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Headspace-solid phase microextraction (HS-SPME) analysis of oxidized volatiles from free fatty acids (FFA) and application for measuring hydrogen donating antioxidant activity

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Abstract

Volatile compounds from thermally oxidized free fatty acids (FFA) at 93 °C for 200 min were analyzed by headspace-solid phase microextraction (HS-SPME)-gas chromatography (GC). Commercially available mixtures of FFA were used instead of selecting specific vegetable oils with various fatty acid compositions. As oxidation time increased, total volatiles and some individual volatiles including hexanal, 2-hexenal, 2-heptenal, 2,4-heptadienal, 2-octenal, and 2,4-decadienal increased linearly with 0.99 coefficient of determination (R^2) at specific oxidation time against conjugated dienoic acid (CDA) or *p*-anisidine value (*p*-AV). Total volatiles showed the highest linearity (R^2) of 0.99 and 2-heptenal showed the highest increasing slope in peak areas for 200 min oxidation. Not all oxidized volatiles increased linearly during oxidation. Availability of HS-SPME method for determining hydrogen donating antioxidant activity was tested using FFA containing serially diluted butylated hydroxytoluene (BHT) at 93 °C for 60 min. 2-Heptenal and total volatiles showed higher linearity against BHT concentration than hexanal. HS-SPME could be a useful method to determine the hydrogen donating antioxidant activity from FFA using total volatiles or 2-heptenal as oxidation markers.

Keywords: HS-SPME; Oxidized volatiles; Conjugated dienoic acid; *p*-Anisidine value; Antioxidant activity measuring method

1. Introduction

Oxidized lipids can accelerate the deterioration of sensory quality in foods and decrease the consumer acceptances. Lipid oxidation is influenced by many factors including antioxidants, metals, photosensitizers, oxygen contents, intensity of heat and light energy, and number of double bonds in fatty acids (Channon & Trout, 2002; Min, 1998; Min & Ahn, 2005). Some reactive oxygen species such as hydroxyl radical (·OH) and singlet oxygen ($^1O_2$) are related to lipid oxidation directly or indirectly and reported to be associated with aging and disease process (Channon & Trout, 2002; Choe & Min, 2005). Therefore, finding out effective antioxidants and determining their antioxidant activity are challenging and important research areas in foods.

Many methods determining antioxidant activity have been developed depending on the mechanisms of action of antioxidants. Total peroxyl radical trapping parameter, oxygen radical absorbance capacity, trolox equivalent antioxidant capacity, and 2,2-diphenyl-1-hydrazyl radicals have been used for measuring free radical scavenging activity (Choi, Ku, Chang, & Lee, 2005; Gliszczynska-Swiglo, 2006; Tsai, Hsu, Kong, Lin, & Lu, 2000; Yilmaz & Toledo, 2006). Conjugated dienoic acid (CDA), *p*-anisidine value (*p*-AV), hydroperoxides, total carbonyls, and thiobarbituric acid reactive substances (TBARS) are typical parameters determining antioxidant activity on the inhibition of lipid peroxidation in foods and model systems (Alamed, Julian, & Decker, 2006; Beltran, Pla, Yuste, & MoroMur, 2003;

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Estevez & Cava, 2006; Juntachote, Berghofer, Siebenhandl, & Bauer, 2006; Rehman, Habib, & Shah, 2004). One of the parameters indicating the degree of oxidation is oxidized volatile compounds. Concentration of volatile markers such as hexanal has been monitored for determining the degree of oxidation in foods such as potato chips or pork (Fernando, Berg, & Grün, 2003; Sanches-Silva, Rodríguez-Bernaldo de Quirós, López-Hernández, & Paseiro-Losada, 2004).

