

Pan-Heating of Low-Linolenic Acid and Partially Hydrogenated Soybean Oils

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ABSTRACT: Genetically modified low-linolenic acid soybean oil (LL-SBO) was compared to partially hydrogenated soybean oil (PH-SBO). Samples were heated on a Teflon pan at ~180°C until a selected end point of ≥20% polymer content was reached. High-performance size-exclusion chromatography analysis indicated the PH-SBO contained >20% polymer after 20 min of heating, whereas the LL-SBO sample contained >20% polymer after 10 min. Supercritical fluid chromatography analysis indicated degradation rates of $0.161 \pm 0.011 \text{ min}^{-1}$ for LL-SBO and $0.086 \pm 0.004 \text{ min}^{-1}$ for PH-SBO. The volatile compounds were identified and quantitated with static headspace-GC-MS. 1-Heptene (239.9 ppm) and hexanal (1486.1 ppm) were present at the greatest concentration among the volatile compounds in LL-SBO. The volatile compounds present in the greatest concentrations in heated PH-SBO were hexanal (376.9 ppm) and pentane (82.1 ppm). After 10 min of heating, the LL-SBO oil FFA value (2.66%), *p*-anisidine value (386.5 abs/g oil), Food Oil Sensor reading (18.75), and color intensity ($Y = 4.0$, $R = 1.0$) were significantly greater than those of PH-SBO after 14 min of heating (4.28%, 298.5 abs/g oil, 16.08, $Y = 1.0$, $R = 0.1$, respectively). There was a significant difference in the degradation rates between LL-SBO and PH-SBO ($P < 0.05$). The PH-SBO was more stable than the LL-SBO.

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Genetic improvements in vegetable oils will continue to play an important role in the development of new frying oils. In 1997, an estimated 23 million acres of genetically modified oilseeds were planted in North America (1). The topic of genetically improved oils has become an area of intense research because most consumers prefer to avoid the saturated and *trans* FA found in hydrogenated oils. In one set of genetically engineered oils, the FA components have been altered to provide high-stability oil without hydrogenation (2). The frying performance of genetically modified oils has been investigated to determine the effect of changing the FA composition on oil stability and odor intensity. A deep-fat frying study of soybean and canola oils, modified by hydrogenation and/or mutation breeding, indicated a reduction in odor intensity for low-linolenic acid oil (3).

Billek (4) investigated the effect of different frying methods on polar-material formation. Ten minutes of pan-frying

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was comparable to approximately 20 h of deep-fat frying, because these are the heating times required to reach approximately the same percentage of polar materials.

Many investigators have published work on deep-fat frying, but very few have studied oil deterioration during pan-frying (5), even though pan- and grill-frying are convenient and common cooking methods used in households and restaurants for preparation of meat, eggs, and vegetables (6). Stir-frying, a form of pan-frying, is a common practice in Chinese cooking. In a study of volatile compounds found during stir-frying, oil was heated at ~200°C in a Chinese wok for 3 min with constant stirring (7). In another study, a thin film of trilinolein was applied to pans made of platinum, of iron, and of silica. Thermal decomposition and polymerization occurred in the outermost 120 μm of oil films heated for 4 min at 200°C (5).

Pan-frying can be an alternative to deep-fat frying. It can be used for continuous cooking of meat products in the food industry, and has been considered as a possible alternative to reduce excessive fat uptake that can occur during deep-fat frying (8). During pan frying, the oil is heated as a thin film at high temperature for a short time. Based on the acid values, iodine values, and TBARS values of oils heated at 100 and 180°C, thin-film heating appeared to be a very deteriorative process (9).

The objectives of this experiment were twofold. The first goal was to design experiments to assess frying oil performance during pan-heating. The second was to determine the extent of changes that occurred during heating of genetically modified low-linolenic acid and partially hydrogenated soybean oils with regard to volatile and nonvolatile components.

EXPERIMENTAL PROCEDURES

Materials and methods. Two oils, low-linolenic acid soybean oil (LL-SBO) and creamy partially hydrogenated soybean oil (PH-SBO), were obtained from Kraft Food Ingredients (Memphis, TN). The FA profiles of the unheated oils were determined by FAME analysis according to AOCS Official Method Ce 2-66 (10). Boron trifluoride in methanol (Sigma Chemical Co., St. Louis, MO) was used for FA transesterification. The column used was a 30 m DB-Wax, 0.25 mm i.d., $d_f = 0.25 \mu\text{m}$ (J&W Scientific, Folsom, CA). The initial temperature was 170°C, initial time was 2 min, and rate was 5°C/min. The final temperature was 230°C and final hold time was 5 min. The mobile phase gas (He) flow rate was 0.8 mL/min. The mobile phase velocity was 25 cm/s. The injector split was 50:1.

