Flavor and Oxidative Stability of Soybean, Sunflower and Low Erucic Acid Rapeseed Oils[1]

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Three samples each of soybean, sunflower and low erucic acid rapeseed (LEAR) oils were evaluated for flavor and oxidative stability. The commercially refined and bleached oils were deodorized under identical conditions. No significant differences were noted in initial flavor quality. After storage at 25°C or 60°C in the dark, soybean oils—with or without citric acid—were more stable than either sunflower or LEAR oils. However, in the presence of citric acid, soybean oils were significantly less stable to light exposure than either LEAR or sunflower oils. In contrast, in the absence of citric acid, soybean oils were significantly more light stable than LEAR oils. In either the presence or absence of citric acid, sunflower oil was significantly more stable to light than soybean oil. Analyses by static headspace gas chromatography showed no significant differences in formation of total volatile compounds between soybean and LEAR oils. However, both oils developed significantly less total volatiles than the sunflower oils. Each oil type varied in flavor and oxidative stability depending on the oxidation method (light vs dark storage, absence vs presence of citric acid, 100°C vs 60°C).

Soybean oil (SBO), sunflower oil (SFO) and low erucic acid rapeseed oil (LEAR) accounted for 60% of the world production of edible vegetable oils in 1986 (1). In the United States, SBO is the major edible oil used in margarines (83%), salad and cooking oils (80%), solid shortenings (62%), and salad dressings (90%) (2). In addition, SBO is used in many frozen foods or packaged dry mixes. Increased SFO production in the U.S., and approval of LEAR for food use by the Food and Drug Administration in 1985 (3), has generated interest in the use of SFO and LEAR for salad and cooking oils and as ingredients in formulated foods. Previous research on the oxidative stability of SBO (4-7), SFO (8-10) and LEAR (11-14) has been reported, although few studies on the flavor evaluation of autoxidized LEAR are in the literature. Several researchers have evaluated the stability of vegetable oils by the active oxygen method (A.O.M.) and automated or modified A.O.M. methods conducted at temperatures of 100°C or higher (15-26) with peroxide values (PV) of 50 and above as the endpoints. deMan and deMan (22) reported that the PV at A.O.M. endpoints for LEAR and corn oil were 95 and 225 meq/kg, respectively. Erkilla (13) found that LEAR had a PV above 50 at the end of the AOM induction period as determined by refractive index measurements. Based on reports in the literature on flavor data and PV, the levels of deterioration produced under A.O.M. test conditions are too high to have any relation to flavor quality in shelf life studies. In general, polyunsaturated oils show flavor deterioration at a PV of 5, which corresponds to less than 0.1% oxidation (27). Other work has shown that SBO was rancid at peroxide values of 20 (28) and that SFO was strong-flavored at a PV of 8 (9) or was off-flavored at a PV of 13 (10). Weiss (29) reported that oils with PV as low as 2 meq/kg were rancid. Therefore, stability tests should be conducted at low levels of oxidation to be relevant to flavor deterioration, and the instrumental or chemical methods used to measure the oxidation should be sensitive enough to detect less than 1% oxidation in order to develop correlations with flavor evaluation.

Rapid methods are needed to assess oil stability. Traditionally, a modified Schaal oven test is used to oxidize oils at 60°C to predict their flavor and oxidative stability (28). Tests reported by Evans et al. (30) showed that the flavor scores of oils aged four days at 60°C were equivalent to scores for oils aged four mo at ambient temperature. However, oil processors would like to predict the stability of an oil within hours after the processing run.

The objective of this research was to compare the flavor and oxidative stability of SBO, SFO and LEAR after deodorizing and aging under identical conditions. A variety of aging conditions were conducted at 25°C, 60°C, 80°C and 100°C in the dark, as well as storage under fluorescent light at 30°C. Methods to measure oxidative stability based on volatiles, PV, AOM and Rancimat, were correlated with sensory analyses. In addition, rapid methods were developed to evaluate oil stability.

MATERIALS AND METHODS

Materials. Three samples each of commercially refined and bleached SBO, SFO and LEAR were obtained from nine processing plants over a six-mo period. The oils were selected to reflect a variety of geographical locations, processing conditions prior to deodorization, and time of year for processing to better represent the quality of oil being produced at the time of the study. The oils were laboratory-deodorized at 220°C for three hr according to previously standardized procedures (31, 32). Citric acid (100 ppm as a 20% aqueous solution) was added to one-half of all oils on the cooling side of deodorization.

