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The Extraction of Wheat Germs, Milkweed Seeds, and Cottonseed by Trichloroethylene

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THE use of trichloroethylene as a solvent for soybean oil has received considerable study and development (10). However little has been reported previously on its suitability as a solvent for other vegetable oils. Preliminary to more complete extraction studies, data for specific gravity-miscella composition curves and for comparative extraction rates of wheat germs, milkweed seeds, and cottonseed have been determined and are presented together with preliminary pilot plant extraction data. Since no evaluation of the nutritional value or toxic properties of the residual meals produced in this study has been made and since certain batches of soybean meal produced by trichloroethylene extraction have been reported to have toxic properties when fed to cattle, these meals are not recommended for feeding until their suitability for this purpose has been determined.

Wheat germs are a by-product of the milling of wheat flour. The oil is principally of interest because of its high content (about 0.5%) of tocopherols (vitamin E), and it is used largely for medicinal purposes. The germs used in this study were received in the form of flakes 0.004 to 0.005 in. thick with an average diameter of about ⅛ in. They had a bulk density of 20.4 lb. per cubic foot.

Milkweed seed oil was produced experimentally by Gerhardt (2) as a part of his research on the possibilities of the milkweed as a farm crop. Considerable attention has been paid to the possibility of utilizing the milkweed floss of which the oil would be a by-product. Over a million and a half pounds of the floss were collected during World War II for use as a kapok substitute (5). Hollowell (3) made several pilot plant runs of seed from this floss, using trichloroethylene as a solvent. He produced a good oil but left from 4.3 to 8.0% residual oil in the meal. Gerhardt (2) reports the oil content of the seeds as 21.2%. Neish and Burns (7) give an average oil content of 22% with some seed as high as 28%. The iodine number was found to vary from 106 to 142. Oil extracted in this laboratory by trichloroethylene had an iodine number of 117.2. The seeds used in the current work were old and dry, making steaming necessary prior to flaking. Their oil content was 25.6%.

Cottonseed meats have been used as a source of oil for many years, and until recently most of it has been produced by pressure methods. Several plants which are now either extracting the oil directly (8, 11) or after prepressing (6, 9) are using commercial hexane as a solvent. Experimental work has been done on extraction with trichloroethylene (1, 3). In the present studies both whole delinted seeds and the hulled seeds or meats were used. Both seeds and meats were steamed to give a moisture content of approximately 11% prior to flaking on a set of smooth rolls. The oil content of the whole seed was 23.4%, of the meats 34.2%. Both the whole seed and the meats were flaked to a thickness of 0.012 in.

TABLE I

Rate of Extraction of Wheat Germs, Milkweed Seeds, and Cottonseed Meats at 87°C. (188.6°F.) with Trichloroethylene (Soxhlet Extraction [a])

Time in Minutes	Residual Oil in Extracted Flakes, Percentage		
	Wheat Germs	Milkweed Seeds	Cottonseed Meats
0	13.90	28.19	37.78
15	0.24	4.17	4.94
30	0.17	2.25	1.95
50	0.15	1.46	1.05
Miscellaneous Data			
Moisture, Percentage	8.76	5.46	8.76
Flake Thickness, Inches	0.005	0.006	0.014

[a] Average syphoning time 2½ minutes.

Specific Gravity-Composition Relationships

One of the routine tests in the operation of solvent extraction plants is the determination of the oil content of the miscella as it comes from the extractor as well as during the concentration and during the desolventizing operations. This can be done by determining the specific gravity of the miscella by means of a hydrometer or Westphal balance if the specific gravity-concentration relations are known for the

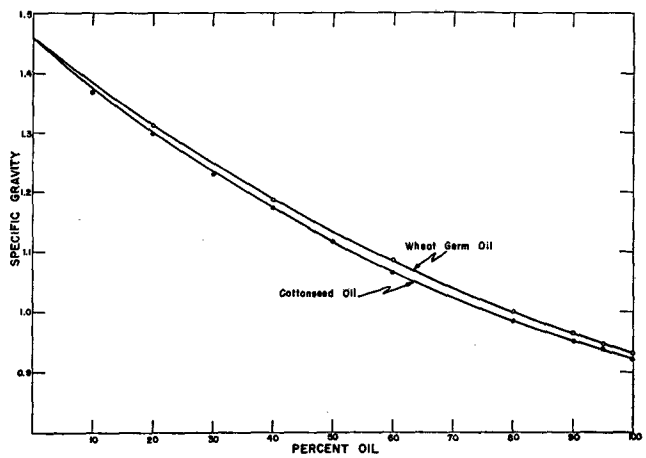

FIG. 1. Specific gravity-percent oil curves for wheat germ oil and cottonseed oil miscellas with trichloroethylene as a solvent at 25°C. (77°F.).