Headspace-solid phase microextraction (HS-SPME) is a rapid, solvent-free, and simple method for volatile analysis and has been widely applied to many types of foods including fruit juices (Foley et al., 2002), fermented foods (Lee, Diono, Kim, & Min, 2003; Lee, Kang, & Min, 2003), and dairy products (Kim, Lee, & Min, 2003; Lee et al., 2003). Also, HS-SPME has been used to analyze volatiles in vegetable oils including corn, soybean, olive, sunflower, and rapeseed oils (Beltran, Aguilera, & Gordon, 2005; Jelen, Obuchowska, Zawirska-Wojtasiak, & Wasowicz, 2000; Kalua, Bedgood, & Prenzler, 2006; Steenson, Lee, & Min, 2002).

If volatile compounds can be used as markers for determining the degree of lipid oxidation, they could be applied for measuring antioxidant activity. To be selected as useful parameters for measuring antioxidant activity, oxidized volatiles should have high correlation with the degree of oxidation and be detected in high amounts. However, limited reports are available in the literature on the oxidized volatiles from the mixtures of FFA using HS-SPME.

The objectives of this study were to monitor the profile changes of volatile compounds from thermally oxidized FFA by HS-SPME and to test the availability of HS-SPME for measuring hydrogen donating antioxidant activity using butylated hydroxytoluene (BHT).

2. Materials and methods

2.1. Materials

Boron trifluoride (BF_3), hexanal, 2-heptenal, 2-octenal, 2-pentylfuran, and other standard volatile compounds were purchased from Sigma–Aldrich Inc. (St. Louis, MO, USA). Commercially available mixtures of FFA were purchased from Sigma–Aldrich Inc. (catalogue number L1626-500ML). Mixtures of FFA were composed of linoleic (57.46%), oleic (29.89%), linolenic (5.57%), palmitic (3.17%), stearic (1.69%), and arachidic (1.68%) acids from preliminary study. Butylated hydroxytoluene (BHT) and isooctane were purchased from Junsei Chemical Co. (Tokyo, Japan). *p*-Anisidine was purchased from Kanto Chemical Co. (Tokyo, Japan). Teflon-coated rubber septa, aluminum caps, serum bottles, 65 μm polydimethylsiloxane/divinylbenzene (PDMS/DVB), glass liners, and the fiber assembly holder were purchased from Supelco, Inc. (Bellefonte, PA, USA).

2.2. Sample preparation

Mixtures of FFA (0.40 g) were transferred into a 10 mL serum bottle and sealed air-tight with at teflon-coated rubber septum. Sample bottles were put in a dry oven (Win Science Co., Seoul, Republic of Korea) at 93 °C for 200 min, and a sample was taken every 20 min. The temperature of 93 °C was selected for accelerating the conditions of autoxidation. From each sample bottle, volatile compounds were analyzed by HS-SPME under air-tight condition and then CDA and *p*-AV were determined after the aluminum cap was removed from the bottle. One sample bottle was prepared for each sampling time and the entire experiment was repeated three times.

Availability of HS-SPME method for determining antioxidant activity was tested using FFA with serially diluted butylated hydroxytoluene (BHT), a well-known hydrogen donating antioxidant. BHT was mixed with FFA to make 1 000 ppm (w/w) and serially diluted to 0, 25, 50, and 100 ppm BHT in air-tight 10 mL bottles, respectively. Sample bottles were put in a dry oven at 93 °C for 60 min and volatile compounds were determined. The entire experiment was repeated four times.

2.3. CDA and p-AV analysis

CDA measures the conjugated dienes formed from polyunsaturated fatty acids such as linoleic or linolenic acids during lipid oxidation. CDA of samples was measured according to the AOCS (1980) method Cd 18-90. One hundred milligrams of sample were dissolved in 25 mL isooctane. The mixture was diluted with tenfold isooctane (v/v) and absorbance at 233 nm was read using a UV–VIS spectrophotometer (Model UV-1650PC, Shimadzu, Kyoto, Japan).

p-AV measures secondary oxidation products, such as 2-alkenal and 2,4-alkadienal. *p*-AV of samples was determined according to the AOCS (1990) method Ti la-64. The sample (100 mg) was dissolved in 25 mL isooctane and the absorbance of this mixture was read at 350 nm using a UV–VIS Spectrophotometer. Five milliliters of the mixture was vigorously mixed with 1 mL 0.25% *p*-anisidine in acetic acid (w/v) and the absorbance at 350 nm was measured.

