# Report of the Bleaching Methods Committee 1941-1942

The formation of this Committee resulted primarily from the publication in the oil and fat literature of the results of an investigation which showed that our official method for the bleach test may give erratic test results when applied to certain oils, for example, soybean oil. (OIL & SOAP, August, 1939: "A Study of Some of the Factors Affecting the Laboratory Bleach of Soybean Oil," by C. J. Robertson, R. T. Munsberg, and A. R. Gudheim.)

It was observed that the results were higher according to the length of time the oil might be agitated after it had been heated to the bleach temperature; and next, even though the oil may be bleached immediately after reaching temperature, the results were somewhat higher when the rate of heating to 120° C. was relatively slow. A carefully planned series of experiments elucidated these effects in some detail, and the expression "heat darkening" was applied to the occurrence. It was then shown that this "heat darkening" was eliminated if the oil were heated to the bleaching temperature after the standard fuller's earth had been added, using agitation during heating. The test results were then found to be independent of the heating time, and the values of the bleach test results obtained were just what would be expected in the absence of "heat darkening."

At this Committee chairman's suggestion, the Refining Committee of 1939-1940 performed some work incidental to the refining program, comparing the official method with the bleaching method which was recommended by the authors of the above paper for use on soybean oil; and the results on the average confirmed the magnitude of the difference obtained in bleaching under these two conditions, i.e., 0.2 to 0.3r lower results by the elimination of "heat darkening."

In view of the increased and expanding importance of soybean oil in the domestic oil and fat supply, and with the knowledge that a bleaching test method formulated primarily for cottonseed oil was being applied generally to soybean oil, it was considered desirable to confirm the above mentioned results by committee action.

Each Committee member was asked to make comparative bleach tests on at least two samples of refined soybean oil of his own choosing, using the A.O.C.S. official, and the suggested modified, methods. In addition, two check samples were sent out to the Committee membership for similar comparative tests. In one series of samples, two different heating times were employed. The results, given in the accompanying table, again show that heating the oil for test in contact with bleaching earth seems to eliminate the "heat darkening" and the average difference in the results is the same as reported previously.

The effect of using different heating times in the case of the official method is not too clearly shown by these results. This effect, however, within a relatively narrow range of heating times, say 3 to 6 minutes, is quite small, only .1 to .2r, and consequently might easily be masked by uncontrolled variations.

Since the investigation mentioned above (see reference) included a study of the behavior of cottonseed oil from the standpoint of "heat darkening," which was found not to occur during the heating of that oil for the bleach test, this Committee confined its attention solely to soybean oil. This was considered to be especially desirable in view of the long and proven experience behind the use of the official bleach test on cottonseed oil, and the fact that the test is used extensively as the basis of trade settlements, which is not true in the case of soybean oil.

In view of the now well-established occurrence of the "heat darkening" effect when our present bleach test is applied to soybean oil, as shown by this Com-

COMPARATIVE BLEACH TEST RESULTS OF EXTRACTED SOYBEAN OIL TEST REFINED AS PART OF THE WORK OF THE REFINING COMMITTEE OF 1939-1940

H. S. MITCHELL, Swift & Company, Chairman

Two bleaching conditions: (1) American Oil Chemists' Society Official Method (matched yellow).
(2) Lever Brothers Modification (Ref. OIL & SOAP, August, 1939).

Eighteen laboratories participated in the work of that Refining Committee, of which six made their bleach tests by both methods. Only the results of those six are considered here since our main concern at this time is a comparison between the A.O.C.S. Method and the Lever modification.

