SPME/GC-MS characterization of volatile compounds of Chinese traditional-chopped pepper during fermentation

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ABSTRACT
Fermented-chopped pepper is a traditional-fermented vegetable in China. Chopped pepper is generally made with pepper, garlic, and ginger. In present study, the volatile compounds, obtained from chopped peppers during spontaneous fermentation, were analyzed by solid-phase microextraction (SPME) and gas chromatography-mass spectrometry (GC-MS). One hundred and forty volatile compounds were identified by solvent-assisted flavor evaporation, including 42 hydrocarbons, 34 esters, 16 alcohols, 21 aldehydes, 8 acids, 4 ketones, 4 ethers, and 9 other compounds. Alcohols, esters, and ketones were the dominant volatile fractions. The terpenes were found to be the main contributors to chopped pepper samples. It is important to note that among terpenes, such as β-phellandrene, α-curcumene, elemene, geraniol and nerolidol may originated from ginger. In addition, diallyl disulfide and diallyl sulfide, the two major sulfur components, may originated from garlic volatile oils. The results indicated that the ginger and garlic as raw materials played an important role in the flavor of chopped pepper. The contents of esters significantly increased during the fermentation process, the ethyl palmitate and ethyl linoleate were the main esters. The content of β-ionone as an degradation compound of carotenoids increased significantly with the fermentation period. Principal component analysis (PCA) was applied to discriminate the fermented-chopped peppers samples according to different times, and the fermented-chopped pepper samples were clearly differentiated on PCA plots. The fermentation stage was mainly affected by esters, alcohols, aldehydes and terpenes.

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INTRODUCTION
Peppers are one of the most valuable vegetables and have been extensively used over the years as a spice to add flavor in food preparation. In addition, peppers are a good source of several health-promoting compounds, such as flavonoids, carotenoids, vitamin C, and capsaicinoids. Nowadays, peppers are widely used as ingredient in the foods, especially the fermented foods, such as kimchi. Kimchi is a traditional-fermented food in both North and South Korean and have many different types of dishes. The most famous type of kimchi is composed of cabbages or radishes, and several seasonings including scallions, garlic, ginger and pepper powders are always used to flavor the kimchi. In China, peppers are an important seasoning in the foods, especially in the Hunan and Sichuan cuisine. The fermented peppers are often used as a seasoning in cuisines, like steamed fish head with chopped pepper (also called Duojiao-yutou). Chopped pepper is a traditional-fermented condiment in the southern regions of China. Chopped pepper is generally made with pepper, garlic,
and ginger. Unlike kimchi, peppers are the main vegetable used in the traditional-fermented chopped pepper, and fermented at a higher temperature.

Traditional-chopped pepper is prepared by spontaneous fermentation for 1–2 weeks without the use of starter cultures, leading to the growth of various microorganisms derived from the surrounding environment, including incubation shelves, air, and raw materials. The chopped pepper represents a very complex microbial ecosystem comprising bacteria and yeasts, which are responsible for the production of various metabolites such as amino acids, organic acids, and active metabolites that contribute to its nutrition, taste, flavor, and functionality.

Literature on the flavor of the fermented-chopped pepper is abundant and mainly focuses on the characterization of volatile components in the peppers under different salt concentrations or inoculation with different starters.\[^{1-3}\] Luo et al.\[^{1}\] extracted the volatile components of three samples of fermented-chopped pepper. The major compounds identified were benzaldehyde, linalool, methyl salicylate, α-himachalene, ethyl caprate and 1-caryophyllene. Fermentation with microorganisms can not only change the aroma, flavor, and textures of substrates, but improve the quality of foods. Zhao et al.\[^{4}\] demonstrated Lactobacillus isolated from traditional-chopped pepper were able to ferment peppers, and further study found that the chopped pepper fermented with Lactobacillus showed the highest content of lactic acid and ethyl acetate. Xu et al.\[^{5}\] used headspace gas chromatography analysis to determine the volatile compounds formed from chopped pepper fermented with Zygosaccharomyces rouxii. They found that the Z. rouxii can produce high contents of some major flavor compounds (ethanol, linalool, α-terpineol and ethyl palmitate). Han et al.\[^{6}\] compared the volatile compounds produced from chopped pepper fermented by L. fermentum, L. plantarum, Pediococcus acidilactici, and Streptococcus lactis, they found that chopped pepper fermented with L. fermentum showed the highest content of linalool, cedrene and furans, whereas S. lactis produced higher amounts of ketones and phenols.

