

# Volatile Compounds Produced from Monosodium Glutamate in Common Food Cooking

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A mixture of soybean oil and/or water, sugar and/or monosodium glutamate (MSG) was water-boiled or oil-heated at 100, 120, 140, 160, or 170 °C, respectively, and volatile compounds produced were isolated by solid-phase microextraction and then identified by GC and GC/MS. Oxidative thermal degradation products of fatty acids (OTDPFA) were the major volatile compounds detected for all water-boiled samples. When MSG and sugar were heated together, 2,5-dimethyl pyrazine and methyl pyrazine were also detected at or after 160 min heating. Water added in soybean oil increased OTDPFA production. In oil-heated samples of soybean oil alone, soybean oil with MSG, and soybean oil with sugar, OTDPFA were also the major compounds found. The samples containing MSG also produced 2-pyrrolidone, and the samples containing sugar also produced furfural and 5-hydroxy methyl furfural. The samples containing soybean oil, sugar, and MSG produced 23 pyrazines, OTDPFA, and  $\gamma$ -butyrolactone as the major volatile compounds. The contents of OTDPFA were lower in MSG plus sugar and MSG-added samples, and it is postulated that antioxidant activities were produced in cooking.

**Keywords:** *Monosodium glutamate; MSG; oil oxidation; Maillard reaction; antioxidant activities*

## INTRODUCTION

One of the most common natural amino acids, glutamic acid, and its salts have a long history of use in foods. Glutamate is ubiquitous in nature and is present in food and in the human body, either as one of the amino acid building blocks of protein and peptides or in its free form (IFT, 1980; 1987). The most important contribution of added monosodium glutamate (MSG) in food is due to its flavor-enhancing property and its meaty taste. MSG is used commonly in food cooking or in processed foods, and it is not common to consume it directly. Therefore, heating or interaction with other ingredients or foods may change MSG into different compounds. We should pay attention to it, although the safety of MSG was recently confirmed again (Filer and Stegink, 1994; IFT, 1995).

A new variation of adsorption technique called solid-phase microextraction (SPME) was developed about a decade ago by Pawliszym and co-workers (Arthur and Pawliszym, 1990; Arthur et al., 1992). This technique has been studied mainly for analysis of pollutants in environmental water samples. Recently, more and more papers have reported using the SPME method for flavor isolation (Yang and Peppard, 1994; Jia et al., 1998). The SPME–GC method is simple to use, is inexpensive, does not require solvent extraction, and requires less heat treatment.

This paper reports volatile compounds produced from MSG in common food cooking, determined by using the SPME–GC method.

## MATERIALS AND METHODS

**Materials.** Food additives or ingredients were all food grade and were bought from common food markets; SPME holder

(S-7730) and fiber (poly(dimethylsiloxane), 100  $\mu$ m film) were from Supelco, Inc. (U.S.).

**Water Cooking.** Soybean oil (20 g), distilled water (40 g), and/or MSG (4 g) and/or cane sugar (4 g) were put into a 250 mL Erlenmeyer flask. The flask was connected to a condenser (6 °C) and then put into a boiling water bath. A magnetic stir bar was put into the sample flask, so the mixture was well mixed when the flask was heated. The heating time was 80, 160, 240, or 320 min. After heating, the sample flask was removed and cooled, and 120 g of distilled water was added followed by 1 g of tetradecane soybean oil solution (internal standard). Then the flask was stopped with a septum (product 210145–1, Aldrich Co., U.S.). The sample flask was heated in a 60 °C water bath again for 30 min. A SPME fiber was inserted into the headspace of the flask to isolate the volatile compounds. Another set of samples containing soybean oil (20 g), distilled water (0, 10, 20, 30, or 40 g), MSG (4 g), and cane sugar (4 g) was water cooked for 320 min, and the above-described analysis procedure was completed. For flavor isolation by SPME coating of these samples, distilled water was added to make up to a total of 160 g water weight of every sample.