2.4. Analysis of volatile compounds by HS-SPME

Samples taken from the dry oven were put in the dark at room temperature for 15 min for the equilibrium of volatiles in the headspace of bottles. The volatile compounds in samples were isolated using a 65 μm PDMS/DVB solid phase at 30 °C for 30 min in a water bath. The isolated volatile compounds were determined using a gas chromatograph equipped with a flame ionization detector (Shimadzu GC-17 A, Kyoto, Japan).

2.5. Gas chromatography condition for volatile compound analysis

A Shimadzu GC-17A gas chromatograph was equipped with a 0.75 mm ID glass injection liner, a flame

ionization detector, and a 30 m × 0.32 mm ID, 0.25 μm film, DB-5 column (Agilent J & W Scientific, Folsom, CA, USA). The oven temperature of GC was programmed starting at 40 °C for 2 min and increasing from 40 to 160 °C at 6 °C/min and from 160 to 220°C at 10 °C/min and holding for 3 min. The temperatures of injector and detector were 250 and 300 °C, respectively. Nitrogen gas was used as carrier gas with 1.0 mL/min flow rate. Splitless mode was used and the isolated volatile compounds in solid phase of SPME were desorbed at 250 °C for 2 min in a GC injector.

2.6. Identification of volatile compounds

A Hewlett-Packard 5890 GC-5971 A mass selective detector (MS) (Agilent Technology 5973, Palo alto, CA, USA) equipped with a Hewlett-Packard 59822 B ionization gauge controller was used. All mass spectra were obtained at 70 eV and 220 °C ion source temperature. The identification of compounds was made by the combination of NIST Mass Spectra and gas chromatographic retention times of standard compounds. Volatile compounds without standard compounds were tentatively identified using GC–MS spectra only. Helium carrier gas at 1.0 mL/min and a 30 m × 0.25 mm i.d., 0.25 μm film thickness, DB-5 column (Agilent J & W Scientific, Folsom, CA, USA) were used. The oven conditions for GC–MS were the same as the gas chromatographic analysis conditions described previously.

2.7. Statistical analysis

Results were expressed as mean ± standard deviation. Coefficient of determination (R^2) or linearity, of data was statistically analyzed using a commercially available software package, SPSS software program (SPSS Inc., Chicago, IL, USA) and Microsoft Excel program.

3. Results and discussion

3.1. CDA and p-AV analysis from thermally oxidized FFA

CDA and *p*-AV of thermally oxidized FFA at 93 °C for 200 min are shown in Fig. 1. CDA increased linearly from 0 to 120 min and was not changed significantly after 120 min while *p*-AV increased linearly from 40 to 180 min. Coefficient of determination (R^2) or linearity, between CDA and oxidation time was 0.99 at the range of 0–80 min and that of *p*-AV was 0.99 from 40 to 180 min oxidation time, suggesting that CDA and *p*-AV are valid parameters for determining the primary and secondary oxidation of lipids, respectively. CDA and *p*-AV have been used for determining the degree of oxidation in foods including pork and vegetable oils (Juntachote et al., 2006; Kim & Choe, 2005). In this study, CDA and *p*-AV were chosen as reference parameters to compare the availability of oxidized volatiles.

