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Physicochemical Properties and Aroma Compounds Analysis in Watermelon Soy Sauce

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Abstract

Watermelon soy sauce (WSS) is a liquid condiment usually prepared using watermelon juice, soybeans, and wheat flour through the process of making *koji* and natural fermentation. It is widely used in Chinese culinary art, despite the lack of knowledge about its aromatic compound content. Here, we characterized the physicochemical properties, free amino acid composition, and volatile compounds of WSS using SPME-GC/MS and E-nose. We noticed that WSS had the highest total acid content but the lowest amino nitrogen and reducing sugar contents compared with commercial soy sauce. Moreover, the highest amounts of Glu and Pro were observed in WSS. A total of 173 volatile compounds were identified in WSS, including alcohols, hydrocarbons, esters, ketones and aldehydes. The E-nose analysis showed a good capacity of differentiating braised samples mainly through W5S, W1S, W1W, W2W, and W3S sensors. The analysis of relationships between flavor components and free amino acids in soy sauce samples showed that Ser, Gly, Val, Ile, Leu, Ph,e and Lys had a strong positive correlation with alcohol and acidic compounds. Moreover, Pro was found to be correlated with aldehyde, ketone, heterocyclic compounds, sulfur compounds, and benzene, while Glu was correlated with hydrocarbons, aldehyde, and benzene. This study could provide important information regarding WSS quality control, characterization, and aroma improvement.

Keywords: watermelon soy sauce; physicochemical properties; volatile compounds; SPME-GC/MS; E-nose

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1. Introduction

Soy sauce is characterized as a salty liquid condiment that is produced in a traditional manner by subjecting soybeans and wheat to a fermentation process by the action of *Aspergillus sojae* or *Aspergillus oryzae* molds. Originally, soy sauce was known to originate from a traditional Chinese product called "*Chiang*" about 3000 years ago [1]. Soy sauce, as a common and novel flavoring agent, is widely popular in China as well as Southeast Asian countries like Korea, Indonesia, and Japan [2]. It has become the most favored traditional fermented soybean product among Asian and Western nations [3]. Soy sauce is not only nutritious but also contains various physiologically active compounds, such as furanone, phenolic acids, organic acids, and peptides [4]. The presence of these compounds provides soy sauce with various properties, such as antioxidant effects, anti-cancer benefits, the ability to reduce high uric acid levels, and the potential to slow the progression of cataracts [5].

Generally, the quality of soy sauce depends on some factors, such as the fermentation method and raw materials used in the production. Soy sauce fermentation can be considered a traditional fermentation based on the raw materials and method or can be classified in terms of salt content (high-salt fermentation and low-salt fermentation) [6,7]. High-salt fermentation employs a high-brine solution (17–20%) and operates at low temperatures (15–30 °C) [8,9]. Under these specific conditions, microbial metabolism and biochemical reactions are inhibited, and so this process requires a longer period of fermentation that can range from several months to about 4 years [6]. For example, wild-type yeast strains, recognized for their aroma-producing capabilities, are difficult to grow and ferment in this condition [10]. Therefore, a variety of traditional and innovative techniques, such as random mutagenesis, adaptive evolution, and genome shuffling, among others, have been developed to create robust strains suitable for food fermentation [11–13].

Watermelon soy sauce (WSS) is a liquid condiment used in the south of China, which is usually prepared using watermelon juice, soybeans, and wheat flour as the primary raw materials through the process of *koji* making and natural fermentation (Figure 1). Unlike the fabrication of soy sauce, the traditional manufacturing procedure of WSS is that matured *koji* was mixed with solid salt and watermelon juice (approximately 3-fold) rather than saline. The production of watermelon sauce commonly takes a long time to ferment and age. During this process, complex chemical reactions lead to the formation of a variety of volatile compounds [14]. Moreover, during fermentation, the various metabolites are produced, involving peptides and reducing sugars in the soy sauce. These bioactive compounds could increase the degree of the salty taste, whereas bitter amino acids may lead to a rising tendency in the mellowness of WSS. These components play variegated significant roles in the fermentation process and together help to achieve the unique flavor profile and fragrance of the soy sauce [15]. Moreover, the unique aroma and attractive color of soy sauce were considered related to the probable Maillard reaction. Studies have shown that furan 2-formaldehyde was the main flavoring substance in soy sauce produced by pentose through the Maillard reaction [16].

