**PROCESSING AND PRODUCTS**

Characterization of the physicochemical changes and volatile compound fingerprinting during the chicken sugar-smoking process

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**ABSTRACT** Sugar-smoking contributes to improving flavor attributes of meat products. However, there is rather limited information concerning the relationship between sugar-smoking process parameters and volatile compound (VC) fingerprinting as well as related quality attributes of sugar-smoked chicken. In this work, the changes in VC across the whole sugar-smoking process were determined and analyzed and physicochemical properties, free fatty acid, thiobarbituric acid reactive substances values, and E-nose were also performed to characterize the quality properties of sugar-smoked chicken breast (CB) and chicken skin (CS). Results suggested that a higher amount ($P < 0.05$) of total VC was observed in CS compared with CB during the whole processing, which may be correlated with higher thiobarbituric acid reactive substances values, and higher polyunsaturated fatty acid/saturated fatty acid ratio. According to E-nose analysis, the volatile flavor is clearly separated in the sugar-smoking stage. Volatile fingerprinting results revealed that heterocycles were the characteristic flavor formed during sugar-smoking process and hexanal, nonanal, furfural, 5-methyl-2-furancarboxaldehyde, and 2-acetyl-5-methyl furan were the major volatiles of the CS, which was closely related to lipid oxidation and caramelization reaction. Above all, the flavor of sugar-smoked chicken was mainly derived from CS and sugar-smoked process improved the flavor of CS. This study could provide theoretical guidance for regulation of the color and flavor of sugar-smoked chicken and further promote the development of the industry.

**Key words:** sugar-smoked chicken, lipid oxidation, volatile fingerprinting, E-nose

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**INTRODUCTION**

Chicken meat is the most commonly consumed meat source in many countries and simultaneously have an increase in consumption and production around the world (Takran et al., 2020). Chicken meat products are available as either sugar-smoked chicken products (Chen et al., 2013) or roast chicken products (Huang et al., 2020). The generation of characteristic flavor and aroma compounds of chicken meat is thermally derived, particularly those involved in the Maillard reaction and thermal degradation of lipids as well as their interaction between the 2 reactions (Chen et al., 2009; Jayasena et al., 2013). Chicken meat flavor mainly relies on several processing procedures and related factors including free fatty acids, cooking, pH, water content and salt content, etc (Jayasena et al., 2013), defining the flavor development and affecting the acceptability and volatile flavor component transformation of final products (Sañudo et al., 2000). Among them, traditional processing procedures for chicken meat include salting, saucing, smoking, and frying, etc (Shi and Ho, 1994). With regard to smoking technology, although using wood smoking to cure meat products has a very long history, the use of wood smoke incurs the formation polycyclic aromatic hydrocarbons (PAH) partially because of incomplete
combustion (Yurchenko and Mölder, 2005). In recent years, evidence has shown that PAH contamination of smoked foods can be significantly reduced by replacing conventional (traditional) wood smoking with sugar smoking (Chen et al., 2013).

Sugar-smoking, a popular smoking method involving preprocessing of meat, such as salted and/or dried, and then exposed to sugar, has been used as it imparts a characteristic flavor to meat products favored by average consumers (Chen et al., 2013). Besides, sugar-smoking improves sensory properties and decreases moisture content (Pittia and Antonello, 2016). Previously, many researchers only focused on determining the flavor composition of wood-smoked, cold, or hot smoked meat products (Turan et al., 2008; Yu et al., 2008; Gómez-Estaca et al., 2011). For example, Yu et al. (2008) revealed that 48 volatile compound (VC) of Chinese traditional smoke-cured bacon using wood smoke. Gómez-Estaca et al. (2011) reported that cold-smoked sardine (Sardina pilchardus) and dolphinfish increased fish lipid oxidation stability and revealed the presence of typical phenol and carbonyl derivatives, as well as some oxidation products and PAH. However, there is rather limited information concerning the relationship between sugar-smoking process parameters and VC fingerprinting as well as related quality attributes. Clarifying these relationships is meaningful to modify and design the final flavor profiling by combining with sugar-smoking technology in a targeted way.

The aim of the present study was, therefore, to determine the change of pH values, water and salt content, fat oxidation, and VC during the processing of sugar-smoked chickens, which will provide more information related to the sugar-smoked products.

MATERIALS AND METHODS

Processing of Sugar-Smoking Chickens and Sample Preparation

Twenty-five three-yellow chickens (cold carcass weight 500 ± 40 g) were randomly selected 24 h post-slaughter (conducted following the European Community, 1099/2099/EC 2009 guidelines). Animals were reared in a farm (Wenzhou, Zhejiang, China) and slaughtered at their market weight in a local commercial slaughter house. The facilities of the slaughter house met the requirements of the Institute of Animal Care and Use Committee. Immediately after slaughter, the carcasses were chilled at 4°C in a ventilated room for 24 h. Detailed process flow chart of sugar-smoked chickens is shown in the Figure 1.

