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Abstract
This project was designed to evaluate the oxidative stability of corn oils with increased total saturated fatty acid composition and to test the feasibility of using the AromaScan, an "electronic nose," to detect the odors/aromas produced by oxidation. Corn oils with traditional (13.1%) and elevated (14.7 to 17.1%) total saturated fatty acid percentages were evaluated for their oxidative quality. Oils from five corn genotypes were extracted, refined, bleached, and deodorized (RBD) in the laboratory. Two replications, separated at the point of extraction, were evaluated for each genotype. The RBD corn oils (18.0 g) were stored in 50-mL beakers at 60°C in the dark, and peroxide values were measured every other day for 8 d. Corn oils with elevated saturated fatty acid compositions were more stable ($P<0.05$) than the traditional corn oil. Aroma intensity of the oils was measured with an AromaScan at days 0, 4, and 8. The AromaScan provided a useful tool to detect odors/aromas produced by oxidation during an oxidative stability study; this tool might be used to partly replace human sensory panel evaluation of oxidized samples.

Keywords
Aroma AromaScan, corn oil, electronic nose, fatty acids, oil stability, oxidation, peroxide value. volatile compounds

Disciplines
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Oxidative Stability and AromaScan Analyses of Corn Oils with Altered Fatty Acid Content

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ABSTRACT: This project was designed to evaluate the oxidative stability of corn oils with increased total saturated fatty acid composition and to test the feasibility of using the AromaScan, an "electronic nose," to detect the odors/aromas produced by oxidation. Corn oils with traditional (13.1%) and elevated (14.7 to 17.1%) total saturated fatty acid percentages were evaluated for their oxidative quality. Oils from five corn genotypes were extracted, refined, bleached, and deodorized (RBD) in the laboratory. Two replications, separated at the point of extraction, were evaluated for each genotype. The RBD corn oils (18.0 g) were stored in 50-mL beakers at 60°C in the dark, and peroxide values were measured every other day for 8 d. Corn oils with elevated saturated fatty acid compositions were more stable ($P < 0.05$) than the traditional corn oil. Aroma intensity of the oils was measured with an AromaScan at days 0, 4, and 8. The AromaScan provided a useful tool to detect odors/aromas produced by oxidation during an oxidative stability study; this tool might be used to partly replace human sensory panel evaluation of oxidized samples.

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KEY WORDS: Aroma, AromaScan, corn oil, electronic nose, fatty acids, oil stability, oxidation, peroxide value, volatile compounds.

An increased use of corn oil has resulted from the recognition of corn oil's high content of unsaturated fatty acids, lack of linolenic acid (18:3) (1), and its flavor stability (2,3). An optimal level of tocopherols also contributes to its quality. Increasing the total saturated fatty acid (TSFA) composition of corn oils, however, is likely to increase its oxidative stability, resulting in specialty oils suitable for deep-fat frying in the food industry or, if the saturated fatty acid percentage is great enough, it also could be used for margarine production without hydrogenation.

Oils are hydrogenated to reduce 18:3 and other polyunsaturated fatty acids, unfortunately producing some *trans* fatty acids at the same time. Mensink and Katan (4) reported that *trans* fatty acids in a diet increased total and low-density lipoprotein cholesterol levels and lowered the high-density lipoprotein cholesterol levels compared to *cis* fatty acids in a diet. The majority of *trans* fatty acids (80%) in the human diet come from consumption of hydrogenated vegetable oils, with the average per capita intake ranging from 6.5 to 12.0 g/d of *trans* fatty acids (5).

Researchers at the Agricultural Research Service at the USDA have developed corn lines with elevated TSFA composition (15 to 17% vs. 13% in traditional corn oil), and anticipate that even greater TSFA percentages will be obtained from continued line development. These oils should have superior oxidative stability and their use as unhydrogenated oils in place of hydrogenated oils would reduce the consumption of *trans* fatty acids. Naturally saturated corn oils also should have fewer processing costs and should result in more profit for the farmers and/or less cost for the consumers.

The study of oxidative stability of oils sometimes involves sensory evaluation. There can be difficulties in recruiting sensory panelists to taste oxidized samples and in acquiring accurate and reliable data. Advances in the technology of multisensor arrays, however, have enhanced the development of "electronic noses" for the detection of the odor/aromas of oils produced by oxidation. Bartlett *et al*. (6) suggested that the electronic nose might be used to supplement or even replace traditional sensory techniques. The application of an electronic nose, such as the AromaScan, to monitor oxidation could be a useful way to enhance the understanding of oxidation studies, to provide an objective analysis of oxidation, and to partly supplement human sensory panels.

