Production of Volatile Compounds from Irradiated Oil Emulsion Containing Amino Acids or Proteins

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ABSTRACT: Oil emulsions containing amino acids, glutathione, bovine serum albumin, gelatin, or myofibrillar proteins were prepared. The emulsions were irradiated at 0, 2.5, 5.0, or 10.0 kGy absorbed doses and analyzed for volatile compounds. Irradiation increased the production of aldehydes (for example, hexanal, heptanal, octanal, and nonanal) indicating that lipid oxidation of oil emulsion was accelerated by irradiation. Irradiation produced, by radiolytic degradations, new volatile compounds from oil emulsions containing leucine, valine, isoleucine, phenylalanine, methionine, or cysteine. This indicated that radiolysis of protein may play an important role in off-odor generation of irradiated meat.

Key Words: volatile compounds, irradiation, off-odor, radiolytic degradation, lipid oxidation

Introduction

IRRADIATION IS AMONG THE BEST METHODS FOR CONTROLLING pathogenic bacteria in raw meat, and low dose (< 7 kGy) irradiation is permitted for use in poultry and red meats to control pathogenic bacteria (Olson 1998). However, irradiation produces a characteristic aroma as well as other significant alterations in meat flavor that significantly and negatively affect consumer acceptance.

Huber and others (1953) reported that meat sterilized by irradiation developed a characteristic odor, which has been described as "metallic," "sulfide," "wet dog," "wet grain," or "burnt." These investigators assumed that the off-odor was the result of free-radical oxidation initiated by the irradiation process. Merritt and others (1978) postulated that carbonyls are formed in irradiated meats due to the reactions of hydrocarbon radicals with molecular oxygen, which follows the same pathway as normal lipid oxidation. Hansen and others (1987) reported that the amount of octane, 1-octene, hexanal, and nonane in irradiated chicken increased with the irradiation dose, but the volatile compounds were not unique products of irradiation. Gas chromatographic separation and odor evaluation of volatile compounds indicated that most sulfur and carbonyl compounds had low odor thresholds and were considered important to irradiation odor (Angellini and others 1975). Batzer and Doty (1955) found that methyl mercaptan and hydrogen sulfide were important to irradiation odor, and the precursors of the undesirable odor compounds in irradiated meat were sulfur-containing compounds that were water-soluble. These results suggest that sulfur-containing compounds could be the major volatile components responsible for irradiation odor in meat.

Ahn and others (1999b) suggested that the volatile compounds responsible for off-odor in irradiated meat are produced by the impact of radiation on protein and lipid molecules and are distinctly different from those of lipid oxidation. Patterson and Stevenson (1995) studied odorous volatile compounds in irradiated chicken meat and found that dimethyltrisulfide is the most potent off-odor compound, followed by *cis*-3- and *trans*-6-nonenals, oct-1-en-3-one, and bis(methylthio-) methane. They also provided evidence to support the concept that the changes detected after irradiation are distinctly different from those of warmed-over flavor in oxidized meat. Heath and others (1990) reported that irradiating uncooked chicken breast and leg at 2 or 3 kGy produced a "hot fat," "burned oil," or "burned feathers" odor that remained after the thighs were cooked. Hashim and others (1995) reported that irradiating uncooked chicken breast and thigh produced a characteristic "bloody and sweet" aroma that remained after the thighs were cooked but was not detectable after the breasts were cooked. Ahn and others (1999b) reported that panelists detected irradiation odor from irradiated pork loin and described it as "barbecued corn-like." However, the panelists showed no preference to the odor.

Although previous evidence has indicated that radiolytic products of protein components were more important than lipid oxidation in the production of irradiation odor in meat, the precise mechanisms by which off-odor is produced are unknown. Substantial differences between the radiation chemistry of food components in model systems and complex food systems exist, but the differences are mostly quantitative rather than qualitative. This fact indicates that off-odor production by irradiation is likely influenced not only by meat composition but also by potential chemical interactions among volatile components. However, this cannot be established without pursuing mechanistic studies using both model and meat systems.

