

Volatile Compounds of Blanched, Fried Blanched, and Baked Blanched Garlic Slices

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After being blanched in boiling water, intact peeled garlic cloves were sliced and then deep-oil-fried or oven-baked to study the contribution of nonvolatile flavor precursors of garlic to thermal flavor generation. Sixteen volatile compounds were detected from blanched garlic; 60 volatile compounds were identified from fried blanched and/or baked blanched garlic. The volatile compounds identified can be divided into four groups: those generated from thermal degradation of nonvolatile flavor precursors of garlic, those generated from thermal interactions of sugars and nonvolatile flavor precursors of garlic, those generated from thermal interactions of lipids and nonvolatile flavor precursors of garlic, and those generated from thermal interactions of sugars, lipids, and nonvolatile flavor precursors of garlic.

INTRODUCTION

Garlic (*Allium sativum* Linn.) has been extensively used as an important vegetable, spice, and seasoning. Several garlic products, such as garlic oil, garlic powder, garlic salt, garlic paste, garlic sauce, garlic flakes, fried garlic slices, and baked garlic slices, are commercially available. These garlic products can be divided into the following forms according to their preparation methods: raw garlic, dried garlic, boiled garlic, baked garlic, and fried garlic. The difference in preparation methods can result in different flavor qualities of these garlic products.

It has been reported that intact garlic cloves contain a group of flavor precursors, i.e., alliin (*S*-allylcysteine sulfoxide), *S*-(*E*)-1-propenylcysteine sulfoxide, and *S*-methylcysteine sulfoxide. It is also well-known that the enzyme, alliinase, which is activated when the cellular tissue of garlic is disrupted, converts these alk(en)ylcysteine sulfoxides into alk(en)yl thiosulfinates, the pungent principles of raw garlic (Stoll and Seebach, 1951; Sreenivasamurthy et al., 1961; Thompson et al., 1964; Raghavan et al., 1983; Fenwick and Hanley, 1985; Block, 1985, 1992; Ziegler and Sticher, 1985; Blania and Spangenberg, 1991; Block et al., 1992a,b; Lawson et al., 1991a; Lawson, 1993). After alliinase was deactivated by boiling the garlic bulb or homogenizing the garlic bulb with alcohol containing limited quantities of water, the flavor precursors were not converted to thiosulfinates and no pungent odor was detected from the garlic samples (Stoll and Seebach, 1951; Ueda et al., 1990).

Besides alk(en)ylcysteine sulfoxides, another group of nonvolatile flavor precursors was found in garlic. They were γ -glutamylalk(en)ylcysteines; three γ -glutamylalk(en)ylcysteines also were found in garlic. They were γ -glutamylallyl-, γ -glutamyl-(*E*)-1-propenyl-, and γ -glutamylmethylcysteines (Thompson et al., 1964; Ziegler and Sticher, 1985; Ueda et al., 1990; Iberl et al., 1990; Lawson et al., 1991a; Lawson, 1993; Block, 1992; Block et al., 1992a). It was found that during storage of garlic cloves these γ -glutamylalk(en)ylcysteines could be converted to alk(en)ylcysteine sulfoxides (Lawson et al., 1991; Lawson, 1993). After the garlic cloves were disrupted, these alk-

(en)ylcysteine sulfoxides could be converted enzymatically to the related alk(en)yl thiosulfinates, the primary flavor compounds of raw garlic. After being heated in boiling water, γ -glutamylallylcysteine was converted to allylcysteine (deoxyalliin), γ -glutamyl-(*E*)-1-propenylcysteine was converted to (*E*)-1- and (*Z*)-1-propenylcysteines, and alliin was found to be completely lost in 8 h to unknown compounds (Fenwick and Hanley, 1985; Ueda et al., 1991; Lawson, 1991a-c; Block, 1992; Block et al., 1992a,b, 1993).