The main objective of this research was to investigate the oxidative stability of corn oils with elevated and traditional TSFA compositions during storage. A secondary objective was to study the possible application of an electronic nose, the AromaScan, in assessing the oxidative stability of the corn oils during storage.

MATERIALS AND METHODS

Materials. The commercial corn cultivar P3394 (Pioneer Brand Hybrid 3394, Pioneer Hi-Bred International, Inc., Johnston, IA) and four experimental corn lines (TS143, TS43-45, TS42-44, and TS86) with elevated TSFA compositions were grown in isolated fields near Ames, Iowa, in 1996. These experimental corn lines were obtained by cross-polli-

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nating Corn Belt inbred lines with material introgressed with *Tripsacum dactyloides*, a wild relative of corn.

Germ separation. Corn was wet-milled according to a pilot-plant procedure proposed by Singh *et al.* (7). After the separation, germs were dried, placed into polyethylene bags, and stored in the dark at 4°C until used.

Laboratory-scale oil extraction. Corn germs were flaked in a smooth roller mill (Roskamp Manufacturing Inc., Waterloo, IA) to about 0.25 mm in thickness. Flaked samples from each genotype were divided into two lots before extraction to give two replications for each of the five corn genotypes.

The extraction method was described by Shen *et al*. (8). Each replicate of flakes (0.40 kg or less) was put into one of the two vessels. Two replicates were extracted simultaneously at 60°C, at a ratio of 2:1 hexane/flakes in the laboratory extraction simulator. During extraction, micella was pumped to vessels containing flakes through which it percolated. The pumping rate was controlled to maintain 1.0 cm of miscella over the flakes. Flakes from each replicate were extracted three consecutive times (stages) with fresh hexane for 6 min of recirculation, plus 3 min for draining. The miscella collections from the three stages were pooled.

The miscella was desolventized in a rotary evaporator (Wheaton, Heidolph, Germany) at 60°C at 80 rpm. After desolventizing, the crude oils were stored at −14°C under nitrogen until needed.

Refining, bleaching, and deodorizing. Crude oils from each replicate were refined, bleached, and deodorized as described by Shen *et al.* (9). Crude oils were refined according to AOCS Official Method Ca-9d-52 (10), bleached based on AOCS Official Method Cc-8b-52 (10), and deodorized by high vacuum (<0.5 Torr) and high temperature (230 to 240°C for 2 h) as described by Stone and Hammond (11) and modified according to Moulton (12). After processing, oils were stored under nitrogen at −14°C until needed. The yields of the corn oils after each stage of processing are listed in Table 1. During oil extraction and refining, no antioxidants or preservatives were added to the oils.

Accelerated stability tests at 60°C in the dark. Corn oils (18 g per replicate of five treatments) were stored in 50-mL beakers without covers at 60°C in the dark until sufficiently oxidized. Every other day, an aliquot (1.2 g) of each corn oil was removed for analysis. Replicates were measured twice and averaged.

Peroxide values (PV) were measured according to the modified Stamm test (13). Fatty acid compositions were measured after triacylglycerides had been converted into fatty acid methyl esters (FAME) according to a method described by Hammond (14). The FAME were injected onto a Hewlett-Packard 5890 Series II gas chromatograph (Kennett Square, PA) equipped with a flame-ionization detector, a split/splitless injector, and an automatic sampler. A DB-23 fused-silica capillary column with dimensions of 0.25 mm × 15 m × 0.25 µm film thickness was used (J&W Scientific Inc., Rancho Cordova, CA). Chromatographic parameters were set as follows: injector temperature 250°C, detector temperature 250°C, column temperature 200°C, and column head pressure 15 kPa.

