

Report of the Refining Committee 1939-40

The 1939-40 activities of the Refining Committee were confined to further study of a method applicable to extracted soybean oil. For the purpose of the record, this year's program is covered in some detail in this report. A summary, with recommendations, is given at the end.

Before sending out cooperative samples, a preliminary study of the tentative method for extracted oil suggested by the Committee in the final report of the 1937-38 program was made in Swift & Company's Laboratory. It was found that the presence of impurities consisting primarily of gums, resins, lecithin, etc., exert a marked influence on the refining procedure. Tests with varying strengths and amounts of lyes indicated that 14° Be' lye gave the best results in a comparison with 10°, 12°, 16° and 18° Be' strengths, in that there was less tendency for water separation with this strength than with 10° and 12° Be' lyes and a lower loss than with the 16° and 18° Be' lyes. It was found necessary to use $\frac{3}{4}$, $\frac{1}{2}$ and $\frac{1}{4}$ maximum strengths to obtain one or more satisfactory settles. Using the 14° Be' lye, the times of cold-fast and hot-slow agitation were varied in an attempt to find the best method of handling. The observations made indicated that the time of cold-fast agitation might be appreciably reduced. It was also indicated that the time of hot-slow agitation could be shortened. As a result of this work the following method suggested itself as most satisfactory for the several types of extracted oil examined:

Refining Method Used on Cooperative Samples No. 1 and No. 2

Sodium hydroxide solution strength of lye shall be 14° Be' for extracted soybean oil. Three tests shall be made on each oil, using the $\frac{3}{4}$, $\frac{1}{2}$, and $\frac{1}{4}$ maximum amounts of sodium hydroxide with the maximum amount of lye calculated using the

$$\text{formula } \frac{\text{F.F.A.}}{5.2} + .54.$$

Extracted soybean oil shall be agitated at 20-24°C. for 45 minutes from the time the sodium hydroxide solution is added with the agitator running 250 ± 10 R.P.M. It shall then be immediately transferred to the 65°C. bath and stirred at 70 ± 5 R.P.M. for exactly 8 minutes. Temperature of oil must then be 60-65°C., adjusting the temperature of the water bath, if necessary, within the limits specified to obtain this final oil temperature.

At the end of the slow agitation period of refining, the extracted soybean oil shall be allowed to settle in the 65°C. bath for one hour. Cool by settling in a cold water bath at 12-15°C. for one hour. The oil shall then be allowed to set overnight before pouring off. After settling overnight the oil should again be chilled in a bath at 12-15°C. for 30 minutes before pouring off, unless the temperature has been maintained within this range.

The soap stock must be hardened after each remelting by chilling in water at a temperature of 12-15°C. or lower to permit satisfactory draining of the oil.

The result of cooperative samples No. 1 and No. 2, refined by the Committee following the method outlined above were not in satisfactory agreement.

As a result of the wide variation in the results on the first two cooperative samples, further work was done in the Swift Laboratory following the suggestions which came from various members of the Committee. It appeared that apparently the longer periods of agitation were necessary if satisfactory foots were to be obtained with extracted oil. It was decided, therefore, to revert back to the tentative method of the previous year, and cooperative samples No. 3 and No. 4 were refined following the procedure given below:

Tentative Refining Method as Suggested by Refining Committee in Their Report Covering the 1938-39 Program

Sodium hydroxide solution strength of lye shall be 12° and 14° Be' for extracted soybean oil. Four tests shall be made on each oil, using the $\frac{2}{3}$ and $\frac{1}{2}$ Max. 12, and $\frac{7}{8}$ and $\frac{2}{3}$ Max. 14° amounts of sodium hydroxide with the maximum amount of

$$\text{lye calculated using the formula } \frac{\text{F.F.A.}}{5.2} + .54.$$

Extracted soybean oil shall be agitated at 20-24°C. for 90 minutes from the time the sodium hydroxide solution is added with the agitator running 250 ± 10 R.P.M. It shall then be immediately transferred to the 65°C. ± 2°C. bath and stirred at 70 ± 5 R.P.M. for exactly 20 minutes. Temperature of oil must then be 60-65°C. adjusting the temperature of the water bath, if necessary, within the limits specified to obtain this final oil temperature.

At the end of the slow agitation period of refining, the extracted soybean oil shall be allowed to settle in the 65°C. bath for one hour. Cool by settling in a cold water bath at 12-15°C. for one hour. The oil shall then be allowed to set overnight before pouring off. After settling overnight the oil should again be chilled in a bath at 12-15°C. for 30 minutes before pouring off, unless the temperature has been maintained within this range.

The soap stock must be hardened after each remelting by chilling in water at a temperature of 12-15°C. or lower to permit satisfactory draining of the oil.

Results on sample No. 3 were not in satisfactory agreement but those on sample No. 4 were fairly good. It was evident that while the suggested method might be satisfactory for some oils it gave poor results with others, especially those containing little or no lecithin.