In our study of fried garlic flavor, we (Yu et al., 1993) found that both alkenylcysteine sulfoxides and γ -glutamylalkenylcysteines could be important contributors of the flavor of fried garlic. In model systems, we (Yu et al., 1994a-c) reported that the synthesized alliin and deoxyalliin could self-degrade or interact with 2,4-decadienal at 180 °C and generate volatile flavor compounds in an aqueous solution. We (Yu et al., 1994d,e) also found that the synthesized alliin and deoxyalliin could react with glucose or inosine 5'-monophosphate and generate Maillard-type flavor compounds which contributed to the roasted meaty or fried garlic flavor of the model reaction systems in an aqueous solution.

In the present study, blanched garlic was used to study the high-temperature deep-oil-frying and baking conditions to investigate the role of nonvolatile flavor precursors of garlic on thermal flavor generation.

EXPERIMENTAL PROCEDURES

Materials. Garlic cloves were purchased in a local market in the United States. The vegetable oil used was soybean oil produced in the United States.

Sample Preparation. (A) *Blanched Garlic.* A total of 400 g of peeled garlic cloves was heated in 2 L of boiling distilled water for 20 min. After being blanched, these garlic cloves were taken out and air-cooled.

(B) *Fried Blanched Garlic Slices.* A total of 400 g of peeled garlic cloves was heated in 2 L of boiling distilled water for 20 min. After being taken out and air-cooled, these garlic cloves were cut into pieces (around 2-mm thickness) and then put into 400 mL of hot (180 °C) soybean oil and heated in a pot on an electric stove. Immediately after the garlic slices were added, the temperature of the oil dropped to around 105 °C. After the temperature of the oil again reached 180 °C (22 min), the mixture of garlic slices and oil was immediately cooled to room temperature using an ice-water bath. The total mixture of oil and fried garlic slices was used for flavor isolation.

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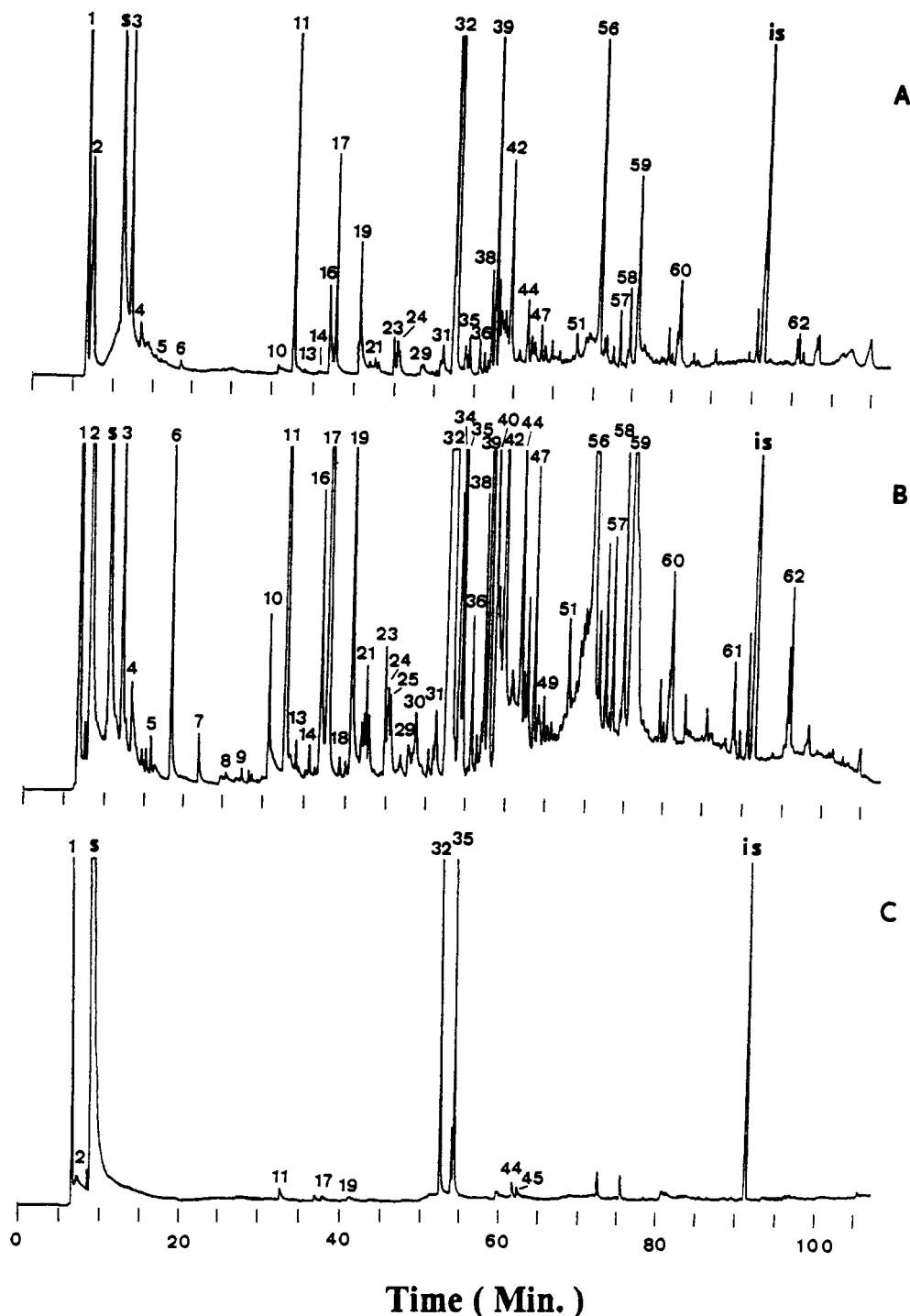