Storage conditions. Oil samples in eight-oz, narrow-mouthed, clear glass bottles were aged in the dark at 60°C in a forced-draft air oven, or under ambient conditions at 25°C, for stability tests based on sensory evaluation, GC-volatiles and PV analyses. Each bottler was first filled 2/3 with oil, leaving 1/3 of the bottle with air in the headspace, and then was loosely stoppered with a cellophane-covered cork. For stability tests based on volatile analyses only, oils were placed in

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either sealed 10-ml headspace vials (0.5 g oil), or nine-cm (diameter) covered glass petri dishes (5 g oil) and aged at 80°C for 2-24 hr. For light stability tests, oils in eight-oz, narrow-mouthed, clear glass bottles were exposed to fluorescent light at 7535 lux (700 ft candles) at 30°C (33). All samples were aged with air in the headspace.

Sensory evaluation. A 15-member trained panel experienced in tasting oils and fat-containing foods evaluated the oils for flavor on a 10-point intensity scoring scale with bland samples scored as 10 and strong-flavored oils as 1 (34, 35). Panelists entered flavor scores and descriptions directly into a mainframe computer from terminals located in each panel booth. Our methods of data handling by computer were described previously (36). In all sensory evaluations the LEAR and SFO were compared separately, with SBO as the undesignated control. To evaluate the flavor of the bleached nondeodorized oils, each oil was diluted (5:95) with deodorized oil and evaluated by the sensory panel for flavor characteristics as described previously (37).

Gas chromatographic volatile analyses. Volatile compounds of oils aged under each storage condition were analyzed in a Perkin-Elmer 8320 capillary gas chromatograph (GC), fitted with a flame ionization detector and equipped with a headspace analyzer (Model HS-6) (Perkin-Elmer Co., Oak Brook, Illinois). For volatiles analyses, 0.5-g samples of oil, in triplicate, were taken from each storage container—bottle or petri dish—at each storage period and were placed in 10-ml headspace vials. The vials were sealed with a teflon-lined septum and aluminum cap (38). The 0.5-g samples of oil aged in the sealed headspace vials, in triplicate for each storage period, were analyzed for volatiles in the same vials that were used for the storage test. Each vial was placed in the headspace analyzer and heated to 180°C for 10 min to generate volatiles. The volatile compounds formed were automatically injected onto a DB-5 fused-silica capillary column (30 m × 0.32 mm, 1 micron film thickness) (J & W Scientific, Rancho Cordova, California). The column temperature was programmed from 0-200°C at 5°C/min, after an initial 10-min hold at 0°C, and then from 200-240°C at 20°C/min. Other gas chromatographic (GC) conditions were: injector temperature, 200°C; detector temperature, 250°C; carrier gas, helium at flow rate of one ml/min. Volatile compounds were identified by matching retention times with those of authentic compounds. Identifications were confirmed by mass spectrometry (39).

Instrumental and chemical analyses. Fatty acid compositions of the oils were determined by gas chromatographic analyses in a Perkin-Elmer Sigma 3B GC (Oak Brook, Illinois) equipped with a wide-bore (30 m × 0.32 mm) capillary column (DB-225, J & W Scientific, Rancho Cordova, California) at 175°C. Spectrophotometric absorption measurements were obtained by using a Spectronic 2000 spectrophotometer (Fischer Scientific, Springfield, New Jersey). Chlorophyll contents of the oils were calculated from measurements at wavelengths of 710, 670 and 630 nm (Official AOCS method Cc 13d-55) (40), and carotenoid contents from wavelength measurements at 444 and 454 nm (41). PV was determined on 10-g aliquots of oil by AOCS Method Cd 8-53 (40). Oxidative stability was estimated at 100°C by both the Rancimat technique (25) and by AOM (AOCS Cd 12-57) (40). Iron contents in both the bleached and the deodorized oils were determined by atomic absorption spectroscopy (AOCS method Ca 15-75) (40).

