Influence of Ascorbic Acid and Ascorbyl Palmitate on the Aroma Composition of an Oxidized Vegetable Oil and Its Emulsion

Saskia M. van Ruth[a,*], Jacques P. Roozen[a], Maarten A. Posthumus[a], and Frans Jos H.M. Jansen[b]

[a]Wageningen Agricultural University, Department of Food Technology and Nutritional Sciences, 6700 EV Wageningen, The Netherlands, and [b]Unilever Research Vlaardingen, 3130 AC Vlaardingen, The Netherlands

ABSTRACT: The formation of conjugated diene hydroperoxides and hexanal was compared to the development of aroma profiles during initial lipid oxidation of a vegetable oil and its 40% oil-in-water emulsion at 60°C. The aroma profiles of the oil and the emulsion with and without addition of ascorbic acid or ascorbyl palmitate were compared. The aroma compounds were isolated under a model mouth system and analyzed by gas chromatography/sniffing port analysis. Detectable odors were found and corresponded to 11 and 14 volatile compounds in the oil and the emulsion, respectively. The emulsion had higher lipid oxidation rates than the oil. Addition of ascorbic acid and ascorbyl palmitate had little influence on the aroma composition of the oil. In the emulsion, addition of these compounds resulted in diminished generation of odor active compounds. Results of measurements of conjugated diene hydroperoxides and headspace hexanal corresponded to that of the lipid oxidation rate in general, but predicted insufficiently the alterations in the aroma compositions by antioxidants.

Paper no. J9122 in *JAOCS 76,* 1375–1381 (November 1999).

KEY WORDS: Antioxidant, aroma, ascorbic acid, ascorbyl palmitate, emulsion, lipid oxidation, oil, volatile compounds.

Lipid oxidation plays a major role in the quality of lipid-containing foods (1). During lipid oxidation, unsaturated fatty acids react with molecular oxygen *via* a free-radical chain mechanism to form fatty acid hydroperoxides (2). Although these products are colorless, tasteless, and odorless, they degrade to form low-molecular-weight compounds with distinctive odors (3).

Understanding of the factors that affect lipid oxidation in systems in which the fat is dispersed as emulsion droplets is still fairly poor. This lack of understanding is surprising when one considers the large number of foods consisting of emulsions. However, there are considerable differences in lipid oxidation between bulk oil and emulsified fats (4).

Lipids influence flavor through their effects on aroma generation and aroma release. Aroma release depends on the volatilities of the compounds and on mass transfer, both of which are influenced by the matrix composition (5). The volatilities of compounds depend on interactions between the volatile compounds and the nonvolatile components in a food, e.g., presence of lipids in the matrix (6). Lipophilic volatile compounds tend to be more odorous in aqueous rather than in lipid media, thus they have higher odor thresholds in vegetable oils than in water solutions (7).

The most commonly used method of retarding lipid oxidation is by the addition of antioxidants. The effectiveness of antioxidants was extensively studied in bulk oils. However, the effectiveness of an antioxidant in an emulsion may be considerably different from that of the same antioxidant in a bulk oil (8,9). Predominantly apolar antioxidants were reported to be more effective in an oil-in-water emulsion than in bulk oil, whereas the opposite was observed for polar antioxidants (9).

Although antioxidants were studied in bulk oils, different substrates, conditions, and methods used to determine lipid oxidation have led to results that are significantly different and sometimes contradictory. The determination of primary products (conjugated diene hydroperoxides) and volatile secondary oxidation products (hexanal) are two commonly used methods to assess lipid oxidation. However, it is uncertain to what extent these methods are related to aromas released in the mouth. In this study, the influence of ascorbic acid and ascorbyl palmitate and the changes in aroma composition of a vegetable oil and its oil-in-water emulsion are studied during initial lipid oxidation at 60°C. Aroma profiles are compared with the formation of conjugated diene hydroperoxides and the formation of hexanal.

