

Report of the A.O.C.S. Refining Committee 1942-1943

Two refining tests have been under investigation by the Refining Committee during the past year. One of these was a continuation of the work on extracted soybean oil. The activities of the Regional Soybean Laboratory were moved during the year to the Northern Regional Research Laboratory at Peoria, Illinois. Fortunately for our committee work, Dr. R. T. Milner, Chief of Analytical and Physical Chemical Division of Peoria and Vice-Chairman of the Refining Committee, has arranged to carry on the investigational program which had previously been followed by Dr. D. H. Wheeler. Dr. Milner's report of the work done since February, 1943, follows:

Refining Loss of Soybean Oil

On April 24, 1940, a meeting of the Refining Committee was held at the Soybean Laboratory at Urbana to discuss and outline a program of work. Complete reports of the Refining Committee on this work have been made in 1941 and 1942. This report is shorter, not because the problems have been solved, but because the chemical work on soybeans was transferred from Urbana to Peoria with consequent delay in reassembling apparatus, and because the war has limited the personnel available for this problem. The work presented here was started February 15, 1943, and it is hoped that the present progress may be maintained so that formal action by the committee may be taken during the coming year.

A general summary of the problem from the viewpoint of those now working on it seems desirable. A recent article in *Oil and Soap*, 19, 97 (1942), has discussed the refining of vegetable oils with emphasis on the history of the method and the apparatus used. The "refining loss" of soybean oil may be considered as caused by three factors:

- I. Non-oil constituents present in crude oil, such as free fatty acids, phosphatides, pigments, sterols, gums, proteins, etc.
- II. Soaps formed by the alkali used in refining, acting on the neutral oil.
- III. Neutral oil entrained by the above two types of materials.

The foots obtained by the refining procedure are the sum of these three fractions. The amount of any one fraction may influence the amounts of the other fractions.

The present official A.O.C.S. refining methods are satisfactory for almost all soybean expeller and hydraulic oils and for most extracted oils. Some solvent-extracted oils cannot be refined by the present tentative method. It is of considerable importance to know the cause and remedy for this failure.

The amount of sodium hydroxide used is of primary importance for fraction II, above, and the 1941 Committee report (*Oil and Soap* 18, 208 [1941]) in Series I presented data on this factor. It has proved convenient to express the amounts of NaOH used per 100 grams of oil as "theory plus excess," as discussed in the 1941 report. By "theory" is meant the amount of NaOH needed to neutralize the fatty acids present. [Theory = (% F.F.A. \times .142) gm. NaOH]. The excess caustic therefore may be regarded as responsi-

ble for the saponification of neutral oil. The results obtained to date in 1943 are given in rather complete detail in Table I, headed "Solvent Extracted Oils." The third column shows the method used, "AOCS" referring to the present tentative A.O.C.S. method, and "Cent." to the centrifugal refining method proposed in the 1942 report (*Oil and Soap*, 19, 161 [1942]) except for variations in the total amount of caustic used in both methods. Column 4 shows the strength of alkali used. The next four columns show the amounts of alkali used, based on 100 grams of oil, since the centrifuge method uses 200 grams while the A.O.C.S. method requires 500 grams. Column 5, headed "Max." shows the amount of alkali used expressed as a fraction of that calculated from the present A.O.C.S. official procedure. Column 6 (A) shows the weight of dry NaOH required by the F.F.A. in 100 grams of oil; column 7 (B) the weight of dry NaOH used, in excess of that required, and column 8 (C) the total weight of alkali solution used per 100 grams of oil. It is evident that A + B represents the total weight of dry NaOH used, and that C - (A + B) gives the total weight of water used for refining 100 grams of oil. No comment is needed on the headings of the next three columns. In describing the condition of the foots and refined oil, columns 12 and 13, an attempt was made to grade the foots as obtained, before remelting. By inspection the foots were classed as very firm, firm, medium, soft, and so forth, and the oil as clear, slightly cloudy, cloudy, or turbid.

