Pilot-Plant Fractionation of Cottonseed. I. Disintegration of Cottonseed Meats[1]

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ABSTRACT

Disintegration of cottonseed meats is an important unit operation in the cottonseed fractionation process. The results of tests utilizing a high-speed, "dissolver-type" impeller for disintegration by liquid shear are presented including curves to show the effect of moisture, hulls, solvents, peripheral speed, etc.

Over 90% of through-80-mesh material can be obtained. Moisture content over 5% appreciably reduced the efficiency and increased power consumption and the viscosity of the slurry. Whole flakes resulted in a finer end product than flakes pre-pulverized in the dry state. It was found that the presence of hulls slightly increased disintegration, power consumption, and viscosity; that higher speeds (up to 6,000 FPM peripheral speed) were more efficient; and that the effect of solvents was small.

Introduction

PRESENT methods of processing cottonseed yield two products, oil and meal, obtained by hydraulic pressing, screw pressing, or by solvent extraction (10). Solvent extraction gives higher yields of oil (11) than do the other methods, and activity in this field is increasing (7, 9, 11, 12, 13, 14, 16). But all three methods produce a cottonseed meal of a grade suitable primarily for use as cattle feed.

The processing of cottonseed, whatever the method, is complicated by a complex pigment system (2) which occurs in rigid, heavily walled glands distributed throughout the seed. While these glands have a high mechanical strength, they tend to rupture in the presence of water (2, 3) and certain solvents. The pigment material thus escapes into the oil and meal. Pigments in the oil necessitate careful control of temperatures during processing to prevent excessive fixation of color (16). Pigments in the meal render it unfit for food or industrial use without further processing. Besides causing discoloration, the pigment material is toxic (1, 5, 6). Heating in the presence of water partially detoxifies the meal but leaves it a discolored mass fit primarily for cattle feed.

In an investigation of these problems a laboratory method (2) was developed for removing whole pigment glands from cottonseed meats, leaving a mixture of meal and hulls and an oil-solvent mixture. In this method the cottonseed meats are flaked, violently agitated in a liquid medium, and separated by flotation in a mixture of inactive solvents (2, 3, 17), the specific gravity of which is adjusted so that the glands tend to float and the meal and hulls to sink, the oil remaining in solvent solution.

A recent publication (17) described the engineering development on a pre-pilot plant scale of a modification of this mixed-solvent flotation process and the initiation of a quantitative investigation of the factors and operations involved in large-scale fractionation. Among the unit operations previously described were: disintegration of the meats to physically remove the meal tissue from the pigment glands; separation of this mixture (2, 17) into pure fractions of glands, fine meal, and hulls; solvent extraction to separate the oil; filtration to remove the miscella from the meal; drying to free the meal of remaining solvent; and evaporation and stripping to remove the solvent from the oil. The engineering problems encountered in applying this fractionation method to cottonseed are similar to those experienced in the industrial application of solvent extraction to cottonseed (10). Each of these unit operations requires engineering study to be evaluated quantitatively with the idea of finding possible alternate solutions of the problems involved and of providing at least one quantitative solution usable on a pilot-plant basis. The unit operation considered here from this viewpoint is disintegration.

Preliminary Investigations

Disintegration of the cottonseed meal to detach the meal tissue from the pigment gland makes possible the separation of the whole gland (2, 17). Microscopic studies, summarized in Table I, indicated that pigment glands in meal of smaller than 80-mesh particle size were relatively free of meal tissue. A screen analysis of glands, given in Table II, showed that 99.7% passed through 80-mesh screen and that only 2.4% passed through the 270-mesh screen. Hence the percentage of through-80-mesh-screen material produced has been taken as the criterion of the efficiency of disintegration.

