Oxidation of Soybean Oil During Storage of Starch–Oil Composites

G.A. Garzón[a], K. Warner[b], F.C. Felker[a,]*, D.E. Palmquist[c], and F. Eller[d]

[a]Cereal Products and Food Science, [b]Food and Industrial Oil, [c]Midwest Area,
and [d]New Crops and Processing Research, NCAUR, ARS, USDA, Peoria, Illinois 61604

ABSTRACT: The oxidative deterioration of dry starch–oil composites was investigated by chemical and sensory methods. The composites were stored at 37°C for 63 d, and changes in the hexanal content and odor attributes were monitored. Analysis of the extracted oil showed that the first run through the drum dryer presented higher hexanal concentrations than the subsequent runs. Starch–oil composites from the first run though the drum dryer showed higher metal concentrations and higher odor ratings during storage time than the subsequent runs. There was a significant correlation between odor attributes and hexanal concentration in the first run. Since both the oxidation and the metal content were higher in the first run and decreased in further runs, we concluded that oxidation might have been induced by the presence of metal in the drum dryer. It is likely that during drum drying, the metal drum and knife surfaces became coated with soy oil, reducing both abrasion and metal contact with the product. Since many applications of the starch–oil composite technology require drum drying, it will be necessary to take measures to minimize metal contamination of the product, perhaps by discarding the initial portion of product, which contains the most metal.

Paper no. J10826 in *JAOCS 81*, 861–866 (September 2004).

KEY WORDS: Hexanal, oxidation, sensory, starch–oil composite.

Starch–oil composites are unique compounds produced by excess steam-jet cooking technology. The excess steam-jet cooking procedure combines high temperature, pressure, and shear as a mixture of starch and oil passes through a small orifice. This treatment causes complete gelatinization and solubilization of the starch and intimate mixing of the carbohydrate with the lipid (1). Dispersions obtained by jet cooking can be drum dried to yield a solid product that can be subsequently mixed with water to obtain dispersions similar to the original freshly cooked composite. Electron microscopy has shown that the oil present in these composites is encapsulated as droplets of about 1–10 µm in diameter within the starch/water matrix (2) and that oil droplets are stabilized by thin films of starch at the oil–water interface (3).

Starch–oil composites represent a versatile technology for delivering starch-coated oil droplets in an aqueous medium. Potential uses include as water-based lubricants, agricultural seed coatings, cosmetic and drug delivery systems, flavor-delivery systems, and reduced-fat food products (1,4,5). Recent studies report the use of such composites as fat replacers in high-fat baked goods such as cookies (6), soft-serve ice cream (7), and beef patties (8,9).

Work in our laboratory has suggested that under the same storage conditions, samples of starch–oil composites from different runs through the drum dryer performed on the same day present different degrees of lipid oxidation. The degree of difference in lipid oxidation seems to be related to the metal content in the samples, with this metal being a contaminant from the drum dryer. A high correlation between soybean oil stability and metal content has been shown, with copper and iron reported as the most important pro-oxidants. The catalytic effect of copper is about 10 times as great as that of ferric ions at small concentrations of metals (10).

No studies have been undertaken to examine the oxidative stability of milled starch–oil composites. Therefore, the main objective of this study was to measure secondary oxidative changes in drum-dried starch–oil composites during storage and to determine their relationship to metal contamination from drum-drying operations. Changes in the odor attributes and hexanal content were determined and correlated to predict the shelf life of such composites.

EXPERIMENTAL PROCEDURES

Materials and reagents. Normal, unmodified food-grade cornstarch was purchased from A.E. Staley Manufacturing Company (Decatur, IL). Soybean oil was acquired from Central Soya Company, Inc. (Fort Wayne, IN). The PV was 0.21 ± 0.011 meq/kg.

