Solvent Extraction. III. The Effect of Particle Size on Extraction Rate[1]

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PARTICLE size is one of the more important variables influencing the rate of extraction of oil, or more properly crude lipids, from oil-bearing materials with solvent. Particle size can be changed in plant operation to control the residual oil content of an extracted meal. However the minimum particle size considered in design or operation is limited by the production of fines, which have an adverse effect on the other steps of the process. This study was made to establish the magnitude of the effect of changing particle size on the rate of extraction.

Particle size for the purpose of extraction studies should be defined in terms of "effective extraction size." For example, in the case of flakes, the flake thickness is a proper measure of the effective extraction size whereas the diameter of the flake as such is of little consequence. In the case of "grits," or ground materials, the average grit diameter as determined by a standard sieve analysis is a measure of effective extraction size. Any method of preparation which produces a material that has roughly equal dimensions in all directions will produce the material designated as "grits." Examples of grits would be oil seeds ground in attrition-type mills, hammer mills, or prepared by multiple passes through smooth rolls in a 5-high stand.

The literature contains numerous qualitative references to the importance of particle size in solvent extraction of oil seeds (1,3,7,8), but very little quantitative data are available. The data of King (5), which will be discussed later, may be used to demonstrate the effect of changing average flake thickness of soybean flakes on the rate of extraction of the oil with trichlorethylene. Fan, *et al.* (2) extracted peanut slices cut with a microtome.

Experimental Procedure

Extraction rates were determined by the Percolation Method (10) in which fresh solvent is percolated through the sample and the recovered oil is measured at successive time intervals. All the extractions performed in this study used commercial hexane essentially at its boiling point as the solvent. All variables, other than effective extraction size, which might possibly influence extraction rate were maintained constant in each series of experiments. All the samples for a series were prepared from the same seed with the same conditioning; solvent, temperature, moisture content, and solvent rate were identical for each rate determination.

Moisture contents were determined by toluene distillation and vacuum oven. [Methods 27.4 and 27.3 of the Association of Official Agricultural Chemists (8).] Average flake thickness was the arithmetic average of the thicknesses of at least 100 flakes taken at random and measured by a micrometer. Sieved fractions were prepared by the hand-sieving procedure, as recommended by the National Bureau of Standards (9).

[1] Presented at the fall meeting, American Oil Chemists' Society, Chicago, Oct. 31-Nov. 2, 1949.

TABLE I

Extraction Data for Flakes

Average Flake Thickness (Inches)	Residual Oil Content (Per Cent—Dry Basis)					
Extraction Time	5 Minutes	10 Minutes	20 Minutes	30 Minutes	60 Minutes	120 Minutes
Series 1—Soybeans						
0.0085	0.78	0.39	0.25	......	......	......
0.0105	1.37	0.67	0.43	......	......	......
0.015	3.49	1.46	0.84	......	......	......
0.0175	......	2.08	1.04	0.76	......	......
0.009[a]	0.92	0.49	0.35	......	......	......
0.011	1.58	0.78	0.56	......	......	......
0.0135[a]	2.48	1.09	0.68	......	......	......
0.017[a]	......	2.11	1.13	0.84	......	......
Series 2—Soybeans						
0.0095	2.29	1.29	0.91	......	......	......
0.0115	......	1.84	1.27	1.03	......	......
0.017	......	3.43	2.22	1.75	1.19	......
Series 3—Soybeans						
0.011	1.61	0.75	0.57	0.37	0.28	0.14
0.021	3.69	2.82	1.82	1.42	0.98	0.66
Series 4—Soybeans						
0.012	0.90	0.48	0.34	0.29	0.19	......
0.0185	2.71	1.14	0.77	0.63	0.46	......
Series 7—Cottonseed						
0.0079	......	5.05	3.35	2.58	1.52	0.84
0.0082	......	5.00	3.33	2.50	1.47	0.85
0.0089	......	5.13	......	2.78	1.66	0.99
0.0107	......	6.56	......	3.43	2.13	1.24
0.0128	......	8.08	......	4.43	2.65	1.44
Series 8—Cottonseed						
0.0079	......	5.10	......	3.25	2.23	1.46
0.0083	......	5.05	......	3.20	2.20	1.44
0.0060	......	4.20	3.20	2.58	1.72	1.13
0.0080	......	......	4.20	3.60	2.37	1.47
0.0080	......	......	4.40	3.60	2.43	1.45
0.0075	......	......	5.08	3.82	2.39	1.34
0.010	......	......	4.28	3.64	2.62	1.77
Series 10—Flaxseed						
0.008	......	7.49	6.14	5.26	3.94	2.67
0.0065	3.03	2.51	1.69	1.38	0.97	0.65
0.004	2.24	1.44	0.80	0.65	0.39	......
0.004	1.79	1.37	0.80	0.67	0.45	......
0.007	9.18	......	5.80	4.61	3.32	2.23
0.0055	......	3.69	2.45	1.85	1.16	0.62
Series 12—Peanuts						
0.024	6.63	2.05	1.14	0.80	0.51	......
0.032	8.38	3.57	2.04	1.41	0.83	......