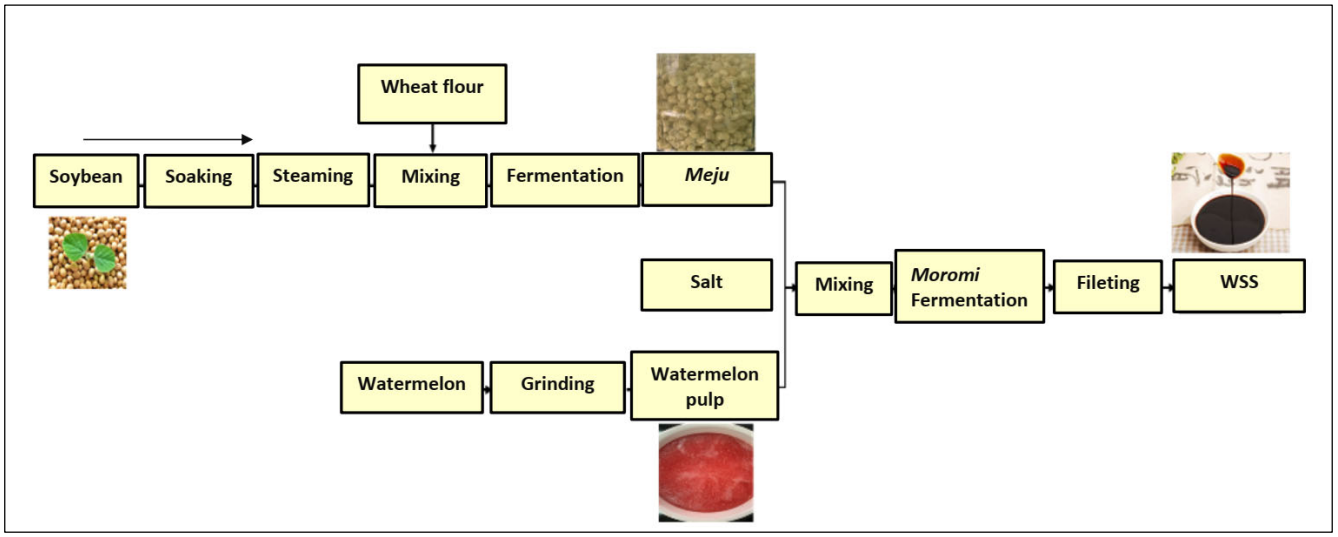

Figure 1. Flowchart of the fermentation process for the production of WSS in the traditional method.

Gas chromatography mass spectrometry (GC-MS) is a reliable method used to detect, quantify, and separate the aromatic compounds in foodstuffs. GC-MS spectra do not provide the particular information regarding sensory perception, and therefore GC-MS is not adequate for characterizing the foodstuffs for quality purposes [17]. In contrast, E-sensing technologies, such as electronic nose (E-nose), have mimicking ability similar

to that of human olfactory function regarding flavor and aroma recognition [18,19]. Metal oxide semiconductor (MOS) E-nose has been successfully employed for analyzing aromas in various foodstuffs. E-nose exhibits a sensor system and chemical imaging function with associated advantages of broad-spectrum responses of analyzed volatile compounds and cross-sensitivity in complex mixtures [19]. A combination of E-nose and GC-MS has been employed successfully for evaluating aromatic compounds in Chinese jujube [18], stinky tofu brine [19], and various berries [17]. Moreover, Zhu et al. [20] elucidated the main synthesis pathway of aromatic compounds in sauce-flavor Daqu at the genetic, proteomic, enzymatic, and metabolomic levels, providing new ideas for the investigation of key flavor substances in Maotai flavor.

Watermelon juice contains abundant amounts of sucrose, fructose, and glucose [21], suggesting that WSS prepared from soybeans and watermelon juice might contain higher alcohols, aldehydes, and other aromatic compounds than soy sauce prepared from soybeans and saline under the same conditions. The characteristics of various soy sauces prepared in different conditions (fermentation times, temperatures, and ratios of ingredients), such as *Koikuchi Shoyu*, *Usukuchi Shoyu*, and *Amakuchi Shoyu* in Japan [22], *Ganjang* in South Korea [23], and *Kecap manis* in Indonesia [1], have been studied. However, to our knowledge, no published report on the characterization of physicochemical properties and volatile compounds in WSS is available in the literature. This study was undertaken to characterize the physicochemical properties of the aroma compounds of WSS by using SPME-GC-MS/MS and E-nose, in comparison with commercial soy sauces.

2. Materials and Methods

2.1. Sauce Samples

The traditional natural sun-dried watermelon soy sauce was obtained from a local market based in Henan Province, China. It was fermented for a year in open-air clay pots using soybean, watermelon juice, and salt as materials. Commercial soy sauces—samples SS1 and SS2—were bought from a local market in Zhengzhou, Henan, China. The samples were stored in an environment of 4 °C before further analysis.

2.2. Physicochemical Properties Analysis

The pH of all samples was accurately measured using a pH meter. The total acid content was measured by the sodium hydroxide titration method and expressed as lactic acid [24]. The previously reported method was used to determine salinity [25]. The formaldehyde value, or the colorimetric method, was used to detect the amino acid nitrogen as described earlier [26]. Reducing sugar was quantified following the methods described by the Chinese National Standard GB 5009.7-2016 [27].