Of the twelve crude extracted oils examined, numbers 258, 265, and 268 were oils studied by this committee previously, but the older results, published in the 1941 report, are not comparable with those presented here since some material has settled out of the oil on standing. Oils S-868 and S-869 were obtained from the same processor who commented that S-868 had given sloppy foots by the official A.O.C.S. method, and that S-869 was taken from current production before drying and would be expected to give trouble because of the presence of some solvent in the oil. Moisture and volatile matter determined on these oils showed .37 per cent for S-868 and .50 per cent for S-869, with both oils having a break of less than .1 per cent. It is evident that the amount of solvent present in S-869 was small, but it is of interest to note that this was one of the few cases in which no results at all could be obtained by the A.O.C.S. method. Oil AOCS-1 is a crude extracted oil sent out by the Referee Committee. The average of results obtained on this sample by the 30 chemists reporting was 3.3 per cent for $\frac{7}{8}$ Max. and 3.2 per cent for $\frac{2}{3}$ Max. with standard deviations of .33 and .25 per cent, respectively. The remaining six oils, AO-6, AO-7, AO-9, AO-11, AO-12, and AO-13, represent samples of crude extracted oil from six different processors from the current crop of beans.

One encouraging aspect of the present results is that the centrifuge method has given firm foots for all the oils. Regardless of whether this method is adopted, it offers a sure means for evaluating an oil when the present official method fails. With oils 258