MATERIALS AND METHODS

Sample materials. A vegetable oil and its 40% oil-in-water emulsion (40% oil, 59% deionized water, 1% Tween 60) were supplied by Unilever Research Vlaardingen (Vlaardingen, The Netherlands). The fatty acid methyl ester composition of the oil determined by gas chromatography (GC) was 5.8% 16:0, 4.2% 18:0, 22.9% 18:1, 56.8% 18:2, and 8.5% 18:3. The

*To whom correspondence should be addressed at University College Cork, Department of Food Science and Technology, Nutritional Sciences, Western Road, Cork, Ireland. E-mail: s.vanruth@ucc.ie

oil contained 610 mg α-, 22 mg β-, and 73 mg γ-tocopherol/kg oil (10). The emulsion was prepared using a homogenizer (APV Gaulin model LAB 40-10 RBFI, APV Gaulin GmbH, Lübeck, Germany) at 150 bar for 10 min. For antioxidant experiments, ascorbic acid (AA) and ascorbyl palmitate (AP) were added to the oil and the emulsion (250 µmol/L oil). AP was added to the oil before emulsion preparation, AA afterward. The average particle size in the emulsions was 1.0 µm (Coulter Laser measurements) and stable during storage at 60°C for 8 d. Duplicate oil and emulsion samples (65 mL) were stored in glass jars (350 mL) in the dark at 60°C for each day of analysis. Samples were collected at days 0, 4, 8, 12, and 16 for the oil, and at days 0, 4, and 8 for the emulsion. The emulsion was stored for a shorter time because of changes in particle size distribution after 8 d of storage.

Analysis of conjugated diene hydroperoxides (CDH). CDH were measured in the oils and in the oil extracted from the emulsions for each of the samples stored in duplicate by a modified version of the method described by Frankel *et al.* (9). For the extraction, 5 g of emulsion was added to 25 mL of methanol. After 15 min the methanol–water layer was removed, and the remaining oil was used for analysis of the dienes. An aliquot of pure or extracted oil was dissolved in 5 mL cyclohexane in duplicate and the absorbance measured at 234 nm (CECIL 2020; Cecil Instruments Ltd., Cambridge, United Kingdom). Results were calculated as hydroperoxides in mmoles per kilogram oil, using a molar absorptivity of 26,000 for linoleate hydroperoxides (11).

Analysis of hexanal: static headspace analysis. For static headspace gas chromatography (SHGC), a modification of a previously described method was used (12). Two milliliters of oil or emulsion was transferred into a 10-mL vial and incubated at 60°C for 10 min in the headspace unit of a Carlo Erba MEGA 5300 GC (Interscience bv, Breda, The Netherlands). The GC was equipped with a DB-Wax column (J&W Scientific, Folsom, CA), 30 m length, 0.53 mm i.d., 1 µm film thickness and a flame-ionization detector (FID) at 275°C. An initial oven temperature of 60°C for 5 min was used, followed by a rate of 3°C min^{-1} to 110°C and then by 4°C min^{-1} to 170°C. Each stored sample was analyzed in duplicate vials. Peak areas were standardized with known concentrations of hexanal in oil and emulsion samples. Results were calculated as hexanal in mmoles per kilogram oil. In the relative hexanal concentration experiments, data of the storage experiments were used, and the proportion of headspace hexanal was calculated as percentage of headspace propanal, pentanal, and hexanal. These three compounds accounted for 95% of the total peak area and possessed detectable odors in preliminary gas chromatography/sniffing port analysis (GC/SP).

Isolation of volatile compounds under mouth conditions. The model mouth system was described in detail previously (13). Artificial saliva (4 mL) was transferred to the sample flask (70 mL) of the model mouth system, which was kept at 37°C and a sample of oil or emulsion (4 mL) was added. The headspace was flushed with purified nitrogen gas (100 mL min^{-1}) for 2 min to trap the volatile compounds in 0.1 g Tenax TA (diameter 0.25–0.42 mm, Alltech Nederland bv, Zwijndrecht, The Netherlands), positioned in a glass tube, 100 mm long and 3 mm i.d.. During the isolation of the volatiles, a plunger made up-and-down screwing movements in order to mimic mastication. For the artificial saliva, a stock solution was prepared and subsequently diluted to single strength. This stock saliva consisted of mucin (2.160 g), potassium phosphate dibasic trihydrate (1.369 g), sodium chloride (0.877 g), calcium chloride dihydrate (0.477 g), and sodium nitrate (0.500 g) in 100 mL distilled water. The stock saliva was purged with purified nitrogen gas (50 mL min^{-1}) for 15 h in order to diminish volatile compounds in the saliva. After purging, sodium bicarbonate (0.521 g) and 20,000 units porcine α-amylase were added to 10 mL stock saliva and diluted 1:9 with distilled water. The single-strength saliva was adjusted to pH 7 with 2 M HCl.