2.3. Amino Acids Composition

An Agilent 1200 high-performance liquid chromatography (HPLC) equipment (Wilmington, DE, USA) linked to a C18 column (AAA, 4.6 × 250 mm, 5 µm) was used to examine the amount of amino acids in this investigation. WSS and commercial soy sauce samples were first acidified and underwent a 24 h acid hydrolysis with 6 M HCl before being derivatized using AccQ Fluor Reagent WAT052880 (Waters Corporation, Milford, MA, USA). A 10 µL solution was then applied to the column after passing through a 0.22 µm filter and eluted at a flow rate of 1.0 mL/min. The mobile phase consisted of solvent A [0.1 mol/L sodium acetate:acetonitrile = 93.0:7.0] and solvent B (water:acetonitrile = 20%:80%), and column temperature was 40 °C. Elution was in accordance with the method of Hou et al. [28], and the mobile phase gradient was as follows: 100–93% A to 0–7% B in 0–11 min, 93–88% A to 7–12% B in 11–13.9 min, 88–66% A to 12–34% B in 13.9–29 min, and 66–30% A to 34–70%

B in 29–32 min. Amino acids in the samples were detected at 254 nm and compared with the standard.

2.4. Headspace Solid-Phase Microextraction (HS-SPME)

The HS-SPME method was used for the extraction of volatile substances, and the fibers were pretreated for 5 min at the gas chromatographic inlet at 250 °C, following the manufacturer's instructions. After, 1 mL of WSS, SS1, and SS2 samples were taken into a 20 mL headspace sample bottle with 10 µL internal standard (2-octanol 50 µg/mL in ethanol) and sealed with a magnetic Teflon/silicone cap. The vials were incubated at 100 °C for 5 min to achieve equilibrium, then the SPME fibers were exposed to the headspace at 100 °C for 15 min for adsorption [29].

2.5. Gas Chromatography–Mass Spectrometry (GC-MS) Analysis

This experiment was conducted following the method described by Jiang et al. [7]. Briefly, helium (99.999% purity) was used as a carrier gas at a flow rate of 1.0 mL/min, and the inlet temperature was set at 250 °C. The split injection mode (split ratio = 5:1) was adopted during volatile insertion. The temperature program was initially set at 40 °C for 1 min and increased to 180 °C at a rate of 4 °C/min. It was finally raised to 260 °C at 7 °C/min and was held for 3 min, with the entire procedure [30]. Initial identification of the flavor substances was carried out by comparing the mass spectral libraries of the National Institute of Standards and Technology (NIST17), and further confirmed by the retention indices (RIs) calculated by comparing the compounds with those reported in the literature. The contents of volatile substances were estimated by the internal standard method.

2.6. E-Nose Analysis

This experiment was conducted as described by Du et al. [31]. The types of compounds corresponding to the different sensors are shown in Table S1. In order to achieve the headspace equilibrium, around 2 mL of each sample was added to a 100 mL glass vial and incubated for 30 min at room temperature. The measurement took 80 s, during which time the sample headspace gas was continuously pumped into the sensor array at a rate of 400 mL/min. For the following study, each sensor's average response value for a span of 69 s was used.

2.7. Statistical Analysis

ANOVA was the statistical method used to examine all the data, which were reported as means ± SD (standard deviation). SPSS 19.0 software (SPSS Inc., Chicago, IL, USA) and Duncan's multiple tests were used to estimate the significant difference established at $p < 0.05$. For volatile chemicals and E-nose analysis, a biplot based on principal component analysis (PCA) was carried out using the SIMCA program (version 14.1, Umea, Sweden).

3. Results and Discussion

3.1. Physicochemical Properties

The results of the physicochemical properties of WSS and commercial soy sauces are presented in Table 1. We noticed that WSS had the lowest pH values compared to those of SS1 and SS2, which was in line with the highest level of total acidity. The SS2 showed the highest total solids content, followed by SS1 and WSS. The amino nitrogen and reducing sugar contents ranged from 2.02 to 3.04 and 0.25 to 1.09 g/100 mL, respectively. This is consistent with the study by Chou and Ling [32], which showed that the contents of total nitrogen, amino nitrogen, free amino acids, and reducing sugars in soy sauce prepared using an extrusion pretreatment of raw soybeans were higher than those using

traditional raw materials. The presence of amino nitrogen in soy sauces can be attributed to the activity of proteases and peptidases within the fermentation system, which break down the protein from the raw material into peptides, amino acids, and small ammonia fragments [33]. The use of amino nitrogen as a reliable agent of the degree of fermentation is well documented [34]. In this work, SS1 showed the highest amino nitrogen amount, followed by SS2 and WSS, which might be related to the lowest salinity in the SS1 samples. Moreover, the highest reducing sugar content was also observed in SS1. The high salt content not only inhibits the growth of microorganisms but also limits the enzymatic activity of protease and amylase during fermentation. Many reports demonstrated that salt content in fermentation systems could affect the characteristics of the flavor and quality of fermented vegetables and soybean products by exerting influence on the microbial structure during fermentation [35,36]. Although a decrease in salt content can modulate protease activity and result in an increase in amino acid nitrogen, Liu et al. [37] found that soy sauce with 20% salt concentration displayed the greatest amino acid nitrogen content when compared to soy sauce with 15.6% and 17.8% salt concentrations. The taste and flavor characteristics of soy sauce are under the influence of free amino acids (FAAs). During moromi fermentation, soybean protein stepwise releases FAAs through the catalysis of extracellular and intracellular proteases and peptidases. In our study, FAA distribution aligned with the features of traditional Chinese soy sauce as reported by previous studies [38,39]. As presented in Table 2, WSS showed the highest contents of Glu and Pro, while SS1 and SS2 had high amounts of bitter amino acids such as Val, Ile, Leu, Tyr, His, Lys, Met, and Phe, which may indicate the intense flavor of WSS. This variation could be due to the difference in the preparation process of soy sauces and the raw materials used in fermentation. The amount of Glu in WSS, SS1, and SS2 was 61.21%, 29.26% and 35.50%, respectively. Indeed, Glu plays a vital role as the main contributor to the umami taste. Glu has two effects in foods: one is to induce a unique taste called umami, which is one of the five basic tastes, and the other is to make food palatable (i.e., flavor-enhancing or seasoning effects). The mechanism by which high Glu content in soy sauce may enhance taste is not clear. Some food scientists believe that the umami taste itself plays an important role [40,41]. On the other hand, the upregulation of N-methyl-D-aspartate (NMDA) and non-NMDA ionotropic glutamate receptors in taste cells may be involved [42–44], but further mechanistic studies are highly needed.