TABLE II

Rate of Extraction of Wheat Germs, Milkweed Seeds, and Cottonseed Meats at 24°C. (75.2°F.)

Time in Minutes	Residual Oil in Extracted Flakes, Percentage		
	Wheat Germs	Milkweed Seeds	Cottonseed Meats
0	13.90	28.19	37.78
4	2.22	10.95	15.19
8	1.95	8.92	9.84
12	1.62	7.74	7.92
16	1.53	7.10	7.00
20	1.41	6.64	6.38
24	1.34	6.24	5.88
28	1.30	5.62	5.45
32	1.19	5.62	5.07
40	0.99	5.03	4.75
50	0.87	4.55	4.40
60	0.77	3.91	4.05
70	.....	.....	3.72
Miscellaneous Data			
Average Thickness of Flakes, Inches	0.005	0.006	0.014
Weight of Sample, Grams	18.6	10.0	15.0
Moisture, Percentage	8.8	5.5	6.8
Solvent Rate, cc./min.	25.0	25.0	25.0

solvent-oil combination. To provide such information specific gravities of trichloroethylene-oil miscellas at 25°C./20°C. for wheat germ, milkweed seed, and cottonseed oils were determined by means of a Westphal balance, using crude oils extracted in the laboratory by trichloroethylene. Specific gravities of the oils were determined by using a specific gravity bottle. Specific gravity data for wheat germ and cottonseed oil miscellas are plotted against percentage of oil to give the curves in Figure 1. Curves for milkweed seed oil and for soybean oil fall on or between the curves shown.

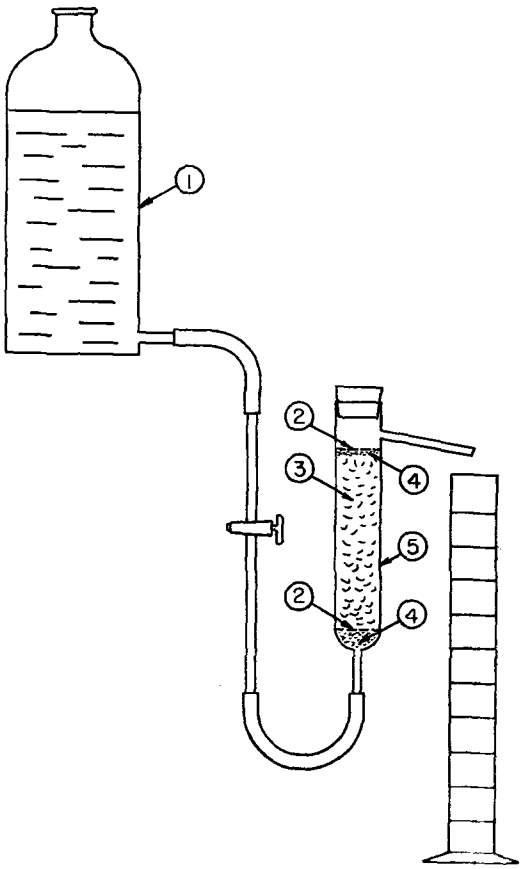

FIG. 2. Extraction rate apparatus: (1) solvent supply; (2) screen; (3) sample; (4) cotton; (5) extraction tube.

Extraction Rates

In the design of continuous solvent extraction plants for oil seeds the rate of extraction of the flaked, or otherwise prepared, seed by the solvent to be used is of primary importance in determining the extraction time necessary to reduce the amount of residual oil to a practical value. To obtain this necessary information the rates of extraction of wheat germ flakes, milkweed seed flakes, and cottonseed meat flakes were determined by two methods. Determinations at the boiling point of the solvent were made in Soxhlet extraction apparatus (Table I). Determinations at room temperature (24°C.) (Table II) were carried out in the apparatus shown in Figure 2. A sample of the material weighing from 10 to 20 g., depending on density, was placed in the tube, and solvent was passed through at a constant rate. The effluent from the side arm was collected and filtered at timed intervals, then the weight of oil was determined by evaporating the solvent until a constant weight of residue was obtained. At the end of the run the whole sample was extracted for two hours, then it was reground and extracted for another two hours to determine the residual oil.

