Flavor and Oxidative Stability of Northern-Grown Sunflower Seed Oil

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ABSTRACT

Flavor and oxidative stabilities of a northern-grown sunflower seed oil were investigated. Taste panel and oxidative evaluations were made on alkali-refined, deodorized, unbleached samples treated with commercial antioxidant mixtures, phenolic antioxidants, metal scavengers and added trace metals. Similar evaluations were conducted on a sample of the same oil after bleaching. Commercial antioxidant mixtures containing both phenolic antioxidants and a metal scavenger improve the flavor and oxidative stabilities of refined unbleached oil. Although phenolic antioxidants alone improve oxidative stability as measured by the active oxygen method test, flavor stability did not improve significantly for antioxidant-treated refined, unbleached samples after accelerated storage. Conversely, alkali-refined and bleached sunflower oil responded to treatment with certain phenolic antioxidants. Although iron and copper are deleterious to oil stability at concentrations of 0.1 ppm, such metal-inactivating agents as citric acid are effective in improving flavor stability.

INTRODUCTION

Sunflower is not a major oilseed crop of the U.S., but it has risen to major prominence in the world oilseed market, second only to soybean oil (1). Domestic interest in sunflower oil has increased considerably over the past few years and was stimulated by introduction of Russian varieties having high oil content and excellent agronomic characteristics (2,3).

Sunflower oil is a rich source of polyunsaturated fatty acids. Recent reports indicate that the oil may contain 65-75% linoleic acid with only traces (0.1-0.3%) of linolenate (3,4). Despite its rather high IV and semidrying properties, sunflower oil has been used primarily in edible shortenings, margarines and salad oils. Putt et al. (4) point out that sunflower oil may polymerize when heated and suggested that its use as a frying fat for potatoes and related food products may be restricted.

The effects of antioxidants in sunflower oil have been studied (5-7). However, except for a preliminary account of this work (8), little has been published in readily accessible journals on flavor stability of edible sunflower oils. We report here data on the flavor and oxidative stability of northern-grown sunflower seed oil.

MATERIALS AND METHODS

The sunflower oil used in this study came from seed grown in the Red River Valley of Minnesota during 1967 and was supplied as a commercially alkali-refined but unbleached sample. Its percentage fatty composition as determined by gas liquid chromatography (GLC) showed: palmitate, 6.6; stearate, 4.4; oleate, 15.7; linoleate, 73.3; and linolenate, a trace. Calculated IV was 140.5.

A portion of the oil was vacuum bleached in pilot plant equipment with 2% activated clay (Super Filtrol). In the laboratory the oil was bleached with 6% Super Filtrol. The oil was protected from air with nitrogen during filtering and packaging.

Deodorizations were carried out in an all glass, four unit deodorizer as described previously (9). Antioxidants, metal scavengers, metals and combinations thereof were added on the cooling side of deodorization, except where otherwise noted. 2,4,5-Trihydroxybutyrophenone (THBP), tertiary-butylhydroquinone (TBHQ), propyl gallate (PG) Tenox 6 (containing butylated hydroxyanisole [BHA], butylated hydroxytoluene [BHT], PG and citric acid) and Tenox 2 (containing BHA, PG and citric acid) were obtained from Eastman Chemical Products, Inc., Kingsport, Tenn. The G-50 antioxidant (containing BHT, BHA, PG and monoglyceride citrate) was purchased from Griffith Laboratories, Chicago, Ill. BHA and BHT came from Universal Oil Products Co., Chicago, Ill. Nordihydroguaiaretic acid (NDGA) came from Norigard Corporation, Chicago, Ill. Citric acid, copper nitrate and ferric chloride were reagent grade chemicals supplied by J.T. Baker Chemical Co., Phillipsburg, N.J.

Organoleptic evaluations were conducted by methods described by Moser et al. (10). Flavor data were supplied by a 20 member taste panel. Most results were obtained by comparison of two samples except for some initial evaluations of freshly deodorized oils.

