Volatile Compounds Identified in Headspace Samples of Peanut Oil Heated under Temperatures Ranging from 50 to 200 °C

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Gas chromatography (GC) and gas chromatography/mass spectrometry (GC/MS) were used to identify 99 volatile compounds in headspace samples of peanut oil heated to 50, 100, 150, or 200 °C for 5 h. There were 42 hydrocarbons, 22 aldehydes, 11 fatty acids, 8 alcohols, 8 ketones, 4 furans, 2 esters, and 2 lactones identified. Identification of formaldehyde, acetone, acetaldehyde, propanal, 2-pentanone, butanal, and 2-hexanone was achieved only after their transformation to corresponding thiazolidine derivatives. The total amount of all identified volatiles increased as the temperature of the oil was increased.

INTRODUCTION

Peanut seed (*Archis hypogaea* L.) is an important oil crop throughout the world and is grown is large quantities in Africa, India, China, and the United States. The oil is manufactured by pressing the shelled and skinned peanut seeds. Peanut oil is also known as arachis oil, groundnut oil, earthnut oil, or katchung oil. It is used in shortenings, margarines, salad oils, and mayonnaise and also for deep-frying foods. Peanut oil contains up to 6% of long-chain saturated fatty acids, including arachidic (20:0), behenic (22:0), and sometimes lignoceric (24:0) (Worthington, 1977). Furthermore, oleic acid (18:1) and linoleic acid (18:2) have been shown to comprise 46.5 and 31.4% of the total fatty acid content in peanut oil (White, 1992).

Cooking and frying oils are used not only for transferring heat to cooking foods but also for enhancing flavor. Since oils undergo a variety of chemical changes during cooking and frying, chemical changes brought about by heat treatment are extremely important to both the consumer and the processor because of their impact on flavor, nutrition, and possibly toxicology. Several recent studies have reported on the volatile compounds produced when various vegetable oils are heated (Wu and Chen, 1992; Macku and Shibamoto, 1991; Snyder et al., 1985), and the flavor chemistry of deep fat frying has been reviewed by Pokorný (1989). Although considerable research on the content of fatty acids in peanut oil has been conducted, the volatile compounds produced by thermal treatment have not received much attention. Gioielli et al. (1992) have investigated the formation of volatile compounds from crude peanut oil heated at relatively low temperatures ranging from 40 to 60 °C.

The isolation of flavor components from fats and oils is particularly difficult because most flavor components are fat soluble and the presence of lipids significantly reduces isolation efficiency. The use of a simultaneous purging and solvent extraction apparatus for headspace sampling of heated butter has recently been employed to overcome these associated problems (Lee et al., 1991). The purpose of this study was to determine the differences in the production of headspace volatile compounds from peanut oil heated under a broad range of temperatures (50–200 °C) simulating mild, deep-frying, and near-pyrolysis cooking conditions. In the present investigation volatile flavor components formed in the headspace of heated peanut oil were isolated and identified by gas chromatography (GC) and gas chromatography/mass spectrometry (GC/MS).

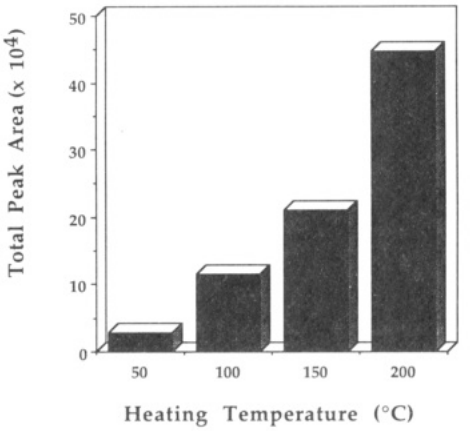

Figure 1. Total peak area of volatile compounds produced from heating peanut oil at four different temperatures. FID total peak area is calculated by subtracting the peak area of the solvent from that of all other components.