TABLE I
Solvent Extracted Oils

Oil Number	F. F. A. (%)	Method	Bé NaOH	Max.	Theory Dry NaOH gm. A	Excess Dry NaOH gm. B	Weight NaOH Solution gm. C	Number Remelts	Refining Loss %	Color 70 Y + R	Foots	Oil	Remarks
258	.54	AOCS	14°	7/8	.08	.48	5.88	1	3.9	Firm	Turbid	
258	.54	AOCS	14°	7/8	.08	.48	5.88	1	4.2	Firm	Turbid	
258	.54	AOCS	14°	7/8	.08	.48	5.88	2	4.8	Soft and oily	Turbid	
258	.54	AOCS	14°	7/8	.08	.48	5.88	2	4.8	Soft and oily	Turbid	
258	.54	AOCS	14°	1.06	.08	.60	7.08	1	4.3	7.9	Firm	Turbid	Some water in oil
258	.54	AOCS	14°	1.22	.08	.70	8.28	1	7.7	Firm	Cloudy	
258	.54	Cent.	30°	0.13	.08	.00	0.35	1	1.35	17.3	Medium	Sl. Cloudy	
258	.54	Cent.	30°	0.20	.08	.05	0.55	1	1.4	16.5	Medium	Sl. Cloudy	
258	.54	Cent.	30°	0.28	.08	.10	0.75	1	2.1	13.1	Firm	Clear	
258	.54	Cent.	30°	0.44	.08	.20	1.20	1	2.9	11.0	Firm	Clear	Theory X 5
258	.54	Cent.	30°	0.59	.08	.30	1.65	1	3.7	Firm	Clear	
258	.54	Cent.	30°	0.75	.08	.40	2.00	1	4.5	9.3	Medium	Clear	
258	.54	Cent.	30°	1.06	.08	.60	2.90	1	5.7	9.0	Medium	Clear	
258	.54	Cent.	12°	0.16	.08	.03	0.16	1	1.6	11.7	Soft	Turbid	
258	.54	Cent.	12°	0.20	.08	.05	0.20	1	2.1	10.4	Slippery	Clear	Oil contained some water
258	.54	Cent.	30°	0.59	.08	.30	1.65	1	4.0	Firm	Sl. Cloudy	Theory X 5
258	.54	Cent.	30°	0.59	.08	.30	1.65	1	3.6	Firm	Clear	
258	.54	Cent.	30°	0.59	.08	.30	1.65	1	3.6	Firm	Clear	
265	0.27	AOCS	14°	7/8	.04	.48	5.38	1	6.8	9.6	Soft	Turbid	Foots slid into oil after 10 min. drain
265	0.27	AOCS	14°	7/8	.04	.48	5.38	Slippery	Turbid	Foots slid into oil after 10 min. drain
265	0.27	AOCS	14°	7/8	.04	.48	5.38	Slippery	Turbid	
265	0.27	Cent.	30°	0.07	.04	.00	0.17	Soft	Sl. Cloudy	
265	0.27	Cent.	30°	0.12	.04	.03	0.30	1	1.5	12.7	Medium	Clear	
265	0.27	Cent.	30°	0.20	.04	.08	0.50	1	2.1	12.4	Firm	Clear	
265	0.27	Cent.	30°	0.32	.04	.15	0.80	1	2.8	10.8	Firm	Clear	
265	0.27	Cent.	30°	0.41	.04	.20	1.00	1	3.7	10.5	Firm	Clear	Theory X 5
265	0.27	Cent.	30°	0.54	.04	.32	1.50	1	5.6	8.7	Firm	Clear	Foots progressively softer
265	0.27	Cent.	30°	0.75	.04	.44	2.00	1	7.3	8.6	Firm	Clear	Foots progressively softer
265	0.27	Cent.	30°	0.95	.04	.56	2.50	1	8.3	8.6	Firm	Clear	Foots progressively softer
265	0.27	Cent.	30°	1.14	.04	.67	3.00	1	9.9	8.3	Firm	Clear	Foots progressively softer
265	0.27	Cent.	30°	1.34	.04	.79	3.50	1	11.0	Medium	Cloudy	Oil contained water
265	0.27	Cent.	30°	1.54	.04	.91	4.00	1	1.0	8.6	Soft	Cloudy	Foots firm after remelts
265	0.27	Cent.	12°	0.90	2	2.6	
265	0.27	Cent.	14°	7/8	.11	.49	6.26	1	5.6	Firm	Turbid	
S-868	0.74	AOCS	14°	7/8	.11	.49	6.26	1	4.3	Firm	Turbid	
S-868	0.74	AOCS	14°	7/8	.11	.49	6.26	1	4.3	Firm	Turbid	
S-868	0.74	Cent.	30°	0.45	.11	.20	1.30	1	3.6	9.0	Firm	Clear	Theory X 5