Peroxide values were determined by a modification of the Wheeler method (11). Active oxygen determinations were carried out according to the AOCS official method (12). Tocopherol was determined according to Stern and Baxter (13), except 10 min was allowed for color development. Oil samples were treated at 210 C under nitrogen for 15 min before tocopherol analysis to remove any interfering peroxidic material (14). The mean tocopherol content of the refined and bleached oils based on three replicates each was 859 ± 17 μg/g and 869 ± 39 μg/g, respectively.

TABLE I

Flavor and Oxidative Stability of Sunflower Seed Oils Without Antioxidants or Metal Scavengers

Oil treatment	Storage condition: 0 Time	Storage condition: 4 days, 60 C	AOM 8 hr, PV
	Flavor score[a]	Flavor score[a]	
Refined, not bleached	7.8	5.6	39.0
Refined, bleached in pilot plant	7.9	4.4	102.4
Refined, bleached in lab	7.3	3.7	138.0

[a] Flavor scores and active oxygen method (AOM) peroxide values (PV) are pooled and averaged data from multiple tests.

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[1] N. Market. Nutr. Res. Div., ARS, USDA.

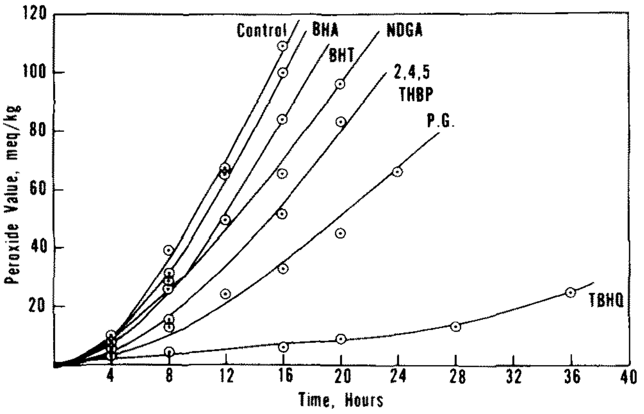

FIG. 1. Peroxide development in refined sunflower oil treated with antioxidants, active oxygen method (AOM) conditions. See Tables II and III for abbreviations.

Trace iron and copper were determined by atomic absorption spectroscopy (15). Flame emission data were provided by a private concern specializing in performing these analyses.

RESULTS

Flavor and oxidative stability data for deodorized sunflower oils having no antioxidants or metal-inactivating agents are presented in Table I. Included are pooled data for refined unbleached, pilot plant bleached and laboratory bleached samples. Hereafter the alkali-refined, unbleached oil will be referred to as refined oil, and the refined and bleached sample will be designated as bleached oil. Initially all three oils received acceptable flavor scores ranging from 7.6-8.0 for the refined samples and from 7.3-8.5 for bleached oils.

After 4 days' storage at 60 C both the refined and bleached oils had undergone considerable flavor deterioration. In every test conducted the refined samples was more stable than its bleached counterpart, as reflected in accelerated storage flavor scores and peroxide values determined by the 8 hr active oxygen method (AOM).

Results in Table I suggest that antioxidants, metal-inactivating agents or combinations thereof might be beneficial in improving the flavor and oxidative stability of edible sunflower oil. Accordingly we studied the effects of various stabilizers on the flavor and oxidative stability of refined and bleached oils.

We examined the effects of phenolic antioxidants on oxidative stability by aerating deodorized sunflower samples under AOM conditions for various periods of time. Peroxide values were determined and plotted against time.

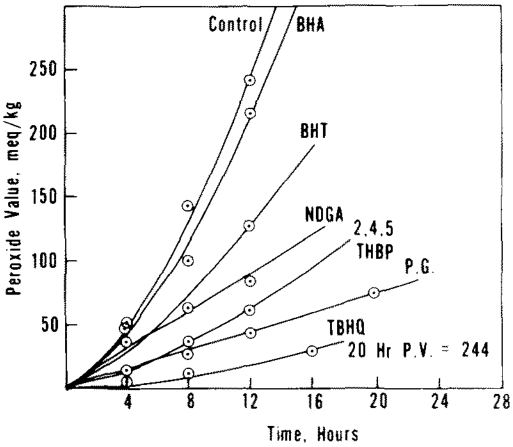

FIG. 2. Peroxide development in refined and bleached sunflower oil treated with antioxidants, AOM conditions. See Table II and III for abbreviations.