Preparation of starch–oil composites. Normal cornstarch and distilled water were mixed to a solids content of 16% (dry-weight basis). This dispersion was stirred in a stainless-steel Waring blender (model 37BL84; Dynamics Corporation of America, New Hartford, CT) for 2 min and subsequently pumped through a Laboratory Model steam-jet cooker consisting of a progressive cavity pump (model 2M1 SS03 AAA; Robbins and Myers, Inc., Springfield, OH) and a manual stainless steel hydroheater (model M101-030; Hydrothermal, Waukesha, WI). The jet cooker was operated under excess steam conditions (11); outlet pressure was maintained at

---

*To whom correspondence should be addressed.
E-mail: felkerfc@ncaur.usda.gov
Present address of G.A.G.: 6011 N. Rosemead Dr., Peoria, IL 61614.

275.8 kPa (140°C), steam line pressure was 448.2 kPa (155 °C), and the pumping rate was 1 L/min. Soybean oil was added to the resulting dispersion in a proportion of 20 parts of oil per 100 parts of dry starch; the slurry was then stirred again in the Waring blender. This oil–starch suspension was fed a second time through the jet cooker under the conditions just described. The final liquid composite was placed in a Dewar flask prior to drying operations on a pilot-scale double-drum dryer (model 20; Drum Dryer and Flaker Company, South Bend, IN) heated with steam at 206.8 kPa (135°C). Dry flakes from the drum dryer were milled using a type ZM1 Retsch mill (Brinkmann Instruments, Inc., Westbury, NY). The amounts of these products used in subsequent analyses were corrected for moisture and are reported on a dry-weight basis. Two experiments were performed with three runs in each experiment. Samples from each run were stored and tested in triplicate, with the exception of hexanal analysis, which was done in duplicate. The starch–lipid composites are described in U.S patents 5,676,994 and 5,882,713 assigned to the U.S. Department of Agriculture (12,13).

Chemical analysis. PV of the starting soybean oil was determined according to AOCS Official Method Cd 8-53 (14). Since copper and iron are known to catalyze nonenzymatic lipid oxidation, the concentrations of these transition metals were measured in the starting soybean oil and in the composites after drum-drying operations. The metal analysis was performed by inductively coupled plasma atomic emission spectroscopy according to AOAC Official Methods 968.08 D(b) and 990.08 (15). Method detection limits were 0.03 ppm for copper and 0.05 ppm for iron.

Storage of composites. Starch–oil composites were stored under accelerated oxidation conditions. Samples (27 g) of the drum-dried starch–oil composites were transferred to 250-mL screw-capped glass jars and subsequently stored in the dark in an incubator (Queue Model 2220, Kendro Laboratory Products, Asheville, NC) at 37 ± 1°C for 8 wk. Preliminary trials (data not shown) indicated high variability in lipid oxidation values among replications. For this reason, two separate final experiments were conducted. In experiment 1, stored samples were removed from the oven at 0, 6, 12, 16, 23, 27, 30, 44, and 63 d, whereas in experiment 2, samples were removed every 7 d. Samples for sensory analysis were stored in a freezer at −70°C for further testing. For hexanal measurements, oil expression from the samples was done immediately after they were removed from the incubator.

Oil expression. Oil was expressed from the starch–oil composites by placing 10 g of sample in a stainless-steel pellet die (28 mm diameter) and pressing it with a Carver press (Model C; Fred Carver, Inc., Wabash, IN) for approximately 30 s each at 5,000, 10,000, 15,000, and then 20,000 lb of pressure. The expressed oil flowed onto a stainless-steel sheet, from which it was collected for further analysis. We observed that the yield of oil from samples decreased as oxidation proceeded, and eventually no oil could be expressed even after a prolonged application of pressure. We attributed the decrease in oil yield to polymerization of the oxidized oil. Studies characterizing the nonvolatile degradation products formed during oxidation of soybean oil show that high-M.W. compounds are formed as a result of thermal oxidation (16).