Radiolysis and lipid oxidation are the 2 major possible reaction mechanisms by which volatile compounds could be generated from meat. However, the irradiation dose-dependent generation of hydroxyl radicals and its mechanism on the off-odor production and the initiation of lipid oxidation in biological systems are more suggestive than conclusive. Also, the exact nature and identity of the volatile compounds that contribute to irradiation-induced off-odor are not known. Since animal tissues are composed of 3 major components, proteins, lipids, and water, volatile compounds from model systems prepared with the basic elements of meat will allow us to define the substrates and elucidate the mechanisms of off-odor production in irradiated meat.

The objectives of this research were to elucidate the distinction between volatile compounds previously implicated for oxidized flavor and irradiation odor and to understand the mecha-

nisms of off-odor production in irradiated meat at the chemical basis.

Results and Discussion

IRRADIATED OIL EMULSIONS PRODUCED LARGER NUMBERS OF volatile compounds than nonirradiated controls regardless of compounds added to the oil emulsions (Tables 1 to 4). More volatile compounds than those listed in the tables were produced by irradiation, but the amounts of some volatile compounds were negligible or inconsistent. Chloroform was produced from all samples, but the amount was not influenced by irradiation dose.

Previous reports (Kanatt and others 1998; Jo and others 1999; Jo and Ahn 2000) indicated that irradiation increased lipid oxidation as measured by 2-thiobarbituric acid-reactive substances (TBARS) and/or carbonyl content. Jo and others (1999) reported that lipid oxidation-dependent volatile compounds, such as aldehydes, ketones, and alcohols, were not influenced by irradiation at d 0 but were increased during 7-d storage in aerobic conditions. Several aldehydes quantified and listed in Tables 1 to 4 were produced only in irradiated samples. The amounts of 1-heptene and 1-nonene produced by irradiation were dose dependent as reported previously (Ahn and others 1999a; Jo and others 1999). However, 1-heptene was produced only in the irradiated oil emulsion containing no amino acid, isoleucine, valine, or glutathione (Tables 1 and 4) because of differences in the column and method used. 1,1-Oxybis ethane was 1 of the major compounds produced from irradiated oil emulsion containing aliphatic, hydroxyl, basic side-chain group amino acids, and myofibrillar protein (Tables 1 to 4). The 2-methyl-1-propene, 2,2,6,6-tetramethylheptane, and 2,2,4-trimethyl-1-pentane were found in almost all samples, but the effect of irradiation was not significant ($p > 0.05$).

Isobutyraldehyde and 3-methylbutanal were produced in irradiated oil emulsion containing leucine, and the amount of 3-methylbutanal increased in a dose-dependent manner up to 10 kGy (Table 5). The irradiation dose-dependent production of 2-methylbutanal from oil emulsion containing isoleucine suggests deamination and decarboxylation of isoleucine by irradiation. Diehl (1995) explained that deamination and decarboxylation are the primary reactions of free radicals by irradiation in aliphatic amino acid. He also indicated that deamination plays a greater role than decarboxylation in irradiated alanine. Valine, which has a similar structure to leucine, produced isobutyraldehyde, as leucine did, but the production rate was much higher than that of leucine, indicating that deamination and decarboxylation were major reactions of radiolysis (Table 5). Among the amino acids with aromatic side chains, phenylalanine produced benzaldehyde and benzene acetaldehyde upon irradiation. Phenylalanine readily reacts with the transient species of water radiolysis and induces hydroxylation of the aromatic ring as the principal reaction (Diehl 1995). Therefore, *o*-, *m*-, and *p*-tyrosine can be formed by irradiation, and subsequent oxidation converts these to various isomers of dihydroxyphenylalanine. Simic (1983) explained that the reactions of electron adducts to aromatic residues would give hydrogenated derivatives, thus benzene rings can be converted to cyclohexadiene derivatives. The determination of *o*-tyrosine is 1 of the methods used to detect irradiated food (Meier and others 1996). Other aromatic amino acids (tyrosine, tryptophane, and histidine) were also known as irradiation-sensitive, but no characteristic volatile compounds were found in this study.

