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# Plant Scale Comparisons of Various Refining Methods for Cottonseed Oil

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Three different refining processes were commercially compared by processing 15,148 metric tons of cottonseed with free fatty acid content varying between 7.1% and 8.9%. All of the seed was prepressed and solvent extracted in the Sanbra plant at Bauru, Brazil. The Ranchers Miscella refining process operating on seed averaging 8.8% F.F.A. yielded more oil of lighter color per ton of seed processed than either of the other processes compared, even though the average F.F.A. of the seed processed during the Ranchers Miscella Refining test averaged 1.7% higher than the seed used in the Sanbra process and 1.1% higher than the average F.F.A. for the seed used in the Low Loss Refining test.

In another comparison, screw pressed oil, Modified Soda Ash refined was compared to Ranchers Miscella refining with seed containing about 0.5% F.F.A. The results showed 42% lower refining loss and a color of 3.5 Red Lovibond units less for Ranchers Miscella refined oil than for Modified Soda Ash refined oil.

The average cost of converting crude cottonseed oil to prime bleachable summer yellow oil by the miscella refining process described is 20.8¢ per hundred weight of oil (not including refining loss). These costs include the prorated cost of control laboratory, plant labor and supervision, fuel, power, chemicals, depreciation, taxes and insurance.

OF THE commercially significant edible vegetable oils processed, one of the most difficult to refine with respect to color removal is cottonseed oil (12). Vegetable oil refiners, processing cottonseed oil or similar highly pigmented oils by conventional methods, continuously compromise between high refining loss resulting from the use of strong caustic on the one hand and highly colored oils which meet with consumer resistance on the other. Re-refining and bleaching may improve the color of the product but these processes add to the production cost, decrease the yield of finished product and contribute to the instability of the finished product (1,8).

In April 1956 a paper was presented before the A.O.C.S. describing a new integrated miscella refining process for edible oils (2). E. M. James favorably reported on miscella refining in April 1957 in a paper comparing various refining methods (10). An acetone miscella refining process was described in the March 1961 J.A.O.C.S. (15). The advantages of lighter color and greater yields of excellent quality finished oil per ton of source material are real and demonstrable when oil is immediately miscella refined subsequent to solve extraction with the exclusion of air and light (2).

This report presents plant refining data comparing four different refining processes operating on cottonseed oil of moderately high and very low free fatty acid content. The refining methods compared are Ranchers Miscella, Low Loss, Modified Soda Ash, and Sanbra. (Sanbra is the Brazilian affiliate of Bunge.)

## Description of Refining Processes

Ranchers Miscella Refining Process actually starts with the cooking of the meats and consists of the following steps: 1) Conditioning meats to contain 10–12% moisture at cooker discharge; 2) adding granular soda ash to the cooked meats to control the F.F.A. of the crude oil within desired limits (3); 3) batching crude 50% miscella in make-up tanks. (Two tanks make continuous operation possible.) 4) Continuously adding 8° to 20° Bé caustic soda through a rotometer into the suction side of the single crude miscella pump; 5) intimately contracting the dilute caustic with the crude miscella in an homogenizer (4); 6) heating the miscella to cause the soapstock to melt; 7) cooling the miscella to form a two-phase system for centrifugal separation (5); 8) separation of refined miscella and soapstock in vapor tight tubular bowl centrifuges. 9) Soapstock containing approximately 15% by weight of hexane may be desolventized in commercially available equipment and subsequently processed as ordinary caustic soapstock, or if the F.F.A. of the crude oil does not exceed 3% and economic conditions warrant, it may be added to the solvent-wet meal from the extractor with decidedly beneficial results to the quality of the meal (9). 10) The refined miscella may be contacted with a soap removing acid wash (6) or, in some instances, filtering through diatomaceous earth in a totally enclosed filer is preferred to water washing. Oils miscella refined according to the above procedure are very light in color. If bleaching is desired, bleaching earth can be substituted for the diatomaceous earth in the filter press and colors as light as required can be obtained with virtually no loss of oil in the filter clay. 11) If winterization is desired, this can be very effectively done in solvent at this stage of processing. Continuous separation of the stearine in valve operated disk type centrifuge gives about 87% yield of 20 hr. cold test cottonseed salad oil and 13% of 73 Iodine Value stearine. 12) Solvent is recovered in conventional equipment with the decided advantage that the distillation equipment operating on refined miscella never has to be cleaned. Ranchers Miscella Refinery and winterizer are preferably located in the solvent extraction plant so that the same operator(s) control all phases of the solvent extraction, refining, winterizing, solvent recovery, and deodorization processes.