Analysis of hexanal. Since hexanal has been reported as the main indicator of linoleic acid oxidation (17), hexanal concentrations were determined in both the starting soybean oil and in the soybean oil extracted from the composites. Headspace analysis using a solid-phase microextraction (SPME) method was used. A 0.5-g sample of oil was placed in an 8-mL headspace vial sealed with a Teflon-lined septum and held at 25°C with constant stirring. The septum was pierced with a 50/30-µm divinylbenzene/Carboxen/polydimethylsiloxane SPME needle (Supelco Inc., Bellefonte, PA), and the fiber was exposed to the oil headspace for 30 min. The fiber was then retracted into the needle and immediately transferred to the injection port of a gas chromatograph, where it was used for hexanal determination. SPME fibers were desorbed by a 0.5-min split-delay splitless injection onto a Hewlett-Packard 5890 Series II gas chromatograph equipped with an FID and an SP-2340 column (60 m × 0.25 mm i.d., 0.20 mm film thickness, Supelco Inc.). The temperature program was 60°C for 1 min, 2°C/min to 76°C, 20°C/min to 250°C, and hold for 5 min. The carrier gas was helium at a linear flow velocity of 18 cm/s. The injector and detector temperatures were 200 and 250°C, respectively. A standard curve of hexanal GC area counts was developed for five concentrations of hexanal in soybean oil to determine the concentration of hexanal in experimental samples. Solutions with 0.001, 0.01, 0.1, 1, and 10 ppm hexanal in soybean oil were analyzed by SPME as described above. A linear regression analysis was applied to determine the hexanal concentrations in the experimental samples.

Sensory analysis. A 12-member, experienced analytical-descriptive sensory panel conducted the evaluation of odor attributes of the starch–oil composites. Metallic, rancid, and painty descriptors were used. Odor attributes were determined in preliminary testing of fresh and aged composite samples, and panelists rated the intensities of the odors on a 10-point intensity scale (0 = none; 10 = strong). Samples were assigned random three-digit numbers, and panelists evaluated three to four samples at a time in a randomized balanced incomplete block design during each panel session. One-gram samples of composite powder were placed in sealed 2-oz plastic souffle cups and held at 25°C for 1 h before testing. Evaluations were carried out in a laboratory with individual booths under red lighting to limit any perceptions of differences in the color of the samples. Samples of fresh and aged composite samples were used to train the panel on odor attributes.

Statistical analysis. A completely randomized design (CRD) was used to examine the effects of storage time on hexanal in the starch–oil composites. A general linear model was applied by fitting three linear regression equations, comparing three consecutive runs through a drum dryer, for hexanal concentration as a function of storage time. Instrumental data from the two experiments were pooled to obtain linear

TABLE 1
Iron and Copper Contents of Starting Soybean Oil and Starch–Oil Composites^a

	Experiment 1				Experiment 2			
Metal	Soy oil	Run 1	Run 2	Run 3	Soy oil	Run 1	Run 2	Run 3
Fe (ppm)	<0.05	7.18 (0.571)	3.78 (0.814)	3.53 (0.248)	<0.05	3.78 (0.185)	1.73 (0.141)	2.34 (0.161)
Cu (ppm)	<0.03	0.57 (0.012)	<0.03	<0.03	<0.03	0.87 (0.090)	<0.03	<0.03

[a] Values are means of three subsamples from each run for each experiment. Values in parentheses are SD of three subsamples.

regression equations. An F-test for full and reduced models was used to compare the hexanal from each run as a function of storage time in weeks. If a significant test statistic was found, indicating unequal hexanal responses over storage time for the three runs, intercept and slope comparisons were made to determine differences between the runs. Mean predicted values of the dependent variable were estimated from the resultant equations at several storage times, along with 95% confidence intervals for those predicted means, and values were then compared among the three runs separately for each storage time. Runs at each storage day were not significantly different from one another when the 95% confidence intervals for predicted hexanal concentrations overlapped.

A CRD was used to examine the effects of storage time on sensory attributes of the composites. Instrumental data from the two experiments were pooled, and linear regression equations were calculated for odor evaluations as a function of storage time for each of the three runs through the drum dryer. Mean predicted values of odor responses were calculated from the regression equations at several storage times, with 95% confidence intervals. The sensory attributes were then used in a simple linear regression analysis to predict panelist responses from hexanal measurements. Correlation coefficients between sensory evaluation ratings and instrumental responses were obtained from the linear regression analysis. Statistical analyses were performed using the PROC GLM of SAS, version 8.0 (18).