Table 1. The physicochemical characteristics of different soy sauce samples.

	pH	Salinity (%)	Total Acid (g/100 mL)	Amino Acid Nitrogen Content (g/100 mL)	Total Solids (%)	Reducing Sugar Content (g/100 mL)
WSS	4.37 ± 0.01^{a}	9.75 ± 0.19^{b}	2.04 ± 0.04^{a}	2.02 ± 0.03^{a}	24.05 ± 0.48^{c}	2.53 ± 0.21^{c}
SS1	4.72 ± 0.01^{a}	4.68 ± 0.32^{c}	1.85 ± 0.11^{a}	3.04 ± 0.03^{a}	26.13 ± 0.24^{b}	10.92 ± 0.17^{a}
SS2	4.82 ± 0.01^{a}	13.46 ± 0.58^{a}	1.76 ± 0.07^{a}	2.58 ± 0.07^{a}	33.2 ± 0.54^{a}	7.65 ± 0.22^{b}

Note: the characteristic values are represented as mean ± SD obtained across triplicate measurements. $^{a–c}$ indicate the significant difference ($p < 0.05$).

Table 2. Free amino acid compositions in different soy sauce samples.

Free Amino Acid	WSS (mg/mL)	SS1 (mg/mL)	SS2 (mg/mL)
Asp	ND	21.10 ± 0.04^{a}	21.07 ± 1.48^{a}
Glu	74.90 ± 2.78^{a}	38.84 ± 2.17^{c}	53.54 ± 1.69^{b}
Ser	1.41 ± 0.004^{b}	6.97 ± 0.10^{a}	6.65 ± 0.16^{a}
Gly	0.91 ± 0.14^{b}	3.82 ± 0.18^{a}	3.61 ± 0.49^{a}
His	ND	0.87 ± 0.07^{a}	2.54 ± 0.71^{a}

Table 2. *Cont.*

Free Amino Acid	WSS (mg/mL)	SS1 (mg/mL)	SS2 (mg/mL)
Arg	2.22 ± 1.12 [a]	1.80 ± 1.02 [a]	3.01 ± 0.16 [a]
Thr	2.21 ± 0.23 [b]	5.16 ± 1.10 [a]	3.90 ± 0.17 [a]
Ala	7.96 ± 1.49 [a]	7.78 ± 0.41 [a]	7.01 ± 0.34 [a]
Pro	17.26 ± 0.19 [a]	6.58 ± 0.44 [b]	7.38 ± 1.35 [b]
Tyr	2.09 ± 0.76 [a]	2.16 ± 0.33 [a]	0.73 ± 0.01 [b]
Val	3.77 ± 0.36 [b]	7.86 ± 0.77 [a]	8.38 ± 0.57 [a]
Met	2.55 ± 0.34 [b]	2.11 ± 0.40 [b]	4.02 ± 0.01 [a]
Cys	0.98 ± 0.01 [b]	1.65 ± 0.39 [b]	3.73 ± 1.50 [a]
Ile	1.43 ± 0.19 [b]	5.10 ± 0.07 [a]	5.40 ± 0.09 [a]
Leu	2.12 ± 0.68 [c]	9.18 ± 1.35 [a]	8.55 ± 0.08 [a]
Phe	1.60 ± 0.43 [c]	6.66 ± 0.11 [a]	6.20 ± 0.03 [a]
Lys	0.95 ± 0.16 [c]	5.08 ± 0.33 [a]	5.09 ± 0.13 [a]
DAA	83.77 ± 4.27 [a]	71.54 ± 2.69 [a]	85.23 ± 4.22 [a]
SAA	31.97 ± 3.17 [a]	32.11 ± 3.25 [a]	31.56 ± 2.67 [a]
BAA	14.51 ± 2.92 [c]	39.02 ± 3.43 [a]	40.91 ± 1.63 [a]
sAA	74.9 ± 2.78 [a]	59.94 ± 2.21 [b]	74.61 ± 3.17 [a]
AAA	74.9 ± 2.78 [a]	60.81 ± 2.28 [b]	77.15 ± 3.88 [a]
TAA	122.36 ± 8.88 [a]	132.72 ± 9.28 [a]	150.81 ± 8.97 [a]

ND: not detected or below the detection limit; mean ± SD, $n = 3$; [a–c] indicate the significance difference ($p < 0.05$); TAA: total free amino acid content; DAA (Umami amino acid): Glu + Asp + Gly + Ala; SAA (Sweet amino acid): Gly + Ala + Ser + Thr + Pro + Arg; BAA (Bitter amino acid): Lys + Met + Val + Ile + Leu + Tyr + His + Phe; sAA (Salty amino acid): Glu + Asp; AAA (Sour amino acid): Glu + Asp + His.