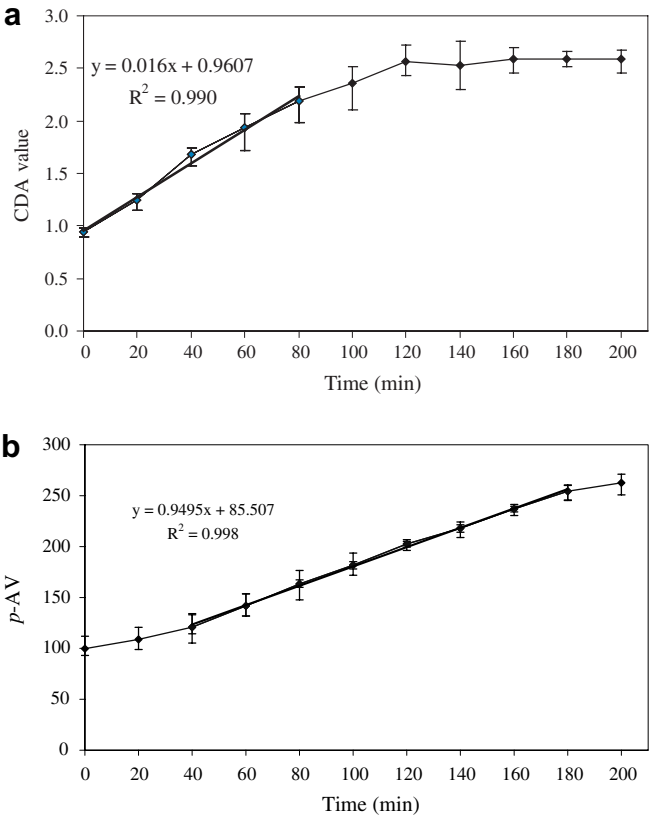

Fig. 1. Changes of CDA (a) and *p*-AV (b) in thermally oxidized FFA at 93 °C ($n = 9$).

3.2. Changes of volatiles from thermally oxidized FFA

Typical HS-SPME-GC chromatogram for volatiles from thermally oxidized FFA at 93 °C is shown in Fig. 2. The coefficient of variation (CV) of peak areas of total volatiles using PDMS/DVB solid phase was 8.7%. It has been reported that reproducibility of HS-SPME analysis for volatile compounds from oils was dependent on the experimental conditions. Steenson et al. (2002) reported that CV of commercially available SPME solid phases was in the range from 3.2% to 10.7% for spiked hexanal in soybean oil. Beltran et al. (2005) showed that CV for total volatiles from oxidized sunflower oil by HS-SPME was 6.1% in oil-in-water emulsion. Jelen et al. (2000) reported that 0.78–5.92% CV was achieved by HS-SPME for added typical standard volatiles in freshly refined rapeseed oil at 10 mg/L concentration.

Changes of total volatiles and some major volatiles including pentane, hexanal, 2-hexenal, 2-heptenal, 2,4-heptadienal, 2-octenal, and 2,4-decadienal are shown in Fig. 3. Peak areas of total volatiles increased linearly throughout oxidation time. Two major oxidized volatiles from FFA were 2-heptenal and hexanal. Hexanal is a typical oxidation volatile from linoleic acid at 13-hydroperoxide and has been commonly monitored for measuring lipid oxidation in foods (Beltran et al., 2005; Fernando et al., 2003; Mielnik, Olsen, Vogt, Adeline, & Skrede,

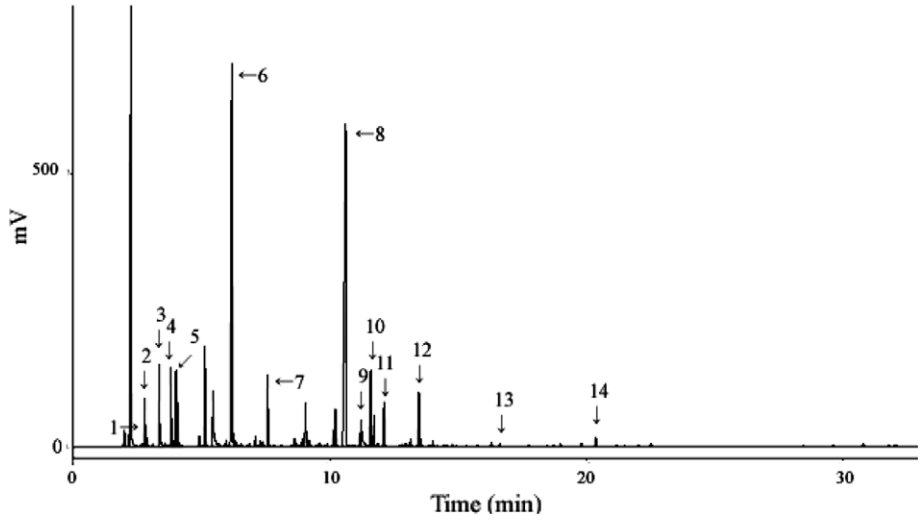

Fig. 2. HS-SPME-GC chromatogram for volatiles from thermally oxidized FFA at 93 °C. Peaks with numbered arrow are listed in Table 1.

