Solvent Extraction of Cottonseed and Peanut Oils. VIII. Effects of Moisture on the Preparation and Flaking of Cottonseed

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THE solution to the many problems involved in the solvent extraction of a material must lie mainly in two considerations: one, the nature of the feed material itself (5) and the other, a suitable extractor design (5). Perhaps the first of these is the more important for a well designed extractor may prove unsatisfactory when operated with a feed material which does not lend itself to extraction.

All types of solvent extractors being considered for the processing of cottonseed make, to a lesser or greater extent, two demands upon the feed material: that it contain relatively few fines which can be carried out with the miscella (2) and that it allow free percolation of the solvent (2). For example, the basket-type extractor requires a close control of the percolation rate to prevent overflowing of the basket if the rate were too low or to prevent the upper layer of flakes from being unimmersed as would occur if the rate were too high (5). Other types involving agitation, demand primarily a material with few fines.

The influence of the nature of the material is the interest of the present study. In particular, the study is concerned with the effect of moisture content of delintered cottonseed on the preparation of flakes having relatively few fines and allowing the ready percolation of solvent. The work consisted of hauling, separating, and flaking in the pilot plant delintered seed of different moisture levels and then evaluating in the laboratory the flakes produced.

Materials and Procedure

The cottonseed used had been delintered and was from the Eufaula, Alabama, crop of 1947. It had been stored for approximately two years. The solvent used was commercial hexane having a specific gravity of 0.678.

Six experiments were made, each with seed at a different moisture level.

Pilot Plant Hulling, Separating, and Flaking. No moisture adjustment was used for Experiment No. 1 in which a 200-lb. batch of the delintered seed was hulled at its natural moisture. In experiments Nos. 2, 5, and 6 a calculated amount of water was added to 200-lb. batches of the seed three days before hulling. In experiments Nos. 3 and 4 200-lb. batches of the seed were dried in a tray oven at 130° F. for 24 and 4 hours respectively, three days before the seed was hulled. This time lapse between moisture adjustment and hulling was considered necessary to allow the seed to come to equilibrium in the drum container (1).

In all experiments the seed was then double-hulled and separated with identical settings using the equipment described in the flow diagram, Figure 1. All screens indicated in this diagram have round holes. The four fractions indicated on the figure were obtained. Following this operation, a portion of the whole meats was cracked and flaked, and in a separate step a portion of the fine meats was flaked. In addition, a combined meats fraction was made by mixing whole meats and fine meats in their collected proportions. This combined meats fraction was cracked and flaked. All flakes made were of 0.010 in. thickness.

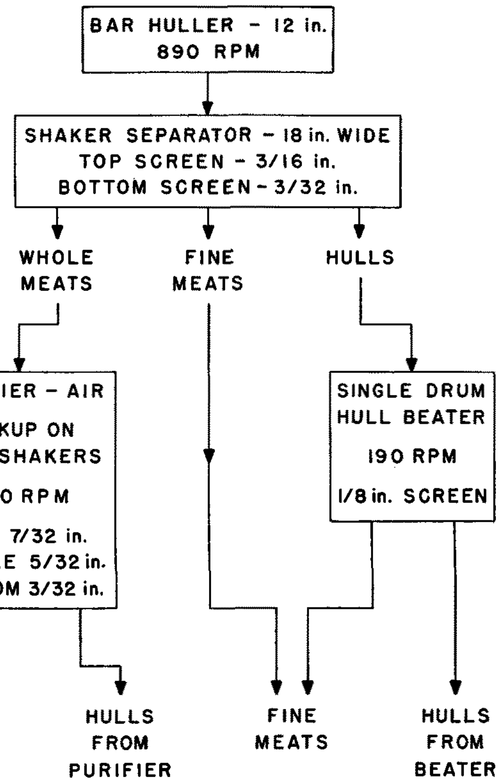

FIGURE 1. Flow diagram of hulling and separating equipment.

The cracking rolls used were corrugated rolls, 12 x 12 in., with a differential speed of 1:2.35, and with a speed of fast roll of 220 r.p.m. The clearance between rolls was .025 in. The flaking rolls used were smooth rolls, 12 x 12 in., with identical speeds of 220 r.p.m.

