can be attributed to the iron contamination. Observed increases in the iron content have been as high as 40-fold for this one processing operation.

9. Laboratory experiments have shown that the presence of 0.3 p.p.m. of iron during deodorization for 3 hours at 210°C. is very detrimental to the quality and stability of soybean oil. Iron at levels of 0.1 p.p.m. also lowers the initial flavor score but not significantly.

10. Oil from new beans was shown to be significantly higher in quality than oil obtained from beans crushed at the end of the season.

11. Variability in commercial deodorization of oil was sufficiently great to prevent recognition of any flavor superiority resulting from use of fresh beans.

12. Deodorization is the processing step in which improvements most logically could be made on both the quality and stability of soybean oil. A reduction of the metal contamination and the use of a metal scavenger will improve both the quality and stability of soybean oil.

Acknowledgment

The authors are grateful to Robert Beal of the Engineering and Development Division of the Northern Regional Research Laboratory for the collection of many of the commercial oils; to members of the research staffs of Allied Mills Inc., Clinton Foods Inc., The Capital City Products Company, and to the other refiners and processors who cooperated in this investigation but prefer to remain anonymous. Tribute must also be paid to the 18 members of the Northern Laboratory's Taste Panel for their continued support and services in the organoleptic evaluation of edible oils.

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Re-Refining Cottonseed Oil at High Rates of Shear[1]

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RE-REFINING, which is essentially a process for removing color bodies from an oil, is an important operation in the production of cottonseed oils intended for use in the manufacture of shortening and sometimes for other uses as well. Recent records indicate that 20-40% of the crude cottonseed oil produced in some areas is off-grade with respect to color under the trading rules of the National Cottonseed Products Association (5).

Re-refining consists of mixing a small amount of an aqueous solution of sodium hydroxide with a once-refined oil and separating the soapstock and oil. Basically, refining and re-refining are very similar processes except for variations in the strength of the sodium hydroxide solution used and several other details (2, 3), according to the ideas of the operator and the equipment available.

Cavanagh (4) has reported that crude cottonseed oil which has been degummed by the continuous soda ash process will yield a lighter-colored oil if it is subsequently treated batchwise with a solution of relatively concentrated caustic soda while being agitated at very high rates. Although Cavanagh reported data with respect to the effect of a number of variables involved in the process, he did not mention other equally essential data. For example, he reported data for the bleach colors of the re-refined oils, but not for the corresponding oils prior to bleaching, and, more important, he reported no data for the refining losses.

The following report is concerned with the application of high rates of agitation and shear in re-refining caustic soda-refined cottonseed oils, and particularly with the effects produced by the systematic variation of the factors involved in the process as applied to oils of widely divergent origins. These factors are a) rate of agitation and shear of the oil-caustic soda mixture, b) duration of agitation and shear, c) temperature of the mixture, d) amount of caustic soda used, and e) concentration of the caustic soda. The rate of agitation and shear is itself a complex variable, but at present this variable cannot be resolved into simpler components.

The relative effectiveness of the color removed by conventional and high-shear re-refining is compared, and data are reported for the comparative refining losses by the two methods.

Apparatus and Test Procedures

All re-refinings at high rates of agitation and shear were made in a one-liter stainless steel beaker, 12.4 cm. inside diameter. Three baffles, consisting of strips of stainless steel (1.5 x 15.0 cm.), were attached equi-

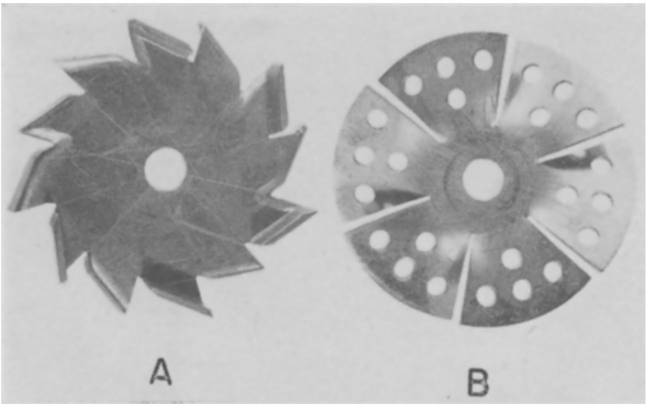

FIG. 1. Impellers employed in high-shear re-refining.

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distantly to the inner sides of the beaker which was also equipped with a gate valve for taking samples. The lower portion of the beaker was wrapped externally with copper tubing, through which water could be circulated to control the temperature of the beaker and its contents.

Agitation was provided by one or the other of the two impellers shown in Figure 1. The impeller, which was attached to a shaft inserted from the top and along the centerline of the beaker, was mounted midway between the bottom of the stainless steel beaker and the top of the oil surface. The speed could be varied step-wise between 325 and 5,000 r.p.m.