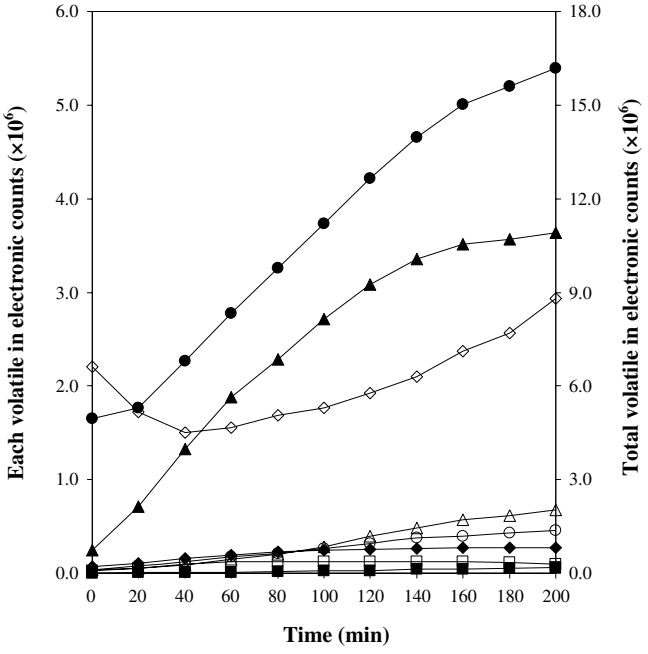

Fig. 3. Changes of volatile compounds from thermally oxidized FFA at 93 °C ($n = 3$). Error-bars were not shown. Pentane (□), hexanal (◇), 2-hexenal (♦), 2-heptenal (▲), 2,4-heptadienal (○), 2-octenal (△), 2,4-decadienal (■), total volatiles (●).

2006; Sanches-Silva et al., 2004; Steenson et al., 2002). High correlations between the content of hexanal and TBARS have been reported in cooked and cold stored turkey meat (Mielnik et al., 2006) and in lean pork (Wettasinghe & Shahidi, 1996). Beltran et al. (2005) reported that hexanal analyzed by HS-SPME from oxidized sunflower oil at 60 °C in oil-in-water emulsion was not changed much for 4 h and increased drastically after 4 h. In this experiment, peak areas of hexanal decreased until 40 min and started to increase after 40 min. 2-Heptenal, which is a typical oxidized volatile from linoleic acid (Frankel, 1985), increased linearly up to 160 min. Peak areas of 2-hexenal, 2,4-heptadienal, 2-octenal, and 2,4-decadienal increased with various slopes (Fig. 3). 2-Hexenal is a typical oxidized volatile from linolenic acid (Frankel, 1985) and increased linearly up to 100 min. 2,4-Heptadienal, 2-octenal, and 2,4-decadienal, which are major oxidized volatile products from linolenic, linoleic, and linoleic acids (Frankel, 1985), respectively, increased throughout oxidation period. Pentane is one of the major volatiles from oxidized linoleic acid at 13-hydroperoxide (Frankel, 1985). Peak areas of pentane increased up to 60 min oxidation and started to decrease. Although HS-SPME has limitation to detect volatiles depending on the volatility and concentration of compounds (Beltran et al., 2005), this study clearly show that not all oxidized volatiles are increasing proportionally along with oxidation time.