Laboratory Evaluation of the Flakes Produced. Small portions of all flakes produced in the six experiments were evaluated on a laboratory scale by the four tests and the analytical methods described below.

Dry screening test. One hundred grams of the prepared flakes were screened on a Rotap Machine[2] for 10 minutes, using 5-8-14-20- and 40-mesh screens to determine flake size.

Wet screening test. One hundred grams of the prepared flakes were placed on a 300-mesh screen. A 300-mesh screen was chosen because screen analyses

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[2] Mention of trade products does not imply their endorsement by the U. S. Department of Agriculture over similar products not mentioned.

TABLE I
Moisture Contents of Seeds and Separated Fractions

Experiment Number	3	4	1	6	5	2
Moisture content of seed before adjustment, %	8.50	8.50	8.50	8.50	8.50	8.50
Moisture content of seed after adjustment, %	3.72	7.48	8.50	12.10	12.97	13.69
Moisture content of whole meats fraction, %	3.58	6.20	7.69	10.10	11.15	11.50
Moisture content of fine meats fraction, %	3.97	6.97	8.55	11.06	13.20	13.10
Moisture content of hulls from beater, %	5.08	10.30	12.32	14.55	14.77	15.88
Moisture content of hulls from purifier, %	5.16	10.40	11.85	13.42	14.41	14.68

of the solids carried out with the miscella from the continuous solvent extraction plant (4) showed that approximately 80% of the solids were through 300-mesh. The screen was partially submerged in hexane so that the flakes were completely covered with solvent and then shaken for approximately five minutes. The screen was then removed from the miscella and the process repeated twice in fresh hexane. After the third screening the three miscellas were combined and filtered. The filtered solids were washed with hexane, air dried, and weighed, and this weight showed the amount of "initial" solids through 300-mesh. The percentage of through 300-mesh solids was calculated by dividing the weight of filtered solids by the weight of flakes used less the weight of their oil.

Percolation test. Percolation tubes were fabricated from 51-mm. glass tubing with an overflow outlet 19 in. from the bottom. An 80-mesh screen in the bottom kept the flakes in the tube. The flakes were compacted (after each 100-g. addition) under a 4.1-lb. weight until the tube was filled 10 in. from the bottom. This method of loading was found empirically to give results that could be duplicated satisfactorily. The weight of the flakes used was recorded. With a constant head (excess overflowing through the outlet), fresh hexane was percolated through the flake bed for 25 minutes. The volume of miscella discharged from the bottom of the tube was measured and this value, with the ones of the tube and the specific gravity of the miscella, was used in calculating the mass velocity values reported in Table III. The meal in the tube was removed, air-dried, and analyzed for percentage of lipids.

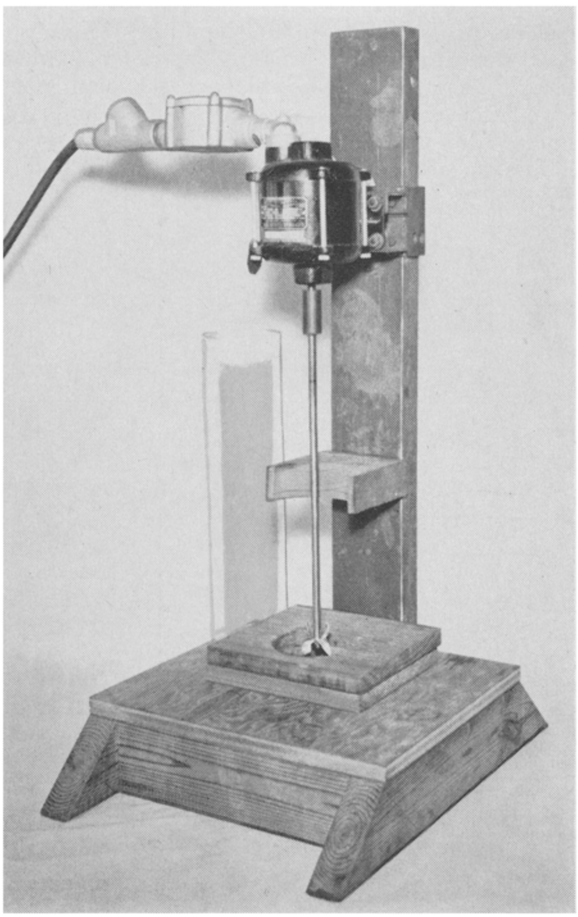

FIGURE 2. Container and impeller used in disintegration test.