Results for refined and bleached oils are presented in Figures 1 and 2, respectively. All antioxidants tested improved oxidative stability as measured by inhibition of peroxide development under conditions of the AOM test. Figure 2 substantiates previous data (Table I) which indicated that bleaching lowers oxidative stability of sunflower oil. With each antioxidant tested peroxides developed at a faster rate in the bleached oil than in the refined sample.

Organoleptic evaluation of refined sunflower oils stabilized with BHA, BHT and PG are summarized in Table II. Initially, refined oil received acceptable flavor scores ranging from 7.2-7.6 with no significant differences evident. However oil treated with phenolic antioxidants and aged at 60 C received flavor scores not significantly different from the control oil; consequently BHA, BHT and PG must not improve flavor stability of refined sunflower oil. Although some improvement in oxidative stability is evident from 8 hr AOM peroxide values, little or no improvement was obtained in storage peroxide values over those of the untreated controls.

Flavor and oxidative stability data for bleached sunflower oil treated with various antioxidants are given in Table III. In contrast to the refined oil, bleached sunflower oils after 4 days' storage at 60 C improved significantly in flavor scores after treatment with certain antioxidants. While BHT, TBHQ and NDGA were ineffective, PG, BHA and THBP were highly effective in improving flavor stability. Most surprising is that while TBHQ is the most effective in inhibiting peroxide development according to the AOM test (Figs. 1 and 2), no improvement in flavor

TABLE II

Flavor and Oxidative Stability of Refined Sunflower Oil Containing Phenolic Antioxidants[a]

Storage, 60 C, days	Flavor scores and significance				
	Control	PG[b]	BHA	BHT	Significance[c]
0	7.6 (0.0)[d]	7.7 (0.0)	7.2 (0.0)	7.5 (0.0)	+
4	6.0 (7.2)	6.3 (6.9)			+
4	5.9 (6.5)		6.2 (6.7)		+
4	6.1 (7.4)			5.6 (9.5)	+
AOM 8 hr, PV	34.0	13.8	31.3	25.5	

[a] Antioxidants added at 0.02% by weight.

[b] PG = propyl gallate; BHA = butylated hydroxyanisole; BHT = butylated hydroxytoluene.

[c] In all table the (+) indicates no statistical significance at the 5% level; (*) denotes significance at the 5% level; (**) shows significance at the 1% level.

[d] Numbers in parentheses are peroxide values at time of tasting.

TABLE III

Effects of Antioxidants on Flavor Stability of Bleached Sunflower Oil

Treatment	Concentration, %	Flavor scores 0 Time	Flavor scores 4 days, 60 C	Significance of aged treated sample to aged control[a]
Control[b]	---	7.9	4.5	---
BHT	0.02	8.1	5.1	+
TBHQ[c]	0.02	7.6	3.7	+
NDGA	0.02	6.7	4.0	+
PG	0.02	8.2	5.6	**
BHA	0.02	8.1	5.1	**
THBP	0.02	7.5	5.4	**
Tenox-2	0.02	8.0	7.1	**
Tenox-6	0.02	8.2	6.4	**
G-50	0.02	7.5	7.1	**

[a] See Footnote c, Table II.

[b] Average five deodorizations.

[c] TBHQ = tertiary butyl hydroquinone; NDGA = nordihydroguaiaretic acid; THBP = trihydroxybutyrophenone.

TABLE IV

Effect of Added Iron on Flavor and Oxidative Stability of Sunflower Oil

Storage, 60 C, days	Flavor scores and significance: Control	+ 0.1 ppm Fe	+ 0.75 ppm Fe	+ 1.5 ppm Fe	Significance[a]
0	8.4 (0.0)	6.1 (0.0)	6.5 (0.3)	5.9 (0.3)	**
4	5.8 (4.7)	5.2 (7.8)			*
4	5.9 (5.8)		4 6 (6.7)		**
4	5.7 (5.3)			4.8 (7.9)	**
AOM 8 hr, PV	33.0	50.6	152.2	206.9	

[a] See Footnote c, Table II.