**Oil Cooking.** Soybean oil (20 g) and/or cane sugar (4 g) and/or MSG (4 g) were put into 250 mL Erlenmeyer flasks. A hot plate with six heating sites was used for heating. A 1000 mL beaker containing 300 g of silicone oil as heating medium was heated to 100, 120, 140, 160, and 170 °C, respectively. The sample flasks were connected to condensers (6 °C) and put into the silicone oil bath, and heating was started for different times. Each flask had a magnetic stirrer in it, and the stir rate was 300 rpm. After heating, the sample flask was cooled in air and then in cold water until it reached room temperature. Cold distilled water (160 mL) and 1 g of tetradecane soybean oil solution (internal standard) were added. The determination of volatile compounds isolated by SPME fiber was carried out by the method described for water-cooked samples. Also, volatile compounds produced from the mixture of soybean oil and/or MSG and/or sucrose at 170 °C for 40 min were also evaluated by the same procedure.

**Volatile Separation by Gas Chromatography.** A HP 6890 gas chromatograph with an FID and a nonpolar fused

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**Table 1. Volatile Compounds Produced from a Mixture of Soybean Oil and/or MSG and/or Sucrose and Water after Cooking with Boiling Water**

compounds <sup>e</sup>	RI <sup>a</sup>	O <sup>b</sup>	OW	OSW	OMW	OSMW	OSMW	OSMW	OSMW
		320 min	320 min	320 min	320 min	80 min	160 min	240 min	320 min
hexanal	782	0.014	0.194 <sup>c</sup>	0.158	0.060	0.017	0.041	0.051	0.076
methyl pyrazine	802	<i>d</i>						0.005	0.007
2,5-dimethyl pyrazine	892						0.002	0.015	0.018
( <i>E</i> )-2-heptenal	937	0.012	0.029	0.070	0.031	0.003	0.008	0.029	0.022
1-octen-3-ol	965	0.007	0.035	0.034	0.013	0.002	0.001	0.014	0.021
2-pentyl furan	984	0.008	0.023	0.020	0.035	0.011	0.016	0.034	0.027
( <i>E,E</i> )-2,4-heptadienal	987	0.005	0.021	0.021	0.012	0.003	0.007	0.016	0.015
( <i>E</i> )-2-octenal	1039	0.002	0.050	0.041	0.008	0.003	0.004	0.013	0.007
nonanal	1088	0.003	0.020	0.016	0.009	0.004	0.007	0.012	0.011
( <i>E,E</i> )-2,4-nonadienal	1211								0.003
( <i>E,Z</i> )-2,4-decadienal	1276	0.003	0.038	0.032	0.013	0.003	0.004	0.022	0.008
( <i>E,E</i> )-2,4-decadienal	1298	0.004	0.108	0.083	0.041	0.011	0.010	0.013	0.021
total volatiles		0.06	0.52	0.47	0.22	0.06	0.10	0.22	0.24

<sup>a</sup> Retention indices using paraffin (C<sub>5</sub>–C<sub>25</sub>) as references. <sup>b</sup> Symbols: O indicates 20 g of soybean oil; S indicates 4 g of sugar; M indicates 4 g of MSG; W indicates 40 g of distd water; 320 min indicates heating for 320 min, etc. <sup>c</sup> Unit of volatile compounds is GC peak area relative to internal standard, average of three experiments. <sup>d</sup> Nondetectable. <sup>e</sup> All compounds were positively identified by authentic compounds.

silica capillary column (60 m × 0.25 mm i.d., 1 μm film, DB-1, J&W Scientific) was employed to analyze the volatile compounds desorbed from SPME fiber in the GC injector. The GC equipment was operated with both injector and detector temperature of 250 °C and a nitrogen carrier gas flow rate of 1.0 mL/min. It was in splitless and constant flow mode. The temperature of the GC column was increased from 30 to 200 °C at a rate of 5 °C/min and held at 200 °C for 30 min. Linear retention time indices (RI) for the volatiles were determined by comparing their retention times with those of *n*-paraffin standards, according to the method of Majlat et al. (1974).

**GC/MS Analysis.** The samples were also analyzed by GC/MS, using a HP 6890 gas chromatograph coupled with a HP 5973 mass selective detector equipped with the same column used for the gas chromatography. The operating conditions of the GC were the same as described above. Mass spectra were obtained by using an electron ionization of 70 eV and an ion source temperature of 230 °C.

**Compound Identification and Quantification.** The identification of compounds was made on the basis of their retention indices and mass spectral data. All mass spectra were identified by using an on-line library (Wiley 275) or authentic compounds. Each compound was quantified by calculating its peak area relative to that of the internal standard.