| | Caustic | Laboratory No. / Method | 4 | 7 | 12† | 14 | 15 | 18 | Red Average | Difference A.O.C.S.—Lever |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | Y R | Y R | Y R | Y R | Y R | Y R | | |
| Sample No. 3 | 12° Be. ⅔ Max. | A.O.C.S. | 30—1.4* | 25—2.3 | 35—2.6 | 35—1.9 | 30—2.4 | 20—2.5 | 2.34 | .52 |
| | | Lever | 20—1.3* | 20—1.7 | 30—2.2 | 35—1.5 | 25—1.9 | 20—1.8 | 1.82 | |
| | 12° Be. ½ Max. | A.O.C.S. | 30—1.4* | 25—2.3 | 35—2.4 | 35—2.0 | 30—2.3 | — | 2.25 | .45 |
| | | Lever | 20—1.3* | 20—1.7 | 30—2.0 | 35—1.6 | 25—1.9 | — | 1.80 | |
| Extracted type | 14° Be. ⅞ Max. | A.O.C.S. | 30—1.4 | 25—2.0 | 30—2.3* | 35—1.7 | 30—2.1 | 25—2.1 | 2.04 | .30 |
| | | Lever | 20—1.3 | 20—1.6 | 25—2.0* | 35—1.6 | 25—1.9 | 20—1.6 | 1.74 | |
| | 14° Be. ⅔ Max. | A.O.C.S. | 30—1.4 | 25—2.1 | 35—2.7* | 35—1.8 | 30—2.1 | 25—2.1 | 2.02 | .37 |
| | | Lever | 20—1.3 | 20—1.6 | 25—2.4* | 35—1.5 | 25—1.9 | 20—1.6 | 1.65 | |
| Sample No. 4 | 12° Be. ⅔ Max. | A.O.C.S. | 30—1.8 | 25—1.8 | 35—2.1 | 35—1.3 | — | 20—2.0 | 1.77 | .17 |
| | | Lever | 30—1.6 | 20—1.5 | 30—2.4* | 20—1.4 | — | 20—2.0 | 1.60 | |
| Extracted type | 12° Be. ½ Max. | A.O.C.S. | 35—1.9 | 25—2.0 | 35—2.5* | 35—1.5* | — | 20—2.0 | 1.97 | .35 |
| | | Lever | 30—1.6 | 20—1.5 | 25—2.1* | 20—1.5 | (Lab. 16) | 20—1.9 | 1.62 | |
| | 14° Be. ⅞ Max. | A.O.C.S. | 30—1.8 | 25—1.7 | 25—2.0 | 35—1.3* | 20—1.9 | 20—1.9 | 1.86 | .26 |
| | | Lever | 30—1.6 | 20—1.5 | 25—2.0 | 35—1.5 | 20—1.9 | 20—1.5 | 1.60 | |
| | 14° Be. ⅔ Max. | A.O.C.S. | 30—1.8 | 25—1.8 | 30—2.4* | 35—1.6 | — | 20—1.9 | 1.80 | .30 |
| | | Lever | 30—1.6 | 20—1.5 | 30—2.4* | 20—1.5 | — | 20—1.5 | 1.50 | |
| | | | | | | | | Average difference between A.O.C.S. and Lever | | .34 |

* Omitted from average.
† Reported also .1 to .4 blue.

Refining Committee members represented here may be identified by referring to report of Committee mentioned above. The mean (+ and —) deviations of the red colors of all the laboratories whose data was included in the average red ranged from as low as .035r up to .19r for the various conditions. There was no significant difference in the deviations as between the A.O.C.S., and Lever modifications.

mittee's work and the work of others, this Committee recommends the adoption of a method employing a slightly modified procedure, designed to eliminate this "heat darkening" from the results. This procedure is to be designated for use exclusively on soybean oil, so that the present official method as it now stands will remain unchanged.

The Committee therefore recommends that the section in our methods covering the bleach test be rewritten as follows:

REFINED OILS—BLEACHING

(a) *Apparatus.* Scales, weights, refining cups and stirring machine are to be similar to those specified under REFINING, but with T-shaped paddles one-half inch wide instead of one inch wide.

Gas burners or electric heaters to heat the oil in the cups.

Official fuller's earth: This is obtained from the Secretary of the American Oil Chemists' Society. A fresh supply must be used each year beginning August 1.

(b) *Determination.* Cottonseed and other oils, except soybean—Weigh 300 grams of refined oil into a refining cup; heat to 120° C. and add 6 per cent of official fuller's earth. Stir mechanically at 250 R.P.M. (plus or minus 10) for five minutes, not allowing temperature to fall below 105° C.