Aroma profiles of corn oils were measured *via* the "sensors" of an AromaScan A32S (AromaScan Inc., Hollis, NH). The procedure of measuring aroma profiles was set per the manufacturer's recommendation (15). The total time of measurement was 200 s, including referencing, sampling, washing, and referencing. The referencing (zeroing) was done in 20 s with water vapor, sampling in 120 s, washing in 30 s with the vapor of 2% isopropyl alcohol in water to wash off the sample residuals from the polymer heads of the sensors, and referencing (zeroing) in 30 s with water vapor, as recommended in the manual. Preliminary investigation indicated the operation procedure was satisfactory and no carryover of volatile compounds was detected. The data were collected during the interval of 108 to 138 s. Within these 30 s, six slices (intervals) were used to collect data. The working conditions of the AromaScan oven were 15% relative humidity and 35°C. The samples were equilibrated in the oven for 5 min to reach the oven temperature. For each AromaScan measurement, a 1-mL aliquot of oil from each replication was transferred with an Oxford BenchMate pipetter into a 500-mL air bag fitted with a one-way valve. The aroma profiles were generated from the aroma intensity, with only arbitrary or relative units applied.

Statistical analysis. A completely randomized design was used for this experiment. Data from all treatments were analyzed by analysis of variance procedures on the Statistical Analysis System release 4.0 for Microsoft Windows (16). Differences in mean values among treatments were determined by the least significant difference test at $P = 0.05$ (16). Each type of corn oil was considered as a treatment, with two replicates per treatment.

RESULTS AND DISCUSSION

The yields of the corn oils, after each stage of processing from the crude oil (refining, bleaching, deodorizing, and final yield) are listed in Table 1; these yields are similar to ones reported by Shen *et al.* (9). Yields among corn oil types seemed to differ; however, the ranges overlapped so their differences were not likely important in the current study. The FAME and TSFA compositions of the corn oils, initially (day 0) and after

TABLE 1
The Oil Yields[a] (%) of Traditional Corn and Corn with Elevated Total Saturated Fatty Acid Composition After Stages of Processing

Corn oil	Refining	Bleaching	Deodorizing	Final
P3394	78.6–86.4	84.5–89.6	97.3–97.4	63.5–74.2
TS143	84.1–85.4	89.7–92.0	96.9–97.1	71.1–74.8
TS43-45	83.5–83.8	93.0–93.2	96.3–96.6	73.3–73.8
TS42-44	88.8–90.0	81.7–85.7	95.4–96.4	70.0–72.8
TS86	92.0–92.3	84.3–87.1	91.8–95.0	70.0–74.5

[a]Values are the range from two replications. Percentage reflects yield for the stage listed. Final yield shows overall percentage yield from beginning to ending of processing.

TABLE 2
FAME Percentage of Corn Oils Obtained by Laboratory-Scale Extraction Before and After Storage at 60°C in the Dark for 8 d

		FAME by GLC (relative area %)[a]						
Corn oil	Days	16:0[b]	18:0[b]	18:1[b]	18:2[b]	18:3[b]	TSFA[c]	CO[d]
P3394	0	11.7 ± 0.08[e]	1.4 ± 0.04	26.4 ± 0.14	59.6 ± 0.10	0.9 ± 0.04	13.1	6.6
	8	11.8 ± 0.09	1.5 ± 0.07	26.7 ± 0.19	59.1 ± 0.20	0.9 ± 0.05		
TS143	0	12.6 ± 0.07	2.1 ± 0.11	32.3 ± 0.35	52.2 ± 0.43	0.7 ± 0.07	14.7	5.9
	8	12.8 ± 0.10	2.2 ± 0.04	32.8 ± 0.08	51.6 ± 0.11	0.7 ± 0.03		
TS43-45	0	13.1 ± 0.04	2.5 ± 0.03	22.0 ± 0.08	61.7 ± 0.15	0.7 ± 0.09	15.6	6.7
	8	13.2 ± 0.09	2.5 ± 0.11	22.1 ± 0.39	61.6 ± 0.48	0.6 ± 0.03		
TS42-44	0	13.5 ± 0.08	2.4 ± 0.03	22.1 ± 0.07	61.4 ± 0.15	0.6 ± 0.03	15.8	6.7
	8	13.6 ± 0.10	2.4 ± 0.04	22.2 ± 0.10	61.3 ± 0.19	0.6 ± 0.03		
TS86	0	14.5 ± 0.06	2.6 ± 0.06	27.9 ± 0.25	54.5 ± 0.33	0.6 ± 0.03	17.1	6.0
	8	14.7 ± 0.08	2.6 ± 0.05	28.2 ± 0.10	54.0 ± 0.16	0.5 ± 0.04		