## RESULTS AND DISCUSSION

**Volatile Compounds Produced in Water Cooking.** Table 1 shows the volatile compounds produced during boiling water cooking of the samples. Most volatile compounds detected were OTDPFA such as 2,4-decadienals, 2,4-heptadienals, hexanal, (*E*)-2-heptenal, nonanal, and 1-octen-3-ol. For MSG or sugar only in water–oil solution samples, no special compound derived from MSG or sugar was detectable, although one MSG-added was somewhat pinkish or purple. The one with MSG and sugar added together in the water–oil solution sample showed 2,5-dimethyl pyrazine after heating for 160 min, and after 240 min methyl pyrazine was also detectable. This indicated that a Maillard reaction had occurred and might produce antioxidant activities (Lingnert and Waller, 1983). Thermal autoxidation of fatty acids has been studied for many years in different kinds of samples (Chang et al., 1978; Henderson et al., 1980; Crnjar et al., 1981; Lomanno and Nawar, 1982), but this is the first report on autoxidation of fatty acids in common food cooking, in which water was present at a higher percentage than soybean oil in boiling water cooking. The effect of water

**Table 2. Volatile Compounds Produced from Soybean Oil (20 g) with Different Amounts of Water after 320 min of Cooking with Boiling Water**

compounds <sup>b</sup>	water (g)				
	0	10	20	30	40
hexanal	0.014 <sup>a</sup>	0.040	0.049	0.059	0.194
( <i>E</i> )-2-heptenal	0.012	0.027	0.029	0.030	0.029
1-octen-3-ol	0.007	0.011	0.021	0.013	0.035
2-pentyl furan	0.008	0.010	0.010	0.007	0.022
2,4 heptadienal	0.005	0.011	0.014	0.014	0.021
( <i>E</i> )-2-octenal	0.002	0.006	0.008	0.009	0.050
nonanal	0.003	0.008	0.012	0.011	0.020
( <i>E,Z</i> )-2,4 decadienal	0.003	0.004	0.004	0.006	0.038
( <i>E,E</i> )-2,4-decadienal	0.004	0.010	0.011	0.012	0.108
total volatiles	0.058	0.127	0.158	0.161	0.517

<sup>a</sup> Unit of volatile compounds is GC peak area relative to internal standard, average of three experiments. <sup>b</sup> All compounds were positively identified by authentic compounds.

on the production of aroma compounds or stability in foods or oils has been studied (Rho et al., 1986; Wu and Liou, 1990; Okumura, 1991a; Okumura, 1991b; Krishna and Prabhaker, 1995). Water may be an antioxidant or a prooxidant depending on its concentration or the presence of other ingredients. Table 2 shows volatile compounds produced from soybean oil with different amount of water after 320 min of cooking in boiling water. OTDPFA contents were higher for water-added samples, and the more water added, the higher OTDPFA contents were detected in the tested ranges. Water promoted OTDPFA production in cooking in boiling water significantly. However, retarding autoxidation in raw peanut oil by addition of water was also reported (Krishna and Prabhaker, 1995). Only 0.25–1.00% of water was added to raw peanut oil stored at 27 °C in that study. In this study, water content percentages in soybean oil were much higher, and the samples were heated at 100 °C. At higher temperature heating or with higher ratio of water to oil, 2,4-decadienal production was accelerated. It was postulated that the cleavage of unsaturated fatty acids at the C<sub>9</sub>–C<sub>10</sub> position produced 2,4-decadienals. Alkanal content such as hexanal and nonanal was the highest in all boiling water samples. However, in fried, stir fried, or canned soybean oils, alkanediensals such as 2,4-decadienal was the highest (Wu and Liou, 1990; Wu and Chen, 1992). It was postulated that linoleic acid, the highest fatty acid constituent in soybean oil, was easily cleaved at the