[a] Presented previously by G. Karnofsky in Short Course lectures (4).

Flaked materials hereafter designated as "laboratory mill flakes" were prepared in the laboratory flaking mill. "Commercial flakes" were made in the flaking mills of a commercial solvent extraction plant in normal operation, where flakes of several average thicknesses were produced by changing the clearance between the rolls of the mill. The samples designated as "hydraulic press flakes" were made by pressing cracked seed between steam-heated platens in a Carver laboratory hydraulic press. The moisture content and temperature of all the seeds or cracked seeds were adjusted, and the material was tempered prior to flaking to duplicate the commercial practice of preparation for extraction as nearly as possible. In the flaking operation all conditions of preparation except roll setting were the same for any series.

The soybean and cottonseed grits were prepared by grinding in laboratory attrition mill. The corn germ grits were sieved directly from a commercial grind resulting from a dry milling operation.

Experimental Results

Table I presents extraction data for flakes of various thicknesses made from soybeans, cottonseed, peanuts, and flaxseed. The data for each lot of material are recorded in a single series, in which the only variable is flake thickness. Figure 1 illustrates the data of Series 1. This is a log-log plot of percentage of residual oil content, on a moisture-free basis, against extraction time in minutes (10). The curves approximate straight lines and can be easily interpolated and extrapolated. Such curves plotted for the data of the other series are similar in appearance to those from Series 1.

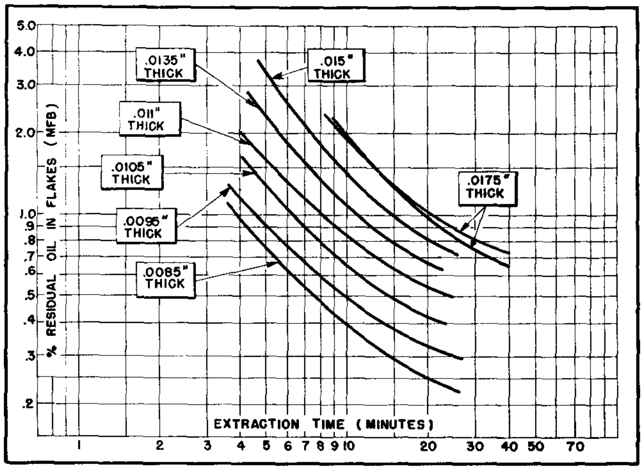

FIG. 1. Relation of extraction time and residual oil. Soybean flakes—Series No. 1.

The time to reach a residual oil of 1.0% is taken as an index of extraction rate. This time, termed "time to 1%," will be used frequently in the discussion of extraction data. It will be noted that the time to 1% increases in a regular manner with increased flake thickness although the degree varies for different oil seeds.