Disintegration test. Two hundred grams of flakes were added to a cylindrical container 85-mm. in diameter which contained 1,000 cc. of solvent. The slurry was then disintegrated for five minutes using a laboratory-type impeller 1½ in. in diameter which was similar to a Waring Blender impeller.[2] The container and impeller are shown in Figure 2. The impeller speed was 1,700 r.p.m. The solids were then wet-screened on a 300-mesh screen. The through portion was filtered and the solids washed with hexane, air-dried, and weighed. The percentage of through 300-mesh solids was calculated by dividing the weight of the filtered solids by the weight of flakes used less the weight of their oil.

Analytical methods. All fractions produced from the hulling, separating, and flaking operations were analyzed for water, oil, and nitrogen, using Standard A.O.C.S. methods.

Results

Moisture Adjusting. Table I shows, arranged in increasing order, the moisture contents of the seeds for the different experiments and the moisture contents of the four fractions from the hulling and separating equipment. The differences in moisture between the whole meats fractions (practically pure meats) and the hulls from the beater (practically pure hulls) show that the moisture-adjusting procedures used gave artificially moistened seed which was similar in moisture distribution between meats and hulls to seed of natural moisture content.

Material Balances. Table II indicates the following effects of moisture content of the meats upon the material balances from the hulling and separating equipment:

1. As moisture content increases, the percentage of total meats recovered in the whole meats fraction increases and the percentage in the fine meats fraction decreases.
2. The hull content of the combined meats is at a minimum at approximately 10% moisture in the meats. It increases rapidly at lower moisture levels and tends to increase slowly at higher moisture levels.
3. The oil content of the hulls from the beater remains at approximately 0.5% until the moisture level is quite high, approximately 11% moisture in the meats, and then it about doubles. This confirms previously reported results (6).

TABLE II

Effect of Moisture Contents of Seed on the Material Balance of the Hulling and Separating Operations

Experiment Number	3	4	1	6	5	2
Moisture content of meats after adjustment, %	3.58	6.20	7.69	10.10	11.15	11.20
Fraction of the total meats present in the whole meats fraction, % [a]	40.4	46.3	60.8	77.0	86.2	82.7
Fraction of the total meats present in the fine meats fraction, % [b]	58.9	53.1	34.3	20.9	12.4	13.9
Fraction of the total meats present in the hulls from beater, % [a]	0.0	0.0	0.0	0.0	0.3	0.2
Fraction of the total meats present in the hulls from purifier, % [b]	0.7	0.6	4.9	2.1	1.1	3.2
Hull content of whole meats, % [a]	4.6	3.7	3.3	0.4	3.4	2.2
Hull content of fine meats, % [b]	22.4	18.0	22.4	22.1	34.8	32.4
Hull content of combined meats, % [b]	16.0	11.9	11.2	6.0	8.9	9.7
Oil content of hulls from beater, % m.f.b.	0.55	0.52	0.46	0.45	1.26	0.91

[a] Hull and meat content of fraction determined by hand-picking. [b] Hull and meat content of fraction determined by nitrogen analyses.

Table III shows the effect of moisture content of flakes upon their percolation characteristics, extractibilities, and fines-producing tendencies.

Percolation. It has been shown (3) that the rate of flow (percolation) of a fluid (hexane) through a bed of porous media (flake bed) is dependent upon the diameter of the particles constituting the bed, the particle shape and roughness, the porosity of the bed (volume of voids per volume of bed), the depth of the bed, the height of the fluid head above the bed, and the viscosity and density of the fluid. In the percolation tests reported herein the last four named variables and the particle shape were kept essentially constant. So the remaining variables which could affect percolation rates are the particle diameter, particle roughness, and the porosity of the bed. It was observed that air rose from the bed during the first 30 seconds of the experiment but ceased abruptly, indicating that substantially all of the air had been displaced. This variable was considered small enough to neglect.