RESULTS AND DISCUSSION

Metal content of soybean oil and starch–oil composites. As reported in Table 1, in both experiments, the copper and iron contents of the starting soybean oil were below detection level. Conversely, in both experiments the iron concentration in the composites from the first run was about twofold the concentration in samples from runs 2 and 3. Similarly, the copper concentration was higher in samples from run 1 as compared with samples from runs 2 and 3 in both experiments.

Increases in copper and iron concentrations due to drum-drying operations have been reported previously. Hakansson and Jagerstad (19) observed that the iron concentrations in white flours and whole meal was increased two- and fivefold, respectively, after drum-drying operations. In those experiments, copper also increased markedly after drum drying of whole meal. Similarly, after mild drum drying of whole-meal wheat flour, Wennermark *et al.* (20) found an increase from 1.6 to 2.7 mg iron/100 g dry matter and from 0.24 to 0.38 mg copper/100 g dry matter. The authors attributed such increases to leakage from the drum.

Hexanal. A linear increase in hexanal concentration during storage of the composites was observed. This increase is described by the equation $C = C_0 + kt$, in which C is the hexanal concentration at any storage time, C_0 is the initial concentration of hexanal at time 0 of storage, k is the rate constant of the reaction, and t is the storage time. The rate constants, regression coefficients, correlation coefficients (r), and coefficients of determination (r^2) are given in Table 2.

All three runs had significant, positive slopes ($P < 0.0001$), with the slope of the first run being significantly higher than the slopes for runs 2 and 3 ($P < 0.05$). The first and third runs through the drum dryer had negative intercepts that were not significantly different from zero, whereas the second run had the only positive intercept that was significant ($P = 0.0453$). In all cases, the high, significant correlation coefficients of the linear regression equations suggest that soybean oil oxidation was a zero-order reaction.

The hexanal concentration values and linear regression results are shown in Figure 1. Unlike runs 2 and 3, samples

TABLE 2
Regression Models for Hexanal Values During Storage for Starch–Oil Composites

Run	Model	r	P-value	r^2
1	Hexanal (ppm) = $-1.1491^a + 0.632$ time (0.882) (0.049)	0.937	≤0.01	0.88
2	Hexanal (ppm) = $1.174 + 0.176$ time (0.566) (0.018)	0.847	≤0.01	0.72
3	Hexanal (ppm) = $-0.762^a + 0.195$ time (0.658) (0.021)	0.848	≤0.01	0.72

[a] Coefficients are not significantly different from zero. Values in parentheses are SD of regression coefficients. r, Pearson correlation coefficient; P-value, significance levels of regression models; r^2, coefficient of determination.

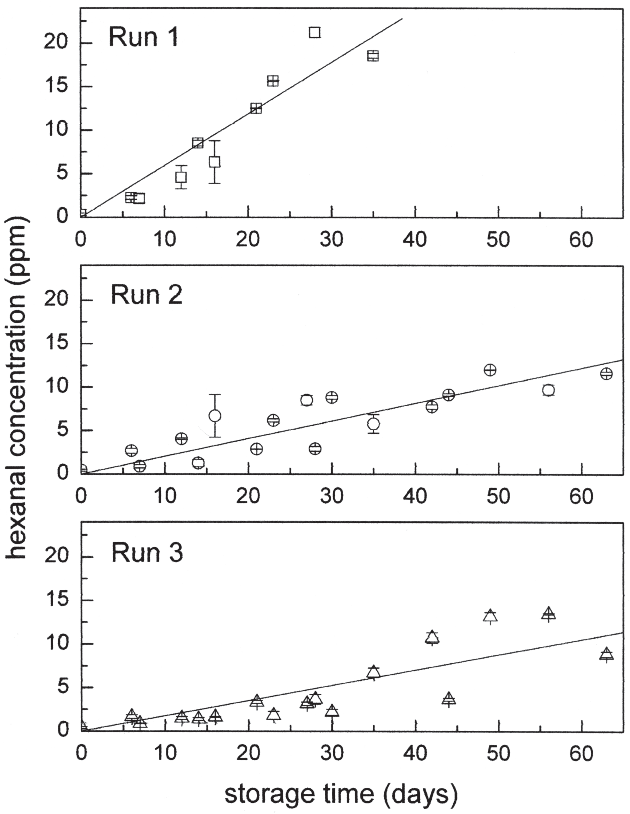

FIG. 1. Simple linear regressions, means ± SE, of hexanal content (ppm) of soybean oil obtained from three consecutive runs of soybean oil composites stored at 37°C in the dark.