3.2. Volatile Compounds Analysis

Aromatic volatile flavoring compounds, which are influenced by the microbiota and their metabolic processes, are thought to be the main characteristics in assessing the quality of fermented soybean foods. As shown in Table S2, from the three samples, a total of 188 volatile chemicals were found, comprising 9 aldehydes, 4 sulfur compounds, 9 esters, 7 acids, 47 hydrocarbons, 25 ketones, 37 heterocyclic compounds, 5 phenols, and 9 other compounds. There were clear variations in the quantity and proportion of volatile chemicals throughout the samples, with 173 compounds in WSS, 174 in SS1, and 175 in SS2, which is consistent with the work of Wang and Cha [45]. The flavor components in soy sauce are extremely complex, and the formation of soy sauce flavor has been considered due to the combined effect of microbial metabolism and raw material composition during fermentation, which brought microorganisms together. According to previous research, various aromatic substances such as aldehydes, ketones, alcohols, acids, esters, and sulfur compounds have been identified in soy sauce [46]. Feng et al. [15] have implied that aldehydes, acids, alcohols, and esters are the most typical aromatic chemicals found in soy sauce. Similarly, Wang and Cha [45] found that aldehydes and ketones, aromatic hydrocarbons, alcohols, ethers, and S-containing compounds were the most abundant in soy sauce by reaction flavor technology. In the present study, the most abundant volatile substances in WSS were esters, followed by heterocyclic compounds and ketones. Thus, the raw ingredients and protocol utilized in the soy sauce manufacturing process can all affect the differences in aromatic compounds.

As shown in Figure S1, the high sugar content in watermelon juice, which is consumed by yeast and mold during WSS fermentation, may be the reason for the significantly ($p < 0.05$) higher relative concentrations of alcohols, esters, and acids in WSS samples compared to SS1 and SS2 [47]. In addition, through the deamination or transamination of extracellular amino acids, the Ehrlich route generates α-keto acids, which are essential intermediates and are crucial in the production of alcohol molecules [48].

The main alcohols detected in soy sauces were trans-2-Dodecen-1-ol, 9-Ethylbicyclo (3.3.1)nonan-9-ol, 2,4-Dimethyl-2,4-pentanediol, Phenylethyl alcohol, and 2-Furanmethanol. Among alcoholic compounds, the relative concentrations of phenylethyl alcohol and 2-furanmethanol were obviously higher in WSS. Phenylethyl alcohol is a volatile substance with a relatively strong rose-like odor, which has been widely detected and identified as the key aroma compound in fermented soybean foods, such as fermented soybean paste, douchi, and sufu [49]. Moreover, the compound 2-furanmethanol was reported to exhibit a burnt meat and vitamin-like aroma formed through the Maillard reaction [50]. 2,3-Butanediol, detected in WSS, SS1, and SS2, and also found in various soy sauces by Gao et al. [51], had a close relation to the flavor of soy sauce. It has been proven that 2,3-butanediol itself has an offensive odor, but it has a subtle blending function with other components [52]. Besides the direct aroma activity of alcoholic compounds in soy sauces, the semi-quantitative data regarding alcohols indicated that they may significantly contribute to the flavor profile of soy sauces by enhancing the solubility of other aromatic compounds, thereby assisting in the production of aroma [51].

For the ester compounds, most of the esters detected in our study were ethyl esters, including octadecanoic acid, hexadecenoic acid, tetradecanoic acid, and 2-furancarboxylic acid, indicating their stable contributions to the aroma of WSS. Most esters had a distinct fruity flavor and sweetness [53,54]. According to reports, the majority of ethyl esters are linked to the high ethanol level generated during fermentation, which can give off a sweet, fruity, or floral scent. Esters are primarily formed by the esterification and dehydration of alcohols and acids under the influence of esterification enzymes [55,56]. The present study corroborated a previous report, which showed that the main ester in soy sauce is ethanol esters, including various fatty acid ethyl esters and aromatic acid ethyl esters [51,52].

In this study, it was found that the relative concentration of volatile acidic flavor substances in WSS was significantly greater than that detected in SS1 and SS2, particularly for 4-hexenoic acid, butanoic acid, and 4-methyl-pentanoic acid. Most of those acids are free fatty acids, which are most likely released by lipases during digestion [57], which are described as having a cheesy flavor, such as butanoic acid with a cheesy, sharp, rancid, sweaty, and sour flavor; 4-methyl-pentanoic acid with a cheesy, rancid, and sweaty flavor; and 4-hexenoic acid with a sweaty, fatty, cheesy, sour, rancid, and pungent flavor. These acids were generated by a variety of microorganisms during the fermentation process.