EXPERIMENTAL PROCEDURES

Materials. Refined peanut oil was purchased from a local market and was used immediately. The oil contained no added antioxidants (only endogenous tocopherols). After initial use, the oil was stored at 4 °C under nitrogen. Cysteamine hydrochloride was purchased from Aldrich Chemical Co. (Milwaukee, WI). Dichloromethane was purchased from J. T. Baker Chemical Co. (Phillipsburg, NJ). All authentic specimens were obtained from reliable commercial sources. Standards of 2-alkylthiazolidines were previously synthesized according to the method of Yasuhara and Shibamoto (1989). The purity of the standards as determined by gas chromatography varied between 98.5 and 99.9%.

Collection of Headspace Volatiles. Peanut oil (100 g) was placed in a 500-mL two-neck, round-bottom flask. The flask was connected to a simultaneous purging and solvent extraction apparatus designed by Umano and Shibamoto (1987). Peanut oil was heated at 50, 100, 150, or 200 °C while being stirred by a magnetic stirrer. The headspace volatiles were purged into 250 mL of deionized water by a purified nitrogen stream at a flow rate of 10 mL/min. The volatiles trapped by the water were simultaneously and continuously extracted with dichloromethane (50 mL) for 5 h. The water temperature was kept at 10 °C by

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Table I. Volatile Compounds Identified in Headspace Samples of Peanut Oil Heated under Four Different Temperature Conditions