[a]Values are the average of duplicate analysis of two replications. FAME, fatty acid methyl ester; GLC, gas–liquid chomatography.
[b]16:0 = palmitic acid, 18:0 = stearic acid, 18:1 = oleic acid, 18:2 = linoleic acid, and 18:3 = linolenic acid.
[c]TSFA, total saturated fatty acids.
[d]CO, calculated oxidizability = [18:1% + 10.3(18:2%) + 21.6(18:3%)]/100.
[e]Data presented are the mean of two replicates with four injections each ± standard deviation.

storage (day 8), are listed in Table 2. The TSFA initial compositions in the experimental lines ranged from 14.7 to 17.1%, with 13.1% TSFA in the traditional corn oil. The 18:3 composition was 0.9% in the traditional corn oil and ranged from 0.6 to 0.7% in the experimental oils; the linoleic acid (18:2) compositions for all oils at 0 d varied from 52.3 to 61.8%. The oleic acid (18:1) composition of corn oils originally was 26.4% for P3394 and varied from 22.0 to 32.3% for corn oils with TSFA. The higher 18:1 over 18:2 usually increases the stability of corn oils. The relative percentage of most fatty acids changed slightly during storage, as preferential oxidation of the polyunsaturated fatty acid progressed. The 18:3 compositions of the corn oils were either unchanged or decreased slightly during storage, as noted by other researchers (9,17,18). The calculated oxidizabilities of corn oils were calculated according to a formula listed at the bottom of Table 2, which was proposed by Fatemi and Hammond (19). According to the calculation, the corn oils from P3394, TS43-45, and TS42-44 should oxidize the most quickly, with calculated oxidizabilities of 6.6, 6.7, and 6.7, respectively.

The PV is commonly used as an indicator of lipid oxidation, especially during the beginning oxidation stages. The PV plateaus after reaching a certain point, then finally decreases. The PV (Table 3) clearly showed the benefits of increased TSFA and decreased 18:3 percentages in the original oils. For corn oils on day 0, the PV were very similar to each other. Even though P3394 had a statistically higher PV than the other corn oils, the practical differences (0.04 to 0.08 meq/kg) likely were not important. From day 2 to day 8 of storage, P3394 always had a significantly greater PV than did the other oils. There were no significant differences among TS143, TS43-45, TS42-44, and TS86 oils on days 2, 4, and 6. On day 8, TS42-44 was significantly lower in PV than P3394 and TS43-45, and TS143 and TS86 had PV intermediate to TS43-45 and TS42-44.

The electronic nose was used to measure aroma intensity. It is generally assumed that a greater aroma intensity indicates the presence of more volatile compounds, which means the samples are more oxidized than those with lower aroma intensity. The AromaScan is designed to give three-dimensional pictures of the aroma profiles of a substance. An example of the aroma profile of P3394 during storage at days 0, 4, and 8 is shown in Figure 1. The *x, y,* and *z* axes indicate only the multidimensional relationship among measurements and have no physical or chemical meanings. Each small square in Figure 1 represents an individual measurement of aroma intensity. There were six individual measurements for each duplicate treatment and two replications for each of the duplicates. Therefore, four sets of six boxes each are shown in Figure 1 for each day of analysis. To analyze these data statistically, the data were exported to a spreadsheet and reorganized to give a two-dimensional graph. The aroma intensities, measured by 32 sensors in AromaScan, of all corn oils after 8 d of storage are shown in Figure 2. The average aroma score of 32 sensors (0 to 31) was used to calculate the total aroma intensity of corn oils.

Values of the total aroma intensity of corn oils and the results of the statistical analysis are listed in Table 4. Values in this table reflect the relative intensities among samples; nega-

TABLE 3
Peroxide Values (meq/kg) of Corn Oils During Storage at 60°C in the Dark

	Corn oil genotypes[a]				
Day	P3394	TS143	TS43-45	TS42-44	TS86
0	0.26[a]	0.18[b]	0.22[b]	0.22[b]	0.20[b]
2	4.42[a]	1.36[b]	1.55[b]	1.60[b]	1.29[b]
4	33.65[a]	15.95[b]	19.44[b]	11.48[b]	15.16[b]
6	90.12[a]	45.64[b]	59.34[b]	39.13[b]	48.54[b]
8	134.64[a]	75.88[b,c]	100.00[b]	72.18[c]	79.04b[c]

[a]Values in the same row with different superscript letters are significantly different ($P \leq 0.05$).