A laboratory pan-heating system was designed to treat the samples. Oil samples were heated at approximately 180°C in a

Teflon pan fryer, 679 cm² surface area (10.25" × 10.25", T-FAL Ultra-base Griddle, TEFAL, Rumilly Cedex, France). The oil temperature was monitored with an infrared thermometer (Model HP A2235M; Cole-Parmer, Vernon Hills, IL). Temperatures were taken at each corner and the center of the pan every minute and recorded during heating to ensure isothermal treatment at 180°C for both samples. Heating periods were randomized. A TLC sprayer (5 mL tube capacity, 14/20 joints; Fisher Scientific, Pittsburgh, PA) was used to apply (spray) 5 mL of oil on the interior heating surface of the pan. The pan was preheated to 100°C before the oil sample was placed in the pan to decrease the come-up time for the oil to the temperature setpoint of 180°C. All of the heating experiments were performed in duplicate and analyzed in triplicate. To check on the reproducibility of the experiments, the average of all replicates in the first heating was compared to that of a second heating. Statistical significance was expressed at the 95% confidence interval, and no significant differences were found between the two experiments (SAS Institute, Cary, NC).

To determine the weight of the sample deposited on the pan, 20 replicate applications of 5-mL oil samples to the pan surface were completed, the oil was collected, and isopropanol was used to remove the oil from the pan surface. The oil sample was weighed after solvent evaporation, and an average weight was determined. When 5 mL of an oil sample was applied, a thin film of oil weighing 2.5 ± 0.1 and 2.6 ± 0.3 g for LL-SBO and PH-SBO, respectively, was deposited on the pan. The heat source was a 30.5 × 30.5 cm hot plate (Thermolyne, Dubuque, IA). After each heating experiment, the oil was removed with isopropanol and/or THF (Fisher Scientific). THF was used to remove oil samples used for high-performance size-exclusion chromatography (HPSEC) analysis, because THF was the solvent used for HPSEC. The solvent was removed under a continuous nitrogen purge, and then the sample was sealed in an amber vial, blanketed with nitrogen, and stored in the dark at approximately 3 to 5°C until analysis the following day. As a result of the oil lost during heating, each experiment produced only ~1 g of sample. To collect sufficient material for all of the analyses, several oil samples were heated under identical conditions and then pooled together to create a single large sample. For the analysis of volatile components, the procedure used was slightly different. Several samples were heated and then combined, but they were collected without the use of a solvent. An internal standard (I.S.) was added, and then an accurately weighed oil sample was placed directly into vials for GC-MS analysis.

Physicochemical analysis. The FFA values, *p*-anisidine values (*p*-AV), and color intensity of each oil sample, heated and unheated LL-SBO and PH-SBO, were determined in triplicate according to the AOCS Official Methods, 1990, Ca 5a-40, Cd 18-90, and Cc 13b-45, respectively (10). Color analysis was done with oil samples that were then used for the FFA determination, because a large amount of sample was required for the FFA analysis. The dielectric constant was measured in triplicate with a Foodoil Sensor (FOS) (Northern Instruments Corp., Lino Lakes, MN).

Chromatographic analysis. HPSEC and supercritical fluid chromatography (SFC) analysis were done according to the

method previously published by Artz *et al.* (11). The TAG polymer content was determined with HPSEC, whereas the unaltered TAG substrate concentration was measured with SFC. The HPSEC system included four Phenogel columns each with 5- μ m particles and particle pore sizes of 500, 100, 100 (three columns × 500 mm in length × 8.0 mm in diameter), and 50 Å (300 mm × 7.8 mm) (Phenomenex, Torrance, CA) connected in series to an ELSD (ELSD II A, Varex Corp., Burtonsville, MD). The mobile phase was THF (Fisher Scientific). The capillary column used for SFC was a 14-m SB-cyano-25 (50 μ m i.d., d_f = 0.25 μ m, 25% cyanopropyl and 75% polymethyl siloxane). The mobile phase was SFC-grade CO₂ (MG Industries, Malvern, PA).