Soybean Oil—Weigh 300 grams of refined oil into a refining cup; add 6 per cent of official fuller's earth, and using mechanical agitation at approximately 250 R.P.M. heat immediately to 120° C. taking not more than five minutes. Then stir mechanically at 250 R.P.M. (plus or minus 10) for five minutes, not allowing temperature to fall below 105° C.

Filter through filter paper. After sufficient oil has passed the filter to insure clearness, collect a sample for color reading. Cool and read color immediately as prescribed under Color.

The Committee's recommendation is that the procedure constituting the paragraph headed "Soybean Oil" be adopted as a tentative method of the Society.

## Notes

The Committee realizes that the Society's methods as a whole are in the process of undergoing a somewhat comprehensive revision of form and arrangement, and accordingly submits the above with the thought that the Uniform Methods and Planning Committee, which is supervising the revision, may wish to change the wording of the method here recommended or to fit it in differently. The Committee believes that the arrangement submitted makes for clarity and is more desirable than one which attempted to embody both bleach test procedures in one method or in one paragraph.

*Heating with agitation.* Considerable thought was given to this subject in formulating the new method, especially with respect to designating a procedure which on the one hand would not permit any variations which might cause errors, and on the other hand would not be unnecessarily rigid so as to occasion the design of elaborate or expensive additional apparatus or render obsolete any apparatus now in use. Specifically, it is apparent that *during the brief heating period,* especially considering that the mixture is in the effective bleaching temperature range perhaps less than one minute, neither the *speed* of agitation nor the *type* of agitator used is critical. On the other hand, we believe that it is important for the agitation to be continuous and vigorous enough to prevent the earth from settling. Hence the designation of me-

SOYBEAN OIL BLEACH TEST RESULTS 1941-42 COMMITTEE

Lovibond red and yellow. Time taken in heating to 120° C. in parentheses

COLLABORATIVE

| | Spencer Kellogg | Proctor & Gamble | Durkee Famous Foods | Staley | Central Soya | Urbana * |
|---|---|---|---|---|---|---|
| Check Sample No. 1 | | | | | | Heating times 5 to 6′ |
| (A) Quick Heat, A.O.C.S. | 25y—3.6r(2′) | 35y—3.4r | 35y—3.6r(3¼′) | | | 35y—3.72r |
| (B) Slow Heat, A.O.C.S. | 25y—3.6r(6′) | | | 21y—3.3r(5′) | 35y—2.7r | 20y—2.68r |
| (C) Lever Modified | 25y—3.4r(4′) | 35y—3.3r | 35y—3.3r(2¾′) | 20y—2.8r(5½′) | 35y—2.4r | 1.04 |
| Difference, r | 0.2, 0.4 | 0.1 | 0.3 | 0.5 | 0.3 | 20y—1.95r |
| Color Reading Check | 35y—3.3r | 28y—2.8r | 35y—2.5r | 20y—3.1r | 35y—2.0r | |
| Check Sample No. 2 | | | | | | |
| (A) Quick Heat, A.O.C.S. | 35y—2.5r(4′) | 25y—2.5r | 35y—2.3r(2½′) | | | |
| (B) Slow Heat, A.O.C.S. | | 25y—2.6r | | 19y—2.3r(5½′) | 25y—1.8r | 15y—1.63r |
| (C) Lever Modified | 35y—2.3r(3′) | 21y—2.3r(3′) | 35y—2.3r(3½′) | 18y—2.0r(5¼′) | 25y—1.7r | 15y—1.5r |
| | | 21y—2.2r(6′) | | | | |
| Difference, r | 0.2 | 0.2, 0.4 | 0.0 | 0.3 | | 0.13 |