RESULTS AND DISCUSSION

These experiments were designed to compare the stability of SBO to that of LEAR and SFO. All oils were deodorized under identical conditions. To evaluate stability differences, the oils were exposed to storage temperatures of 25°C, 60°C and 80°C in the dark and to fluorescent light at 30°C. Finally, all oils were evaluated for flavor by trained taste panelists and for oxidative deterioration by GC-volatiles, PV, AOM and Racimat. The fatty acid compositions of the oils varied only slightly within each oil type (Table 1). The compositions did vary significantly between oil types for oleic, linoleic and linolenic acid contents. Iodine values averaged 140 for SFO, 130 for SBO and 115 for LEAR. Free fatty acid contents averaged 0.09 for SBO, 0.10 for SFO and 0.13 for LEAR (Table 2). Beta carotene values for refined, bleached LEAR and SBO varied widely within each oil type (Table 2). Deodorization reduced carotene contents of all oils to below 1 ppm. Analyses of chlorophyll in the refined, bleached oils showed that SBO had the highest average content of 0.8 ppm, followed by SFO at 0.6 ppm and LEAR at 0.3 ppm (Table 2). Deodorization further reduced the chlo-

TABLE 1

Fatty Acid Compositions of Soybean, Sunflower and Low Erucic Acid Rapeseed (LEAR) Oils

	Soybean oil			LEAR oil			Sunflower oil		
Fatty acid composition	I[a]	II	III	I	II	III	I	II	III
C16:0	11.7	12.8	10.8	4.7	4.5	4.8	7.1	6.4	6.8
C18:0	3.5	3.3	3.8	1.5	1.3	1.7	4.4	4.1	3.9
C18:1	22.2	26.7	22.9	62.2	61.4	61.0	15.7	16.0	14.2
C18:2	55.3	51.5	54.5	20.7	21.0	22.4	72.8	73.2	74.5
C18:3	7.3	5.8	8.1	9.4	10.4	9.0	—	—	—
C22:1	—	—	—	1.1	1.3	1.2	—	—	—
Iodine values	133.	125.	133.	114.	116.	114.	139.	140.	141.

[a]Roman numerals indicate different samples of each oil type.

TABLE 2

Instrumental and Chemical Analyses of Refined, Bleached (RB) and Refined, Bleached, Deodorized (RBD) Oils

	Soybean oil			LEAR oil[a]			Sunflower oil		
Analyses	I	II	III	I	II	III	I	II	III
Free fatty acids, RB (%)	.07	.08	.11	.12	.12	.16	.10	.10	.10
Carotene (PPM)									
RB	2.91	4.72	15.01	63.63	6.57	5.74	.56	.71	1.10
RBD	.29	.80	.33	.93	.56	.48	.14	.25	.34
Chlorophyll (PPM)									
RB	.01	.16	.06	.06	.02	0.	.02	.04	.11
RBD	0.	.13	.05	.01	0.	0.	.02	.03	.08
Iron (PPM)									
RB	.77	.99	.79	.50	.61	.45	.56	.69	.51
RBD	.52	.83	.62	.57	.57	.47	.52	.61	.62

[a]Low erucic acid rapeseed oil.

rophyll contents of all oils to 0.13 ppm or less. Iron contents in the refined, bleached oils averaged 0.85 ppm for SBO, 0.59 for SFO and 0.52 for LEAR (Table 2). The iron levels in the deodorized oils changed only slightly from the amounts in the bleached oils.

Flavor stability. Initial evaluations of oils processed with and without citric acid showed no significant differences in flavor scores between SBO and SFO or LEAR (Table 3). As expected, oils without citric acid had lower initial flavor scores than oils containing citric acid. After storage at 60°C in the dark, SBO containing citric acid had significantly higher flavor scores ($P < 0.05$) than the corresponding SFO and LEAR in 10 of 11 trials (Table 3).

After storage at 60°C in the dark for two and four days, SBO without citric acid had significantly higher flavor scores ($P < 0.05$) than the LEAR oils (Table 3). SBO also had significantly higher flavor scores ($P < 0.05$) than the SFO in three of the four comparisons. SFO had significantly higher scores ($P < 0.05$) than the LEAR oils in three of four comparisons. These latter results for oils without citric acid were in contrast to those for oils with citric acid. The LEAR and SFO can be compared indirectly, because they were tested separately with the same SBO control. In the presence of citric acid, all LEAR, except LEAR II, received scores slightly higher than those for the corresponding SFO.

