Processing of Cottonseed.

III. Color Development in Cottonseed Oil During Storage of the Seed and Crude Oil[1]

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Summary

Processing of cottonseed by the hydraulic press method has been carried out at two mills, one located about 100 miles farther south than the other. The varieties of seed processed and conditions during processing, which included cooking of moistened seed, were essentially the same at both mills. Seed was also processed at the more southerly mill by the screw press method for which seed was cooked without added moisture.

The crude hydraulic- and screw-pressed oils produced at the two mills were stored at different temperatures for a total of ten months, and samples of the oils were periodically refined and bleached. Seed was also stored at the two mills, and was periodically processed for comparison of the oils produced from stored seed with the stored oils. The absorption spectra of the crude, refined, and bleached oils were determined.

The rate of increase of bleach color in the oils during storage of the seed and crude oils has been correlated with temperature of storage and changes in the absorption spectra of the oils.

Introduction

THE increase in bleach color[4] during storage of crude cottonseed oil, a phenomenon known as *color reversion,* has been of considerable concern in certain segments of the cottonseed oil industry for many years. In a previous investigation of this phenomenon Fash (1) noted that crude oil from *bollie* seed deteriorated rapidly during storage, and admixture of *bollie* seed oil with normal oil caused increase in the bleach color of the mixture in excess of that which could be accounted for on the basis of the added *bollie* oil. King (2) reported that *color reversion* does not occur during storage of *good quality* crude oils and that such oils could be stored for as long as five months without serious deterioration or appreciable increase in processing costs.

During periods when the majority of mills in a given area, however, are crushing cottonseed in excess of the capacity of the refineries to handle the oil, it may be necessary to store both *sub-quality* and prime quality oils for later refining. In western Texas it has been observed (3, 4, 5) that some crude oils revert so rapidly in color that they develop excessive bleach color during the time elapsing between loading of the oils at the mills and their receipt at the refineries.

In the course of a recent investigation (6) of the pigmentation of cottonseed oils and meals produced at two mills by the hydraulic- and screw-press methods of processing, it was found that, of the hydraulic-pressed oils from seed of the same variety which were processed at both mills under similar conditions, the bleach colors of the oils produced at the more southerly mill were higher than those of the oils produced at the mill located farther north. Comparison of the absorption spectra of the crude, refined, and bleached hydraulic-pressed oils showed that corresponding oils from the two mills contained similar pigments, but the darker oils from the more southerly mill contained larger amounts of these pigments. Oils produced by the screw-press method from seed cooked without addition of water were more deeply colored than the corresponding hydraulic-pressed oils produced at the same mill. The principal pigments of the screw-pressed oils occurred in higher concentrations and the absorption spectra of the crude, refined, and bleached oils were entirely different from those of the corresponding hydraulic-pressed oils.

The present investigation was undertaken in order to determine the factors which influence the increase in bleach color during storage of seed and crude oils. A preliminary analysis of the production records of several cottonseed oil mills in southeast Texas revealed that as the season progressed, the bleach color of oils produced at a given mill increased without concurrent increase in the content of free fatty acid. The most rapid increase in color occurred toward the end of the milling season during warm weather, and it occurred to a greater extent in oils produced at the more southerly mills. It was evident, therefore, that bleach color increased during storage of seed as well as crude oils, and it seemed probable that the development of bleach color was accelerated by increased temperature during storage.

Experimental Procedure

Processing of cottonseed by the hydraulic- and screw-press methods was carried out at the same two mills and by the same procedure as previously reported (6). The hydraulic-pressed oils were prepared at these two mills according to the general procedure of pre-cooking moistened flaked seed in steam-jacketed cookers for a total of 50-80 minutes to a final temperature of 232° F. followed by expression in hydraulic presses. Water was added to the meats before flaking at the more southerly mill to yield flakes having a moisture content of 11.30%. At the

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[4] The term "bleach color" is used to designate the residual color, in terms of Lovibond red and yellow units, of an oil which has been alkali-refined and bleached by American Oil Chemists' Society official methods.

TABLE 1.

Content of Free Fatty Acid and Refining Loss of Stored Oils and Oils From Stored Seed.

Storage conditions			Hydraulic-pressed oils (CS-167-HO) from more northerly mill			Hydraulic-pressed oils (CS-166-HO) from more southerly mill			Screw-pressed oils (CS-166-EO) from more southerly mill		
Product	Time (months)[b]	Temp. °F.	FFA %	Lye °Baumé	Refining loss,[a] %	FFA %	Lye °Baumé	Refining loss,[a] %	FFA %	Lye °Baumé	Refining loss,[a] %
Seed	0	......	0.89	12; 14	6.1; 6.4	0.74	12; 14	4.1; 4.3	1.0	16	7.6
Oil	1	90	0.80	14	......	0.8	14	......	......	......	......
Oil	1	100	0.90	14	......	1.0	14	......	......	......	......
Oil	4.5	70	0.80	14	5.6	0.8	14	6.0	......	......	......
Oil	4.5	90	0.90	14	6.5	0.7	14	3.1	......	......	......
Oil	4.5	100	1.0	14	6.8	0.8	14	3.6	......	......	......
Oil	10	38	0.9	12	......	0.8	12	......	......	......	......
Oil	10	70	1.2	12	......	0.9	12	......	......	......	......
Oil	10	100	1.3	12	......	1.2	12	......	......	......	......
Seed	5	......[c]	1.0	12; 14	4.0; 4.0	1.2	12; 14	6.6; 6.4	1.5	16; 20	13.2; 19.6
Seed and oil[d]	10	38	1.3	12	......	1.1	14	......	1.5	16	......
Seed and oil[d]	10	70	1.1	12	......	1.2	12	......	1.5	16	......
Seed and oil[d]	10	100	1.4	12	......	1.7	16	......	1.6	16	......
Seed	10	......[e]	......	......	......	0.9	12	......	1.3	16	......