Closely sized fractions of ground material were prepared, and the extraction rate with hexane was determined for each fraction. The average opening of the two sieves (U.S. Standard Series) between which the fraction was collected was taken as the effective extraction size. For example, the effective extraction size of grits which pass a U.S.S. Sieve No. 35 and are retained on a No. 40 would be 0.0181 in., the average of the openings of the two sieves. Table II lists the extraction data obtained for two sets of sieved fractions of soybean grits, one series for ground cottonseed, and one series of sieved fractions of the corn germ grits described above. Again, the time to 1% increased in a regular manner with increased extraction size when the data were treated as illustrated in Figure 1.

Discussion

Curves of the data in Tables I and II were plotted for each series in a manner similar to that illustrated in Figure 1 for Series 1, where each curve corresponds to a certain thickness, or effective extraction size. From these curves, the times to 1% were read and cross plots made showing the relation between time to 1% and effective extraction size. Figure 2 is the cross plot for flakes, and Figure 3 for grits.

TABLE II
Extraction Data for Grits

Average Grit Diameter (Inches)	Residual Oil Content (Per Cent—Dry Basis)					
Extraction Time	5 Minutes	10 Minutes	20 Minutes	30 Minutes	60 Minutes	120 Minutes
			Series 5—Soybeans			
0.0256	......	6.16	5.02	4.69	3.44	2.32
0.0215[a,b]	......	4.37	3.42	2.94	2.19	1.52
0.0181[a,c]	......	2.99	2.22	1.83	1.33	0.82
0.0152[a]	2.46	1.65	1.28	0.97	0.63	0.55
0.0128[a]	1.08	0.80	0.60	0.50	......	......
			Series 6—Soybeans			
0.0181[d]	......	......	......	3.40	2.44	1.53
0.152[e]	......	......	......	2.35	1.64	0.97
0.0128	2.88	2.20	1.54	1.23	0.81	......
			Series 11—Corn Germ			
0.0256	4.34	2.69	1.76	1.34	0.87	......
0.0181	2.23	1.16	0.67	0.49	0.33	......
0.0152	1.53	0.69	0.39	0.31	0.16	......
			Series 9—Cottonseed			
0.0181[f]	19.4	15.7	12.4	10.4	7.2	2.79
0.0128	9.98	6.88	4.74	3.57	2.02	0.98

[a] Presented previously by G. Karnofsky in Short Course lectures (4).
[b] 0.0215 inches—1.09% in 180 minutes.
[c] 0.0181 inches—0.55% in 180 minutes.
[d] 0.0181 inches—1.00% in 240 minutes.
[e] 0.0152 inches—0.68% in 180 minutes.
[f] 0.0181 inches—1.30% in 360 minutes.

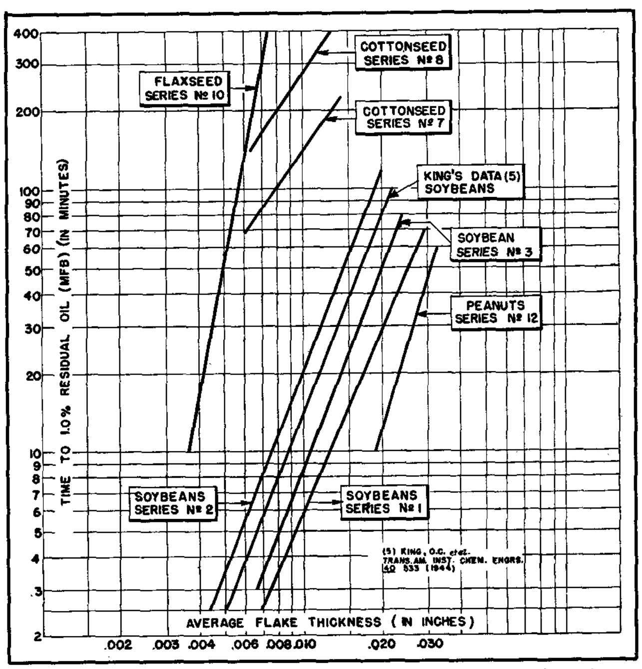

FIG. 2. Flakes. Effect of flake thickness on extraction time.