Static headspace with GC-MS was used to collect, separate, quantitate, and identify the major volatile compounds in triplicate. A static headspace sampler, Tekmar 7000 (Tekmar Co., Cincinnati, OH), with a heated 3 m × 0.32 mm transfer line, was used to transfer the volatile compounds to the GC capillary column (Durabond, DB-5, 50 m × 0.32 mm i.d. × 1.0 μ m film; J&W Scientific). A 5890 series II Hewlett-Packard GC connected to a mass selective detector, model MSD 5973 (Hewlett-Packard, Naperville, IL) was used for identification, whereas a GC with an FID was used for quantification of the volatile compounds. The GC conditions were as follows: initial temperature 40°C, followed by a temperature ramp of 5°C/min to 85°C and then 25°C/min to 250°C. The headspace gas column pressure was 12 psi, and the FID was held at 300°C. Helium was the carrier gas with a run time of 30 min. Volatile compounds were cryo-focused at -165°C with a Tekmar Cryo-Focusing Module (Tekmar Co.) before introduction of analytes into the column. The headspace autosampler conditions were as follows: platen temperature 90°C, platen equilibrium time 5 min, sample equilibrium time 30 min, vial pressurization time 0.25 min, pressure equilibrium time 0.05 min, loop fill time 0.15 min, loop equilibrium time 0.05 min, injection time 2 min, cryo injection time 0.75 min, cryo injection temperature 200°C, sample loop temperature 150°C, and line temperature 150°C. The GC cycle time was 60 min.

Approximately 15 g of sample, immediately collected without solvent after each heating, was placed in an Erlenmeyer flask and spiked with an I.S., methyl hexanoate, to assist in quantitation. The I.S. was added directly with a 1- μ L syringe to an Erlenmeyer flask containing collected heated samples. The weight change (~0.0001–0.0004 g) was recorded, and the concentration of I.S. as parts per million (ppm) was determined. The Erlenmeyer flask was sealed, and the I.S. and the oil sample were mixed with a stir bar on a stir plate for 10 min before placing 1-g samples into headspace vials. Polytetrafluoroethylene/silicone vial septa (PerkinElmer, Norwalk, CT) secured by aluminum caps were used to seal the sample vials. The septa were kept at 55°C for at least 48 h in a vacuum desiccator prior to use to remove any contaminating volatile compounds. External standards were prepared (~300 ppm) by adding flavor standards to a bland sample of sunflower oil (Hunt-Wesson, Fullerton, CA).

Heating experiments were done in duplicate and all the GC-MS analyses were performed in triplicate; therefore, each data point was an average of six measurements from the quantitation calculations.

Statistical analysis. Changes occurring during the heating of LL-SBO and PH-SBO were compared by statistical analysis using a Statistical Analysis System, Version 8.0 (SAS Institute, Cary, NC). Data were analyzed with a General Linear Model (GLM) program expressed at 95% confidence interval. The program was used to analyze the data based on comparison of rates for the changes in the physicochemical analyses results, the polymer content, and increase in volatile compound concentrations for both the LL-SBO and PH-SBO samples.

RESULTS AND DISCUSSION

The FAME composition of PH-SBO (Table 1) shows a greater percentage of oleic acid and much less linoleic acid than the LL-SBO. HPSEC analysis indicated a substantial increase in TAG polymer content (>20%) after 10 min of heating for the LL-SBO sample and after 20 min of heating for the PH-SBO sample. The increase in polymer content that occurred during heating is shown in Figure 1. A rapid increase was expected considering that a thin layer of oil was exposed to high temperatures. During pan-frying there is a greater oxygen adsorption per unit oil than in deep-fat frying, which results in a large increase in the rate of oxidation and a large reduction in the frying life of the oil (6). The HPSEC results also correlated well with the SFC results for the two samples. Capillary SFC was used to determine the concentration and, subsequently, the degradation rate for the unaltered TAG substrate remaining in each oil sample after each pan-frying or heating interval. Based on substrate concentration determined with capillary SFC, the degradation rate for PH-SBO was $0.086 \pm 0.004 \text{ min}^{-1}$ with 16% unaltered TAG remaining in the oil sample at the end of the 20-min heating period, whereas the degradation rate for LL-SBO was $0.161 \pm 0.011 \text{ min}^{-1}$ with 22% unaltered TAG at the end of a 10-min heating period (which was one-half the heating period for PH-SBO).

Table 2 contains the physicochemical analysis results for the heated LL-SBO and PH-SBO samples. As expected, the values increased rapidly during the heating period because of a high surface-to-volume ratio, which allowed substantial air exposure at elevated temperatures. The values are similar for both samples, although it is important to note that the PH-SBO sample required twice as much heating time as the LL-SBO sample to reach approximately the same target endpoint.