Twenty-five chicken breast (CB) and chicken skin (CS) were sampled at 5 different processing points: raw (RC, RS), the end of pickling (PC, PS), the end of air-drying (DC, DS), the end of baking (BC, BS), and the end of sugar-smoked (SC, SS). Immediately after sampling, all the samples were cut to small piece (about 1 cm × 1 cm × 1 cm) and were wrapped in aluminum foil, frozen, and stored at −40°C before analysis, except the analysis of moisture content.

Determination of pH Value, Moisture Content, and Salt Content

The moisture content was determined in duplicate according to GB/T5009.3-2003; the salt content was evaluated as chloride in duplicate and was assayed according to GB/T12457-2008. The results of the moisture and the salt content were both expressed as g per 100 g muscle. For pH determination, 5 g of samples and 20 mL of distilled water were mixed with DY89-I high-speed homogenizer (Ningbo, Zhejiang, China) at 10,000 rpm for 30 s at 4°C, and then pH was determined by a pH meter (Mettler Toledo Instruments Co., Ltd., Shanghai, China) in duplicate. The 5 replicates were considered as mean values.

Determination of TBARS Values

The thiobarbituric acid reactive substances (TBARS) values were measured according to the method of our previous study (Yang et al., 2017). The values were expressed as mg of malondialdehyde per 100 g of muscles.

Analysis of FAA

Lipid Extraction and Free Fatty Acids Purification

Lipid was extracted as described, and free fatty acids (FAA) were purified by our previous study (Yang et al., 2017).

Determination of FAA

Free fatty acids were quantified according to the method of our previous study (Yang et al., 2017).
et al., 2017) with slight modifications. The fatty acid methyl esters were analyzed using a TSQ8000EVO (Thermo Fisher, Waltham, MA) equipped with a flame ionization detector and a split injector. 1 μL of solution was injected in split mode (10:1) onto a RtxWax capillary column (Restek, Bellefonte, PA; 30 m × 0.25 mm id × 0.25 lm film thickness). The temperature of the column was programmed as follows: 3 min at 160°C, increments of 10°C/min to 200°C, and maintained at 200°C for 2 min, then increments of 2°C/min to 230°C and maintained at 230°C for 10 min. The flow rate of the carrier gas (He) was 1.2 mL/min. Identification of fatty acids was performed by comparison of the retention times with internal standards. The results are expressed as the absolute content of free fatty acid.

Analysis of Volatile Compounds The extraction of VC of CB and CS was performed using solid-phase microextraction (SPME). The Supelco device containing (70 μm length) PDMS/DVB (Polydimethoxysilane/divinylbenzene) was used. For head-space SPME extraction, 4.5 g of ground sample was placed in a 20 mL vials. 2-Methyl-3-heptanone was used as internal standard. Analyses were performed using TSQ8000EVO. The VC were separated on TG-5MS (30 m × 0.25 mm × 0.25 μm) flexible quartz capillary column (ThermoFisher). The fiber was desorbed and maintained in the injection port at 270°C for 2 min. The GC oven temperature was first isothermal for 10 min and then raised to 200°C at a rate of 4°C/min, held for 2 min, next raised to 250°C at a rate of 20°C/min, and then kept for 7 min. Mass spectra were obtained using electron impact with ionization energy of 70 eV, scan mode ranging 33 to 350 m/z. The identification and quantity of compounds was achieved by comparing their mass spectra with those in NIST and Wiley eighth spectra libraries (minimum 90% accuracy).

E-NOSE ANALYSIS

The E-nose analysis was conducted using a commercial PEN 3.5 E-nose (Airsense Analytics GmBh, Schwerin, Germany) containing 10 metal-oxide semiconductors (Supplementary Table 1). Accurately weigh the 4.5 g of the minced CB and CS sample into a 40 mL headspace sample bottle, respectively. These kept at temperature (30°C) for 10 min for gas generation in the headspace. The headspace gaseous compounds were pumped into the sensor arrays. The responses of sensors were expressed as the ratio of conductance G/G0 (G and G0 represent the conductances of sensors exposed to sample gas and zero gas, respectively). An assessment time of 120 s was used to make sure the stable response signs. Each test was conducted in 5.

Statistical Analysis

The ANOVA was performed to compare means for pH value, moisture content, salt content, TBARS, FAA, and VC. When a significant difference (P < 0.05) was
RESULTS AND DISCUSSION

The Change of Physicochemical Properties

The change of physicochemical properties during processing, including pH values, moisture content, and salt content of the sugar-smoked chickens was summarized in Table 1. The pH values of CB increased, ranging from 5.91 to 6.39. The pH values of CS significantly increased ($P < 0.05$) in the pickling stages and then slowly decreased. Huang et al. (2014) reported that the pH values of smoked bacon were 5.73 to 5.96, which was lower than CB. The rise in the pH values was mainly related to the lipid oxidation and the generation of amino acids through protein degradation (Huang et al., 2014). Compared with other stages, the pH value of the sugar-smoking stage (SS) was the lowest, possibly because the citric acid and phosphate could be attached to the sugar-smoked CS in the pickling stage, which further resulted in relatively high content of acidic substances after sugar-smoking. Furthermore, some studies indicated that glucose more easily formed furfurals in the caramelization reaction process under acidic conditions (Ajandouz et al., 2010), which could further contribute to the improvement of flavor profiles of sugar-smoked chicken during the processing of sugar-smoking.