TABLE V

Flavor Evaluation of REfined Sunflower Oil Treated with Added Metals and Citric Acid

Storage, 60 C days	Flavor scores and significance: 1 0.1 ppm Cu	2 0.1 ppm Fe	3 0.1 ppm Cu + 0.01% citric	4 0.1 ppm Fe + 0.01% citric	Significance[a]
0	8.5 (0.5)	7.1 (0.6)	7.6 (0.3)	8.3 (0.2)	$\underbrace{\underbrace{1 ** \rlap{\overbrace{}^{**}}2 + 3}_{**} ** 4}_{+}$
4	4.5 (9.9)		6.5 (10.2)		**
4		6.1 (10.6)		6.7 (9.9)	+
4			6.3 (7.3)	5.9 (8.2)	+
4	3.9 (8.7)	5.8 (10.4)			**
8 hr AOM PV	145.2	62.9	30.3	43.2	

[a] See Footnote c, Table II.

TABLE VI

Flavor and Oxidative Stability of Refined Sunflower Oil Treated with Commercial Antioxidants

Storage, 60 C, days	Flavor scores: Control	Tenox-2 0.02%	Tenox-6 0.02%	G-50 0.02%	Significance[a]
0	7.9 (0.0)	7.8 (0.0)	8.3 (0.0)	8.3 (0.0)	+
4	5.2 (6.0)	6.8 (3.2)			**
4	5.4 (6.1)		6.9 (2.6)		**
4	4.7 (6.3)			8.0 (3.6)	**
8 hr AOM PV	41.4	13.6	13.7	19.0	

[a] See Footnote c, Table II.

TABLE VII

Effects of Various Stabilizers on Sunflower Oil Flavor[a]

	Treatment, flavor scores and significance[b]					
Oil	Control		Antioxidants	Citrated		Commercial antioxidants
Refined	5.6	+	5.9	6.9	+	7.2
	*		+	+		+
Bleached	4.4	*	5.4	7.2	+	6.9

[a]Accelerated 4 day storage at 60 C. Pooled data from multiple tests, s (1σ) for all observations = ±0.46 flavor units.

[b]See Footnote c, Table II.

stability was achieved.

Sunflower oil, like other highly unsaturated fats, would be expected to be sensitive to trace metal contamination. Studies were undertaken to determine the effects of added iron and copper on sunflower oil stability. When refined sunflower oil was deodorized in the presence of iron (added as the chloride), there was a detrimental effect on both flavor and oxidative stability (Table IV). Initially a highly significant difference in flavor scores was obtained for the three concentrations of added iron over that of the control oil. As little as 0.1 ppm iron reduced the flavor score to a point barely acceptable as an edible oil, i.e., 6.0. After 4 days' storage at 60 C, the sample containing 0.1 ppm Fe was significantly lower than the untreated control, whereas at 0.75 and 1.5 ppm highly significant differences were observed. Further evidence of oxidative deterioration is shown by increasing 8 hr AOM peroxide values as iron concentration increased. In addition storage peroxide values are somewhat higher than those of the untreated control oil.

Similar tests conducted on bleached oil demonstrated that iron at levels of 0.1, 0.75 and 1.5 ppm affected initial quality in a manner analogous to the refined sample. Iron-contaminated oils were scored highly significantly lower than the uncontaminated control. However after accelerated storage iron-treated samples had flavor scores not significantly lower than the control.

In additional studies on the effects of iron and copper on flavor stability (Table V), metals were added at the beginning of deodorization. Two samples containing 0.1 ppm copper and 0.1 ppm iron served as controls, whereas citric acid (0.01%) was added to another pair on the cooling side of deodorization.

Table V data confirm previous results (Table IV) on the deleterious effect of iron on initial flavor. Conversely the effect of copper on initial quality is quite unexpected. This metal at 0.1 ppm had no effect on the initial flavor of both refined and bleached oil. However after accelerated storage copper had a more pronounced effect than did iron at the same concentration. The aged copper-treated sample was scored some two units lower than the aged iron-treated sample.