**Table 3. Volatile Compounds Produced from the Mixture of Soybean Oil (20 g) and/or MSG(4 g) and/or Sucrose (4 g) after 40 min of Cooking at 170 °C**

compound	RI <sup>a</sup>	O <sup>b</sup>	OS	OM	OSM
pentane <sup>e</sup>	500	c	0.68 <sup>d</sup>		
hexane <sup>e</sup>	600		0.06		
pentanal <sup>e</sup>	680	0.39	0.19	0.21	
acetoin <sup>e</sup>	687				0.09
heptane <sup>e</sup>	700		0.06		
1-pentanol <sup>e</sup>	753		0.11		
hexanal <sup>e</sup>	782	1.19	1.02	0.37	0.31
octane <sup>e</sup>	800	0.39	0.08	0.04	
methyl pyrazine <sup>e</sup>	802				0.54
furfural <sup>e</sup>	804		0.27		
furfuryl alcohol <sup>e</sup>	830				0.17
(E)-2-hexenal <sup>e</sup>	831	0.22	0.07		
γ-butyrolactone <sup>e</sup>	867				1.61
heptanal <sup>e</sup>	883		0.07	0.03	0.05
2,5-dimethyl pyrazine <sup>e</sup>	892				1.16
ethyl pyrazine <sup>e</sup>	897				0.10
2,3-dimethyl pyrazine <sup>e</sup>	899				0.11
(E)-2-heptenal <sup>e</sup>	937	0.83	0.56	0.23	0.16
I-octen-3-ol <sup>e</sup>	965	0.14	0.10	0.05	
(Z,E)-2,4-heptadienal <sup>e</sup>	975	0.08	0.07	0.03	
2-pentyl furan <sup>e</sup>	984	0.18	0.25	0.13	0.10
(E,E)-2,4-heptadienal <sup>e</sup>	987	0.23	0.42	0.29	
2-ethyl-6-methyl pyrazine <sup>e</sup>	981				0.09
2-ethyl-3-methyl pyrazine <sup>e</sup>	985				0.69
2-ethyl-5-methyl pyrazine <sup>e</sup>	988				0.27
2-ethenyl-6-methyl pyrazine <sup>e</sup>	996				0.04
2-ethenyl-5-methyl pyrazine <sup>e</sup>	1001				0.03
2-pyrrolidone <sup>f</sup>	1017			0.13	0.23
furaneol <sup>e</sup>	1023				0.07
2-acetyl pyrrole <sup>f</sup>	1028				0.06
(E)-2-octenal <sup>e</sup>	1039	0.23	0.21	0.03	0.04
3-ethyl-2,5-dimethyl pyrazine <sup>e</sup>	1063				0.15
3-ethyl-2,6-dimethyl pyrazine <sup>e</sup>	1068				0.07
2-ethyl-3,5-dimethyl pyrazine <sup>e</sup>	1070				0.07
2,5-diethyl pyrazine <sup>e</sup>	1074				0.13
dimethyl-2-vinyl pyrazine <sup>f</sup>	1082				0.05
nonanal <sup>e</sup>	1088	0.26	0.20	0.09	0.08
undecane <sup>e</sup>	1100	0.17			
2-acetyl-3-methyl pyrazine <sup>f</sup>	1097				0.04
2,3-dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one <sup>f</sup>	1107				0.06
2,3-diethyl-5-methyl pyrazine <sup>f</sup>	1122				0.05
2,3,5-trimethyl-6-ethyl pyrazine <sup>f</sup>	1127				0.03
3,5-dimethyl-2-propyl pyrazine <sup>f</sup>	1130				0.04
2,5-diethyl-3-methyl pyrazine <sup>f</sup>	1141				0.05
5-hydroxymethyl furfural <sup>e</sup>	1176		0.52		
2-methyl-5H-6,7-dihydrocyclopentapyrazine <sup>f</sup>	1179				0.12
3-octanone <sup>e</sup>	1212		0.13	0.06	
2,5-dimethyl-3-cis-propenyl pyrazine <sup>f</sup>	1213				0.07
3,5-dimethyl-6,7-dihydro-5H-cyclopentapyrazine <sup>f</sup>	1220				0.05
(E)-2-decenal <sup>e</sup>	1246	0.16	0.12	0.04	0.08
(E,Z)-2,4-decadienal <sup>e</sup>	1276	0.38	0.25	0.18	0.26
(E,E)-2,4-decadienal <sup>e</sup>	1298	1.21	0.81	0.45	0.50
2-undecenal <sup>f</sup>	1346	0.17	0.10	0.06	
(E,E)-2,4-undecadienal <sup>f</sup>	1349		0.09	0.14	
total volatiles		6.21	6.41	2.56	7.82

<sup>a</sup> Retention indices using paraffin (C<sub>5</sub>–C<sub>25</sub>) as references.