Regardless of CB or CS, the moisture content kept decreasing during processing. Puangsombat et al. (2012) reported that the moisture level of fresh meat products ranged between 69 and 82%, except in the high-fat parts (bacon, breast skin, and thigh skin), which contained low moisture levels (approximately 37%). In our study, the average moisture content of the final CB and CS was 37.35 and 11.35%, respectively, which was lower than that of other cured meat products (Marra et al., 1999; Martin et al., 2001; Jin et al., 2010) and higher than that of the reported smoked bacon (Huang et al., 2014). It could be caused by osmotic dehydration in the early stages and the water evaporation in the mid-late stages of processing. The low moisture content is a typical property of Chinese traditional smoke-cured meat products and also one of the most important factors for prolonging shelf life under room temperature.

Simultaneously, the salt content of CB also significantly increased ($P < 0.05$) during processing, whereas it markedly increased ($P < 0.05$) in the first stages and then slowly decreased in CS. The rise of salt content for CB was related to salt diffusion of CS and great dehydration in the pickling and air-drying stages, whereas the last 2 stages was ascribed only to dehydration because of temperature (Huang et al., 2014). The average salt content of the final product was 7.34%, which was similar to that of a typical dry-cured ham (6–8%) (Motilva and Toldrá, 1993).

The Change of Lipid Oxidation

As shown in the Figure 2, TBARS of CB and CS gradually increased during processing and the oxidation degree of CS was higher than CB. This result was consistent with the report by Pikul et al. (1984). To our knowledge, CS contained the highest contents of fat, which was more susceptible to oxidation. It could be also seen that sugar-smoking process accelerated the speed of lipid oxidation. This was different from the result of Hobson et al. (2019) probably because of the difference in processing procedures. Heat treatment and oxygen are also factors which promote lipid oxidation (Pikul et al., 1984). As the process continued, the salt concentration enhanced and the moisture content declined, which could inactivate the neutral lipase and phospholipase and to some extent favored the activity of acid lipase (Toldrá, 2006). Huang et al. (2014) suggested that salt varied closely with both POV and TBARS. Pikul et al. (1984) reported that the increase of pH may be helpful for phospholipid hydrolysis and the TBARS numbers of roasted muscles and skin were further elevated because of water evaporation and loss of juiciness, which effectively increased the total lipid content of roasted samples, which could be the reason for further acceleration in the SS.

The Change of FAA

Table 2 showed that the sugar-smoked CB and CS both contained 14 kinds of FAA. It was founded that...
Table 2. Changes in the free fatty acid content of chicken breast (CB) and skin (CS) during processing (ng/g).  

