND	ND	127.92a	97.55b	70.98c	ND
2-Heptanone	RI, MS	1186	ND	50.76f	ND	83.19e	ND	186.12d	ND	408.9b	477.99a	354.29c
3-Hydroxy-2-butanone	RI, MS	1278	78.16c	ND	ND	60.25d	ND	ND	ND	78.96b	ND	127.09a
2-Cyclohexen-1-one	RI, MS	934	ND	ND	33.26a	ND	ND	ND	23.23b	ND	18.09c	9.19d
2,3-octanedione	RI, MS	987	56.34a	ND	ND	9.43f	ND	31.28c	54.09b	11.26e	25.24d	ND
3-Octen-2-one	RI, MS	1040	ND	22.58c	ND	ND	69.02b	ND	ND	ND	ND	111.24a
2-Nonanone	RI, MS	1094	ND	ND	ND	55.67a	ND	ND	ND	34.42c	43.09b	ND
3-Nonen-2-one	RI, MS	1140	ND	ND	18.65	ND	73.28	ND	42.86	ND	ND	48.98
3-Hexanone	MS		ND	ND	ND	ND	ND	ND	37.24b	42.59a	ND	ND
2-Heptadecanone	RI, MS	1901	ND	ND	ND	117.25a	55.33d	18.42e	ND	6.48f	85.87b	69.98c
2-Decanone	RI, MS	1195	88.24a	ND	58.35c	ND	ND	ND	18.55d	ND	ND	85.41b
2,3-Butanedione	MS		ND	68.98a	ND	20.78c	ND	ND	ND	5.89d	24.19b	ND
2-Undecanone	RI, MS	1592	52.29c	17.58e	ND	ND	ND	ND	40.21d	83.16a	ND	76.55b
2-Pentadecanone	RI, MS	2016	ND	ND	45.56b	ND	ND	33.29d	ND	63.43a	38.97c	ND
2,3-Octanone	MS		ND	ND	ND	ND	ND	41.75b	ND	ND	ND	48.98a
6-Methyl-5-heptane-2-ketone	MS		ND	ND	ND	ND	9.01c	ND	11.25b	17.86a	5.09d	ND
Amines (3)												
Trimethylamine	RI, MS	702	32.16j	77.232i	207.72h	278.088f	25.112g	370.224e	422.13c6	402.24d	430.272b	468.672a
Dimethylamine	MS	<600	8.19i	19.74f	65.42a	45.26b	ND	18.24g	35.21c	26.89e	11.25h	32.39d
N-nitrosodimethylamine	RI, MS		15.27c	55.38a	39.09b	ND	8.07f	ND	ND	8.85e	10.28d	ND
Esters (4)												
Methyl 2-methylbutanoate	RI, MS	1018	ND	0.61a	0.17c	ND	ND	ND	ND	0.44b	ND	0.56ab
Ethyl butyrate	RI, MS	804	ND	ND	ND	ND	ND	ND	6.4a	4.6b	2.85c	ND
Acetic ether	RI, MS	885	ND	ND	ND	ND	0.95a	ND	ND	0.43b	ND	ND
Ethyl 2-methylbutanoate	RI, MS	1059	ND	ND	0.14d	ND	ND	ND	0.82b	ND	1.18a	0.56c
Alcohols (10)												
1-Pentene-3-ol	RI, MS	1151	22.58a	10.22c	ND	ND	ND	3.28e	ND	ND	6.25d	18.11b
1-Pentanol	RI, MS	770	ND	9.85d	ND	ND	31.42a	ND	ND	16.54c	21.28b	ND
1-Octanol	RI, MS	1072	9.64e	ND	18.24c	ND	19.54b	ND	3.21f	ND	10.28d	30.97a
Alcohol	RI, MS	925	ND	15.26d	ND	4.17g	25.57b	10.87e	7.18f	26.83a	ND	15.82c
3-Methyl-3-buten-1-ol	RI, MS	1238	5.23c	ND	25.12a	10.22b	ND	ND	ND	ND	ND	ND
Pentanol	RI, MS	1243	ND	ND	18.29b	8.43e	ND	9.24	13.24c	42.56a	12.25d	5.45f
3-Octanol	RI, MS	1397	ND	5.13d	ND	ND	7.38c	ND	ND	ND	18.42a	11.86b
Undecanol	RI, MS	1389	11.25c	ND	9.12d	ND	ND	ND	25.17b	4.38e	31.28a	ND
1-Heptanol	RI, MS	1461	ND	26.27a	ND	18.55b	4.21e	18.27c	ND	2.28f	ND	7.41d
Methanethiol	MS		ND	ND	7.11b	ND	ND	ND	ND	16.13a	ND	5.24c