After light exposure of oils containing citric acid, SBO had significantly lower flavor scores ($P < 0.05$) than the corresponding SFO and LEAR (Table 4). The scores for the LEAR and SFO were similar. In contrast, in the absence of citric acid, light-exposed LEAR had significantly lower flavor scores than SBO in 3 of 4 comparisons (Table 4). Light-exposed SFO, on the other hand, was rated significantly higher than either SBO or LEAR. The cause for differences in light stability of LEAR oil in the absence or presence of citric acid may be due to the effect of minor constituents such as metals, although the average iron content is higher for the SBO than for the LEAR oil (Table 2). Other reasons for the change in light stability of LEAR oil in the presence or absence of citric acid were not specifically investigated in this study and will be the subject of future research. These results on oils containing citric acid agree with those reported by Sattar et al. (42). They evaluated the flavor and oxidative stability of LEAR and SBO exposed to 5400 lux and found that LEAR was significantly more light stable than SBO. However, they did not indicate whether their oils contained citric acid.

All freshly deodorized oils tasted nutty and buttery. Each of the three oil types had distinct flavor characteristics during the early stages of oxidation, before the development of rancid and painty flavors. Aged SBO was described as grassy and beany, whereas LEAR had characteristic cabbage and sulfur flavors as well as a grassy flavor. SFO was described as pine/cedar, weedy and acrid. These flavors in slightly aged oil were the same as those detected in diluted, bleached oil, but were at much lower intensity (37). In later stages of oxidation, all oils were described as rancid. In addition, the linolenic acid-containing SBO and LEAR were described as painty. The LEAR also had fishy flavors. The distinctive flavors that develop in oils as they age have been attributed to the decomposition products of the oxidized fatty acids (27). Photooxidized oils developed distinctly different flavors than did the oils autoxidized in the dark. SBO was described as grassy, sour, metallic or buttery. Light-exposed LEAR had flavor descriptions similar to those of SBO; these may be characteristic of linolenic acid-containing oils. On the other hand, light-exposed SFO was described as stale or sour.

The reproducibility of the data in Tables 3 and 4 can be evaluated by comparing the replicate scores of the aged control soybean oil samples. The variation in replicate scores ranged from 0 to 0.5 with averages of 0.15 for data in Table 3 and 0.22 for data in Table 4, which are indicative of good reproducibility. On the other hand, the variability of the data, which is apparent by comparing the scores of the three samples within each oil type, showed that oils varying in quality were obtained as had been planned. By obtaining oils varying in quality, the stability characteristics of these oils were shown to be representative of many of the oils produced today. Our results on oils aged in the dark at 60°C, in either the presence or absence of citric acid, showed that SBO had better flavor stability than the

TABLE 3

Effects of Storage at 60°C in the Dark on the Flavor Scores[a] of Soybean (SBO), Low Erucic Acid Rapeseed (LEAR) and Sunflower (SFO) Oils in the Presence and Absence of Citric Acid

	Series I			Series II			Series III			
60°C In Dark (Days)	SBO	LEAR	SFO	SBO	LEAR	SFO	SBO	LEAR	SFO	Sig.[b]
A. + 100 PPM Citric acid										
0	8.0	7.7	8.1							NS
				8.1	7.6	8.0				NS
							8.3	8.2	7.8	NS
4	6.7	6.4								NS
	6.7		5.4							**
				6.3	4.5					*
							6.9	6.0		*
							6.9		5.2	**
8	6.1	4.9								**
	6.1		4.8							**
				5.9	3.2					**
				5.7		4.9				*
							5.9	4.5		**
							5.4		4.0	**
B. No Citric acid										
0	7.6	7.3	7.1							NS
				6.7	6.3	6.5				NS
							7.3	7.5	7.9	NS
2				5.0	4.2					*
							6.4	4.0		**
							6.6		5.7	*
4	6.3	5.4								*
	6.2		5.4							*
				4.7	3.3					*
				4.7		5.3				NS
							6.4	3.2		**
							6.7		4.3	**

[a]Based on 10-1 scale; 10=bland, 1=strong intensity. Least significant difference, 0.8.
[b]NS, not significant; *, significant at 95% confidence level; **, significant at 99% confidence level.

SFO or LEAR. However, SBO was less stable to light than the other oils in the presence of citric acid, but more stable than the corresponding LEAR in the absence of citric acid.