Impeller A is a laboratory model of a type used to reduce and disperse solids in slurries. Impeller B, which was designed by the authors, may be designated as a perforated, turbine type. Both impellers were fabricated from 0.0318 cm. (1/8 inch) stainless steel plate and were 7.0 cm. in diameter. The two impellers behaved alike with respect to the removal of color at high rates of shear. Impeller A produced a greater rise in the temperature of the oil than impeller B when operated at the same speed. When operating at 1,730 r.p.m. in 500 g. of oil initially at room temperature, the former produced sufficient friction to raise the temperature 0.8°C. per minute.

In all re-refining tests in which the effect of changing the variables was investigated, impeller A, unless stated otherwise, was used on 500-g. batches of oil. In each case the desired amount of caustic soda solution was added with a pipette after the impeller had been started. After agitation for a specified length of time at a given temperature, the oil-soapstock mixture was separated by rapid filtration through coarse diatomaceous earth and filter paper. In order to avoid unnecessary complications the soapstock was not separated by heating and settling.

A number of conventional re-refining tests were made in order to compare the results with those obtained at high rates of agitation and shear. The former tests were made by the official cup method (Ca 9a-41) of the American Oil Chemists' Society (1) except that a refined oil was used in place of the crude oil and the amount and strength of the caustic soda solution were changed.

Various high-shear re-refining tests were made in which the procedure for the conventional tests was followed except that the 15 minutes of mixing at 250 r.p.m. was replaced with 15 minutes of high-shear mixing, usually under conditions of temperature and amount and strength of caustic soda solution other than those used in the conventional re-refining tests.

Bleachability tests were made in accordance with the official cup method (Cc 8a-49) of the American Oil Chemists' Society except that 6% neutral, activated clay was used instead of the official clay. Colors before and after bleaching were determined by the Wesson method using Lovibond glasses. The official method (Cc 13b-45) was followed except that in some instances, which will be noted, a 70 yellow glass was used in matching the refined oils.

Effect of Variables

The effect of systematically changing each of the effective variables was investigated with two cottonseed oils designated as A and B. Oil A was prepared by refining a hydraulic-pressed crude cottonseed oil, obtained from a mill in the Southeast with an amount of 20° Bé. caustic soda sufficient to neutralize the free fatty acids (4.9%) and provide 0.3% excess sodium hydroxide on an oil-weight basis. The crude oil and caustic soda were mixed in a stainless steel kettle at room temperature for 15 minutes and the soapstock separated after warming the mixture to 65°C. The refined oil was washed and dried before re-refining. The color of the refined oil was 70 yellow and 17.0 red on the Lovibond scale, and after bleaching with 6% clay it was 35 yellow and 5.7 red. The colors of the oils obtained on re-refining oil A were always measured with the aid of a 70 yellow glass, which gave a better match color.

Oil B was prepared in a similar manner by refining a composite sample of ten crude cottonseed oils with an amount of 16° Bé. caustic soda sufficient to neutralize the free fatty acids (1.6%) and provide an excess of 0.4% sodium hydroxide on an oil-weight basis. The composite sample contained hydraulic- and screw-pressed oils. The color of the refined oil was 35 yellow and 16.2 red on the Lovibond scale, and after bleaching with 6% clay it was 35 yellow and 10.1 red.

Rate of Agitation and Shear. Samples of oil A were re-refined at various rates of agitation and shear, using 0.2% of solid sodium hydroxide as 20° Bé, caustic soda solution, a temperature of 30° C., and a mixing time of 10 minutes. The colors of the re-refined oils, determined immediately after filtration of the oil-soapstock mixture, and the colors of the corresponding bleached oils are shown in Figure 2.

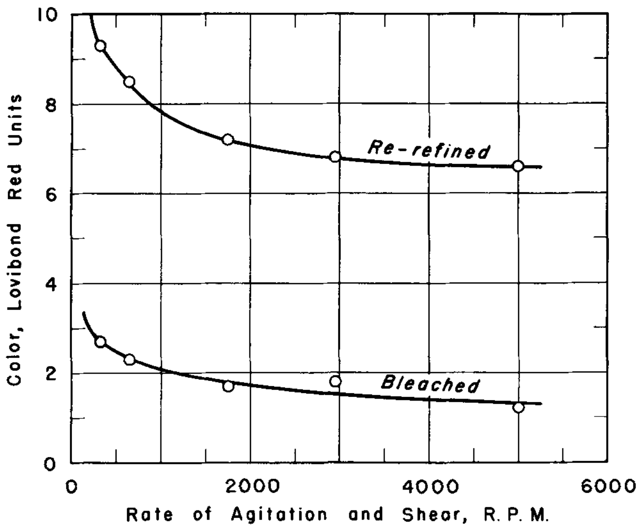

FIG. 2. Effect of agitation on color of oil A re-refined at 30° C. for 10 minutes with 0.2% NaOH as 20° Bé. solution.