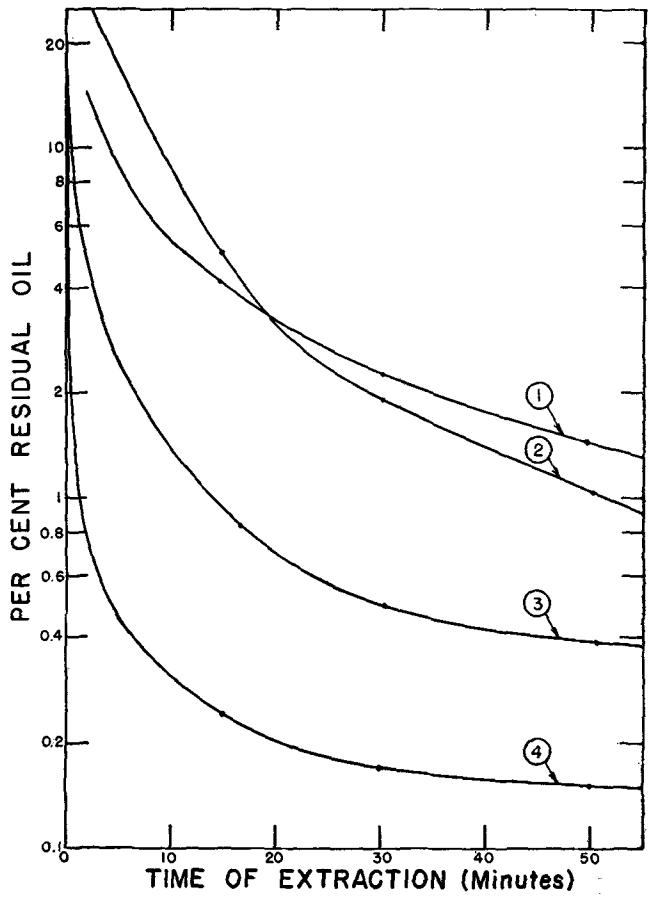

FIG. 3. Extraction rates in a Soxhlet extractor at 87°C. (188.6°F.): (1) milkweed seed flakes 0.006 inch thick; (2) cottonseed flakes 0.014 inch thick; (3) soybean flakes 0.009 inch thick; (4) wheat germ flakes 0.005 inch thick.

The extraction data for the three seeds together with similar data from flaked soybeans are shown graphically with the residual oil as a function of extraction time in Figures 3 and 4. Theoretically any

TABLE III

Pilot Plant Operation Data

Material Processed	Flakes Fed per Hour Lb.	Extraction Time in Minutes	Flake Thickness Inches	Miscella Concentration Percentage Oil	Percentage Oil		Solvent Flake Ratio [a]	Plant Capacity Referred to Soybeans [b]
					Flakes	Meal		
Wheat Germs	84	25.5	0.005	11.8	13.9	0.61	2.0	0.70
Wheat Germs	150	15.3	0.005	21.8	13.9	0.76	1.1	1.25
Milkweed Seeds	41	25.5	0.007	17.0	27.8	3.34	3.8	0.34
Milkweed Seeds	59	15.3	0.007	10.0	27.8	4.58	3.0	0.49
Cottonseed	56	25.5	0.012	23.0	23.0	1.4	2.9	0.51
Cottonseed Meats	90	25.5	0.012	14.5	34.5	2.7	2.7	0.75

[a] Ratio of the weight of solvent fed per hour to the weight of flaked material fed per hour.
[b] Capacity of pilot plant referred to the capacity for soybeans taken as 1.

comparison between the extraction rates of the four materials is invalid because of the differences in flake thicknesses. From a practical point of view this is not true since the thicknesses shown are practical ones suitable for the actual extraction of these materials. The relatively slow extraction rate of cottonseed as compared to that of soybeans, both with hexane as a solvent, has been shown by Karnofsky (4) and with trichloroethylene by Hollowell (3). Karnofsky (4) has pointed out that large diameter soybean flakes extract more readily than smaller ones of the same thickness, presumably because of the greater distortion occurring in their production. This may explain in part the slow rate of extraction of the milkweed seeds, which, being initially thin, suffered a relatively small amount of distortion during flaking. A considerable part of the difference in the extraction rates of soybeans and wheat germs is probably the result of difference in thickness.

