fairly accurate theoretical curves for the production of conjugated diene and triene for different geometrical isomers of linolenic acid. Consequently it will be possible to assign a geometrical configuration to a particular linolenic acid, once its diene and triene time curves have been determined. With linoleic acid, determination of configuration is possible by combining a knowledge of the geometrical isomers formed and certain theoretical predictions of the geometrical isomers produced from a given configuration. In the case of linoleic acid the available experimental evidence is in good agreement with the conclusion that naturally occurring linoleic acid is of the cis-cis configuration and that Kass's linoelaidic acid is of the trans-trans configuration. Altogether it seems highly probable that adequate experimental evidence combined with the theory presented would allow accurate assignment of geometrical configuration to the various isomers of linoleic acid, linolenic acid, and arachidonic acid.

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Pilot-Plant Fractionation of Cottonseed. III. Process Development of Differential Settling[1, 2]

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ABSTRACT

Chemical engineering data are presented to show the pilot-plant process development of cottonseed fractionation employing the differential settling principle. The purpose of the process is to produce a cottonseed meal fraction essentially free of pigment glands and hulls, and a second fraction in which the pigment glands are concentrated sufficiently to serve as a raw material if pharmaceutical or other industrial use is developed for the glands or the pigments. The non-lipids fraction will make available a meal of high nutritive value and a source of industrial protein.

Unit operations involved, including machinery and other equipment required, and proposed flow diagrams for commercial application are discussed. In brief the unit operations are as follows: material preparation; disintegration for proper size reduction of cottonseed flakes (either defatted or undefatted) in solvent slurries; separation by tank differential settling or by centrifugal differential settling at 62 times gravity; meal recovery to recover separated fractions by either centrifuging at 1450 times gravity or by pressure filtration; desolventization of solvent-damp meal; and oil and solvent recovery.

IT has long been felt by the cottonseed industry that the nutritional and industrial value of cottonseed meal could be materially improved by removal of the glands which contain gossypol and other pigments of cottonseed.

The pigments of cottonseed can be removed from the flakes by two general methods. One method is to extract the flakes with an organic solvent in which the pigments are soluble, for example, acetone, ethyl ether, isopropanol, and methyl alcohol. Although by this method a meal low in gossypol is produced, the oil-solvent mixture (miscella) contains most of the pigments and other non-oil compounds which complicate recovery of the oil (4).

The second general method is to detach the pigment glands intact from the flakes or meal by disintegration of the flakes or meal in a solvent slurry (3) followed by separation (fractionation) of these two components. This method takes advantage of the high mechanical strength of the glands and of their natural detachability from the remainder of the kernel tissue as well as of the fact that these glands are not affected by certain solvents, such as the low-boiling petroleum cuts and some chlorohydrocarbons.

Utilizing this second general method, a mixed-solvent flotation process of cottonseed fractionation was developed first on a laboratory scale (3) and then on a pre-pilot-plant scale (8). This process employed a mixture of the two solvents, hexane and tetrachlorethylene, in a proportion to give a resulting specific gravity of 1.378. At this specific gravity the pigment glands tended to float and the meal and hulls to sink since the solvent specific gravity was intermediate to that of the cottonseed components being separated. The pre-pilot-plant scale development proved adequate for producing sufficient pigment glands and meal (essentially free of glands) for early nutritional studies (2). But the method was not considered commercially feasible because it required a high-boiling solvent such as tetrachlorethylene, whose removal from the oil or the meal calls for high vacuum or high temperatures. The use of high vacuum is costly; and the use of high temperatures lowers the value of the oil by color fixation (6) and the nutritive value of the meal by decreasing the protein solubility (1). Moreover, a 3-component system of oil, hexane, and tetrachlorethylene is formed, complicating the recovery of both solvents and oil.

Consequently a second fractionation process was developed. It is called "differential settling" and has been developed on a laboratory-scale (7) and a pilot-plant-scale using the "disintegration and separation" method. Differential settling overcomes the inherent disadvantages of the mixed-solvent flotation process primarily by the use of only one solvent, com-

[1] Report of a study made under the Research and Marketing Act of 1946.
[2] Presented at the spring meeting of the American Oil Chemists' Society, held in New Orleans, La., May 1949.
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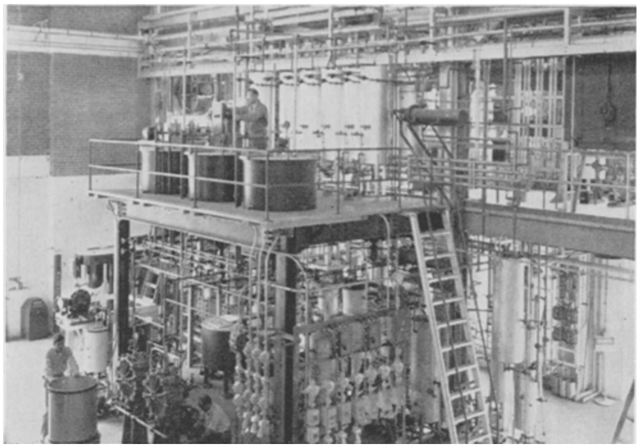

FIG. 1. Cottonseed fractionation pilot plant.

mercial hexane, which is widely used in the solvent extraction of oilseeds.

This differential settling process is based upon a principle that takes advantage of the different frictional resistances of the various components of cottonseed to a liquid medium. For example, when cottonseed flakes are sufficiently disintegrated in hexane to detach the pigment glands completely from the meal, a fine fluffy meal is produced which, regardless of its higher specific gravity, settles at a slower rate than the smooth, ovoid-shaped pigment glands. The laboratory-scale development showed that when a well-disintegrated cottonseed flake-hexane slurry was screened through 80-mesh, diluted, and settled for 20 minutes, 95% of the fine meal (2 to 40 microns) or 77% of the total meal through 80-mesh was still in suspension, and that the hulls, coarse meal, and pigment glands had settled to the bottom. The fine meal free of pigment glands could be decanted and recovered.