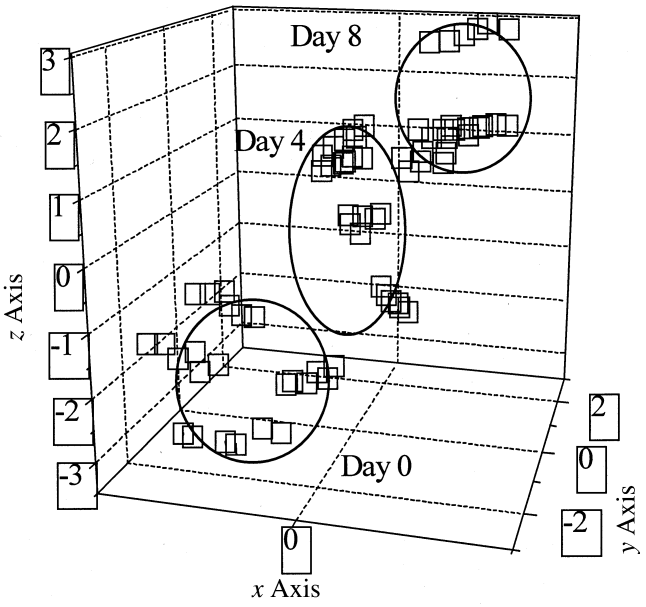

FIG. 1. Three-dimensional chart of aroma profiles of corn oil (P3394) derived from the AromaScan during storage at 60°C in the dark.

TABLE 4
Aroma Intensities of Corn Oils During Storage at 60°C in the Dark

	Corn oil genotypes[a]				
Day	P3394	TS143	TS43-45	TS42-44	TS86
	Total aroma intensity				
0	-0.037^{a}	-0.147^{b}	-0.127^{b}	-0.143^{b}	-0.149^{b}
4	0.218^{a}	0.109^{b}	$0.089^{b,c}$	0.009^{c}	$0.028^{b,c}$
8	0.153^{a}	$0.086^{a,b}$	0.010^{b}	0.147^{a}	$0.051^{a,b}$
	Aroma intensity detected by sensor 7				
0	-0.015^{a}	-0.143^{b}	-0.110^{b}	-0.125^{b}	-0.153^{b}
4	0.283^{a}	0.070^{b}	0.098^{b}	0.010^{b}	0.030^{b}
8	0.243^{a}	$0.120^{a,b,c}$	0.020^{c}	$0.180^{a,b}$	$0.070^{b,c}$
	Aroma intensity detected by sensor 10				
0	0.023^{a}	-0.145^{b}	-0.103^{b}	-0.135^{b}	-0.163^{b}
4	0.395^{a}	0.185^{b}	0.168^{b}	0.093^{b}	0.115^{b}
8	0.318^{a}	$0.130^{b,c}$	0.043^{c}	$0.210^{a,b}$	$0.098^{b,c}$
	Aroma intensity detected by sensor 14				
0	0.113^{a}	-0.058^{b}	-0.003^{b}	0.045^{b}	0.100^{b}
4	0.485^{a}	0.245^{b}	0.245^{b}	0.150^{b}	0.153^{b}
8	0.440^{a}	0.208^{b}	0.088^{b}	0.240^{b}	0.180^{b}

[a]Values in the same row with different superscript letters are significantly different ($P \leq 0.05$).

tive values mean only that intensities are less than positive values, but otherwise the negative sign is meaningless. The fresh corn oil of P3394 had a significantly greater aroma intensity than did the other oils. There were no significant differences in aroma intensity among the experimental oils on day 0. The aroma intensity of TS86 tended to have the lowest score of all corn oils at days 0 and 4, except for TS42-44. This oil also had the greatest TSFA content (Table 2). On day 4, the aroma intensity of P3394 was significantly greater than that of other corn oils. The aroma intensity of TS42-44 was the lowest of all corn oils on day 4 of storage. The oil with the lowest aroma intensity on day 8, however, was TS43-45, which had one of the greatest calculated oxidizabilities and the second-greatest PV. These conflicting results need further investigation before a reasonable explanation can be drawn. On day 8, the aroma intensity of P3394, the traditional oil with the lowest saturated fatty acid composition, still tended to be greater than that of TS143, TS86, and TS42-44, and it was significantly greater than that of TS43-45.

