Volatile Components of Ripe Tomatoes and Their Juices, Purées and Pastes

Tae-Yung CHUNG,* Fumitaka HAYASE and Hiromichi KATO**

*Department of Agricultural Chemistry, The University of Tokyo,
Bunkyo-ku, Tokyo 113, Japan*

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Volatile components in neutral, basic, phenolic and acidic fractions of the steam-distillate and in headspace gas trapped from two varieties of fully ripe tomato fruits and their juices, purées and pastes were analyzed by GC and GC-MS using a glass capillary column. One hundred and fifty-four compounds were identified: they were 3 sulfides, 10 aromatic hydrocarbons, 4 terpene hydrocarbons, 27 alcohols, 26 aldehydes, 13 ketones, 17 esters, 2 lactones, 7 furans, 4 pyrroles, 2 pyridines, 2 pyrazines, 2 thiazoles, 12 phenols, 16 carboxylic acids and 7 others. Of these, 39 compounds were identified for the first time in tomato fruits and their products.

Generally, with the advance of the processing degree from tomato fruits to pastes, decrement of low-boiling components and increment of middle to high-boiling components were observed. In tomato purées and pastes, 14 components, which were detected in tomato fruits and juices, disappeared and 11 components, pyrrole, 2,5-dimethylpyrrole, 2-acetylpyrrole, 2-formylpyrrole, 2-methylpyrazine, 2,6-dimethylpyrazine, dibenzofuran, *o*-phenylanisole, farnesol, indole and diphenylamine, were newly produced. 2-Acetylpyrrole, 2-formylpyrrole and the above pyrazines, which have respectively a burnt odor, are considered to be important for the characteristic heated aroma of tomato purée and paste.

A number of studies concerning the volatile components of tomatoes have been carried out since 1954.^{1~5)} More than one hundred and fifty compounds were characterized in fresh tomato fruits. As important components for the odor of tomato fruits, hexanal, *trans*-2-hexenal, *cis*-3-hexenal, *cis*-3-hexenol and 2-isobutylthiazole were evaluated.^{6,7)} In a previous paper,⁸⁾ we reported the changes of flavor components of tomato fruits during artificial ripening. The results obtained were that the amounts of components in the steam-distillate of field-grown tomatoes generally increased with ripening, particularly those of middle and high boiling components.

On the other hand, the volatile components of heated tomato and tomato products have not been characterized in detail. Miers⁹⁾ and Nelson and Hoff¹⁰⁾ reported that the dimethyl sulfide content increased in the volatile fraction of canned tomato juice during the heat

processing. Sieso and Crouzet¹¹⁾ investigated the effect of processing on the volatile components present in tomato products, mainly canned tomato juice and paste, by packed column gas chromatography. They identified sixty components and found that important heat-induced volatile products were furfural, linalyl acetate and 2-methyl-2-hepten-6-one. In the present work, we investigated the volatile components of fully ripe tomato fruits and their products (juices, purées and pastes) in detail. Both the steam distillation method and headspace gas trapping method were applied for collecting the volatiles, and glass capillary column gas chromatography was used for separating them.

MATERIALS AND METHODS

Tomatoes. Fully ripe tomato fruits of two varieties, Wasedaruma and Kagome 77, which were harvested at a

* Present address: Busan National University, College of Home Economics, Busan, Korea.

** Address correspondence to this author.

farm of the Institute of Kagome Co. Ltd., Tochigi Prefecture, in 1979 and 1980, were used. These varieties are those for processing use.

Preparation of tomato products. Tomato juices, purées and pastes were prepared using an experimental process line. The preparation conditions in the case of Wasedaruma tomatoes were as described below. Tomato juice was obtained with a pulper and finisher after a hot break (90~96°C for 22 min). The juice was pasteurized in boiling water for 20 min. Tomato purée was obtained by evaporating the juice under vacuum at 41.7°C for 107 min. Tomato paste was obtained by evaporating further the purée at 42.5°C for 89 min. The purée and paste were pasteurized at 95°C for 15 min. The solid contents of the juice, purée and paste were 4.56, 14.90 and 21.41%, respectively. Tomato juice, purée and paste from Kagome 77 tomatoes were also prepared under essentially similar conditions. Tomato fruits and their products were stored at -20°C until use.