The pilot-plant development of this differential settling process is reported in this paper. Included are data on the relationships between operating variables and products as well as on the commercial possibilities of the process illustrated with proposed flow diagrams.

Equipment and Unit Operations

The pilot-plant equipment is shown in Figure 1. The plant was designed and constructed in a manner to permit the maximum flexibility of the engineering study of as many unit operations and processes as possible.

Equipment includes: a 10-h.p., dissolver-type disintegration unit (Figure 2); an 18-x28-inch continuous, horizontal-bowl, stainless-steel centrifuge equipped with cake and effluent discharge tanks (Figure 3); two vaportight, rotating-leaf, pressure filters; three 300-gallon tanks with stirrers and swing suction pipes; a solvent recovery system; and auxiliary equipment, such as evaporators, condensers, storage tanks, filter crocks, and explosion-proof motors and switches.

The unit operations studied were: *material preparation,* the production of suitable flakes, meal, or pulverized meats; *disintegration,* the particle size reduction of the prepared cottonseed material in solvent slurries to permit the separation of whole pigment glands from the meal tissue; *separation,* the removal of the fine meal fraction from the coarse meal fraction within the solvent slurry by means of tank settling or continuous centrifugal settling; *meal recovery,* the recovery of the two meal fractions from the solvent by the use of filters and centrifuges; *desolventization,* the removal of solvent from solvent-damp meal under such conditions as to produce a meal suitable for nutritional and industrial uses; *oil and solvent recovery,* the removal of solvent from the miscella to obtain an oil equal in quality to that commercially produced by other processes.

Material Preparation

Cottonseed flakes totaling 3,203 lb. were fractionated in 10 runs of the plant. Material for Run No. 1 consisted of hexane-extracted flakes from Eufaula, Alabama, 1946 prime seed; and for Run Nos. 2, 3, 4, 5, 8, 9, and 10 it consisted of undefatted flakes from Eufaula, Alabama, 1947 prime seed. The seeds were delinted, dehulled, and flaked in conventional semi-commercial cottonseed preparation equipment. Flake thicknesses averaged 0.008 inch. Material for Run Nos. 6 and 7 was the bottom or coarse fractions obtained from the differential settling of other runs.

To study the effect of hulls upon meal disintegration and final meal purity the flakes for Run No. 9 were prepared from the fine meats obtained from the cottonseed preparation equipment. These fine meats had a high hull content (19.9%) as compared to the much lower hull content (8.0 to 9.6%, solids basis) of the whole meat fractions used in most of the other runs. An extremely low hull content material (1.97%, solids basis) was prepared for Run No. 10 by dry-pulverizing the flakes, followed by screening over a 20-mesh screen to remove a major portion of the hulls.

Previous studies had indicated that a low moisture content of the feed material was necessary for effi-

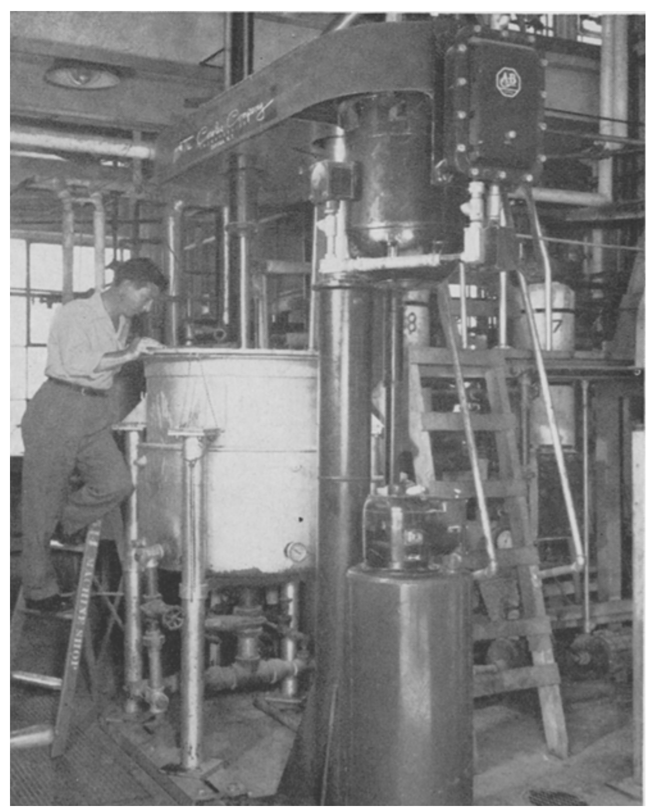

FIG. 2. Ten horsepower dissolver-type disintegration unit.

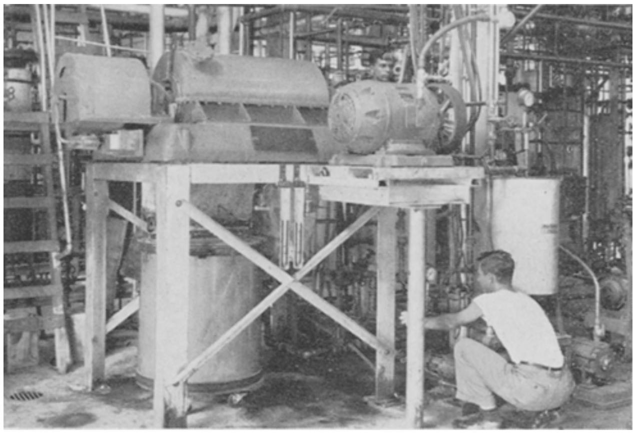

FIG. 3. Continuous horizontal bowl centrifuge (18"x28").

cient disintegration (5) but that high drying temperatures had a tendency to denature or decrease the protein solubility of the subsequent meal (1). Therefore in these runs a tray drying procedure was developed and maintained for reducing the moisture content of the feed material from an average of 7.8% to an average of 3.4% by drying at 140°-145°F. for three hours.