GC/SP analysis. In GC//SP, desorption of volatile compounds from Tenax was performed by a thermal desorption (245°C, 5 min)/cold trap (–120°C/260°C) device (Carlo Erba TDAS 5000; Interscience bv, Breda, The Netherlands). GC was carried out on a Carlo Erba MEGA 5300 (Interscience bv, Breda, The Netherlands) equipped with a Supelcowax 10 capillary column, 60 m length, 0.25 mm i.d., film thickness 0.25 µm, and a FID at 275°C. At the end of the column the effluent was split 1:2:2 for FID, sniffing port 1, and sniffing port 2, respectively. An initial oven temperature of 40°C was used, followed by a rate increase of 2°C min^{-1} to 92°C and then by 6°C min^{-1} to 272°C. Ten assessors were selected on the basis of their sensitivity, memory, ability to recognize odors, and availability. Prior to sniffing the effluent of the oil and emulsion samples, the assessors were trained on the technique of sniffing. Assessors used portable computers with a program in Pascal for data collection. The data were converted from the field disks into Lotus 123 software in order to process the raw data. Aroma descriptors were generated during preliminary GC/sniffing experiments and clustered after group sessions of the panel. This resulted in a list of 19 descriptors (green, mushroom, spicy, fruity, sweet, flowers, fatty, oil, rancid, rotten, musty, chemical, nuts, almond, burned, caramel, chocolate, vanilla, sharp/irritating). These descriptors and "other/I do not know" were used for each compound detected by the assessors at the sniffing port. Tenax tubes without adsorbed volatile compounds were used as dummy samples for determining the signal-to-noise level of the group of assessors. During a GC/SP analysis two assessors were sniffing and the FID response was recorded simultaneously.

GC/mass spectrometry (MS) analysis. Volatile compounds were isolated as described under GC/SP and identified by combined GC (Varian 3400; Varian, Walnut Creek, CA) and MS (Finnigan MAT 95; Finnigan MAT, Bremen, Germany) equipped with a thermal desorption/cold trap device (TCT injector 16200; Chrompack bv, Middelburg, The Netherlands). The capillary column and the oven temperature program were the same as those used in GC/SP analyses. Mass spectra were obtained with 70 eV electron impact ionization, while the

mass spectrometer was continuously scanning from *m/z* 24 to 400 at a scan speed of 0.7 s/decade (cycle time 1.05 s).

Statistical analyses. Analysis of variance (ANOVA) was used to determine significant differences between the means of quadruple analysis of hydroperoxides, hexanal, and aroma compounds released in the model mouth (FID data). If significant differences were found, Fisher's Least Significant Difference tests (LSD) were performed (14). The GC/SP data were subjected to Friedman two-factor ranked analysis of variance (15) and correlation coefficients were calculated for the data obtained by the various analysis techniques. Significance level is $P < 0.05$ throughout the study.

RESULTS AND DISCUSSION

Conjugated diene and hexanal concentrations: the effect of antioxidants. Initial lipid oxidation of the oil and the emulsion was studied by following the formation of conjugated diene hydroperoxides and the formation of hexanal in the oil and the emulsion (Table 1). The oil demonstrated a gradual increase for the CDH and hexanal. Generally, the oil showed higher hydroperoxide values and lower hexanal peak areas than the emulsion. The CDH degraded at a lower rate in the oil. Storage significantly influenced the conjugated diene and hexanal concentrations in both the oil and emulsion ($P < 0.05$). When all measurements were taken into account, the addition of AA or AP had no significant effect on the formation of primary products in the oil and emulsion and on the formation of secondary products in the oil. An overall significant effect of the antioxidants on the hexanal concentration in the emulsion was determined ($P < 0.05$). Lowest concentrations were observed for the emulsion with added AP, followed by the emulsion with added AA and the control, respectively. Very low correlation was determined between the CDH and hexanal data ($r^2 = 0.0327$).