Oxidative stability. Oils were evaluated for oxidative stability based on PV, AOM, Rancimat and GC volatiles techniques. The PV presented in Table 5 were obtained from the same oil samples that were aged at 60°C and evaluated by the sensory panel (Table 3). After four days of storage, the SBO and LEAR showed few differences in PV. However, the PV for SFO was significantly higher than those of the LEAR and SBO. The PV of oils aged eight days at 60°C averaged 8.3 for SBO, 11.0 for LEAR and 13.6 for SFO. The average value for SFO is skewed because of an unusually high PV for SFO III. In addition to the PV of oils obtained at the time of taste panel evaluation, we determined induction periods based on PV development (28) (Fig. 1). At 60°C SFO in the presence of citric acid had the shortest induction period, i.e., four days, followed by LEAR with an induction period of five days, and SBO with an induction period of six days. Under the same test conditions, oils in the absence of citric acid showed greater differences in stability between oil types. Both SFO and LEAR had induction periods of less than one day, whereas the corresponding SBO had an induction period of five days.

PV measurements of light-exposed oils showed few differences between oil types. Sattar et al. (43, 44) previously reported significantly higher PV for light-exposed (5400 lux) LEAR than for SBO aged under the same conditions. In that study, the PV of the oils exposed to light for 12 hr were 13 for LEAR and 4 for SBO.

Evaluations of the oils with the Rancimat and under AOM conditions were similar (Table 5). The LEAR had the longest induction periods with an average of 16.9 hr under Rancimat conditions, followed by 15.9 hr for SBO and 11.75 hr for SFO. The induction periods determined under AOM conditions were 16.7 hr, 14.2 hr and 13.5 hr for LEAR, SBO and SFO, respectively. A correlation coefficient calculated between the two methods was 0.78, which was significantly ($P< 0.05$) below the correlation coefficient of 0.98 reported by Laubli and Bruttel (25) between Rancimat and AOM results for six oils and fats.

Induction periods were also determined by GC volatiles analyses on all oils treated with citric acid (Fig. 2a-c). Three conditions of accelerated storage were used to determine oxidative stability based on volatiles. Oils aged in the eight-oz bottles at 60°C (Fig. 2a) were the same as those used for the PV tests (Table 5)

TABLE 4

Effects of Light Exposure on the Flavor Scores[a] of Soybean (SBO), Low Erucic Acid Rapeseed (LEAR) and Sunflower (SFO) Oils in the Presence and Absence of Citric Acid

	Series I			Series II			Series III			
30°C In light 7500 lux (hr)	SBO	LEAR	SFO	SBO	LEAR	SFO	SBO	LEAR	SFO	Sig.[b]
A. + 100 PPM Citric acid										
0	8.0	7.7	8.1							NS
				8.1	7.6	8.0				NS
							8.3	8.2	7.8	NS
8	4.8	6.9								**
	5.2		7.2							**
				4.8	6.0					*
				5.0		7.1				**
							5.4	7.4		**
							5.2		6.5	*
16	4.6	6.8								**
	4.8		6.3							**
B. No Citric acid										
0	7.6	7.3	7.1							NS
				6.7	6.3	6.5				NS
							7.3	7.5	7.9	NS
4	4.8	4.7								NS
	4.8		6.5							**
				4.9	3.6					**
				5.3		6.6				**
							4.9	4.0		*
							4.8		6.5	**
8	4.8	3.5								**
	5.1		6.0							*

[a]Based on 10-1 scale; 10=bland, 1=strong intensity. Least significant difference, 0.8.
[b]NS, not significant; *, significant at 95% confidence level; **, significant at 99% confidence level.

TABLE 5

Effects of Storage at 60°C in the Dark on Peroxide Values[a] and Effects of AOM and Automated AOM Tests on Induction Period Endpoints for Soybean, Low Erucic Acid Rapeseed (LEAR) and Sunflower Oils[b]

	Peroxide values (meq/kg)								
	Soybean oil			LEAR oil			Sunflower oil		
60°C	I	II	III	I	II	III	I	II	III
0 days	0.	0.	0.	0.	0.	0.	0.	0.	0.
4 days	1.3	1.3	1.5	1.0	2.3	1.2	6.6	--[c]	2.4
8 days	7.6	7.6	9.7	7.7	13.8	11.5	9.0	10.8	21.0
	Induction periods (hr)								
100 C									
AOM	13.5	15.0	14.0	16.5	17.0	16.6	12.2	15.8	12.4
Rancimat	15.0	17.25	15.5	18.25	15.25	17.25	10.75	13.5	11.0

[a]Determined at time of flavor evaluation.
[b]Oils contain 100 ppm citric acid.
[c]Not determined.