| Kinds  | RC      | PC       | DC       | BC      | SC       | RS       | PS       | DS       | BS       | SS       |
|--------|---------|----------|----------|---------|----------|----------|----------|----------|----------|----------|
| C14:0  | 3.35 ± 0.02e | 5.45 ± 0.3d  | 6.02 ± 0.05d,e | 6.36 ± 0.06d,e  | 5.88 ± 0.26d  | 4.92 ± 0.16h  | 7.46 ± 0.08a  | 6.66 ± 0.2d,e  | 7.21 ± 0.14h  | 6.99 ± 0.17h,e  |
| C16:0  | 61.52 ± 2.24d  | 70.34 ± 0.38d  | 75.3 ± 0.99b,c  | 76.25 ± 0.47a  | 62.03 ± 0.62d  | 60.8 ± 3.11d  | 64.37 ± 0.63d  | 62.22 ± 2.73d  | 61.78 ± 2.5d  | 72.65 ± 0.16h,c  |
| C18:0  | 43.82 ± 1.12b,c  | 43.8 ± 1.02b,c  | 45.43 ± 0.64b  | 48.6 ± 1.68a  | 43.42 ± 2.62b,c  | 41.26 ± 1.19c  | 40.87 ± 0.3b  | 41.74 ± 0.35b  | 41.29 ± 2.04d  | 40.73 ± 0.84c  |
| C18:1  | 15.12 ± 0.06b  | 18.02 ± 0.97e  | 25.22 ± 1.58b  | 20.84 ± 0.01d  | 21.15 ± 0.21d  | 26.2 ± 0.1b,c  | 19.4 ± 1.05b  | 24.07 ± 2.16b  | 33.34 ± 1.85a  | 27.39 ± 1.25b  |
| C18:2  | 4.76 ± 0.02c  | 48.15 ± 1.83a,b  | 46.22 ± 0.86a,b  | 40.18 ± 0.87c  | 45.18 ± 7.32a,b  | 47.35 ± 0.19b,c  | 49.29 ± 0.15a,c  | 48.16 ± 1.61b  | 48.39 ± 10.94b  | 54.18 ± 2.09b  |
| C22:0  | 0.49 ± 0.01c  | 0.56 ± 0.05b  | 0.57 ± 0.06b  | 2.32 ± 0.02a  | 0.53 ± 0.05b  | 0.31 ± 0.01d  | 0.34 ± 0.01d  | 0.31 ± 0.04d  | ND        | ND        |
| C22:2  | 11.12 ± 0.06a  | 74.27 ± 4.14a  | 84.15 ± 1.50a,b  | 80.67 ± 0.9a  | 75.22 ± 5.7a  | 67.47 ± 1.33b,c  | 71.71 ± 0.78b,c  | 74.03 ± 0.10b,d  | 72.24 ± 1.26b,c  | 67.03 ± 0.39b,c  |
| C22:4  | 7.61 ± 0.45  | 10.57 ± 0.54a  | 11.28 ± 0.07a  | 8.82 ± 0.19b  | 6.29 ± 0.17d  | ND        | 2.94 ± 0.6f  | 4.77 ± 0.01e  | 4.92 ± 0.41e  | 4.64 ± 0.04f  |
| C20:0  | 13.62 ± 0.18a  | 13.18 ± 0.01a  | 11.48 ± 0.59b  | 12.79 ± 1.15a  | 9.05 ± 1.05a  | 3.14 ± 0.36d  | 3 ± 0.13d  | 3.12 ± 0.07d,e  | 2.79 ± 0.19d  | 2.96 ± 0.41f  |
| C20:4  | 44.45 ± 0.18b  | 43.14 ± 1.19b  | 45.77 ± 0.12b,c  | 47.61 ± 0.12a  | 43.25 ± 1.99b  | 15.71 ± 2.74f  | 12.22 ± 0.02d  | 11.61 ± 0.09d  | 9.74 ± 1.29d  | 10.15 ± 1.06c  |
| C22:4  | 13.88 ± 2.45a  | 18.03 ± 0.86a  | 17.14 ± 0.12a  | 18.77 ± 0.42a  | 16.9 ± 0.27a  | 1.09 ± 0.06a  | 1.3 ± 0.01c  | 1.25 ± 0.01e  | 1.17 ± 0.00f  | 1.16 ± 0.05g  |
| C22:5  | 13.88 ± 2.45  | 18.03 ± 0.86a  | 17.14 ± 0.12a  | 18.77 ± 0.42a  | 16.9 ± 0.27a  | 1.09 ± 0.06a  | 1.3 ± 0.01c  | 1.25 ± 0.01e  | 1.17 ± 0.00f  | 1.16 ± 0.05g  |
| C22:6  | 11.17 ± 1.27a  | 12.3 ± 0.17a  | 11.79 ± 0.27a  | 11.56 ± 0.51a  | 10.11 ± 0.18b  | 3.17 ± 0.04f  | 2.81 ± 0.05b  | 3.94 ± 0.31c  | 3.58 ± 0.29f  | 3.14 ± 0.28g  |
| ∑SFA   | 112.1 ± 2.88d  | 129.32 ± 1.53b  | 133.41 ± 1.75b,c  | 136.3 ± 1.13a  | 117.14 ± 1.39a  | 106.99 ± 1.75b  | 112.7 ± 0.84b  | 110.63 ± 0.58b,c  | 111.56 ± 4.69b  | 121.41 ± 0.81b  |
| ∑MUFA  | 58.37 ± 0.07d  | 66.73 ± 0.9b,c,d  | 72.01 ± 2.43a,b,c  | 63.34 ± 0.9a  | 66.85 ± 7.47b,c,e  | 73.86 ± 0.28b  | 69.03 ± 1.21b  | 72.54 ± 3.73b,c  | 81.73 ± 9.09a  | 81.56 ± 3.34a  |
| ∑PUFA  | 171.29 ± 3.59b  | 200.74 ± 3.65b  | 209.22 ± 0.9e  | 205.57 ± 3.17a,b  | 187.4 ± 7.7f  | 93.47 ± 5.05f  | 101.72 ± 1.4d  | 107.19 ± 0.03e  | 106.85 ± 2.74e  | 97.59 ± 0.77f  |
| Total  | 341.76 ± 0.78e  | 396.79 ± 1.21c  | 414.63 ± 5.12a  | 405.22 ± 3.41b  | 371.4 ± 1.62d  | 274.32 ± 3.02b  | 283.45 ± 3.45e  | 290.36 ± 4.28b  | 300.14 ± 7.14d  | 300.56 ± 1.76c  |