from run 1 showed a significant increase in hexanal concentration beginning at day 12 of storage and continuing to day 35 ($P \leq 0.05$). At that time, it was not possible to obtain oil from the powder from run 1. This behavior agrees with reports by Hardas *et al.* (21), who had difficulty extracting the encapsulated fat from oxidized powders; in their study the amount of recoverable encapsulated fat decreased with time. Samples from runs 2 and 3 had additional storage times of 42, 44, 49, 56, and 63 d. From zero time up to day 23, run 2 had a significantly higher hexanal content than run 3 ($P \leq 0.05$), but there were no differences in hexanal concentration between these two samples after 23 d of storage.

When comparing different drying methods, Hall (22) found that hexanal was the most abundant volatile product of whole-meal and white-flour lipid oxidation. The author reported that roller drying caused a high initial hexanal concentration and that the increase of this volatile was rapid during storage.

Odor attributes. The mean predicted values of the sensory attribute ratings from the descriptive analysis are presented in Table 3. The change in metallic and rancid odors was described by the equation: mean rating = β_1 (time) + β_0, where β_0 is the intercept, and β_1 is the slope for all three runs. The change in painty character was described by the equation: mean rating = β_1 (time)^2 + β_0 for all three runs. This quadratic model indicates that a maximum painty odor was detectable at a certain storage time but that it decreased with further storage time.

We observed large SE in mean predicted values during the first 2 wk of sensory testing, indicating that the odor attributes

TABLE 3
Mean Predicted Values of Sensory Attributes During Storage of Starch–Oil Composites from Regression Equations[a]

Odor Attribute	Storage time (wk)						Slope (β_1)
	0	2	4	6	7	8	
Run 1							
Metallic	$0.105^{a(1)}$	$0.333^{a(1)}$	$0.561^{a(1)}$	$1.02^{a(1)}$	$1.47^{a(1)}$	NR	0.228
	(0.285)	(0.228)	(0.189)	(0.202)	(0.314)		(0.080)
Rancid	$-0.498^{d(1)}$	$0.723^{c,d(1)}$	$1.94^{c(1)}$	$4.38^{b(1)}$	$6.84^{a(1)}$	NR	1.22
	(0.443)	(0.354)	(0.292)	(0.315)	(0.488)		(0.125)
Painty	$0.148^{c(1)}$	$0.314^{c(1)}$	$0.811^{c(1)}$	$2.80^{b(1)}$	$6.11^{a(1)}$	NR	0.166
	(0.230)	(0.221)	(0.20)	(0.175)	(0.335)		(0.012)
Run 2							
Metallic	$0.119^{a(1)}$	$0.190^{a(1)}$	$0.262^{a(2)}$	$0.333^{a(2)}$	$0.369^{a(2)}$	$0.404^{a(1)}$	0.036
	(0.092)	(0.065)	(0.052)	(0.062)	(0.074)	(0.089)	(0.019)
Rancid	$0.405^{a(1)}$	$0.992^{a(1,2)}$	$1.58^{a(2)}$	$2.17^{a(2)}$	$2.46^{a(2)}$	$2.75^{a(1)}$	0.294
	(0.630)	(0.442)	(0.352)	(0.426)	(0.508)	(0.607)	(0.127)
Painty	$0.193^{c(1)}$	$0.256^{b,c(1)}$	$0.445^{a,b,c(2)}$	$0.760^{a,b,c(3)}$	$0.965^{a,b(3)}$	$1.202^{a(2)}$	0.016
	(0.159)	(0.145)	(0.113)	(0.119)	(0.158)	(0.218)	(0.005)
Run 3							
Metallic	$0.129^{c(1)}$	$0.183^{b,c(1)}$	$0.238^{a,b,c(2)}$	$0.293^{a,b(2)}$	$0.320^{a,b(2)}$	$0.347^{a(1)}$	0.027
	(0.036)	(0.025)	(0.022)	(0.028)	(0.034)	(0.040)	(0.008)
Rancid	$-0.225^{c(1)}$	$0.740^{c(2)}$	$1.704^{b(2)}$	$2.67^{a,b(2)}$	$3.15^{a(2)}$	$3.63^{a(1)}$	0.482
	(0.30)	(0.210)	(0.181)	(0.236)	(0.283)	(0.336)	(0.065)
Painty	$0.087^{c(1)}$	$0.24^{c(1)}$	$0.700^{c(2)}$	$1.47^{b(2)}$	$1.96^{a,b(2)}$	$2.54^{a(1)}$	0.038
	(0.164)	(0.150)	(0.121)	(0.143)	(0.191)	(0.260)	(0.005)

