REFINING METHOD FOR CRUDE COTTONSEED OIL

The following is a progress report of work done for the Refining Committee, in the laboratory of the Procter & Gamble Company, since the last convention. The program undertaken consists of three parts.

First, a detailed study of each step in the operation, to determine the effect of variations in same.

Second, on the basis of the data from No. 1, a revised method to be written up involving all the optimum conditions and eliminating all chance for personal difference in procedure.

Third, a comparison of results obtainable with this revised method, in the hands of different operators, as compared with results by the present official method.

The first portion is sufficiently far advanced to present results at this time, and correspondence and suggestions are invited from all interested parties.

Two lots of well settled oil were set aside for this work, one containing approximately 4 per cent F.F.A. and the other about 20 per cent The former will be referred to here as the "Low F.F.A. Oil" and the latter as the "High F.F.A. Oil." The procedure followed was substantially that of the present official method, varying only one condition at a time. In order to insure constant speed, the usual friction type drive was replaced with gears, and in order to maintain a standard rate of heating in all tests this was accomplished by transferring the dish after mixing in cold water bath directly into a hot water bath maintained at 60° to 65°C. In eight minutes, under these conditions, the low F.F.A. oil reached a temperature about 5° below the temperature of the bath and the high F.F.A. oil about 10° below the bath temperature. The greater amount of lye in the latter case made the heating somewhat slower.

The principal points studied so far, with results obtained, are as follows: all figures shown being checked by two or more tests; color readings over 50 RED were made on a short column and corrected to approximate a 5¼" column.

Paddles

Various sizes and shapes of paddles were first tried on various oils, noting color and loss obtained, and especially the time required to cause a visible mixing movement on the surface of the mixture in case of high F.F.A. oils. Some average results are shown below:

Size and Form of Paddle	Average Results on Four Oils F. F. A.	Color	Loss	Time to Mix
½" Straight Paddle	15.3%	65 R	44.0%	10 minutes
½" Twisted Paddle	15.3	71	44.1	12+ "
1" Straight Paddle	15.3	61	42.2	1 minute
1" Twisted Paddle	15.3	61	43.2	7 "

The 1 inch paddle, especially on high F.F.A. oils, appeared so much

superior to the present ½ inch paddle, especially in time required for mixing, that it was adopted for all subsequent work. The straight form is definitely superior to the twisted form.

The effect of varying the distance of the paddle above the bottom of the dish, up to a 1 inch clearance, was found to be negligible.

Refining Vessel

A comparison was made of a wide and shallow dish vs. a deep and narrow dish, as it was felt that on bad oils the large amount of foots formed might settle more completely and could be drained more thoroughly if in a shallow layer instead of a deep layer. No difference in results, however, could be found.

Rate of Agitation Cold

	Low F. F. A. Oil		High F. F. A. Oil	
R. P. M.	Color	Loss	Color	Loss
100	9.5 R	11.4%	—	—
150	8.9	11.4	255 R	52.3%
200	8.9	11.7	220	54.2
250	8.9	11.7	210	50.5

On the basis of the above results, a speed of 250 R.P.M. was adopted. Higher speeds could not be used safely with the 1 inch paddle.

Time for Agitating Cold

	Low F. F. A. Oil			High F. F. A. Oil	
Time	Color	Loss	Time	Color	Loss
3 minutes	9.7 R	10.0%	3 Min.	230 R	51.2%
12 "	9.4	12.3	10 "	220	52.1
21 "	9.3	12.8	16 "	180	55.5
30 "	9.3	13.9	20 "	180	54.9
			30 "	180	58.0

It is obvious that the loss increased steadily with the time of agitating. Color does not improve proportionately on these oils after about 10 to 15 minutes. Consequently, 10 minutes was adopted for subsequent work.

