

## Chemical and Physical Changes in Cottonseed Oil During Deodorization

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The effects of deodorization time and temperature on the physical and chemical properties of cottonseed oil were investigated. Higher temperatures and longer times lead to increases in free fatty acids, peroxide value, viscosity and refractive index while iodine value, unsaponifiable matter and induction period decreased.

**KEY WORDS:** Color, cottonseed oil, deodorization, fat peroxides, free fatty acid, oxidative stability, unsaponifiable matter.

The object of refining an edible oil is to remove unacceptable materials with the least possible changes on desirable components and with the least possible loss of oil. Vegetable oils are usually refined in four successive operations—degumming, neutralizing, bleaching and deodorizing (1).

Deodorization is primarily a high-temperature and high-vacuum steam distillation process. This process improves the oil's flavor and oxidative stability by nearly complete removal of free fatty acids and of other volatile odor and flavor materials by thermal destruction of fat hydroperoxides. However, it has been shown that when deodorization is carried out at higher temperatures for longer times, some undesirable changes occur in the physical and chemical properties of deodorized oils (2).

### MATERIALS AND METHODS

A degummed, neutralized and bleached cottonseed oil was used. Deodorizations were carried out in a 3-neck, 2-L, round-bottomed glass flask. Water was introduced to the flask through a capillary tube in one neck while a vacuum

TABLE 1

Effect of Deodorization Temperature and Time on Cottonseed Oil

Oil treatment	Free fatty acid (oleic %)	Peroxide (meq/kg)	Iodine value (Wijs)	Induction period (at 100°C, hr)	Viscosity (poise at 25°C)	Refractive index (at 25°C)	Lovibond red color (5/4' cell)	Total USM (%)	Total sterols (mg/100 g)	Total tocopherol (mg/100 g)
Degummed, neutralized, bleached oil	0.19	3.6	116	20	0.50	1.4704	6.0	1.39	355	86.8
180°C <sup>a</sup>										
1/2 hr	0.17	2.6	116	22	0.51	1.4704	4.6	1.35	331	82.4
1 hr	0.15	2.0	116	22	0.51	1.4705	4.5	1.33	312	81.3
2 hr	0.14	1.2	116	22	0.52	1.4705	3.8	1.28	305	80.9
3 hr	0.10	0.8	115	22	0.53	1.4705	3.8	1.21	301	74.2
4 hr	0.10	0.5	115	22	0.53	1.4707	3.9	1.17	277	73.9
200°C										
1/2 hr	0.11	1.7	115	22	0.51	1.4705	4.6	1.33	323	81.9
1 hr	0.11	1.8	115	22	0.51	1.4706	4.6	1.30	309	76.1
2 hr	0.11	0.8	114	22	0.52	1.4706	3.8	1.25	298	76.0
3 hr	0.11	0.8	113	22	0.53	1.4709	3.8	1.18	272	69.6
4 hr	0.09	0.8	113	22	0.53	1.4709	3.5	1.15	262	59.8
220°C										
1/2 hr	0.13	1.4	114	23	0.52	1.4706	3.8	1.33	297	79.4
1 hr	0.14	1.5	114	23	0.52	1.4707	3.8	1.25	280	76.0
2 hr	0.11	0.8	113	23	0.52	1.4709	2.9	1.23	251	75.1
3 hr	0.08	0.6	111	22	0.55	1.4711	2.9	1.22	245	67.9
4 hr	0.07	0.6	111	22	0.56	1.4711	3.0	1.15	233	58.1
240°C										
1/2 hr	0.09	1.6	113	23	0.52	1.4706	3.8	1.33	277	69.0
1 hr	0.10	0.8	111	23	0.53	1.4707	3.1	1.22	263	56.7
2 hr	0.10	0.5	109	20	0.54	1.4710	2.3	1.02	261	51.4
3 hr	0.06	1.6	108	19	0.56	1.4712	3.1	1.00	235	40.0
4 hr	0.09	2.4	107	17	0.58	1.4715	3.1	1.00	218	39.5

<sup>a</sup>Deodorized at that temperature.