**Note:** Each value is expressed as mean ± SD.  
Abbreviations: BC = baking chicken breast and skin; DC = air-drying chicken breast and skin; MUFA = monounsaturated fatty acid; PC = pickling chicken breast and skin; PUFA = polyunsaturated fatty acid; RC = raw chicken breast and skin; SC = sugar-smoking chicken breast and skin; SFA = saturated fatty acid; SS = sugar-smoking chicken breast and skin.
| Compounds                                      | RT   | RC    | FC    | DC    | BC    | SC    | RS    | PS    | DS    | BS    | SS    |
|-----------------------------------------------|------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| Alcohol                                       |      |       |       |       |       |       |       |       |       |       |       |
| 3,7-dimethyl-1,6-dodecadiene                  | 8.38 | 0.67  | 0.86  | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    |
| 6-Undecen-3-ol                                |      |       |       |       |       |       |       |       |       |       |       |
| Terpinen-4-ol                                 |      |       |       |       |       |       |       |       |       |       |       |
| Isopropenyl alcohol                           |      |       |       |       |       |       |       |       |       |       |       |
| Phytol                                        |      |       |       |       |       |       |       |       |       |       |       |
| 4-(2,6-dimethyl-1,3-cyclohexadiene)           |      |       |       |       |       |       |       |       |       |       |       |
| 3-Buten-2-ol                                  |      |       |       |       |       |       |       |       |       |       |       |
| (Z)-9-Octodec-1-en-3-ol                       |      |       |       |       |       |       |       |       |       |       |       |
| 2-methyl-1-Hexadecanol                        |      |       |       |       |       |       |       |       |       |       |       |
| 3-isocoranol                                   |      |       |       |       |       |       |       |       |       |       |       |
| 3,7,11-trimethyl-1,6,10-dodecatriene           |      |       |       |       |       |       |       |       |       |       |       |
| Bencenic alcohol                              |      |       |       |       |       |       |       |       |       |       |       |
| Ethanol                                       |      |       |       |       |       |       |       |       |       |       |       |
| 1-Tricosenol                                   |      |       |       |       |       |       |       |       |       |       |       |
| 2-Hexyl-1-Deconol                             |      |       |       |       |       |       |       |       |       |       |       |
| 2-Hexyl-1-octanol                             |      |       |       |       |       |       |       |       |       |       |       |
| Total                                         |      |       |       |       |       |       |       |       |       |       |       |
| Aldehydes                                     |      |       |       |       |       |       |       |       |       |       |       |
| Hexanal                                       | 17  | 0.02  | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    |
| Heptanal                                      | 6.24| ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    |
| Benzaldehyde                                  | 7.6 | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    |
| (E)-2-Octenol                                 | 10.6| ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    |
| Nonanal                                       | 11.8| 1.38  | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    |
| 8-Octodecane                                  | 18.9| 0.27  | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    |
| 5-Octodecane                                  | 25.7| 1.02  | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    |
| Total                                         |      |       |       |       |       |       |       |       |       |       |       |
| Hydrocarbons                                  |      |       |       |       |       |       |       |       |       |       |       |
| n-Hexane                                      | 2.92| 1.37  | 1.65  | 0.14  | 0.28  | 0.38  | 0.40  | 0.52  | 0.63  | 0.75  | 0.83  |
| p-Xylene                                      | 5.67| ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    |
| Styrene                                       | 6.07| ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    |
| Undecane                                      | 11.0| ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    |
| 9-Methylheptadecane                           | 13.7| ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    |
| decane                                        |      |       |       |       |       |       |       |       |       |       |       |
| Dodecane                                      | 13.4| 0.89  | 0.28  | 0.22  | 0.64  | 0.81  | 0.89  | 0.93  | 0.97  | 1.01  | 1.09  |
| 2,6,10-trimethylheptadecane                   |      |       |       |       |       |       |       |       |       |       |       |
| Tetradecane                                   | 15.9| ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    |
| Tetracosane                                   | 19.7| 0.73  | 0.17  | 0.21  | 0.46  | 0.49  | 0.52  | 0.54  | 0.57  | 0.60  | 0.64  |
| 2,6,10,15-tetramethyl-1,3,5,7-hexadecatriene   |      |       |       |       |       |       |       |       |       |       |       |
| Nonadecane                                    | 20  | 0.95  | 0.12  | 0.25  | 0.37  | 0.43  | 0.47  | 0.51  | 0.55  | 0.58  | 0.63  |
| Hexadecane                                    | 21.9| 1.26  | 0.61  | 0.79  | 1.15  | 1.49  | 1.92  | 2.18  | 2.44  | 2.69  | 3.17  |
| Heptadecane                                   | 23.8| 1.05  | 0.82  | 1.13  | 1.54  | 1.95  | 2.41  | 2.91  | 3.40  | 3.89  | 4.44  |
| Octadecane                                    | 25.8| 1.14  | 1.87  | 2.60  | 3.27  | 4.00  | 4.85  | 5.73  | 6.64  | 7.55  | 8.45  |
| 9-Hexyl Heptadecane                           | 27.8| 0.52  | 1.00  | 1.35  | 1.80  | 2.47  | 3.25  | 4.06  | 4.88  | 5.70  | 6.52  |
| decane                                        |      |       |       |       |       |       |       |       |       |       |       |
| Total                                         | 55.4| 0.05  | 0.95  | 1.37  | 1.83  | 2.40  | 3.01  | 3.63  | 4.25  | 4.88  | 5.52  |
| Esters                                        |      |       |       |       |       |       |       |       |       |       |       |
| Hexanoic acid                                 | 6.8 | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    |
| methyl ester                                  | 9.3 | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    | ND    |
| 1-methyl-4-(1-methyl ethenyl)-Cyclohexanol, acetate | 10.8 | ND | ND | ND | ND | ND | ND | ND | ND | ND | 462.65 | 1.55 |