Preparation and fractionation of steam-distillate concentrate. One kilogram of crushed tomato fruits, tomato juice, purée or paste was placed in a 10 liter glass vessel and steam distillation was performed for 1.5 hr under atmospheric pressure. The steam-distillate (ca. 400 ml) was saturated with sodium chloride and then extracted with diethyl ether (ca. 400 ml). The ether extract was concentrated at 36~38°C under atmospheric pressure. This process was repeated six times and thus a steam-distillate concentrate (ca. 1.3~1.5 g) was obtained from 6 kg of each sample. The concentrate was fractionated into neutral, basic, phenolic and acidic fractions by the usual method. Each fraction was concentrated by a similar method. The acidic fraction was treated with diazomethane for methylation of the acids in the fraction. These fractionated volatile components were analyzed by GC and GC-MS.

Trapping of headspace gas. The Tenax GC trapping technique described in previous papers^{12,13} was applied. Five hundred grams of crushed tomato fruits or tomato product was placed in a flask (500 ml) and heated in a water bath at 60°C, and the headspace volatiles were trapped in a Tenax GC trapping tube (4 mm i.d. × 15 cm) by passing through a stream of nitrogen (50 ml/min) for 1 hr. In order to remove the water contained in the Tenax tube, the nitrogen flow was continued through the Tenax tube for 30 min. GC and GC-MS analyses were carried out by the direct injection technique.¹²⁾

Gas chromatography (GC). Each fraction obtained from the steam-distillate concentrate was analyzed with a Shimadzu Model 5A Gas Chromatograph equipped with a flame ionization detector (FID). A glass capillary column (60 m × 0.28 mm i.d.) coated with PEG 20M was used. The column oven temperature was programmed from 60°C to

190°C at a rate of 4°C/min. The injection port and detector temperatures were kept at 200°C. Nitrogen was used as the carrier gas at a flow rate of 1.5 ml/min with a split ratio of 1:15.3.

Headspace gas trapped in Tenax GC was analyzed with a Shimadzu Model 7A Gas Chromatograph equipped with a FID and a flame photometric detector (FPD), using the same glass capillary column. Nitrogen was used as the carrier gas at a flow rate of 1.54 ml/min with a split ratio of 1:12. Other analytical conditions were the same as described above.

Gas chromatography-mass spectrometry (GC-MS). GC-MS spectra were recorded with a Hitachi Model RM-50 Mass Spectrometer connected with a Hitachi Model 063 Gas Chromatograph. The ionization voltage was 25 eV and the ion source temperature was kept at 200°C. A glass capillary column (50 m × 0.28 mm i.d.) coated with PEG 20M was used. The column oven temperature was programmed from 60 to 170°C at a rate of 3°C/min and the injection port temperature was 200°C. The carrier gas (helium) was controlled at an inlet pressure of 0.2 kg/cm².

RESULTS AND DISCUSSION

Yield and odor of each fraction obtained from steam-distillates of tomatoes and their products

Table I shows the yield and odor description of each fraction obtained from the steam-distillate concentrates of two varieties of tomato fruits and their products. Most of the volatile components of each sample were contained in the neutral fraction and the odor of the neutral fraction of each sample was almost the same as the odor of the steam-distillate concentrate, respectively. The neutral fraction of fresh tomato fruits had a fresh tomato-like aroma with an oily odor. The heated odor of each tomato product, which is characteristic of heated tomato juice, purée and paste, was also perceived in their neutral fractions. Basic, phenolic and acidic fractions of each sample, of which the yields were very low, had a burnt, medicinal or ground roasted soybeans-like and rancid or sweat-like odor, respectively, and almost no difference among the samples was seen. These results indicate that important components of the fresh odor or heated odor were contained in the neutral fraction of each sample.