TABLE I

Microscopic Studies of Fractions of Pulverized Cottonseed Meats

Particle Size		Observations
Through	On	
20 mesh	30 mesh	Average number of pigmtnt glands, 7.4 per particle. Average diameter of particle, 1.28 mm. Particles were of uniform size and did not stick together.
30 mesh	40 mesh	Average number of pigment glands, 2.6 per particle. Average diameter of particle, 0.67 mm. Particles mostly were of uniform size and showed only a slight tendency to stick together.
40 mesh	60 mesh	Average number of pigment glands, 1.24 per particle. Average diameter of particle, 0.51 mm. Particles were of uniform size and showed little tendency to stick together.
60 mesh	80 mesh	Particle sizes were indeterminate because of adhesion to each other. The pigment glands seemed to be relatively free of meal, as they were not imbedded in the particles.
80 mesh	100 mesh	Same as above.

Selection of Equipment

Pre-pilot plant investigations (17) showed that propeller-type mixers were inefficient as disintegrators of cottonseed in solvent slurries although suitable for

[1] Presented at 39th annual meeting of the American Oil Chemists' Society, New Orleans, La., May 4-6, 1948.

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TABLE II

Screen Analysis of Cottonseed Pigment Glands

Mesh	Weight of Meal Fraction	Retained	Through
	Grams	%	%
40	0.005	0.0	100
60	0.033	0.1	99.9
80	0.040	0.2	99.7
100	1.010	4.0	95.7
120	3.445	13.7	82.0
140	7.800	30.9	51.1
170	6.266	24.9	26.2
200	4.075	16.2	10.0
230	1.345	5.3	4.7
270	0.572	2.3	2.4
—270	0.612	2.4	......

qualitative laboratory investigations (2). Consideration of the elementary theory of the propeller (4, 8, 15), in which the revolving helical blades constantly push forward a continuous cylinder of material, led to the conclusion that disintegration was primarily due to the incidental impact of the cutting edge of the propeller against the meal particles and not to the turbulence which was the main effect of the propeller. To increase the effect of this cutting edge a cage-type mixer was adopted. Movement of the slurry through the cage was maintained by propellers mounted above and below the cage. Disintegration efficiency was increased, but the disintegration zone was limited to the immediate vicinity of the propeller cage system.

To extend this disintegration zone a specially designed high-speed, "dissolver-type" impeller having a series of vanes at its periphery was used which set up successive slurry surfaces of constant velocity, each moving at a different rate, thus introducing an interfacial shear acting upon the cottonseed meats. The magnitude of this shear will increase with the viscosity of the slurry.

Such an impeller blade was finally selected for the disintegration unit (Fig. 1) for this series of experiments. A 4-inch impeller blade mounted on a 1-inch steel shaft was driven through V-belts by a 2 h.p. motor and could be raised or lowered by an hydraulic lift arrangement. During runs the impeller blade was 1 inch from the bottom of a 5-gallon cylindrical stainless steel container.

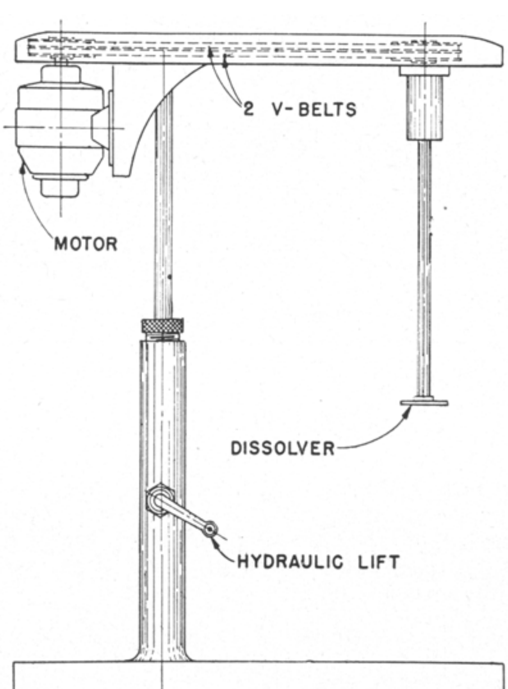

FIG. 1. Experimental dissolver.