| Table 3. Changes in volatile compounds (VC) of chicken breast (CB) and skin (CS) during processing. |
| Compound                                         | BC          | BS          | DC          | DS          | PC          | PS          | RC          | RS          | SC          | SS          |
|-------------------------------------------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
| 10,13-Octadecadiynoic acid, methyl ester         | 12.56       | ND          | ND          | ND          | ND          | ND          | 14.26 ± 0.66 | 31.15 ± 0.05 | 11.36 ± 1.4 | 81.94 ± 0.55 |
| Lavandulyl acetate                              | 14.78       | ND          | ND          | ND          | ND          | ND          | 0.51 ± 0.08  | 1.45 ± 0.01  | 3.18 ± 0.17 | 2.65 ± 0.39  |
| 9-Octadecen-12-ynoic acid, methyl ester         | 15.75       | 0.49 ± 0.08b| 1.29 ± 0.48b| 7.41 ± 2.73b| 26.63 ± 9.49| 20.81 ± 13.29| 3.73 ± 0.21  | 3.77 ± 1.00  | 9.55 ± 0.98b| 7.40 ± 1.00  |
| Geranyl isovalerate                              | 16.94       | ND          | ND          | ND          | ND          | ND          | 8.04 ± 2.05b| ND          | ND          | ND          |
| 7-Methyl-Z-tetra decen-1-ol acetate             | 21.4        | 0.82 ± 0.50c| 0.66 ± 0.12c| 6.79 ± 0.83c| 50.29 ± 11.69c| 41.3 ± 4.7c  | 0.67 ± 0.12c| 0.54 ± 0.07c| 1.66 ± 0.78c| 1.99 ± 0.54c| 7.28 ± 1.1c |
| Total                                           | 1.02 ± 0.1  | 1.95 ± 0.02de| 4.19 ± 3.56de| 84.95 ± 23.23b| 62.11 ± 61.99de| 19.72 ± 1.96de| 45.39 ± 2.33de| 59.91 ± 5.28b| 55.16 ± 2.73hde| 620.31 ± 19.96c |
| Heterocycles                                     |             |             |             |             |             |             |             |             |             |             |
| Furfural                                        | 4.87        | ND          | ND          | ND          | ND          | ND          | 929.43 ± 132.7b| ND          | ND          | ND          | 18.087.59 ± 717.3a |
| 5-methyl 2-Furancarboxaldehyde                  | 7.8         | ND          | ND          | ND          | ND          | ND          | 167.74 ± 6.6b | ND          | ND          | ND          | 4.039.33 ± 3.46c |
| 2-Acetyl-5-methylfuran                          | 9.59        | ND          | ND          | ND          | ND          | ND          | ND          | ND          | ND          | ND          | 469.46 ± 1.6  |
| 5-Hydroxymethylfurfural                         | 14.8        | ND          | ND          | ND          | ND          | ND          | ND          | ND          | ND          | ND          | 244.3 ± 16.54 |
| 1-(2-furanyl)Ethanolone                         | 6.52        | ND          | ND          | ND          | ND          | ND          | ND          | ND          | ND          | ND          | 687.3 ± 1.67  |
| 2-hydroxy-3-methyl-2-Hydroxyethylone            | 9.4         | ND          | ND          | ND          | ND          | ND          | ND          | ND          | ND          | ND          | 407.75 ± 1.05  |
| 2-Cyclopenten-1-one                             | ND          | ND          | ND          | ND          | ND          | ND          | 1,097.18 ± 219.00b| ND          | ND          | ND          | 23,422.99 ± 792.61a |
| Total                                           | ND          | ND          | ND          | ND          | ND          | ND          | 12.88 ± 2.00d| ND          | 0.27 ± 0.16c| ND          | ND          | 248.02 ± 1.45b |
| Ketones                                         |             |             |             |             |             |             |             |             |             |             |             |
| Acetoin                                         | 2.94        | 12.88 ± 2.00a| ND          | 0.27 ± 0.16b| ND          | ND          | 17.96 ± 3.32a| ND          | ND          | ND          | ND          |
| 2-methyl-3-Octanone                              | 8.21        | ND          | ND          | ND          | ND          | ND          | ND          | ND          | ND          | ND          | 254.51 ± 1.13a| 19.65 ± 1.16b |
| 1-(1,2-Dimethyl-cyclopent-2-enyl)-ethanone       | 10.3        | ND          | ND          | ND          | ND          | ND          | ND          | ND          | ND          | ND          | 248.02 ± 1.45b |
| Total                                           | 12.88 ± 2.00d| ND          | 0.27 ± 0.16c| ND          | ND          | ND          | 17.96 ± 3.32c| 254.51 ± 1.13c| 19.65 ± 1.16c| ND          | 248.02 ± 1.45b |
| Others                                          |             |             |             |             |             |             |             |             |             |             |             |
| Estragole                                       | 15.62       | ND          | ND          | ND          | ND          | ND          | ND          | ND          | ND          | ND          | 103.1 ± 2.75  |
| 3-hydroxy-Dodecanic acid                        | 13.64       | 0.15 ± 0.05c| ND          | ND          | ND          | ND          | ND          | ND          | ND          | ND          | 16.02 ± 1.32b  |
| Total                                           | 0.15 ± 0.052d| ND          | ND          | ND          | ND          | ND          | 16.02 ± 1.32c| 125.61 ± 2.88a| ND          | 103.1 ± 2.75b |