It is apparent from the curves in Figure 2 that the amount of residual color decreased rapidly as the rate increased up to about 2,000 r.p.m. Increased speed of agitation beyond this rate had comparatively little effect on the amount of residual color in the re-refined oil or on the color of the corresponding bleached oil.

When oil B was similarly re-refined over a range of stirring from 370 to 3,000 r.p.m., its color decreased rapidly with increasing rate of agitation up to about 1,000 r.p.m. Further increase in the rate of agitation failed to reduce the color below a value of 6.7 Lovibond red units.

It can be concluded that, as the rate of agitation and shear increases, the amount of color removed increases until an optimum value is reached beyond which a further increase in the rate of agitation has little effect when considered with respect to the amount of color originally present. All of the rates of agitation at which effective color removal occurred (Figure 2) are above those used in conventional re-refining.

Duration of Agitation and Shear. Two series of re-refining were made with oil A, one at 30° C. with 0.3% solid sodium hydroxide as a 26° Bé. caustic soda solution and the other at 65° C. with 0.2% solid sodium hydroxide as a 20° Bé. caustic soda solution. Both series of re-refinings were made using a mixing speed of 1,730 r.p.m. From the curves in Figure 3 it is apparent that agitation in excess of 10 minutes does not result in appreciable further reduction in color of the re-refined oil. Data obtained with other oils confirmed this observation. All samples of oil B, when re-refined at 65° C. and 1,730 r.p.m. with 0.2% solid sodium hydroxide as 20° Bé. caustic soda solution and times of agitation in excess of 10 minutes, had a residual color of 5.5 Lovibond red units. However substantially more than 10 minutes are required to reach a constant minimum color value when the rate of agitation and shear is below the optimum rate. For example, when oil B was re-refined at 30° C. and 660 r.p.m. with 0.2% solid sodium hydroxide as 20° Bé. caustic soda solution, a constant minimum color (5.7 Lovibond red units) was obtained only after agitation for 30 minutes.

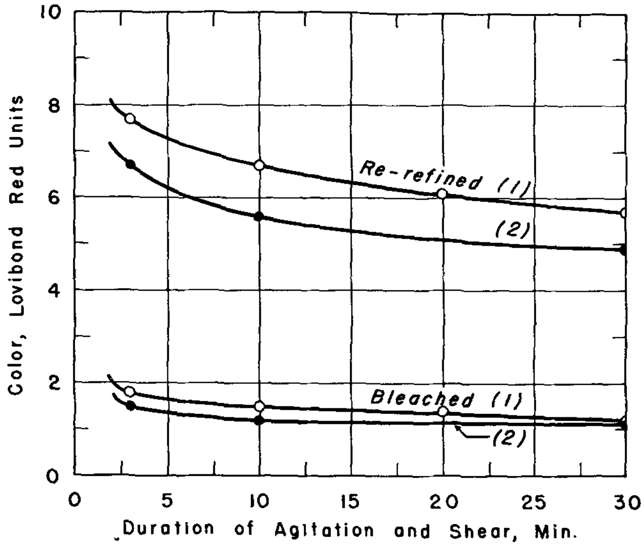

FIG. 3. Effect of time on color of oil A re-refined (1) at 30° C., 1,730 r.p.m. with 0.3% NaOH as 26° Bé. solution and (2) at 65° C. and 1,730 r.p.m. with 0.2% NaOH as 20° Bé. solution.

Temperature. The influence of the temperature of re-refining on the color of the oil was determined in two series of experiments with oil A, one with 20° Bé. and the other with 26° Bé. caustic soda solution. In both series 0.2% sodium hydroxide was used with stirring for 10 minutes at a speed of 1,730 r.p.m. Each oil was re-refined at 15, 30, 50, and 65° C. with the results shown in Figure 4. Similar results were obtained with oil B.

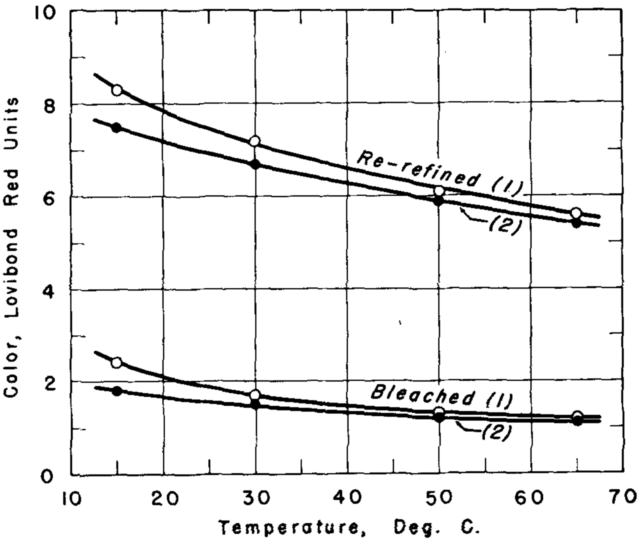

FIG. 4. Effect of temperature on color of oil A re-refined (1) for 10 minutes at 1,730 r.p.m. with 0.2% NaOH as 20° Bé. solution; (2) for 10 minutes at 1,730 r.p.m. with 0.2% NaOH as 26° Bé. solution.