Different letters indicate significant differences ( $p < 0.05$ ) in the same row. MS, mass spectrum; RI, retention index; ND, not detected. S0, S1, S2, S3, S4, S5, S6, S7, S8 and S9 represent hot-air-dried shrimp for 0 h, 1 h, 2 h, 3 h, 4 h, 5 h, 6 h, 7 h, 8 h and 9 h, respectively.

Table 1. Continued...

Compounds	Identification	RI	Concentration(ng/g)									
			S0	S1	S2	S3	S4	S5	S6	S7	S8	S9
Aldehydes (9)												
Benzaldehyde	RI, MS	1496	60.76g	119.84e	ND	ND	34.72h	90.44f	208.88c	187.66d	610.43b	719.62a
3-Methylbutyraldehyde	RI, MS	916	ND	31.86f	ND	ND	33.63e	ND	111.51d	165.2b	208.86a	163.43c
Caproaldehyde	RI, MS	1080	5.32a	1.84f	1.28g	ND	ND	ND	3.88b	2.56d	2.25e	2.96c
Amyldehyde	MS	1001	38.88a	ND	ND	ND	ND	ND	5.49b	ND	ND	ND
3-(Methylthio)propionaldehyde	RI, MS	1432	ND	ND	ND	ND	ND	9.02e	20.44d	83.56a	70.45c	80.84b
2-Methylhexanal	MS		ND	ND	ND	ND	ND	182.28d	ND	361.23a	327.61b	218.43c
2-Methylpropionaldehyde	RI, MS	912	ND	ND	ND	3.83b	2.51c	2.51c	5.63a	3.83b	ND	1.12d
2-Methylbutyraldehyde	RI, MS	916	4.18d	ND	9.17c	21.74b	ND	ND	ND	ND	49.29a	ND
Butyraldehyde	MS		ND	10.21e	ND	31.54c	ND	ND	26.19d	49.35b	ND	77.69a
Heterocyclic compounds (3)												
2-Pentylfuran	RI, MS	1224	ND	ND	ND	ND	ND	42.05d	30.57e	49.88c	64.96b	120.06a
Pyridine	RI, MS	1181	ND	ND	ND	ND	ND	ND	460.88d	3219.53c	3801.19b	5213.65a
2-Aminopyridine	MS		ND	ND	ND	ND	ND	2.12e	10.18d	23.51b	25.56a	17.82c
<b>S-containing compounds (2)</b>												
Dimethyl disulfide	RI, MS	802	ND	ND	ND	ND	ND	0.14e	0.74d	3.24c	18.28b	40.22a
Methyl propyltrisulfide	RI, MS	1083	ND	ND	ND	ND	ND	ND	13.42d	23.27c	43.82b	65.28a

Different letters indicate significant differences ( $p < 0.05$ ) in the same row. MS, mass spectrum; RI, retention index; ND, not detected. S0, S1, S2, S3, S4, S5, S6, S7, S8 and S9 represent hot-air-dried shrimp for 0 h, 1 h, 2 h, 3 h, 4 h, 5 h, 6 h, 7 h, 8 h and 9 h, respectively.



Table 2. Odor thresholds and aroma-active compounds during the hot-air-drying process of shrimp.