The percentages of the flakes through 20-mesh by the dry screen analyses and the bulk densities of the flake beds were used as indicators of particle diameters and bed porosities for the flakes tested. No attempt was made to compare particle roughnesses, but it should be kept in mind that this property varies from flake to flake because of variations in hull content and possibly moisture content. With these factors in view it was possible to understand the large variations in percolation rates which were measured with different flakes. The percolation data in Table III shows that an increase in rate occurs with increase in moisture until a maximum is reached at a moisture of 9 to 10%. Then the rate decreases rapidly, particularly with whole meat flakes.

These variations may be explained in the light of the particle size and the bulk density data of Table III. At the lowest moisture level the particle sizes are very small, approximately 80% through 20-mesh. The fines fill the void in the bed more completely, resulting in a slower rate of percolation. As the moisture content increases in the succeeding experiments, the average particle size increases and the rate increases. At the two highest moisture levels however the rate of percolation decreases, but the particle size is still large. The decrease is explainable from the bulk density data, which show a rapid increase with moisture, and the bulk density rise is undoubtedly due to the fact that at high moisture content the meats are soft and pliable and the voids are decreased due to packing. This explanation is evidenced further by the fact that at the highest moisture levels the whole meat flakes (approximately 3% hulls) have a much lower percolation rate than their corresponding fine meat flakes (approximately 35% hulls).

TABLE III

Effects of Moisture Contents of Seed on Percolation Characteristics, Extractibilities, and Fines-Producing Tendencies of Flakes

Description of flakes	Exp. No.	Moisture content of flakes when tested [a]	Percolation Data			Extractibility		Fines	
			Mass velocity of miscella thru bed	Particle size. Fraction of undefatted flakes thru 20 mesh [b]	Bulk density of flakes in bed	Oil content of flakes defatted by percolation test		Fraction of solids in undefatted flakes thru 300 mesh	
						M.F.B.	M.F. & hull free basis [c]	Before disintegration	After disintegration
		%	Lb./sq. ft./hr.	%	Lb./cu. ft.	%	%	%	%
Flakes from whole meats	3	3.65	625	81.3	27.3	2.57	2.73	11.4	13.5
Flakes from fine meats		4.05	433	86.0	28.6	2.55	3.54	8.3	9.8
Flakes from combined meats		3.82	645	76.2	27.3	3.10	3.90	11.0	12.7
Flakes from whole meats	4	6.19	945	62.3	27.3	3.00	3.14	6.1	7.5
Flakes from fine meats		6.80	727	66.3	28.6	3.19	4.14	7.4	7.3
Flakes from combined meats		6.60	740	73.3	27.3	2.93	3.44	6.2	6.0
Flakes from whole meats	1	7.65	1430	14.1	28.6	2.50	2.61	1.6	3.6
Flakes from fine meats		8.60	1210	40.6	28.6	2.85	3.89	1.8	2.8
Flakes from combined meats		8.29	1110	27.0	28.6	2.14	2.48	1.5	2.7
Flakes from whole meats	6	9.21	2880	6 7	26.0	2.01	2.02	0.6	3.7
Flakes from fine meats		10.53	2610	35.0	26.0	3.45	4.31	0.8	2.6
Flakes from combined meats		10.02	2620	17.6	26.0	2.57	2.71	0.6	2.8
Flakes from whole meats	5	11.00	894	8.1	29.3	2.27	2.39	0.9	3.8
Flakes from fine meats		12.60	1737	33.9	27.3	2.66	4.31	1.3	2.8
Flakes from combined meats		11.27	1172	9.8	28.6	2.34	2.62	1.1	4.1
Flakes from whole meats	2	11.60	625	4.7	37.8	2.75	2.83	0.9	4.0
Flakes from fine meats		12.98	1615	40.6	32.6	2.63	4.20	1.1	3.4
Flakes from combined meats		11.90	1217	10.0	32.6	2.08	2.38	1.2	3.8

[a] The variations between the moisture contents of the flakes when tested and the moisture contents of the meats from which they were prepared are probably due to losses or gains in moisture while handling. [b] Determined by dry-screen analyses. [c] Calculated oil content of extracted meats in flakes, assuming the hulls have 0.4% oil.