[a] Oils for which refining loss is indicated were refined by the appropriate American Oil Chemists' Society official refining method; others were refined by the recently published (8) small-scale adaptation of the American Oil Chemists' Society official methods.
[b] Analyses made on stored oil or oils expressed from stored seed as indicated.
[c] Temperatures during this period were 60° to 70°F. at the more northerly mill, and 75° to 85°F. at the more southerly mill.
[d] Seed stored for five months; oils then stored for five months; total storage, 10 months.
[e] Temperatures during the last five months of storage at the more southerly mill were 80° to 90°F.

other mill moisture was introduced in the form of steam in the first stack of the cooker, to yield a moisture content of 10.86% in the meats. Thus, the moisture content of the seed during cooking was approximately the same at both mills. Seed processed by the screw-press method at the more southerly mill was ground in a disc huller, and the ground seed of 3.92% moisture content was heated, without addition of water, for one hour in a steam-jacketed cooker to a final temperature of 224°F. The oil was then expressed in Anderson No. 1 Expellers.

Laboratory samples of the oils were refined by the appropriate American Oil Chemists' Society official refining methods (7), and the absorption spectra of chloroform solutions of the crude, refined, and bleached oils were determined with a Beckmann quartz spectrophotometer.

For determining the effect of temperature during storage of the crude oils, samples of the hydraulic-pressed oils were stored in closed tin cans at 38°, 90°, and 100°F. The quantity of screw-pressed oil collected from the first processing of seed was insufficient for making storage tests.

After storage of the crude hydraulic-pressed oils for periods of one, four and a half, and ten months, samples corresponding to each storage period were refined, either by the appropriate American Oil Chemists' Society official refining method (7) or by a recently described (8) modification of the official method especially adapted to small samples. Absorption spectra of chloroform solutions of the crude, refined, and bleached oils were determined for comparison with the corresponding original oils.

In order to ascertain the effect of storage of the seed on the bleach color of the expressed oils, a portion of the seed from selected locations in the seed houses of the two mills was processed early in the milling season to produce the first series of oils, and another portion was processed after storage for five months. Samples of the crude oils from the stored seed were refined, and the absorption spectra of chloroform solutions of the crude, refined, and bleached oils were determined. Samples of the crude oils were also stored for five months at different temperatures in closed tin cans.

Storage of seed was discontinued after five months at the more northerly mill, but was continued at the more southerly mill for a total of ten months after which this seed was also processed. The oils which were obtained from the stored seed by the hydraulic-

TABLE 2.

Colors of Refined and Bleached Oils Processed From Stored Oils and Oils From Stored Seed.

Storage conditions			Hydraulic-pressed oils (CS-167-HO) from more northerly mill				Hydraulic-pressed oils (CS-166-HO) from more southerly mill				Screw-pressed oils (CS-166-EO) from more southerly mill			
			Lovibond colors (5.25-in. cell)				Lovibond colors (5.25-in. cell)				Lovibond colors (5.25-in. cell)			
			Refined		Bleached		Refined		Bleached		Refined		Bleached	
Product	Time (months)[a]	Temp. °F.	Yellow	Red	Yellow	Red	Yellow	Red	Yellow	Red	Yellow	Red	Yellow	Red
Seed	0	......	35	5.4	20	1.2	35	5.6	20	1.9	35	10.3	35	4.2
Oil	1	90	35	5.6	15	1.6	35	6.1	20	1.9	....	......	....	......
Oil	1	100	35	5.7	20	1.8	35	6.9	20	2.7	....	......	....	......
Oil	4.5	70	35	5.2	20	1.2	35	5.8	20	1.8	....	......	....	......
Oil	4.5	90	35	5.7	20	1.7	35	6.7	35	2.4	....	......	....	......
Oil	4.5	100	35	6.8	35	2.4	35	9.3	35	3.5	....	......	....	......
Oil	10	38	35[b]	3.1	5[b]	0.9	35[b]	4.48	20[b]	1.29	....	......	....	......
Oil	10	70	35	7.02	35	2.27	35[b]	4.99	20[b]	1.83	....	......	....	......
Oil	10	100	35[b]	3.6	20[b]	1.36	35[b]	4.56	20[b]	2.56	....	......	....	......
Seed	5	......[c]	35	5.5	20	1.4	35	6.6	20	2.6	35	18.4	35	10.2
Seed and oil[d]	10	38	35[b]	3.96	20[b]	3.30	35[b]	6.65	35[b]	4.19	35[b]	6.75	35[b]	3.39
Seed and oil[d]	10	70	35[b]	5.17	20[b]	3.22	....	......	....	......	35[b]	10.83	35[b]	7.11
Seed and oil[d]	10	100	35[b]	4.89	20[b]	7.12	35[b]	13.1	35[b]	7.75	....	D[e]	....	D[e]
Seed	10	......[f]	....	......	....	......	35[b]	2.91	15[b]	1.58	35[b]	11.0	35[b]	4.48

[a] Analyses made on stored oils or oils expressed from stored seed or both as indicated.
[b] One-inch cell was used in reading these colors.
[c] Temperatures during this period were 60° to 70°F. at the more northerly mill, and 75° to 85°F. at the more southerly mill.
[d] Seed stored for five months; oils then stored for five months; total storage, 10 months.
[e] Colors too dark to match in Lovibond tintometer.
[f] Temperatures during the last five months of storage at the more southerly mill were 80° to 90°F.

and screw-press methods were refined, and the absorption spectra of chloroform solutions of the crude, refined, and bleached oils were determined.

Results

The refining characteristics of the various stored crude oils and the oils from stored seed are shown in Table 1. From these data, it can be noted that there was very little, if any, increase in free fatty acid or refining loss during storage of the seed or oils.