This type of disintegration unit is used commercially for effecting rapid solution of gums, resins, and plastics in organic solvents and in a specially designed arrangement for the disintegration and hydration of fiber and pulp. It will be referred to as a dissolver type of disintegrator.

Cottonseed and Solvents

The two lots of prime cottonseed used for this investigation were from the 1945 (Arkansas) and the 1946 (Alabama) crops. The type, source, amounts, and analyses of these seeds are given in Table III and the corresponding data for the cottonseed flaked meats are given in Table IV.

TABLE III

Cottonseed for Disintegration Investigation

Type of Cottonseed	Source	Amount Processed	Moisture	Lipids as Rec'd	F.F.A. of Oil	Nitrogen as Rec'd
		Pounds	%	%	%	%
Prime lot	1945 Arkansas	215	11.91	17.05	1.69	2.85
Prime lot	1946 Alabama	884	12.00	18.90	1.00	3.01

TABLE IV

Flaked Cottonseed Meats for Disintegration Investigation

Type	Source	Quantity	Moisture	Lipids	Nitrogen as Rec'd
		Pounds	%	%	%
Prime lot	1945 Arkansas	70	8.72	35.59	5.19
Prime lot	1946 Alabama	265	8.00	34.0	5.13

The cottonseed was carefully cleaned, delinted, and hulled and the whole meats were air-purified to reduce the hull content to approximately 4% by weight. Sixty to 65% whole meats were obtained; the remaining meats were in the form of fines which contained a considerable amount of hulls. Whole meats only, flaked to a thickness of 0.005 to 0.008 inch, were used as a feed, thus permitting more accurate evaluations and comparisons of the disintegration results obtained. The processing of the cottonseed flaked meats prior to each disintegration run is shown schematically in Figure 2.

The solvents used were a commercial hexane and perchlorethylene which have the following properties:

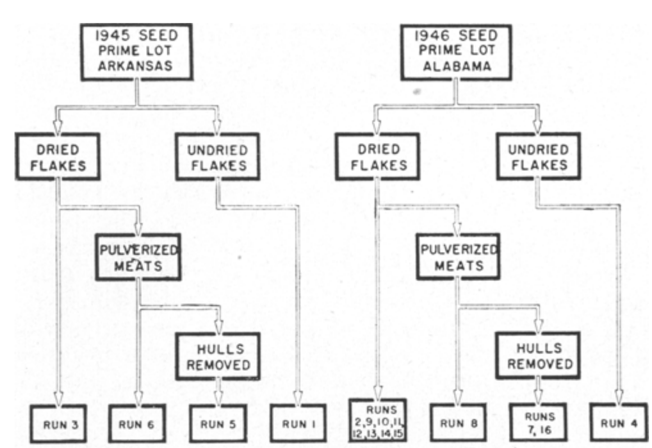

FIG. 2. Processing of cottonseed flaked meats prior to disintegration.

TABLE V

Data for Run No. 5

Time of Run	Sample No.	Ave. Amps.	Ave. Volts	Power Factor	Total K. W. Hrs.	H.P. Motor Input	H.P. Motor Output	Bath Temp. °F.	Slurry Temp. °F.
0′	1	6.00	206	84		2.41	1.99	....	....
5	2	5.50	206	82	0.1418	2.15	1.77	58	99
10	3	6.50	206	84	0.1480	2.61	2.15	....	....
15	4	5.75	206	82	0.1510	2.25	1.86	61	100
20	5	5.50	206	82	0.1325	2.06	1.70	61	106
25	6	5.25	206	82	0.1280	2.06	1.70	60	108
30	7	5.00	206	82	0.1250	1.96	1.62	62	110
40	8	5.00	206	81	0.2420	1.94	1.58	54	103
50	9	5.00	206	81	0.2420	1.94	1.58	54	104
60	10	5.00	206	80	0.2390	1.91	1.56	54	100
					1.5493				

