Meta Science 133 (2017) 1–9

Contents lists available at ScienceDirect

Meat Science

journal homepage: www.elsevier.com/locate/meatsci

Evaluation of lipid oxidation of Chinese-style sausage during processing and storage based on electronic nose

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ARTICLE INFO

Keywords:
Chinese-style sausage
Lipid oxidation
Electronic nose
Sensors
Classification
Prediction

ABSTRACT

A portable electronic nose was used for extracting flavour fingerprint map of Chinese-style sausage during processing and storage, in parallel with detection of acid value (AV) and peroxide value (POV) for evaluating lipid oxidation. Sausage samples during processing and storage were divided into three and five quality phases, respectively. After comparison of sensors response to lipid oxidation, optimal sensor array was determined. Several classification and regression models were developed to classify samples into their respective quality phase and predict lipid oxidation using full and optimal sensor array. Results indicated classification accuracy for sausage samples were, respectively, above 95% and 82% during the processing and storage. For support vector machine (SVM) and artificial neural networks (ANN) regression models, good performance in predicting AV and POV were obtained, with the coefficients of determination (R^2s) > 0.914 and 0.814 during processing and storage, respectively. Thus, E-nose demonstrated acceptable feasibility in evaluating the degree of lipid oxidation of Chinese-style sausage during processing and storage.

1. Introduction

Chinese-style sausage is one of the most famous traditional meat products in China and is popular worldwide for its unique flavour. This sausage remains stable for a long period, mostly due to its high salt content and low water activity. In general, the quality deterioration of Chinese-style sausage is mainly due to lipid peroxidation. A moderate degree of lipid oxidation plays a positive role in sausage quality, but excessive oxidation results in product deterioration (Lorenzo, Bedia, & Bañón, 2013). Currently, high-temperature drying is used to accelerate lipid oxidation and shorten the drying time of Chinese-style sausage in commercial production instead of natural drying, which is mainly responsible for peroxidation and off-flavour phenomena (Zhang, Lin, Leng, Huang, & Zhou, 2013). However, Chinese-style sausage, which is mainly composed of pork fat and lean tissues, is a high-fat meat product. Although a high fat content makes sausages tender and succulent, this feature also increases the accumulation of lipid peroxide (Bradley et al., 2011). Furthermore, during a long shelf period, lipid oxidation caused by the oxidation of unsaturated fats occurs, limiting human consumption due to the formation of oxidative rancidity and off-flavours (Estévez, 2011; Ravyts et al., 2010). Therefore, it is crucial to monitor the degree of lipid oxidation in Chinese-style sausage products during the processing and storage.

According to current research results, some lipid oxidation indices, such as acid value (AV) (Zhang et al., 2015), peroxide value (POV) (Azeredo, Faria, & Da Silva, 2004; Grossi, Di Lecce, Arru, Gallina Toschi, & Riccò, 2015) and 2-thiobarbituric acid reactive substances (TBARS) (de Almeida et al., 2015; Fan, Zhang, Chen, Sun, & Yi, 2014), are usually developed to evaluate the degree of lipid oxidation of Chinese-style sausage. However, certain disadvantages accompany these chemical analytical methods, including a waste of time and labour, destruction of samples, high requirements for operation and vast use of organic solvents. In addition, sensory analysis is often performed to evaluate the quality of sausages. Some sensory properties (e.g., colour, odour, texture) are usually linked to the changes of lipid oxidation of the products (de Almeida et al., 2015; Kulkarni, De Santos, Kattamuri, Rossi, & Brewer, 2011). However, the method is very expensive because it needs highly-trained specialists, who can distinguish the flavour and appearance regardless of olfactory and visual fatigue. Thus, the detection of lipid oxidation indices need to be replaced by more convenient and appropriate methods following the needs to monitor in real time the quality of Chinese-style sausage.