The colors of the re-refined oils, before and after bleaching, always decreased as the temperature increased, up to 65° C. Increases beyond this temperature had little effect on the reduction of color of either the re-refined or bleached oils. The fact that the higher temperatures produced lighter colored re-refined oils as well as lighter colored bleached oils indicates that the higher temperatures did not "set" the residual colors.

Amount of Caustic Soda. Three series of re-refining tests were made with oil A to establish the influence of the amount of sodium hydroxide on the color of the re-refined oil. A mixing time of 10 minutes and a 26° Bé. caustic soda solution was used in each series. Two of the series of tests were made at a temperature of 30° C. with mixing speeds of 1,730 and 5,000 r.p.m., respectively. In the third series a mixing speed of 5,000 r.p.m. and a temperature of 65° C. were used. The effect of the amount of sodium

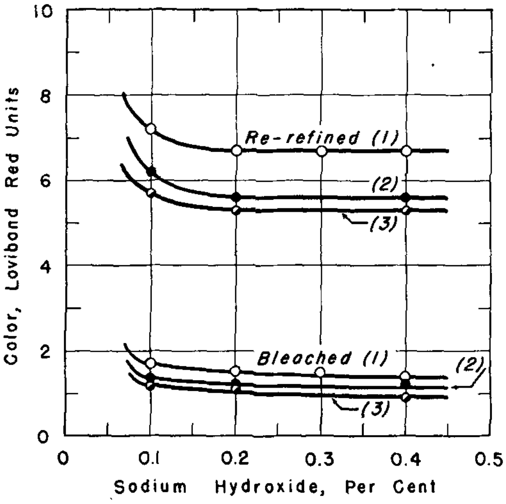

FIG. 5. Effect of NaOH (weight-percent) on color of oil A re-refined (1) for 10 minutes, 30° C., and 1,730 r.p.m. with 26° Bé. caustic soda; (2) for 10 minutes, 30° C., and 5,000 r.p.m. with 26° Bé. caustic soda; (3) for 10 minutes, 65° C., and 5,000 r.p.m. with 26° Bé. caustic soda.

hydroxide used on the color of the re-refined and bleached oils in the three series of tests is shown in Figure 5. These results are similar to those obtained with oil B.

The amount of sodium hydroxide used generally appears to have no effect on the color of oils re-refined by the high shear method, provided the amount equals 0.2% or more on an oil weight basis. With amounts of sodium hydroxide below 0.1% satisfactory reduction in color could not be obtained while amounts in excess of 0.2% increased the re-refining loss without producing further reduction in color. The optimum amount of sodium hydroxide required to obtain a minimum residual color is approximately 0.2%.

Additional data on the effect of rate of agitation on color removal are also shown graphically in Figure 5. A comparison of curves 1 and 2 (Figure 5) with the curves in Figure 2 shows that an increase in the rate of agitation above 2,000 r.p.m. has a somewhat greater effect on the residual color of the re-refined oil when the more concentrated solution of caustic soda is used.

Strength of Lye. The influence of the concentration of the caustic soda solution on the reduction of color on re-refining oil A was established by two series of tests which were alike except one series was made at 30° C. and the other at 65° C. The other conditions were a mixing speed of 1,730 r.p.m., a mixing time of 10 minutes, and an amount of sodium hydroxide equal to 0.2% on an oil-weight basis. The results of these tests are shown in Figure 6.

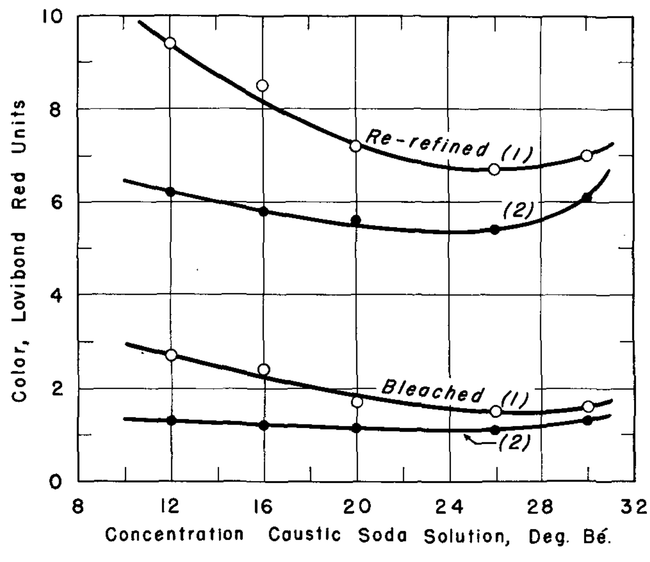

FIG. 6. Effect of NaOH (solution strength) on color of oil A re-refined (1) for 10 minutes, at 30° C., and 1,730 r.p.m. with 0.2% NaOH; (2) for 10 minutes, at 65° C. and 1,730 r.p.m. with 0.2% NaOH.

