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Effects of Linalyl Oleate on Soybean Oil Flavor and Quality in a Frying Application

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Abstract Bread pieces were fried at 180 °C in soybean oil (SBO) containing no additives (control), 0.1% linalyl oleate (LO), or 10 ppm methyl silicone (MS). After 2 h of heating, the MS-containing oil was the most stable, followed by the oil with LO and the control, based on conjugated dienoic acid percentage (CD) and the ratio of linoleate%/palmitate%. Oil extracted from the fresh fried bread showed similar, but not significant, trends for CD and PV. Fresh and stored (60 °C, 2 days) bread fried in LO-containing oil had less hexanal than the other two treatments, and the stored LO bread had less *t*,*c*- and *t*,*t*-2,4-heptadienal than the control. Fresh bread fried in LO-containing oil had a less rancid flavor than did the other two treatments, and the LO treatment had less fishy flavor than the control. In stored bread, the MS treatment was less rancid than the control. In oil extracted from the stored bread, the amounts of *t*,*c*-2,4-heptadienal and 2-decenal correlated ($p \leq 0.05$) with the amounts of individual unsaturated fatty acids and with CD, but only *t*,*c*-2,4-heptadienal correlated with the PV. The *t*,*c*-2,4-heptadienal correlated with individual Polyunsaturated fatty acids (PUFA) in freshly fried bread. In general, oil and fried bread had improved flavor quality and/or oil stability when they contained MS or LO.

Keywords Linalyl oleate · Oxidation inhibitor · Flavor · Soybean oil · Methyl silicone · Sensory evaluation · Volatile compounds

Introduction

Soybean oil (SBO) has a good nutritional profile because of its high proportion of unsaturated fatty acids (FA), but it has poor oxidative stability and is prone to flavor deterioration. The linoleate (18:2), and especially linolenate (18:3), in SBO oxidize quickly and are the major contributors to the poor stability of SBO [1]. Hydroperoxides formed by the oxidation of 18:2 and 18:3 can break down to many undesirable flavor compounds [1–3]. Oil quality loss through oxidation is important, because the oil that cooks the food affects the food's final quality. To improve the oxidative stability and flavor quality of SBO, antioxidants and antifoaming agents may be added. Methyl silicone (MS) (dimethylpolysiloxane) is commonly used as an antifoaming agent in frying oils, and can extend the frying oil life. Various plant extracts [4, 5] and several plant sterols [6, 7] also reduce the chemical changes that occur in vegetable oils during frying. The activity of linalyl oleate (LO) as a frying oxidation inhibitor has been reported [8, 9]. LO additions at 0.05 and 0.1% were as effective as MS at 5 and 10 ppm [8] in slowing the formation of CD and the decrease in 18:2%/16:0% or 18:3%/16:0% during heating of soybean oil at 180 °C. Thus, the objectives of this study were to further evaluate the impact on oil and food quality and flavor of linalyl oleate added to the oil before frying.

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Materials and Methods

Materials

Bleached, deodorized soybean oil, containing citric acid as the only additive was a gift from ADM (Decatur, IL). The PV of the fresh soybean oil was 0.5 meq/kg by AOCS method Cd 8–53 [10]. White sandwich bread was purchased from a local market and cut into pieces (2.54 × 2.54 × 1.27 cm). Food-grade methyl silicone fluid (MS 0.97 g/mL at 25 °C, viscosity 350 centistokes) was a gift from Dow Corning Co., Midland, Michigan. Linalyl acetate and external standards for gas chromatography (GC) retention times were purchased from Sigma-Aldrich Chemical Co., Milwaukee, WI.

Synthesis of Linalyl Oleate

Methyl oleate (MO) was concentrated from olive oil by urea fractionation as described by Onal-Ulusoy et al. [8]. LO was synthesized by interesterification of linalyl acetate with methyl oleate, and the LO was purified by liquid chromatography to remove unreacted MO as described previously [9].