The main sources of aldehydes and ketones in soy sauce are the breakdown of precursor amino acids and the conversion of alcohol. In this study, various aldehydes such as piperonal, 2,4-dimethylbenzaldehyde, methional, and 3-fluoro-4,5-dihydroxybenzaldehyde were detected in all samples. Aldehydes and ketones are also thought to be the primary volatile chemicals that improve the flavor of soy sauce, despite the fact that their relative concentration is typically modest. For instance, 3-(methylthio)-propanal possesses a strong flavor reminiscent of onions and roasted meat [52]. It was identified as the primary aroma compound and a significant contributor to the fragrance in soy sauces [58]. Benzene and acetaldehyde detected in WSS, SS1, and SS2 were found to be positively related to 'musty' and 'soy sauce-like' odors and exhibited the highest levels of aroma intensity [59].

As for the phenolic compounds, nine phenols, including 2-(1,1-Dimethylethyl)-6-(1-methylethyl)-phenol, 2,4-Di-tert-butylphenol, 4-ethyl-2-methoxyphenol, and 4-ethylphenol, were identified in all samples. 4-ethyl-2-methoxyphenol, formed from lignin degradation by *asperillus* during fermentation [52], is known as a potent contributor to the good aroma of soy sauce. 2-methoxyphenol, typically described as smoky, spicy, and medicine-like [58], detected in WSS, SS1, and SS2, is commonly found in soy sauces [51]. These results are consistent with the study of Kan et al. [60].

From the results of volatile compound analysis, it was also found that higher relative concentrations of esters, alcohols, acids, aldehydes, and phenol substances were observed in SS1, which was prepared by high-salt liquid state (HSLS) fermentation, compared to those of SS2, manufactured by low-salt solid-state (LSSS) fermentation. This result is consistent with that of Gao et al. [61], who showed that alcohols, esters, aldehydes, and ketones are the main volatile compound groups of HSLS fermented soy sauce. Moreover, Feng et al. [6] compared the aroma profiles of two varieties of Chinese soy sauce by GC-O/MS and demonstrated that HSLS fermented soy sauce exhibited much higher alcoholic, cooked potato-like, and caramel-like features, while LSSS fermented soy sauce showed strong sour and burnt attributes.

3.3. Electronic Nose Analysis

E-nose primarily uses a combination of gas sensors and pattern recognition technology to mimic a biological system to realize gas detection and recognition functions [62]. Therefore, the E-nose is always used to evaluate the aroma and flavor substances in fermented foods. Figure 2a displayed the usual E-nose responses to several soy sauce samples, with G/Go being utilized as the response value. Results showed that the E-nose has a good capacity for differentiating braised samples via W5S, W1S, W1W, W2W, and W3S sensors. Whereas, W1S sensors gave the strongest responses to aromatic compounds of samples, followed by W1W and W2W sensors, indicating that all three soy sauce samples may have higher abundances of methyl, sulfur, and terpene compounds. Moreover, W5S showed a high response value in WSS, which suggested that the nitrogen oxide compounds might improve flavor formation. From the results, it was also observed that SS1 showed the highest response in the W5S, W1S, W1W, W2W, and W3S sensors, followed by SS2 and WSS. These results were consistent with the above SPME-GC/MS analysis, suggesting higher relative concentrations of alcohols, aldehydes, esters, acids, and phenol compounds in LSSS fermented soy sauce. The lowest response values in WSS might be explained by the low amino nitrogen and total solid content. Using the E-nose sensor response data shown in Figure 2b, a Principal Component Analysis (PCA) was conducted. This analysis can assist in identifying the most important volatile compounds that contribute to the distinct aroma profiles of various soy sauce products [63]. The percentage of variance of the first principal component was 84.44%, the second one was 9.87%, and the overall score of the 2D principal component was 94.31%. Data points from the three sample groups were different from one to another, and the discrepancies in aroma components across the soy sauce samples were mostly on PC1, suggesting that the E-nose fully and unmistakably separated the sample groups.

3.4. Correlation of the Predominant Flavor Compounds and Amino Acids

In general, the characteristic flavor ingredients can be divided into components that are volatile and non-volatile. The principal non-volatile ingredients in foods made from fermented soybeans are taste 5′-nucleotides, tiny molecular peptides, and free amino acids. The savor of fermented soybean food has been considered not only the mere combination of these ingredients but also their synergistic reaction, which improves the flavor as a whole. Furthermore, the breakdown of amino acids is also a direct source of a large number of volatile compounds. In this study, Figure 3 displayed the relationships between flavor components and free amino acids in several soy sauce samples. Ser, Gly, Val, Ile, Leu, Phe, and Lys had a great positive correlation with alcohol and acidic compounds. Erhlich's pathway, which produces aldehydes and their associated alcohols and acids through a transamination process to an α-keto acid and then a decarboxylation phase, was thought to be the most prevalent pathway of amino acid metabolism during fermentation. Moreover,