When samples of oil B were re-refined for 10 minutes at a temperature of 30° C. and a mixing speed of 1,730 r.p.m., it was found that the color of the re-refined oils decreased from 11.3 Lovibond red units to 5.5 red units as the concentration of the caustic soda used increased from 7° to 27° Bé.

The concentration of the caustic soda solution is important when quantities of sodium hydroxide as small as 0.2% are used in re-refining at high shear. At about room temperature weak caustic soda solutions are relatively ineffective in removing color. As the concentration of the caustic soda (not the amount) is increased, reduction in color increases markedly until an optimum concentration is reached.

A West Texas crude oil which was mildly refined did not conform to the general pattern. The crude oil had been screw-pressed at a low temperature from seed known to produce highly colored refined oils. The Lovibond color of this laboratory-refined oil designated as C was too dark to read accurately in a 5¼-inch tube but was estimated to be about 40 red units. With a 1-inch tube the color was 70 yellow and 8.2 red. The results of the high-shear re-refining tests made with this oil at 25° C., 3,000 r.p.m., and 10 minutes mixing time are shown in Figure 7. The upper curve was obtained using 0.2% sodium hydroxide in each test while the lower curve was obtained with 0.4% sodium hydroxide.

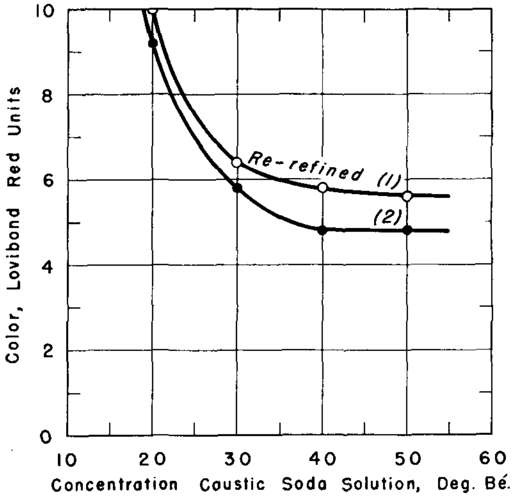

FIG. 7. Effect of NaOH (solution strength) on color of oil C re-refined (1) for 10 minutes, 25° C., and 3,000 r.p.m. with 0.2% NaOH; (2) for 10 minutes, 25° C., and 3,000 r.p.m. with 0.4% NaOH.

This oil re-refined readily but required the use of more concentrated caustic soda than other oils. Also the amount of color removed increased about one red unit as the amount of caustic soda used increased from 0.2% to 0.4%. However the original Lovibond red color was about 40, and this difference in color removal may therefore not be significant.

Comparison of Oils Re-Refined by Normal and High-Shear Methods

Four crude oils were refined by the A.O.C.S. cup method except that the amount of 20° Bé. caustic soda used was sufficient to neutralize the free fatty acids and provide 0.5% excess sodium hydroxide on an oil-weight basis. Portions of the refined oils designated as D, E, F, and G, were re-refined by the conventional method, using the same apparatus and procedure except that the amount of 20° Bé. caustic soda used provided only 0.2% of sodium hydroxide.

The refined and the re-refined oils were bleached with 6% clay, and the color of each oil was determined before and after re-refining, and before and after bleaching, with the results shown in series 1, 2, 4, 5, Table I.

TABLE I

Effect on the Color of Oils Re-Refined With Conventional and High-Shear Agitation

Series No.	Treatment of oil	Color, Lovibond units, yellow/red			
		Oil D	Oil E	Oil F	Oil G
1	Laboratory refined	70/17.9	70/14.7	70/11.3	70/6.8
2	Re-refined, conventional shear, 25° C., 20° Bé. NaOH, 250 r.p.m. (cup)	70/9.9	70/10.7	70/9.2	70/6.7
3	Re-refined, high shear, 65° C., 26° Bé. NaOH, 3,000 r.p.m.	70/4.8	70/5.8	35/4.3	70/4.6
4	Series 1, bleached	35/7.4	35/4.6	35/4.2	15/1.4
5	Series 2, bleached	35/4.1	35/3.1	35/2.7	35/1.2
6	Series 3, bleached	15/1.4	15/1.7	15/1.1	10/0.9
7	Re-refined, high shear, 25° C., 20° Bé. NaOH, 3,000 r.p.m.	70/8.1	70/8.7	70/7.7	70/5.8
8	Re-refined, conventional shear, 65° C., 26° Bé. NaOH, 250 r.p.m. (cup)	70/9.3	70/8.3	70/7.5	70/5.5