When the losses reported on the third and fourth samples indicated the need for further study, we again began in Swift's Laboratory, to make changes in the procedure in an effort to find a set of conditions which would lead to hard foots. During this work an error in the preparation of the caustic lyes led to the incorporation of small quantities of sodium silicate. These mixtures gave losses considerably lower than we had been getting. The use of silicate is, of course, not new in the refining of oil. It was thought advisable, however,

that we should investigate this lead, with particular respect to the Laboratory refining test for extracted soybean oil. Mixtures of $\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$ and NaOH were, therefore, made up containing 0.68%, 1.36%, 2.7%, 4.1% and 6.8% of the silicate and these mixtures were used in quantities of $\frac{2}{3}$ maximum, $\frac{1}{2}$ maximum and $\frac{1}{3}$ maximum, using 12° , 14° , and 16° lyes. Approximately 5% of Na_2SiO_3 with Be' lye was selected as most promising, and a mixture was prepared having the following analysis:

Total Alkalinity as NaOH	9.68%
Na_2SiO_3	4.66%

This mixture of $\text{NaOH-Na}_2\text{SiO}_3$ was sent to the Committee for use in the refining tests on cooperative samples No. 5 and No. 6. The losses on the low acid oil used as sample No. 5 are in fairly good agreement. The findings on the higher acid oil, sample No. 6, were not quite as satisfactory.

Summary and Recommendations

The cooperative results from the refinings made by Committee on extracted oil, samples No. 1 and No. 2, following the present tentative method except that the cold-fast agitation period was 45 minutes and the hot-slow period was 8 minutes, were unsatisfactory. The percentages of losses falling within steps of 0.1% up to 0.5% and that over 0.5% follow:

Refining Loss	Sample No. 1			Sample No. 2		
	$\frac{3}{4}\cdot 14^\circ$	$\frac{1}{2}\cdot 14^\circ$	$\frac{1}{4}\cdot 14^\circ$	$\frac{3}{4}\cdot 14^\circ$	$\frac{1}{2}\cdot 14^\circ$	$\frac{1}{4}\cdot 14^\circ$
Results on Average:	5.88%	None	None	6.67%	12.50%	
Results Within 0.1% of Avg.:	11.76	13.34	6.25%	6.67	18.75	Results Too Variant to Tabulate
Results Within 0.2% of Avg.:	11.76	6.67	12.50	6.67	25.00	
Results Within 0.3% of Avg.:	11.76	20.01	12.50	6.67	6.25	
Results Within 0.4% of Avg.:	None	6.67	12.50	13.34	6.25	
Results Within 0.5% of Avg.:	5.88	None	6.25	None	None	
Results Which Were More than 0.5% of Avg.:	52.96	53.31	50.00	59.98	31.25	

Samples No. 3 and No. 4 were refined by the present tentative method. The percentages of losses falling within steps of 0.1% up to 0.5% and that over 0.5% are given below. It will be noted that there was wide variation in results from the different collaborators.

A caustic-silicate mixture was prepared and sent out to the Committee for use in refining samples No. 5 and No. 6. The 14° Be' mixture used analyzed as follows:

Total Alkalinity as NaOH	9.68%
Na_2SiO_3	4.66%

The percentages of losses falling within steps of 0.1% up to 0.5% and that over 0.5% are given below. It will be noted that the findings on these two samples as reported by the Committee were the most encouraging of the year.

Refining Loss	Sample No. 5		Sample No. 6	
	$\frac{3}{4}\cdot 14^\circ$	$\frac{1}{2}\cdot 14^\circ$	$\frac{3}{4}\cdot 14^\circ$	$\frac{1}{2}\cdot 14^\circ$
Results on Average:	21.42%	33.30%	5.88%	17.65%
Results Within 0.1% of Avg.:	37.16	20.01	17.64	17.65
Results Within 0.2% of Avg.:	7.14	6.67	17.64	17.65
Results Within 0.3% of Avg.:	7.14	20.01	17.64	11.76
Results Within 0.4% of Avg.:	7.14	13.34	5.88	11.76
Results Within 0.5% of Avg.:	None	6.67	None	5.88
Results Which Were More than 0.5% of Avg.:	None	None	35.32	17.65

Recommendations:

The Committee recommends that the tentative method for extracted soybean oil be given further study next year. The program should include the consideration of a suitable combination of alkalies, as well as method of application.

If it is possible to do so, the Committee recommends that the Soybean Regional Laboratory take a very active part in the program next year, collaborating with the Refining Committee in the investigational work necessary for the development of methods to be used in the cooperative study.

NOTE.—Detailed tabulations of the data obtained by the Committee are omitted from publication since no really satisfactory method has been arrived at.

Refining Loss	Sample No. 3				Sample No. 4			
	$\frac{3}{4}\cdot 12^\circ$	$\frac{1}{2}\cdot 12^\circ$	$\frac{1}{4}\cdot 14^\circ$	$\frac{3}{4}\cdot 14^\circ$	$\frac{3}{4}\cdot 12^\circ$	$\frac{1}{2}\cdot 12^\circ$	$\frac{1}{4}\cdot 14^\circ$	$\frac{3}{4}\cdot 14^\circ$
Results on Avg.:	6.67%	7.14%	None	None	12.50%	12.50%	20.01%	None
Results Within 0.1% of Avg.:	6.67	7.14	6.25%	12.50%	18.75	25.00	59.98	18.75%
Results Within 0.2% of Avg.:	13.34	7.14	12.50	6.25	25.00	12.50	13.34	43.75
Results Within 0.3% of Avg.:	6.67	14.28	12.50	12.50	31.25	25.00	None	6.25
Results Within 0.4% of Avg.:	13.34	21.42	12.50	18.75	None	None	None	6.25
Results Within 0.5% of Avg.:	None	14.28	6.25	12.50	None	12.50	None	6.25
Results Which Were More Than 0.5% of Average:	53.31	28.60	50.00	37.50	12.50	12.50	6.67	18.75

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