Disintegration

The dissolver-type disintegrator utilizes the liquid shear principle produced by a high-speed, vaned impeller and was shown in a previous study (5) with a 2-h.p. model to be satisfactory for the particle-size reduction of cottonseed flakes in solvent slurries. In this work a 10-h.p. commercial disintegrator of this type was used. Experimentation with this model showed that the best disintegration action was obtained with an 8-inch, double-vaned (vane width 3/16 inch) impeller turning at a peripheral speed of 6,000 feet per minute. The disintegration tank was dish-bottomed and was 29 inches in diameter, giving a ratio of 3.6 to 1 for the tank and impeller diameters. Four baffles at 45° angle to the tank tangent appeared to give better disintegration action than, for example, two baffles at a 60° angle.

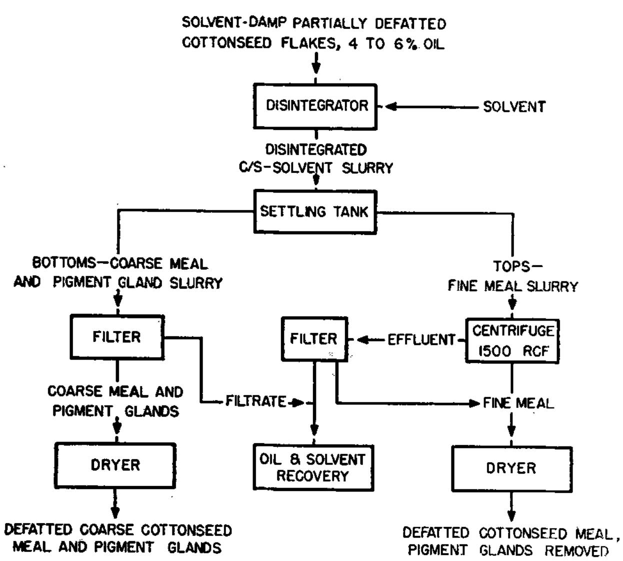

FIG. 4. Fractionation of partially defatted cottonseed flakes using tank differential settling.

The nine disintegration runs are summarized in Table I. Slurry concentrations in all cases were approximately 50% solids by weight. The oil content of the flakes and the solvent used were calculated as part of the liquid portion of the slurry. For example, to prepare a slurry utilizing 240 lb. of flakes containing 34% oil only 82 lb. (14 gallons) of commercial hexane was required. The percentage of meal through 80-mesh was used as a criterion of the degree of disintegration since it was shown that when the meal was sufficiently disintegrated to pass a screen of this mesh the pigment glands were sufficiently detached from the meal. Table I shows the percentage of meal through 300-mesh also since only meal in this fineness range (2 to 40 microns) was recoverable free of pigment glands.

An "intermittent" method of disintegration (used in Run Nos. 8 and 10) represented a major improvement in the disintegration operation. This method consisted of the periodic removal of part of the disintegrated slurry containing a major part of the fine meal and pigment glands followed by addition of fresh flakes to the portion of the slurry remaining in the disintegration tank. For example, in Run No. 8a, 260 lb. of flakes were disintegrated for 10 minutes, diluted and promptly decanted. The decantate was pumped to storage tanks, the bottoms remaining to be further disintegrated with fresh flakes. This step was repeated a second time with the addition of 120 lb. of flakes and 10 minutes of disintegration. In the final step 69 lb. of flakes were added and the slurry was disintegrated 30 minutes. In Run No. 10 three 10-minute periods and a final 33-minute disintegration time was used to disintegrate a total of 800 lb. of flakes. A complete flow sheet for Run No. 10 is shown in Figure 6.

Figure 7 in which the percentage of meal through 80-mesh is plotted versus the disintegration time shows a comparison of intermittent and batch operations (Run Nos. 8a and 5). The curve shows that the intermittent operation resulted in better disintegration at less actual operating time although the capacity (449 lb. of flakes as compared to 366 lb.) was higher with the intermittent run. In intermittent Run No. 10 a capacity of 800 lb. in 63 minutes total operating time was attained. The average disintegration from zero to 50 minutes, expressed as pounds per minute through 80-mesh for the 9 runs are shown in Table III. The two intermittent runs ranged from 4 to 6 lb. per minute through 80-mesh as compared to 1.8 to 2.5 lb. per minute for the batch operations.

It was found that the presence of hulls in the disintegrating material had an appreciable effect upon the degree of disintegration and later upon the quality of separated fine meal in respect to pigment gland content. This was especially true in the intermittent runs in which hulls accumulated in the tank due to the periodic decanting procedure. Table I shows that a higher degree of disintegration was obtained for Run No. 8a which contained 8.0% hulls than for Run No. 10 which contained 2.0% hulls; however the fine meal from Run No. 8a contained 7.0% of the original glands as compared to 1.5% for Run No. 10. Batch Run No. 9 which contained 19.9% hulls gave satisfactory disintegration; but it too contained a high percentage of glands in the resulting fine meal.

The percentage of pigment glands in the samples was determined in all cases by application of the centrifugal and flotation principle (3) to obtain the glands followed by microscopical estimation of the purity of these glands.