Frying Procedure and Oil Sampling

Six frying sessions, three per day, were conducted using three oil treatments: a control (no additives), oil containing 0.1% LO, and oil containing 10 ppm MS. Each treatment was duplicated and randomly assigned to a fryer. The LO or MS was dissolved in distilled ethanol, and the ethanol solution was added to a FryDaddy® fryer (model 05422, National Presto Industries, Eau Claire, WI). The ethanol was allowed to evaporate at room temperature overnight. The next day, 252 g of SBO was weighed into the fryers and heated to 180 ± 5 °C within 12 min and held at 180 ± 5 °C for 2 h. The temperature of each fryer was maintained by a variable transformer and monitored with a thermocouple. Before frying, 3 g of oil was removed and stored under nitrogen at 5 °C until analyzed for conjugated dienoic acid percentage (CD) and FA composition. Seven 12-piece batches of crust-free bread were fried for 2 min per batch at 4-min intervals. Thus, the frying of bread in one oil treatment was completed within 40 min. The first and last batches of the fried bread were not further analyzed because the first pieces had little flavor, and the last pieces had a very strong flavor. The remaining five batches of fried bread were pooled. Half of the pooled bread pieces were cooled to room temperature and immediately tested for sensory and volatiles analyses. The other half of the bread was stored, loosely covered, at 60 °C in the dark for two days before sensory and volatile analyses.

Fried bread used for chemical tests was placed in a Ziploc® freezer bag (Dow Brands, St. Louis, MO), flushed with nitrogen and stored at –10 °C until analyzed. For chemical tests, oils were extracted from bread by two 10-min extractions with high-performance liquid chromatography (HPLC)-grade hexane [11]. About 3–4 g of extracted oils was used for the chemical analyses, PV, CD and FA composition.

GC Analyses

Fatty acid methyl esters (FAMEs) of the frying oils were prepared by transesterifying the oils with sodium methoxide in methanol as described by Hammond [12]. GC conditions used for methyl oleate and linalyl oleate production, and FA composition were the same as described by Onal-Ulusoy et al. [8]. The decrease in the relative percentage of the polyunsaturated fatty acids and increase in saturated fatty acids previously was shown to correlate well with other measures of deterioration during frying [13].

PV of the SBO before Frying and of the Oil Extracted from Bread

PV was determined by the Stamm test as modified by Hamm et al. [14]. The method is useful in determining the initial quality of oil, and in measuring oxidation during storage of foods.

Conjugated Dienoic Acids

Conjugated dienoic acid percentages (CD) were measured by AOCS method Ti 1a-64 [14] with a Hitachi U-2000 model spectrophotometer. The method measures early changes in frying oils, and is particularly useful when comparing treatments applied all to the same oil source.

Sensory Evaluations of the Oils and Fried Bread

To determine panelists' abilities to detect any flavor arising from the LO and MS additives, two triangle tests were conducted on oils containing 0.1% LO and 10 ppm MS that were heated to 100 °C and cooled to room temperature.

Sensory evaluations of the fried bread pieces were conducted according to AOCS recommended practice Cg 2–83 [10]. A 12-member trained descriptive panel evaluated individual flavor and off-flavor intensities of the fried bread pieces. All panelists were trained during three 1-h sessions. During training, panelists were given definitions for five flavor descriptors, including nutty, buttery, fishy, rancid and painty flavors. Standards for these flavors, respectively, included cold-pressed and roasted peanut oil [15], soybean oil containing 0.05 ppm diacetyl, canola oil heated to 190 °C for 30 min [15], soybean oil aged at 0 °C for three years in the dark, and soybean oil aged at 25 °C for several months, until a strong painty flavor was exhibited. Training involved having panelists smell and taste a fried bread piece dipped into the designated flavor standard. The intensity scores were marked on a linear scale, similar to that used in the actual evaluations. Panelists were led in a discussion of their results in relation to the known samples.

For the actual tests, two bread pieces from three different treatments were presented to each panelist at each session. The bread was presented on plastic plates, labeled with random, three-digit codes, and given in random order to panelists in individual, lighted booths. Panelists smelled the pieces first and then tasted them. They were asked to expectorate the sample after tasting and to rinse their mouths with distilled water between tasting samples. The breads were evaluated for intensity of the five individual flavors on a 15-cm scale (from bland at 0 to extreme at 15). The intensity of a flavor was calculated as the average in centimeters among the panelists who detected that flavor in the sample.