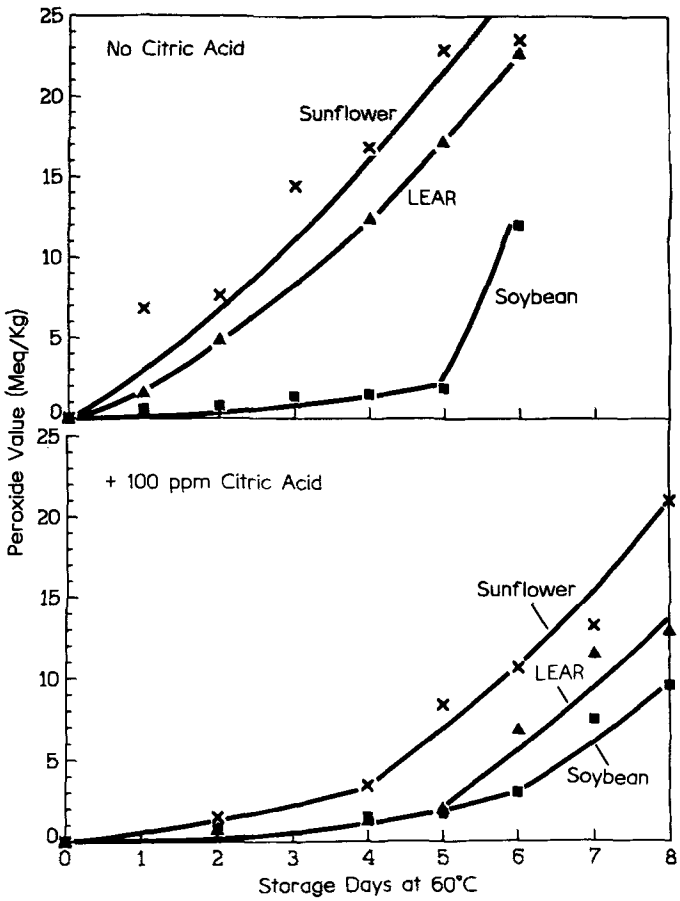

FIG. 1. Induction periods for peroxide development of soybean, low erucic acid rapeseed (LEAR) and sunflower oils with and without citric acid.