During lipid oxidation, odour is a very important indicator. Some volatile compounds, including certain aldehydes, ketones and alcohols, form via lipid oxidation, playing an important role in the flavour development of sausages (Sun et al., 2009). Nevertheless, peroxidation damages the aromatic characteristics, resulting in an unbearable smell of rancidity. This continuous accumulation of off flavours parallels a

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http://dx.doi.org/10.1016/j.meatsci.2017.05.017
Received 16 January 2017; Received in revised form 19 May 2017; Accepted 22 May 2017
Available online 25 May 2017
0309-1740/

higher level of lipid oxidation. And the presence of off flavours universally occurs prior to the deterioration of the appearance of sausages. Hence, smell messages are considered as an effective method of evaluating the degree of lipid oxidation of Chinese-style sausage instead of chemical analysis and sensory analysis. In recent decades, there have been similar studies related to the volatile compounds of Chinese-style sausage. A study of the volatiles of Cantonese sausage based on headspace solid phase micro-extraction and gas chromatography–mass spectrometry (HSPME–GC–MS) was carried out by Sun, Zhao, Zhao, Zhao, and Yang (2010), analysing the main source taste and demonstrating that lipid oxidation is one of the primary sources. However, there are several defects of HSPME–GC–MS, including the high cost of detection and the delay in obtaining results. A faster and better method for acquiring the smell messages of Chinese-style sausage is necessary.

An electronic nose consists of a sampling system, an array of electronic gas sensors with sensitivity and selectivity to multi-component gas mixtures and pattern recognition software for data recording and analysis. When gas mixtures enter the system of an electronic nose, different sensors in an array produce different electric signals due to their different sensitivity and selectivity to each component of gas mixtures. Then these electric signals are converted into a series of response values of sensors through data acquisition and A/D conversion. Next, the response signals of sensors are simplified through signal pre-processing (e.g., eliminating noise, extracting features and amplifying signal) to obtain useful information. Finally, simplified response signals are imported into pattern recognition system to gain the information about the variety and concentration of gas mixtures (Wei, Yu, & Tang, 2014). This nose is applicable in a various range of studies in the meat industry, including (i) quality control, (ii) grade and freshness judgment, (iii) spoilage detection, (iv) category and adulteration determination, (v) production process monitoring and control and (vi) shelf-life assessment (Papadopoulou, Panagou, Mohareb, & Nychas, 2013). Not only does an electronic nose require little sample pretreatment, it also provides on-line and fast-screening application. Furthermore, an electronic nose acquires an overall estimate of the mixture of volatiles, which is evaluated based on a pattern recognition algorithm.

The purpose of this work, therefore, was to evaluate the performance of the electronic nose in classifying sausage samples into their respective quality phase during processing and storage using pattern recognition algorithms. An additional purpose was to predict the lipid oxidation of Chinese-style sausage during processing and storage by establishing regression models between electronic nose data and the lipid oxidation index in conjunction with some pattern recognition algorithms.