Information on the concentrations of various secondary lipid oxidation products in the oil phase of a product is interesting for following the formation of compounds. However, data on headspace concentrations are more valuable with regard to flavor deterioration as they are likely to correlate better with flavor perception than volatile concentrations in the oil phase. Hexanal concentrations in product and vapor phase are often used as a marker for secondary lipid oxidation products and as a measure for flavor deterioration. When the formation of one compound is considered to be representative for the aroma composition of a product, the proportions of compounds are assumed to be relatively constant and volatile composition is considered to change in quantity only. The relative headspace concentration of hexanal was measured for the oil and emulsion, with and without added antioxidants during lipid oxidation (Fig. 1). Although the addition of antioxidants had little influence on the proportion of hexanal, a considerable difference between the oil and the emulsion was observed. Proportionally, more hexanal was measured in the emulsion than in the oil. The proportion of hexanal increased during storage for both the oil and emulsion. These results indicate that the use of one compound only, as a measure of generated aroma compounds, has limited value as an indicator for flavor deterioration by lipid oxidation.

Odor active compounds in the oil and the effect of antioxidants. In order to determine the compounds responsible for the flavor deterioration in the oil and emulsion as perceived during eating, volatile compounds released in a model mouth system were analyzed by GC/SP. The odor active compounds released from the oil and emulsion, with and without added AA or AP, were identified by GC/MS and further characterized by their retention times and the odors described by the assessors at the sniffing port. GC sniffing of dummy samples revealed that detection of an odor at the sniffing port by two or fewer out of 10 assessors could be considered as noise. An example of a chromatogram generated by means of oil sniffing is presented in Figure 2A. GC/SP demonstrated that 11

TABLE 1
Effect of Addition of Antioxidants on Conjugated Diene and Hexanal Concentrations[a]

	Oil			Emulsion		
	Control	AA	AP	Control	AA	AP
CD (mmol/kg oil)						
Day 0	7.7	7.7	7.3	8.0	8.2	7.2
Day 4	25.4	24.6	18.6	27.4	15.4	19.8
Day 8	41.7	43.2	45.5	35.7	34.6	32.5
Day 12	57.4	56.4	57.1			
Day 16	63.1	67.2	70.0			
Hexanal (mmol/kg oil)						
Day 0	0.03	0.03	0.03	0.11	0.14	0.07
Day 4	0.55	0.18	0.14	64.17	6.61	4.63
Day 8	1.02	0.72	0.61	274.34	217.38	131.79
Day 12	1.48	1.64	1.38			
Day 16	2.16	2.50	2.83			

[a]As measured in a vegetable oil and its 40% oil-in-water emulsion. AA, ascorbic acid; AP, ascorbyl palmitate; CD, conjugated diene.

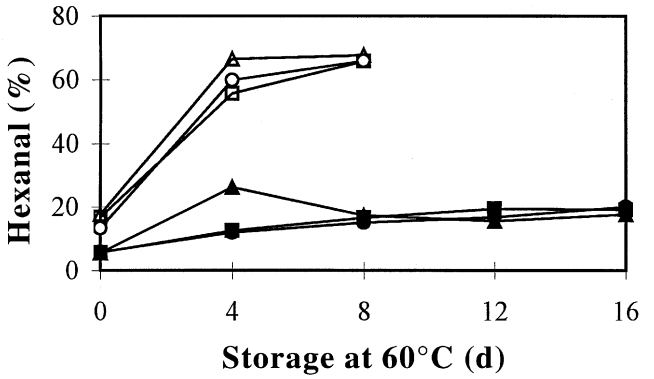

FIG. 1. Proportion of hexanal in the headspace of a vegetable oil (Oil) and its 40% oil-in-water emulsion (Emuls), without (Contr) and with added ascorbic acid (AA) or ascorbyl palmitate (AP) ($n = 4$). ▲, Oil-control; ■, Oil-AA; ●, Oil-AP; △, Emuls-Contr; □, Emuls-AA; ○, Emuls-AP.

