Commercial Extraction of Vegetable Oils by Means of Trichlorethylene

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FOR a number of years chlorinated solvents have been used extensively in the fields of metal degreasing and dry-cleaning. Trichlorethylene, one of the principal chlorinated hydrocarbons used in metal degreasing, has been shown to be well suited for the extraction of vegetable oils from oil seeds. Its value as a vegetable oil solvent has been recognized in some of the European countries for several years, and is now being used to a considerable extent in this country. In 1944 the Detrex Corporation installed their first commercial trichlorethylene soybean plant, which has been in continuous operation since installation. Research and development since 1944 have resulted in many improvements and refinements in the process, some of which were incorporated in an installation completed late in 1947.

Trichlorethylene is a powerful fats and oil solvent and is noninflammable. The latter property is very desirable from the standpoint of safety. Also the elimination of safety switches, spark proof motors, special fire fighting equipment and other safety control measures makes for a great saving in the cost of extraction equipment. Trichlorethylene has been unfairly criticized by some as being too good a solvent for extraction purposes, claiming that dark colored oils result from its use. Our experience has shown that this criticism is without foundation because prime oils are being produced continuously in the Detrex trichlorethylene extraction plants. The information which will be presented in this paper will substantiate this statement.

The data presented in Table I shows the refining loss and color of a number of trichlorethylene extracted soybean oil samples taken from time to time during 1947. The oil from which these samples were taken was stripped at a still temperature of 225-240° F. The Expeller Cup method was employed for refining the samples.

The results given in Table I show an average refining loss of about 6.9% for the 9 samples reported with a range of 5.5 to 8.2%. The average color of the refined and bleached oil was 17 yellow + 1.7 red. The average free fatty acid content of the oil was 0.94%. From these results it may be concluded that soybean oil of satisfactory refining loss and color are produced by trichlorethylene extraction. The residual solvent left in the oil ranged between 0.005 and 0.01% as it came from the distillation equipment.

Work on the extraction of cottonseed by means of trichlorethylene was started in our pilot plant at Detroit in 1945. The pilot plant is a complete continuous extraction unit, equipped with a meats heater, a flaker, an extractor, dryers, a filter, and distillation equipment. Dehulled cottonseed meats were procured from Southern cottonseed mills and shipped to Detroit in cloth bags. The pilot plant handled between 30 and 40 pounds of flaked meats per hour. Over a period of more than one year approximately 12,000 pounds of meats were processed. A study was made of such factors as flake preparation, time and temperature of extraction, quality of oil and meal produced, fines and methods for the removal of fines from the miscella.

TABLE I

Refining Loss and Color of Trichlorethylene Extracted Soybean Oil

Sample No.	F.F.A. %	Refining Loss %	Color (Refined and Bleached)
1	1.1	8.2	21 Y + 2.2 R
2	0.9	7.0	21 Y + 2.2 R
3	0.9	7.6	19 Y + 1.9 R
4	1.1	7.7	20 Y + 2.0 R
5	1.0	7.5	19 Y + 1.9 R
6	0.9	6.7	15 Y + 1.5 R
7	0.8	5.7	13 Y + 1.3 R
8	0.8	5.5	14 Y + 1.4 R
9	0.9	6.3	14 Y + 1.4 R
Average	0.94	6.9	17 Y + 1.7 R

Throughout the experimental work no particular operational difficulties were encountered. The oil content of the meal was reduced to between 1.0% and 2.0%. Fines carried into the miscella ranged from 0.75 to 1.5% based on the weight of flakes fed. The miscella from the extractor was reddish brown in color. The crude oil from the still was dark in color, but it could be easily refined to an acceptable color. The amount of red color in the refined oil varied with the age and free fatty acid content of the meats and the stripping temperature used for the removal of the solvent. The bleaching characteristics of the oil was good. The dried meal from the unit was brownish yellow in color and possessed a pleasant odor. The residual solvent in the meal varied from 0.03 to 0.06%, which is far below the toxicity level for trichlorethylene as reported in the scientific journals. The gossypol content was reduced to less than 0.1%. Reports of feeding trials showed that the meal was essentially non-toxic.

Since we first began work on the extraction of cottonseed, it has been the opinion of the writer that the official expeller and hydraulic refining methods are not necessarily adapted to the refining of trichlorethylene extracted cottonseed oil. Therefore laboratory work was done in order to check this point. It soon became apparent that certain modifications of the official method gave both lower refining loss and lower color. As a result of our studies the following centrifugal method was adapted for our work and is being presented here as a suggestion for further work along this line.