Volatile Profile of the Bread Pieces by GC-Solid-phase microextraction (SPME)

The procedure by Su et al. [3] was used. About 3.0 g finely ground fried bread was placed in a 20-mL flat-bottom headspace vial and sealed. A 2-cm 50/30 μm divinylbenzene/Carboxen/polydimethylsiloxane Stable-Flex fiber (Supelco, Bellefonte, PA) was inserted through the Teflon seal and held at 40 °C for 60 min. The fiber was removed from the vial and inserted into the injection port of a HP 5890 series II gas chromatograph equipped with a 30 m × 0.32 mm HP-5 column with a 0.25μm film thickness. The injection temperature was 250 °C and detector temperature was 270 °C. The initial column temperature of 30 °C was held for three min, programmed at 4 °C/min to 100 °C, followed by 8 °C/min to 220 °C and held for five minutes. After injection, the fiber remained in the injection port for desorption for 10 min in preparation for reuse. External standards were used to identify retention times for each flavor compound. For this procedure, 0.5 μl of a standard was injected into a bread piece fried in a fresh control soybean oil, and the volatiles were determined by GC, and the retention times of the changes in the amounts of volatiles from the ground bread were noted.

Statistical Analysis

All data are the average of replicate experiments and replicate analyses. Data were analyzed statistically by using the analysis-of-variance general linear model of the SPSS 9.0 software package [16]. Differences in mean values among treatments were determined by LSD at $\alpha = 0.05$.

Results and Discussion

CD

The control oil had the greatest CD ($p \le 0.05$) among the treatments at 2 h of heating, followed by the LO and then the MS treatments (Table 1). The CD of oil extracted from fresh fried bread was lowest in the MS treatment. After storage, oils extracted from bread fried in oils with either LO or MS had lower CD than the control, and the MS treatment had the lowest CD. In oil from both the fresh and stored bread pieces, the correlation ($p \le 0.05$) between CD and unsaturated fatty acids was positive for 18:1 (R^2 0.74 for fresh bread, 0.96 for stored bread), but negative for 18:2 (R^2 –0.88 for fresh bread, –0.96 for stored bread), 18:3 (R^2 –0.83 for fresh bread, –0.98 for stored bread) and for the ratio of 18:2%/16:0% (R^2 –0.70 for fresh bread, –0.97 for stored bread; Table 2), The relationship is logical, considering that CD are some of the first degradation products formed from 18:2 and 18:3 during heating [17], and a decrease in 18:2 and 18:3 leads to an increase in 18:1.

PV of the Fresh Oils and of the Oils Extracted from the Fried Bread

The PV of the oil with the LO was not different from the other treatments in the fresh fried bread, and was intermediate between the values for the control and MS treatments after storage. SBO treated with MS after extraction from fresh and stored fried bread, had a lower PV than did the control (Table 1). Storage of the bread increased the PV of the control, LO and MS treatments by about six, six and sevenfold, respectively. The increase in PV was correlated with the decrease in 18:2% or 18:3% for both oils extracted from fresh fried

Table 1 Quality parameters and FAME composition of oil from the fryers and extracted from fried bread pieces

Test protocol treatment	CD[b]	PV	FAME (% relative area)					
			C16:0	C18:0	C18:1	C18:2	C18:3	C18:2/C16:0
Fresh Oil	0.20	0.5	10.4	4.4	23.4	52.5	7.2	5.1
Oil heated (2 h)								
SBO	1.46 (c)	NA	10.9 (c)	4.6 (c)	24.1 (c)	51.5 (a)	6.7 (a)	4.7 (a)
SBO + 0.1% LO	1.01 (b)	NA	10.8 (b)	4.6 (b)	23.9 (b)	51.7 (a)	6.8 (b)	4.8 (b)
SBO + 10 ppm MS	0.57 (a)	NA	10.4 (a)	4.5 (a)	23.5 (a)	52.1 (b)	7.1 (c)	5.0 (b)
Fresh bread pieces								
SBO	1.29 (b)	6.8 (b)	11.1 (b)	4.7 (c)	24.5 (c)	51.2 (a)	6.5 (a)	4.6 (a)
SBO + 0.1% LO	1.33 (b)	5.0 (a,b)	10.8 (a)	4.6 (b)	24.1 (b)	51.4 (a)	6.7 (b)	4.8 (b)
SBO + 10 ppm MS	0.71 (a)	3.1 (a)	10.8 (a)	4.6 (a)	23.9 (a)	52.3 (b)	6.9 (c)	4.9 (c)
Stored bread pieces[a]								
SBO	1.79 (c)	40.6 (c)	11.2 (c)	4.8 (c)	24.6 (c)	50.7 (a)	6.4 (a)	4.5 (a)
SBO + 0.1% LO	1.47 (b)	29.7 (b)	10.9 (b)	4.7 (b)	24.3 (b)	51.4 (b)	6.6 (b)	4.7 (b)
SBO + 10 ppm MS	0.93 (a)	22.4 (a)	10.6 (a)	4.5 (a)	23.8 (a)	52.1 (c)	6.9 (c)	4.9 (c)