Since it is recognized that moisture content does not affect the pliability of hulls to the same degree that it affects the pliability of meats, it is reasonable that the fine meat flakes, where only about 65% of the flake bed (meats portions) was made soft, had a lower bulk density with a corresponding increase in percolation rate over its companion whole meats flake of equal moisture level.

Extractibility. Table III also shows the effect of moisture content upon extractibility. There is little difference between flakes of various moisture contents in the residual oil left in the meal after the 25-minute percolation test. In the case of whole meat flakes the highest extractibility (lowest residual oil) appears to be at a moisture content of approximately 9-10%. In the case of fine meat flakes the extractibility appears to be unaffected by the moisture content. For combined meat flakes there is a trend downward which indicates that an increase in moisture level gives an increased extractibility. However the changes in extractibility are all small; and the results therefore not conclusive.

Fines. Table III shows that moisture content does have a pronounced effect on the fines-producing tendencies of flakes. The fines present before disintegration, which is the case more closely resembling the basket extractor having least flake handling, is at a minimum at a moisture level of approximately 9 to 10%. Decreased moisture causes a large rise in fines so that at approximately 4% moisture the amount of fines is about 12 times greater than at 9% moisture. Increased moisture causes a small rise in fines. At approximately 12% moisture the amount of fines is about 1½ times greater than at 9% moisture.

The fines present after disintegration, which is the case more closely resembling the extractor which agitates the flake bed, is also a minimum at approximately 9-10% moisture. However the variations between the amount of fines at the minimum and the amounts at other moisture levels are not so great as the variations before disintegration.

Conclusions

The results indicate that 9 to 10% moisture in the meats is the best level for the preparation of cottonseed into flakes for solvent extraction. At lower moisture the flakes contain more fines before and after agitation in solvent, and the percolation rate of solvent through a flake bed is lower because of reduced particle size. At higher moisture the amount of oil in the hulls from the beater is greater. Also the flakes produce more fines, and the percolation rate becomes slower because of the softness and pliability of the flaked meat. It is likely that in commercial continuous operations this packing or balling up tendency of the flaked meats may lead to periodic channeling or plugging in the extractor, dryer, conveyors, and filters.

These conclusions have been applied in the continuous pilot plant solvent extraction of three lots of prime cottonseed. The results substantiate the findings herein and will be reported in a separate publication.

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Solvent Extraction IV. The Effect of Temperature on Extraction Rate

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SINCE temperature is a significant factor influencing the rate of extraction of crude lipids from oil seeds with solvents, a knowledge of the relationship between extraction rate and temperature is important in solvent extraction plant design and operation. The literature contains very little quantitative data although several references to the qualitative effect of temperature have been made (1, 3, 5, 6, 8). Karnofsky presented data on the extraction of cottonseed in his Short Course lectures (7). Boucher *et al.* extracted degummed soybean oil from porous clay plates with chlorinated hydrocarbon solvents at several temperatures (2).

In considering the effect of temperature on extraction rate, the experimenter is immediately confronted with the fact that the total "oil" extractable from seeds is itself a variable which may change with the extraction temperature (4, 7, 11). For this reason the results would have little theoretical value unless the residual oil at any time were determined by an analytical method employing the same solvent and temperature as the rate experiment. Of more practical interest is the residual oil as determined by the standard method Ba 3-38 of the A.O.C.S. (10).

For convenience in our laboratory, analyses for residual oil are made by extracting the ground sample with hexane for two hours at the boiling point, which is 150°F. The residuals determined in this way are slightly higher than those found by the standard method.

Experimental Procedure

The Percolation Method (11), modified where necessary, was used in determining extraction rates. The modifications were necessitated by operating conditions other than those specified in the original Percolation Method, namely, the use of solvents other