compound	GC peak area,[a] % 50 °C	100 °C	150 °C	200 °C	identification RI[c]	MS	RT	compound	GC peak area,[a] % 50 °C	100 °C	150 °C	200 °C	identification RI[c]	MS	RT
heptane	0.59	2.75	2.46	0.49	700	+	+	2-nonanone	*b*	*b*	*b*	0.14	1405	+	+
cyclohexane	79.46	49.68	35.55	5.78	712	+	+	nonanal	0.15	1.02	1.11	2.01	1411	+	+
1-heptene	1.12	0.31	0.46	2.29	750	+	+	(*Z*)-3-octenal	*b*	*b*	0.19	0.10	1421	+	+
(*E*)-2-heptene	0.10	0.27	6.23	0.17	778	+	+	*n*-pentylbenzene	*b*	0.23	*b*	0.10	1426	+	+
octane	0.33	0.42	0.12	7.39	800	+	+	(*E*)-2-octenal	0.25	1.51	2.16	1.49	1453	+	+
1-octene	*b*	0.31	0.80	2.05	822	+	+	1-tetradecene[d]	*b*	*b*	*b*	*b*	1454	+	
(*E*)-4-octene	*b*	0.45	0.66	1.08	841	+	+	2-heptylfuran[d]	*b*	*b*	*b*	*b*	1454	+	
(*E*)-2-octene	0.25	0.25	0.36	0.19	854	+	+	1-octen-3-ol	0.16	0.89	1.15	4.30	1462	+	+
(*Z*)-2-octene	*b*	0.35	0.36	*b*	866	+	+	1-heptanol	*b*	0.19	0.25	0.18	1467	+	+
nonane	*b*	*b*	*b*	*b*	900	+	+	1,12-tridecadiene[d]	*b*	*b*	*b*	*b*	1494	+	
1-nonene	*b*	0.08	*b*	0.60	923	+	+	2-decanone[d]	*b*	*b*	*b*	*b*	1503	+	
cyclooctene	0.17	0.16	*b*	0.21	955	+	+	(*E*,*E*)-2,4-heptadienal	*b*	*b*	*b*	0.06	1503	+	+
(*E*)-4-nonene	*b*	*b*	0.27	0.14	973	+	+	decanal	*b*	*b*	*b*	0.06	1512	+	+
pentanal	2.01	3.76	3.58	2.01	1003	+	+	(*Z*)-3-nonenal	*b*	0.14	0.20	0.12	1522	+	+
3-nonyne	*b*	*b*	*b*	0.14	1009	+	+	benzaldehyde	*b*	*b*	*b*	0.21	1558	+	+
2-propylfuran	*b*	*b*	*b*	0.13	1043	+	+	1-pentadecene	*b*	0.26	0.31	0.12	1558	+	+
bicyclo[5.1.0]octane[d]	*b*	*b*	*b*	*b*	1045	+		(*E*)-2-nonenal	*b*	0.27	0.40	1.54	1562	+	+
toluene	*b*	*b*	*b*	0.05	1047	+	+	1-octanol	*b*	*b*	*b*	0.24	1575	+	+
1-decene	*b*	0.06	*b*	0.09	1054	+	+	2,4-pentadecadiene	*b*	*b*	*b*	0.63	1583	+	+
cyclononene	*b*	0.05	*b*	1.02	1059	+	+	(*Z*)-3-decenal	*b*	*b*	*b*	0.10	1641	+	+
hexanal	3.46	13.52	18.26	18.05	1107	+	+	1-hexadecene	*b*	*b*	*b*	0.22	1655	+	+
2-butylfuran	*b*	*b*	*b*	0.17	1112	+	+	(*E*)-2-decenal	2.39	*b*	*b*	0.30	1660	+	+
1-pentylcyclopentene[d]	*b*	*b*	*b*	*b*	1114	+		1-nonanol	0.11	0.48	0.57	1.51	1674	+	+
4,6-decadiene	*b*	*b*	*b*	0.07	1117	+	+	allyl levulinate	*b*	*b*	0.13	0.33	1694	+	+
ethylbenzene	*b*	*b*	*b*	0.06	1135	+	+	isopulegone	*b*	*b*	*b*	0.13	1720	+	+
camphane	*b*	*b*	*b*	0.41	1140	+	+	(*E*,*E*)-2,4-nonadienal	*b*	0.06	*b*	0.24	1738	+	+
(*E*)-5-undecene	*b*	*b*	*b*	0.08	1146	+	+	1-heptadecene[d]	*b*	*b*	*b*	*b*	1750	+	
(*E*)-3-undecene	*b*	0.20	0.13	0.13	1155	+	+	1-decanol	*b*	*b*	*b*	0.32	1783	+	+
1-undecene	*b*	*b*	*b*	0.13	1160	+	+	(*E*)-2-undecenal	*b*	0.06	0.18	0.94	1785	+	+
1-penten-3-ol	*b*	0.09	*b*	0.12	1169	+	+	(*Z*,*E*)-2,4-decadienal	*b*	0.29	0.62	0.56	1803	+	+
(*E*)-2-undecene	*b*	*b*	*b*	0.07	1173	+	+	(*E*,*E*)-2,4-decadienal	0.30	0.60	1.53	5.30	1852	+	+
2-heptanone	*b*	*b*	*b*	0.71	1180	+	+	caproic acid	*b*	*b*	0.36	0.32	1866	+	+
heptanal	0.29	1.33	2.43	4.86	1183	+	+	(*E*)-2-dodecenal	*b*	*b*	*b*	0.35	1887	+	+
limonene	*b*	*b*	0.17	0.63	1190	+	+	γ-octalactone[d]	*b*	*b*	*b*	*b*	1910	+	
n-propylbenzene	*b*	*b*	*b*	0.45	1194	+	+	enanthic acid	*b*	*b*	*b*	0.18	1975	+	+
dodecane[d]	*b*	*b*	*b*	*b*	1200	+		eicosane	*b*	*b*	*b*	*b*	2000	+	+
1-hexylcyclopentene	*b*	*b*	*b*	0.58	1203	+	+	2-pentadecanone	*b*	*b*	*b*	*b*	2056	+	+
(*E*)-2-hexenal	0.24	0.99	0.97	0.34	1210	+	+	γ-nonalactone[d]	*b*	*b*	*b*	*b*	2063	+	
2-pentylfuran	*b*	1.94	3.97	2.60	1224	+	+	caprylic acid	*b*	*b*	*b*	0.14	2085	+	+
1-pentanol	0.45	1.57	1.71	0.80	1249	+	+	heneicosane	*b*	*b*	*b*	0.16	2100	+	+
cyclohexanone	*b*	*b*	*b*	0.08	1273	+	+	pelargonic acid	*b*	*b*	*b*	*b*	2194	+	+
2-octanone	*b*	*b*	*b*	0.25	1278	+	+	methyl palmitate[d]	*b*	*b*	*b*	*b*	2254	+	
octanal	0.10	0.55	0.88	0.70	1282	+	+	tricosane[d]	*b*	*b*	*b*	*b*	2300	+	
(*Z*)-3-heptenal[d]	*b*	*b*	*b*	*b*	1312	+		caproic acid	*b*	*b*	*b*	*b*	2304	+	+
n-butylbenzene	*b*	*b*	0.37	2.23	1323	+	+	lauric acid	*b*	*b*	*b*	*b*	2524	+	+
(*E*)-2-heptenal	1.03	5.84	7.25	6.97	1350	+	+	myristic acid	*b*	*b*	*b*	*b*	2744	+	+
1-tridecene	*b*	*b*	0.49	0.36	1357	+	+	palmitic acid	0.56	*b*	*b*	0.94	2963	+	+
6-methyl-5-hepten-2-one	*b*	*b*	*b*	0.08	1363	+	+	stearic acid	*b*	*b*	*b*	*b*	3239	+	+
1-hexanol	*b*	0.16	0.21	0.15	1380	+	+	oleic acid	0.62	*b*	*b*	1.49	3241	+	+
linoleic acid	*b*	*b*	*b*	0.63	3314	+	+								
								known compounds	94.14	91.09	97.35	90.37			
								unknown compounds	5.86	8.91	2.65	9.63			