The Low Loss process is a two stage refining process usually followed by double water washing to remove soap. The crude oil is first conditioned by admixture with 0.1% of citric or orthophosphoric acid. In the neutralization stage the conditioned oil is heated to 150°F and then a stoichiometric amount of caustic soda is added to the oil and the resultant soapstock

centrifugally separated. A color removal step using a small amount of strong caustic soda follows the initial soapstock removal step. This refining method is extensively used in South America and parts of Europe.

Modified Soda Ash process is similar to the Low Loss process, the main differences being that the acid conditioning step is eliminated and a 250% excess of 20° Bé soda ash is used in the initial neutralization stage instead of a stoichiometric amount of caustic soda. Briefly described, the Modified Soda Ash process consists of the following steps: 1) A continuous stream of crude oil, heated to approximately 140°F., is admixed with about 2.5 times the theoretical amount of 20° Bé soda ash required to neutralize the free fatty acid in the oil, and this mixture is further heated to about 200°F. 2) The soapstock and coagulated gums are centrifugally separated. On oils of 2% or higher F.F.A. it is common practice to introduce a degassing tank for release of carbon dioxide between the heater and the primary centrifugals. 3) The partially refined dark oil is cooled to about 100°F and mixed continuously with a small amount of 20°–40° Bé caustic soda solution, the oil-caustic mix is agitated, heated to 160–180°F, and then centrifugally separated. 4) Finally the oil is washed once or twice with water, vacuum dried, and sent to storage. The details of this process are well known and are adequately described in the literature (10, 13, 14, 1, 7, 11).

The Sanbra process is an experimental process which was used in South America as a basis for comparison between Low Loss and Ranchers Miscella Refining processes.

The Sanbra process is described as a double neutralization process in which the crude miscella is first degummed with a stoichiometric amount of caustic soda-soda ash solution at room temperature, followed by centrifugation. In the second step the miscella is violently agitated with a small amount of concentrated caustic soda solution, heated to 40°C, and centrifuged.

The Sanbra process and the Ranchers Miscella Refining process were both carried out in the same refinery at Bauru, Brazil. The caustic soda miscella mixture was not agitated in an homogenizer in the Sanbra process.

Essentially the Sanbra process is the Low Loss process carried out in approximately 50% concentration miscella.

## Experimental

Moderately high free fatty acid seed were processed and refined in the Sanbra prepress solvent extraction plant in Bauru, by the Low Loss, Sanbra, and Ranchers Miscella Refining processes. This plant has a capacity of approximately 250 metric tons of cottonseed per 24 hr. The miscella refinery has a capacity of 150,000 pounds of refined oil per 24 hr. day. Table I shows the weight and analysis of the seed processed during the various trial runs. It is apparent from these data that the seed processed during the Ranchers Miscella Refining test was 4% higher in moisture and over 1% higher in F.F.A. than the seed processed by the other two refining methods.

Table II compares the oil yields and refining data of the three refining methods in lb. per short ton. The total oil per ton of seed was calculated from the analysis in Table I. The yield of crude oil per ton was calculated from the weight and analysis of residual oil in the meal and hulls. The refined oil was weighed and refining loss was determined from

TABLE I
Seed Analysis

| Refining method | Metric tons of seed | Moisture | F.F.A. | Hexane soluble (oil) |
|---|---|---|---|---|
| | | (%) | (%) | (%) |
| Low Loss No. 1 | 5130.834 | 9.7 | 8.0 | 15.84 |
| Low Loss No. 2 | 2419.355 | 10.5 | 7.4 | 15.75 |
| Low Loss No. 3 | 2521.781 | 10.2 | 7.4 | 15.56 |
| | 10071.970 | 10.0 | 7.7 | 15.75 |
| Sanbra | 858.426 | 10.2 | 7.1 | 15.53 |
| Ranchers No. 1 | 1706.363 | 11.0 | 8.7 | 16.29 |
| Ranchers No. 2 | 2511.652 | 16.7 | 8.9 | 15.58 |
| | 4218.015 | 14.4 | 8.8 | 15.87 |

the calculated weight of crude oil and the actual weight of refined oil. Because the soda ash added to the cooked meats in the Ranchers Miscella Refining Method neutralized 1.34% of the F.F.A. in the crude oil, the refining loss factor in all cases was determined by dividing the refining loss by the F.F.A. in the seed.