Temperature of Agitating Cold

	Low F. F. A. Oil			High F. F. A. Oil	
Temp.	Color	Loss	Temp.	Color	Loss
10°-12°C	9.3 R	12.2%	10°C	210 R	50.0%
17°-19°C	9.3	12.0	18°-20°C	225	52.0
26°-28°C	9.4	12.2	28°-30°C	210	57.4
36°-38°C	9.6	11.8	36°-38°C	220	73.1

The best results on color and loss seem to be obtained at the lower temperatures, the difference being especially marked on high F.F.A. oil. 20°C. was considered to be the best and most practical temperature for use.

Rate of Agitation Hot

R. P. M.	Low F. F. A. Oil Color	Loss	High F. F. A. Oil Color	Loss
40	9.1 R	11.8%	—	—
50	9.1 R	11.7	180 R	52.0
75	9.2	11.8	180	51.9
100	9.9	12.7	180	53.4
125	10.5	12.6	180	54.2

On the basis of these results 70 R.P.M. was adopted for agitating oil while heating. Higher speeds are distinctly injurious.

Temperature of Agitation Hot

Bath temperatures of 54°, 64° and 74°C. were used with eight minutes allowed for heating in each case. Practically no difference was found in the results on the low oil, but on the high oil a slightly better recovery of oil was made from the first settling, before remelting the foots, and also slightly better color was obtained with the 64°C. bath.

Rate of Heating

Time to reach 40°C	55°C	Low F. F. A. Oil Color	Loss	Time to reach 40°C	55°C	High F. F. A. Oil Color	Loss
2.6 Min	5.2 Min.	9.5 R	12.35%	2.3 Min.	4.2	240 R	54.8%
10.0 "	13.4 "	10.0 R	15.0 %	7.5 "	11.4	260 R	58.1%

It is very clear that the best results are obtained by heating as rapidly as possible. Slow or irregular heating by different operators will cause serious variations in results.

Temperature of Settling

No differences were found on the low F.F.A. oil when settled at temperatures between 40° and 75°C. Likewise, on the high F.F.A. oil, no appreciable difference in the ultimate loss or color was found, but more oil is recovered on the first settling, when the temperature is about 70°. 65° will probably be a safe and desirable temperature for settling.

Time of Settling Hot (60° —65°C).

Time	Low F. F. A. Oil Color	Loss	High F. F. A. Oil Color	Loss
½ hour	10.8 R	14.0%	190 R	57.4%
1 "	10.8	14.0	190	57.1
2 "	10.6	14.0	200	56.1
4 "	10.4	14.1	190	52.6
8 "	10.2	13.9	210	54.6
16 "	10.0	13.7	230	51.1

Note that on the low oil, the color improves but on the high oil it darkens on long settling. The loss shows an improvement on the high oil. The results obtained in the four-hour test are probably out of line. Two hours settling are probably sufficient for practical purposes.

Temperature of Cooling Foots

Rapid preliminary cooling on ice with subsequent standing at room temperature gave the same final results as when the preliminary ice cooling was omitted.

Angle of Dish During Draining of Oil

It was found that on the high F.F.A. oil a solid foots could not be obtained. Hence the angle was limited to that at which the foots would not flow out of the dish. On the low F.F.A. oil no appreciable difference, due to different angles, was noted.

Time of Draining Oil from Foots

Low F. F. A. Oil		High F. F. A. Oil		
Time	Loss	Time	Loss	
15 min.	13.60%	15 min.	60.53%	(Foots not remelted,
30 "	13.52%	30 "	60.43%	" " "
60 "	13.47%	60 "	60.31%	" " "

Practically nothing is gained after 30 minutes. A difference of .1 per cent on a 60 per cent loss is negligible.

Air Bubbles in Crude Oil

The effect of air bubbles introduced when shaking the sample was studied, by comparing results on samples containing the maximum possible amount of air bubbles with samples perfectly clear. No difference in results could be found.

Remelting of Foots

It was found on the low oil, that even one remelting was seldom necessary, but on high oil at least three remeltings were necessary in all cases.

Caustic Soda in Different Beaumé's

Refinings were made on each oil with lyes of different Beaumé but equal NaOH, using the amount specified as a maximum in the present rule.