<sup>b</sup> Symbols: O indicates 20 g of soybean oil; S indicates 4 g of sugar; M indicates 4 g of MSG. <sup>c</sup> Unit of volatile compounds is GC peak area relative to internal standard, average of three experiments.

<sup>d</sup> Nondetectable. <sup>e</sup> Compounds were positively identified by authentic compounds. <sup>f</sup> Identified by an on-line library (Wiley 275) with the quality (match) higher than 78%.

position of C<sub>12</sub>–C<sub>13</sub> in boiling water to produce hexanal. The other possibility is that the lower content of 2,4-

**Table 4. Volatile Compounds Produced from the Mixture of Soybean Oil (20 g), MSG (4 g), and Sucrose (4 g) after 40 min of Heating at Different Temperatures**

compound	heating temperature (°C)				
	100	120	140	160	170
acetoin	0.05 <sup>a</sup>	0.02	0.18	0.10	0.09
hexanal	0.10	0.09	0.74	0.22	0.31
methyl pyrazine	b		0.16	0.19	0.54
furfuryl alcohol			0.04	0.03	0.17
γ-butyrolactone			0.38	0.54	1.61
heptanal	0.02	0.01	0.04	0.02	0.05
2,5-dimethyl pyrazine			0.35	0.44	1.16
ethyl pyrazine			0.03	0.04	0.10
2,3-dimethyl pyrazine			0.03	0.04	0.11
(E)-2-heptenal	0.02	0.02	0.27	0.14	0.16
2-pentyl furan	0.02	0.03	0.04	0.05	0.10
2-ethyl-6-methyl pyrazine			0.02	0.04	0.09
2-ethyl-3-methyl pyrazine			0.29	0.39	0.69
2-ethyl-5-methyl pyrazine			0.37	0.15	0.27
2-ethenyl-6-methyl pyrazine			0.02	0.03	0.04
2-ethenyl-5-methyl pyrazine			–	0.02	0.03
2-pyrrolidinone			0.06	0.11	0.23
furaneol			0.04	0.04	0.07
2-acetyl pyrrole			0.03	0.02	0.06
(E)-2-octenal		0.01	0.01	0.02	0.04
3-ethyl-2,5-dimethyl pyrazine			0.04	0.09	0.15
3-ethyl-2,6-dimethyl pyrazine			0.03	0.04	0.07
2-ethyl-3,5-dimethyl pyrazine			0.03	0.04	0.07
2,5-diethyl pyrazine			0.04	0.06	0.13
dimethyl-2-vinyl pyrazine			0.02	0.01	0.05
nonanal	0.02	0.03	0.10	0.06	0.08
2-acetyl-3-methyl pyrazine				0.02	0.04
2,3-dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one			0.01	0.02	0.06
2,3-diethyl-5-methyl pyrazine				0.01	0.05
2,3,5-trimethyl-6-ethyl pyrazine			0.01	0.02	0.03
3,5-dimethyl-2-propyl pyrazine			0.02	0.01	0.04
2,5-diethyl-3-methyl pyrazine			0.03	0.03	0.05
(E)-2-nonenal	0.02	0.01			
2-methyl-5H-6,7-dihydrocyclopentapyrazine			0.01	0.05	0.12
2,5-dimethyl-3-cis-propenyl pyrazine			0.03	0.03	0.07
3,5-dimethyl-6,7-dihydro-5H-cyclopentapyrazine			0.02	0.05	0.05
(E)-2-decenal	0.04	0.03	0.06	0.04	0.08
(Z,E)-2,4-decadienal	0.02	0.02	0.12	0.10	0.26
(E,E)-2,4-decadienal	0.04	0.04	0.35	0.30	0.50
total volatiles	0.35	0.28	4.00	3.57	7.82

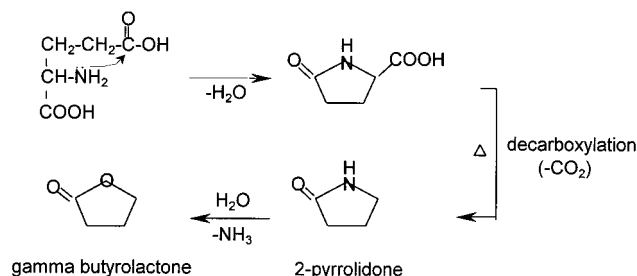
<sup>a</sup> Unit of volatile compounds is GC peak area relative to internal standard, average of three experiments. <sup>b</sup> Nondetectable.

decadienals detected in the water cooking samples was due to lower stability of that compound in water/oil solution (Josephson and Lindsay, 1987).