Commercial hexane: gravity at 60°F., 74.4° A.P.I. (sp. gr. 0.678 at 86°F.); boiling range, 140-160°F.; Reid vapor pressure at 100°F., 5.1 pounds per square inch; evaporation residue by weight, 0.0016%; and color, water-white. Perchlorethylene: boiling point at 760 mm., 250.2°F., specific gravity at 68°F., 1.623; refractive index, 1.5044; and evaporation residue by weight, 0.0106%. These solvents are virtually miscible in all proportions and were mixed in the proper quantities to obtain the desired specific gravities.

Development of Procedures

Pilot Plant. Four trial runs were made to determine a practical operating procedure. Six or 7 quarts of solvent and 15 lb. of flaked meats or 5 quarts of solvent and 20 lb. of pulverized meal gave satisfactory operation. An industrial electric analyzer was placed in the disintegrator motor line to give power consumption. The disintegration container was placed in a cold water bath to keep the slurry temperature from rising and causing the pigment glands to rupture.

Sixteen disintegration runs were then made to determine the feasibility of disintegrating cottonseed meats in a solvent slurry and to evaluate the principal factors concerned. In a typical run (No. 5, Table IX) 20 lb. of pulverized, dried cottonseed meal and 5 quarts of a 1.378 sp. gr. mixture of perchlorethylene and commercial hexane were placed in a 5-gallon stainless steel disintegration container. The data for this run are given in Table V.

Laboratory Control. A sample of the cottonseed feed was taken immediately prior to each run for moisture determination.

Samples of slurry were taken periodically throughout each run (for run No. 5, samples Nos. 1 to 10, Table V). The following determinations were made:

Total solids. A 50.0-g. sample of each slurry sample was filtered on a Buchner funnel, washed with commercial hexane, vacuum-dried, and weighed. The percentage of total solids was then calculated.

Percentage of meal through 80-mesh screen. A 50.0-gram sample of each slurry was wet-screened on an 80-mesh screen using commercial hexane. The fraction on the screen was then air-dried and weighed. This gave the weight of the *solids* (meal and hulls) on the 80-mesh screen. In order to calculate the percentage of *meal* on the 80-mesh screen, based on the total *meal* present in the slurry, the following method of estimating the percentage of hulls was devised:

The solids on the 80-mesh screen were dry-screened on 60-, 40- and 30-mesh screens, separating the fraction into several smaller fractions of about the same particle size. As standard samples for comparison, samples of through-60, through-40, through-30-, and on-30-mesh particle size, containing known percentages of hulls by weight, were prepared.

Comparisons were made by at least two trained estimators and the average values recorded. From these values the total weight of hulls was obtained and the percentage of hulls in the original fractions calculated. Table VI gives the percentage in the on 80-mesh fractions obtained by wet screening slurries from the typical run, No. 5.

The percentage of meal through the 80-mesh screen could then be calculated as shown in Table VII.

TABLE VI

Determination of Hull Percentages in Coarser Than 80-Mesh Meal Fractions from Cottonseed Slurry (Run No. 5)