Sulfur-containing amino acids (cysteine, cystine, and methionine) react with free radicals more easily than aliphatic amino acids (Simic 1983). Electron reaction with sulfhydryl derivatives leads to formation of H_2S and is of considerable concern in irradiation technology due to its unpleasant odor (Simic 1983). However, H_2S was not detected in this study because the molecular scan range used was 46.1 to 550 to eliminate CO_2 peak. The volatile compounds detected in oil emulsion containing cysteine were carbon oxide disulfide and carbon disulfide (Table 5). Simic

Table 1—Major volatile compounds found in irradiated oil emulsion containing no amino acids or amino acids with aliphatic groups

	None		Leu		Ile		Val		Gly		Ala		Pro	
Volatile compounds	**N**	**I**	**N**	**I**	**N**	**I**	**N**	**I**	**N**	**I**	**N**	**I**	**N**	**I**
1,1-Oxybis ethane	×	×	×	×		×	×	×		×		×		×
2-Propanone*			×	×				×						
Hexane	×	×	×	×	×	×				×		×		×
Isobutyraldehyde*				×				×						
1-Heptene		×				×		×						
3-Methylbutanal*				×										
2-Methylbutanal*						×								
Chloroform	×	×	×	×	×	×	×	×	×	×	×	×	×	×
Hexanal*		×				×	×	×	×	×	×	×		
Heptanal*	×	×		×		×		×	×	×	×	×	×	×
Octanal*	×	×		×						×				
Nonanal*	×	×			×	×	×	×	×	×		×		×
2-Methyl-1-propene	×	×	×	×	×	×	×	×	×	×	×	×		×
2,2,6,6-Tetramethyl-heptane	×	×	×	×	×	×	×	×	×	×	×	×		×
2,2,4-Trimethyl-1-pentane	×	×	×	×	×	×	×	×	×	×		×		×

*Volatile compounds verified by standards and the rest are tentative.
N = nonirradiated; I = irradiated
Abbreviations: Leu = Leucine; Ile = iosleucine; Val = valine; Gly = glycine, Ala = alanine; Pro = proline

Table 2—Major volatile compounds found in irradiated oil emulsions containing amino acids with basic, acidic, or amide side-chain groups

	His		Arg		Lys		Asp		Glu		Asn		Gln	
Volatile compounds	**N**	**I**	**N**	**I**	**N**	**I**	**N**	**I**	**N**	**I**	**N**	**I**	**N**	**I**
1,1-Oxybis ethane		×		×										
Chloroform	×	×	×	×	×	×	×	×	×	×	×	×	×	×
Hexanal*		×	×	×		×		×		×			×	×
Nonanal*			×	×	×		×		×		×			
2-Methyl-1-propene	×	×	×	×	×	×	×	×	×	×	×	×		×
2,2,6,6-Tetramethyl-heptane	×	×	×	×	×	×	×	×	×	×	×	×		×
2,2,4-Trimethyl-1-pentane	×	×	×	×	×	×	×	×	×	×	×	×		×

*Volatile compounds verified by standards and the rest are tentative.
N = nonirradiated; I = irradiated
Abbreviations: His = histidine; Arg = arginine; Lys = lysine; Asp = aspartic acid; Glu = glutamic acid; Asn = asparagine; Gln = glutamine

Table 3—Major volatile compounds found in irradiated oil emulsion containing amino acids with aromatic or those with hydroxyl side-chain groups

	Tyr		Trp		Phe		Thr		Ser	
Volatile compounds	**N**	**I**	**N**	**I**	**N**	**I**	**N**	**I**	**N**	**I**
1,1-Oxybis ethane							×	×		×
Propanal*								×		
2-Propanone*								×		
Chloroform	×	×	×	×	×	×	×	×	×	×
Benzaldehyde						×				
Benzeneacetaldehyde						×				
Hexanal*	×	×	×	×	×	×	×	×	×	×
Nonanal*		×			×	×	×	×		×
2-Methyl-1-propene		×	×	×	×	×	×	×		×
2,2,6,6-Tetramethylheptane							×	×	×	×
2,2,4-Trimethyl-1-pentane				×			×	×		×