*aDifferent letters in the same row indicate that there is significant difference ($P < 0.05$, along the lines).
Each value is expressed as mean ± SD.
Abbreviations: BC, BS = baking chicken breast and skin; DC, DS = air-drying chicken breast and skin; PC, PS = pickling chicken breast and skin; RC, RS = raw chicken breast and skin; SC, SS = sugar-smoking chicken breast and skin.
The Change of Volatile Compounds

As summarized in the Table 3, a total of 40 and 54 VC were isolated and identified using SPME-GC-MS technique in CB and CS, respectively. The HCA was given to search for sample distribution pattern according to overall comparison of the VC (Supplementary Figure 1). The HCA demonstrated a clear clustering tendency of the samples, including the PS; RS, BS, DS, and DC; RC, PC, and BC as well as SC and SS. Figure 3 showed that a higher amount (P < 0.05) of total VC was observed in the CS compared with CB, except the baking stages. Our research was in agreement with Pippen and Nonaka (1963) who founded that the overall yield of volatile material was greater and a more complex nature from skin and skin fat than from lean leg and breast muscle. During baking stages, this was not surprising, because CB contained an appreciable amount of protein, amino acids, etc, and high temperature during baking stages, which resulted in the faster speed of Maillard reaction and fat oxidation and could be expected to give rise to VC. Besides, Ang (1988) suggested that protein, water, and total lipid in various broiler tissues might affect the specific tissue stability by influencing the physical or environmental conditions for the oxidation process. It was concluded that physical and chemical characteristics could result in generating intense VC. In addition, the reason for CS also may be because of the directly exposure to the air (Ang and Lyon, 1990), which further promoted oxidation rate and, to the extent, slowed down the degree of autooxidation of the internal meat.

Figure 4 showed that the content of heterocyclic compounds reached the highest compared with other kinds of compounds. The heterocyclic compounds were produced only in the SS and was greater in the CS than CB, in agreement with the result reported by Sung (2013) who observed that furan compounds (furfural, 5-methyl-2-furancarboxaldehyde, 5-hydroxymethyl-2-furaldehyde) were the major volatile constituents found in the smoke condensates. And, these compounds were considered as characteristic flavor compounds of the sugar-smoking process. Among them, furfural, with a sweet, woody, bready, and caramel aroma (Kerth and Miller, 2015), could be produced as a flavor active intermediate present in the early stages of the Maillard reaction and a precursor of other heterocyclic compounds (Fors, 1983), with relatively low threshold. It was mainly produced by the sucrose pyrolysis of the smoked chicken (Sung, 2013). 5-Methylfurfural has a sweet, spicy, coffee, and caramel flavor (Fors, 1983). 2-Acetyl-5-methylfuran has a sweet, moldy, nutty, and caramel-like aroma note (Steen et al., 2017). 5-Hydroxymethyl furfural has a sweet, coffee, and caramel flavor characteristic. The formation of 5-Hydroxymethyl furfural was produced by an acid-catalyzed dehydration of fructose (Román-Leshkov et al., 2006), which was not detected in the CB. In particular, 1-(2-furanyl)-ethanone has strong sweet balsamic-cinnaminc odor note and are founded in processed foods (Cho et al., 2010). However, its formation mechanism had not been clearly shown.

Figure 4 showed that the total content of aldehydes compounds gradually increased from the raw to the air-drying stages in the CS. When it reached the baking stages, the total content dropped significantly (P < 0.05). This may be because the flavor compounds produced by the oxidation of the CS were diffused inward during the baking stages, so that the content of the CB was significantly higher, indicating the dynamic
change process of the flavor composition of CB and CS. According to Table 3, most aldehydes belong to the straight-chain aldehydes with more than 5 carbon atoms such as hexanal, heptanal, and nonanal. This finding was consistent with Dominguez et al. (2014) who reported that hexanal was the main VC of foal meat. Mottram (2007) suggested that all were produced from fat oxidative degradation and strecker amino acid reaction. The content of hexanal in the CS was higher than that in CB, which was derived from ω-6 unsaturated fatty acid (Wettasinghe et al., 2001) and corresponded well with the higher TBARS values. Hexanal is described as intense grass-like aroma note, whereas heptanal has a fatty and oily aroma, and nonanal shows grassy, oily and bitter (Wettasinghe et al., 2001; Takakura et al., 2014). The presence of benzaldehyde in the processing of CS may be associated to the degradation of phenylalanine (Xie et al., 2008). Owing to low odor thresholds and distinctive odors, the above volatile aldehydes should belong to potent contributors to the chicken flavor (Xie et al., 2008). Besides, long-chain aliphatic aldehydes, for example 8-octadecenal and 5-octadecenal were also found. However, these high-molecular weight aldehydes probably act as precursors of the volatile saturated and unsaturated aldehydes, because lower volatility makes them less important to meat flavor (Xie et al., 2008).