S-868	0.74	Cent.	30°	0.78	.11	.42	2.20	1	5.4	Firm	Clear	
S-868	0.74	Cent.	30°	0.82	.11	.45	2.35	1	5.6	Firm	Clear	
S-868	0.74	Cent.	30°	1.04	.11	.60	2.95	1	7.9	8.8	Firm	Clear	
S-869	0.27	AOCS	14°	7/8	.04	.48	5.42	Slippery	Could not complete detn.
S-869	0.27	AOCS	14°	7/8	.04	.48	5.42	Slippery	Could not complete detn.
S-869	0.27	Cent.	30°	0.32	.04	.15	0.80	1	2.5	Very firm	Clear	Theory X 5
S-869	0.27	Cent.	30°	0.75	.04	.40	1.95	1	4.3	8.5	Firm	Clear	
AOCS I	0.5	AOCS	14°	7/8	.07	.49	5.84	1	3.5	5.9	Firm	Turbid	Theory X 5
AOCS I	0.5	Cent.	30°	0.67	.07	.36	1.50	1	3.1	8.4	Firm	Clear	
AO-6	0.49	AOCS	14°	7/8	.07	.48	5.88	1	4.2	Green	Firm	Turbid	
AO-6	0.49	AOCS	14°	7/8	.07	.48	5.88	1	4.2	Green	Firm	Turbid	
AO-6	0.49	Cent.	30°	0.56	.07	.28	1.45	1	3.9	Green	Firm	Clear	Theory X 5
AO-6	0.49	Cent.	30°	0.87	.07	.48	2.30	1	4.7	Green	Firm	Clear	
AO-7	0.27	AOCS	14°	7/8	.04	.48	5.48	2	5.9	9.0	Slippery	Cloudy	
AO-7	0.27	AOCS	14°	7/8	.04	.48	5.48	2	5.9	9.0	Slippery	Cloudy	
AO-7	0.27	Cent.	30°	0.37	.04	.15	0.80	1	4.7	8.8	Firm	Sl. Cloudy	Theory X 5
AO-7	0.27	Cent.	30°	0.75	.04	.40	1.85	1	2.5	9.1	Firm	Clear	
AO-9	0.50	AOCS	14°	7/8	.07	.49	5.96	1	4.3	8.5	Firm	Cloudy	Theory X 5
AO-9	0.50	AOCS	14°	7/8	.07	.49	5.96	1	4.3	8.5	Firm	Cloudy	
AO-9	0.50	Cent.	30°	0.56	.07	.29	1.50	1	3.9	10.2	Slippery	Clear	Foots floating
AO-9	0.50	Cent.	30°	0.73	.07	.40	2.00	1	4.7	10.2	Firm	Sl. Cloudy	Theory X 5
AO-11	0.37	AOCS	14°	7/8	.05	.48	5.68	1	3.3	7.0	Firm	Cloudy	Foots light green
AO-11	0.37	AOCS	14°	7/8	.05	.48	5.68	1	3.3	6.4	Firm	Sl. Cloudy	Foots light green
AO-11	0.37	Cent.	30°	0.43	.05	.21	1.10	1	3.3	5.7	Firm	Clear	Foots and Oil dark green
AO-11	0.37	Cent.	30°	0.74	.05	.40	1.90	1	3.4	6.4	Firm	Clear	Foots and Oil dark green
AO-12	0.60	AOCS	14°	7/8	.09	.49	6.12	1	6.4	8.7	Slippery	Sl. Cloudy	Foots soft after remelt
AO-12	0.60	AOCS	14°	7/8	.09	.49	6.12	1	6.4	8.7	Slippery	Sl. Cloudy	Foots soft after remelt
AO-12	0.60	Cent.	30°	0.65	.09	.36	4.68	1	4.7	8.8	Soft	Sl. Cloudy	Foots soft after remelt
AO-12	0.60	Cent.	30°	0.74	.09	.34	1.80	1	4.7	9.2	Firm	Clear	Theory X 5
AO-12	0.60	Cent.	30°	0.74	.09	.34	1.80	1	4.9	9.1	Firm	Clear	
268	0.5	AOCS	14°	7/8	.07	.48	5.74	1	2.1	8.4	Firm	Turbid	Water in remelt oil
268	0.5	AOCS	14°	7/8	.07	.48	5.74	1	2.1	8.4	Firm	Turbid	Water in remelt oil
268	0.5	Cent.	30°	0.45	.07	.28	1.45	1	2.7	8.8	Loose grains	Sl. Cloudy	Theory X 5
AO-13	0.59	AOCS	14°	7/8	.08	.49	6.04	1	3.9	6.0	Firm	Cloudy	Foots floating
AO-13	0.59	AOCS	14°	7/8	.08	.49	6.04	2	3.9	6.1	Firm	Cloudy	Theory X 5
AO-13	0.59	Cent.	30°	0.63	.08	.33	4.60	1	3.9	6.9	Firm	Clear	
AO-13	0.59	Cent.	30°	0.74	.08	.40	2.00	1	3.8	7.3	Firm	Clear	