Code	Compounds	Threshold(ng/g)	Odorant description	OAV									
				S0	S1	S2	S3	S4	S5	S6	S7	S8	S9
Pyrazines (10)													
1	3-Ethyl-2,5-dimethyl-pyrazine	1	Roasted, smoke	<1	<1	<1	2.41g	8.64f	45.62e	156.87d	297.61b	303.85a	295.74c
2	2,5-Dimethyl pyrazine	0.8	Nutty, roasted	<1	3.23i	5.87h	25.43g	34.28f	83.56e	119.67d	186.5c	221.19a	208.46b
3	2,3,5-Trimethylpyrazine	297	Nutty, caramel	<1	<1	<1	1.94g	6.83d	3.54e	2.28f	9.71b	10.54a	9.23c
4	2-Methyl-3,5-diethylpyrazine	60	Smoke, roasted	<1	<1	<1	<1	<1	1.28e	6.87b	5.4d	6.07c	8.43a
5	2,3-Dimethyl pyrazine	300	Nutty, roasted, meat	<1	<1	<1	<1	<1	<1	2.54b	1.43c	ND	3.86a
6	2,3,5,6-Tetramethylpyrazine	180	Coffee, roasted	<1	<1	<1	1.83b	2.48a	1.07e	3.7c	2.2d	5.27b	7.71a
7	2,6-Dimethyl pyrazine	200	Roasted,	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
8	2-Ethyl-6-methylpyrazine	240	Potato, roasted	<1	<1	<1	<1	<1	<1	1.26b	<1	2.85a	<1
9	2-Isoamyl-6-methylpyrazine	9000	Fatty, coffee, nutty	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
10	Methylpyrazine	10500	Fatty, roasted	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
Hydrocarbons (2)													
11	Tridecane	2040	Unpleasant, rubber	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
12	Dodecane	2140	Unpleasant, woody	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
Ketones (3)													
13	2-Octanone	39	Green, grass	<1	<1	<1	<1	<1	<1	3.28a	2.5b	1.82c	<1
14	2-Heptanone	141	Vegetable, fruity,	<1	<1	<1	<1	<1	1.32c	<1	2.9b	3.39a	<1
15	3-Hydroxy-2-butanone	800	Milk, sweet	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
Amines (1)													
16	Trimethylamine	2.4	Grass, fishy	13.4fj	32.18i	86.55h	115.87f	104.63g	154.26e	175.89c	167.6d	179.28b	195.28a
Esters (4)													
17	2-Methyl-Butyrate ethyl	0.5	Sweet, fatty	<1	1.21a	<1	<1	<1	<1	<1	<1	<1	1.12a
18	Ethyl butyrate	5	Sweet, fruity	<1	<1	<1	<1	<1	<1	1.28a	<1	<1	ND
19	Acetic ether	0.9	Vegetable, fruity	<1	<1	<1	<1	1.05a	<1	<1	<1	<1	<1
20	Ethyl 2-methylbutyrate	1.2	Metallic, fatty	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
Aldehydes (7)													
21	Benzaldehyde	28	Unpleasant, metallic	2.17g	4.28e	<1	<1	1.24h	3.23f	7.46c	6.7d	21.8b	25.7a
22	3-Methylbutyraldehyde	59	Vegetable, fruity	<1	<1	<1	<1	<1	<1	1.89c	2.8b	3.54a	2.77b
23	Caproaldehyde	4	Grass, creamy	1.33a	<1	<1	<1	<1	<1	<1	<1	<1	<1
24	Amylaldehyde	9	Fruity	4.32a	<1	<1	<1	<1	<1	<1	<1	<1	<1
25	3-(Methylthio) propionaldehyde	2	Potato, meat	<1	<1	<1	<1	<1	4.51e	10.22d	41.78a	35.21c	40.44b
26	2-Methylhexanal	84	Leather, smoke	<1	<1	<1	<1	<1	2.17d	ND	4.33a	3.90b	2.62c
27	2-Methylpropionaldehyde	4.4	Malt	<1	<1	<1	<1	<1	<1	1.28a	<1	<1	<1
Heterocyclic compounds (2)													
28	2-Pentylfuran	5.8	Potato, fruity, metallic	<1	<1	<1	<1	<1	7.25d	5.27e	8.6c	11.2b	20.7a
29	Pyridine	2000	Unpleasant, metallic	<1	<1	<1	<1	<1	<1	<1	1.6c	1.9b	2.6a
S-containing compounds (1)													
30	Dimethyl disulfide	0.6	Sulfury, unpleasant	<1	<1	<1	<1	<1	<1	1.24d	5.4c	13.8b	33.7a