The primary aim of this research was to determine the volatiles in Chinese traditional-chopped pepper during fermentation, using headspace solid-phase microextraction. The volatiles were analyzed and the concentration changes in the volatile compounds during fermentation were described.

Materials and methods

**Preparation of chopped pepper**

In this study, chopped pepper samples were prepared according to the procedure described by Ye et al.\[^{7}\] Peppers (Capsicum annuum L.) were grown at Hunan Academy of Agricultural Sciences. All peppers received similar water and fertilizer treatments. The longline pepper fruits were harvested when the whole fruit color was red. The garlic (Allium sativum L.) and ginger (fresh rhizome of Zingiber officinale Roscoe) were purchased from a local market. Briefly, the peppers were washed, dried, chopped into small pieces (0.5–1 cm × 0.5–1 cm), salted with 10% (w/w) salt, and put into 5 L pickle jars along with spices including garlic (5%), ginger (3%), calcium chloride (0.1%), and a white spirit (0.5%). The jars were sealed with water to exclude air and fermented in a 30°C incubator. The chopped pepper samples were obtained at 0, 6, 12, 18 and 24 days and kept in a deep freezer at −80°C for volatile compounds analysis.

**Extraction of volatile components of chopped pepper samples**

The headspace solid-phase microextraction (HS-SPME) method was employed to extract the volatile compounds from the chopped pepper samples. The volatile components were extracted according to the method described by Luo et al.\[^{1}\] with slight modification. Samples (30.0 g each) were blended with 30.0 mL of distilled water and homogenized, and 2.000 g of the sample was immediately transferred into 15 mL SPME vials (Supelco, Bellefonte, PA, USA) within 2 min followed by addition of 5 μL 5 mg/kg 2-methyl-3-heptanone (Sigma-Aldrich Co. USA) in methanol as an internal
standard. After sample preparation, each vial was placed in a water bath at 70°C for 15 min with agitation to reach an equilibrium state. Subsequently, a fiber coated with 50/30 μm DVB/CAR/PDMS (Supelco, Bellefonte, PA, USA) was injected into the vial for 30 min to absorb volatile compounds. All reported results constitute the mean of triplicate measurements.

**Gas chromatography-mass spectrometry analysis**

GC-MS analysis was carried out using a Shimadzu GC-2010 gas chromatograph connected to a QP-2010 mass spectrometry system (Shimadzu Corp., Kyoto, Japan). A DB-Wax fused silica capillary column (30 m long × 0.25 mm internal diameter × 0.25 μm film thickness) was used with helium as the carrier gas at a constant flow rate of 1 mL/min. The heating gradient program was 40°C for 2 min, followed by increasing at 4°C/min to 80°C and remaining for 1 min. Thereafter, the temperature was raised to 240°C at 3.5°C/min and held on this stage for 4 min. Helium (purity 99.999%) carrier gas flow was at a constant pressure of 2 psi. All mass spectra were acquired in the electron impact (EI) mode (70 eV ionization energy, source temperature 225°C). EI mass spectra ranged from 30 to 550 a.m.u.

**Volatile compound identification and quantification**

Volatile compounds were identified by comparing the mass spectra of the samples with the data system (NIST 08 and WILEY 05). Quantitative results were calculated from the peak areas of the GC-MS chromatograms with three extractions for each sample with the use of the internal standard method and reported as μg/kg weight of the sample.

**Statistical analysis**

The mean values and standard deviations were calculated using the data obtained from three separate experiments. Means were compared using Duncan’s multiple range test method in SAS, version 8 (SAS Institute, Cary, NC, USA). Principal component analysis (PCA) was conducted to investigate the volatile compounds in the chopped peppers according to fermentation times using SPSS software (version 12.0, Chicago, IL, USA).