2. Materials and methods

2.1. Sample preparation

Fresh lean pork, back fat and casings were purchased from the local meat market where slaughtered pigs were transported within 5 h. Non-meat ingredients, including salt, sugar and alcohol, were purchased from Nanjing Suguo Supermarket (Nanjing, China). Sodium nitrite was obtained from Anhui Chemical Reagent Factory (Anhui, China). Fresh lean pork and back fat were ground through 10-mm and 5-mm sieve plates, respectively. Then, 70% lean pork and 30% back fat were mixed thoroughly with 3.5% salt, 12% sugar, 4% white wine (ethanol content: 55%, in Volume, Yanghe Qu liquor, Yanghe Inc., Jiangsu, China), 0.02% sodium nitrite and 20% water prior to stuff into natural pork casings (Sun et al., 2010). Simultaneously, the sausages were holed and deflated with a fine needle at 15-mm intervals. For the consistency, raw Chinese-style sausages were linked into 150 ± 20 mm lengths, a diameter of 25 ± 2 mm and 125 ± 5 g weight. Home-made sausages were rinsed in warm water at 35 °C and then drained and oven dried according to the specific processing conditions. Samples were oven dried for 50 h under the temperature of 50–55 °C with about 40% relative humidity in the constant temperature and humidity box (Table B1 in supplementary information files). After being oven dried for 50 h, some of the sausages were taken out, cooled to room temperature, and then sealed in polyethylene plastic bag and stored at room temperature (20 ± 2 °C) for 20 weeks. The remaining sausages were still oven dried for 70 h at 55 °C with 40% relative humidity. Throughout the oven-drying process of 120 h, the flavour fingerprinting maps of Chinese-style sausage were acquired at 0 h, 12 h, 24 h, 36 h, 42 h, 48 h, 54 h, 60 h, 72 h, 96 h and 120 h, respectively, and 25 sausage samples were acquired volatiles by E-nose at each detecting point during the processing; meanwhile, during the storage of 20 weeks, the volatile compounds were extracted at 0 w (week), 2 w, 4 w, 6 w, 8 w, 12 w and 20 w, respectively, and volatiles of 25 samples were acquired by E-nose at each detecting point during the storage. Among these samples, when establishing classification and regression models, 15 samples were used for the calibration datasets and 10 samples for the prediction datasets at each detecting point during processing and storage.

2.2. Electronic nose

An odour fingerprint map of Chinese-style sausage during processing and storage was extracted using a portable electronic nose (PEN 3, Win Muster Air-sense Analytics Inc., Germany) with an array of 10 sensors (S_1: sensitive to aromatic compounds, sensitivity: 10 ppm; S_2: sensitive to nitrogen oxides, sensitivity: NO_2, 1 ppm; S_3: sensitive to ammonia and aromatic compounds, sensitivity: Benzene, 10 ppm; S_4: sensitive to hydrogen, sensitivity: H_2, 100 ppb; S_5: sensitive to alkenes and aromatic compounds, sensitivity: Propane, 1 ppm; S_6: sensitive to methane broad range, sensitivity: CH_3, 100 ppm; S_7: sensitive to sulphur compounds, sensitivity: H_2S, 1 ppm; S_8: sensitive to alcohols and partially aromatic compounds, sensitivity: CO, 100 ppm; S_9: sensitive to aromatics compounds and sulphur organic compounds, sensitivity: H_2S, 1 ppm; S_{10}: sensitive to alkane, sensitivity: CH_3, 100 ppm). For each measurement, a sausage sample was placed in a 1000 mL polyethylene plastic case sealed with tinfoil and left at 25 °C for 20 min to equilibrate the volatiles in the headspace. Subsequently, the measurement started, in which headspace volatiles were pumped into the sensor chamber at a rate of 300 mL/min, while the generated signals of the 10 sensors continuously changed and were timely recorded by the Win Muster software coupled with an electronic nose in a portable computer. The optimum time for the detection was set as 60 s to obtain a stable signal for further analysis. Following the detection time, the optimum time for the cleaning phase was set as 110 s; automatic zero phase and sample preparation phase were both set as 5 s. Hence, the total measurement time was 3 min for each sample. At the same time, the blank sample was detected at the same condition in order to confirm that responses values of all sensors tended to be 1.0 before each test in the experiment.

2.3. Lipid oxidation index

Acid value and peroxide value during the processing and storage were determined to evaluate the degree of lipid oxidation of Chinese-style sausage. Acid value reflects the free fatty acid content, which is a vital indicator in determining the quality of Chinese-style sausage (Qi & Zhou, 2013). Peroxide value is a parameter of weighing the content of peroxide or hydroperoxide as primary products of lipid oxidation and is often used to characterise the degree of lipid oxidation in meat products (Mehta, Darji, & Aparnathi, 2015). In general, the higher the AV or POV is, the worse is the sausage sample quality (Armenta, Garrigues, & de la Guardia, 2007). In this work, the extraction of lipids from sausage samples was implemented according to China's official standard named 'Method for analysis of hygienic standard of meat and meat products' (GB/T 2003-5009.44, 2003).