[a] Calculated from GC peak area after solvent peak area is subtracted from total area. [b] Peak area percent less than 0.02. [c] Kovats index values calculated on a DB-Wax capillary column. [d] Tentatively identified (on the basis of MS data alone).

a Brinkman RM6 constant-temperature water circulator. The dichloromethane extract was dried over anhydrous sodium sulfate overnight, and then the extract was concentrated to 0.5 mL by fractional distillation.

Analysis of Carbonyl Compounds. Carbonyl compounds in headspace samples of peanut oil heated under four different temperatures were trapped by a method previously developed by Yasuhara and Shibamoto (1991). The deionized water used in the collection apparatus described above was replaced with an aqueous cysteamine solution (1.5 g of cysteamine in 250 mL of deionized water). The pH of the solution was adjusted to 7.0 with 2 N NaOH. Carbonyl compounds formed in headspace samples of heated peanut oil were purged into the cysteamine solution to form thiazolidine derivatives, which were then continuously extracted with dichloromethane or chloroform (50 mL). These extracts were dried over anhydrous sodium sulfate overnight and then brought to a final volume of 20 mL by fractional distillation. The samples were analyzed by GC and GC/MS.

Instrumental Analysis. A Hewlett-Packard (HP) Model 5890 gas chromatograph equipped with a 60 m × 0.25 mm i.d. DB-Wax fused silica capillary column (J&W Scientific, Folsom, CA) and a flame ionization detector (FID) was used for routine analysis. Thiazolidine derivative analysis was performed on a 30 m × 0.25 mm i.d. DB-Wax fused silica capillary column with nitrogen–phosphorus (NPD) and flame photometric (FPD) detection. The oven temperature was held at 60 °C for 4 min and then programmed to 220 °C at 3 °C/min. The same temperature program was also utilized for thiazolidine derivative analysis. The GC peak areas were integrated with a Spectra Physics 4290 integrator. The injector temperature was 250 °C. The detector temperatures were 275, 250, and 230 °C for the