Statistical evaluation of the data indicated that there was a highly significant difference between the two oils (LL-SBO

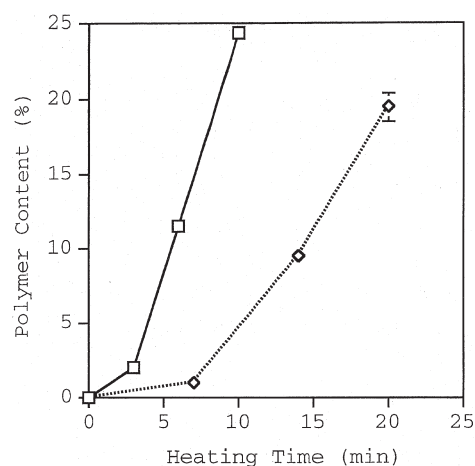


FIG. 1. Increase in polymer content as a function of time for heated LL-SBO (\square , low-linolenic acid soybean oil) and heated PH-SBO (\diamond , partially hydrogenated soybean oil).

and PH-SBO) with regard to the rates of polymer formation, FFA value increase, *p*-AV increase, FOS increase, and Lovibond color formation ($P < 0.05$). The results of polymer content and physicochemical analyses indicate that PH-SBO was more stable than LL-SBO.

The identity and concentration of the major volatile compounds are shown in Tables 3 and 4. Volatile components were tentatively identified by comparison of retention times with standards and were calculated as ppm according to the method described by Ouchi (12). The major volatile compounds found in LL-SBO included butanal, 2-pentanone, 1-heptene, pentanal, *trans*-2-pentenal, 1-pentanol, hexanal, 2-hexanal, heptanal, *trans*-2-heptenal, 2-pentylfuran, and *trans*-2-octenal. 1-Heptene at 240 ppm and hexanal at 1486 ppm were present at the greatest concentrations among the volatile compounds found at the end of heating. Hexanal is the major volatile product formed from the decomposition of linoleic acid and 1-heptene is produced from oleic acid decomposition (13,14). Other linoleic acid decomposition products

TABLE 2
Physicochemical Analysis of Heated LL-SBO and PH-SBO^{a,b}

Time (min)	FFA value ^c (% oleic)	<i>p</i> -AV ^c (abs/g oil)	FOS ^c (reading)	Color ^c	
				Y	R
LL-SBO					
0	0.07 ± 0.01	2.8 ± 0.8	0.00 ± 0.00	1.0	0.0
3	0.41 ± 0.02	227.4 ± 2.8	7.67 ± 0.16	1.0	0.1
6	1.16 ± 0.04	372.3 ± 3.7	15.78 ± 0.21	2.0	0.3
10	2.66 ± 0.02	386.5 ± 8.9	18.75 ± 0.26	4.0	1.0
PH-SBO					
0	0.04 ± 0.00	5.5 ± 0.9	0.00 ± 0.00	1.0	0.0
7	0.27 ± 0.03	134.8 ± 3.6	5.63 ± 0.17	1.0	0.0
14	1.74 ± 0.06	298.5 ± 9.4	16.08 ± 0.09	1.0	0.1
20	4.28 ± 0.04	346.2 ± 6.4	18.62 ± 0.03	3.0	0.4

^aAbbreviations: *p*-AV, *p*-anisidine value; abs, absorbance; FOS, Foodoil Sensor; Y, yellow filter reading; R, red filter reading; for other abbreviations see Table 1.

^bEach value represents the average of triplicate analysis from duplicate heating experiments ± SD.

^cSignificant difference between LL-SBO and PH-SBO with regard to the rates of FFA value increase, *p*-AV increase, FOS increase, and Lovibond color formation ($P < 0.05$).

TABLE 1
FAME Composition (relative %) of LL-SBO and PH-SBO^{a,b}

FA	LL-SBO (%)	PH-SBO (%)
C16:0	10.2 ± 0.1	10.7 ± 0.1
C18:0	4.8 ± 0.1	11.7 ± 0.1
C18:1	28.0 ± 0.1	43.5 ± 0.1
C18:2	53.3 ± 0.1	30.3 ± 0.0
C18:3	3.0 ± 0.0	2.6 ± 0.1

^aAbbreviations: LL-SBO, low-linolenic acid soybean oil; PH-SBO, partially hydrogenated soybean oil.

^bEach value represents an average of six replicates ± SD.