[a] Mean predicted values represent scores rated on a 0 = none to 10 = strong intensity scale. Values in parentheses are SE of the mean predicted values. Predicted values within a row with unlike superscripts indicate a significant difference. For each odor attribute, predicted values in the same column with identical superscript numbers in parentheses are not significantly different based on overlap of the 95% confidence intervals. NR, not recorded.

were difficult to evaluate consistently among panelists and between runs and replicates.

Undesirable volatiles increased with storage time. However, panelists were unable to detect significant increases in the metallic character of samples from runs 1 and 2 during storage. On the other hand, the panelists reported a significant increase in metallic odor after 6 wk of storage in samples from run 3. At weeks 4, 6, and 7, significant increases in rancid odor were detected by the panelists in samples from run 1 ($P \leq 0.05$), but no differences in rancid character were detected during storage of samples from the second run. Samples from run 3 were rated significantly more rancid at weeks 4 and 7 ($P \leq 0.05$). The increase in rancid character was more pronounced in samples from run 1, as confirmed by a slope of 1.22, than from run 2, with a slope of 0.294, and run 3, with a slope of 0.482 (Table 3).

Significant increases in painty odor were detected at weeks 6 and 7 in samples from run 1, at week 7 in samples from run 2, and at weeks 6 and 8 in samples from run 3 ($P \leq 0.05$). Again, slopes were less inclined for the two last runs, indicating less increase in paintiness with time. A painty character has been reported as indicative of lipid oxidation (23).

Statistical comparisons for each odor attribute among the three runs, based on nonoverlap of the 95% confidence intervals, indicated that run 1 had a significantly higher rancid character than run 3 starting at week 2 of storage. This significant difference was observed through week 7 when samples from run 1 were compared with samples from runs 2 and 3 ($P \leq 0.05$) (Table 3). Descriptor scores for metallic sensory attributes increased significantly at week 4 and continued through the end of the storage times examined in the experiment ($P \leq 0.05$). There were no differences between runs 2 and 3 for this sensory attribute. The painty character of samples from run 1 increased significantly at week 4 compared with samples from runs 2 and 3 ($P \leq 0.05$). This significant difference prevailed through the end of the storage period. However, at weeks 6, 7, and 8, samples from run 3 were rated as more painty than samples from run 2 ($P \leq 0.05$). It has been reported that hexanal concentration is positively correlated with painty odors resulting from soybean oil oxidation (24). It is possible that, although the hexanal concentration was not significantly different between runs 2 and 3 at the storage times mentioned, the panelists were able to detect higher hexanal concentrations in samples from run 3.

Odor attributes were correlated with hexanal chemical determinations. Correlation coefficients between the odor descriptors and hexanal values are given in Table 4. Run 1 had significantly high correlations between metallic, rancid, and painty odor descriptors and the hexanal concentration ($P \leq 0.05$). These data suggest that hexanal was a good predictor of the sensory characteristics of the composites during storage for the first run. High correlations between a painty odor and hexanal were reported by Eldrid *et al.* (25) during sensory and chemical studies of lipid oxidation in raw and heat-treated oat flours. On the other hand, the lack of significance in the correlations between instrumental data and odor scores for runs 2 and 3 indicates that with slower rates of oxidation, hexanal is not a good predictor of sensory ratings of starch–oil composites during storage.