The oxidation of lipids results in the formation of primary and secondary decomposition products, including hydroperoxides, carbonyls, alcohols, esters, carboxylic acids, and hy-

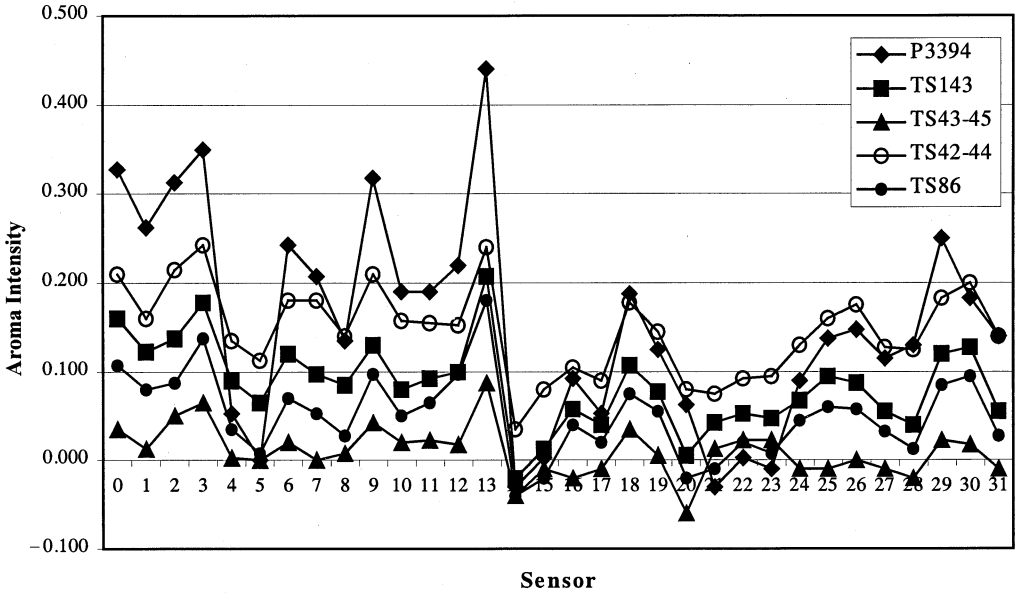

FIG. 2. Aroma profiles of corn oils after 8 d of storage at 60°C in the dark.

drocarbons (20,21,22). The polymer sensors of AromaScan are capable of detecting some of those aroma compounds. As an example, the responses of each sensor for all oils on day 8 are shown in Figure 2. Several sensors gave very intense responses and were chosen to determine the aroma profiles of oxidized corn oils.

According to the operation manual, sensor #7 has a medium response to short-chain alcohols and a strong response to carboxylic acids. During storage, P3394 had a significantly greater aroma intensity measured by sensor #7 on days 0 and 4 than did the other oils (Table 4). There were no significant differences among corn oils with elevated TSFA contents. On day 8, P3394 tended to have the greatest aroma intensity and TS43-45 tended to have the least aroma intensity.

Sensor #10 can detect short- and long-chain alcohols, carboxylic acids, short- and long-chain esters, and ketones. The data in Table 4 also show that on days 0 and 4, P3394 had significantly greater aroma intensities of these volatile compounds than did other oils. On day 8, P3394 tended to have greater aroma intensity than did TS42-44, and it had significantly greater aroma intensities of volatile compounds than did oils of TS143, TS43-45, and TS86. Sensor #14 is capable of detecting short-chain alcohols and carboxylic acids. The P3394 oil had significantly greater aroma intensity by this sensor than did the other oils after 0, 4, and 8 d of storage. There were no significant differences among corn oils with elevated TSFA contents during storage.

The data from the AromaScan are in general agreement with the PV data confirming that corn oils with elevated TSFA contents had lower PV and lower aroma intensities during storage. One exception was the AromaScan data from TS43-45, which generally was the lowest in aroma intensity among all genotypes, despite having PV that tended to be greater than those of the other oils with elevated TSFA. The calculated oxidizability also predicted that TS143 and TS86 were more stable than P3394, but it could not differentiate between P3394 and TS43-45/TS42-44. In general, the oils from the experimental corn lines (TS143, TS43-45, TS42-44, and TS86) were more stable to oxidation than the P3394 oil as evaluated by PV and aroma intensity. The application of the AromaScan may be useful in detecting aroma intensity in corn oils. The electronic nose provided a nondestructive, rapid, and objective way to detect aromas resulting from oil oxidation. Ultimately, these values might be more directly related to certain flavors and off-flavors in oils. These preliminary data from the AromaScan are promising, thus, additional tests will be done to correlate sensory evaluation with AromaScan analyses.

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