DEVELOPMENT OF BLEACH COLOR DURING STORAGE OF SEED AND CRUDE OILS.

Hydraulic-pressed Oils. The bleach colors of the various oils are shown in Table 2. Comparison of the data in this table indicates that the bleach colors of the hydraulic-pressed oils produced at the more northerly mill were initially lower, and increased more slowly during storage of the seed and crude oil than in the case of the corresponding oils produced at the more southerly mill. The bleach colors of the screw-pressed oils were initially higher and increased more rapidly during storage of the seed and oils than was the case with the corresponding hydraulic-pressed oils. The rate of increase in bleach color was greatest during storage of screw-pressed oils produced from stored seed and least during storage of hydraulic-pressed oils produced from the more lightly pigmented seed processed early in the season at the more northerly mill.

The more lightly pigmented hydraulic-pressed oil produced early in the season at the more northerly mill was still prime with respect to bleach color after storage for ten months at 70°F., and after storage for four and a half months at 100°F. The oil produced five months later from the same seed developed excessive bleach color during subsequent storage for five months at both 70° and 100°F. The more highly pigmented hydraulic-pressed oil processed early in the season at the more southerly mill was still prime with respect to bleach color after storage for one month at 100°F. or lower and after storage for four and a half months at 90°F. or lower, but after storage for ten months at 38°, 70°, and 100°F., the bleach colors of the oils exceeded the color specification for prime oils.

The hydraulic-pressed oil produced from seed stored for five months at the more southerly mill developed excessive bleach color during subsequent storage at 38°, 70°, and 100°F. The bleach color of the screw-pressed oil produced early in the season initially exceeded the color specifications for prime oils and continued to increase rapidly during storage of the crude oil.

It is notable that all of the control samples of oil which were stored at 38°F. developed rather high bleach colors during storage for ten months. It is not known whether the increase in bleach color in this case represented true color reversion of the oils at low temperatures or whether it was the result of undetected and intermittent failure of the refrigerating equipment which may have resulted in increases in temperature.

The relative rates of development of red bleach color in representative samples of stored crude oils and oils from stored seed are shown graphically by the curves in Figure 1. Bleach colors which were read in one-inch cells have been multiplied by 5¼ in order to permit approximate comparison with bleach colors read in 5¼-inch cells.

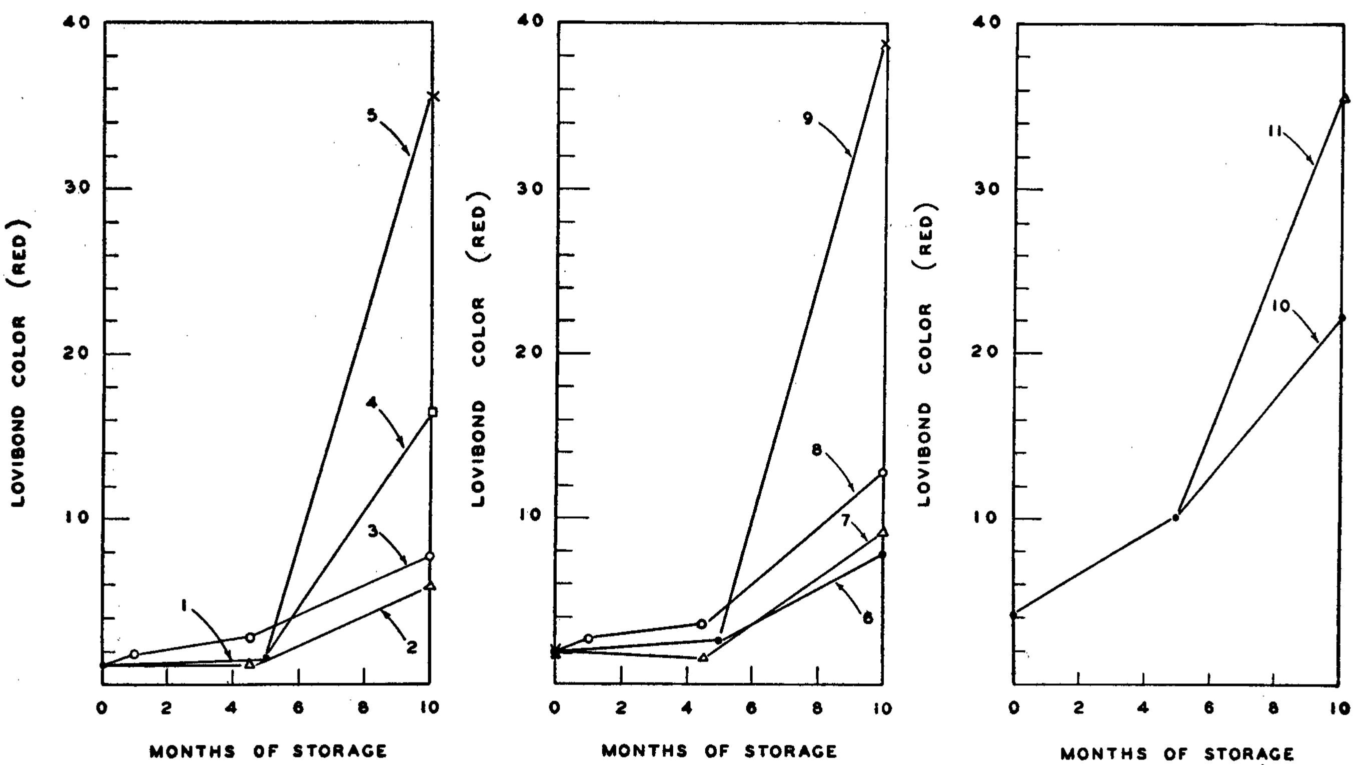

FIG. 1. Development of red bleach color vs. time of storage of hydraulic-pressed (1 to 9) and screw-pressed (10, 11) cottonseed oils: (1) fresh oils from seed stored at northern mill, (2 and 3) oil from fresh seed, oil stored at 70°F. and 100°F., (4 and 5) oil from 5-month old seed, oil stored at 70°F. and 100°F., (6) fresh oils from seed stored at southern mill, (7 and 8) oil from fresh seed, oil stored at 70°F. and 100°F., (9) oil from 5-month old seed, oil stored at 100°F., (10) fresh oils from seed stored at southern mill, (11) oil from 5-month old seed, oil stored at 70°F.