TABLE I. YIELD AND ODOR OF EACH FRACTION OBTAINED FROM THE STEAM-DISTILLATE OF TOMATO FRUITS AND THEIR PRODUCTS

	Fraction	Yield (g)*		Odor**
		(1)	(2)	
Fresh tomato fruit	NF	1.203	1.102	Oily, fresh tomato-like
	BF	0.054	0.051	Burnt
	PF	0.059	0.054	Medicinal, ground roasted soybeans
	AF	0.075	0.071	Rancid or sweat-like
Tomato juice	NF	1.239	1.301	Oily, tomato juice-like
	BF	0.042	0.050	Burnt
	PF	0.044	0.052	Medicinal, ground roasted soybeans
	AF	0.049	0.063	Rancid or sweat-like
Tomato purée	NF	1.229	1.296	Oily, tomato purée-like
	BF	0.038	0.069	Burnt
	PF	0.032	0.064	Medicinal, ground roasted soybeans
	AF	0.043	0.122	Rancid or sweat-like
Tomato paste	NF	1.277	1.331	Oily, tomato paste-like
	BF	0.045	0.060	Burnt
	PF	0.057	0.053	Medicinal, ground roasted soybeans
	AF	0.070	0.073	Rancid or sweat-like

NF, neutral fraction; BF, basic fraction; PF, phenolic fraction; AF, acidic fraction.

(1), Wasedaruma and their products; (2), Kagome 77 and their products.

* Yield from 6 kg of each sample.

** The ether solution of each fraction was absorbed on a filter paper and the filter paper was air-dried to remove the solvent and then subjected to the sensory test by 10 members of our laboratory.

Identified volatile components

Volatile compounds identified in the present investigation are listed in Table II. One hundred and fifty-four components were identified. Of these, thirty-nine were identified for the first time in tomato fruits and their products. Eleven components were found only in tomato purées and pastes, whereas fourteen components were detected only in fresh tomato fruits and tomato juices and disappeared in purées and pastes. These volatiles are described individually below.

Volatiles in the steam-distillate

1) *Neutral fraction.* Figure 1 shows gas chromatograms of the neutral fractions of Wasedaruma fresh tomato fruits and their products. Since the volatile components of the two varieties, Wasedaruma and Kagome 77, were qualitatively similar, gas chromatograms obtained for Wasedaruma samples are only

shown in this paper.

Generally, decrement of low-boiling components and increment of middle to high-boiling components were observed with the advance of the processing degree. However, methyl linoleate (peak 214), ethyl linoleate (peak 218), methyl linolenate (peak 220) and ethyl linolenate (peak 222) disappeared completely in tomato purées and pastes. On the other hand, *trans*-2-hexenal (peak 51) and *cis*-3-hexenol (peak 78) increased remarkably in tomato juices. These results suggest that C6 compounds were partly formed through hydrolysis and breakdown of the above fatty acid esters by enzymatic and/or nonenzymatic reaction. In tomatoes, the major volatiles on enzymatic breakdown of fatty acids are known to be hexanal and *cis*-3-hexenal.^{2,14-16)} However, *cis*-3-hexenal is unstable both to heat and acidity and is easily isomerized to *trans*-2-hexenal.²⁾ In the present investigation,