Volatile compounds should have high correlation with the lipid oxidation and high GC responses to be used as parameters for representing the oxidation state of FFA and determining the antioxidant activity. Volatile compounds with high linearity (R^2) between peak areas against CDA at 0–80 min or *p*-AV at 40–180 min in thermally oxidized FFA are shown in Fig. 4. CDA and *p*-AV were used as reference parameters from 0 to 80 min and from 40 to 180 min oxidation time, respectively. Linearity (R^2) between CDA and total volatiles, hexanal, 2-hexenal, 2-heptenal, 2,4-heptadienal, 2-octenal, or 2,4-decadienal were over 0.99 at the range of 40–80, 40–80, 0–80, 0–80, 0–80, 40–80, and 0–40 min oxidation time, respectively. In case of *p*-AV, peak areas of total volatiles, hexanal, 2-hexenal, 2-heptenal, 2,4-heptadienal, 2-octenal, or 2,4-decadienal showed over 0.99 (R^2) linearity at the oxidation time of 40–180, 120–180, 40–100, 40–140, 40–180, 40–160, and 80–120, respectively (Fig. 4). Pentane was not considered due to its inconsistent changes during oxidation.

Changes of major volatile compounds from thermally oxidized FFA at 0, 60, 120, and 180 min are shown in

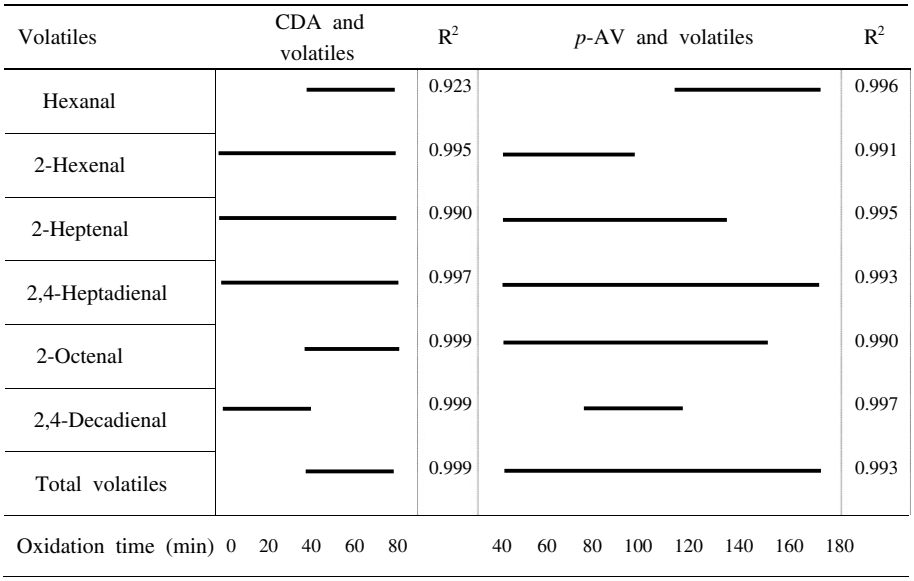

Fig. 4. Oxidation period of volatiles showing high linearity (R^2) against CDA or *p*-AV in thermally oxidized FFA at 93 °C.

Table 1. At 0 min, hexanal was the highest volatile followed by 2-heptenal and 1-heptene by HS-SPME analysis. The highest volatile at 60 min was in the order of 2-heptenal, hexanal, 2-pentylfuran, 1-heptene, and 2-octenal, while at 120 and 180 min, the order was 2-heptenal, hexanal, 2-pentylfuran, 2-octenal, and 2,4-heptadienal (Table 1). Linearity (R^2), slopes, and *y*-intercept of total volatiles and selected volatiles from thermally oxidized FFA at 93 °C for 200 min are shown in Table 2. Total volatiles and 2,4 heptadienal showed the highest linearity throughout oxidation time while hexanal showed the lowest linearity of 0.72. The slopes of peak areas of 2-heptenal was +17602, which was 3.5 time higher than those of hexanal with the second highest increasing slopes (Table 2). Jelen et al. (2000) tested fourteen lipid oxidation volatiles including hexanal, 2-heptenal, and 2,4-decadienal by HS-SPME and confirmed the availability of SPME method for each volatile analysis. Also, they reported that the main sensory