We concluded from this study that the initial run of a liquid starch–oil composite on a drum dryer results in metal

TABLE 4
Regression Models and Correlation Coefficients Between Mean Odor Descriptors and Hexanal Concentration in Stored Starch–Oil Composites

Descriptor	Model[a]	r^2	r	P-value
Run 1				
Metallic	$Y = 0.117 + 0.061X$ (0.188) (0.016)	0.782	0.884	≤0.05
Rancid	$Y = -0.413 + 0.333X$ (0.655) (0.054)	0.902	0.950	≤0.05
Painty	$Y = -0.387 + 0.264X$ (0.806) (0.067)	0.786	0.887	≤0.05
Run 2				
Metallic	$Y = 0.141 + 0.018X$ (0.116) (0.016)	0.057	0.239	NS
Rancid	$Y = 0.527 + 0.179X$ (0.626) (0.087)	0.394	0.628	NS
Painty	$Y = 0.011 + 0.09X$ (0.228) (0.032)	0.638	0.799	NS
Run 3				
Metallic	$Y = 0.158 + 0.014X$ (0.32) (0.0037)	0.743	0.862	NS
Rancid	$Y = 0.308 + 0.251$ (0.517) (0.060)	0.767	0.876	NS
Painty	$Y = 0.303 + 0.053X$ (0.454) (0.053)	0.370	0.608	NS

[a] Model Y = descriptor intensity; X = hexanal concentration (ppm). Values in parentheses are SD of regression coefficients. NS, nonsignificant; r, Pearson correlation coefficient; P-value, significance levels of regression models; r^2, coefficient of determination.

contamination of the product, which causes an increase in the rate of oxidation. When the oxidation rate is as low as the ones observed in the second and third runs, hexanal cannot be used to predict a consumer's response to the stored product. The marked contrast in the accumulation rates of hexanal and the negative odor attributes of the first run vs. subsequent runs suggests that conditioning or coating the metal drum-dryer surfaces with a film of oil from the initial product protects subsequent product from metal contamination. The relatively low rates of oxidation seen in the second and third runs, after storage at 37°C for 9 wk, suggest that the useful shelf life of drum-dried starch–oil composites, when they are stored under more favorable conditions (such as under refrigeration), may be much longer.

ACKNOWLEDGMENT

The authors thank Dr. Jonathan Maynes (Central Soya) for supplying the soybean oil used in this study.