Separation

Two differential settling methods for separation or fractionation of the disintegrated slurry were investigated, tank and centrifugal. Proposed flow diagrams illustrating each method are shown in Figures 4 and 5, respectively. With these two diagrams as references the general procedure for investigating the two separation methods can be briefly outlined as follows:

For both methods of settling the prepared cottonseed material (flakes and pulverized meal—either defatted, undefatted, or partially defatted) was mixed with hexane and thoroughly disintegrated. The disintegrated slurry was diluted from a concentration of 50% solids to 12-15% solids by weight in a cone-bottomed, 300-gallon tank.

In tank differential settling this diluted slurry was settled simultaneously with a test sample which was observed to obtain the time for optimum settling of the pigment glands. At the end of the settling time the top fraction containing the fine meal (2 to 40 microns), essentially free of pigment glands, was decanted, and then either centrifuged at 1450 times gravity or filtered in a rotating leaf, pressure filter for recovery of the fine meal. The sediment or bottom fraction, consisting of hulls, coarse meal, and pigment glands, was recovered by filtering and drying. Variations in the procedure were necessary, depending on the feed material used. For example, when using undefatted flakes as a feed, both the top and bottom fractions required further washing for removal of the residual 6 to 8% oil.

In centrifugal differential settling the disintegrated and diluted slurry from the 300-gallon tank was fed to the continuous horizontal centrifuge operating at 62, 110, or 180 times gravity. At these relative centrifugal forces (r.c.f.) the fraction comparable to the bottom fraction in tank settling was discharged at the cake discharge end of the machine, and the fine meal, essentially free of pigment glands, was discharged with the effluent at liquid discharge end of the machine. Purity and yield of available fine meal by this method is dependent upon the operating r.c.f., slurry concentration, slurry feed rate, and pond depth in the machine. Recovery of the fine deglanded meal in the effluent and of the coarse meal fraction, which contains the hulls and pigment glands in the cake discharge, was similar to the recovery of the corresponding fractions from tank settling except that filtration was not required for the coarse fraction because of the low solvent content. Separation data for tank and centrifugal settling are summarized in Tables II and IV and are illustrated in Figures 8, 9, and 10.

The curve in Figure 8 shows the effect of settling time upon the separation of fine meal from the disintegrated slurry by tank differential settling. The percentage of through-300-mesh meal, separated as the fine meal fraction, decreased sharply to about 80% for settling times up to 25 minutes and then decreased slowly until the fine meal separated practically leveled off at 76% and at 75 minutes settling time. This indicates the need for attaining the shortest settling time consistent with the fine meal purity desired and which also allows the maximum yields.

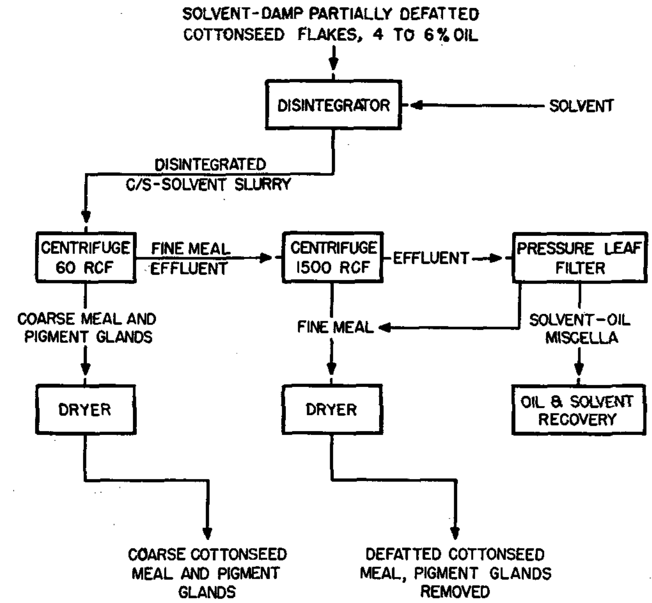

FIG. 5. Fractionation of partially defatted cottonseed flakes using centrifugal differential settling.

The curve in Figure 9 shows the effect of r.c.f. upon the separation of fine meal by centrifugal settling. The percentage of the meal separated decreased sharply from 62 to 110 r.c.f. Figure 10, shows the effect of slurry feed rate upon the separation of fine meal by centrifugal settling. As the feed rate increased, the percentage of fine meal separated increased proportionately. These two figures show that

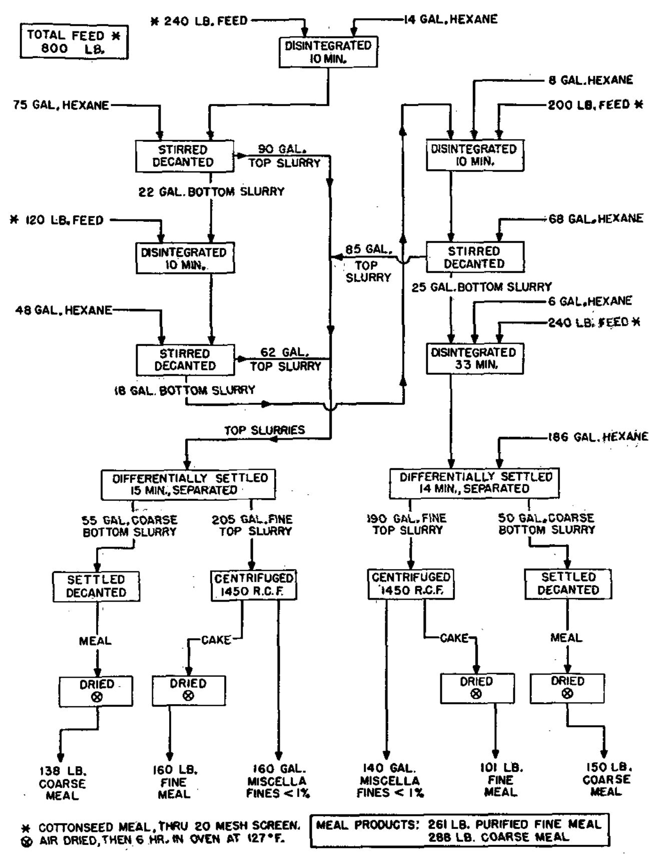

FIG. 6. Flow sheet of Run No. 10.