The metal-inactivating properties of citric acid are illustrated in Table V. After storage, copper-contaminated oils treated with citric acid were scored significantly higher than a copper-contaminated but uncitrated control. Initially citric acid improved the flavor stability of iron-contaminated oil, and after 4 days' storage the contaminated citrated oil scored higher but not significantly higher than the iron-contaminated but uncitrated sample.

When the experiment depicted in Table V was repeated with bleached oil, results were similar. Citration improved the initial quality of the iron-contaminated sample. In addition citration improved stability of the iron-contaminated oil stored 4 days at 60 C.

Because iron and copper have such a marked influence on flavor and oxidative stability, trace metal contents of the natural oil used throughout this study were determined. The respective copper and iron contents as measured by atomic absorption spectroscopy were 0.025 and 0.071 ppm. In addition other analyses by flame emission spectroscopy revealed zinc, barium and calcium. Manganese and chromium were essentially absent. The bleached sample had a trace metal content similar to the refined oil. Complete data for trace metals in sunflower oil were reported earlier (8).

Flavor evaluations conducted on refined sunflower oil stabilized with phosphoric, α,α'-thiodipropionic and tartaric acid revealed that these metal-inactivating agents were significantly less effective than citric acid in improving flavor stability. Oil treated with 0.01% phosphoric acid on the cooling side of deodorization received low initial flavor scores because of characteristic melony flavors associated with this additive.

Commercial antioxidant mixtures were also evaluated as stabilizers for sunflower oil. Flavor data are assembled in Table VI for refined oil stabilized with Tenox 2, Tenox 6 and G-50 antioxidant mixtures. Initially all samples received high flavor scores with no significant differences evident between the control and stabilized oils. Accelerated storage data show a highly significant improvement in flavor scores after stabilization with these antioxidant mixtures. Improvements were similar with bleached oil treated with these same mixtures.

The antioxidant mixtures contain up to eight components including: phenolic antioxidants (BHT, BHA and PG), a metal inactivator common to all three and various carrier solvents, such as vegetable oil or propylene glycol. Thus improvements in flavor and oxidative stability derived from stabilization with commercial antioxidants mixtures could result through trace metal inactivation or antioxidant synergism. The effects of various stabilization treatments for refined and bleached oil are summarized in Table VII. Data given here are averaged flavor scores from 31 observations for each oil type. As shown previously, penolic antioxidants alone did not improve flavor stability in the refined oil but did in the bleached sample.

Flavor scores for oils stabilized with commercial antioxidants were not significantly higher than those treated with citric acid alone. Flavor stability probably improves from metal inactivation rather than antioxidant synergism.

Predominant flavors for refined and bleached sunflower oil containing neither added antioxidants nor metal-inactivating agents are described in Table VIII. Also included are flavor intensity values (FIV) which allow a quantitation of flavor responses (16). Data given in Table VIII indicate that initially both refined and bleached oils have predominantly buttery, beany and nutty flavors. Little difference in these flavor intensities is evident between the refined and bleached oils.

After accelerated storage, flavor for both oils changed from predominantly buttery to predominantly rancid. However the bleached oil had a rancid FIV of 1.5 compared to 0.9 for the refined oil. In addition some painty

TABLE VIII

Predominant Flavor Descriptions of Sunflower Oil

Flavor description	Refined FIV[a]		Bleached	
	Initial	4 Days, 60 C	Initial	4 Days, 60 C
Buttery	0.7	0.3	0.5	0.2
Beany	0.2	0.2	---	0.4
Nutty	0.2	0.2	0.4	0.3
Rancid	---	0.9	---	1.5
Painty	---	---	---	0.4

[a] Average values from three deodorization runs; oils not stabilized with antioxidants or metal scavengers. Flavor intensity value (FIV) =

$$\frac{1\text{ (weak responses)} + 2\text{ (moderate responses)} + 3\text{ (strong responses)}}{\text{no. of tasters}}.$$

responses, essentially absent in aged refined oil, were given to the bleached sample.