**Results and discussion**

**Volatile compounds in fermented-chopped pepper**

The total ion chromatograms of the fermented-chopped peppers are given in Figure 1. A total of 138 identified volatile compounds were listed in Table S1. These compounds comprising 16 alcohols, 34 esters, 21 aldehydes, 42 hydrocarbons, 8 acids, 4 ketones, 4 ethers, and 9 other compounds.

In the raw materials, hydrocarbons and esters were the most important chemical family, accounting for at least 70% total chromatographic area, followed by aldehydes and alcohols with 19.86% and 4.62%, respectively (Figure 2). However, the proportion of hydrocarbon content in the total content decreased during fermentation, which may be due to the conversion of some terpenes to terpene derivatives. Among the hydrocarbons, 24 terpenes, including α-pinene, camphene, β-pinene, β-myrcene, α-phellandrene were identified in the fermented-chopped peppers. It is noteworthy that β-phellandrene, α-camphene, 1,8-cineole, geraniol and nerolidol were reported as the main volatile components of fresh ginger.[8,9] Geraniol is responsible for the typical floral aroma of grapes and contributes to the aroma of wines, and nerolidol is a volatile sesquiterpene that contributes to the floral aroma of teas.[10,11]

The contents of most terpenes were higher in samples fermented for 6 days and then gradually decreased in the following fermentation period, including those of α-phellandrene, β-elemene, α-zingiberene and himachalene. Previous report indicated that the contents of terpenes were higher in fermented peppers...
for 12 days.\textsuperscript{[12]} The differences in trend may due to the different raw materials and fermentation method used. Terpenes occupied a great part of the multiple compounds and are the most variegated class in natural plants, and they exist in a glycoconjugated form in some fruits and vegetables.\textsuperscript{[13,14]} The contents of α-phellandrene and α-zingiberne originated from ginger was relatively higher at the end of fermentation, suggesting those compounds result in a more significant contribution to the overall aroma of chopped pepper.

Esters, which contribute to the fruity and sweet flavor, accounted for 22.99\% to 31.07\% of the components during the fermentation of chopped pepper. Among the 34 esters, the content of ethyl salicylate was increased slightly during the fermentation period, which was previously reported as an aroma-active compound in gochujang with peppermint aroma.\textsuperscript{[15]} The contents of ethyl palmitate, methyl linoleate, ethyl linoleate and ethyl stearate were significantly increased during the fermentation. Previous studies demonstrated that palmitic acid, oleic acid and linoleic acid were the major fatty acid in fresh red pepper and the pepper seeds, and the ethyl palmitate, methyl linolenate, ethyl linoleate, ethyl stearate were the main esters in fermented peppers.\textsuperscript{[16–18]} These esters in chopped pepper may be formed from alcohols and acids by the esterases produced by lactic acid bacteria.

Figure 1. GC-MS total ion chromatograms of volatile compounds in fermented chopped peppers (a: raw materials, b–e: 6, 12, 18, and 24 d of fermentation, respectively).
should be noted that the contents of linoleates were increased during the fermentation with the decrease of linolenates, and the linolenates were only detected in the initial stage of fermentation. The possible causes for decrease of linolenates may be the autoxidation, and most intense odor compounds were formed at room temperature. [19]

Previous studies reported that the ethyl linoleate was the main ester in fermented pepper and soybean paste.[12,20] There is now a general consensus that the ethyl linoleate can lower hepatic and plasma cholesterol levels[21,22], and a higher content of ethyl linoleate suggested that the chopped pepper may have good health-promoting activities.

In present study, the contents of acids increased drastically from 0 d to 12 d but decreased from 12 d to 18 d. These compounds, which have coconut aromas and greasy, pungent odors depending on their concentration, may be formed either via bacterial degradation of amino acids or lipid oxidation.[23] Previous study indicated that citric acid, malic acid and acetic acid were detected in the spontaneous fermented-chopped pepper.[7] However, these organic acids were not detected in this study. It has been reported that the number and relative content of volatile compounds are related to pepper varieties, while the number and relative contents of volatile compounds in the same pepper variety were different with the amounts of added NaCl.[1] The main acids in samples were linoleic acid, tetradecanoic acid, neric acid and nonanoic acid. The neric acid was identified for the first time in this study, and the content of neric acid was only detected in the late fermentation period. The other three acids were previously reported as main acids in peppers. The content of linoleic acid was detected after 6 days, and sharply deceased in the following fermentation period. Linoleic acid was the main fatty acid in the red pepper seeds oil[17], and the decreased of linoleic acid was mainly due to the involvement in the formation of linoleat. Tetradecanoic acid only detected in the end of fermentation, it may be formed by the oxidation of tetradecal.