Figure 1. Gas chromatograms of volatile compounds isolated from (A) baked blanched garlic, (B) fried blanched garlic, and (C) blanched garlic.

(C) *Baked Blanched Garlic Slices.* A total of 400 g of peeled garlic cloves was heated and cut into pieces as described in (B). The slices were spread on steel plates and then put into an electric oven preheated to 180 °C. When the baked blanched garlic slices turned slightly dark brown (after approximately 25 min), they were cooled to room temperature and then used for flavor isolation.

Flavor Isolation. (A) *Blanched Garlic Flavor Isolate.* All blanched garlic cloves prepared were blended with 3 L of distilled water in a stainless blender for 3 min. The homogenate was then extracted twice by stirring with 400 mL of redistilled diethyl ether containing 4 mL of hexadecane stock solution (0.062 g of hexadecane in 100 mL of ether) in a flask for 4 h. After being separated, combined, and dried over anhydrous sodium sulfate and filtered, the ether layer was concentrated to about 5 mL using a Kurdena-Danish apparatus fitted with a Vigreux distil-

lation column and then slowly concentrated further under a stream of nitrogen in a small sample vial to a final volume of 0.2 mL.

(B) *Fried Blanched Garlic Flavor Isolate.* All fried blanched garlic slices and oil prepared were mixed with 2 L of distilled water and distilled and extracted into 60 mL of redistilled diethyl ether in a modified Likens-Nickerson apparatus for 3 h. After distillation/solvent extraction, 4 mL of hexadecane stock solution (0.062 g of hexadecane in 100 mL of ether) was added to the isolate as the internal standard. After being dried over anhydrous sodium sulfate and filtered, the distillate was concentrated to about 5 mL using a Kurdena-Danish apparatus fitted with a Vigreux distillation column and then slowly concentrated further under a stream of nitrogen in a small sample vial to a final volume of 0.2 mL.

(C) *Baked Blanched Garlic Flavor Isolate.* All baked blanched

garlic slices were extracted twice by 400 mL of redistilled diethyl ether containing 4 mL of hexadecane stock solution (0.062 g of hexadecane in 100 mL of ether) in a flask for 4 h. After being separated, combined, and dried over anhydrous sodium sulfate and filtered, the ether layer was concentrated to about 5 mL using a Kurdena-Danish apparatus fitted with a Vigreux distillation column and then slowly concentrated further under a stream of nitrogen in a small sample vial to a final volume of 0.2 mL.