From the Figure 4, the total content of alcohol compounds kept increasing in the CB. And the content of CS decreased significantly during the baking stages. The phenomenon occurred may be related to the action of enzymes, which could reduce the aldehydes produced by the catabolism of fatty acids and amino acids to the corresponding alcohols (Chen et al., 2009). Among alcohol compounds (Table 3), 3,7-dimethyl-1,6-octadien-3-ol is commonly known as linalool and has a
floral, citrus-like aroma (Adebo et al., 2018), of which the content was higher in CS. Terpinen-4-ol has a woody, earthy, and musty aroma (Adebo et al., 2018). It has been demonstrated that terpenoids including 3,7-dimethyl-1,6-octadien-3-ol, terpinen-4-ol, isopinocarveol, ß-acorenoi, and 9-octadecen-1-ol were the major sources of natural flavor additives in foods and fragrances (Singh and Sharma, 2015), with those content was slower despite in the CB or CS. The linear saturated alcohols have a higher threshold value, thus exerting little effect on the flavor perception (Dirinck et al., 1997). As shown in the Figure 4, the content of hydrocarbons in the CB and CS gradually increased. For CB, the content reached the maximum during the SS. Among them, the aromatic and aliphatic hydrocarbons might derive from the thermal degradation of lipid by thermal homolysis or autooxidation of long-chain fatty acid (Song et al., 2011). Nevertheless, as a result of high odor thresholds, they were generally believed to have little contribution to meat flavor (Xie et al., 2008). Simultaneously, some compounds may be important intermediates in the formation of heterocyclic compounds, which was beneficial to improve overall meat flavor.

The esters of C1-C10 fatty acids have a typical fruity aroma, while the long-chain esters of long-chain fatty acids show an oily taste (Flores, 2018). The presence of ethyl hexanoate produces the aroma of fruit (banana, green apple, etc.) and may be related to the alcohols added during the pickling process, as well as the fermentation of microorganisms (Tešević et al., 2009). It was reasonable to assume that aldehydes compounds in the SS may be oxidized to form acid, such as furfural, and further to form ester so that result in the existence of 2-furoyl methyl. 3-Hydroxy-dodecanoylic acid is a fatty acid that could act as a precursor to volatile ketones (2-undecanone) (Labows et al., 1980). Estragole is the main flavor of star anise and has the aroma of star anise (Sun et al., 2014). It only was detected in the CS. The ketones could arise from lipid degradation, and they have been implicated in the butyric aroma note of cooked meats (Peterson et al., 1975).

E-Nose

Examination of the score plot (Figure 5) in the area defined by the first 2 principal components (93.85%) revealed a clear separation between CB and CS. The predicted accuracy of this model was more than 85% (Guohua et al., 2012), indicating that PC1 and PC2 already contained a large amount of information of the samples. Figure 5 showed that the distribution of PC, DC, and BC; PS, DS, and BS were relatively concentrated, suggesting that the overall flavor had no significant in the PC1. This result was similar to the above-mentioned result of HCA. Besides, RC, SC, RS, and SS could be well distinguished in the PC1. These results demonstrated that the processing procedures greatly caused the shifts of the flavor precursors as the results of the biochemical reactions occurring during the different stages, especially including lipid oxidation, lipolysis, Maillard reaction, and caramelization reaction, thus leading to the differences in the contents and types of volatile flavor substances.

CONCLUSION

The VC fingerprinting mainly included heterocyclic, aldehydes, alcohol and hydrocarbons compounds in sugar-smoked chicken. The flavor of sugar-smoked chicken was mainly derived from CS and sugar-smoked process improved the flavor of CS. The changes in physicochemical attributes and the composition of free fatty acids, as well as the increased of TBARS values, Maillard reaction and caramelization reactions favored the generation of intense flavor during the sugar-smoking CS. Hexanal, nonanal, furfural, 5-methyl-2-furancarboxaldehyde, 2-acetyl-5-methylfuran, and 1-(2-furanyl)-ethanone were the major volatiles of sugar-smoked chickens. However, the quantitative mechanism toward the flavor contribution of sugar-smoked technology needs further investigation.

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DISCLOSURES

The authors declare no conflicts of interest.

SUPPLEMENTARY DATA

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