*Volatile compounds verified by standards and the rest are tentative.
N = nonirradiated; I = irradiated
Abbreviations: Tyr = tyrosine; Trp = tryptophane; Phe = phenylalanine; Thr = threonine; Ser = serine

Table 4—Major volatile compounds found in irradiated oil emulsions containing sulfur-containing amino acids, glutathione (GSH), bovine serum albumin (BSA), gelatin, or myofibrillar protein (MFP)

	Met		Cys		Cystine		GSH		BSA		Gelatin		MFP	
Volatile compounds	N	I	N	I	N	I	N	I	N	I	N	I	N	I
Pentane										×	×	×		×
Carbon oxide disulfide				×				×						
Methanethiol		×												
1,1-Oxybis ethane													×	×
3-Methylbutanal*													×	×
2-Methylbutanal*													×	×
Methylbenzene										×				×
Carbon disulfide*				×	×									
2-Propenal*		×												
1-Heptene								×						
Chloroform	×	×	×	×	×	×	×	×	×	×	×	×	×	×
Dimethyldisulfide*		×												
Benzaldehyde*										×		×		×
Hexanal*		×				×		×	×	×	×	×	×	×
Ethylbenzene*										×				×
Dimethyltrisulfide		×												
3-Methylthiopropanal*		×												
Nonanal*		×				×		×		×	×	×	×	×
2-Methyl-1-propene		×	×	×		×	×	×						
2,2,6,6-Tetramethyl-heptane						×	×	×						
2,2,4-Trimethyl-1-pentane						×	×	×						

*Volatile compounds verified by standards and the rest are tentative.
N = nonirradiated; I = irradiated
Abbreviations: Met = methionine; Cys = cysteine; GSH = glutathione; BSA = bovine serum albumin; MFP = myofibrillar protein

Table 5—The relative amounts of characteristic volatile compounds in irradiated oil emulsions containing amino acids or proteins

Amino acid or proteins	Volatile compounds	Irradiation dose (kGy) 0	2.5	5.0	10.0	SEM[1]
		total ion counts × 10³				
Leucine						
	Isobutyraldehyde*	0^{b}	197^{b}	243^{b}	1110^{a}	55
	3-Methylbutanal*	0^{d}	9000^{c}	22200^{b}	46700^{a}	2573
Isoleucine						
	Butanal*	0^{b}	77^{ab}	166^{a}	160^{a}	37
	2-Methylbutanal*	0^{d}	20500^{c}	40500^{b}	73500^{a}	1390
Valine						
	Isobutyraldehyde*	0^{d}	5700^{c}	10100^{b}	18500^{a}	520
Phenylalanine						
	Benzaldehyde*	0^{d}	224^{c}	284^{b}	390^{a}	17
	Benzene acetaldehyde	0^{d}	1770^{c}	4060^{b}	8000^{a}	755
Methionine						
	Methanethiol	0^{d}	3560^{c}	6000^{b}	24400^{a}	502
	2-Propenal*	0^{c}	389^{c}	2470^{b}	6240^{a}	222
	Dimethyldisulfide*	0^{d}	7030^{c}	13900^{b}	27500^{a}	645
	Dimethyltrisulfide	0^{d}	33^{c}	273^{b}	432^{a}	11
	3-Methylthiopropanal*	0^{c}	1280^{bc}	3320^{b}	9370^{a}	668
Cysteine						
	Carbonoxide disulfide	0^{b}	0^{b}	19^{b}	145^{a}	13
	Carbon disulfide*	0^{d}	384^{c}	1060^{b}	1530^{a}	101
Cystine						
	Carbon disulfide*	742^{a}	0^{b}	0^{b}	0^{b}	28
Glutathione						
	Carbonoxide disulfide	0^{b}	0^{b}	69^{b}	261^{a}	40
Bovine serum albumin						
	Methylbenzene	0^{b}	0^{b}	130^{a}	137^{a}	21
	Ethylbenzene*	0^{b}	0^{b}	132^{a}	148^{a}	23
	Benzaldehyde*	0^{c}	42^{b}	98^{a}	117^{a}	13
Gelatin						
	Benzaldehyde*	0^{b}	0^{b}	152^{ab}	275^{a}	44
Myofibrillar proteins						
	Methylbenzene	0^{b}	0^{b}	210^{a}	227^{a}	37
	Ethylbenzene*	0^{b}	0^{b}	214^{a}	199^{a}	39