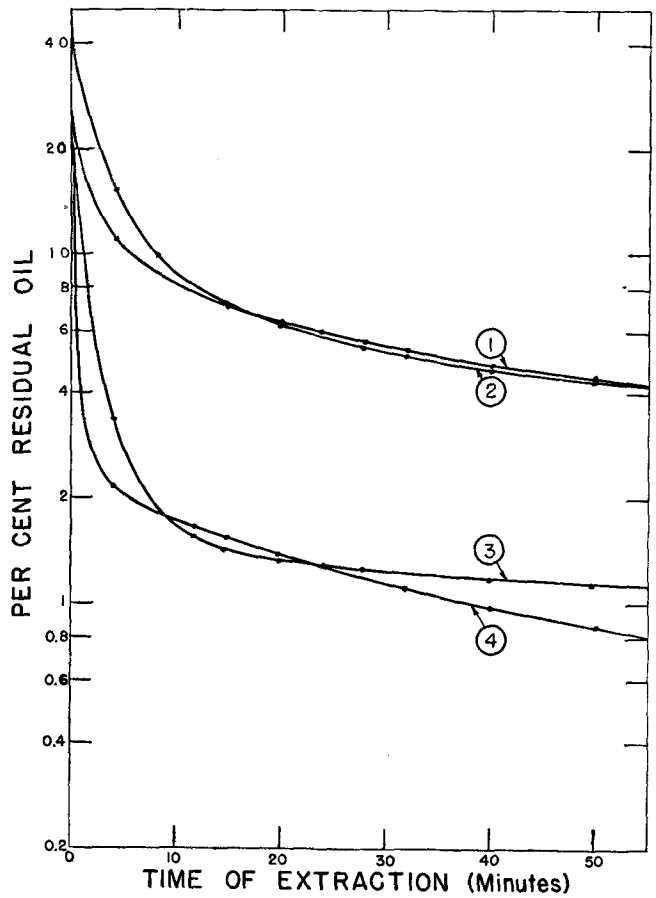

FIG. 4. Extraction rates at 24°C. (75.2°F.): (1) milkweed seed flakes 0.006 inch thick; (2) cottonseed flakes 0.014 inch thick; (3) soybean flakes 0.009 inch thick; (4) wheat germ flakes 0.005 inch thick.

Pilot Plant Extractions

Flaked wheat germs, milkweed seeds, cottonseed, and cottonseed meats were extracted with trichloroethylene in a continuous laboratory pilot plant, resulting in the data in Table III. The pilot plant extractor (10) consisted essentially of the lower part of a rectangular loop about 12 feet long and 6 feet high constructed of a steel casing having a cross-section of 4 by 5½ inches through which the flakes were moved slowly by a continuous Redler conveyor. Solvent was fed in at the end opposite the flake entrance to give countercurrent extraction. The rate at which the material being extracted passed through the extractor was a function of both the chain speed and the bulk density of the material. Extraction time was a direct function of chain speed. Solvent-flake ratio was the ratio of the weight of solvent fed to the weight of material fed per unit of time. Miscella concentration was a function of solvent-flake ratio, extraction time, temperature, and extraction characteristics of the individual flakes being processed. The plant capacity has been calculated in its relation to that for soybeans since commercial units similar to the pilot plant have been operated on soybeans with trichloroethylene as a solvent (10).

The extraction results obtained in the pilot plant (Table III) check in a general way with the rate extraction data. This indicates that the solvent extraction of wheat germs should be practical. The higher residual oil content of the cottonseed meats agrees with previous results. The results with milkweed seeds confirm earlier experience in this laboratory and show that they are difficult to extract. The results are preliminary in nature, but it is believed that they will be of value in that they show the possibilities of solvent extraction of these materials and may guide future experimental work. No evaluation has been made of either oil or meal quality.

Summary

Specific gravity-concentration data have been determined for wheat germ oil, milkweed seed oil, and cottonseed oil miscellas where trichloroethylene is used as a solvent. Extraction rate data at two temperatures and pilot plant runs on wheat germs, cottonseed, cottonseed meats, and milkweed seeds indicate increasing extraction time in the order given.