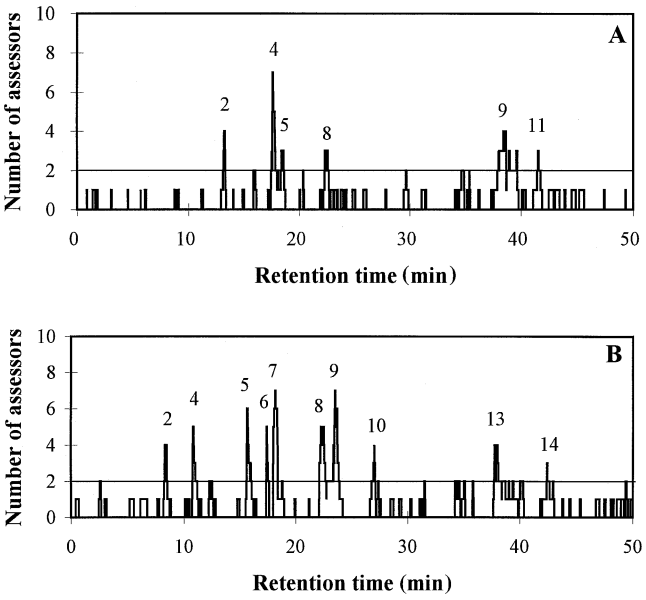

FIG. 2. Sniffing chromatogram of the volatile compounds of a control vegetable oil after 16 d of storage (A) and its 40% oil-in-water emulsion after 8 d of storage (B). Numbers in chromatograms A and B refer to compounds in Tables 2 and 4, respectively.

volatile compounds isolated from the oil possessed odors (Table 2). Although assessors described odors differently, mutual descriptors were green and chemical. 1-Penten-3-one and hexanal were most frequently detected, while *trans*-3-butenal and 3-octen-2-one were the least detected. The cumulated number of assessors perceiving odors over all compounds (Table 2) is a measure for overall aroma intensity and shows a gradual but slow increase during storage. The sniffing data and the FID data (Table 3) did not show significant differences between the control, AA, and AP ($P < 0.05$), although storage had a significant effect.

Odor active compounds in the emulsion: comparison with the oil. Fourteen odor active compounds were determined in the emulsion system (Table 4). An example of a sniffing chromatogram of the volatile compounds of the emulsion is shown in Figure 2B. Among these compounds, seven were the same as in the oil samples. These include pentanal, 1-penten-3-one, *cis*-2-butenal, hexanal, 1-octen-3-one, *trans*-2-heptenal, and 3-octen-2-one. The compounds pentanal, *cis*-2-butenal, and hexanal were most frequently perceived. Generally, compounds were more frequently perceived in the emulsions than in the oils as indicated by the total number of perceptions (Tables 2 and 4). FID peak areas of the odor active compounds showed significant differences between the oil (Table 3) and the emulsion (Table 5). Differences in lipid oxidation between oils and their oil-in-water emulsions were reported previously (9,15) and might be explained on the basis that the physical state of the lipid system has an effect on oxidative stability of lipid systems (9). In addition, emulsifiers have been reported to affect lipid oxidation and antioxidant activity through their presence at the oil/water interface (16,17).

Odor active compounds in the emulsion: effect of antioxidants. Several odor active compounds were determined in common in the emulsion samples during storage. However, some remarkable differences in the sniffing chromatograms were shown. Fewer numbers of assessors perceived 1-penten-3-one and 3-octen-2-one in the samples containing AA compared to the control sample after 8 d of storage. In addition to the latter two compounds, 3-pentanol was perceived by a remarkably lower number of assessors in the samples with added AP. Nevertheless, these three compounds were present in all three samples, as was shown by the FID data (Table 5). Lower concentrations, probably near threshold values, contributed to the observed lower numbers of perceptions for these compounds. The FID data showed significant differ-

TABLE 2
Number of Assessors Perceiving Odor Active Compounds in GC/Sniffing Port Analysis[a]