Strecker degradation was previously assumed to be the source of several aldehydes, including benzaldehyde and benzeneacetaldehyde [64]. It was also observed that Pro was found to correlate significantly positively with aldehyde, ketone, heterocyclic compounds, sulfur compounds, and benzene. Whereas, Glu also showed an apparent positive association with various aromatic compounds, including hydrocarbons, aldehydes, sulfur compounds, and benzene. Similarly, Pei et al. [65] reported that Lys and Glu possess a strong positive correlation with (E)-6-nonenal, (Z)-6-nonen-1-ol, 3-hexenol acetate, (E)-2-nonenal, dimethyl-2-methylpropionate, and ethanol. Zhang et al. [66] proposed a close relationship between aldehydes, pyrazine, and pyrrole and certain amino acids, such as Ala and Phe. Moreover, a high level of umami, bitter, and sweet amino acids may occur as a result of the enzymatic breakdown of proteins during microbial reactions of fermentation, which could result in a bitter taste. Kim et al. [25] stated that all free amino acid concentrations increase during natto fermentation, with Glu, Lys, Tyr, and Phe showing the largest increases. It has also been shown that increased production of dipeptides and free amino acids gives fermented soybeans a meatier and umami taste. The high perception of the umami taste of inosine monophosphate in soy sauce is attributed to the amino acids Ser and Gly. It is well recognized that aspartic acid and Glu contribute to the umami and kokumi flavors.

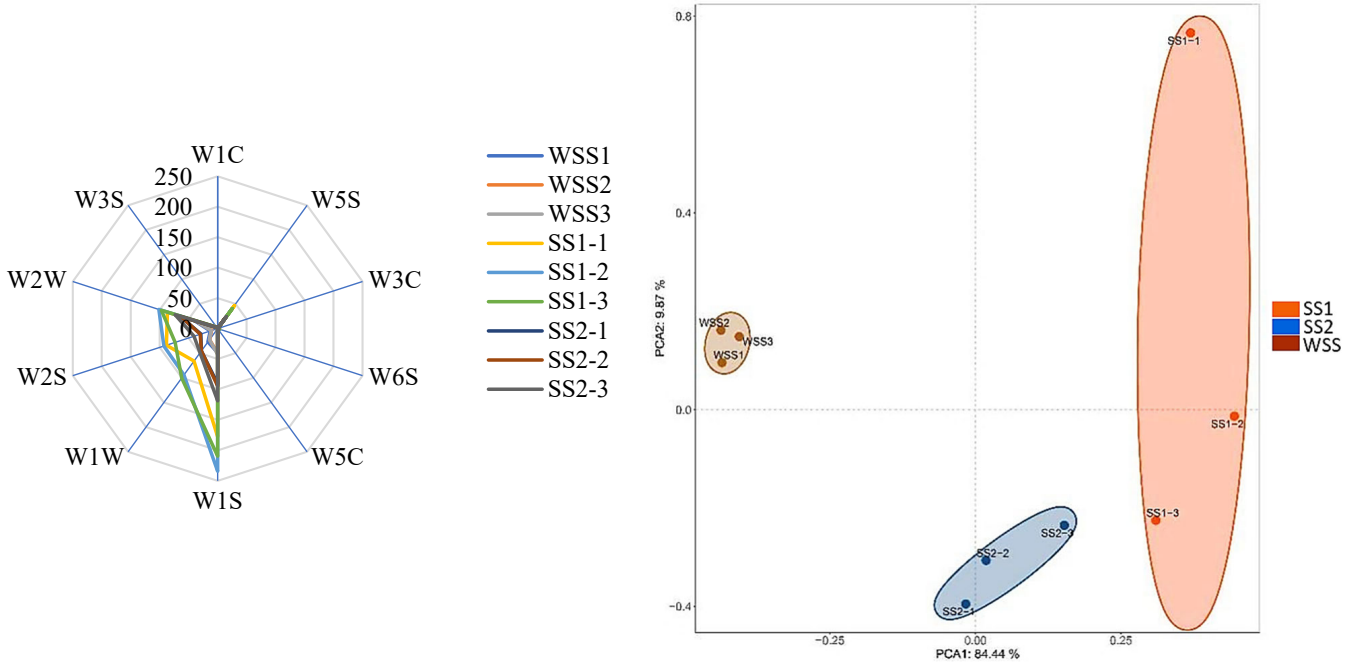

Figure 2. (**a**) Radar chart of sensor responses of different soy sauce samples and (**b**) the results of PCA based on E-nose data. E-nose has good capacity for differentiating braised samples via W5S, W1S, W1W, W2W, and W3S sensors. The percentage of variance of the first principal component was 84.44%, the second one was 9.87%, and the overall score of the 2D principal component was 94.31%.