	Est. Hulls	Wt. Fr.	Wt. Hulls	Est. Hulls	Wt. Fr.	Wt. Hulls	Est. Hulls	Wt. Fr.	Wt. Hulls	Est. Hulls	Wt. Fr.	Wt. Hulls
Time (minutes)		0			5			10			15	
	%	*Grams*	*Grams*	%	*Grams*	*Grams*	%	*Grams*	*Grams*	%	*Grams*	*Grams*
On 40 mesh	18	2.71	0.49	60	0.02	0.01	....	......	......	....	......	......
Through 40 mesh	9	2.15	0.19	22	1.82	0.40	22	1.34	0.29	25	1.27	0.32
Through 60 mesh	....	......	......	13	2.01	0.26	11	1.86	0.20	8	1.49	0.12
Totals		4.86	0.68		3.85	0.67		3.20	0.49		2.76	0.44
Ave. % Hulls		14			18			15			16	
Time		20′			25′			30′			40′	
On 40 mesh	....	......	......	....	......	......	....	......	......	....	......	......
Through 40 mesh	23	1.02	0.23	27	0.91	0.25	28	0.86	0.24	28	0.70	0.20
Through 60 mesh	10	1.64	0.16	12	1.36	0.16	14	1.34	0.19	13	1.20	0.16
Totals		2.66	0.39		2.27	0.41		2.20	0.43		1.90	0.36
Ave. % Hulls		15			18			20			19	
Time		50′			60′							
On 40 mesh	....	......	......	....	......	......						
Through 40 mesh	28	0.62	0.17	30	0.51	0.15						
Through 60 mesh	14	1.15	0.16	14	1.01	0.14						
Totals		1.77	0.33		1.52	0.29						
Ave. % Hulls		19			19							

TABLE VII

Progress of Disintegration of Cottonsed Meal (Run No. 5)

Time Sample Taken (minutes)	0	5	10	15	20	25	30	40	50	60
Wt. slurry, grams	50.0	50.0	50.0 [1]	50.0	50.0	50.0	50.0	50.0	50.0	50.0
Wt. total solids, defatted, grams	18.255	18.370	18.435 [1]	18.455	18.52	18.585	18.650	18.780	18.915	19.045
Wt. solids on 80 mesh, grams	4.865	3.895	3.235 [1]	2.835	2.765	2.320	2.280	1.955	1.820	1.635
Est. % hulls on 80 mesh	14	18	15 [1]	16	15	18	20	19	19	19
Wt. hulls, grams on 80-mesh	0.681	0.701	0.485 [2]	0.454	0.415	0.418	0.456	0.372	0.346	0.312
Wt. hulls in solids, grams	0.681	0.681	0.681 [3]	0.681	0.681	0.681	0.681	0.681	0.681	0.681
Wt. meal in solids, grams	17.574	17.689	17.754 [4]	17.774	17.839	17.904	17.969	18.099	18.234	18.364
Wt. meal on 80-mesh, grams	4.184	3.194	2.750 [5]	2.381	2.350	1.902	1.824	1.583	1.474	1.323
Per cent of total meal on 80-mesh	23.81	18.06	15.49 [6]	13.40	13.17	10.62	10.15	8.75	8.08	7.20
Per cent of total meal through 80-mesh	76.19	81.94	84.51 [7]	86.60	86.83	89.38	89.85	91.25	91.92	92.80

[1] Using 10-minute sample as an example, the weight slurry, 50.0-gram sample taken; weight solids, 18.435 grams, determined experimentally; weight solids on 80 mesh, 3.235 grams determined experimentally; and estimated per cent hulls on 80 mesh, 15 determined experimentally.

[2] $\frac{15 \times 3.235}{100} = 0.485$ grams.

[3] This is the value for the weight hulls on 80 mesh determined for the initial sample. This value is assumed to be the weight hulls in solids throughout the run.

[4] $18.435 - 0.681 = 17.754$ grams.

[5] $3.235 - 0.485 = 2.750$ grams.

[6] $\frac{2.750}{17.754} \times 100 = 15.49\%$.

[7] $100 - 15.49 = 84.51\%$.

Viscosity. Viscosity determinations were made on each sample of slurry using a Stormer viscosimeter with forked spindle. In Table VIII are given the determinations made on samples of slurry from run No. 5.

TABLE VIII

Viscosity of Slurry, Run No. 5

Sample Number	Mixing Time	Viscosimeter Data	
		Temp. of Slurry	Viscosity
	Minutes	°C.	*Poises*
1	0	28	49.4 [1]
2	5	28	7.6
3	10	28	3.3
4	15	28	3.7
5	20	28	2.5
6	25	28	2.5
7	30	28	2.9
8	40	28	2.5
9	50	28	3.3
10	60	28	2.5

[1] Outside of calibration range of viscosimeter.