In this study, the amounts of sulfur-containing compounds gradually increased during the fermentation period, which consistent with the results of volatile composition of kimchi.[24] There were four sulfur-containing compounds detected in present study, such as dialyl sulfide, methyl 2-propenyl disulfide, diallyl disulfide and di-2-propenyl trisulfide. The sulfur-containing compounds are a particular class of natural products in plants, especially in pepper, garlic, onion, cabbage, and they can attribute to sharp, harsh, and pungent flavors.[25] Sulfur compounds were reported as a major volatile components of kimchi, and the formation of various sulfide derivatives were affected by fermentation conditions.[24] Among the sulfur

Figure 2. Contents of volatile fractions in the chopped peppers during fermentation.
compounds, the amount of diallyl disulfide was much higher than that of the other sulfur-containing volatiles. Previous study reported that diallyl disulfide and diallyl sulfide were the major components in garlic volatile oils. The increase of sulfur compounds may be related to metabolic transformation of microorganisms involved in the fermentation of chopped pepper.

Four ketones were detected in samples. 2-Undecanone was the major ketone in the initial stage of fermentation, and gradually decreased in the following fermentation period. 2-Undecanone was reported as a major volatile compound in chili pepper, and has a characteristic greasy scent, ruta-like aroma, and citrus-like odor notes.

In contrast, the content of β-ionone was increased significantly with the fermentation period, but was not detected in the early stage of fermentation. As a terpene derivative, β-ionone occurs in carotenoid-rich plants, including red peppers, tomatoes, carrots, and teas. It is well accepted that β-ionone is one of the main compounds formed by the degradation of carotenoids, and it can contribute to woody, violet, fruity and raspberry-like odor notes. The macromolecules including carotenoids in the plant can be degraded by the lactic acid bacteria present in the fermented vegetable.

The contents of all alcohols were gradually increased from 4.62% to 4.81% during the fermentation. 16 alcohol compounds were detected in chopped pepper, citronellol and eucalyptol were the main alcohol. Citronellol and eucalyptol were previously reported as main alcohol in fresh ginger, and the contents of that two alcohols was relatively stable. The α-panasinsen and nonadecanol-1 were detected in the later stage of fermentation.

The contents of aldehydes were highest during the initial stage of fermentation, and then gradually deceased since they could be converted into alcohols and acids during the fermentation process. Among aldehydes, most compounds were previously found in peppers, such as hexanal, benzaldehyde, benzeneacetaldehyde, nonanal, and tridecanal. The major aldehydes were citral and tetradecanal, which were also main volatile compound in ginger with citrus and milk-like aroma, respectively. C6 and C9 aldehydes are volatile compounds that have a fresh green and cucumber-like flavor in plant. In chopped pepper samples, the contents of hexanal, nonanal and nonenal decreased as the fermentation time increased. The hexanal, nonanal and nonenal can be formed from linolenic acid through oxygenation by lipoxygenase (LOX) and fatty acid hydroperoxide lyase (HPL). During fermentation, hexanal can be reduced to 1-hexanol by alcohol dehydrogenase. The content of 1-hexanol increased in the later fermentation period with the decrease of hexanal. Hexanol is a common and important flavor component of some vegetables and plants like peppers. It is derived from the bioremediation of unsaturated fatty acid and is also a prerequisite for the formation of long-chain esters.

For the other 9 compounds, 3,4-dimethylthiophene, 1,3-dithiane, and 1,2-benzisothiazole are heterocyclic compounds, and 4-ethylphenol, 4-ethyl-2-methoxyphenol, and 2-methoxy-4-vinylphenol are phenolic compounds. In present study, the three heterocyclic compounds were relatively low in content. All phenolic substances appeared at the end of the fermentation process (24 d). It has been reported that 4-ethylphenol and 4-ethyl-2-methoxyphenol are present in fermented soybean paste (Doenjang). Therefore, it is highly probable that 4-ethylphenol and 4-ethyl-2-methoxyphenol are derived from the fermentation of pepper raw materials by microorganisms.

