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Characterisation of aroma profiles of commercial soy sauce by odour activity value and omission test

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ABSTRACT

Twenty-seven commercial soy sauces produced through three different fermentation processes (high-salt liquid-state fermentation soy sauce, HLFSS; low-salt solid-state fermentation soy sauce, LSFSS; Koikuchi soy sauce, KSS) were examined to identify the aroma compounds and the effect of fermentation process on the flavour of the soy sauce was investigated. Results showed that 129 volatiles were identified, of which 41 aroma-active components were quantified. The types of odorants occurring in the three soy sauce groups were similar, although their intensities significantly differed. Many esters and phenols were found at relatively high intensities in KSS, whereas some volatile acids only occurred in LSFSS. Furthermore, 23 aroma compounds had average OAVs > 1, among which 3-methylbutanal, ethyl acetate, 4-hydroxy-2-ethyl-5-methyl-3(2*H*)-furanone, 2-methylbutanal and 3-(methylthio)propanal exhibited the highest average OAVs (>100). In addition, omission tests verified the important contribution of the products resulting from amino acid catabolism to the characteristic aroma of soy sauce.

1. Introduction

For thousands of years, soy sauce has been one of the most popular seasonings in Asian countries including China and Japan (i.e. with an annual production over 7 million tons), and is also increasingly consumed in the Western world, due to its intense umami taste, characteristic aroma and desirable components, including protein and antioxidants, such as isoflavones and superoxide dismutase (Gao et al., 2010; Li, Zhao, Zhao, & Cui, 2010; Steinhaus & Schieberle, 2007). Three fermentation processes are used globally for producing soy sauce: High-salt liquid-state fermentation soy sauce (HLFSS), low-salt solid-state fermentation soy sauce (LSFSS) and Koikuchi soy sauce (KSS) (as shown in Fig. 1). HLFSS and LSFSS are mainly used in China, while KSS accounts for about 85% of the total soy sauce consumed in Japan (Kaneko, Kumazawa, & Nishimura, 2012). Different fermentation processes employ different raw materials, brine solution concentrations, strains of microorganisms, *moromi* fermentation time and temperature, which in turn influence the flavour of soy sauce (Gao, Zhao, Zhao, Cui, & Ren, 2009; Kaneko et al., 2012).

The volatile profiles of soy sauce have been extensively studied over the past 50 years, and more than 300 volatile compounds belonging to 12 chemical classes have been identified (Gao et al., 2010; Giri, Osako, Okamoto, & Ohshima, 2010; Nunomura, Sasaki, Asao, & Yokotsuka, 1976; Sun, Jiang, & Zhao, 2012; Wanakhachornkrai & Lertsiri, 2003; Yan, Zhang, Tao, Wang, & Wu, 2008). The results have indicated alcohols, acids, esters and aldehydes as the most abundant volatile classes in soy sauce. Soy sauces are typically characterised for their malty, roasty, caramel-like, seasoning-like and flowery aromas (Steinhaus & Schieberle, 2007), which have been thought to result from a complex mix of many volatile constituents at appropriate proportions (Nunomura et al., 1976). However, it has been shown for a considerable number of foods that only a limited number of so-called key aroma-active components in a food actually contribute to the overall aroma (Steinhaus & Schieberle, 2007). Thereafter, investigations have been focused on the identification of key aroma-active volatiles, using techniques like gas chromatography-olfactometry (GC-O) and aroma extract dilution analysis (AEDA) (Baek & Kim, 2004; Kaneko, Kumazawa, & Nishimura, 2013; Kaneko et al., 2012; Lee, Seo, & Kim, 2006). Baek and Kim (2004) were the first to study the volatiles in Korean soy sauce using SPME–GC-O and reported the highest odour activities of 3-(methylthio)propanal followed by 3-methylbutanoic acid and 2,5-dimethyl-4-hydroxy-3(2*H*)

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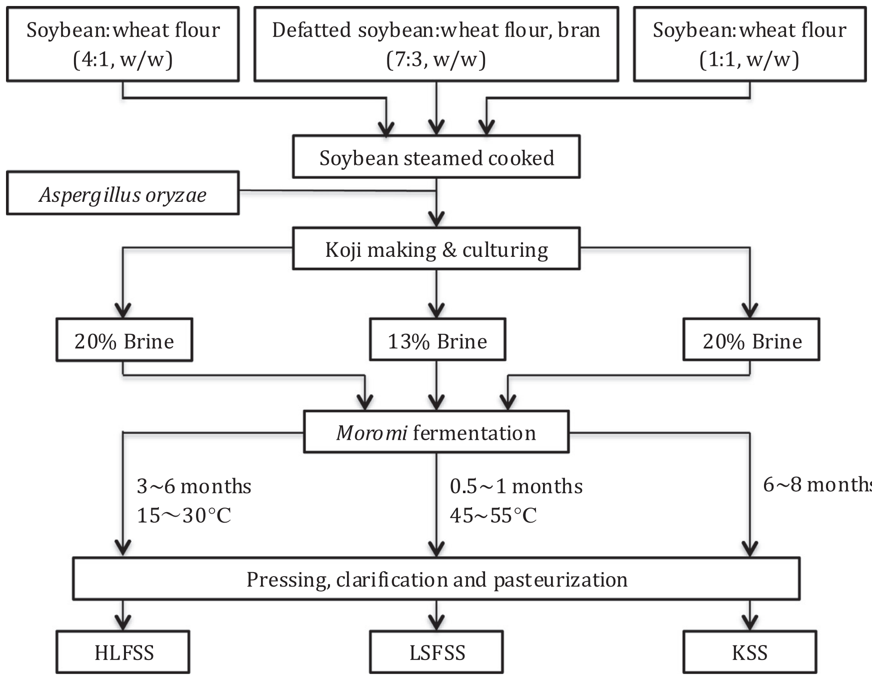