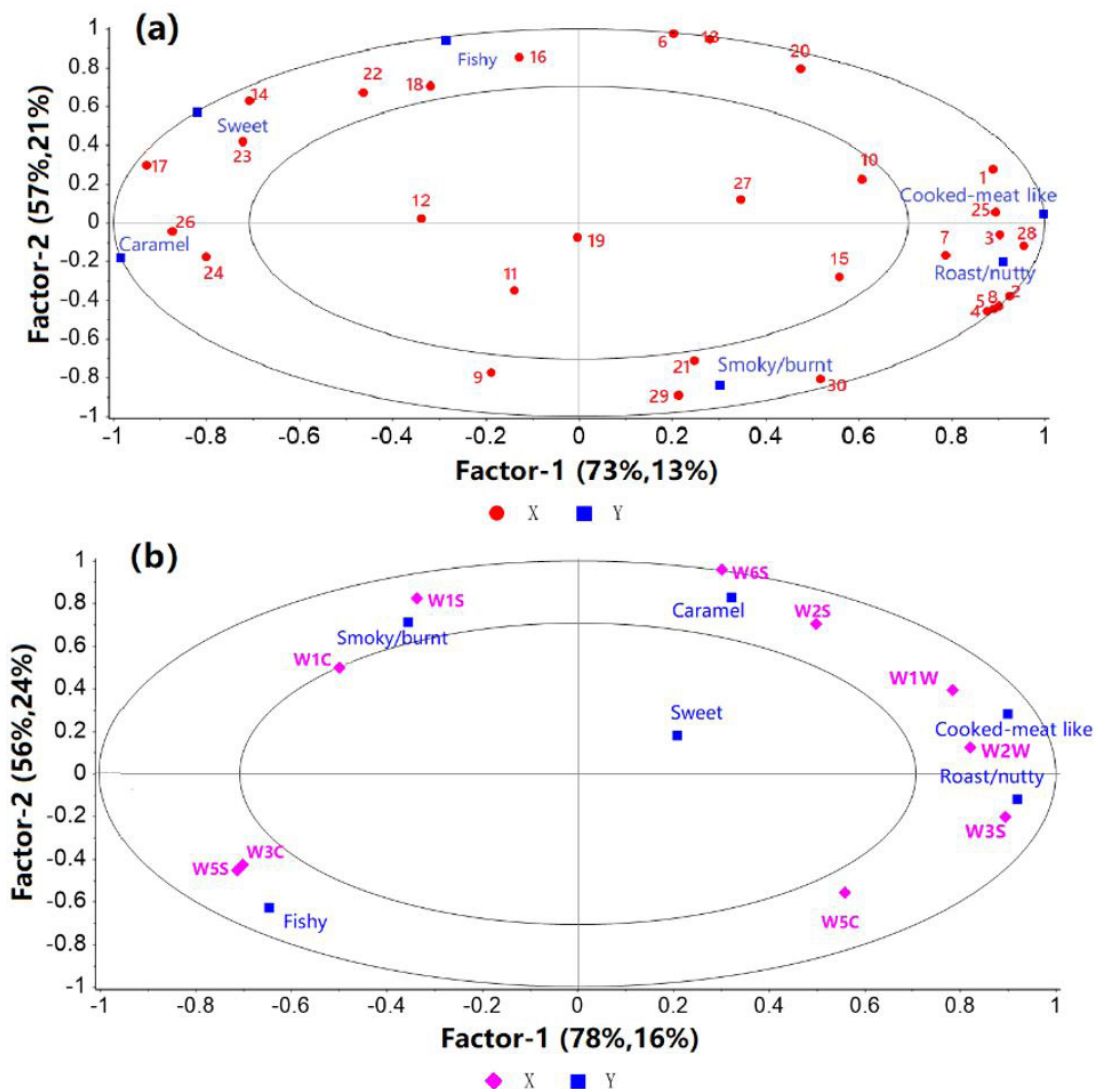
Different letters indicate significant differences ( $p < 0.05$ ) in the same row. S0, S1, S2, S3, S4, S5, S6, S7, S8 and S9 represent hot-air-dried shrimp for 0 h, 1 h, 2 h, 3 h, 4 h, 5 h, 6 h, 7 h, 8 h and 9 h, respectively.