The curves obtained are essentially straight lines on log-log paper within the range of times and sizes of practical interest. The general equation of such lines is:

$$\log T = n \log D + \log K$$
$$\text{or } T = KD^n$$

where T = time to 1.0% oil content on a dry basis, minutes
D = effective extraction size in inches
K = intercept at the 1.0 inch ordinate
n = slope of the line

TABLE III
Preparation of Samples for Extraction

Series No.	Material	Moisture Content Per Cent	Source	Crop Year	Preparation Method
1	Soybean Flakes	10.7	Illinois	1946	Flaked in Commercial Mill
2	Soybean Flakes	9.5	Kansas	1948	Flaked in Laboratory Mill
3	Soybean Flakes	7.0	Illinois	1945	Flaked in Laboratory Hydraulic Press
4	Soybean Flakes	11.5	Ohio	1947	Flaked in Laboratory Hydraulic Press
5	Soybean Grits	9.5	Illinois	1945	Sieved fractions from Lab. Attr. Mill
6	Soybean Grits	7.0	Illinois	1947	Sieved fractions from Lab. Attr. Mill
7	Cottonseed Flakes	12.0	Arkansas	1947	Flaked in Laboratory Mill
8	Cottonseed Flakes	10.5	Tennessee	1948	Flaked in Laboratory Mill
9	Cottonseed Grits	6.0	Illinois	1944	Sieved fractions from Lab. Attr. Mill
10	Flaked Flaxseed	10.5	....	1947	Flaked through Commercial Rolls
11	Corn Germ Grits	12.0	Illinois	1947	Sieved fractions from dry-milling grind
12	Flaked Peanuts	7.9	Virginia	1946	Flaked in Laboratory Hydraulic Press

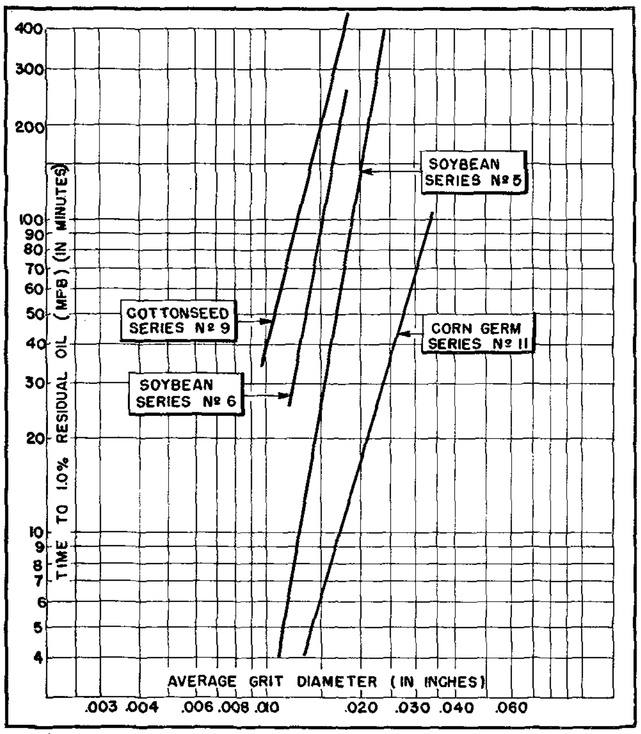

FIG. 3. Grits. Effect of grit size on extraction time.

Since the time to 1.0% residual oil content varies with the nth power of the effective extraction size, the value of n is a convenient way of expressing the effect of size on extraction rate.