Each of the once-refined oils was also re-refined with 0.2% sodium hydroxide used as a 26° Bé. caustic soda solution. The oil-caustic soda solution was agitated at 3,000 r.p.m. and a temperature of 65° C. The mixture was transferred to a refining cup, stirred at 70 r.p.m. and the soapstock separated from the oil. The re-refined oils were bleached with 6% of clay, and their Lovibond colors determined before and after bleaching (series 3 and 6, Table I).

It may be seen from the data in Table I that a given amount of alkali will remove much more color when mixed into the oil with high speed and shear than when mixed at a conventional rate. The oils re-refined at high rates of shear also bleach as readily or more so than conventionally re-refined oils.

In the foregoing comparison of the high-shear and conventional methods of re-refining the term "high-shear method" refers to mixing at a high rate of agitation and shear plus those other conditions, such as concentration of caustic soda and temperature which are essential for maximum reduction of color.

A comparison was also made in which the only difference in the re-refining methods was the rate at which the oil and caustic soda solution were mixed.

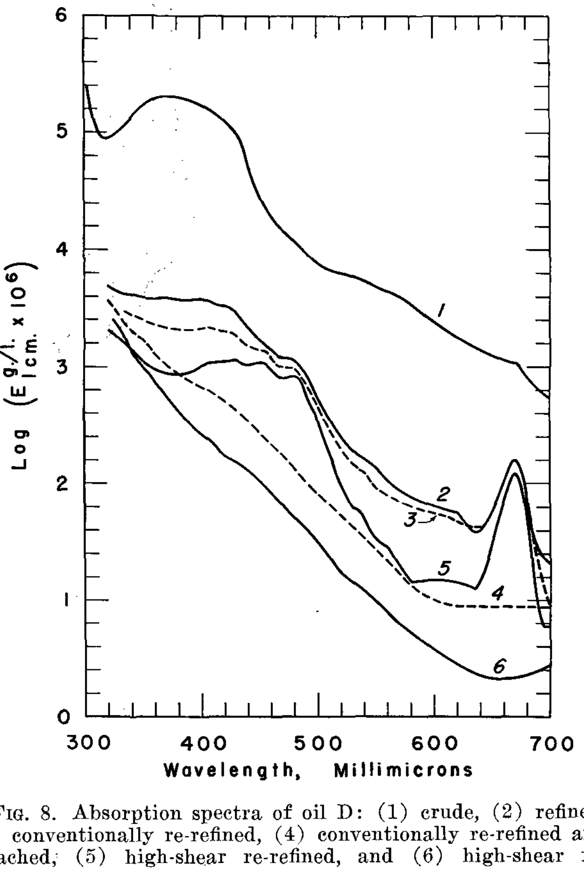

FIG. 8. Absorption spectra of oil D: (1) crude, (2) refined, (3) conventionally re-refined, (4) conventionally re-refined and bleached, (5) high-shear re-refined, and (6) high-shear re-refined and bleached.

An additional series of oils (D to G) was re-refined under the conditions shown under No. 2 in Table I except that the agitation was 3,000 r.p.m. A further series of oils (D to G) was refined under the conditions shown under No. 3 in Table I except that the agitation was 250 r.p.m. (cup method). The results of these re-refinings are given in series 7 and 8 of Table I.

Comparison of the data for series 2 and 7 and for series 3 and 8 shows that high shear agitation was more effective in removing color than conventional agitation. The difference in the reduction of color by the two methods of agitation was most marked at the higher temperature.

Oil G did not respond well to re-refining because the original refining removed practically all of the acidic color bodies.

Absorption Spectra. In order to obtain information concerning the nature of the color bodies removed in re-refining, absorption spectra of oil D were examined before and after processing by several methods. The spectral measurements were made in isooctane solution over the wave length region 300-700 mμ, using a Cary automatic recording spectrophotometer. These spectral curves are shown in Figure 8. Similar curves were obtained for oil F.

The spectral curves reproduced in Figure 8 can be divided into four principal absorption regions, each centered about the maximum absorption of one of the principal pigments or group of pigments of cottonseed oil.

In the near ultraviolet region (300-420 mμ) the absorption of the crude oil (curve 1) is caused principally by the strongly acidic gossypol and gossypol-like pigments. In the refined oil (curve 2) these pigments have been largely removed. If the absorption at 380 mμ for the refined oil is assumed to be 100%, then conventional re-refining (curve 3) removes 45% of the pigments causing this absorption compared to 78% removed by high-shear re-refining (curve 5). It should be noted that removal of pigments having maximum absorption in a given region affects to some extent the entire spectrum by decreasing the background absorption in other regions.