*Volatile compounds verified by standards and the rest are tentative.
[a-d]Means with different superscripts in the same row differ significantly ($p < 0.05$).
[1]Standard errors of the mean among different irradiation doses (n = 16)

(1983) reported that aqueous electrons (e^-_{aq}) generated by irradiation exclusively attack -SH and not the -NH^{3+} group of cysteine, and induce γ-radiolysis. It was also assumed that cysteine underwent dimerization reaction to form cystine as suggested by others (Simic 1983; Diehl 1995). In contrast to cysteine, cystine produced carbon disulfide from nonirradiated oil emulsion, but carbon disulfide was not found in irradiated oil emulsions (Table 5). The carbon oxide sulfide was also produced from the irradiated oil emulsion containing glutathione due probably to cysteine in the structure. The amount of carbon oxide sulfide from glutathione was much smaller than that from cysteine, and probably the cysteine present as part of a peptide is less susceptible to free-radical reaction (Diehl 1995).

Four sulfur-containing compounds were produced by irradiation from oil emulsion containing methionine (Table 5). These compounds have been suggested as the main off-odor generators (Schweigert and others 1954; Patterson and Stevenson 1995). This indicated that the major source of off-odor production in irradiated meat could be sulfur-containing amino acids via the radiolytic degradation of their side chains. Interestingly, 2-propenal, which generates unpleasant odor, was also produced by irradiation in a dose-dependent manner. Removal of -SCH_3 moiety from methionine followed by deamination and decarboxylation could have produced 2-propenal. Since the dimethylthio-group (-C-S-C-) has a low reactivity with aqueous electrons (e^-_{aq}, Simic 1983), methionine is more likely to react with hydroxyl radicals from water radiolysis.

Bovine serum albumin (BSA), gelatin, and myofibrillar protein produced many benzene-containing compounds, but irradiation at the 2.5-kGy dose could not create these products except for benzaldehyde from BSA (Table 5). The rigid spatial structure of protein molecules could have protected amino acid side chains from radiolytic degradation, in contrast to the effect on free amino acids. Diehl (1995) reported that radicals formed by irradiation can be held in position and be recombined. Therefore, radiation damage to certain amino acids in proteins is quite limited, and the proteins tested may not be affected at low-dose irradiation (<2.5 kGy). WHO (1994) reported that little changes in amino-acid composition were observed at doses below 50 kGy. Bachman and others (1973) reported that free-radical yield measured by electron spin resonance (ESR) spectroscopy was increased by irradiation, but there were no changes in organoleptic characteristics and chemical composition in gelatin irradiated at the 5-kGy dose. Oil emulsion containing myofibrillar protein produced 3-methylbutanal and 2-methylbutanal, but the amounts of these compounds were not influenced by irradiation doses between 0 and 10 kGy.

Ahn and others (1998) and Shahidi and Pegg (1994) reported that hexanal is the most sensitive indicator for lipid oxidation and flavor deterioration in meat products. Our results showed that irradiation significantly increased the amount of hexanal in oil emulsion containing different compounds (Table 6). Apparently, lipid oxidation was accelerated by irradiation. Irradiation-induced oxidative chemical changes were dose-dependent, and the presence of oxygen had a significant effect on the rate of lipid oxidation. Other aldehydes such as propanal, heptanal, octanal, and nonanal were also produced but the trends were similar to that of the hexanal (data not shown, $p < 0.05$.) Ang and Lyon (1990) reported that hexanal and pentanal had a very strong correlation with TBARS, and off-odor related to lipid oxidation in meat.

