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Evaluation of the aroma quality of Chinese traditional soy paste during storage based on principal component analysis

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ABSTRACT

Soy paste, a fermented soybean product, is widely used for flavouring in East and Southeast Asian countries. The characteristic aroma of soy paste is important throughout its shelf life. This study extracted volatile compounds via headspace solid-phase microextraction and conducted a quantitative analysis of 15 key volatile compounds using gas chromatography and gas chromatography–mass spectrum analysis. Changes in aroma content during storage time were analyzed using an acceleration model (40 °C, 28 days). In the 28 days of storage, results showed that among key soy paste volatile compounds, alcohol and aldehyde contents decreased by 35% and 26%, respectively. By contrast, acid, ester, and heterocycle contents increased by 130%, 242%, and 15%, respectively. The overall odour type transformed from a floral to a roasting aroma. According to sample clustering in the principal component analysis, the storage life of soy paste could be divided into three periods. These three periods represent the floral, roasting, and pungent aroma types of soy paste.

1. Introduction

Soy paste, a traditional flavouring, is processed using cooked soybean seeds as a raw material and then fermented by koji. Soy paste has been widely used for umami-enhancing applications in East and Southeast Asia for hundreds of years. It endows food with a roasting and sauce aroma despite its different names in various areas, such as dajiang (in China), doenjang (in South Korea), and miso (in Japan) (Blandino, Al-Aseeri, Pandiella, Cantero, & Webb, 2003). Soy paste has long been used in kitchen cooking, but it is now extensively applied in the industrial manufacturing of noodles and instant foods. The flavour qualities of fermented soybean products vary significantly because of their differences in raw material, processing procedures, and fermentation environment.

Many studies have reported and identified several fundamental compounds in Japanese and South Korean soy paste using different extraction methods, such as 2-phenyl-2-butene aldehyde, 4-hydroxy-2 (or 5) -ethyl-5 (or 2) -methyl-3-dihydrofuran, methylmercaptan and 2-phenylethanol (Chung, 1999; Chung, Fung, & Kim, 2005; Ku, Chen, & Chiou, 2000; Leejeerajumnean, Duckham, Owens, & Ames, 2001; Mori, Kiuchi, & Tabei, 1983; Sugawara, 1991). Although the contents and numbers of aromatic compounds vary significantly with different extraction and quantitative analysis methods (Lee & Ahn, 2009; Zhao, Dai, Liu, Zhang, et al., 2011), soy paste aromatic compounds can be mainly divided into five groups: alcohols and phenols, aldehydes and ketones, acids, furans, and pyrazines.

In our previous work, 103 volatile compounds were extracted from traditional Chinese soy paste using simultaneous distillation and extraction (SDE), and 18 volatile compounds were identified as key constituents of the characteristic soy paste aroma (Zhang, Li, Lo, & Guo, 2010). Furthermore, Zhang et al. (2010) clarified that trimethylpyrazine (roasting cereal odour), 4-ethyl-2-methoxyphenol (barbecue flavour) and isovaleric acid (dirty socks smell) were three critical volatile compounds that formed the characteristic soy paste aroma.

Owing to the complicated microbial activities and biochemical reactions, the components, contents, and overall aroma characteristics of soy paste continuously change (Choi, Sohn, & Jeon, 1997; Park, Lee, Kim, & Lee, 1994; Seo, Chang, Ji, Lee, et al., 1996). This variation in aroma quality hinders the application of soy paste in the modern manufacturing of noodles and instant foods. Thus, it is important to maintain the aroma characteristics of the soy paste package, for a quality assurance purpose. Manufacturers, who produce soy paste or instant noodles, need to consider the effect of storage conditions on changes in aroma attributes and quality of soy paste. At present, the quality of soy paste is mainly decided by sensory evaluations. No attempts have been made to clarify how storage conditions affect the soy paste aroma quality. In addition, the procedures involved in the change of soy paste aroma during storage, as well as the type and shelf-life of soy paste suitable for particular applications, remains unclear.

The objectives of the present study were to identify and quantify the key volatile compounds in Chinese traditional soy paste,

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and to mathematically analyse their change during storage by applying principal component analysis (PCA). The storage experiments were performed using an acceleration model (40 °C, 28 days).

2. Materials and methods

2.1. Materials

Soy paste was obtained from Fangyuan Food Co., Ltd., Laizhou City, Shandong Province, China. The soy paste was produced by a Chinese traditional method and the commercially available soybeans (harvested in Northeast China) were cooked and fermented with koji for 90 days and aged for 30 days. Thereafter, 2 g of soy paste sample was placed in a 5 ml sample vial with a rubber stopper. Samples were stored at 40 °C in a thermostat incubator. In the 28 days storage experiment, new samples were placed in the incubator every 4 days.

2.2. Headspace solid-phase microextraction (HS-SPME) preparation of volatile compounds

HS-SPME was applied for the extraction of soy paste aromatic compounds during storage experiments. The soy paste sample was accurately weighed at 2 g, placed in a 15 ml headspace vial, stirred in a 60 °C water bath for 20 min before extraction, and kept at this temperature throughout the sampling. Thereafter, the extraction fibre (DVB/CAR/PDMS solid microextraction head, 50 μm, Supelco, Bellefonte) was inserted into the headspace of a headspace vial, and the tip was kept 0.5–1 cm above the liquid level for 20 min. The fibre was then drawn into a needle and introduced into a gas chromatograph injector. The injector temperature was held at 250 °C.