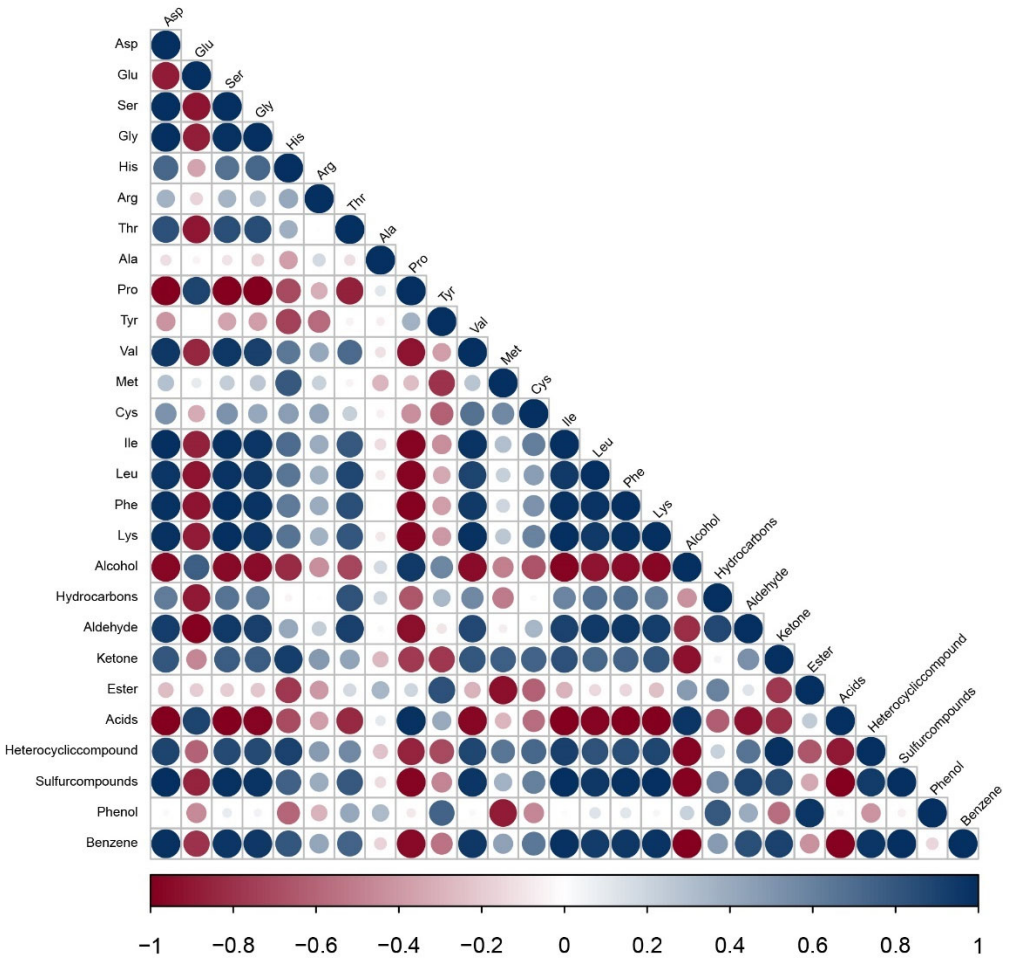

Figure 3. The Pearson's correlation analysis of aroma components and free amino acids. The red color represents negative correlations, and the blue color represents positive correlations. The size of each circle and the color intensity are proportional to the correlation coefficient.

4. Conclusions

There is no previous report on the characterization of physicochemical properties and volatile substances of watermelon soy sauce. WSS exhibited the lowest amino nitrogen and reducing sugar contents, which are probably related to the lowest total solid contents, compared with those of commercial soy sauce samples (SS1 and SS2). When comparing WSS to commercial soy sauce samples, there were clear differences in the quantity and proportion of volatile compounds. For instance, it was observed that the relative levels of alcohols and esters in WSS samples were greater than those in SS1 and SS2. Among alcoholic compounds, the relative levels of phenylethyl alcohol and 2-furanmethanol were obviously higher in WSS. Additionally, the relative concentration of volatile acidic flavor substances in WSS was higher than those identified in SS1 and SS2, particularly for 4-hexenoic acid, butanoic acid, and 4-methyl-pentanoic acid. These compounds were considered to be the main contributors to the aroma activity of WSS. Despite the novelty of this work, we only analyzed three soy sauce samples (WSS, SS1, and SS2), which can be considered the major limitation of the current study. To ensure reliability and generalizability, it would be more appropriate to analyze a large number of soy sauce samples from different manufacturers. Further studies should be performed to investigate the mechanism of formation of aromatic compounds in WSS under the reactions of microorganisms during the natural fermentation process.

Supplementary Materials: The following supporting information can be downloaded at https://www.mdpi.com/article/10.3390/gastronomy3040020/s1, Table S1. Corresponding aroma types of different sensors of E-nose; Table S2. The relative contents (μg/mL) of volatile compounds in different soy sauce samples; Figure S1. Functional groups of volatile compounds identified in different soy sauce samples.

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Abbreviations

The following abbreviations are used in this manuscript:

BAA	Bitter amino acid
DAA	Umami amino acid
GC/MS	Gas chromatography/mass spectrometry
HPLC	High-performance liquid chromatography
HS-SPME	Headspace solid-phase microextraction
HSLS	High-salt liquid-state
LSSS	Low salt solid-state
MOS	Metal oxide semiconductor
ND	Not detected
NIST	National Institute of Standards and Technology
PCA	Principal component analysis
RIs	Retention indices
sAA	Salty amino acid
SAA	Sweet amino acid
TAA	Total free amino acid content
WSS	Watermelon soy sauce

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