2,5-dimethylpyrazine and 3-ethyl-2,5-dimethylpyrazine) were the highest AACs of the three samples, and they accounted for more than 85% of the total. This indicated that S7, S8 and S9 had similar aroma attributes. It is worth noting that OAVs of benzaldehyde in S8 and S9 increased by 2.25 and 2.84 times that of S7, respectively. Benzaldehyde has an unpleasant almond and smoky odors, and too high concentration would lead to the deterioration of the overall aroma (Cai et al., 2016). This phenomenon explained the reason for the increase of smoky odor during the late period. GC-MS results were consistent with results of sensory evaluation and E-nose.

In conclusion, aroma compounds of shrimps apparently changed during the hot-air drying process. The middle period (2-7 h) was the crucial period for the development of aroma characteristic of dried shrimps, which gradually converted trimethylamine and aldehydes of fresh shrimps to pyrazines, amines, aldehydes and heterocyclic compounds of dried shrimps.

### 3.5 Correlation between GC-MS results, E-nose data and sensory attributes

In an attempt to study the relationships between sensory attributes, E-nose data and volatile compounds, PLSR models were performed. As shown in Figure 4a, the X-matrix was projected as volatile compounds with thresholds; the Y-matrix was projected as six sensory properties (roasted/nutty, smoky/burnt, sweet, cooked-meat-like, fishy and caramel). The derived PLSR model explained 82% of the variance in X and 86% of the variance in Y. The inner ellipse showed that 50% of the explained variance and the outer ellipse showed 100% of the explained variance (Kovács et al., 2010). Most of the sensory attributes and volatile compounds were located between the small and big ellipses, except methylpyrazine, tridecane, dodecane, 3-hydroxy-2-butanone, acetic ether and 2-methylpropionaldehyde. 3-Ethyl-2,5-dimethylpyrazine, 2,5-dimethylpyrazine, 2,3,5-trimethylpyrazine, 2-methyl-



**Figure 4.** PLSR correlation loadings plot of indicator variables of volatile compounds, E-nose data and sensory evaluation (1-30 denote the 30 compounds defined in Table 2) during the hot-air-drying process of shrimp. (a) The model was derived from volatile compounds as the X-matrix (red point) and sensory attributes as the Y-matrix (blue point); (b) The model was derived from the signals of the E-nose as the X-matrix (purple point) and sensory attributes as the Y-matrix (blue point). The small and big ellipses represent  $R^2 = 50$  and 100%, respectively.

3,5-diethylpyrazine, 2,3-dimethylpyrazine, 2,6-dimethylpyrazine, 2-ethyl-6-methylpyrazine, 3-(methylthio)propionaldehyde and 2-pentylfuran had correlations with cooked-meat-like and roasted/nutty odors. Trimethylamine, ethyl butyrate and 3-methylbutyraldehyde have correlations with fishy odor. 2-Heptanone, ethyl 2-methylbutyrate and caproic aldehyde showed positive correlations with sweet odors. Amyl aldehyde and 2-methylhexanal have correlations with caramel odor. Benzaldehyde, pyridine and dimethyl disulfide have correlations with smoky/burnt odor. PLSR were consistent with results of sensory evaluation and AACs.

In Figure 4b, the X-matrix was designed as the signal values from the E-nose and designated as independent variables, six sensory properties were designated as dependent variables. The derived PLSR model included two significant PCs explaining most of the E-nose data and sensory attributes, except sweet odor. Variables of W1W, W2W and W3S from the X-matrix have correlations with roasted/nutty and cooked-meat-like odors from the Y-matrix. Variables of W5S and W3C showed positive correlations with a fishy odor. Variables of W6S, W2W and W2S showed positive correlations with caramel odor. Variables of W1C and W1S showed a good correlation with smoky/burnt odor. PLSR were consistent with results of E-nose, sensory evaluation and AACs.

#### 4 Conclusions

The aroma characteristics of shrimps changed significantly during the hot-air-drying process. Along with hot-air drying, the aroma intensity in shrimp increased while the aroma characteristics including mainly roasted and meat-like odors had come into being gradually. In shrimp with  $A_w$  0.274 (hot-air drying for 7 h), the number of AACs increased to 16, and the aroma attributes as a whole were much better than the others. Four kinds of AACs made important contributions, namely, pyrazines, amines, aldehydes and heterocyclic compounds. The results will provide a theoretical basis for the control of flavor and quality of hot-air-dried shrimp during the drying process.

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