Table II. Thiazolidine Derivatives Identified in the Cysteamine Trap of Headspace Samples of Peanut Oil Heated under Four Different Temperature Conditions

peak[a]	original carbonyl	derivative	GC peak area,[a] % 50 °C	100 °C	150 °C	200 °C
1	unknown		1.59	0.51	0.25	0.12
2	unknown		2.63	1.03	0.85	0.44
3	acetone	2,2-dimethylthiazolidine	b	0.25	0.35	0.13
4	acetaldehyde	2-methylthiazolidine	15.91	13.55	11.33	10.74
5	formaldehyde	thiazolidine	58.60	15.91	12.26	4.64
6	propanal	2-ethylthiazolidine	1.29	3.49	2.53	2.56
7	2-pentanone	2-methyl-2-propylthiazolidine	b	b	0.12	0.07
8	butanal	2-propylthiazolidine	0.91	2.44	1.88	1.86
9	2-hexanone	2-methyl-2-butylthiazolidine	b	b	0.17	b
10	pentanal	2-butylthiazolidine	2.98	14.10	11.31	10.60
11	unknown		b	b	0.07	0.06
12	3-heptanone	2-ethyl-2-butylthiazolidine	b	b	0.14	0.48
13	2-heptanone	2-methyl-2-pentylthiazolidine	b	b	0.90	0.13
14	hexanal	2-pentylthiazolidine	4.73	39.46	45.63	55.23
15	unknown		b	b	0.09	0.07
16	unknown		b	b	0.47	0.09
17	unknown		b	b	0.26	0.07
18	heptanal	2-hexylthiazolidine	b	1.77	3.05	7.12
19	unknown		b	b	0.61	0.48
20	unknown		b	b	0.24	0.18
21	unknown		b	b	0.47	0.21
22	octanal	2-heptylthiazolidine	b	0.93	1.33	1.00
23	unknown		b	b	0.20	0.05
24	unknown		b	b	0.30	0.06
25	nonanal	2-octylthiazolidine	b	1.57	2.18	1.51
26	unknown		b	b	0.19	0.11
27	unknown		b	b	0.43	0.28
28	decanal	2-nonylthiazolidine	b	0.69	0.56	0.38
29	undecanal	2-decylthiazolidine	b	b	b	0.09
30	dodecanal[c]	2-undecylthiazolidine	0.65	0.32	0.16	0.10
31	tridecanal[c]	2-dodecylthiazolidine	7.38	2.78	1.22	0.74
32	unknown		0.91	0.36	0.14	0.17

[a] Peak identified on a DB-Wax capillary column. [b] Peak area percent less than 0.02. [c] Tentatively identified (on the basis of MS data alone). [d] Calculated from GC peak area using NPD dectection.

FID, NPD, and FPD, respectively. The linear velocity of the helium carrier gas was 26.5 cm/s, with a split ratio of 1:40.

A HP Model 5890 gas chromatograph interfaced to a VG Trio II mass spectrometer was used for MS identification of the GC components using the same column and oven conditions as stated above. Mass spectra were obtained by electron impact ionization at 70 eV and a source temperature of 165 °C. The spectral data were recorded on a VG 11-250 computer data system.

RESULTS AND DISCUSSION

The total peak area of headspace samples of peanut oil heated at four different temperatures is shown in Figure 1. The total peak area of volatile compounds increased remarkably as the temperature of the oil was increased. This is due to the combined effects of vapor pressure promotion and increased thermal oxidative degradation of fatty acids by subsequent increases in heating temperature (Lomanno and Nawar, 1982).

The volatile compounds identified from headspace samples of peanut oil heated under four different temperatures are listed in Table I in elution order from a DB-Wax column. The gas chromatographic retention index and MS fragmentation pattern of each component were compared to those of the authentic chemical. Since some compounds were not positively identified because of the lack of authentic chemical, a Kovats index could not be compared and only tentative identification could be made.

Ninety-nine volatile compounds among over 150 peaks on the gas chromatograms were identified in this study. These compounds included 42 hydrocarbons, 22 aldehydes, 4 furans, 8 alcohols, 8 ketones, 2 esters, 11 fatty acids, and 2 lactones. The hydrocarbons identified were 18 *n*-alkenes, 7 *n*-alkanes, 5 *n*-alkylbenzenes, 3 alkadienes, 2 cycloalkenes, 2 *n*-alkylcycloalkenes, 2 monoterpene hydrocarbons, 1 bicycloalkane, 1 cycloalkane, and 1 alkyne. Hydrocarbons were identified as the most abundant class of identified compounds; they were followed by aldehydes. Among the hydrocarbons identified, Alencar et al. (1983) reported that *n*-alkanes and *n*-alkenes were the predominant constituents in pyrolyzed (300–500 °C) vegetable oil samples. They also found many *n*-alkylcyclohexanes and *n*-alkylcyclohexenes as pyrolysis products of oil samples. Only 1-pentylcyclopentene and 1-hexylcyclopentene were, however, identified in the present investigation; this may be due to the relatively low temperatures (50–200 °C) used.