TABLE II. VOLATILE COMPONENTS IDENTIFIED IN THE STEAM-DISTILLATE AND HEADSPACE GAS OF TOMATO FRUITS AND THEIR PRODUCTS

Peak no.	Compound	Remarks**	Peak no.	Compound	Remarks**
<i>Aromatic hydrocarbons</i>			16	Pentanal	H
12	Benzene	H	30	Hexanal	H
24	Toluene	H	47'	Heptanal	
38	Ethylbenzene	H	51	<i>trans</i> -2-Hexenal	H
40	<i>p</i> -Xylene	H	65	Octanal	<i>a</i>
43	<i>m</i> -Xylene		71	<i>trans</i> -2-Heptenal	H
50	1-Phenylpropane	H	83	<i>trans,trans</i> -2,4-Hexadienal	
58	Styrene	H	87	<i>trans</i> -2-Octenal	H
61	<i>p</i> -Cymene		91	2-Furfural	
64	Pseudocumene	H	94	<i>trans,trans</i> -2,4-Heptadienal	<i>c</i>
75	1,2,3-Trimethylbenzene		95	Citronellal	
<i>Terpene hydrocarbons</i>			97	Decanal	<i>c</i>
41	Sabinene*	H	103	Benzaldehyde	H
44	δ -3-Carene*		104	<i>trans</i> -2-Nonenal	H
45	Myrcene*		111	5-Methylfurfural	
67	Terpinolene*	<i>a</i>	119	<i>p</i> -Torualdehyde	
<i>Alcohols</i>			121	Phenylacetaldehyde	
9	Ethanol	H	127	Neral	
19	2-Butanol	<i>c</i>	133	Geranial	
21	1-Propanol		144	<i>trans,trans</i> -2,4-Decadienal	
21'	2-Methyl-3-buten-2-ol		147	<i>trans,cis</i> -2,4-Decadienal	
27'	2-Methyl-1-propanol		154	<i>m</i> -Methoxybenzaldehyde	<i>a</i>
32	1-Propen-3-ol		<i>Ketones</i>		
39	1-Butanol	H	1	Acetone	H'
42	1-Penten-3-ol	H	14	Diacetyl	
48	3-Methyl-1-butanol	H	27	2-Hexanone	
55	1-Pentanol	H	35	3-Penten-2-one	H
73	1-Hexanol	H, <i>c</i>	46'	2-Heptanone	
78	<i>cis</i> -3-Hexenol	H	47	Cyclopentanone	
82	<i>trans</i> -2-Hexenol	H	72	2-Methyl-2-hepten-6-one	H
88	1-Octen-3-ol		86	<i>trans,trans</i> -3,5-Octadien-6-one	
89	<i>trans</i> -Linalooloxide (furanoid)	<i>a</i>	113	2-Methyl-2,4-heptadien-6-one	
90	2-Methyl-2-hepten-6-ol		116	Undecanone	
93	<i>cis</i> -Linalooloxide (furanoid)		151	Geranylacetone	
106	Linalool	H	162	β -Ionone	
108	<i>cis</i> -4-Hexenol*	<i>a</i>	205	Farnesylacetone	
122	Furfuryl alcohol		<i>Pyrroles</i>		
128	α -Terpineol		102	Pyrrole	<i>a, b</i>
138	Citronellol	<i>c</i>	114	2,5-Dimethylpyrrole*	<i>a, b</i>
149	Geraniol		164	2-Acetylpyrrole	<i>a, b</i>
152	Benzyl alcohol		170	2-Formylpyrrole	<i>a, b</i>
158	2-Phenylethanol		<i>Lactones</i>		
174	Nerolidol	<i>a</i>	171''	γ -Nonalactone	
203	Farnesol	<i>a, b</i>	202	Dihydroactinidiolide	
<i>Aldehydes</i>			<i>Esters</i>		
H-1	Methanal	H'	2'	Methyl acetate	
H-2	Ethanal	H'	2	Ethyl formate	H
4	Acrolein		6	Ethyl acetate	H
10	3-Methyl-1-butanal	H	48'	Methyl hexanoate	

TABLE II. (continued).