A sample of the crude oil, usually 100 grams, was weighed into a 250-ml. centrifuge bottle; the sample was placed in a water bath at 64 to 66°C. and the oil was rapidly stirred with an electric laboratory stirrer. When the oil had reached a temperature of 65°C., which required approximately 7 minutes, the calculated amount of NaOH solution was added and the stirring and heating was continued for 12 to 15 minutes. The break occurred after one or two minutes and the soapstock rapidly settled when the stirrer

was stopped at the end of the stirring period. The sample was transferred to a batch type centrifuge and centrifuged at approximately 2000 R.P.M. for about 5 minutes. The bottle and contents were then weighed, the oil decanted, and the soapstock allowed to drain for about 30 minutes. The oil clinging to the walls of the bottle was wiped off with a clean cloth and the bottle and contents again weighed. The difference in weight represents weight of refined oil from which the refining loss was calculated. The decanted oil was filtered with suction on a small Hirsch type filter fitted with filter paper. A small amount of filter aid, such as Johns-Manville's Hyflo Supercel, was usually added to the filter to aid filtration and to assure a perfectly clean oil. The color of the oil was then read in a a Lovibond color comparator.

The amount of sodium hydroxide used in the refining procedure just described was approximately 80% of that required for hydraulic or hot pressed oil as specified in the official methods of analysis. The NaOH was made up to give a solution of 20 to 25% concentration. The values in Table II show how closely results can be checked by this procedure.

TABLE II

Refining Cottonseed Oil Samples by the Modified Procedure

	Trial No.	Refining Loss (%)	Color (Lovibond)
Sample A	1	7.37	35 yellow + 6.4 red
	2	7.21	35 yellow + 6.2 red
	3	7.45	35 yellow + 6.3 red
	4	7.24	35 yellow + 6.3 red
Sample B	1	6.97	35 yellow + 6.3 red
	2	6.93	35 yellow + 6.4 red
	3	7.00	35 yellow + 6.4 red
	4	7.00	35 yellow + 6.4 red

The results in Table II show that the determinations can be duplicated satisfactorily by the procedure.

Table III shows the results obtained by the modified procedure. Also a few comparisons are shown between this procedure and the Expeller Cup refining method. Most of the cottonseed meats on which the extractions were made came from the 1945 crop. Shipments were received in the fall, winter, and spring of the following storage period. In all cases the dehulled meats were held from one week to three months before processing was complete. For this reason the free fatty acids were high, resulting in higher refining losses than would be expected on fresh meats.

According to the results shown in Table III, the modified procedure gave considerably lower refining loss and less red color than that given by the official method. The refined and bleached color of the samples ranged from 10 yellow + 1.0 red to 23 yellow + 2.3 red with an average of 14 yellow + 1.4 red. The low color of the refined and bleached samples indicate that the bleaching characteristics of the oil is very good. These results substantiate the statement made earlier that a prime oil is produced by the extraction of cottonseed with trichlorethylene. By the use of vacuum distillation equipment oils of even lower color would be expected.

TABLE III

Refining Loss and Color of Trichlorethylene Extracted Cottonseed Oil by the Expeller Cup Method and by the Modified Procedure

Sample No.	Expeller Cup Method		Modified Method		F.F.A. %
	Refining Loss %	Red Color (With 35 Yellow)	Refining Loss %	Red Color (With 35 Yellow)	
1	9.3	5.8	7.1	5.5	2.6
2	8.4	6.3	6.7	5.6	2.4
3	8.8	6.5	6.7	5.8	2.5
4	9.9	6.3	8.3	5.8	2.6
5	8.8	7.3	7.2	5.8	2.3
6	10.0	7.4	7.4	5.2	2.5
7	9.2	7.9	7.0	5.2	2.3
8			6.6	5.5	2.8
9			6.7	5.6	2.7
10			10.2	3.5	3.4
11			10.0	3.7	3.4
12			11.0	5.5	3.4
13			10.3	6.0	3.4
14			11.2	8.0	3.6
15			11.7	7.0	3.6
16			10.5	7.4	3.5
17			10.8	7.0	3.4
18			10.9	6.0	3.4
19			10.9	7.3	3.5

The soapstock from the refining bottle was firm and separated easily from the oil. The consistency of the soapstock could be controlled as desired by the amount of water added with the sodium hydroxide solution. The resulting product as well as that from extracted soybean oil should lend itself to fatty acid recovery as readily as that of soapstocks from hydraulic or expeller oils. The modified procedure for refining cottonseed oil should be well adapted to a continuous commercial centrifugal refining process.

Considerable pilot plant work has also been done on the extraction of copra and flaxseed by means of trichlorethylene. From both of these products good quality prime oils were produced. In the case of copra only minor extraction difficulties were encountered. A commercial copra extraction plant is being installed at the present time. Due to the size and characteristics of the flaxseed some difficulty has been encountered in reducing the oil content to as low a value as desired. However, oil contents in the meal as low as 1.5% have been reached. The preparation of the seeds for extraction results in considerable fine material which would present some difficulties in large units. The work on flaxseed is in progress at the present time.