Data given in Table VIII indicate that sunflower oil, on extended storage, undergoes flavor changes much like other nonlinolenate-containing vegetable oils (17). Rancid flavors resulting from autoxidative breakdown are observed rather than grassy, painty and fishy flavors associated with linolenate-containing edible oils (18).

DISCUSSION

Bleaching of sunflower oil lowers its oxidative stability and our data confirm the findings of Ostric-Matijasevic et al. (19).

Sherwin and Luckadoo (7) found that refined, bleached and deodorized sunflower oil processed from a crude oil treated with the antioxidant TBHQ had a much poorer stability than oils similarly processed from crude oils treated with other antioxidants.

Mikolajczak et al. (20) compared oxidation rates of crude sunflower oils extracted from several American and Russian varieties and found that domestic oils oxidized considerably faster than the Russian oils. Since the Russian oils had better oxidative stability despite being more unsaturated, it was suggested that factors other than fatty acid composition were responsible. Such factors include minor constituents like phosphatides, tocopherols and trace metals.

A Russian report (21) indicates that the ratio of vitamin E to linoleic acid determines the inherent oxidative stability of sunflower oil. Of nine varieties the Russians examined, the ratio of vitamin E (mg%) to linoleic acid (%) was the same, and each oil had the same induction period in oxidative tests. Conversely a factor-refined sample in which the tocopherol content had been significantly lowered was less oxidatively stable. Another study (22) indicates that the oxidative stability of safflower oil, an oil whose composition is similar to sunflower, does not vary with α-tocopherol content.

Although these published findings suggest that bleaching lowers to tocopherol content of sunflower oil and that this loss results in a concurrent reduction in oxidative stability, our analytical data (see Materials and Methods) on refined and bleached oils indicated little, if any, loss of tocopherols from bleaching. Nevertheless bleaching can lower oxidative stability. Sunflower oils treated in the laboratory with a high concentration of bleaching earth (6%) have had poorer flavor and oxidative stability (Table I) than the same oil bleached with a lower concentration of earth (2%). Since high bleach concentrations yield poorer quality oils, this effect depends on the concentration of bleaching earth. Apparently bleaching removes, destroys or inactivates some unknown minor constituent essential for optimum keeping qualities.

It should be pointed out that 2% bleach probably represents a concentration somewhat higher than that used in commercial practice. Thus sunflower oil bleached commercially may have somewhat different oxidative properties than the oil described in this paper.

Another factor in sunflower oil stability not easily explained involves the behavior of refined and bleached oils toward antioxidant treatment. Judging from their fatty acid composition, antioxidants might not be expected to improve flavor scores because of the highly unsaturated nature of the fat. The failure of antioxidants in soybean oil is attributed to its highly unsaturated fatty acid composition (23). Antioxidants are effective in fats containing low levels of natural antioxidants (tocopherols). The failure of antioxidants to improve flavor scores of the refined oil suggests that sunflower oil is not deficient in tocopherols, whereas results with the bleached oil suggest the opposite is true. Limited published reports (19,21) indicate that sunflower oil contains approximately 500-770 μg tocopherol per gram; our sample contained somewhat more than these levels.

Our studies on the trace metal content of refined and bleached sunflower oils revealed nothing unusual regarding the concentrations or kinds of metals present. Copper and iron, known to be the most powerful prooxidants for unsaturated fats, were present in concentrations (see Results) much like those reported for other fully processed edible fats (24). Consequently we do not attribute differences in oxidative stability between refined and bleached oils to possible metallic contamination from bleaching earth.

Published reports (25) indicate that copper, iron, manganese and nickel occur naturally in crude sunflower oil as complexes. Although compleses are unimportant in promoting autoxidative deterioration, either metals added from corrosion of processing equipment or intentionally added metal chlorides and metal soaps are deleterious in the order Cu>Fe>Mn>Ni. Tables IV and V indicate that iron may be more detrimental than copper to the initial quality of freshly deodorized sunflower oil. However, in accelerated storage tests, copper is more deleterious and has greater prooxidant effects than iron when added in equal concentrations.

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