TABLE I
Disintegration Data

Run No.	Disintegration		Dry Wt. of Cottonseed Material	Disintegrated Meal % by Wt. Through		Hulls
	Time	Method		80-Mesh	300-Mesh	
	Min.		*Lb.*	%	%	%
1	60	Batch	200	67.3	56.3	14.0
2	70	Batch	240	72.1	58.2	8.5
3	60	Batch	350	67.2	54.2	9.6
4	88	Batch	300	80.6	69.3	8.6
5	70	Batch	336	74.7	60.6	8.5
7	70	Batch	188[1]	84.2	58.8	15.6
8a	10 10 30	Intermittent	260 120 69	80.6	67.7	8.0
8b	30	Batch	146[2]	73.2	51.9	19.1
9	60	Batch	340	76.8	62.8	19.9
10	10 10 10 33	Intermittent	240 120 200 240	62.2	47.8	2.0

[1] Bottom or coarse fraction obtained from tank settling of Run No. 6 which was the resettling of bottoms from Run No. 5.
[2] Bottom or coarse fraction obtained from tank settling of the intermittent Run 8a.

final control of the percentage of fine meal separated should be made by varying the feed rate since small changes in r.c.f. will undoubtedly change the percentage of fine meal enormously; however the feed rate will in turn depend upon the fine meal purity required.

Centrifugal settling was compared with tank settling, employing the same slurry. This slurry was first centrifugally settled and sampled, and then the products were remixed for tank settling. A fine meal yield of 92% was obtained for tank settling as compared to 72% obtained for centrifugal settling at r.c.f. of 62; however the percentage of original pigment glands removed was 93.0 and 98.9%, respectively. It is reasonable to believe that at a lower r.c.f., or preferably at a high feed rate, the yield of fine meal by centrifugal settling could approach the 92% of tank settling without lowering the purity of the fine meal beyond that for the tank settling.

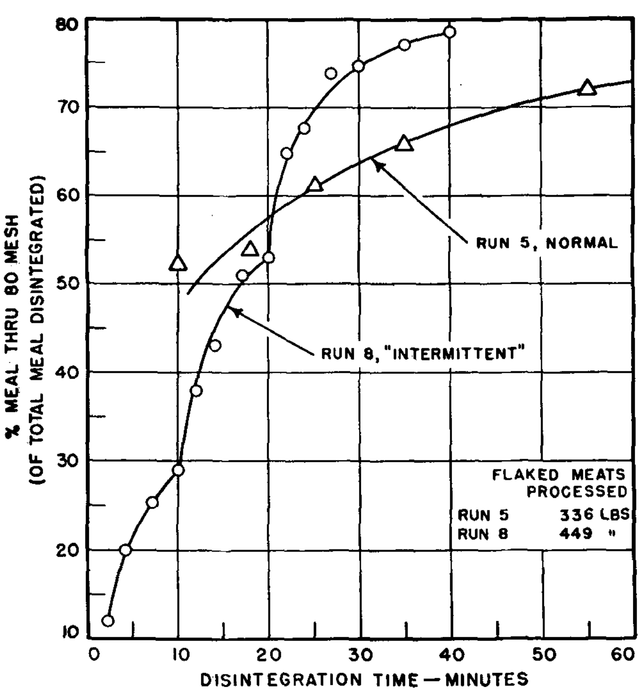

FIG. 7. Comparison of "intermittent" with normal batch disintegration.

The presence of hulls during disintegration tends to break up some of the pigment glands into segments, which settle at a much slower rate than the whole glands. This has a detrimental effect upon the yield of fine meal due to the increased settling time required to obtain a comparable meal essentially free of pigment glands. To illustrate this effect of hulls batch-type Run Nos. 2 and 9 are given in Table IV since the hull contents of the feed materials differed appreciably, 8.5 and 19.9, respectively, and since the purity of the resulting meals were comparable. Although the high hull content Run No. 9 gave better disintegration (percentage through 80- and 300-mesh) the recovery of available fine meal was only 76% as compared to 95% for Run No. 2, because the increased settling time (7.5 times) was necessary to obtain comparable fine meal purity.

Meal Recovery

The fine meals essentially free of pigment glands in the meal-miscella slurries obtained from either tank or centrifugal settling were recovered either by filtration or by centrifugation.

For meal recovery by filtration two rotating-leaf, pressure filters each with a filtering area of 2.6 square feet and a capacity of 1 cubic foot of meal cake, were used. Meal slurries containing 3.5 to 11.8% solids were fed to the filters. The solvent content of the resulting meal cakes varied from 28.7 to 40.4% depending on operating conditions; the filtrates were crystal clear. Figure 11 shows the filtration time versus the rate of filtrate discharge for a slurry containing 11.8% solids. Increase of the slurry feed pressure after four minutes of operation from 30 to 50 lb. per square inch owing to the rapid decrease in the rate of filtrate discharge had the effect of immediately increasing the rate of filtrate discharge, which again decreased fairly rapidly up to the first 20 minutes of operation and then tended to level off at about 0.55 g.p.m.

For meal recovery by centrifugation an 18-x28-inch continuous horizontal bowl centrifugal operating at a r.c.f. of 1450 times gravity was used. Meal slurries containing 3.4 to 11.4% solids were fed to the centrifuge at rates varying from 2.3 to 7.9 g.p.m. The solvent content of the cake discharge varied from 3.0 to 27.9% depending on operating conditions. The effluent or miscella discharge contained 0.2 to 1.5% solids of very fine particle size and could only be filtered with the aid of excessive quantities of filter aid. Detailed data for the recovery of fine meal by centrifugation are given in Table V.