2.3. Chromatographic conditions

Gas chromatography–mass spectrometry (GC–MS) analysis was conducted on Agilent 6890NGC-5973IMS GC–MS equipped with an HP-INNO Wax Polyethylene Glycol capillary column (60 m × 0.25 mm i.d. × 0.25 μm film thickness, Agilent, USA). Carrier gas (helium) was applied at a flow rate of 1 ml/min. The GC oven temperature was held at 40 °C for 3 min, increased from 40 °C to 130 °C at a rate of 3 °C/min, maintained at 130 °C for 2 min, increased from 130 °C to 200 °C at a rate of 4 °C/min, and held at 200 °C for 5 min. The mass spectrometer was used in the electron impact (EI) scan mode with a scan range of masses from 33 *m*/*z* to 450 *m*/*z*. The ionisation energy was 70 eV. Electron multiplier voltage was 350 V, and scan rate was 0.2 s/scan.

GC–olfactometry (GC–O) analysis was conducted using an Agilent 7980 gas chromatograph interfaced to an Alpha-MOS sniffing device (Alpha-MOS Corporation, France). The gas chromatograph was equipped with an Rtx-Wax capillary column (30 m × 0.25 mm i.d. × 0.25 μm film thickness, Agilent, USA). Carrier gas (helium) was introduced at a flow rate of 1 ml/min. The GC temperature programme was the same as that for GC–MS. The split ratio of the effluent into the FID and the Alpha-MOS sniffer was 1:1.

2.4. Aroma extract dilution analysis (AEDA)

The soy paste (200 g) was mixed with distilled water (300 ml) and a small amount of zeolite in a 1000 ml round-bottomed flask. The mixture was extracted using 40 ml diethyl ether by means of SDE at 40 °C for 3 h. The extract was diluted using diethyl ether according to the volume ratios of 1:3, 1:9, 1:81, 1:243, and so on. The diluted sample (2 μl) was injected for GC–O analysis until the evaluators at the GC–O terminal could not smell it. The highest dilution ratio obtained was defined as the flavour diluting (FD) factor. Three professional evaluators from the Chinese Agricultural University performed the AEDA. Each sample was assessed for three times. Timing and description of aromas were recorded if they were agreed by at least two of the three evaluators.

2.5. Compound identification and quantification

Identification was based on the retention indices and mass spectra of reference standards matching in the National Institute of Standards and Technology (NIST 2.1) mass spectral library. All the standards that were analysed in this study were available. A positive identification of volatile compounds was confirmed by matching the retention indices and mass spectra of reference standards analyzed under the same experimental conditions. Retention indices were calculated using a C_6–C_{22} *n*–alkane series (Supelco) under the same conditions.

A series of concentrations of standard compounds with an internal standard 2-methyl-3-heptanone (1 mg/L compound solutions) was prepared. For key volatile compound quantification, a five-point calibration curve was developed for each of the identified key volatile compound. The calibration curve was drawn by plotting the concentration ratio of a standard compound to the internal standard against the corresponding peak area of the standard compound to that of the internal standard. The concentration of each compound was calculated based on the standard curve derived from the area ratio and the concentration ratio of each compound.

2.6. Statistical analysis

Every sample was measured thrice, and all experiments were conducted in duplicate. The significance of differences was evaluated by one-way ANOVA using the SPSS 16.0 software package. Values of $p < 0.05$ were considered statistically significant. The average value of aroma compound content in different samples was evaluated using principal component analysis (PCA) with maximum variation rotation.

3. Results and discussion

3.1. Key aromatic compound identification and quantitation of soy paste

Volatile compounds of soy paste were extracted by HS-SPME and subjected to GC–MS analysis. The total ion chromatogram and gas chromatogram are shown in Fig. 1. As shown in Table 1, 15 volatile compounds could be smelled by three evaluators in GC–O and AEDA analysis after diluted for 81 times ($\log_3 FD \geqslant 4$). Thus, these compounds could be defined as key volatile compounds of soy paste according to Zhang et al. (2010). In accordance with the numerical order of labeled peaks in Fig. 1B, the 15 identified key volatile compounds are as follows: 2-methyl-1-propanol, 3-methyl-1-butanol, 2,5-dimethylpyrazine, 2,6-dimethylpyrazine, ethyl lactate, trimethylpyrazine, 3-methylthio-propionaldehyde, furfural, benzaldehyde, isovaleric acid, ethyl phenylacetate, 2-phenylethanol, 2-acetylpyrrole, 4-ethyl-2-methoxyphenol, and 4-ethylphenol. Compared with the 18 key volatiles detected using SDE by Zhang et al. (2010), long-chain acids and esters such as *n*-octanol, acetic acid, and Ding esters were not detected in this study, which was mainly attributed to the difference in processing and fermentation conditions. Three additional key volatile compounds in soy paste that were not found in Zhang's study, i.e., 2-methyl-1-propanol, 3-methylthio-propionaldehyde and 3-methyl-1-butanol, were also identified