Effect of Flake Thickness. Table III contains a description of the samples used in preparing the range of sizes for each series. Included in Figure 2 are the data for four series of soybean flakes. The lines for the data of King (5), the series for flakes prepared in the laboratory mill, a series of flake samples in the hydraulic press, and the series for commercially-made flakes show a surprisingly uniform slope in view of the different sources of seeds, the different manner of production, and the two solvents, hexane and trichlorethylene, used. This constant appears to be characteristic of flaked soybeans. This slope varies from 2.4 to 2.7, averaging 2.5. The importance of this value of n can be appreciated by noting, for example, that a decrease in flake thickness from 0.015 in. to 0.010 in. in Series 2 decreases the time to 1% residual oil from 57 minutes to 21 minutes.

In Figure 2 two series are given for cottonseed that show almost identical values for n. However there is reason to believe from other data not given here that the value for n for cottonseed may be influenced by the method of preparation. The value of n for cottonseed flakes determined from Figure 2 is 1.5. This indicates that the extraction time is influenced to a lesser degree by change in flake thickness than is the case for soybeans.

The values of n for the different materials shown on Figure 2 are: cottonseed flakes, 1.5; peanut flakes, 2.2; soybean flakes, 2.5; and flaxseed flakes, 7. Comparison of these values for the various oil seeds indicates the relative effect on extraction time of changing flake thickness. Another point of interest that is illustrated by Figure 2 is the relative extractability of the different oil seeds. For example, at a flake thickness of 0.010 in. it is seen that peanuts are most readily extracted, followed by soybeans, cottonseed, and flaxseed in that order.

Effect of Grit Diameter. Figure 3 illustrates the effect of the size of screened grits on their extraction rate. As is the case of soybean flakes, the lines for the two series of soybean grits have practically the same slope. The average slope of the lines for soybeans in Figure 3 is 5.5, meaning that the time required to reduce the residual lipid content of soybean grits is a function of the 5.5 power of the average grit diameter. The change of extraction rate with effective size is therefore much greater than for soybean flakes. This indicates the importance of a short-range particle size distribution in material prepared by grinding. It is apparent that a relatively small amount of coarse material would extend the extraction time of the entire sample considerably. The extraction rates of sieved fractions of corn germ grits and cottonseed grits are less affected by particle size than are the rates for soybean grits. The value of n for the different materials are: corn germ grits, 3.4; cottonseed grits, 4; and soybean grits, 5.5. The value of n is much greater for grits than for flakes of the same material. A comparison of the extractability at the same grit size shows corn germ grits to be the most extractable, followed by soybeans and cottonseed.

Summary

Quantitative data have been presented for the effect of "effective extraction size" on the rate of extraction of oil from soybeans, peanuts, cottonseed, corn germ grits, and flaxseed. Both flakes and grits have been studied. The results are best correlated by a straight-line plot on log-log paper of "time to 1% residual" and effective extraction size. The slope of the line is a measure of the change in extraction rate with extraction size. The slope was found to be different for the different oil seeds and to be larger for seeds prepared as grits than for the same seed prepared as flakes.

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REFERENCES

1. Collins, F. I., and Kroher, O. A., Oil & Soap, *22*, 307-311 (1945).
2. Fan, H. P., *et al.*, Ind. Eng. Chem., *40*, 195-199 (1948).
3. Gastrock, A. E., and D'Aquin, E. L., Oil Mill Gaz., *53*, No. 4, 13-21 (1948).
4. Karnofsky, G., J. Am. Oil Chem. Soc., *26*, 564-570 (1949).
5. King, O. C., *et al.*, Trans. Am. Inst. Chem. Engrs., *40*, 533-556 (1944).
6. MacGee, A. E., Oil Mill Gaz., *53*, No. 12, 13-20 (1949).
7. McCormack, R. H., J. Am. Oil Chem. Soc., *24*, 299-303 (1947).
8. "Methods of Analysis," Association of Official Agricultural Chemists, 6th Ed. 1945, as revised to Oct. 1948, page 404.
9. "Profitable Use of Testing Sieves," Catalog 53, p. 21, W. S. Tyler Co., Cleveland, 1940.
10. Wingard, M. R., and Shand, W. C., J. Am. Oil Chem. Soc., *26*, 422-426 (1949).