		Control			AA		AP	
Compound	Odor descriptor[b]	Day 0	Day 4	Day 16	Day 4	Day 16	Day 4	Day 16
1. Unk	CSFUH?	—[c]	3	—	4	—	—	—
2. 2-Propanol	C?SOXEU	—	—	5	—	7	4	4
3. Pentanal	XTR	—	3	—	—	4	—	4
4. 1-Penten-3-one	COTGUN?LAI	—	5	7	6	8	—	—
5. *Cis*-2-butenal	COUFRI	—	—	4	—	3	—	6
6. *Trans*-2-butenal	GWOC	—	—	—	—	—	—	4
7. 2,3-Pentanedione	RSTUN	—	—	3	—	—	—	4
8. Hexanal	GLFXTE	5	3	5	5	5	6	3
9. 1-Octen-3-one + *trans*-2-heptenal[d]	MUWRCBX?TNAI	8	—	7	3	3	4	7
10. Unk	MCEUA	3	—	—	4	—	—	3
11. 3-Octen-2-one	FUCABI	—	—	3	—	—	3	—
Total number of perceptions		16	14	34	22	30	17	35

[a]Compounds released in a model mouth system from a vegetable oil with and without added antioxidants after storage at 60°C for various times.
[b]Descriptors in order of decreasing frequency: G (green), M (mushroom), S (spicy), F (fruity), W (sweet), L (flowers), T (fatty), O (oil), R (rancid), E (rotten), U (musty), C (chemical), N (nuts), A (almond), B (burned), X (caramel), H (chocolate), V (vanilla), I (sharp/irritating), and ?(other/I do not know).
[c]At or below noise level.
[d]Compounds eluted simultaneously. GC, gas chromatography; for other abbreviations see Table 1.

TABLE 3
FID Peak Areas of Odor Active Compounds Released in a Model Mouth System from a Vegetable Oil

	Control			AA		AP	
Compound[a]	Day 0	Day 4	Day 16	Day 4	Day 16	Day 4	Day 16
1. Unk	79	17	220	28	24	17	45
2. 2-Propanol	696	585	778	601	342	795	575
3. Pentanal	16	14	84	60	111	61	173
4. 1-Penten-3-one	1	7	57	38	27	9	58
5. *Cis*-2-butenal	7	4	150	57	55	3	140
6. *Trans*-2-butenal	——	—	—	4	—		19
7. 2,3-Pentanedione	—	3	12	5	8	4	15
8. Hexanal	215	121	519	813	1376	855	1699
9. 1-Octen-3-one + *trans*-2-heptenal	3	25	412	52	429	45	630
11. 3-Octen-2-one	—	2	11	26	26	26	41

[a]Compounds were isolated from a vegetable oil with and without added AA and AP after storage at 60°C for various times.
[b]The peak areas of the unknown active odor compound 10 were below detection level. FID, flame-ionization detector; peak areas in units of mV s^{-1}; for other abbreviations see Tables 1 and 2.

TABLE 4
Number of Assessors Perceiving Odor Active Compounds in GC/Sniffing Port Analysis

		Control			AA		AP	
Compound	Odor descriptor[a]	Day 0	Day 4	Day 8	Day 4	Day 8	Day 4	Day 8
1. Heptane	FGSWLB	—	4	—	3	—	—	—
2. Propanal	C?FLGWTRN	—	—	7	—	6	—	8
3. Acetone	FWX	—	—	—	3	—	—	—
4. Butanal	HTORWLECXI	—	—	6	—	5	—	6
5. Pentanal	GWTESFLRUN XHIMOCA?	—	7	8	—	8	6	10
6. 1-Penten-3-one	CUWLTONX?	—	3	6	4	3	—	3
7. *Cis*-2-butenal	COTGRUBXI?	—	8	7	6	8	6	7
8. Hexanal	GLFTU?	—	9	5	5	7	6	9
9. 3-Pentanol	GCOFLTUMWRI	—	6	9	5	8	—	4
10. 1-Penten-3-ol	?FSLGC	—	—	4	—	6	—	3
11. Unk	WTH		—	—	—	—	—	3
12. 1-Pentanol	EMLTUV	—	—	—	—	4	3	—
13. 1-Octen-3-one + *trans*-2-heptenal[b]	MUTC?WRNABXH	—	7	5	5	4	5	4
14. 3-Octen-2-one	RWT	—	—	3	—	—	—	—
Total number of perceptions		0	44	60	31	59	26	57

[a]Odor active compounds released in a model mouth system from a 40% vegetable oil-in-water emulsion with and without added antioxidants after storage at 60°C for various times.
[b]Compounds eluted simulatenously. For abbreviations see Tables 1, 2, and 4.