SUMMARY OF COOPERATIVE REFINING TEST RESULTS ON PEANUT OIL SAMPLES

Refined March 15, 1943

Loss		No. of Results That Vary from Average by				Color—Red			No. of Results That Vary from Average by										
Method	Average	High	Low	0.1%	0.2%	0.3%	0.4%	0.5%	More Than	Method	Average	High	Low	0.1%	0.2%	0.3%	0.4%	0.5%	More Than
TEXAS OIL																			
1-a	7.05	8.70	5.30	3	1	1	2	1	4	1-a	2.1	2.3	1.9	6	2
1-b	6.4	8.6	5.2	2	1	...	3	1	6	1-b	2.0	2.3	1.5	5	2	1
1-c	7.4	8.3	5.9	4	2	2	3	...	3	1-c	1.9	2.2	1.4	5	2	1
2-a	6.7	7.8	5.6	1	1	4	1	...	6	2-a	2.0	2.4	1.5	6	1	1	1	1	...
2-b	6.5	8.6	4.9	...	2	...	1	4	6	2-b	2.1	2.3	1.9	6	3
2-c	7.3	8.2	6.2	4	1	2	2	...	4	2-c	1.9	2.1	1.4	7	1	1
3-a	5.8	8.2	4.0	3	1	1	9	3-a	2.1	2.3	1.9	4	4
3-b	6.2	8.8	5.0	3	5	...	2	...	5	3-b	2.0	2.3	1.5	3	2	3
3-c	6.7	9.3	5.8	3	...	4	1	...	5	3-c	1.9	2.1	1.4	5	1
4-a	8.1	8.8	4.8	1	4	4-a	1.9	2.2	1.6	2	...	2
4-b	6.8	8.2	5.5	...	1	2	4-b	1.9	1.9	1.8	2
4-c	7.1	7.6	6.8	1	...	2	...	1	...	4-c	1.7	1.9	1.4	1	1	1
GEORGIA OIL																			
1-a	9.7	11.2	9.0	1	...	4	1	1	8	1-a	7.1	7.4	6.8	2	4	3
1-b	9.5	10.6	8.9	4	7	1	1	...	3	1-b	6.9	7.5	6.4	3	...	2	1	2	...
1-c	10.9	12.7	9.3	4	...	1	2	2	7	1-c	6.8	7.2	6.2	3	4	1
2-a	9.6	11.0	8.5	3	3	1	1	2	4	2-a	7.0	7.3	6.8	4	4	1
2-b	9.4	10.5	9.0	8	...	3	3	...	1	2-b	7.0	7.3	6.6	2	4	1	2
2-c	11.0	12.1	10.0	4	1	1	2	2	5	2-c	6.8	7.2	6.2	3	2	2	2
3-a	9.7	12.2	8.6	8	...	1	7	3-a	7.1	7.4	6.8	3	3	3
3-b	9.6	10.4	9.3	7	5	2	1	3-b	6.8	7.3	6.3	3	1	1	1	2	...
3-c	11.4	8.6	10.1	2	...	2	2	1	9	3-c	6.7	7.0	6.0	2	...	6	1
4-a	9.4	9.8	8.8	4	2	2	1	1	1	4-a	7.0	7.3	6.6	3	3	1	1
4-b	9.7	10.6	9.6	4	3	1	1	1	2	4-b	6.9	7.3	6.6	3	1	2
4-c	11.2	13.0	10.0	2	1	...	3	...	5	4-c	6.9	7.0	6.6	5	...	1

Method 1—Regular Method, 90-minute setting period.
Method 2—Regular Method, overnight setting period.

Method 3—Slow Breaking Method, 90-minute setting period.
Method 4—Slow Breaking Method, overnight setting period.

Georgia Oil:

- a = 60% Maximum 16° Beaume
- b = 80% Maximum 16° Beaume
- c = 80% Maximum 20° Beaume

Texas Oil:

- a = 60% Maximum 12° Beaume
- b = 60% Maximum 16° Beaume
- c = 80% Maximum 16° Beaume

COMMENTS

Mr. King of the Interstate Cotton Oil Company at Sherman, Texas, has reported that peanut oil from certain sections of Texas has not refined well with the regular peanut oil refining method. The same information has since been confirmed by other people. It was suggested that this oil did not "break" satisfactorily in the regular test and that a "slow-breaking" procedure might be better. This work was undertaken in an effort to see if refining results could be improved by using the cottonseed oil "slow-breaking" method.

In general, the soapstock from the Texas oil was soft and did not settle well. The soapstock from the Georgia oil was firm and did settle well.

There was insufficient sample of the Texas oil to complete the tests that had been outlined. In the case of the Georgia oil, there is no indication that the slow-breaking method shows any advantage. In the case of the Texas oil, the issue is incomplete and the test should be repeated.

and 265, the effect of varying amounts of alkali was studied. On oil 258, the excess NaOH over theory per 100 grams of oil was varied from .35 to .70 gram for the A.O.C.S. method, and from 0.0 to .60 gram excess for the centrifuge method. On oil 265 the excess NaOH ranged from 0.0 to .91 gram on the centrifuge method. For both oils a rather consistent picture was obtained of the refining loss increasing with increasing alkali used, and this seems generally true for all the oils examined here. This is also supported by the 1941 results presented in Series I, referred to above. It appears that factor II, the formation of soaps by excess alkali, comprises the greater part of the foots when the centrifuge method is used, since, in general, the refining loss for a given oil bears more relation to the excess of alkali used than to the F.F.A. of the oil. This receives further support from the experiment on the 265 refined oil, where, when .90 gram of alkali was used in a "re-refining," a loss of 2.6 per cent was obtained, whereas a loss of 2.5 per cent was found using .80 gram alkali on the original oil containing .27 per cent F.F.A.