ON MISCELLANEOUS SAMPLES

| Method | A.O.C.S. | | | | |
|---|---|---|---|---|---|
| Condition | (A) Quick Heat | (B) Slow Heat | (C) Lever Modified | Difference | Sample |
| S. K. (Freyer) | 35y—5.2r(3′) | 55y—5.4r(6′) | 35y-4.9r | 0.3r | X |
| | 30y—2.5r | | 30y—2.2r | 0.3 | Y |
| | 35y—3.8r | | 35y—3.6r | 0.2 | Z |
| P. & G. (Sanders) | 25y—2.9r | | 25y—2.6r | 0.3 | Sample A |
| | 25y—2.7r | 25y—2.6r | 20y-2.4r(3′), 20y-2.3r(6′) | 0.3 | Sample B |
| | 25y—2.6r | 25y—2.6r | 20y-2.4r(3′), 20y-2.3r(6′) | 0.2, 0.3 | Sample C |
| Durkee (Spielman) | 35y—2.7r(2½′) | 35y—2.6r(6′) | 35y—2.4r(3′) | 0.3 | Extracted Oil |
| | 40y—4.8r(3′) | 40y—5.1r(6¾′) | 40y—4.4r(3′) | 0.4 | Expeller Oil |
| Staley (M. M. Durkee) | 21y—3.8r(3′) | 21y—3.7r(5′) | 21y—3.4r(4½′) | 0.3 | Sample No. 1 |
| | 20y—4.2r(3′) | 20y—4.2r(5⅓′) | 20y—3.7r(5¼′) | 0.5 | Sample No. 2 |
| Central Soya (Kruse) | | 25y—1.3r | 20y—1.2r | 0.1 | |
| | | 25y—1.3r | 20y—1.3r | 0.0 | |
| Urbana (Wheeler)* | | 35y—4.17r(5′—6′) | 35y—3.35r(5′—6′) | 0.82 | "Commercial Alkali Refined Oil" |

(A) Requested to heat in "less than 3 minutes."
(B) Requested to heat in about 6 minutes.
(C) Requested to heat with agitation in from 3 to 6 minutes.

* U. S. Regional Soybean Industrial Products Lab.

chanical agitation during heating. On the basis of observations of the apparatus used in a number of oil laboratories, and considering various possibilities, especially one wherein the simplest arrangement of apparatus is that in which the source of heat, whether it be electric or gas, is integral with the bleaching apparatus (and hence not requiring any transfer of the test after it reaches bleaching temperature), it appears best to designate for the heating stage the same kind of paddle as now designated for the bleaching, and to specify that the agitation be at "approximately 250 R.P.M." Since, as stated above, the agitation *during the heating period* is not critical, it is not necessary to designate any specific speed, such as the "250 (plus or minus 10) R.P.M." for the bleaching stage, in the present method. Thus, in laboratories in which adequate heating apparatus is *not* integral with the multi-unit, fixed speed stirring apparatus, the use of small, controlled and variable speed electric or air-driven agitators becomes permissible. With little experience one can judge 250 R.P.M. plus or minus 25 R.P.M. quite easily.

One outstanding feature of the results shown here is represented by the rather wide discrepancies between the results of different laboratories in the case of the check samples. A single sample of bleached soybean oil sent to the Committee members for a check on the color reading factor alone yielded results which indicate strongly that these discrepancies are largely due to the color reading factor. At least two possible causes for this were brought out in the study: (1) variability in the clarity of the filtered bleached sample; (2) uncertainty, and hence variability in the amount of yellow to use in making the color match, which emphasizes the need of conducting further work on this test. The Committee therefore recommends that it be continued another year: (1) to determine the effect of using different kinds of filter papers, and (2) to attempt to settle the question of the yellow component of the color reading, and (3) to follow any other lines of investigation which may be indicated.

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# Report of the Cellulose Yield Committee 1941-42

During the year the Cellulose Yield Committee sent out two sets of samples of linters and hull fibre. Each set consisted of four linter and two hull fibre samples. These samples were sent to laboratories which had the necessary equipment for determining the yields as outlined in the A.O.C.S. method for determining cellulose yields.

The following table gives the results of samples sent out on January 10. All results are calculated to 7 per cent lint moisture basis.