Low F. F. A. Oil

F. F. A. = 4.4%

Lye %	Bé	Color	Loss
14.2	14°	11.8R	13.4%
10.6	18	10.6	13.6
9.3	20	10.5	14.3
8.4	22	10.2	14.8
7.5	24	10.0	14.9

High F. F. A. Oil

F. F. A. = 20.5%

Lye %	Bé	Color	Loss	Foots
38.0	16°	290	58.2	Very soft
29.0	20	230	52.3	Fairly soft

%	Bé	Color	Loss	Foots
23.5	24	260	52.7	Semi soft
19.3	28	Over 500	57.5	Granular
Second Series, using more Lye				
40.5	16	270	58.6	
31.0	20	220	54.7	
25.0	24	210	55.4	
20.6	28	385	66.4	

On both oils it appears that the weakest lye gives darker color, and on the high oil higher loss also. 20° seems to be best on the high oil and 18° to 20° on the low oil. Stronger lyes increase the loss without a compensating improvement in color.

Effect of Different Percentages of 20° Lye

Low F. F. A. Oil				High F. F. A. Oil			
Lye				Lye			
%	Bé	Color	Loss	%	Bé	Color	Loss
5.3	20°	21.7 R	32.2%	25	20°	390 R	48.9%
7.3	20°	12.6	12.0	27	20°	340	48.7
9.3	20°	10.6	14.5	29	20°	250	50.3
11.3	20°	10.1	17.3	31	20°	190	54.2
13.3	20°	9.4	19.8	33	20°	180	61.1

On the lower F.F.A. oil 9.3 per cent of 20° lye gives the best results. This is the amount specified as allowable maximum in our present rule. On the high F.F.A. oil 29 per cent of 20° is the maximum in the present rule, but 31 per cent gives a much lighter color with some increase in loss, but not enough in our opinion to offset the improvement in color. Apparently, from these tests and others not here recorded, the present table does not allow sufficient lye for best results on the higher F.F.A. oils.

Impurities in Lye

Studies are now under way of the effect of different impurities such as sodium carbonate, iron, aluminum, calcium and silica.

Summary

The principal points in the present method, which cause differences in results when conditions are varied, and which, therefore, are the probable causes of differences reported by different chemists are the following. These points must be rigidly standardized in any revision of the method if we are to obtain more concordant results for settlement purposes:

1. Agitation must be improved. The 1 inch straight paddle appears to be the best yet tried.

2. The rate of agitation in the cold must be closely controlled. A gear driven machine should be used at 250 R.P.M.

3. The rate of agitation hot must be likewise closely controlled by a gear driven machine at 70 R.P.M.

4. The time of agitation in the cold with the 1 inch paddle should be 10 minutes.

5. The temperature of agitation cold should be rigidly kept between proper limits. 18° to 22°C. appears most suitable.

6. Maximum temperature 60° to 65°C.

7. Rate of heating should be the quickest possible. 8 minutes in a water bath maintained at 65°C. appears the best and safest method. (Iron vessels without enamel were used in this work.)

8. Temperatures of settling hot: 65°C.

9. Time of settling hot: 2 hours.

10. Remelting foots. Remelt several times if necessary, until recovery is less than .5 per cent.

11. Choice of lyes must be rigidly limited.

12. Purity of lye. Effect to be determined later.

Future Work

As soon as the few remaining points now under investigation are completed, a revised method will be written up and comparative tests made on a number of crude oils, to determine two points: First, a comparison between the new and old method by different operators; and secondly, whether more consistent results are obtainable on the new method by different operators than on the old method, when working on the same oils.

It is probable that the apparatus used in different laboratories will also have to be standardized, not only as to size and shape of paddle but also size, shape and metal of refining vessel and the type of agitating device. The last must be so designed as to give always the same speed, without slipping. Two speeds—250 R.P.M. and 70 R.P.M. seem to meet all requirements.

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