Table 2
Linearity (R^2), slopes, and *y*-intercept of hexanal, 2-hexenal, 2-heptenal, 1-octene-3-ol, octanal, 2,4-heptadienal, and total volatiles from thermally oxidized FFA at 93 °C for 200 min in electronic counts

Volatiles	Slopes	*y*-Intercept	Linearity
Hexanal	4970	1534086	0.72
2-Hexenal	992	113575	0.91
2-Heptenal	17602	633071	0.96
1-Octene-3-ol	1780	58821	0.98
Octanal	356	9363	0.95
2,4-Heptadienal	2212	40504	0.99
Total volatiles	61889	4698255	0.99

Table 1
Changes of peak areas of major volatile compounds from 93 °C thermally oxidized free fatty acids at 0, 60, 120, and 180 min in electronic counts ($\times 10^4$)

No.	Volatile compounds	Retention time (min)	Oxidation time (min)			
			0	60	120	180
1	1-Hexene[a]	2.73	3.39 ± 0.40[b]	2.21 ± 0.06	4.18 ± 1.14	5.19 ± 0.23
2	Pentane	2.79	2.70 ± 0.58	12.17 ± 1.21	12.72 ± 1.93	11.55 ± 1.65
3	Butanal[MSc]	3.49	1.71 ± 0.21	14.6 ± 2.21	20.37 ± 2.69	20.75 ± 1.63
4	1-Heptene	3.98	23.33 ± 1.80	22.88 ± 3.90	24.42 ± 8.38	25.63 ± 6.48
5	Pentanal	4.06	7.09 ± 1.50	8.21 ± 2.89	10.43 ± 1.13	13.42 ± 1.25
6	Hexanal	6.16	220.89 ± 27.36	155.19 ± 20.33	192.69 ± 10.93	256.84 ± 7.84
7	2-Hexenal[MS]	7.54	6.63 ± 1.34	19.49 ± 1.74	25.48 ± 2.01	27.26 ± 3.44
8	2-Heptenal	10.54	24.63 ± 7.56	187.56 ± 55.60	308.34 ± 62.06	357.03 ± 57.63
9	1-Octen-3-ol	11.22	4.01 ± 2.18	18.27 ± 7.61	29.32 ± 9.19	35.36 ± 11.34
10	2-Penthyl furan	11.56	19.48 ± 11.55	24.38 ± 8.10	46.57 ± 10.55	79.64 ± 12.08
11	2,4-Heptadienal[MS]	12.1	4.27 ± 1.54	17.52 ± 6.41	31.75 ± 10.08	43.45 ± 14.65
12	2-Octenal	13.46	3.17 ± 1.07	14.61 ± 5.40	39.59 ± 13.13	61.76 ± 20.87
13	2-Nonenal	16.28	20.67 ± 17.11	2.39 ± 1.03	3.30 ± 1.26	4.69 ± 1.89
14	2,4-Decadienal	20.34	1.27 ± 0.50	3.42 ± 1.52	9.09 ± 4.05	16.93 ± 7.49

[a] Volatiles were identified with a combination of mass spectrometry and retention time of standard compounds.
[b] Mean ± standard deviation (average of triplicate in electronic counts).
[c] Volatiles were tentatively identified with mass spectrometry only.

descriptors for the stored vegetable oils are oxidized, acidic, and green odor, which are influenced mainly by 2-heptenal, free fatty acids, and 2-hexenal contents, respectively. Especially, the contents of 2-heptenal was closely correlated with worst sensory quality of rapeseed oil (Jelen et al., 2000).

Considering high linearity with CDA or *p*-AV and high peak responses in GC, total volatiles and 2-heptenal could be selected as possible volatile parameters for determining antioxidant activity using commercially available mixtures of FFA under current experimental conditions.