Gas Chromatographic (GC) Analysis. A Varian 3400 gas chromatograph equipped with a fused silica capillary column (60 m \times 0.25 mm i.d.; 1- μ m thickness, DB-1, J&W Inc.) and a flame ionization detector was used to analyze and quantify the volatile compounds. The operating conditions were as follows: injector temperature, 270 °C; detector temperature, 300 °C; helium carrier flow rate, 1 mL/min; temperature program, 40 °C (5 min), 2 °C/min, 260 °C (60 min). A split ratio of 50:1 was used. The quantification was based on the ratio of peak areas of flavor components to that of internal standard. The FID response factors of all flavor components and internal standard were assumed to be 1.

Gas Chromatography–Mass Spectrometry (GC–MS) Analysis. The concentrated isolate was analyzed by GC–MS using a Hewlett-Packard GC-5890 coupled to a Hewlett-Packard 5971 MSD equipped with a direct split interface and the same column used for the gas chromatography. The operating conditions were the same as described above. Mass spectra were obtained by electron ionization at 70 eV and an ion source temperature of 200 °C. The temperature of the transfer line was 200 °C.

Identification of the Volatile Compounds. Identification of the volatile compounds in the isolate was mainly based on GC–MS and information from the GC retention index using C₆–C₂₆ paraffins as standards (Majlat et al., 1974). The structural assignment of volatile compounds was accomplished by comparing the mass spectral data with those of authentic compounds available from the Browser–Wiley computer library, the NBS computer library, or previously published literature (Yu et al., 1989, 1993, 1994a–c).

RESULTS AND DISCUSSION

Flavor Description. The blanched garlic prepared in this study possessed a cooked popcorn flavor with sweet and slightly pungent garlic character. The fried blanched garlic slices possessed a typical fried garlic flavor but lacked the pungency of raw garlic or the sulfury note of air-dried garlic that appeared in the fried unblanched garlic slices. The baked blanched garlic slices possessed a typical baked garlic flavor but also lacked the sulfury air-dried garlic flavor that appeared in the baked unblanched garlic.

Volatile Compounds Identified. The gas chromatograms of volatile compounds isolated from garlic samples are shown in Figure 1. The identification and quantification of the volatile compounds generated from garlic samples are shown in Table 1. Comparison of the yields of volatile compounds generated in the garlic samples prepared in this study is shown in Table 2. As shown in Tables 1 and 2, the volatile compounds identified in the garlic samples can be grouped into those generated from thermal degradation of nonvolatile flavor precursors of garlic, those generated from thermal interactions of sugars and nonvolatile flavor precursors of garlic, those generated from thermal interactions of lipids and nonvolatile flavor precursors of garlic, and those generated from thermal interactions of sugars, lipids, and nonvolatile flavor precursors of garlic. The total yields of the volatile compounds detected in blanched garlic, baked blanched garlic, and fried blanched garlic were 8.07, 39.90, and 147.72 ppm, individually. The results showed that the 20-min boiling treatment of garlic could efficiently deactivate the flavor enzyme. Although the volatile compounds detected in the blanched garlic sample would probably be generated

from the transformation of the nonvolatile flavor precursors of garlic through the action of the residual undeactivated flavor enzyme into the primary flavor compounds (the thiosulfinates), and then through the thermal degradation of these primary flavor compounds, they also could be some of the thermal degradation products of the nonvolatile flavor precursors themselves during the boiling treatment of garlic cloves. The difference in the yields of the garlic samples also showed that nonvolatile flavor precursors of garlic, indeed, could contribute to the thermal flavor generation of garlic.