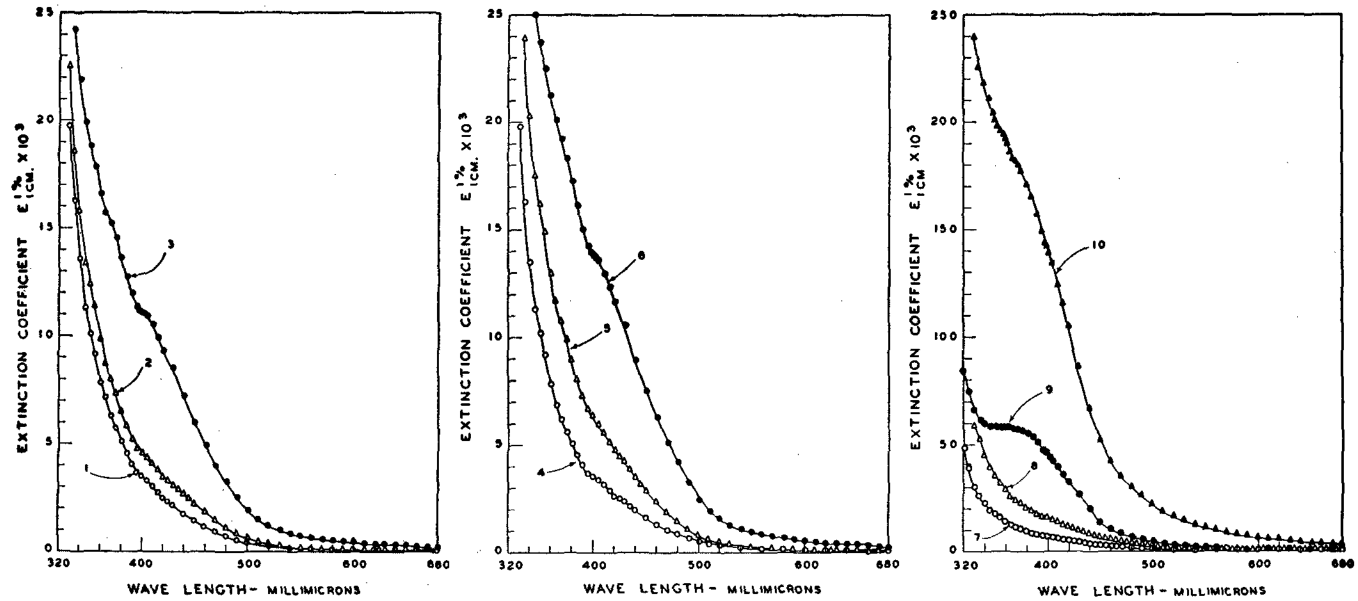

FIG. 2. Absorption spectra of bleached, hydraulic-pressed (1 to 6) and screw-pressed (7 to 10) cottonseed oils: (1) fresh oil produced from fresh seed at northern mill, (2 and 3) oil from fresh seed, oil stored at 100°F. for four and a half and ten months, (4) fresh oil produced from fresh seed at southern mill, (5 and 6) oil from fresh seed, oil stored at 100°F. for four and a half and ten months, (7) fresh oil produced from fresh seed at southern mill, (8) fresh oil from 5-month old seed, (9) fresh oil from 10-month old seed, (10) oil from 5-month seed, stored at 100°F. for five months.

In the case of oils produced at the more northerly mill, the bleach color of the oil obtained from seed stored at the mill for five months (Figure 1, Curve 1) was about the same as that of the crude oil produced five months earlier and then stored for four and a half months at 70°F., (Figure 1, Curve 2), however, the color was lower than that observed in the same crude oil after storage for the same period at 100°F. (Figure 1, Curve 3). The average temperature of the mill up to this point of the storage experiments was 70°F.

Crude oils, produced from stored seed (Figure 1, Curves 4 and 5), developed bleach color more rapidly during subsequent storage than did the oils produced from the original seed which had been stored at corresponding temperatures (Figure 1, Curves 2 and 3). Oils stored at 100°F., developed bleach color more rapidly than the corresponding oils which were stored at 70°F.

The bleach colors of hydraulic-pressed oils produced at the more southerly mill were initially higher and developed more rapidly during storage of the seed and oils than those produced at the more northerly mill. The bleach color of oil produced from seed stored at the mill for five months (Figure 1, Curve 6) was intermediate between the colors of the crude oils stored at 70°F. (Figure 1, Curve 7) and 100°F. (Figure 1, Curve 8), respectively. During this period of the storage experiments the average temperature of the mill was about 85°F.

The bleach color of oil freshly expressed from seed which had been stored for ten months (Figure 1, Curve 6) was less than that of any of the stored oils even though the average temperature at the mill during the last period of storage was relatively high. Bleach color developed very much more rapidly during storage at 100°F. of the oil obtained from seed stored for five months (Figure 1, Curve 9) than during storage at the same temperature of oil obtained from the original seed (Figure 1, Curve 8). As may be seen on comparison of curves 7 and 8 of Figure 1, elevated temperature during storage of the crude oils accelerated the development of bleach color.