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Choice and Application of a Detergency Test Method[1]

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THE correct evaluation of detergent compositions in a variety of end uses is of vital and continuing interest to the detergent industry. A growing number of test procedures have been independently described in the literature (2, 3, 4), particularly during the past 10 years which have witnessed the greatest expansion of the synthetic detergent business. Some of these tests are relatively simple and closely parallel actual use conditions. Others are more complex, having diverged markedly from conditions simulating end application in the quest of more exact reproducibility. When properly interpreted however, the majority of these tests have proven to be capable of consistent results.

As a contribution to the information on detergency test methods herewith is presented a typical launderometer procedure which has been successfully applied over a number of years to the evaluation of detergents based on the Ultrawets, an homologous series of alkyl aryl sulfonates.

This test procedure was developed specifically for the evaluation of heavy duty detergents, that is detergents for heavily soiled white cotton goods. To be satisfactory it was considered essential that the test first, accurately evaluate the efficacy and utility of the products in the field of intended application; second, give consistent comparative detergency patterns with respect to a standard reference detergent; and last, be sufficiently simple in nature to permit rapid performance with a minimum of specialized equipment and skills.

To insure meeting the first and most important of these requirements, relative performance ratings on cotton were first established on an accepted proprietary detergent (a built fatty acid soap) and on a typical unbuilt alkyl aryl sulfonate (35% active, 65% sodium sulfate) known to be deficient in this application. This was accomplished by subjecting white cotton shirts and towels to repeated cycles of soiling, by normal wear and use in families including young children, and laundering in typical household equipment. Series of three, five, 10, and 20 cycles were employed to establish relative field performance ratings on different detergents. All field tests ratings were based on visual comparisons made significant by carrying the tests through a number of cycles sufficient to produce marked differences.

Working from these field ratings on products of different merit, a launderometer procedure was then sought which would show like differences in performance under test conditions approximating actual use conditions. Paralleling what was considered to be typical home laundry practice, use conditions of 100 p.p.m. hardness water, a temperature of 120°F., and a wash cycle time of 20 minutes were adopted.

The choice of test swatch size, 2″ x 4″, was based on the A.A.T.C.C. Method for Color-fastness to Domestic Washing of Cotton (6). The relatively small 2″ x 4″ swatch is sufficiently mobile in the Launderometer test jar to insure a uniform degree of mechanical flexing which is important to uniform detergency results. While the standard test swatch containing on the average 0.0215 g. (3.0 wt. %) of soil represents a light poundage load of soiled fabric on the volume of detergent solution used, this has proven to be a minor factor in test correlation with field performance. Then by experimentation a combination of test fabric and soil was found which was capable of giving the desired comparative detergent patterns which are plots of detergency versus detergent concentration. Using the above standardized conditions, experiments were run to find the combination of cloth plus soil composition which would give the results desired.

A white oxford cloth was adopted as the preferred fabric in this test because it seemed to give more uniform results, possibly because of the loose weave and the very light starch processing size which can be completely removed by simple means. Permanently sized cloth did not give the desired results with the varied soils tested.

The composition of, and detergency patterns obtained with a number of the different soils tested, are shown in Figure 1. The 1 AX soil was chosen for our test method since, with it, the satisfactory proprietary detergent gave a near white deterged swatch in a single wash cycle and yet left sufficient residual soil to permit the method to be used in evaluating improved or superior products; and, of most importance, the 1 AX soil gave clearly distinguishable differences in detergency patterns between the known satisfactory and unsatisfactory reference compositions. It is of interest to note in passing that

[1] Presented at fall meeting, American Oil Chemists' Society, Oct. 31-Nov. 2, 1949, in Chicago.