REFERENCES

1. Knutson, C.A., K. Eskins, and G.F. Fanta, Composition and Oil-Retaining Capacity of Jet-Cooked Starch–Oil Composites, *Cereal Chem. 73*:185–188 (1996).
2. Eskins, K., G.F. Fanta, F.C. Felker, and F.L. Baker, Ultrastructural Studies on Microencapsulated Oil Droplets in Aqueous Gels and Dried Films of a New Starch–Oil Composite, *Carbohydr. Polym. 29*:233–239 (1996).
3. Fanta, G.F., F.C. Felker, K. Eskins, and F.L. Baker, Aqueous Starch–Oil Dispersions Prepared by Steam Jet Cooking. Starch Films at the Oil–Water Interface, *Ibid. 39*:25–35 (1999).
4. Fanta, G.F., and K. Eskins, Stable Starch–Oil Compositions Prepared by Steam Jet Cooking, *Ibid. 28*:171–175 (1995).
5. Eskins, K., and G.F. Fanta, Fantesk: Carbohydrate–Oil Composites with Low-Fat Foods, Cosmetics, Drugs and Industrial Applications, *Lipid Technol. 8*:53–57 (1996).
6. Garzón, G.A., C.S. Gaines, and D.E. Palmquist, Use of Wheat Flour–Lipid and Waxy Maize Starch–Lipid Composites in Wire-Cut Formula Cookies, *J. Food Sci. 68*:654–659 (2003).
7. Byars, J.A., Effect of a Starch–Lipid Fat Replacer on the Rheology of Soft-Serve Ice Cream, *Ibid. 67*:2177–2182 (2002).
8. Warner, K., K. Eskins, G.F. Fanta, T.C. Nelsen, and J.W. Rocke, Use of Starch–Lipid Composites in Low-Fat Ground Beef Products, *Food Technol. 55*:36–41 (2001).
9. Garzón, G.A., F.K. McKeith, J.P. Gooding, F.C. Felker, D.E. Palmquist, and M.S. Brewer, Use of Starch–Lipid Composites in Reduced-Fat Meat Patties, *J. Food Sci. 68*:1–7 (2003).
10. Evans, C.D., A.W. Schwab, H.A. Moser, J.E. Hawley, and E.H. Melvin, The Flavor Problem of Soybean Oil. VII. Effect of Trace Metals, *J. Am. Oil Chem. Soc. 28*:68–73 (1951).
11. Klem, R.E., and D.A. Brogley, Methods for Selecting the Optimum Starch Binder Preparation System, *Pulp Paper 55*:98–103 (1981).
12. Eskins, K., and G.F. Fanta, Non-separable Starch Oil Composites, U.S. Patent 5,676,994 (1997).
13. Eskins, K., and G.F. Fanta, Non-separable Compositions of Starch and Water-Immiscible Organic Material, U.S. Patent 5,882,713 (1999).
14. AOCS, *Official Methods and Recommended Practices of the American Oil Chemists' Society*, 5th edn., AOCS Press, Champaign, 1998, Method Cd 8-53.
15. AOAC International, *Official Methods of AOAC International*, 17th edn. (rev. 2), AOAC International, Gaithersburg, MD, 2003, Methods 968.08 D(b), 990.08.
16. Kupranycz, D.B., M.A. Amer, and B.E. Baker, Effects of Thermal Oxidation on the Constitution of Butterfat, Butterfat Fraction, and Certain Vegetable Oils, *J. Am. Oil Chem. Soc. 63*:332–337 (1986).
17. Labuza, T.P., Kinetics of Lipid Oxidation in Foods, *Crit. Rev. Food Technol. 2*:355–405 (1971).
18. SAS, SAS PROC GLM, Statistics, Version 8.0; SAS Institute, Inc., Cary, NC, 2000.
19. Hakansson, B., and M. Jagerstad, The Effect of Thermal Inactivation of Lipoxygenase on the Stability of Vitamin E in Wheat, *J. Cereal Sci. 12*:177–185 (1990).
20. Wennermark, B., H. Ahlmen, and M. Jagerstad, Improved Vitamin E Retention by Using Freshly Milled Whole-Meal Wheat Flour During Drum Drying, *J. Agric. Food Chem. 42*:1348–1351 (1994).
21. Hardas, N., S. Danviriyakul, J.L. Foley, W.W. Nawar, and P. Chinachoti, Effect of Relative Humidity on the Oxidative and Physical Stability of Encapsulated Milk Fat, *J. Am. Oil Chem. Soc, 79*:151–158 (2002).
22. Hall, G., The Influence of Heat Processing Conditions on Lipid Oxidation in Wheat Whole Meal and White Flour, *in Cereal Science and Technology in Sweden: Proceedings from an International Symposium, June 13th–16th, 1988, Ystad, Sweden,* edited by N.-G. Asp, Lund, Sweden, 1989, pp. 286–291.
23. St. Angelo, A.J., Lipid Oxidation in Foods, *Crit. Rev. Food Sci. Nutr. 36*:175–224 (1996).
24. Brewer, M.S., J.D. Vega, and E.G. Perkins, Volatile Compounds and Sensory Characteristics of Frying Fats, *J. Food Lipids 6*:47–61 (1999).
25. Eldrid, L.M., E.M. Mangus, J.M. Bjørge, and A. Nilsson, Sensory and Chemical Studies of Lipid Oxidation in Raw and Heat-Treated Oat Flours, *Cereal Chem. 73*:579–587 (1996).

[Received March 17, 2004; accepted September 7, 2004]