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Composition of Castor Oil by Optical Activity

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CASTOR oil occupies a unique position in the field of natural fats and oils. While, like other common oils, it is a glyceride, it is unusual in that the acid components of the glyceride are primarily hydroxy compounds. No other common natural fat or oil contains an appreciable quantity of combined hydroxy acids. This acid in castor oil has been named ricinoleic acid.

The exact amount of ricinoleic acid that occurs in the mixed acids from castor oil has been the subject of a number of analytical investigations. The methods used have been all chemical and involved the determination of acetyl or hydroxyl values as well as the use of the thiocyanate procedure. The values obtained for the percentage of ricinoleic acid by the various investigators are as follows:

Investigator	Date	Ricinoleic Acid
A. Eibner and E. Munzing (4)	1925	84.0%
P. Ponjutin and M. Rappoport (12)	1930	87.5
H. P. Kaufmann and H. Bornhardt (7)	1939	87.0
G. W. McBride (11)	1940	90.0
H. G. Kirschenbauer (8)	1944	87.8
H. K. Dean (3)	1946	87.0
R. L. Terrill (15)	1950	88.0
S. S. Gupta and T. P. Hilditch (5)	1951	93.1
J. P. Riley (13)	1951	92.6

It may be noted there has been a trend towards higher values in later years. This could be explained by the composition of castor oil tending towards higher ricinoleic acid content, but a more likely explanation is simply experimental error or refinement of analytical procedures. As far as is known, no attempt had previously been made to determine the percentage of ricinoleic by physical means, such as optical activity, refractive index, specific gravity, etc.

The formula for ricinoleic acid is: $CH_3(CH_2)_5CHOHCH_2CH:CH(CH_2)_7COOH$. It may be noted ricinoleic acid is identical with the more common vegetable fatty acid, oleic acid, except that it has a hydroxyl group on the 12th carbon atom. This atom is therefore asymmetric and should give the molecule optical activity since the oil is also a natural product. Actually castor oil and its ricinoleate derivatives have a pronounced ability to rotate a plane of polarized light. The observed specific rotation should therefore be adaptable for use as a measure of the ricinoleic content. It should also be highly specific since no other common fatty acid has an asymmetric carbon atom and thus optical activity. Although most natural vegetable oils contain unsymmetrical triglycerides and thus an asymmetric carbon atom in the glyceryl radical, no optical activity has been noted due to this structure (2).

The nonricinoleate components of castor oil are linoleic, oleic, stearic, and some dihydroxystearic. The actual amounts reported by different investigators varies considerably. The relative amounts should be further investigated by the newer analytical techniques. Actually dihydroxystearic acid has two asymmetric carbon atoms and could be optically active. Apparently the two sources of optical activity are nearly internally compensating, and the optical activity is slight. Since only small amounts of this acid are present, their effect on total optical activity has been disregarded in this paper. Thus the mixed fatty acids of castor oil are considered to be ricinoleic acid and nonricinoleic acid. This paper is concerned only with their relative proportion.

The optical activity of castor oil and its derivatives has been recognized for some time. In 1905 Lythgoe (9) measured the optical rotation of 44 samples of castor oil at 20° C. He reported that they varied in specific optical rotation from +8° to +9°. Markley (10), in his book on "Fatty Acids" published in 1947, reported the specific optical rotation of ricinoleic acid to be +6.7°. Hawke (6) in 1949 reported +7.79° and Brown and Green (1), +6.25°. It is suspected all these values are high and their variation due to some lactone or estolide formation. Brown and Green (1) in an excellent article on the "Preparation of Pure Methyl Ricinoleate," reported the optical rotation with the sodium line at 20° to be +5.19° for the pure methyl ester. There was apparently no attempt to determine the ricinoleic acid content of castor oil or its derivatives by this means.

Measurement of Optical Activity

The optical rotation of castor oil and its various derivatives described below was measured with a Rudolph Precision Polarimeter, Model 70, using the standard technique. This polarimeter is stated to have an accuracy of 0.01°. It was used with a 200 cm. tube and mercury arc lamp. Thus the data given are in terms of the 5461 Å mercury line as recommended by the Bureau of Standards instead of the usual sodium line. All readings were made at 25°C or corrected to 25°C. The correction was made by observing the change in specific rotation with temperature from which the necessary correction factor was readily calculated. The total rotation observed was calculated to specific rotation, alpha. The formula used was:

$$\text{Alpha} = \frac{\text{Observed Angle of Rotation}}{\text{Length in Decimeters} \times \text{Density}}$$