Volatile Compounds Probably Generated from Thermal Degradation of Nonvolatile Flavor Precursors of Garlic. Six nonvolatile flavor precursors, i.e., γ -glutamylallylcysteine, γ -glutamyl-(*E*)-1-propenylcysteine, γ -glutamylmethylcysteine, allylcysteine sulfoxide, (*E*)-1-propenylcysteine sulfoxide, and methylcysteine sulfoxide, have been detected in intact garlic (Stoll and Seebach, 1951; Sreenivasamurthy et al., 1961; Thompson et al., 1964; Raghavan et al., 1983; Fenwick and Hanley, 1985; Block, 1985, 1992; Ziegler and Sticher, 1985; Ueda et al., 1990; Iberl et al., 1990; Blania and Spangenberg, 1991; Block et al., 1992a,b; Lawson et al., 1991a; Lawson, 1993). These flavor precursors were proposed to contribute to the formation of the volatile compounds detected in the garlic samples prepared in this study.

Among the volatile compounds shown in Table 1 and grouped into those probably generated from the thermal degradation of nonvolatile flavor precursors of garlic, 1-propene, acetaldehyde, allyl alcohol, dimethyl disulfide, 2-methylthiophene, allyl sulfide, 2-methylthiazole, methyl allyl disulfide, methyl propyl disulfide, 1,2-dithiacyclopent-3-ene, dimethyl trisulfide, 1,3-dithiane, allyl disulfide, 1,4-dithiacyclohept-5-ene, 3,6-dimethyl-1,4-dithiane, methyl allyl trisulfide, 2-ethyl-1,3-dithiane, methyl-1,2,3-trithiacyclopentane, allyl trisulfide, 1,2,3,4-tetrathiepane, 3,6-dimethyl-1,2,5-trithiepane, 4,6-dimethyl-1,2,5-trithiepane, and 4-ethyl-6-methyl-1,2,3,5-tetrathiane have been detected in the thermal degradation mass of alliin or deoxyalliin or in the thermal interaction mass of alliin or deoxyalliin and glucose or inosine 5'-monophosphate (Yu et al., 1993, 1994a,b,d,e). Although not detected previously in alliin or deoxyalliin model systems, methylthiirane, 2,5-dithiahexane, 1-propenyl allyl disulfide, 3-(allylthio)propionic acid, 3-vinyl-4*H*-1,2-dithiin, 2-vinyl-4*H*-1,2-dithiin, 2-vinyl-1,3-dithiane, and propyl 1-propenyl disulfide could also derive from thermal degradation of nonvolatile flavor precursors of garlic or interactions of the degradation products of these precursors. The cyclic S₈ compound identified in this study also has been found in garlic oil (Jirovetz et al., 1992). This compound was believed to generate from the interaction of hydrogen sulfide molecules which, in turn, were the degradation products of alliin or deoxyalliin, during high-temperature thermal treatment of garlic samples or in the high-temperature GC injector.

Volatile Compounds Probably Generated from Thermal Interactions of Sugars and Nonvolatile Flavor Precursors of Garlic. Among the volatile compounds, the pyrazines shown in Table 1 were grouped into those generated from thermal interactions of sugars and nonvolatile flavor precursors of garlic. Methylpyrazine, 2,5-dimethylpyrazine, 2-ethyl-6-methylpyrazine, 2-ethyl-5-methylpyrazine, trimethylpyrazine, and 2-ethyl-3-methylpyrazine have been detected in the model reaction systems of alliin or deoxyalliin plus glucose or inosine 5'-monophosphate (IMP) (Yu et al., 1994b,d,e). Although not previously detected in alliin or deoxyalliin plus glucose