Fig. 1. Three procedures of manufacturing soy sauces (HLFSS, LSFSS and KSS).

furanone (4-HDMF). 3-(Methylthio)propanal, 4-ethylguaiacol and three furanones – 4-HDMF, 3-hydroxy-4,5-dimethyl-2(5H)-furanone (sotolon) and 5(or 2)-ethyl-4-hydroxy-2(or 5)-methyl-3(2*H*)-furanone (4-HEMF) – were proposed as the most aroma-active constituents in Japanese-style soy sauce (Kaneko et al., 2012, 2013; Steinhaus & Schieberle, 2007).

Recently special attention has been devoted to the characterisation of aroma profiles of soy sauce, which led to the conclusion that fermentation technology could significantly contribute to diverse aromas and odorants of various soy sauce (Feng et al., 2014; Kaneko et al., 2012). However, in most cases the speculation lacks sufficient quantification and sample sizes are inadequate. For example, Steinhaus and Schieberle (2007) determined 2-phenylethanol as an important odorant in a model aroma mixture of Japanese soy sauce (which contained 13 volatiles). Contradictorily, Kaneko et al. (2012) did not detect 2-phenylethanol after comparing the key aroma compounds in five different types of Japanese soy sauce. Thus, further research is required to verify quantitatively the roles of these key aroma-active compounds in the overall aroma of a wide range of commercial soy sauces.

The aims of this study were to (i) quantify the aroma compounds that were previously identified in soy sauce by SPME–GC-O, (ii) evaluate these key volatiles through the calculation of their odour activity values (OAVs) and omission tests, and (iii) allocate the aroma-active compounds that account for the characteristic aromas corresponding to different fermentation processes by analysing a representative number of soy sauces.

2. Materials and methods

2.1. Soy sauce samples

Twenty-seven samples of commercial soy sauce, including 14 high-salt and liquid-state fermentation soy sauces (HLFSS) from 9 different brands, 8 low-salt and solid-state fermentation soy sauces (LSFSS) from 6 different brands, and 5 Koikuchi soy sauces (KSS) from 5 different brands, were kept in their original containers and stored in a laboratory refrigerator (4 °C) before extraction.

2.2. Chemicals

2-Methylbutanal, 3-methylbutanal, phenylacetaldehyde, 3-methylbutanoic acid, ethyl isobutyrate, 3-(methylthio)propanal (methional), (−)-ethyl L-lactate, (*E*)-2-octenal, 4-ethylphenol, 2-acetylpyrrole and isobutyl acetate were purchased from Sigma-Aldrich (Steinheim, Germany). Ethanol, 1-propanol, 2,3-butanediol, 3-methyl-1-butanol, 2-methyl-1-butanol, 1-octen-3-ol, 2-ethyl-1-hexanol, benzyl alcohol, 2-methylpropanal, nonanal, 2,4,7,9-tetramethyl-5-decyn-4,7-diol, 5-methyl-2-phenyl-2-hexenal, 2-pentanone, 3-pentanone, 2,3-pentanedione, 3-hydroxy-2-butanone, 2,3-butanedione, 5-methyl-2-hexanone, 2-heptanone, acetic acid, 2-methylpropanoic acid, butanoic acid, 2-methylbutanoic acid, ethyl acetate, ethyl butanoate, butyl acetate, ethyl isovalerate, methyl benzoate, ethyl benzoate, diethyl succinate, ethyl phenylacetate, 2,5-dimethylfuran, furfural, 2-furanmethanol, 5-methyl-2-furancarboxaldehyde, benzofuran, 4-ethylguaiacol, dimethyl sulphide, dimethyl trisulphide, 3-(methylthio)propanol, 2,5-dimethyl-pyrazine, 2-ethyl-3-methyl-pyrazine and 2,3,5-trimethylpyrazine were purchased from Aladdin (Shanghai, China). Sodium chloride (NaCl) was purchased from the China National Pharmaceutical Ground Corporation (Shanghai, China). 2-Methyl-3-heptanone that was employed as an internal standard in this study was purchased from Sigma-Aldrich (Steinheim, Germany).

2.3. Extraction of volatile compounds

The extraction of volatile compounds from the 27 soy sauces was performed following our newly published protocol (Feng et al., 2014) that was modified based on the method of Yan et al. (2008). The SPME Trisplus automated sampler equipped with a 75 μm carboxen/polydimethylsiloxane fibre (CAR/PDMS, Supelco, Inc., Bellefonte, PA) was employed in this study.

2.4. GC–MS analysis

Identification and analyses of the volatiles in the 27 soy sauces were carried out following the method described in our previous