Screw-pressed Oils. The colors of crude screw-pressed oils were initially higher and *reverted* very much more rapidly than those of the corresponding hydraulic-pressed oils. Storage of seed for five months resulted in a significant increase in the bleach color of the screw-pressed oil obtained therefrom (Figure 1, Curve 10). However, the bleach color of screw-pressed oil produced from seed which had been stored for ten months (Figure 1, Curve 10) was less than that of oil which had been stored for five months at 70°F. after it had been obtained from seed stored for five months (Figure 1, Curve 11). The bleach color of the corresponding oil stored at 100°F. for five months was too high to be matched in the Lovibond tintometer.

These results demonstrate the existence of a consistent pattern with reference to the change in bleach color during storage of cottonseed and crude cottonseed oils. The rate of development of bleach color during storage of both seed and oil was proportional to the original pigmentation of the seed and crude oils and to the time and temperature of storage. Development of bleach color occurred less rapidly during storage of seed than during storage of the corresponding crude oils.

ABSORPTION SPECTRA OF STORED OILS AND OF OILS FROM STORED SEED.

Bleached Oils. As shown in Figure 2, the curves of the absorption spectra of most of the bleached oils exhibit no selective absorption in the visible and near ultraviolet wavelength regions, but the absorption increases smoothly and sharply toward shorter wavelengths. Absorption spectra were obtained for all of the bleached oils investigated, but, for the sake of clarity, only representative examples have been shown in **Figure 2.**

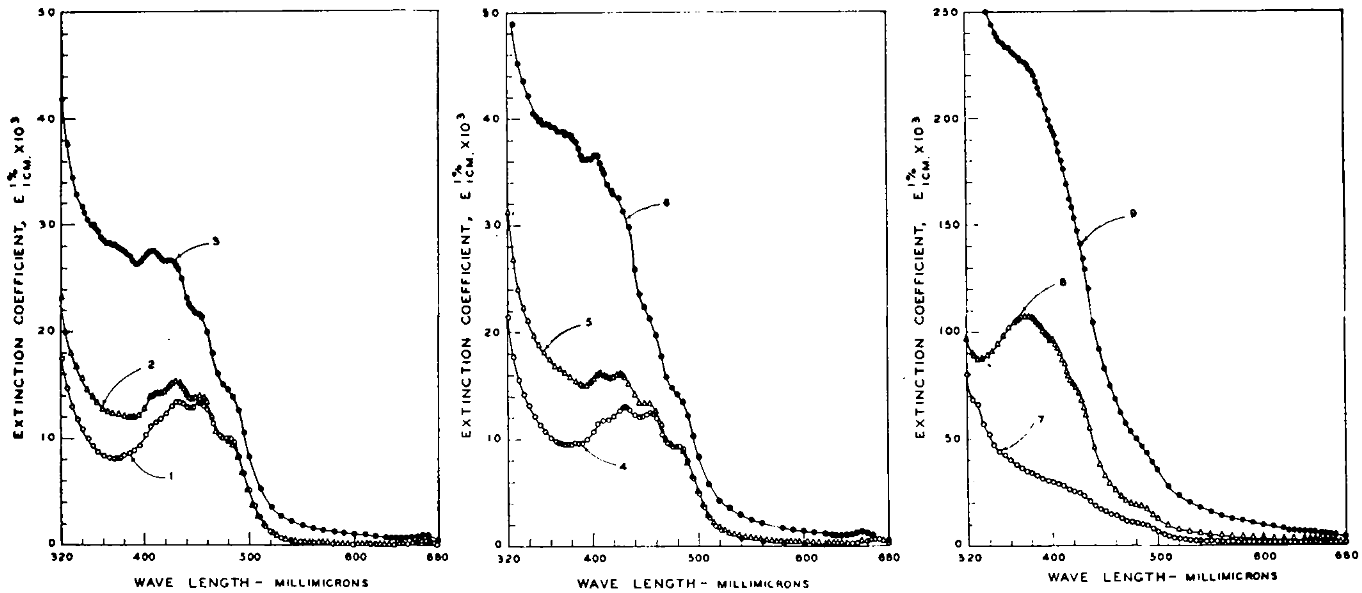

FIG. 3. Absorption spectra of alkali-refined, hydraulic-pressed (1 to 6) and screw-pressed (7 to 9) cottonseed oils: (1) fresh oil produced from fresh seed at northern mill, (2 and 3) oil from fresh seed, oil stored at 100°F. for four and a half and ten months, (4) fresh oil produced from fresh seed at southern mill, (5 and 6) oil from fresh seed, oil stored at 100°F. for four and a half and ten months, (7) fresh oil produced from 5-month old seed at southern mill, (8) fresh oil from 10-month old seed, (9) oil from 5-month old seed, oil stored at 100°F. for five months.

Bleached oils obtained from oils produced at the more northerly mill and subsequently stored at 100°F. (Figure 2, Curves 1, 2, and 3) were lighter in color and exhibited less absorption than the corresponding hydraulic-pressed oils produced at the more southerly mill (Figure 2, Curves 4, 5, and 6). The absorption spectra of the latter oils were lower than those of the corresponding screw-pressed oils (Figure 2, Curves 7, 8, 9, and 10).

The intensity of the absorption of the bleached oils obtained from stored crude oils increased in proportion to the temperature and duration of storage. Furthermore, the absorption spectra of oils which were originally of higher bleach color increased more rapidly during storage than that of the oil from the more northerly mill, the bleach color of which was originally lower and increased less rapidly during storage of the crude oil.

After storage of the crude hydraulic-pressed oils for ten months, the absorption spectra of the bleached oils (Figure 2, Curves 3 and 6) exhibited a slight inflection in the region of 406 mμ. The absorption spectrum of the bleached, screw-pressed oil from seed stored for ten months (Figure 2, Curve 9) exhibited an absorption band having a minimum at 332 mμ and a maximum from 374 to 380 mμ.