Irradiation produced significant amounts of volatile compounds from oil emulsion containing amino acids, indicating that the radiolytic products of amino acids could be important for off-odor production in irradiated meat. However, the responses of singular amino acid and proteins to irradiation and those of oil emulsion and meat systems would be different. Therefore, this study should be considered as a step to determine the sources of volatiles and the mechanisms of off-odor production in irradiated meat.

Conclusions

THE EVIDENCE SUGGESTS THAT THE RADIOLYTIC PRODUCTS OF meat components, such as proteins, lipids, amides, amines, vitamins and so on, are the major sources of off-odor volatiles in irradiated meat. Irradiation produced significant amount of volatile compounds from oil emulsion containing sulfur-containing amino acids and those with aliphatic side-chains. Volatile compounds produced by lipid oxidation during and after irradiation in the presence of oxygen also could contribute to the development of off-odor in irradiated sample.

Table 6—The relative amount of hexanal produced in irradiated oil emulsions containing different amino acids, glutathione, bovine serum albumin, and gelatin

Amino acids or proteins	Irradiation dose (kGy)				
	0	2.5	5.0	10.0	SEM[1]
	total ion counts × 10³				
None	0[d]	484[c]	1088[b]	1680[a]	123.3
Isoleucine	0[b]	0[b]	110[b]	411[a]	31.3
Threonine	282[b]	715[a]	758[a]	918[a]	82.1
Serine	0[c]	517[b]	590[b]	917[a]	34.1
Histidine	0[b]	1078[a]	944[a]	813[a]	101.9
Lysine	0[d]	565[c]	675[b]	915[a]	26.7
Aspartic acid	0[c]	863[b]	821[b]	1236[a]	37.9
Glutamic acid	0[c]	674[b]	724[b]	857[a]	21.9
Asparagine	143[c]	732[b]	678[b]	986[a]	74.9
Glutamine	0[d]	584[c]	739[b]	968[a]	38.8
Tyrosine	225[b]	802[ab]	1185[a]	1343[a]	195.0
Methionine	0[d]	190[c]	303[b]	458[a]	18.2
Cystine	0[d]	744[c]	926[b]	1053[a]	28.7
Glutathione	0[b]	349[b]	1101[a]	1512[a]	139.2
Bovine serum albumin	583[b]	1358[a]	1231[a]	1703[a]	192.6
Gelatin	501[b]	225[b]	675[b]	1176[a]	148.1

[a-d]Means with different superscripts in the same row differ significantly ($p < 0.05$).
[1]Standard errors of the mean among different irradiation doses ($n = 16$)

Materials and Methods

Sample preparation

Oil emulsions were prepared by blending soybean oil (2 mL, Preferred Products, Inc., Eden Prairie, Minn., U.S.A.), cholesterol (5% of soybean oil, w/v), and Triton X-100 (50 μL) with 200 mL maleate buffer (50 mM, pH 5.8). Amino acid (50 mM), glutathione (25 mM), or protein was added to the buffer before blending with a Waring blender for 2 min at high speed. Amino acids used included those with the aliphatic side-chain group (leucine, isoleucine, valine, glycine, alanine, and proline), the hydroxyl side-chain group (threonine and serine), the basic side-chain group (histidine, arginine, and lysine), the acidic side-chain group (aspartate and glutamate), the amide side-chain group (glutamine and asparagine), the aromatic side-chain group (tyrosine, tryptophane, and phenylalanine), and the sulfur-containing side-chain groups (methionine, cysteine, and cystine). Proteins used included bovine serum albumin (BSA, 5%, w/v), gelatin (2%, w/v), and myofibrillar proteins (MFP, 20%, w/v).