**Principal component analysis**

The present study applied PCA to understand how the volatile compounds from the chopped pepper samples as analyzed from GC-MS data sets varied with the fermentation time. As shown in Figure 3, the constructed PCA plot explained 67.3% of the total variance, comprising 44.2% from the first principal component (PC1) and 23.1% from the second principal component (PC2). Classifying the plots according to fermentation time revealed that the profiles of the volatiles moved from right to left along the PC1 dimension with increasing fermentation time. As shown in Figure 4(a), most esters, such as ethyl oleate, ethyl palmitate, octacosyl acetate, methyl linoleate, methyl linolenate, and ethyl stearate, contributed to the negative PC1 axis and exhibited marked concentration changes.
During the fermentation stage. These esters compounds had fruity, flowery, and winey aromas, which are crucial components in the aroma of fermented vegetables. The increases of contents of esters may be formed from alcohols and acids by the esterases produced by microorganisms. In addition, PC1 loading was also negatively related to some alcohols (α-panasinsen, nonadecanol), aldehydes (dodecanal), and terpenes, such as β-pinene, γ-elemene, and dodecanal. On the other hand, 1-octanol, geraniol, and 3-methylbutyric acid hexyl ester contributed to the positive PC2 axis, and the levels of these compounds were highest at the beginning of fermentation, which indicates that they originated mainly from the raw materials and were utilized as precursors for subsequent fermentation by microorganisms (Figure 4(b)). Some volatile compounds (methyl salicylate, α-zingiberene, and pentadecanal) contributed to the negative PC2 axis, and their concentrations increased as fermentation proceeded.

As is well known, cultivar type, fruit maturity, and processing methods and parameters play an important role in the creation of volatile compounds. A large amount of acids, alcohols, and esters were produced in the fermentation process by lactic acid bacteria. A large number of esters detected in the fermented-chopped peppers were produced during the growth of lactic acid bacteria and other microorganisms by biosynthesis and bioconversion.\[36\]

Conclusion

This study investigated the volatile compounds in fermented-chopped peppers during the fermentation process. The data collected from the present study provide quantitative information concerning the development of volatile compounds during the fermentation process of the chopped pepper. Most of the volatiles identified in the fermented-chopped pepper showed that the contents of alcohols, ketones, and esters increased as fermentation proceeded and that the esters and hydrocarbons were the predominant compounds in both number and content. Since ginglers are known to contain high levels of terpenes, most terpenes from chopped peppers (β-phellandrene, α-curcumene, elemene, geraniol and nerolidol) may be originated from ginger. Our results therefore suggested that terpenes can play a crucial role in the flavor of chopped pepper. Similarly, the contents of sulfur compounds were gradually increased with the
fermentation time. Diallyl disulfide and diallyl sulfide, the two major sulfur components, may be originated from garlic volatile oils and released from garlic by metabolism of microorganisms. The results indicated that the ginger and garlic as raw materials were important for the flavor of chopped pepper.

Figure 4. The PCA loading plots for the volatile compounds isolated from different chopped pepper samples; (a) PC1, (b) PC2.
Principal component analysis (PCA) was applied to discriminate the fermented-chopped peppers samples according to different fermentation times, and the fermented-chopped pepper samples were clearly differentiated on PCA plots. Most esters, such as ethyl oleate, ethyl palmitate, octacosyl acetate, methyl linoleate, methyl linolenate, and ethyl stearate increased as fermentation proceeded. It is noteworthy that the pepper seeds are major source of palmitic acid, oleic acid and linoleic acid, and it was useful for improving the flavor of chopped pepper by using pepper seeds as raw material.

The results indicated that the flavor profiles of fermented-chopped pepper can change significantly during the fermentation process. The highlighted volatile compounds during the fermentation of chopped peppers can be used to as indicators for assessment of quality of chopped pepper products, such as inoculated fermented-chopped peppers.

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