The aldehydes identified were 6 *n*-alkanals, 11 *n*-alkenals, 4 *n*-alkadienals, and 1 aromatic aldehyde. Several fatty aldehydes were previously reported in corn oil heated to 180 °C (Macku and Shibamoto, 1991). Some *n*-alkadienals including (*E*,*E*)-2,4-nonadienal, (*Z*,*E*)-2,4-decadienal, and (*E*,*E*)-2,4-decadienal were found in the heated peanut oil but not in the heated corn oil. These aldehydes form by the thermal oxidation of triglycerides through a subsequent C–C scission of linoleate hydroperoxides at elevated temperatures (Nawar, 1989). Methyl ketones were derived from the decarboxylation of β-keto acids produced from triglyceride by heat treatment (Nawar, 1985).

Free fatty acids are also formed from triglycerides upon hydrolysis. Linoleic, oleic, and palmitic acids constitute 67% of total fatty acids in peanut oil (Worthington, 1977). The resulting free fatty acids are transformed to the γ-hydroxy fatty acids by the oxidative attack of OH$^{\bullet}$ at the γ position, followed by further transforamtion to the γ-lactones by cyclization. This transformation is impor-

tant in forming favorable fruitlike aromas characteristic of lactones, such as γ-octalactone and γ-nonalactone found in the present study.

The thiazolidine derivatives identified in the aqueous cysteamine trap are shown in Table II. The *n*-alkanals and *n*-alkanones derived from thermal oxidative degradation of fatty acids are remarkably increased by elevation of heating temperatures. Identifications of 18 thiazolidine derivatives, corresponding to their original carbonyl compounds, were confirmed by a combination of NPD, FPD, retention indices, and mass spectra. These compounds included 3 2,2-dialkylthiazolidines and 15 2-alkylthiazolidines. As shown in Table II, all straight-chain carbonyl compounds corresponded to those identified in the initial analysis shown in Table I, except for the lower molecular weight compounds such as formaldehyde, acetaldehyde, acetone, propanal, 2-pentanone, butanal, and 2-hexanone. These seven carbonyl compounds were detected only with derivatization, which suggests that they may have escaped during the experiment. For example, formaldehyde can easily escape from experimental systems during analysis because of its extremely high volatility (bp = −19.5 °C). Formaldehyde may be produced in this system by decomposition of the 8-alkoxy radical during oxidation and decomposition of the 9-hydroperoxide of linoleic acid and its ethyl ester. Linoleic acid constitutes between 26.7 and 31.4% of peanut oil (Worthington, 1977) and illustrates the potential for high formaldehyde production when peanut oil is heated. Formaldehyde was produced in high amounts even at 50 °C and is possibly formed in peanut oil at room temperature in the presence of oxygen.

In the present investigation heated peanut oil was found to generate fatty, deep-fried, and rancid aromas which intensify as the temperature increases. Formation of off-flavors such as these have been observed in numerous other studies (Frankel, 1991; Ullrich and Grosch, 1988). This can be attributed to the increase in the formation of carbonyl compounds under normal cooking conditions. The direct isolation of low molecular weight carbonyl compounds from the headspace of heated peanut oil proved to be successful only following derivatization to the corresponding thiazolidine compounds. The absence of nitrogen- and sulfur-containing heterocyclic compounds indicates that extremely low levels of proteins and amino acids are present in refined peanut oil; consequently, there are no Maillard-type reactions, and there is a subsequent lack of preferable flavor and aroma.

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