Alkali-refined Oils. The curves of the absorption spectra of typical examples of alkali-refined oils are shown in Figure 3. As might be expected on the basis of the smooth curves of the absorption spectra of the bleached oils, no direct correlation could be observed between the bleach color and the height of the absorption maxima characteristic of the alkali-refined, hydraulic-pressed oils. It could be observed, however, that storage of crude oils resulted in an overall increase in the absorption of the alkali-refined oils, and this increase was greatest in the regions of shorter wavelengths where the absorption of the bleached oils was greatest.

The alkali-refined oil obtained from the crude oil having the least initial bleach color exhibited less absorption (Figure 3, Curve 1) than the alkali-refined oil obtained from the crude oil of higher initial bleach color (Figure 3, Curve 4). The curves for the absorption spectra of the alkali-refined oils obtained from crude oils stored at 100°F. for ten months (Figure 3, Curves 3 and 6) were higher than those of the alkali-refined oils obtained from crude oils stored at 100°F. for four and a half months (Figure 3, Curves 2 and 5). The increase in both absorption and bleach color was greatest during storage of the initially more deeply colored oils from the more southerly mill.

Similar changes were observed in the absorption spectra of alkali-refined oils obtained from crude oils stored at lower temperatures (Tables 3 and 4), but these changes were of lesser magnitude than those occurring at higher temperatures. No qualitative differences could be detected between the absorption spectra of the alkali-refined oils produced from stored crude oils, and those produced from oils obtained on processing the stored seed.

The absorption spectra of the alkali-refined, screw-pressed oils, of which typical examples are reproduced in Figure 3, Curves 7, 8, and 9, exhibited little selective absorption in the visible and near ultraviolet wavelength regions. As might be expected on the basis of the higher bleach colors of the oils, the absorption spectra of the alkali-refined, screw-pressed oils were higher than similarly treated hydraulic-pressed oils. After storage at 100°F. for five months, the bleach color and the absorption spectrum (Figure 3, Curve 9) were higher than those exhibited by freshly expressed oil (Figure 3, Curve 7). The alkali-refined, screw-pressed oil obtained from seed stored for ten months had a lower bleach color and less absorption (Figure 3, Curve 8) than the corresponding product obtained from stored oil (Figure 3, Curve 9), but the absorption spectrum of the former oil exhibited a well-defined maximum at 374 to 380 mμ similar to that exhibited by the corresponding bleached oil (Figure 2, Curve 9).

Crude Oils. The specific extinction coefficients of the crude, hydraulic-pressed oils are shown in Tables 3 and 4, and those of the crude, screw-pressed oils in Table 5. Typical curves for the absorption spectra of the crude oils in the visible and near ultraviolet wavelength regions are shown in Figure 4.

TABLE 5.

Specific Extinction Coefficients of Crude Screw-Pressed Cottonseed Oils (CS-166-EO) After Storage of Seed and Crude Oils.

Temperature during storage of crude oils °F.	Specific extinction coefficients at wavelengths of selective absorption			
	Minimum		Maximum	
	$E^{1\%}_{1cm.}$	Wavelength mμ	$E^{1\%}_{1cm.}$	Wavelength mμ
Original seed				
......	0.889	316-319	2.22	368-71
Oil from seed stored for five months				
......	........[1]	........[1]	1.42	370-72
Oil stored for five months				
38	0.682	322-4	1.33	369-75
70	0.754	322	1.27	369-75
100	0.819	330-2	1.06	370-81
Oil from seed stored ten months				
......	........[1]	........[1]	2.23	368-72

[1] No absorption minimum in near ultraviolet.

Comparison of the data in Tables 3, 4, and 5 or in the curves of Figure 4, showed a direct correlation existed between the increase in bleach color during storage of the oils and the position and magnitude of the principal absorption band of these oils in the near ultraviolet wavelength region. The absorption at the minimum at 326 to 332 mμ and at the maximum at 375 to 381 mμ of the original crude hydraulic-pressed oil (Table 3 and Figure 4, Curve 1) having the lowest bleach color was less than that of the darker hydraulic-pressed oil (Table 4 and Figure 4, Curve 4). Moreover, the absorption of the darker, hydraulic-pressed oil was less than that of the still darker corresponding screw-pressed oil (Table 5 and Figure 4, Curve 7), which exhibited a sharp absorption band with minimum at 322 to 324 mμ and maximum at 368 to 371 mμ.

During the first month of storage of the crude hydraulic-pressed oils the absorption at the minimum in the near ultraviolet wavelength region increased and that at the maximum in the visible wavelength region decreased. The change in absorption was greater during storage of the originally darker crude oil, and it was also greater during storage of the oils at 100°F. than during storage at 90°F. The secondary maximum at 398 to 403 mμ observed in the absorption spectra of the freshly expressed oils decreased and is represented by a shoulder in the absorption spectra of the stored crude oils and the crude oils produced from stored seed.

Subsequent storage of the hydraulic-pressed oils obtained from stored seed resulted in a decrease in these oils of the absorption band in the near ultraviolet wavelength region. The absorption band was broadened and shifted toward longer wavelengths. This change was accompanied by an increase of the absorption minimum and a shift toward longer wavelengths. These changes can be observed by comparison of Curves 3 and 2 with Curve 1, and of Curves 6 and 5 with Curve 4 of Figure 4.

A poor correlation was observed between the bleach colors of the screw-pressed oils and the absorption spectra of the crude oils (Table 5 and Figure 4, Curves 7, 8, and 9). The freshly expressed oil had a lower bleach color and greater absorption (Figure 4, Curve 7) than did the oil expressed from stored seed (Figure 4, Curve 8). However, subsequent storage of the oil expressed from stored seed resulted in increased bleach color and a concurrent broadening and lowering of the absorption band in the near ultraviolet wavelength region as well as a shift toward longer wavelengths (Figure 4, Curve 9).