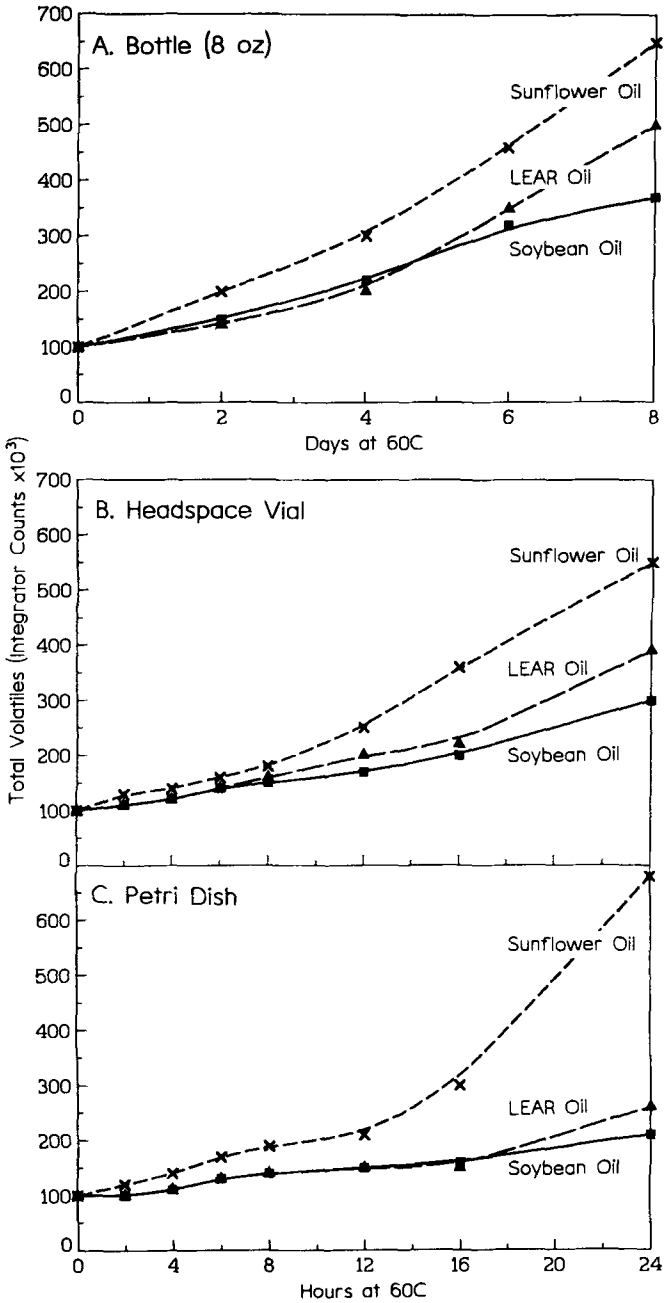

FIG. 2. Induction periods for development of volatiles by gas chromatographic headspace analyses in soybean, low erucic acid rapeseed (LEAR) and sunflower oils in the presence of citric acid: (a) aged at 60°C in 8-oz glass bottles; (b) aged at 80°C in 10-ml headspace vials, and (c) aged at 80°C in covered glass petri dishes.

and for the flavor tests (Table 3). The alternative conditions included storing the oils at 80°C or in small quantities to shorten the time of deterioration from eight days to 24 hr. Although the induction periods for the oils in eight-oz bottles did not have distinct endpoints, SBO and LEAR had similar rates of volatile formation during the first six days of storage (Fig. 2a). After six days, the volatile compounds began to develop at a faster rate in LEAR than in SBO, and the level of total volatiles was significantly higher ($P < 0.05$) in LEAR than in SBO. Volatiles in SFO formed at a significantly faster ($P < 0.05$) rate than for either LEAR or SBO. In order to predict the stability of vegetable oils in less than eight days, we aged either 0.5-g samples of oil in headspace vials at 80°C, or five-g samples of oil in covered glass petri dishes (9 cm diameter) at 80°C. The rate of volatile formation was significantly increased compared to the rate of formation of volatiles in oil stored in the eight-oz bottles. Within 24 hr of storage at 80°C, the amounts of volatiles formed in oils aged in the headspace vials (Fig. 2b) and in petri dishes (Fig. 2c) were equivalent to those formed in bottles after 8 days at 60°C. SFO developed significantly more ($P < 0.05$) total volatiles than either LEAR or SBO in the two oxidation tests at 80°C. These data agree with those obtained in the aging tests at 60°C. The induction periods of volatile compounds for both LEAR and SBO remained the same until after 16 hr of storage at 80°C. After that time, the slope for total volatiles in LEAR began to increase more than that of SBO. After 24 hr of storage, SBO had significantly less total volatiles than LEAR. Aging oils at 80°C for 24 hr in headspace vials (0.5 g) or petri dishes (5 g) was equivalent to aging 150 g of oil in an 8-oz glass bottle for eight days at 60°C. These results agree with the headspace analyses of Snyder et al. (38) who reported that SBO aged for eight days at 60°C developed 40% less volatiles than SFO.

In summary, this study showed marked differences in relative flavor quality and oxidative stability of SBO, SFO and LEAR, depending on aging conditions and type of analyses used to measure oxidative deterioration. SBO was significantly better in flavor stability and peroxide development than either SFO or LEAR, with or without citric acid, after storage in the dark at 60°C. Both SBO and LEAR developed significantly fewer total volatiles than the SFO when aged at 60°C. Stability tests under both Rancimat and AOM conditions at 100°C showed that the LEAR had slightly better oxidative stability than SBO. Therefore, at 60 and 80°C, SBO was more stable than LEAR, whereas at 100°C, LEAR was more stable than SBO.

In light-exposure tests, SFO and LEAR were significantly more stable than the SBO in the presence of citric acid. However, in the absence of citric acid, LEAR was less light-stable than SBO or SFO. In contrast, volatiles analyses of the light-exposed oils showed that both SBO and SFO developed more total volatiles than LEAR.

This study showed that valid comparisons of oils that vary widely in fatty acid composition as well as in minor constituents require a variety of both storage conditions—temperature and light—and evaluation methods—sensory, volatiles and peroxide values. More research on such factors as triglyceride structure, metal contaminants, pigments and other minor constituents is needed to help determine the causes for these differences in oil stability.

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