[a] Fried bread pieces stored for 2 days at 60 °C in the dark

[b] *CD* Conjugated dienoic acid as percentage, *PV* meq/Kg, *SBO* Soybean oil, *LO* Linalyl oleate, *MS* Methyl silicone, *NA* not analyzed. Values in the same column for each test protocol with different letters within parentheses were significantly different ($p \le 0.05$)

Table 2 Correlations among various quality parameters in oils extracted from fried bread pieces

		Fresh bread pieces		Stored bread pieces	
		Correlation coefficient	*p* value	Correlation coefficient	*p* value
Correlation A	FAME				
CD	18:1	0.739	0.003*	0.960	0.000*
	18:2	–0.879	0.000*	–0.959	0.000*
	18:3	–0.830	0.000*	–0.982	0.000*
	18:2/16:0	–0.699	0.006*	–0.974	0.000*
PV	18:1	0.884	0.000*	0.908	0.000*
	18:2	–0.688	0.007*	–0.896	0.000*
	18:3	–0.844	0.000*	–0.921	0.000*
	18:2/16:0	–0.881	0.000*	–0.939	0.000*
t,*c*-2,4-Heptadienal	18:1	0.416	0.090	0.630	0.014*
	18:2	–0.532	0.038*	–0.689	0.007*
	18:3	–0.500	0.049*	–0.646	0.012*
t-2-Decenal	18:1	0.225	0.241	0.520	0.042*
	18:2	–0.377	0.113	–0.620	0.019*
	18:3	–0.335	0.144	–0.540	0.035*
Correlation B					
CD	*t*,*c*-2,4-Heptadienal	0.343	0.137	0.725	0.004*
	t-2-Decenal	0.447	0.072	0.649	0.011*
PV	*t*,*c*-2,4-Heptadienal	0.407	0.095	0.696	0.006*
	t-2-Decenal	0.191	0.552	0.476	0.059

Abbreviations are defined in Table 1

* Correlation is significant at $p \le 0.05$ levels

(R^2 –0.69 and –0.84, respectively) and stored fried (R^2 –0.90 and –0.92, respectively) bread (Table 2). These correlations also make sense, because peroxides are formed especially from 18:2 and 18:3 [18].

FA Composition

Significant differences in FAME percentages among treatments are shown in Table 1. For all treatments, including oils and fried breads, the polyunsaturated percentages (18:2 and 18:3) tended to decrease, whereas the saturated percentages (16:0 and 18:0) and monounsaturated percentage (18:1) tended to increase. LO and MS treatments had significantly lower 16:0%, 18:0% and 18:1%, and greater 18:2% than the control, although 18:2% did not always achieve significance for LO in heated oil and oil from freshly fried bread. The change in 18:2 and 16:0 percentages can be used as indicators of the extent of fat deterioration because linoleate esters are susceptible to oxidation, whereas palmitate esters are stable. The 18:2%/16:0% has been reported to correlate with the iodine value and dielectric constant of oxidized oils [13].

Sensory Evaluations (Table 3)

Two panelists out of 12 reported a difference between fresh oil and fresh oil treated with LO or MS (data not shown). According to Altug [19], 9 out of 12 positive answers are required to determine a significant difference between groups; thus, we concluded that no flavor contribution from the additives was detected.