Figure 12 shows the effect of fine meal slurry feed rates and of various meal concentrations upon the percentage of meal discharged with the effluent. At a feed rate of 4 g.p.m. and slurry concentrations of approximately 8% meal (top fraction from separation step) only 6% of the meal was discharged with the effluent. The remaining meal was recovered as cake discharged from the centrifuge. Increasing the feed rate to 8 g.p.m. increased the meal discharged with the effluent to about 10%. Decreasing the meal concentration in the slurry feed to the centrifuge to 3.0% increased the percentage of meal discharged with the effluent and gave a lower meal recovery as cake discharge.

TABLE II

Separation by Tank and Centrifugal Differential Settling

Run No.	Slurry to be settled				Settling Time[1]	Tank Settling				Operating Time	Centrifugal Settling				R.C.F. Used
	Vol.	Total Solids	Meal Through			Total Meal		Original Pigment Glands			Total Meal		Original Pigment Glands		
			80-Mesh	300-Mesh		In Tops	In Bottoms	In Tops	In Bottoms		In Effluent	In Cake	In Effluent	In Cake	
	Gal.	%	%	%	*Min.*	%	%	%	%	*Min.*	%	%	%	%	
1	258	11.2	67.3	56.3	20	47.8	52.2	3.4	96.6						
2	210	13.3	72.1	58.2	10	55.3	44.7	2.7	97.3						
2	74	16.1	38.3	14.6	10	14.9	85.1	0.6	99.4						
3	250	13.8	67.2	54.2	19	43.0	57.0	0.7	99.3						
3	115	18.7	41.1	17.8	10	12.5	87.5	0.1	99.9						
4	250	11.9	80.6	69.3	....	......	......	......	......	30	27.7	72.3	3.1	96.9	180
5	245	13.3	74.7	60.6	10	61.8	38.2	4.7	95.3						
5	250	13.3	74.7	60.6	....	......	......	......	......	35	23.3	76.7	......	......	110
6	168	11.8	63.8	55.4	15	59.6	40.4	6.1	93.9						
7	199	13.8	84.2	58.8	15	60.6	39.4	5.3	94.8						
8a	260	17.2	80.6	67.7	10	62.9	37.1	7.0	93.0						
8a	260	17.2	80.6	67.7	....	......	......	......	......	30	48.5	51.5	1.1	98.9	62
8b	125	15.6	73.2	51.9	9	41.8	58.2	4.9	95.1						
9[2]	250	14.1	76.8	62.8	19	52.9	47.1	......	......						
9	195	8.7	100.0	100.0	19	84.2	15.8	......	......						
9	270	13.1	76.8	62.8	19	29.2	70.8	1.5	98.5						
9	270	13.1	76.8	62.8	40	50.7	49.3	4.6	95.4						
9	270	13.1	76.8	62.8	75	47.5	52.5	2.2	97.8						
10	260	15.6	72.9	50.9	15	54.5	45.5	1.5	98.5						
10	240	15.4	54.5	44.3	14	41.8	58.2	......	......						

[1] Two settling times denotes that settling was conducted in two batches.
[2] Settled products were remixed several times to obtain settling data.

Desolventization

To produce a cottonseed meal suitable for nutritional and industrial uses the solvent-damp meal from either filtration or centrifugation was desolventized by air-drying followed by oven-drying at 125°F. (1) for five hours in a tray dryer. This procedure is to serve as a basis for the development of a commercial process and the selection of equipment that can economically handle large quantities of meal and recover the solvent vapor.

Oil and Solvent Recovery

Miscellas were concentrated in conventional equipment for recovery of the oil and solvent. Oil from one of the fractionation runs was evaluated for color by refining, bleaching, and determining Lovibond colors all according to the official A.O.C.S. methods. Results showed a refined oil color of 35Y—4.23R and a bleached oil color of 20Y—1.66R, both in the prime color range. No further attempt was made to evaluate this phase of the process since satisfactory equipment is available for oil and solvent recovery and since the conditions required for oil concentration to obtain light-colored oils have been previously determined (6).

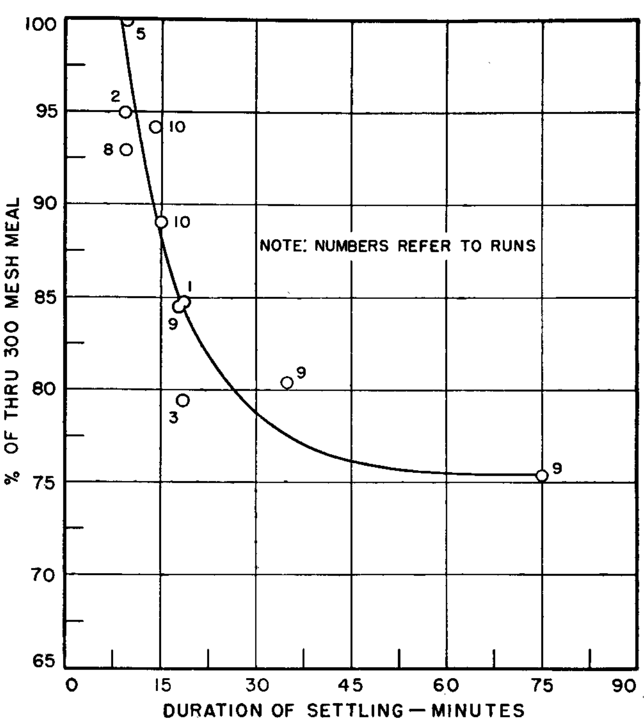

FIG. 8. Effect of settling time upon recovery of fine meal by tank separation.