#### Volatile Compounds Produced in Cooking Oil.

Table 3 shows the volatile compounds produced from the mixture of soybean oil and/or MSG and/or sucrose after 40 min of heating at 170 °C. Table 4 shows volatile compounds produced from the mixture of soybean oil, MSG and sucrose after 40 min of heating at 100, 120, 140, 160, or 170 °C. A total of 58 volatile compounds were identified. Volatile compounds produced were classified as the following groups: (1) Maillard reaction products such as pyrazines, (2) OTDPFA such as (E,E)-2,4-decadienal, (3) sugar degradation or converted products such as furfural, and (4) MSG degradation or converted products such as 2-pyrrolidone and γ-butyrolactone.

Formation of pyrazine compounds in sugar/amino acid systems and the effect of time, temperature, and reactant ratio on them have been reported over the past three decades (Koehler et al., 1969; Shibamoto and



**Figure 1.** Proposed mechanism for the formation of  $\gamma$ -butyrolactone and 2-pyrrolidone from monosodium glutamate.

Bernhard, 1976; Benzing-Purdie et al., 1985). This paper reports pyrazine formation in common food cooking. Twenty-three pyrazine compounds were found in oil cooking samples. The sample heated at 140 °C for 40 min already showed all the compound which were found in the 160 and 170 °C heated samples, except the contents were lower (Table 4). Alkyl pyrazines contribute roast, nutty, or meaty flavors. It is common to regard MSG as a meaty taste contributor and sugar as a sweetener. This paper indicates MSG and sugar can produce odor compounds in foods when they are cooked together.

Important OTDPFA compounds found were hexanal, 2,4-decadienals, 2-heptenal, 2,4-heptadienal, 1-octen-3-ol, 2-octenal, and nonanal. Hexanal has a powerful green, grassy, fatty note, and it may be an off-flavor initiator. 1-Octen-3-ol is herbaceous, earthy like mushroom; 2,4-decadienal is powerful fatty, citrus and is often used in chicken flavor compounding. Therefore, OTDPFA may contribute aroma but also has the possibility to cause off flavor. The amount of OTDPFA was significantly reduced in the samples of MSG plus sugar and only MSG-added oil. It is postulated that antioxidant activities were produced. Lactones formed from heat-degraded pork fat was reported (Watanabe and Sato, 1970). However, except for  $\gamma$ -butyrolactone, no other lactone was found in this study.

2-Pyrrolidone was found in the sample of MSG in oil and MSG plus sugar in oil. As MSG was the only source of nitrogen, it is postulated that this nitrogen-containing compound is from MSG.  $\gamma$ -Butyrolactone was one of the major components found in the MSG with sugar and soybean oil sample by oil heating. The mechanism of formation of 2-pyrrolidone and  $\gamma$ -butyrolactone is proposed in Figure 1.

Furaneol (4-hydroxy-2,5-dimethyl-3(2H)-furanone) was found in the oil-heated MSG plus sugar and soybean oil sample. This compound was also found in corn products (Buttery and Ling, 1997). Schieberle (1992) concluded that "in heated-processed foods containing hexoses furaneol will always be formed". Formation of furaneol through Maillard reaction based on pentose sugars has also been reported (Blank and Fay, 1996). We only detected furaneol in the Maillard reaction product and not in the sugar and oil sample. We also tentatively identified 2,3-dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one in the same sample.

In conclusion, MSG, sugar, and soybean oil are common food ingredients. When they were mixed and cooked by water boiling or oil cooking, volatile compounds were produced. Most of them were common flavor compounds and therefore contributed to the aroma in the food products. Water added in soybean oil promoted oil autoxidation. Maillard reaction products were produced significantly with decreased OTDPFA

contents in oil-heated samples. Some parts of these food ingredients were changed after heating, but no safety-suspected volatile compounds were detected.

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