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## SHORT COMMUNICATION

and nitrogen gas inlet and a thermometer well were placed in the others. An electric heating mantle connected to a variable transformer was used to control the temperature of deodorization. The deodorization equipment was similar to that described in the literature (3-5).

Cottonseed oil was heated to the desired temperature under 2 torr vacuum with 5% stripping steam. After deodorization, the oil was cooled to 100°C, the vacuum was broken with nitrogen, and 200 ppm of aqueous 10% citric acid (w/v) solution was added. Oil samples were stored at 4°C prior to analysis and characterization. Deodorization experiments were carried out in duplicate and reproducible results were obtained.

Free fatty acid, peroxide value, active oxygen method, viscosity, refractive index and color were determined according to Official AOCS Methods (6). Unsaponifiable matter and sterols were determined by IUPAC methods (7). Tocopherols were determined according to Carpenter (8).

## RESULTS AND DISCUSSION

Because many undesirable materials in undeodorized oils possess the same vapor pressures as free fatty acids, free fatty acid content is a measure of the completeness of deodorization. Normally when the free fatty acid content is reduced to 0.01-0.03% and the peroxide value approaches zero, many flavors and odors are eliminated (9), and bland odorless-tasteless fats result.

Table 1 shows that free fatty acid levels of cottonseed oil decreased with temperature and time of deodorization. This reduction was apparent in oils deodorized at temperatures of 220°C and 240°C beyond 2 hr. However, the free fatty acid in cottonseed oil deodorized under these conditions exceeds 0.03% because of citric acid added to the deodorized oil (10). Table 1 also shows that deodorization for 3 hr at 180°C, 2 hr at 200°C and 220°C, and 1 hr at 240°C is sufficient to reduce peroxides to low levels, and peroxide destruction occurs rapidly at temperatures above 200°C.

In addition, deodorizations carried out at 180°C, 200°C, and 220°C yield a constant induction period. However, when the temperature exceeded 220°C for longer periods of time, the stability was reduced sharply. This may have resulted from a reduction in tocopherol content (1).

Results given in Table 1 confirm that the iodine value was progressively reduced when the deodorization temperature was raised; it became pronounced in the oil deodorized at 240°C beyond 1 hr. A decrease in iodine value is indicative of polymerization of unsaturated fatty acids as observed by Jawad *et al.* (1). Further evidence of polymerization occurring at higher temperature is provided by increases in viscosity and refractive index (1,11). Our data confirm the detrimental effect of high tempera-

tures for prolonged periods, as shown by the refractive index and viscosity data in Table 1.

Some data have been presented in the literature concerning color changes during deodorization (12,13). Cottonseed oil is known to heat-bleach during deodorization (12), and the magnitude of this bleaching may range from 1-2 red units for winterized oil to zero for hydrogenated oils. Data in Table 1 show that heat-bleaching is both time- and temperature-dependent, with optimum color reduction occurring at 220°C for 2 hr. Raising the temperature to 240°C offered no advantage.

The detrimental effects of deodorization temperatures beyond 220°C and times beyond 2 hr are further illustrated by the total unsaponifiable matter and tocopherol contents of the deodorized oil (Table 1). Tocopherols, which serve as natural antioxidants, show a marked loss at temperatures beyond 240°C and times beyond 2 hr at 220°C.

Moser *et al.* (14) have demonstrated that with soybean oil, the quality of cottonseed oil is dependent on both time and temperature of deodorization. In general, when temperatures are low, time should be increased, whereas time should be decreased when temperatures are high. Optimum deodorization conditions necessary to produce a cottonseed oil low in free fatty acid, light in color, with minimum damage to the tocopherols, appear to be a time of 2-3 hr at temperatures no higher than 220°C.

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