TABLE III

Average Rate of Disintegration From 0 to 50 Minutes

Run No.	1	2	3	4	5	7	8	9	10
Lb./min. through 80-mesh	1.9	1.8	2.5	2.3	2.6	2.0	4.1	2.5	5.9

Possible Commercial Application

Collectively, the findings gave a good basis for planning a "theoretical" or proposed flow diagram for possible commercial adoption of the fractionation process.

TABLE IV

Fine Meal Separation by Tank Settling in Relation to Settling Time and Hull Content

Run No.	Hulls in Disintegrated Slurry	Meal Through 80	Meal Through 300	Through-300 Meal Recovered as Fines	Fraction of Original Pigment Glands in Fine Meal	Settling Time
	%	%	%	%	%	*Min.*
2	8.5	72.1	58.2	95	2.7	10
9	19.9	76.8	62.8	76	2.2	75

The fractionation process might be used in conjunction with a combination screw-pressing and solvent-extraction plant. Meats as obtained from the cottonseed preparation equipment would be divided into a) fine meats having a high hull content, and

TABLE V

Recovery of Fine Meal by Centrifugation

Run No.	Centrifugation No.	R.C.F.	Pond Depth	Average Feed Rate	Meal in Feed	Meal in Effluent	Hexane in Cake	Total Meal In Effluent	Total Meal In Cake
			Inches	*G.P.M.*	%	%	%	%	%
2	1	1450	15/16	3.8	6.1	0.5	3.0	6.0	94.0
3	1	1450	15/16	7.9	7.8	0.7	27.9	8.1	91.9
	2	1450	15/16	7.8	3.5	0.3	1.0	6.7	93.3
	3	1450	15/16	2.5	0.7	0.2	......	25.0	......
4	1	1450	15/16	{6.8 / 3.5}	3.4	{1.5 / 0.9}	8.2	26.4	73.6
	1	1450	15/16	6.5	7.8	1.2	24.4	13.6	86.4
5	2	1450	15/16	2.3	1.2	0.8	9.0	53.0	47.0
	1	1450	15/16	7.0	7.7	0.8	19.9	8.5	91.5
7	1	1450	7/8	7.1	8.2	1.0	26.9	9.0	91.0
9	1	1450	7/8	6.7	8.4	1.1	24.7	9.3	90.7
10	2	1450	7/8	6.8	11.4	1.5	24.8	9.4	90.6

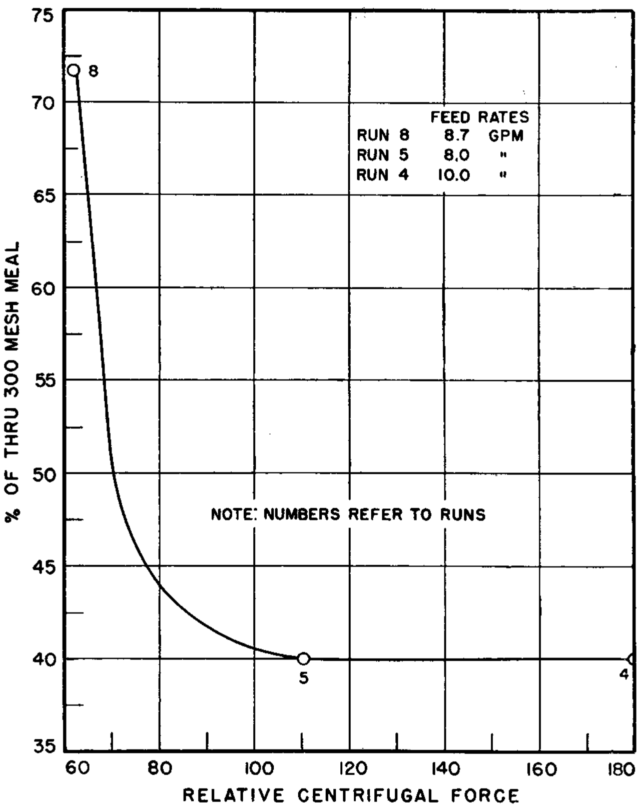

FIG. 9. Effect of r.c.f. upon recovery of fine meal by centrifugal separation.

b) whole meats containing 2% or less hulls, the whole meats representing at least 50% of the total meats. The fine meats would be diverted to the screw-pressing plant for processing and flakes from the whole meats to a solvent-extraction plant.

Extraction of the flakes down to 4 to 6% oil is recommended so that the bulk of the oil can be removed quickly and easily to produce a high-grade oil. Furthermore the capacity of the extraction plant can be increased by at least 50% since removal of the last 4 to 6% oil is the most lengthy and most difficult phase of solvent extraction. This remaining oil will be extracted in the fractionation process; and the meal will be desolventized only once—at the end of the whole process.

The solvent-damp, partially defatted flakes from the solvent-extraction plant will be fed directly to the disintegrator unit of the fractionation plant. As shown in the flow diagram (Figure 4), the disintegrated meal will be fractionated or separated by tank differential settling, the fine meal in the top fraction being recovered by continuous centrifugation at a r.c.f. force of 1500 times gravity followed by filtration of the effluent, and the coarse meal containing hulls and a concentration of pigment glands recovered by filtration and drying. The miscella filtrate can either be used as a feed to the solvent-extraction plant or recovered by conventional methods. Figure 5 shows a flow diagram utilizing the centrifugal settling method at 60 r.c.f. substituted for the tank settling method.