Peak no.	Compound	Remarks**	Peak no.	Compound	Remarks**
68	<i>trans</i> -3-Hexenyl acetate		167	Phenol	
69	<i>cis</i> -3-Hexenyl acetate		167'	<i>o</i> -Cresol	
83'	Methyl octanoate	H, <i>a</i>	171'	<i>p</i> -Ethylguaiacol	<i>a</i>
171	Methyl myristate		177	<i>p</i> -Cresol	<i>a</i>
178	Ethyl myristate	<i>a</i>	187	Eugenol	<i>c</i>
181	Methyl pentadecanoate	<i>c</i>	188'	3,4-Xylenol	<i>a</i>
193	Methyl palmitate	<i>c</i>	189	4-Vinylguaiacol	
196	Ethyl palmitate	<i>a</i>	204	4-Vinylphenol	
211	Methyl oleate	<i>a</i>			
214	Methyl linoleate	<i>a, c</i>		<i>Sulfides</i>	
218	Ethyl linoleate	<i>a, c</i>	H-3	Dimethyl sulfide	H'
220	Methyl linolenate	<i>a, c</i>	H-4	Methyl ethyl sulfide	H', <i>a</i>
222	Ethyl linolenate	<i>a, c</i>	29	Dimethyl disulfide	H
	<i>Furans</i>			<i>Acids</i>	
8	2-Methylfuran	H, <i>a</i>	A-1	Acetic acid	
13	2-Ethylfuran	H	A-2	Propionic acid	
54	2-Pentylfuran	H	A-3	Isobutyric acid	
57	2-Methyl-5-iso-propenylfuran	<i>a</i>	A-4	Butanoic acid	<i>a</i>
100	2-Acetylfuran		A-5	Valeric acid	<i>a</i>
115	5-Methyl-2-acetylfuran		A-6	Hexanoic acid	<i>a</i>
196'	Dibenzofuran	<i>a, b</i>	A-7	Heptanoic acid	<i>a</i>
	<i>Pyridines</i>		A-8	Octanoic acid	
46	Pyridine	H	A-9	Nonanoic acid	<i>a</i>
109	2-Formylpyridine	<i>a</i>	A-10	Benzoic acid	
	<i>Pyrazines</i>		A-11	Myristic acid	<i>a</i>
58'	2-Methylpyrazine	<i>b</i>	A-12	Pentadecanoic acid	<i>a</i>
71'	2,6-Dimethylpyrazine	<i>b</i>	A-13	Palmitic acid	
	<i>Thiazoles</i>		A-14	Stearic acid	
84	2-Isobutylthiazole	H	A-15	Oleic acid	
163	Benzothiazole		A-16	Linoleic acid	
	<i>Phenols</i>			<i>Others</i>	
139	Methyl salicylate	<i>c</i>	25	<i>p</i> -Dioxane	<i>a</i>
142	<i>o</i> -Hydroxyacetophenone		62	Acetoin	H
145	Ethyl salicylate	<i>a</i>	160	Phenylacetnitrile	
150	Guaiacol	<i>c</i>	183	<i>p</i> -Methoxyacetophenone	
			197	<i>o</i> -Phenylanisole	<i>a, b</i>
			208	Indole	<i>a, b</i>
			221	Diphenylamine	<i>a, b</i>

* Compounds identified by MS only.

** H, compounds identified in both the headspace gas and steam-distillate; H', compounds identified only in the headspace gas; *a*, compounds identified for the first time in fresh tomato fruits, juices, purées and paste; *b*, compounds identified only in tomato purée and paste; *c*, compounds not identified in tomato purée and paste.

cis-3-hexenal was not detected, suggesting that it was isomerized during the steam distillation.

As shown in Table II, "a"-marked components were identified for the first time in tomato fruits and their products. Of these components, 2-methylfuran (peak 8) and 2-methyl-5-isopropenylfuran (peak 55) are

known as volatile components formed during thermal degradation of carbohydrates.¹⁷⁾ Two linalooloxides (furanoid) (peaks 89 and 93) were identified as minor components of the neutral fraction. Linalooloxides are known as major volatile components of pouchong tea.¹⁸⁾ Sieso and Crouzet¹¹⁾ reported that linalyl ace-

FIG. 1. Gas Chromatograms of the Neutral Fraction of Wasedaruma Tomato Fruits and Their Products. BHT is a contaminant originated from ether used as solvent.

tate was one of the important major components in tomato paste. However, linalyl acetate was not identified in the present investigation. This discrepancy would be due to the difference of tomato varieties and preparation conditions.

Of the "a"-marked components, pyrrole (peak 102), 2,5-dimethylpyrrole (peak 114), 2-acetylpyrrole (peak 164), 2-formylpyrrole (peak 170), dibenzofuran (peak 196'), *o*-phenylanisole (peak 197), farnesol (peak 203) and indole (peak 208) were detected only in tomato purée and paste. Since 2-acetylpyrrole and 2-formylpyrrole have respectively a burnt odor, they are considered to be important components for the heated odor of tomato purée and paste. These pyrroles are probably formed by the Maillard reaction during the heat treatment.¹⁹⁾ Indole is known to be formed on heating of tryptophan²⁰⁾ and protein.²¹⁾

2) *Basic fraction.* Figure 2(A) shows a gas chromatogram of the basic fraction of Wasedaruma fresh tomato fruits. This fraction

was further analyzed by GC with FPD. As a result, 2-isobutylthiazole (peak 84) and benzothiazole (peak 163) were identified. 2-Isobutylthiazole is known to be an important component for the characteristic flavor of fresh tomatoes.^{2,4,7)} The relative amount of 2-isobutylthiazole was observed to decrease in tomato purée and paste. The decrement of this component is supposed to have a significant effect on the aroma of tomato products.