The fresh and stored bread fried in oil with MS tended to have the lowest nutty, buttery, and painty flavors, but the differences were not significant. In fact, the intensity of all the flavors detected by the panelists was not great, with values ranging from 0.3 to 3.6 on a 15-point scale. Fresh bread fried in oil containing LO had less rancid flavor than the control and the MS treatment, and tended to be less fishy than the control. After two days storage at 60 °C in the dark, rancid and painty flavors tended to be greatest in the control, followed by the LO and the MS treatments. The MS treatment scored significantly lower than the control for rancid flavor.

Volatile Profiles of the Fried Bread by GC-SPME

Both the fresh and stored bread fried in oil with LO had significantly less hexanal, *t*,*c*-2,4-heptadienal and less (although not significantly less than for fresh bread) *t*,*t*-2,4-heptadienal than those fried in the control (Fig. 1a, b). Fresh bread fried in oil with MS had less *t*,*c*-2,4-heptadienal than the control (Fig. 1a). After storage, bread fried in oil with MS had less hexanal and *t*,*c*-2,4-heptadienal than the control. Hexanal was the most abundant volatile, followed by *t*,*t*-2,4-decadienal, in both fresh and stored bread fried in all three oil treatments. Hexanal is dominated by green, oily and fatty characteristics [2]. 2,4-Decadienal is one of the primary volatile compounds of heated linoleate hydroperoxides and also the major contributor to deep-fried flavor [18]. Among the volatile compounds, *t*,*c*-2,4-decadienal is the most flavorful, followed in order by *t*,*t*-2,4-decadienal, *t*,*c*-2,4-heptadienal, 1-octen-3-ol, *n*-butanol, *n*-hexanal, *t*,*t*-2,4-heptadienal, 2-heptanal, *n*-heptanal, *n*-nonanal, and 2-hexenal [3].

Table 3 Flavor characteristics of fried bread pieces

Fried bread pieces test protocol treatment[a]	Nutty	Buttery	Fishy	Rancid	Painty
Fresh					
SBO	1.1 (a)	1.6 (a)	0.9 (b)	2.1 (b)	0.6 (a)
SBO + 0.1% LO	1.0 (a)	1.5 (a)	0.3 (a)	0.7 (a)	0.5 (a)
SBO + 10 ppm MS	0.9 (a)	1.2 (a)	0.6 (a,b)	1.7 (b)	0.4 (a)
Stored					
SBO	0.4 (a)	0.7 (a)	1.1 (a)	3.6 (b)	2.0(a)
SBO + 0.1% LO	0.5 (a)	0.7 (a)	1.3 (a)	3.1 (a,b)	1.6 (a)
SBO + 10 ppm MS	0.3 (a)	0.6 (a)	0.8 (a)	1.8 (a)	0.8 (a)

Mean values obtained from a panel of 12 members. For intensity of individual flavors, *15* extreme, *0* bland

Values in the same column within the same test protocol group with different letters within parentheses were significantly different ($p \leq 0.05$)

[a] See Table 1 for abbreviations

Data Correlations

There was a significant negative correlation between *t*,*c*-2,4-heptadienal and the percentages of 18:2 (R^2 – 0.53 for fresh bread, –0.69 for stored bread) and 18:3 (R^2 –0.50 for fresh bread, –0.65 for stored bread) in the extracted oil (Table 2). Oxidation of 18:3 produces 2,4-heptadienal [3]; thus, the more 18:3 that disappeared, the greater the amount of 2,4-heptadienal formed. The amount of *t*-2-decenal in the stored fried bread pieces positively correlated with the percentage of 18:1 (R^2 0.52) and negatively with 18:2 (R^2 –0.62) and 18:3 R^2 – 0.54). Since *t*-2-decenal arises from the oxidation of oleic acid [18] one would expect a negative correlation. *t*-2-Decenal, and *t*,*c*-2,4-heptadienal, seem to be correlated with the general level of oxidation, which results in an increase in 18:1 and a decrease in 18:2 and 18:3. The greater the CD in the oil extracted from stored bread, the greater the content of *t*,*c*-2,4-heptadienal and *t*-2-decenal (Table 2). The PV in the oil extracted from stored bread correlated with the content of the *t*,*c*-2,4-heptadienal (R^2 0.70).