Although pilot-plant data show about 50% recovery as fine meal essentially free of glands, it is believed that by redisintegration and resettling of the bottom fractions and by decreasing operating losses to a minimum, a yield of 70% can be obtained. Also laboratory tests have shown that screening operations between the disintegration and settling steps can further increase the yield of fine meal having a high nutritive value and possible industrial uses. Furthermore by the insertion of a screening operation which will include a screen for the removal of hulls, it may be possible to use the total meats from

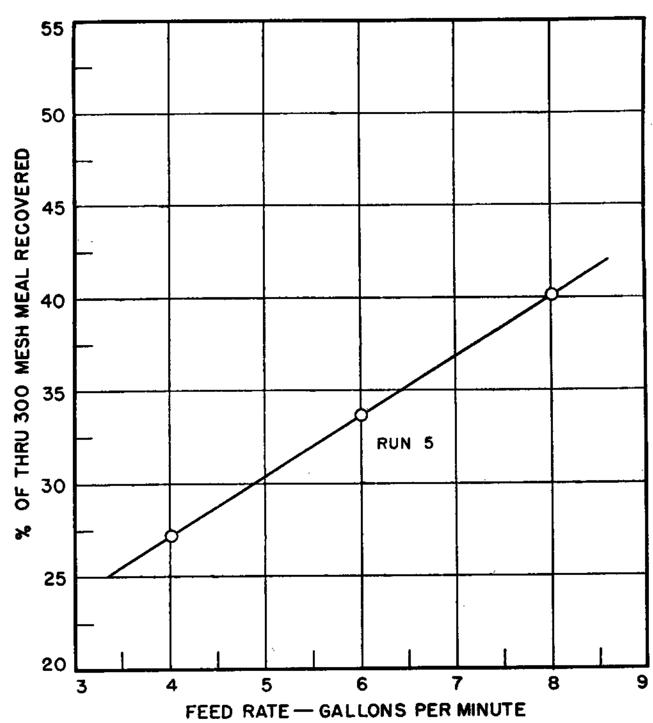

FIG. 10. Effect of feed rate upon recovery of fine meal by centrifugal separation.

the cottonseed preparation equipment in addition to the whole meats fraction as contemplated above. The coarse meal fraction consisting of about 30% of the original meal and containing 8 to 10% pigment glands has many possibilities. This is especially so if a use is found for either gossypol or pigment glands which can be recovered by extraction with proper solvents and by the mixed solvent flotation process, respectively. If no use is found for these materials, the coarse meal can possibly be detoxified and used as cattle feed.

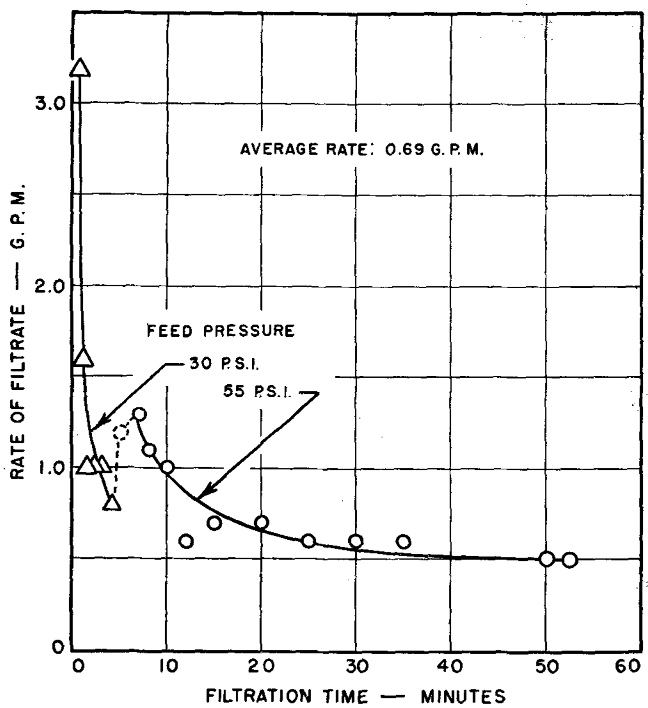

FIG. 11. Filtration of top fraction from differential settling.

Summary and Conclusions

The development of a pilot-plant cottonseed fractionation process has been described, and detailed experimental data regarding the development of the unit operations have been presented. A possible commercial application of the fractionation process has been outlined.

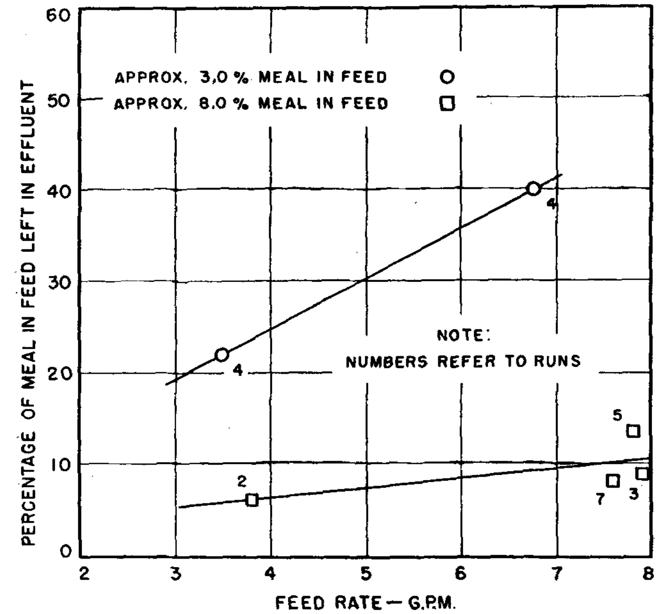

FIG. 12. Effect of feed rate upon amount of meal left in effluent from centrifuge.

In this development a fine meal essentially free of pigment glands and a coarse meal fraction containing a concentration of 8 to 10% pigment glands were produced. Fine deglanded cottonseed meal has a high nutritive value (as reported in other publications), and it is also a potential source of protein for possible industrial utilization.

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