2-Methylpyrazine (peak 58') and 2,6-dimethylpyrazine (peak 71') were not detected in fresh tomato fruits and their juices but were detected in purées and pastes. These pyrazines are considered to be formed by the interaction between sugars and amino acids²²⁾ and to have a significant effect on the heated aroma of tomato purée and paste. 2-Formylpyridine (peak 109) and diphenylamine (peak 221) were identified for the first time in the present investigation but their formation pathways are not known.

3) *Phenolic fraction.* Figure 2(B) shows a gas chromatogram of the phenolic fraction of

FIG. 2. Gas Chromatograms of the Basic Fraction (A), Phenolic Fraction (B) and Acidic Fraction Methylated with Diazomethane (C) of Wasedaruma Tomato Fruits.

BHT is a contaminant originated from ether used as solvent.

fresh tomato fruits. Major components in the fraction were methyl salicylate (peak 139), guaiacol (peak 150), phenol (peak 167'), *o*-cresol (peak 167), eugenol (peak 187) and 4-vinylguaiacol (peak 189). Ethyl salicylate (peak 145), *p*-ethylguaiacol (peak 171'), *p*-cresol (peak 177) and 3,4-xylene (peak 188') were identified for the first time. A remarkable increment of 4-vinylphenol (peak 204) was observed in tomato purée and paste, suggesting that the increment is based on decarboxylation of *p*-coumaric acid during the heat treatment.²³⁾

4) *Acidic fraction.* A gas chromatogram of the methylated acidic fraction of fresh tomato fruits is shown in Fig. 2(C). Major components of the fraction were butanoic acid (peak A-4), hexanoic acid (peak A-6), octanoic acid (peak A-8) and palmitic acid (peak A-13). Valeric acid (peak A-5), hexanoic acid, heptanoic acid (peak A-7), nonanoic acid (peak A-9), benzoic acid (peak A-10), myristic acid (peak A-11) and pentadecanoic acid (peak A-12) were identified for the first time. Each acidic component did not show any remarkable change during the preparation process for each tomato

product.

Volatiles in headspace gas

Volatile components in the headspace gases of fresh tomato fruits, tomato juices, purées and pastes were trapped by the Tenax GC trapping method¹²⁾ and analyzed by GC with both FID and FPD. Figure 3 shows a gas chromatogram of the headspace gas of the fresh tomato fruits. Forty-two components, including four sulfur-containing compounds, dimethyl sulfide (peak H-3), methyl ethyl sulfide (peak H-4), dimethyl disulfide (peak 29) and 2-isobutylthiazole, were identified and they are also listed in Table II. Of these, some low-boiling components, acetone (peak 1), methanal (peak H-1), ethanal (peak H-2), dimethyl sulfide and methyl ethyl sulfide, were identified only by the headspace trapping method. These components were not characterized by the steam distillation method, because of their loss during evaporation of solvent and interference by the solvent in gas chromatographic analysis. Methyl ethyl sulfide was identified for the first time in tomato fruits and their products.

FIG. 3. Gas Chromatogram of Headspace Volatiles of Wasedaruma Tomato Fruits.

Generally, the amounts of volatiles in headspace gas decreased with the advance of the processing degree. Any volatile component, which was detected only in tomato purée and paste, was not found with the headspace trapping method, indicating that high-boiling components such as 2-acetylpyrrole and 4-vinylphenol are hardly evaporated. On the other hand, dimethyl sulfide was observed to increase in tomato juices. Dimethyl sulfide is well known as an important component of the heat-induced odor of tomatoes.^{6,9,10} The formation of dimethyl sulfide during the heating process is ascribable mainly to degradation of the S-methylmethionine sulfonium ion.²⁴⁾

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