TABLE 5
FID Peak Areas of Odor Active Compounds Released in a Model Mouth System

	Control			AA		AP	
Compound[b]	Day 0	Day 4	Day 8	Day 4	Day 8	Day 4	Day 8
1. Heptane	6	66	274	21	143	11	87
2. Propanal	10	484	2221	91	1260	25	801
3. Acetone	8	153	264	19	221	28	226
4. Butanal	4	45	354	5	193	5	107
5. Pentanal	10	835	5361	128	3110	54	1725
6. 1-Penten-3-one	2	114	193	17	118	7	49
7. *Cis*-2-butenal	5	77	275	14	194	3	101
8. Hexanal	62	5111	16203	1096	14491	526	9734
9. 1-Penten-3-ol	6	154	534	19	389	9	221
10. 1-Pentanol	2	178	1629	49	1056	18	580
13. 1-Octen-3-one + *trans*-2-heptenal	1	35	160	28	64	8	68
14. 3-Octen-2-one	3	20	213	11	79	4	106

[a]As measured in a vegetable oil-in-water emulsion with and without added AA and AP after storage at 60°C for various times; peak areas in units of mV s^{-1}.
[b]The peak areas of the unknown odor active compound (11) and 3-pentanol (12) were below detection level. For abbreviations see Tables 1 and 3.

ences between the treatments with the highest values for the control, followed by the emulsion with added AA and AP, respectively.

Volatile compounds were formed at lower rates during storage in the emulsion containing AA and AP. Besides the direct interactions with hydroperoxyl radicals, AA and AP can show interactions with natural tocopherols in the samples. Tocopherols may compete with lipid substrates by donating their hydrogen atoms to alkoxyl radicals to form more stable alcohols and tocopheroxyl radicals. Also alkoxy radicals can be trapped by tocopheroxyl radicals (18). AA and AP can regenerate tocopherols and therefore extend their antioxidant activity (19), which is indicated by significantly lower conjugated diene values in the AA and AP emulsion systems after 4 d of storage ($P < 0.05$). Frankel *et al.* (9) reported an α-tocopherol–ascorbyl palmitate mixture to be more effective than an α-tocopherol–ascorbic acid mixture in a 10% oil-in-water corn oil emulsion. AP (apolar) retarded lipid oxidation in the emulsion more effectively than in the oil, which is in agreement with the polar paradox. The effectiveness of an antioxidant under the "polar paradox" follows the rule that nonpolar antioxidants function better in polar lipid emulsions, whereas polar antioxidants are relatively more effective in lipid (nonpolar) systems (18). In the present study, no effect was observed in the oil treated with AA and AP during initial lipid oxidation. The hydroperoxides were rather stable in the control oil, which might be due to sufficient concentrations of natural tocopherols.

Comparison of conjugated diene concentrations, hexanal concentrations, and composition of odor active compounds. To compare the various methods used in the present work, the data were correlated with GC/SP data of the aroma compounds released in the model mouth (cumulated number of assessors). The latter method showed high correlation with sensory analysis (20–22), which is the ultimate test for flavor deterioration. Correlation coefficients include: conjugated diene concentrations ($r^2 = 0.349$), hexanal concentrations in oil ($r^2 = 0.833$), aroma compounds released in a model mouth: cumulated GC/FID peak area ($r^2 = 0.854$), aroma compounds released in a model mouth: log transformed cumulated GC/FID peak area ($r^2 = 0.951$). These results indicate that the conjugated diene concentrations were not related to flavor changes. Measurements of volatile compounds showed sufficient relationships ($r^2 > 0.8$), however, the proportion of compounds in the aroma may differ among products and may change during storage as shown in the present work. This indicates the limited value of one volatile compound as an indicator for flavor deterioration.

ACKNOWLEDGMENT

The authors wish to thank Jack Seijen ten Hoorn (Unilever Research Vlaardingen) for preparation of the emulsion.

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[Received January 15, 1999; accepted August 12, 1999]