TABLE 3
Flavor Volatile Compounds (ppm) in Heated LL-SBO^{a,b}

Volatile compound	3 min	6 min	10 min
Butanal ^c	2.2 ± 0.6	12.6 ± 2.0	71.7 ± 10.7
2-Pentanone	3.8 ± 0.2	5.2 ± 0.2	19.1 ± 1.7
1-Heptene	14.6 ± 0.1	37.5 ± 1.8	239.9 ± 21.2
Pentanal	5.1 ± 0.0	18.9 ± 0.9	101.1 ± 9.5
<i>trans</i> -2-Pental	ND	4.9 ± 0.2	ND
1-Pentanol	ND	7.8 ± 0.3	33.8 ± 2.4
Hexanal ^c	106.2 ± 1.5	298.1 ± 12.3	1486.1 ± 126.8
2-Hexanal	4.4 ± 0.0	9.9 ± 0.4	35.5 ± 3.2
Heptanal	ND	ND	20.9 ± 9.3
<i>trans</i> -2-Heptenal ^c	9.7 ± 0.2	19.2 ± 0.7	76.5 ± 6.6
2-Pentylfuran	ND	5.2 ± 0.2	29.5 ± 2.6
<i>trans</i> -2-Octenal	ND	25.9 ± 0.8	93.3 ± 7.9

^aAbbreviation: ND, not detected; for other abbreviations see Table 1.

^bEach value represents the average of triplicate analyses from duplicate heating experiments ± SD.

^cSignificant difference between LL-SBO and PH-SBO (Table 4) with regard to the rates of butanal, hexanal, and *trans*-2-heptenal increase ($P < 0.05$).

included butanal, pentanal, 1-pentanol, 2-hexanal, *trans*-2-heptenal, 2-pentylfuran, and *trans*-2-octenal. Butanal and *trans*-2-pental also are products of linolenic acid decomposition. All of the volatile compounds increased in concentration as the heating progressed. Most of the volatile compounds in LL-SBO were similar to those formed in PH-SBO.

PH-SBO flavor volatile compounds included butanal, hexane, pentanone, 1-heptene, pentane, heptane, 2-pental, 1-pentanol, hexanal, *trans*-2-hexenal, heptanal, *trans*-2-heptenal, octanal, *trans*-2-octenal. Hexanal at 377 ppm and pentane at 82 ppm were present in the greatest concentration among all the volatile compounds formed after 20 min of heating. Butanal, pentane, hexane, 1-pentanol, hexanal, *trans*-2-hexenal, *trans*-2-heptenal and *trans*-2-octenal are all decomposition products of linoleic acid. Butanal and 2-pental are derived from linolenic acid. Oleic acid decomposition products included 1-heptane,

TABLE 4
Flavor Volatile Compounds (ppm) in Heated PH-SBO^a

Volatile compound	7 min	10 min	14 min
Butanal ^b	4.3 ± 0.2	21.6 ± 5.3	36.4 ± 8.5
Hexane	ND	15.1 ± 0.6	26.1 ± 2.9
Pentanone	ND	6.0 ± 0.6	10.4 ± 0.8
1-Heptene	ND	5.5 ± 0.3	7.5 ± 0.3
Pentane	10.4 ± 4.5	42.4 ± 1.8	82.1 ± 3.9
Heptane	ND	22.8 ± 0.5	43.8 ± 3.7
2-Pental	ND	7.7 ± 0.1	8.7 ± 0.4
1-Pentanol	ND	5.4 ± 0.2	ND
Hexanal ^b	61.2 ± 2.0	283.8 ± 12.6	376.9 ± 18.2
<i>trans</i> -2-Hexenal	ND	8.2 ± 0.3	11.4 ± 0.5
Heptanal	ND	7.8 ± 0.3	19.0 ± 0.9
<i>trans</i> -2-Heptenal ^b	5.2 ± 0.1	12.4 ± 0.7	16.5 ± 0.7
Octanal	ND	ND	9.4 ± 0.5
<i>trans</i> -2-Octenal	ND	30.1 ± 1.4	30.8 ± 1.2

^aEach value represents the average of triplicate analyses from duplicate heating experiments ± SD.

^bSignificant difference between LL-SBO (Table 3) and PH-SBO with regard to the rates of butanal, hexanal, and *trans*-2-heptenal increase ($P < 0.05$). For abbreviations see Tables 1 and 3.

heptane, heptanal, and octanal. Butanal, hexanal, and *trans*-2-heptenal were found in both samples during all heating intervals. Therefore, the rate of increase in these volatile concentrations was used for statistical evaluation and comparison of the samples. Statistical analysis indicated that there was a significant difference in the rate of volatile compound production ($P < 0.05$), with the heated LL-SBO samples having a greater rate of volatile component production than did PH-SBO.

The results indicate that the PH-SBO sample was more stable than the LL-SBO sample in experiments designed to compare pan-frying performance. PH-SBO must be heated approximately twice as long to reach the same level of oxidative and thermal degradation as the LL-SBO sample under the pan-heating conditions of these experiments.

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