Myofibrillar proteins were obtained using the following procedure: Ground pork muscle was homogenized with a solution [100 mM KCl, 20 mM potassium phosphate (pH 6.8), 2 mM $MgCl_2$, 2 mM EGTA, and 1 mM NaN_3 (standard salt solution)] for 10 s in a Waring blender and centrifuged at 1500 × *g* for 10 min. The sediment was suspended in 8 volumes (v/w) of standard salt solution and homogenized in the blender for another 10 s. The homogenate was filtered through a household nylon net strainer, and the filtrate was centrifuged at 1500 × *g* for 10 min. The sediment was resuspended in 8 volumes (v/w) of 100 mM KCl, homogenized for 10 s in the blender, and centrifuged at 1500 × *g*. The sediment was used as a myofibrillar protein for this study.

A 10-mL portion of oil emulsion was transferred to 20-mL sample vials, irradiated at the 0, 2.5, 5.0, or 10.0 kGy absorbed dose using an Electron Beam irradiator (Circe IIIR Thomson CSF Linac, St. Aubin, France) with a 10 MeV energy level, a 10 kw power level, and an average 86 kGy/min dose rate after storage overnight in a 4 °C cooler. The irradiation process was conducted at room temperature with single-layer display and single-sided dosage. Oil emulsion with no amino acid was prepared as a control. Samples were stored in a 4 °C refrigerator and analyzed for volatile compounds.

Volatile compounds analysis

A Precept II and a Purge-and-Trap concentrator 3000 (Tekmar-Dohrmann, Cincinnati, Ohio, U.S.A.) were used to purge and trap volatile compounds as described by Ahn and others (1998) with modifications. A gas chromatograph (GC; Model 6890, Hewlett Packard Co., Wilmington, Del., U.S.A.) equipped with a mass selective detector (MSD; Model 5973, Hewlett Packard Co.) was used to identify and quantify the volatile compounds. Each sample (2 mL) was transferred to a 40-mL sample vial, and headspace was flushed with helium gas (99.999% purity) for 5 s to minimize oxidation during the waiting period before analysis. Samples were purged with helium (40 mL/min) for 14 min at 40 °C. Volatile compounds were trapped using a Tenax/silica/charcoal column (Tekmar-Dohrmann) and desorbed for 1 min at 220 °C. The temperature of transfer lines was maintained at 135 °C. A modified column was used to improve separation of volatile compounds. An HP-Wax column (7 m, 250 μm i.d., 0.25 μm nominal) was combined with an HP-5 column (30 m, 250 μm i.d., 0.25 μm nominal) using a Glass Press-fit Connector (Hewlett Packard Co.) A split inlet (split ratio, 49:1, inlet temperature 180 °C) was used to inject volatile compounds into the column, and a ramped oven temperature was used (7 °C for 2.5 min, increased to 25 °C @3 °C/min, to 105 °C @8 °C/min, to 200 °C @30 °C/min, and held for 0.33 min). Liquid nitrogen was used to cool the oven below the ambient temperature. Helium was the carrier gas at a constant flow of 1.2 mL/min. The ionization potential of mass spectrum was 70 eV; the scanned mass range was 46.1 to 550, and the scan velocity was 2.94 scan/sec. The identification of volatile compounds was achieved by comparing mass spectral data with those of the Wiley library (Hewlett Packard Co.), and some compounds were verified using external standards. Standards were added to sample vials containing 2 mL of oil emulsion and run with the same method used for oil emulsions with amino acids or proteins. Carbon oxide sulfide, 2,3-dimethyldisulfide, ethylbenzene, isobutyraldehyde were purchased from Aldrich (Milwaukee, Wisc., U.S.A.), and 2-methylbutanal, 3-methylbutanal, 2-propenal, 2-propanone, hexanal, heptanal, octanal, and nonanal were obtained from Chromatography Research Supplies Inc. (Addison, Ill., U.S.A.) The peak area (total ion counts × 10^3)

was reported as the amount of volatile compounds released. The peak area less than 20,000 was discarded and was considered as no production.

Statistical analysis

Analysis of variance was conducted to determine any difference in volatile profiles and volatile contents by using the SAS program (SAS 1989). Four replications were conducted, and significant level used was $p < 0.05$. Mean values were compared by Student-Newman-Keul's multiple range test (Steel and Torrie 1980), and mean values and standard errors of the mean (SEM) were reported.

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