Table 1. Volatile Compounds Identified in Baked Blanched Garlic (BBG), Fried Blanched Garlic (FBG), and Blanched Garlic (BG)

peak no. ^a	compd identified	M _r ^b	RI ^c	yield (ppm)		
				BBG	FBG	BG
Compounds Probably Generated from Thermal Degradation of Nonvolatile Flavor Precursors of Garlic						
1	1-propene	42	<500	7.12	10.55	2.20
2	acetaldehyde	44	<500	1.85	25.24	0.70
3	allyl alcohol	58	549	1.42	2.26	0.05
4	methylthiirane ^d	74	577	0.18	0.70	0.11
6	methyl allyl sulfide	88	678	0.06	1.31	nd ^e
7	dimethyl disulfide	94	732	0.01	0.13	0.02
8	2-methylthiophene	98	765	0.05	0.05	nd
11	allyl sulfide	114	850	1.16	8.99	0.09
15	2-methylthiazole	99	888	0.01	0.02	nd
17	methyl allyl disulfide	120	902	0.80	7.26	0.04
18	methyl propyl disulfide	122	920	0.01	0.09	nd
19	1,2-dithiacyclopent-3-ene ^d	104	936	0.77	2.15	0.06
21	dimethyl trisulfide	126	957	0.09	0.25	nd
22	2,5-dithiahexane ^d	122	961	0.10	0.24	nd
28	1,3-dithiane ^d	120	1002	0.02	0.19	nd
32	allyl disulfide	146	1076	9.14	50.74	2.40
34	1-propenyl allyl disulfide	146	1087	0.19	0.54	0.30
35	1-propenyl allyl disulfide	146	1090	0.21	0.77	1.59
36	3-(allylthio)propionic acid ^d	146	1104	0.18	0.33	nd
37	1,4-dithiacyclohept-5-ene ^d	132	1113	0.11	0.09	nd
38	3,6-dimethyl-1,4-dithiane ^d	148	1123	0.38	0.57	nd
39	methyl allyl trisulfide	152	1130	1.53	4.34	0.17
40	2-ethyl-1,3-dithiane ^d	148	1140	0.52	1.04	nd
42	3,6-dimethyl-1,4-dithiane ^d	148	1152	1.46	2.58	nd
44	3-vinyl-4H-1,2-dithiin	144	1180	0.49	1.44	0.14
45	methyl-1,2,3-trithiacyclopentane ^d	138	1185	0.26	0.32	0.11
47	2-vinyl-4H-1,3-dithiin	144	1199	0.31	0.72	nd
48	2-vinyl-1,3-dithiane ^d	146	1208	0.27	0.30	nd
49	propyl 1-propenyl disulfide ^d	148	1217	0.29	0.28	nd
52	allyl trisulfide	178	1275	0.52	0.66	0.06
56	1,2,3,4-tetrathiepane ^d	170	1295	2.21	5.98	nd
57	3,6-dimethyl-1,2,5-trithiepane ^d	180	1331	0.33	0.69	nd
58	4,6-dimethyl-1,2,5-trithiepane ^d	180	1350	0.64	1.04	0.03
59	4,6-dimethyl-1,2,5-trithiepane ^d	180	1366	1.45	5.90	nd
61	4-ethyl-6-methyl-1,2,3,5-tetrathiane ^d	198	1557	0.28	0.42	nd
62	cyclic S ₈	256	1671	0.26	0.48	nd
Compounds Probably Generated from Thermal Interactions of Sugars and Nonvolatile Flavor Precursors of Garlic						
9	methylpyrazine	94	792	nd	0.04	nd
16	2,5-dimethylpyrazine	108	893	0.51	0.69	nd
23	2-ethyl-6-methylpyrazine	122	981	0.15	0.26	nd
24	2-ethyl-5-methylpyrazine	122	984	0.11	0.13	nd
26	trimethylpyrazine	122	986	0.10	nd	nd
27	2-ethyl-3-methylpyrazine	122	988	0.09	0.33	nd
33	methylpropylpyrazine ^d	136	1085	0.05	0.38	nd
41	3,5-diethyl-2-methylpyrazine	150	1145	0.47	0.45	nd
46	methylallylpyrazine ^d	134	1190	0.31	0.60	nd
Compounds Probably Generated from Thermal Interactions of Lipids and Nonvolatile Flavor Precursors of Garlic						
5	benzene	78	630	0.02	0.11	nd
10	4-heptenal	112	829	0.11	0.68	nd
12	ethylbenzene	106	858	nd	0.01	nd
13	2,5-dimethylthiophene	112	865	0.04	0.05	nd
14	2-vinylthiophene	110	881	0.03	0.07	nd
20	2-ethylpyridine	107	951	0.12	0.20	nd
25	2-pentylfuran	138	986	nd	0.22	nd
29	methylethylpyridine ^d	121	1013	0.06	0.19	nd
30	phenylacetaldehyde ^d	120	1024	0.10	0.44	nd
31	butylbenzene	134	1054	0.25	0.34	nd
43	pentylbenzene	148	1167	0.36	0.76	nd
50	cyclopentylbenzene ^d	146	1244	0.11	0.19	nd
51	hexylbenzene	162	1257	0.43	0.62	nd
53	(E,E)-2,4-decadienal	152	1279	nd	0.44	nd
55	benzothiophene ^d	134	1290	0.70	0.40	nd
60	BHT	220	1439	1.10	0.85	nd
Compounds Probably Generated from Thermal Interactions of Sugars, Lipids, and Nonvolatile Flavor Precursors of Garlic						
54	2,3-dimethyl-5-pentylpyrazine	178	1283	nd	0.61	nd
	total			39.90	147.72	8.07