3.3. HS-SPME method for measuring hydrogen donating antioxidant activity

Peak areas of total volatiles, hexanal, 2-hexenal, 2-heptenal, 2-octenal, and 2,4-heptadienal from oxidized FFA with BHT are shown in Fig. 5. CDA and *p*-AV, which are closely related with primary and secondary lipid oxidation, were valid at 0–80 min and 40–180 min, respectively, and 60 min of oxidation time was chosen for representing both primary and secondary lipid oxidation products. Total volatiles and 2-heptenal were detected higher than any other volatiles. Linearity (R^2) of peak areas of total volatiles, hexanal, 2-hexenal, 2-heptenal, 2,4-heptadienal, and 2-octenal against BHT concentration were 0.917, 0.885, 0.987, 0.956, 0.926, and 0.886, respectively. 2-Heptenal and total volatiles showed high linearity against BHT concentration. Hexanal showed reverse trend comparing with other volatiles and did not give good linearity against BHT concentration in this experimental condition. HS-SPME can differentiate the FFA with different BHT concentration by comparing peak areas of total volatile and 2-heptenal.

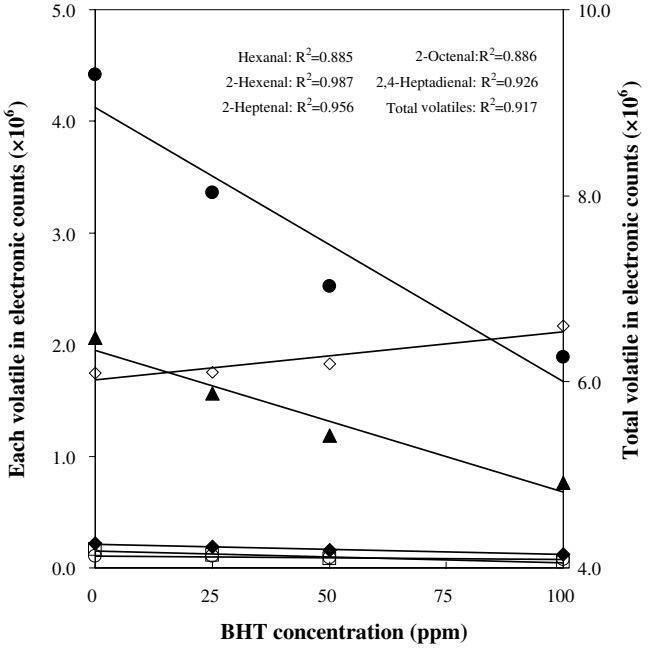

Fig. 5. Changes of peak areas from volatiles against BHT concentration in thermally oxidized FFA at 93 °C for 60 min ($n = 4$). Error-bars were not shown. Hexanal (◇), 2-hexenal (♦), 2-heptenal (▲), 2,4-heptadienal (○), 2-octenal (□), total volatiles (●).

Commercially available mixtures of FFA were selected instead of using specific vegetable oils, which have different fatty acid compositions and may contain various amounts of inherent antioxidants. It is well-documented that the productions of oxidized volatile compounds are greatly influenced by the fatty acid composition of oils (Frankel, 1985; Jelen et al., 2000). Thus the most well correlated volatile compounds for the degree of oxidation would be also different with the different oil systems (Jelen et al., 2000). Due to these reasons, specific vegetable oils may not be proper as substrates for determining antioxidant activity by HS-SPME. However, results of antioxidant activity from commercially available mixtures of FFA can be compared objectively with other reports using commercially available FFA as substrates.

In conclusion, changes of volatile distribution from oxidized FFA were monitored by HS-SPME and availability of this method for determining antioxidant activity for hydrogen donating compounds was studied. As thermal oxidation time increased, some aldehydes including hexanal, 2-hexenal, 2-heptenal, 2,4-heptadienal, 2-octenal, and 2,4-decadienal increased linearly at specific oxidation period. Total volatiles showed the highest linearity (R^2) of 0.99 for 200 min and 2-heptenal showed the highest increase in peak areas. Not all oxidized volatiles increased linearly during oxidation. Thermally oxidized FFA containing different BHT concentration could be differentiated by HS-SPME comparing peak areas of total volatiles and 2-heptenal.

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