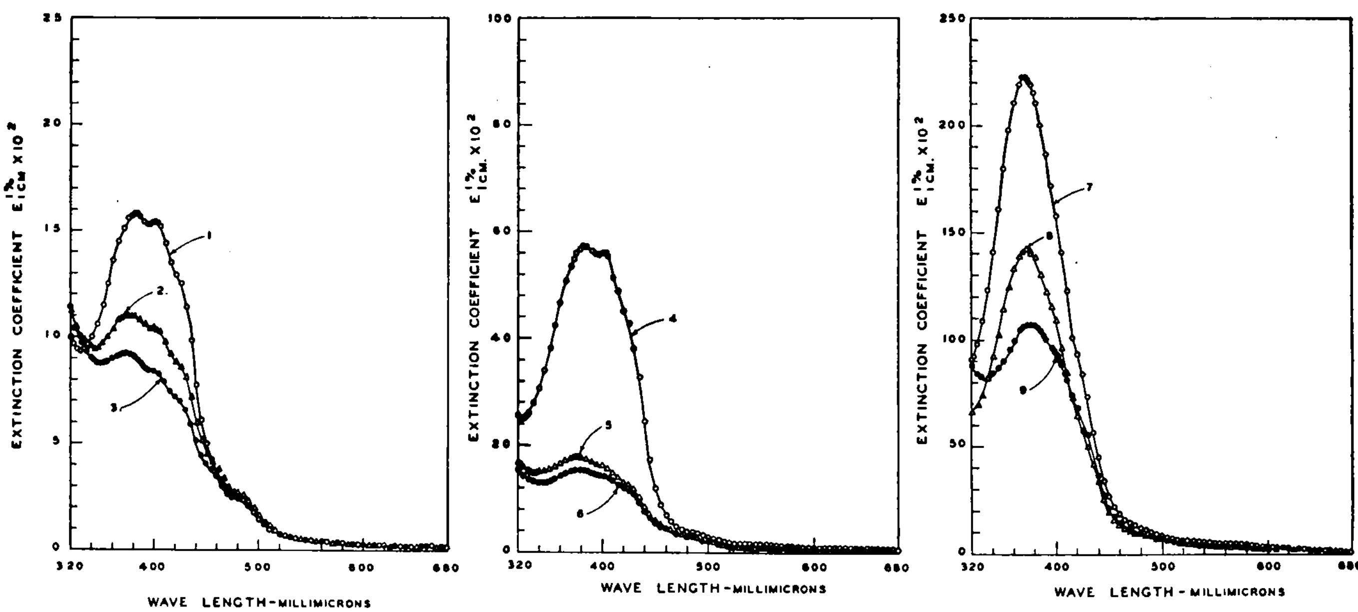

FIG. 4. Absorption spectra of crude, hydraulic-pressed (1 to 6) and screw-pressed (7 to 9) cottonseed oils: (1) fresh oil produced from fresh seed at northern mill, (2 and 3) oil from fresh seed, oil stored at 100°F. for four and a half and ten months, (4) fresh oil from fresh seed processed at southern mill, (5 and 6) oil from fresh seed, oil stored at 100°F. for four and a half and ten months, (7) fresh oil produced from fresh seed at southern mill, (8) fresh oil from 5-month old seed, (9) oil from 5-month old seed, oil stored at 100°F. for five months.

Conclusions

Crude hydraulic- and screw-pressed cottonseed oils were stored at different temperatures for a total of ten months during which time samples were periodically withdrawn and refined. Seed was also stored and periodically processed in order to determine the effect of storage of the seed on the bleach color of the oil. Absorption spectra of the crude, refined, and bleached oils were determined and correlated with the development of bleach color during storage.

Similar changes in the characteristics of cottonseed oils were found to occur during storage of both the seed and the crude oil, but they occurred less rapidly during storage of the seed than during storage of the crude oils.

The bleach color developed in the oil during storage of cottonseed was found to be determined by the amount of pigments in the original seed. Its development appears to be accelerated by elevated temperatures during storage.

The development of the bleach color in stored crude oils was found to be determined by the amount, as well as the kind, of pigments in the original crude oils, and these, in turn were determined by the pigment content and conditions obtaining during processing of the seed. The bleach colors of oils obtained from screw-pressed cottonseed which had been cooked without addition of water were initially high and increased rapidly during storage of the crude oils. The bleach colors of hydraulic-pressed oils obtained from wet-cooked cottonseed were initially lower and developed less rapidly during storage than in the case of screw-pressed oils.

Hydraulic-pressed oils of higher initial pigment content obtained from seed of higher pigment content developed bleach color more rapidly than less highly pigmented crude hydraulic-pressed oils. This was found to occur not only in oils obtained at two different mills but in oils obtained from seed stored at the mills for varying periods of time.

Elevated temperatures during storage of crude oils accelerated the development of bleach color in the oils. Only oils of initially low pigment content could be stored for relatively long periods at temperatures as high as 70°F. without development of excessive bleach color.

Comparison of the Lovibond bleach colors with the absorption spectra of the crude, refined, and bleached oils showed a correlation between the increase in bleach color and the absorption spectra of the crude oils. The decrease in the height of the absorption band of the crude hydraulic-pressed oils at 378 to 405 mμ, and the crude screw-pressed oils at 368 to 370 mμ with concurrent increase in bleach color indicates that deterioration of the pigments in the crude oil or their precursors in the seed is responsible for the development of high bleach color during storage of crude oil and seed.

The failure to detect a similar correlation between Lovibond bleach color and the height of the absorption maxima of the alkali-refined oils suggests that the pigments characteristic of the alkali-refined oils have little, if any, effect on bleach color. This observation is consistent with the poor correlation of the color of alkali-refined oils with the color of the corresponding bleached oils.