Yellow, nonacidic carotenoid pigments have their maxima absorption in the blue region of the spectra (420-480 mμ). Removal of the gossypol-like pigments by refining clearly reveals the characteristic absorption of the carotenes. As might be expected, neither conventional nor high-shear re-refining has any appreciable effect on these pigments, which are however removed by bleaching (curves 4 and 6).

The spectrum of the refined oil shows that several unidentified red pigments absorbing in the region of 480-640 mμ are present but in such low concentrations that no characteristic maxima are detectable in this region. Only a small proportion of these pigments is removed by conventional re-refining whereas they are very effectively removed by high-shear re-refining. This is apparent from the fact that the absorption of the refined oil at 600 mμ is decreased only 15% by conventional re-refining compared to 78% by high-shear re-refining.

The absorption at 670 mμ is characteristic of pheophytin. Neither re-refining process has any effect on the removal of this pigment which is however effectively removed by bleaching.

Re-Refining Losses

The preceding section has dealt entirely with the reduction of the color of cottonseed oils processed by high-shear re-refining. Of equal importance is the effect this method has on the refining loss of the oil.

When strong caustic soda solution is dispersed in a once-refined cottonseed oil, the alkali reacts with the acidic pigments, and simultaneously the alkali saponifies some of the glyceride constituents with the formation of soaps of the fatty acids. It is assumed that the colloidal soap particles absorb the color bodies which are present in the oil. Quantitative data to support this assumption are lacking, but the following experiments indicate adsorption is not of major importance in color removal.

A cottonseed oil was re-refined by the high-shear method before and after the addition of 1% to 5% of purified and practically colorless cottonseed oil fatty acids. Additional caustic soda sufficient to neutralize the free fatty acids was added. In every case the addition of free fatty acids increased the color of the re-refined oil by 1 to 3 Lovibond red units. On the other hand, the colors of rice bran and palm oils, which are almost devoid of acidic pigments, were practically unaffected after being re-refined by the high-shear method.

Apparently adsorption is not a primary factor in color removal by high-shear re-refining. Therefore color removal should be more or less independent of the refining loss produced by saponification. It should be possible to use a concentrated caustic soda solution in the range where it effectively removes color bodies and then remove any excess sodium hydroxide before it begins to act solely as a saponifying agent.

Actual re-refining losses found by filtering the oil-caustic soda mixture immediately on completion of the high-shear mixing indicated that the color removal consumed less than half of the added alkali. For example, when oil A was mixed for 10 minutes at 30° C. and 1,730 r.p.m. with 0.2% of sodium hydroxide in the form of a 26° Bé. caustic soda solution, 99.1% of the oil was recovered by filtration; that is, the re-refining loss was only 0.9%. Another re-refining, otherwise identical except that a temperature of 65° C. and 30° Bé. caustic soda was used, resulted in a re-refining loss of only 0.7%. If all the sodium hydroxide present had been consumed in saponifying the oil, the loss would have been at least 1.4% even though no neutral oil was emulsified in the soapstock.

Re-refinings at high-shear were also made in which the oil-soapstock mixture was warmed to 65° C., stirred at low speed (70 r.p.m., cup apparatus) for 12 minutes, allowed to settle for one hour, cooled, and the oil separated by decantation. The loss with oil A was 1.8% when it was re-refined with 0.2% sodium hydroxide as 20° Bé. caustic soda while agitated for 10 minutes at 30° C. and 5,000 r.p.m. In other re-refinings, which were identical except that in one case a temperature of 65° C. was used and in the other 30° Bé. caustic soda was used with 1,730 r.p.m. agitation, the losses were 1.5% and 2.6%, respectively. These losses either are equal to or differ by only a few tenths percentage unit from those found when the re-refindings were conducted using the A.O.C.S. cup apparatus. In other words, the use of high-shear mixing in the otherwise conventional re-refining procedure results in re-refining losses not significantly different from those obtained with low-speed agitation.

Summary

1. A number of refined cottonseed oils were re-refined under various conditions at high rates of agitation and shear.

2. The effect of the several variables in removing the color of these oils was investigated. The color of a re-refined cottonseed oil was found to decrease as the rate of agitation increased until a rate was reached beyond which a further increase in agitation had little effect. With high rates of agitation and shear, color removal was most rapid during the first five to 10 minutes. After 10 minutes the rate of color removal was very slow. About 0.1% of sodium hydroxide was the minimum and 0.2% the optimum amount required to produce the maximum improvement in color at high rates of shear. These amounts of sodium hydroxide were best utilized in the form of 20° to 30° Bé. solutions. As the temperature of re-refining increased, the color of the re-refined oil decreased. Between 15° C. and 65° C. the removal of color was almost a linear function of the re-refining temperature.