Generally, greater amounts of hexanal, *t*,*c*-2,4-heptadienal and *t*-2-decenal were indicators of loss of the favorable odors (buttery and nutty) and development of off-flavors, such as fishy, rancid and painty. No significant correlations were found between individual flavors of fresh fried and either individual volatile compounds or unsaturated fatty acids (18:1,18:2 and 18:3) at $p \leq 0.05$ (data not shown). This situation was also true for stored fried bread. Brewer et al. [2] reported correlations between buttery, rancid or painty flavor and hexanal, and between fishy and *t*,*t*-2,4-decadienal for potatoes fried in typical soybean oil treated with filter aids. In the current study, oil and fried bread generally had improved flavor quality and/or oil stability when they contained MS and LO. Further work is needed to examine ways to enhance the inhibitory effect of linalool or other active terpenes.

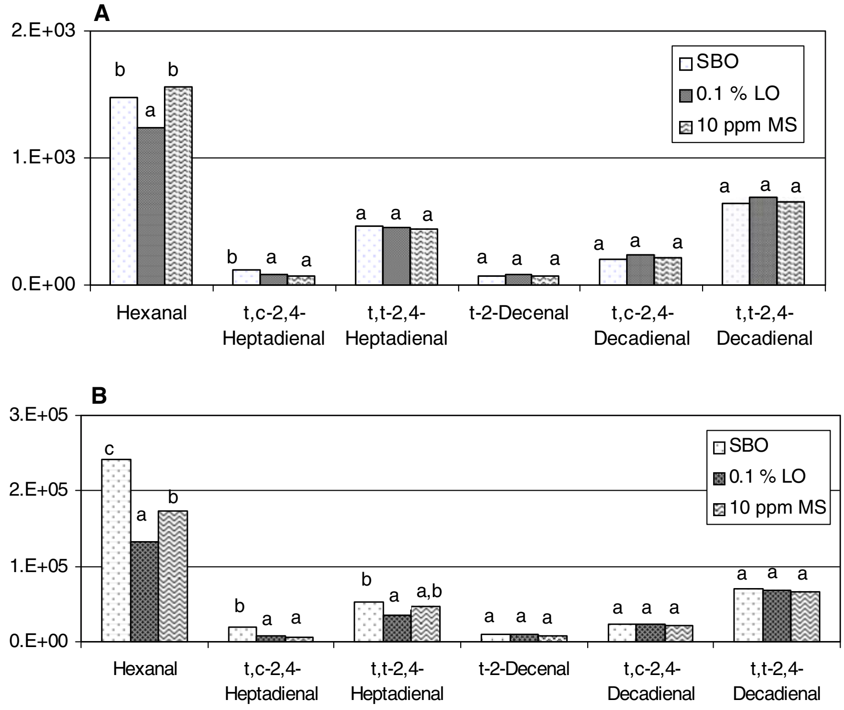

Fig. 1 Volatile compounds from **a** fresh and **b** stored fried bread pieces· For each volatile compound, values with label letters in common were not significantly different ($p > 0.05$). *Y*-axis units: GC area count

In general, the LO was less effective in the frying tests than MS in preventing CD and FAME composition changes, although in our previous tests LO and MS stabilized the soybean oil similarly at the concentrations used [8]. The primary difference in the present conditions and those used previously are the presence of the bread pieces, the boiling action that accompanied frying and the agitation entailed in adding and removing the bread pieces. It is well know that if MS is used in a constantly agitated oxidation test, such as the active oxygen test, it loses its ability to inhibit fat oxidation [20]. Observations in our laboratory with LO indicate that it shows a similar lack of effect in active oxygen conditions. These results might be explained by the theory that MS and LO work by accumulating on the oil surface and forming a partial oxygen barrier. If this is so, these compounds should be less effective in a mildly agitated oil than in the still-oil comparisons we have used in our previous experiments. The present experiments also suggest that LO may recover from agitation more slowly than MS. The tendency and rate of accumulation of these compounds on a surface may also explain why a much higher concentration of LO is needed than MS even in still-oil oxidation.

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