TABLE I

| | Yields | | | | | | |
|---|---|---|---|---|---|---|---|
| Laboratory No. | Hull Fibre | | Linters | | | | Overall Average |
| | A | B | C | D | E | F | |
| 1* | 64.2 | 66.9 | 66.0 | 67.6 | 71.9 | 78.6 | 69.20 |
| 2* | 64.8 | 68.1 | 66.3 | 70.1 | 73.5 | 79.4 | 70.36 |
| 3* | 62.6 | 66.8 | 65.3 | 69.0 | 72.3 | 77.9 | 68.98 |
| 4* | 64.5 | 68.5 | 65.0 | 68.1 | 74.1 | 78.1 | 69.71 |
| 5* | 66.6 | 69.5 | 65.0 | 68.7 | 72.0 | 78.1 | 69.98 |
| 6* | 65.7 | 69.5 | 65.1 | 69.4 | 73.3 | 80.0 | 70.50 |
| 7 | 63.5 | 67.0 | 65.6 | 68.7 | 72.4 | 78.8 | 69.33 |
| 8 | 63.6 | 67.2 | 66.3 | 69.8 | 73.3 | 78.8 | 69.83 |
| 9 | 65.1 | 69.5 | 66.5 | 69.1 | 72.7 | 77.8 | 70.11 |
| 10 | 64.2 | 68.1 | 65.5 | 68.7 | 72.9 | 78.4 | 69.63 |
| Av. | 64.48 | 68.11 | 65.66 | 68.92 | 72.84 | 78.59 | 69.76 |

* Members of Cellulose Yield Committee.

The overall average of all samples is good. A few individual tests could be better.

Table II gives the results of samples sent on February 5.

The results of the last set of check samples are better than those obtained in Table I.

The individual yield variations are due to (1) not following the procedure in detail and (2) nonuniformity of the low yield linters produced for the past season. The samples sent out do not represent the average of lint yield produced but do represent the maximum range.

The results as a whole are satisfactory.

The cellulose yield method was adopted as a tentative procedure by the A.O.C.S. in 1937. Since that time it has been on that basis. At present it is being used 100 per cent as a basis for the purchasing of second cut linters and hull fibre for chemical uses.

TABLE II

| | Yields | | | | | | |
|---|---|---|---|---|---|---|---|
| Laboratory No. | Linters | | | | Hull Fibre | | Average |
| | A | B | C | D | E | F | |
| 1* | 74.2 | 67.7 | 77.2 | 72.7 | 62.0 | 69.6 | 70.57 |
| 2* | 74.3 | 67.6 | 77.1 | 72.7 | 64.3 | 69.8 | 70.97 |
| 3* | 73.2 | 67.4 | 76.1 | 72.6 | 64.4 | 71.1 | 70.80 |
| 4* | 73.8 | 66.0 | 77.3 | 72.0 | 66.7 | 68.4 | 70.70 |
| 5* | 73.1 | 66.8 | 76.2 | 71.6 | 62.2 | 68.8 | 69.79 |
| 6* | 72.4 | 65.8 | 76.6 | 71.4 | 61.8 | 68.8 | 69.47 |
| 7 | 73.4 | 67.7 | 77.3 | 72.2 | 65.0 | 69.7 | 70.88 |
| 8 | 73.1 | 66.4 | 76.2 | 71.3 | 65.8 | 69.1 | 70.32 |
| 9 | 73.1 | 65.8 | 76.8 | 71.9 | 64.2 | 69.0 | 70.13 |
| 10 | 72.8 | 65.7 | 77.8 | 72.5 | 63.3 | 69.5 | 70.27 |
| 11 | 73.9 | 67.8 | 77.0 | 72.8 | 63.0 | 69.4 | 70.65 |
| Av. | 73.39 | 66.79 | 76.87 | 72.15 | 63.88 | 69.38 | 70.41 |

* Members of Pot Cook Yield Committee.

## Recommendations

(1) That the Cellulose Yield procedure be adopted as an official method to be known as "The American Oil Chemists Society Cellulose Yield Method."

(2) That check samples be sent out several times during the next year.

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