Using the refining loss factors from Table II and assuming 8% F.F.A. and a yield of 300 lb. of crude oil per ton of seed, then the Low Loss refining process

TABLE II
Oil Yields and Refining Data

| | Yield | | | | |
|---|---|---|---|---|---|
| | Total oil/ ton seed | Crude oil/ ton seed | Refined oil/ ton seed | Refining loss | Ref. loss factor |
| | (lb.) | (lb.) | (lb.) | (%) | |
| Low Loss No. 1 | 316.8 | 309.6 | 256.8 | 17.05 | 2.13 |
| Low Loss No. 2 | 315.0 | 309.2 | 262.2 | 15.20 | 2.05 |
| Low Loss No. 3 | 311.2 | 296.2 | 252.4 | 14.79 | 2.00 |
| | 315.0 | 306.1 | 257.0 | 16.04 | 2.08 |
| Sanbra | 310.6 | 304.0 | 261.8 | 13.88 | 1.95 |
| Ranchers No. 1 | 325.8 | 320.4 | 271.8 | 15.17 | 1.74 |
| Ranchers No. 2 | 311.6 | 305.8 | 259.4 | 15.17 | 1.70 |
| | 317.3 | 311.7 | 264.4 | 15.17 | 1.72 |

would yield 250.0 lb. of refined oil per ton of seed; the Sanbra process would yield 253.2 lb. and Ranchers Miscella Process would yield 258.7 lb.

Table III compares the once refined oil color, bleached color, and soap in refined oil. The average Lovibond red color was 12.6 Low Loss, 12.8 Sanbra, and 13.5 Ranchers Miscella. Soap in refined oil: Low Loss 3388 P.P.M., Sanbra 1710 P.P.M., and Ranchers 388 P.P.M. In a soap removing step, bleaching earth at 0.61% of the weight of the oil gave 6.5 Red color for Low Loss; 0.02% of the weight of the oil gave 9.2 Red for Sanbra; and 0.30% of the weight of the oil gave 5.9 Red for Ranchers Miscella.

In a second test, Modified Soda Ash refining was compared to Ranchers Miscella refining on seed of

TABLE III
Oil Color and Soap Data

| | Lovibond color ref. oil | Soap in refined oil | Lovibond color oil (bleached) | Bleaching earth used |
|---|---|---|---|---|
| | (Red) | (ppm.) | (Red) | (% wt. oil) |
| Low Loss No. 1 | 12.4 | 3225 | 5.3 | 0.45 |
| Low Loss No. 2 | 11.7 | 2580 | 7.4 | 0.44 |
| Low Loss No. 3 | 13.7 | 4495 | 8.2 | 1.09 |
| | 12.6 | 3388 | 6.5 | 0.61 |
| Sanbra | 12.8 | 1710 | 9.2 | 0.02 |
| Ranchers No. 1 | 13.5 | 600 | 5.5 | 0.25 |
| Ranchers No. 2 | 13.6 | 244 | 6.2 | 0.34 |
| | 13.5 | 388 | 5.9 | 0.30 |

very low free fatty acid content grown within a 150 mi. radius of Fresno, California. The Modified Soda Ash refining was done on screw pressed oil. The Ranchers Miscella refining was done on prepressed solvent extracted crude. Although the seed was grown in the same area and the analysis in Table IV shows them to be quite similar, the difference in processing methods imparts a variable not present in the comparisons shown in Table II and III where all the seed was processed in the same extraction plant.

It would have been desirable to compare the Modified Soda Ash process with the Ranchers Miscella process on seed with F.F.A. content similar to that encountered in the Sanbra plant in Bauru, Brazil. It will be obvious to the reader, however, that during commercial plant comparisons involving over 15,000 metric tons of seed in South America and about 23,000 short tons of seed in Fresno, California, very little control can be exercised over the quality and grade of the source material.