The absorption spectra of crude oils can be used as a basis for predicting the rate at which bleach color will develop during storage of crude oils. It can be expected that crude oils having a single absorption maximum at 368 to 370 mμ will rapidly develop high bleach colors. Hence, processing conditions which result in the production of oils exhibiting this type of absorption should be avoided if possible, or if production of such oils is unavoidable, the oils should be refined and bleached as rapidly as possible.

TABLE 3.

Specific Extinction Coefficients of Crude and Alkali-Refined, Hydraulic-Pressed Cottonseed Oils (CS-167-HO) After Storage of Seed and Crude Oils.

Temperature during storage of crude oils °F.	Specific extinction coefficients at wavelengths of selective absorption									
	Crude oils						Alkali-refined-oils			
	Minimum		Maximum		Shoulder		Minimum		Maximum	
	$E^{1\%}_{1cm.}$	Wavelength mμ	$E^{1\%}_{1cm.}$	Wavelength mμ	$E^{1\%}_{1cm.}$	Wavelength mμ	$E^{1\%}_{1cm.}$	Wavelength mμ	$E^{1\%}_{1cm.}$	Wavelength mμ
Oil from original seed										
......	0.0935	328	0.158	380-2	0.154[1]	398-400	0.00808	370-2	0.0134	430-4
Oils stored one month										
90	0.0951	330	0.143	378-82	0.14	390-402	0.00936	365-70	0.0141	430-32
100	0.0973	332	0.132	378-83	0.13	390-402	0.00958	365-75	0.0139	429-30
Oils stored four and a half months										
70	0.107	330	0.171	378-83	0.16	395-405	0.00944	368-9	0.0140	429-32
90	0.118	328-34	0.168	379-82	0.16	390-405	0.0108	365-90	0.0145	429-32
100	0.0953	340	0.110	370-80	0.10	395-405	0.0119	385-90	0.0153	429-32
Oils stored ten months										
38	0.0866	330	0.138	376-8	0.13	390-400	0.0251	390-4	0.0263	422-6
70	0.108	328-32	0.167	379-83	0.16	392-400	0.0234	390-4	0.0241	422-6
100	0.0874	348-50	0.0920	372-6	0.085	395-400	0.0263	390-4	0.027	410-430
Oil from seed stored five months										
......	0.128	326-30	0.217	380	....	....	0.0090	390	0.0118	429-32
Oils stored five months										
38	0.167	325-30	0.312	377-80	0.30	390-400	0.0648	392-4	0.057[2]	418-422
70	0.126	334-36	0.162	374-81	0.15	390-400	0.047	394-404	0.037[2]	418-422
100	0.136	344-50	0.151	373-9	0.14	390-400	0.034[2]	394-404	0.030[2]	418-422

[1] Absorption maximum. [2] Shoulder.

TABLE 4.

Specific Extinction Coefficients of Crude and Alkali-Refined, Hydraulic-Pressed Cottonseed Oils (CS-166-HO) After Storage of Seed and Crude Oils.

Temperature during storage of crude oils °F.	Specific extinction coefficients at wavelengths of selective absorption									
	Crude oils						Alkali-refined oils			
	Minimum		Maximum		Shoulder		Minimum		Maximum	
	$E^{1\%}_{1cm.}$	Wavelength mμ	$E^{1\%}_{1cm.}$	Wavelength mμ	$E^{1\%}_{1cm.}$	Wavelength mμ	$E^{1\%}_{1cm.}$	Wavelength mμ	$E^{1\%}_{1cm.}$	Wavelength mμ
Oil from original seed										
......	0.244	322	0.574	381-2	0.561[1]	401-3	0.0096	360-5	0.0130	430.1
Oils stored one month										
90	0.140	326-30	0.234	379-81	0.22	392-404	0.00953	366-8	0.0142	430-2
100	0.153	328-30	0.231	379-81	0.22	394-404	0.0126	388-92	0.0144	428
Oils stored four and a half months										
70	0.180	320	0.416	379-85	0.40	393-405	0.0115	390	0.0138	428-9
90	0.137	330	0.196	378-80	0.19	390-405	0.0125	390	0.0144	426-31
100	0.149	334-40	0.178	376-8	0.16	390-405	0.0149	391-4	0.0159	424-9
Oils stored for ten months										
38	0.124	330	0.197	378-81	0.19	390-8	0.0517	392-8	0.0524	402
70	0.141	330	0.210	376-8	0.19	390-8	0.0373	392-6	0.0381	402-4
100	0.129	342-46	0.154	378-80	0.14	395-402	0.0362	392-400	0.0365[2]	402-6
Oil from seed stored five months										
......	0.217	327-30	0.365	377-81	......	....	0.0116	393-6	0.0122	426-9
Oils stored five months										
38	0.217	325-30	0.359	380	......	....	0.060[2]	394-404	....	....
70	0.221	330-35	0.304	375-8	......	....	0.026[2]	394-406	....	....
100	0.222	340-45	0.263	372	......	....	0.063[2]	394-404	....	....
Oil from seed stored ten months										
......	0.185	326-8	0.358	378-82	......	....	0.0331	392-6	0.0335	400-4

[1] Absorption maximum. [2] Shoulder.

Crude oils having a broad absorption band with maxima at 378 to 382 mμ and 390 to 405 mμ can be stored for considerable periods of time without excessive development of bleach color if the extinction coefficients at the maxima and at the minimum (328 to 332 mμ) are relatively low. When the extinction coefficients of the crude oils in the aforementioned wavelength regions are initially high, indicating the presence of excessive amounts of the crude oil pigments, the oils should be held at temperatures not exceeding 70° F., and they should be refined and bleached as rapidly as possible.

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