^a Peak no. refers to that shown in Figure 1. ^b M_r, molecular weight. ^c RI, linear retention indices. ^d Tentatively identified. ^e nd, not detected.

or IMP model systems, the other pyrazines, i.e., methylpropylpyrazine, 3,5-diethyl-2-methylpyrazine, and methylallylpyrazine, could also be the thermal interaction

products of sugars and nonvolatile flavor precursors of garlic. Although the amino acids in garlic could also contribute to the formation of these pyrazines, the total

Table 2. Comparison of the Yields of Volatile Compounds Generated in Baked Blanched Garlic (BBG), Fried Blanched Garlic (FBG), and Blanched Garlic (BG)

compd	yield (ppm)		
	BBG	FBG	BG
compounds generated from thermal degradation of nonvolatile flavor precursors of garlic	34.68	138.66	8.07
compounds generated from thermal interactions of sugars and nonvolatile flavor precursors of garlic	1.79	2.88	0.00
compounds generated from thermal interactions of lipids and nonvolatile flavor precursors of garlic	3.43	5.57	0.00
compounds generated from thermal interactions of sugars, lipids, and nonvolatile flavor precursors of garlic	0.00	0.61	0.00
total	39.90	147.72	8.07

amount of amino acids in garlic was found to be less than that of nonvolatile flavor precursors of garlic (Ueda et al., 1991; Lawson, 1993). Since most of the pyrazines identified in this study have been identified in alliin or deoxyalliin plus glucose or IMP model systems, the nonvolatile flavor precursors of garlic were considered to make a significant contribution to the formation of pyrazines identified in fried or baked garlic.

Volatile Compounds Probably Generated from Thermal Interactions of Lipids and Nonvolatile Flavor Precursors of Garlic. Although most of the volatile compounds shown in Table 1 were grouped into those probably generated from thermal interactions of lipids and nonvolatile flavor precursors of garlic have not been detected in the model reaction systems of alliin or deoxyalliin, they were proposed to generate from thermal degradation of lipids and/or interactions of lipid degradation products and nonvolatile flavor precursors of garlic. Most of these products have been identified from thermal degradation of lipids and/or interactions of lipids and amino-containing compounds (Whitfield, 1992).

Volatile Compounds Probably Generated from Thermal Interactions of Sugars, Lipids, and Nonvolatile Flavor Precursors of Garlic. The 2,3-dimethyl-5-pentylpyrazine identified was proposed to generate from thermal interactions of sugars, lipids, and nonvolatile flavor precursors of garlic. It also has been identified from the model reaction systems containing lipids, sugars, and amino compounds and has been proposed to generate from the thermal interactions of sugars, lipids, and amino compounds (Whitfield, 1992).

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