TABLE IV
Seed Analysis

| | Moisture | F.F.A. | Hexane soluble (oil) |
|---|---|---|---|
| | (%) | (%) | (%) |
| Modified Soda Ash | 8.3 | 0.55 | 18.61 |
| Ranchers Miscella | 9.7 | 0.47 | 18.42 |

Table V shows the total amount of oil in the seed; the amount of crude oil per ton of seed was calculated from the weight and analysis of meal and hulls produced. The refined oil per ton of seed was the actual weighed amount. In the case of the Modified Soda Ash refining process, the 324.2 lb. yield is in lb. of 7.8 Red bleachable prime summer yellow oil per ton of seed. In the Ranchers Miscella process, yield is in lb. of winterized and deodorized salad oil of 1.2 Red, and deodorized stearine with an average of 1.4 Red. The A.O.M. stability of the salad oil averages 19–20 hr. without the use of anti-oxidants. The 347.0 lb. yield of finished oil includes deodorization loss. The actual refining loss with the Ranchers Miscella process was 42.2% less than the Modified Soda Ash refining method under the conditions of this comparison. The refining loss factors of both processes are relatively high, due to the low F.F.A. content of the oil in the seed and moderately high gum content of the oil.

TABLE V
Oil Yields and Refining Data

| | Total oil/ton seed | Crude oil/ton seed | Refined oil/ton seed | Refining loss | Ref. Loss factor |
|---|---|---|---|---|---|
| | (lb.) | (lb.) | (lb.) | (%) | |
| Modified Soda Ash | 374.2 | 337.6 | 324.2 | 3.96 | 7.2 |
| Ranchers Miscella | 368.4 | 355.1 | 347.0* | 2.29 | 4.9 |

* Includes deodorization loss.

Assuming a 350 lb. yield of crude oil for both processes and 0.50% F.F.A. content in the seed, and assuming all other processing conditions to be the same, then the yield of PBSY per ton of seed for the Modified Soda Ash process would be (Refining Loss = .50% F.F.A. × 7.2 = 3.60%) 337.4 lb.

Using the same formula, the yield of finished winterized and deodorized salad oil and deodorized stearine per ton of seed for the Ranchers Miscella process would be (R.L. = 0.50% F.F.A. × 4.9 = 2.45%) 341.4 lb. Of this total, 293.6 lb. would be salad oil, and 47.8 lb. would be deodorized stearine.

TABLE VI
Oil Color and Soap Data

| | Lovibond color ref. oil | Soap in refined oil * | F.F.A. refined oil | AOCS bleach color |
|---|---|---|---|---|
| | (Red) | (ppm.) | (%) | (Red) |
| Modified Soda Ash | 7.8 | 6.0 | 0.06 | 2.1 |
| Ranchers Miscella | 4.3 | 2.0 | 0.02 | 1.2 |

* By Brom Phenol Blue Titration Method.

Table VII compares the Gas Liquid Chromato graphic analysis of screw pressed crude oil, Modified Soda Ash refined oil, and the total fatty acids in the soda ash soapstock with the prepressed-solvent extracted crude oil, miscella refined oil, and total fatty acids in the miscella caustic soapstock. A six ft. column of Diethylene Glycol Succinate was used for all analysis, temperature of the column 215°C, Helium Gas was used as carrier at flow rate of 60 ml. per minute. The triglycerides were methylated with sodium methoxide as a catalyst.

The data presented in Table VII indicate that crude and refined oils of quite similar fatty acid composition are produced by the two different extraction and refining methods from seed of similar analysis. An appreciable difference in the linoleic and oleic acid in the T.F.A. of the caustic soapstocks is observed between the two refining methods. This difference is significant and is presented to encourage further investigation in this area.

TABLE VII
GLC Fatty Acid Composition of Oil and Soapstock

| | Myristic | Palmitic | Stearic | Oleic | Linoleic |
|---|---|---|---|---|---|
| | (%) | (%) | (%) | (%) | (%) |
| Screw-pressed crude oil | 1.2 | 28.0 | 1.9 | 17.5 | 50.6 |
| Screw-pressed M.S.A. refined | 1.1 | 26.1 | 1.5 | 17.1 | 54.1 |
| T.F.A. from M.S.A. soap | 0.6 | 25.8 | 1.9 | 19.6 | 52.2 |
| T.F.A. from caustic soap | 1.0 | 29.6 | 2.3 | 26.8 | 39.5 |
| Prepressed srude oil | 1.0 | 26.2 | 1.6 | 16.9 | 54.3 |
| Miscella refined oil | 1.0 | 26.4 | 1.8 | 17.1 | 53.3 |
| T.F.A. from miscella refined soap | 0.8 | 28.1 | 2.1 | 21.5 | 47.6 |

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