3. Evidence was obtained to show that the removal of color by re-refining cottonseed oil at high rates of agitation and shear is relatively independent of the re-refining loss. Losses by the high-shear and conventional methods of re-refining were almost identical.

4. Usually the residual color of the oils re-refined by the high-shear method was only half that observed in the same oils after conventional re-refining. This proportional difference in color was also observed after bleaching.

5. High-shear re-refining is more effective in removing the acidic gossypol-like pigments from cottonseed oil than is conventional re-refining. In addition, the former method removes a high percentage of unidentified red pigments which are unaffected in conventional re-refining. On the basis of the response of the gossypol pigments to high-shear re-refining it can be assumed that these unidentified red pigments are also acidic in nature. The failure of the conventional method of re-refining to remove these acidic pigments results from the incomplete dispersion of the aqueous alkali in the oil phase at the low speed of stirring and shear used in conventional re-refining. The failure to remove them in the original refining is attributable to the same cause and to the use of insufficiently concentrated alkali to react with the weakly acidic pigments.

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Solvent Extraction of Cottonseed and Peanut Oils. IX. Determination of Fines in Miscella

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IN the continuous solvent extraction of oilseeds the quantity of fine meal solids (fines) that will be carried out from the extractor with the oil-solvent mixture (miscella) depends upon the oilseed, the method of preparation, and the extractor used. Fines (1) create a problem. This problem is in proportion to the quantities involved. In soybean solvent extraction, for example, the miscella usually contains fines less than 0.2% by weight of the extractor feed material. With quantities as small (3) as these the clarification step, or the removal of the fines before evaporation and stripping, can be easily achieved by direct filtration. The problem is different in extracting cottonseed, peanuts, okraseed, and rice bran. With these materials the fines in the miscella may be as high as 5% by weight of the feed material, and settling or centrifugation (3) of the miscella is required before filtration.

It is desirable therefore to control as far as possible the amount of fines produced. A rapid, on-the-spot method for the determination of fines in miscellas has been developed and is presented here. The method is volumetric and replaces the slower gravimetric method, which consists of weighing the filter cake after a complete cycle and calculating what the average fines content of the miscella had been. The volumetric determination is converted to weight values by the use of an appropriate curve. The construction of the curve and its application in pilot-plant operations to cottonseed, peanuts, rice bran, and okraseed are described.

Experimental Data

The miscella samples used for the construction of the curves in Figures 2 and 3 were obtained from a continuous solvent-extraction pilot plant (2), which had a capacity of approximately 150 lb./hr. of flakes. In all runs commercial hexane was used. Miscella samples were taken before filtration.

The solids content of the unfiltered miscella samples was determined both gravimetrically and volumetrically. To determine gravimetrically the grams of solids per liter of clear miscella in a sample the following data were recorded: weight in grams of unfiltered miscella (slurry) sample and of solids from slurry after filtering and the specific gravity of the filtered miscella. These data can be substituted in the following formula to calculate the points along the ordinate of the chart:

$$\frac{(\text{wt. solids})(\text{sp. gr.})(1{,}000)}{\text{wt. slurry}-\text{wt. solids}} = \frac{\text{grams solids}}{\text{liter filtered miscella}}$$

The units involving filtered miscella were chosen for the ordinate of the plot because most solvent-extraction plants meter the clear miscella flow from the filter(s).

To determine volumetrically the solids content, duplicate samples of the miscella were spun for 10 min-

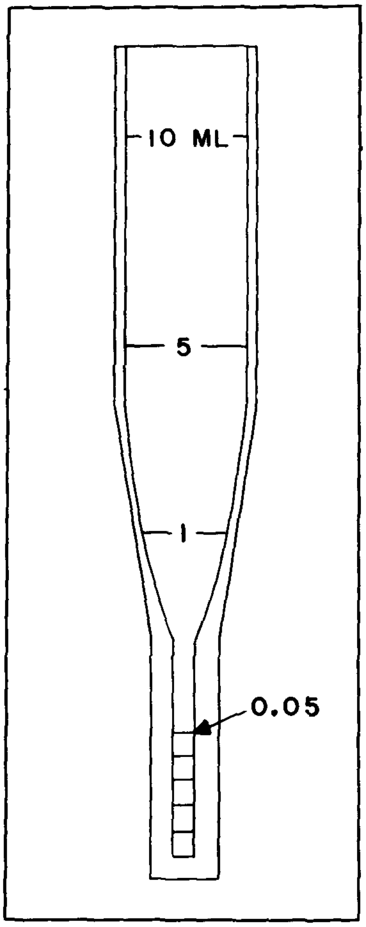

FIG. 1. Centrifuge tube used for experiments.

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