The use of five times theory is incorrect in that, for oils with a high F.F.A., too much alkali will be used, and in oils with very low F.F.A., too little alkali to give good foots may be used. A more logical choice would be to use alkali equivalent to theory plus .1 or .2 gram. It may also be possible to use the centrifuge method successfully with weaker alkali, and thus further reduce saponification. Inspection of column C in the table shows the large amounts of water used in the A.O.C.S. refining as compared with the centrifuge method. By centrifuging there will be more tendency for this water to separate and caution must be exercised to prevent the occurrence of free water in the foots-oil mixture. Alkali of 12° Bé was used for oil 265, and the results obtained with an equal amount of 30° Bé caustic shows the lower loss with the weaker alkali. On oil 258, a similar comparison was unsuccessful because of the unsatisfactory foots obtained with the weaker alkali.

Further work should be carried out on the following points:

- I. Crude undried extracted oils and their effect on the A.O.C.S. method.
- II. Limits of amount and strength of alkali in the centrifuge method.
- III. Use of the centrifuge method on all types of oil.

Our second refining problem was referred to us by Mr. R. R. King of the Interstate Cotton Oil Refining Company, Sherman, Texas. Mr. King, in his letter of January 19, 1943, called attention to the fact that

peanut oil from certain sections of the country was very difficult to refine by the official peanut oil method. He further suggested that the cottonseed oil "slow breaking" procedure was better adapted to this kind of oil.

Accordingly, two samples of peanut oil were sent out. One of these, marked "Georgia" was a normal oil which could be satisfactorily refined by the regular method. The other sample marked "Texas" did not respond to the usual treatment. The results of the tests of the committee are recorded in the attached tabulation and summarized at the end. These results clearly indicate that the "Texas" type of peanut oil does not refine satisfactorily by the official method. However, more work is necessary in the development of a procedure which will effectively handle this type of oil. The "slow breaking" method did not appear to have any particular advantage in the case of the Georgia sample.

Recommendations for Next Year's Refining Committee Activities

(1) It is possible that a refining loss basis of settlement for soybean oil will be established at the start of the next oil season. It is extremely important, in view of this, that we continue to have the assistance of the Northern Regional Research Laboratory. It is the hope of the committee that this will be possible.

(2) Further study should be made of the centrifuge method as outlined by Dr. Milner:

- (a) Limits of amount and strength of alkali in the centrifuge method.
- (b) Use of centrifuge method on all types of oil.

(3) Study of crude undried extracted oils and their effect on the A.O.C.S. method.

(4) Further study of the present A.O.C.S. refining test for peanut oil:

- (a) Application of slow breaking procedure to oils from Texas and Oklahoma.
- (b) Effect of longer settling time with regular method in the case of oils giving unsatisfactory foots with the regular procedure.

H. S. MITCHELL, *Chairman*

R. T. MILNER, *Vice-Chairman*

E. R. BARROW	LAMAR KISHLAR
C. B. CLUFF	N. F. KRUSE
G. A. CRAPPLE	T. C. LAW
M. M. DURKEE	H. E. MOORE
R. H. FASH	L. A. SPIELMAN
E. B. FREYER	B. L. STERNBERG
A. R. GUDHEIM	W. L. TAYLOR
ARTHUR KIESS	

Interim Report of the Refining Committee (A. O. C. S.)

As stated in the report of this Committee presented at the Annual Meeting, May 12-14, 1943, recent work has been on two different methods of refining soybean oil. As a result of a Committee meeting in Chicago on April 30, 1943, the Northern Regional Research Laboratory was requested to compare these two analytical methods (the present official and tentative

A. O. C. S. cup methods and the proposed centrifugal method) on representative samples of all types of soybean oil to be furnished by Committee members. In addition, three sub-committees were established to study modifications of the present A. O. C. S. methods: Mr. S. O. Sorenson was assigned extracted oils; Mr. J. H. Sanders, expeller oils; and Dr. E. G. Freyer,