Refining Method for Crude Cottonseed Oil

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THE work on this subject which was published in the November issue of this journal has been continued with results as shown below:

Time of Settling at Room Temperature before Decanting Oil

	Low Oil		High Oil	
Time	Color Red	Loss Per Cent	Color Red	Loss Per Cent
3 hours	10.2	14.3	21.0	53.0
16 hours	9.8	14.1	23.0	52.0

Apparently only slight change in color or loss occurs after three hours.

Additional Settling of Refined Oil at Room Temperature after Decanting

	Color Red
Filtered immediately after decanting	10.5
Filtered 12 hours after decanting	10.2

Confirms previous observations that a slight improvement in color usually occurs after prolonged settling.

Time of Reheating Foots from High F. F. A. Oil in Hot Bath at 65°C

	Oil Recovered	
Time Heating	1st Heating Per Cent	2nd Heating Per Cent
20 minutes	2.5	.4
60 minutes	2.9	.7
Increased Recovery	.4	.3

Apparently 20 minutes heating of large masses of foots is not quite sufficient.

Stirring Foots During Remelting

	Oil Recovered (High F.F.A. Oil)
Not stirred	2.5%
Stirred	None

Shows necessity for remelting *without stirring.*

Effect of Rusty Refining Cups

Refinings were made in extremely rusty cups and in clean cups for comparison. Low F. F. A. Oil was used.

Cups	Color Red	Loss Per Cent
Clean	10.3	14.4
Rusty	10.3	14.9

Rust has no effect on color but loss apparently increases slightly with the abnormal amount of rust on these cups, which was far greater than would ever be present in ordinary usage. Any ordinary amount of rust would evidently have no measurable effect. We conclude from this that the use of enameled cups to avoid rust is not essential.

Comparative Rates of Heating in Enameled vs. Plain Iron Cups of Identical Dimensions in Bath at 64° C.

	Time and Temperatures				
	0	2	4	6	8 Mins.
Plain Iron.	21°	42°	53°	58°	60°C.
Enameled .	22°	41°	51°	57°	59½°C.

Rate of heating, and final temperature attained in eight minutes are practically identical, showing that enamel does not noticeably retard the transfer of heat under these conditions.

Impurities in Lye (NaOH content unchanged)

(A) Carbonate of Sodium

No2CO3 as % of NaOH used Per Cent	Low Oil Color Red	Low Oil Loss Per Cent	High Oil Color Red	High Oil Loss Per Cent
0	9.8	14.7	275	51.4
5	10.0	14.8	265	50.6
10	10.2	14.8	265	49.8
20	10.2	14.6	270	49.5
30	10.3	14.7	285	48.5
40	10.5	14.7	285	48.7

(*B*) *Iron added as Ferric Hydroxide, freshly precipitated.*

Fe as % of NaOH Used	Color Red	Loss Per Cent
0 per cent.....	10.2	14.7
2 per cent.....	10.5	14.1
4 per cent.....	11.6	14.3
8 per cent.....	11.7	14.1

(*C*) *Aluminum Added as Hydroxide, freshly precipitated.*

Al as % of NaOH used	Color Red	Loss Per Cent
0 per cent.....	10.0	14.8
2 per cent.....	10.1	16.8
4 per cent.....	10.5	15.8
8 per cent.....	10.7	15.2

Note: Aluminum added in this form neutralizes a portion of the NaOH.

(*D*) *Aluminum added as Sodium Aluminate, powder.*

Al as % of NaOH used	Color Red	Loss Per Cent
0 per cent.....	10.2	14.4
2 per cent.....	9.7	18.3
4 per cent.....	9.4	20.2

Note: Aluminum added in this form does not neutralize any of the NaOH used for refining.

(*E*) *Calcium added as Hydroxide, freshly precipitated.*

Ca as % of NaOH used	Color Red	Loss Per Cent
0 per cent.....	10.2	14.7
2 per cent.....	10.1	14.7
4 per cent.....	9.9	14.5
8 per cent.....	9.9	14.8

(*F*) *Silicate of Sodium added as solution at 40° Beaumé. Note that percentage here is per cent Silicate solution on crude oil used.*

Silicate Sol. as % of Oil used Per Cent	Low Oil Color Red	Low Oil Loss Per Cent	High Oil Color Red	High Oil Loss Per Cent
0	10.2	14.4	260	51.2
2	15.5	10.5	270	48.7
4	19.1	10.2	380	48.0

The various impurities studies as described above were those which may occur in caustic soda solutions, due either to the chemicals used in making sodium hydroxide, or to impurities dissolved when the solution stands in glass bottles.

It appears that all these impurities have a bad effect on either color or loss except Calcium Hydroxide which has very little effect on either. The most serious impurities are Aluminum and Silica in the forms of Sodium Aluminate and Sodium Silicate respectively; the latter especially is apt to be present in solutions which have stood in glass bottles for a long time.

The necessity of using only the purest possible solutions of Sodium Hydroxide should be clearly evident to every chemist from these results, both from the standpoint of obtaining the best possible color and loss, and to avoid discrepancies among different operators.

Summary (continued from November issue, page 381).

12. Sodium Hydroxide solutions must be free from all impurities.
13. Time of settling at room temperature, 3 hours.
14. Time required to remelt Foots in water bath at 60-65°C, 30 minutes.
15. Foots must not be stirred while remelting.
16. Enameled refining cups have no advantage over plain iron, but are not in any way objectionable.

Conclusion

Acknowledgment is again given to Mr. Nelson W. Humbaugh who carried out the analytical work here recorded.

A proposed revision of the refining method as at present written which is intended to embody all the points found desirable, is now in the hands of the members of the Refining Committee for consideration and test.

WRITES ABOUT TUNG OIL

J. Francis Cooper, of the Florida Experiment Station, Gainesville, Fla., has written an interesting article in the November issue of *Better Crops*, which he has entitled "Tung Oil."