Experimental Results

The factors considered in evaluating the results of 16 disintegration runs were the efficiency or extent of disintegration, the power consumed, and the viscosity of the slurry, the viscosity being important in connection with the other operations in the fractionation process. These three factors were studied in relation to the moisture content of the meal; the physical state of the meal feed (flaked meats or pulverized meal, with or without hulls); the peripheral speed of the impeller; and the nature of the solvent. A summary of the disintegration run data is given in Table IX. Table X gives the particular size distribution of the solids in slurries from runs Nos. 8 and 9 after 30 minutes' disintegration.

TABLE X

Particle Size Distribution of Solids in Slurries After 30 Minutes Disintegration

Mesh	Run 8		Run 9	
	Solids Retained	Solids Through	Solids Retained	Solids Through
	%	%	%	%
40	6.92	93.08	6.39	93.61
60	6.42	86.66	3.68	89.93
80	4.40	82.26	3.36	86.57
100	2.78	79.48	2.90	83.67
120	2.78	76.70	2.78	80.89
140	3.08	73.62	3.29	77.60
170	2.58	71.04	2.58	75.02
200	1.57	69.47	1.16	73.86
230	1.92	67.55	2.65	71.21
270	0.71	66.84	0.65	70.56
300	0.20	66.64	0.52	70.04

Microscopic examination of pigment glands produced in runs (Nos. 9 to 13) where the impeller speed varied from 2,700 to 7,200 showed that the pigment glands were sufficiently strong mechanically to withstand disintegration under these conditions.

The bar chart in Figure 3 was constructed to give an overall comparison of the runs. This shows for each run the kw. hrs. required per pound of cotton-

TABLE IX

Summary of Disintegration Runs

Run No.	Peripheral Speed [1]	Solvent			Cottonseed Meats				Final Slurry						Time of Run Mins.	K.W. Hrs. Per Lb. Feed	80-Mesh Mat. Prod. % by Wt.
		Type [2]	Sp. Gr.	Vol. Qts.	Lot [3]	Type	Moisture % by Wt.	Lbs.	% Oil by Wt.	% Moisture by Wt.	% Solids by Wt.		% Solv't by Wt.	Viscosity Poises			
											Calculated	Lab. Det.					
1	6000	A	1.485	7	C	Flake	8.0	15	14.7	3.27	23.0	27.6	59.2	2.7	60	0.116	89.52
2	6000	A	1.475	7	D	Flake	3.2	15	14.7	1.31	25.0	29.0	59.2	1.2	60	0.096	97.89
3	6000	A	1.475	7	C	Flake	4.0	15	15.3	1.64	23.9	28.3	59.2	1.1	60	0.097	96.39
4	6000	A	1.475	7	D	Flake	9.0	15	13.8	3.68	23.4	26.7	59.2	28.0	60	0.136	89.22
5	6000	A	1.460	5	C	Pulv.[4]	2.4	20	21.6	1.36	33.8	38.1	43.4	2.5	60	0.062	92.80
6	6000	A	1.460	5	C	Pulv.	2.8	20	21.5	1.59	33.6	38.5	43.4	3.3	60	0.070	93.48
7	6000	A	1.460	5	D	Pulv.[4]	3.2	20	20.3	1.82	34.3	37.9	43.4	2.9	60	0.083	89.55
8	6000	A	1.460	5	D	Pulv.	2.2	20	20.5	1.25	34.6	39.5	43.4	3.8	60	0.085	94.43
9	6000	A	1.460	6	D	Flake	2.6	15	16.3	1.17	27.7	31.0	55.0	1.4	30	0.051	92.64
10	4800	A	1.460	6	D	Flake	2.6	15	16.3	1.17	27.7	31.9	55.0	1.8	30	0.034	86.88
11	3600	A	1.460	6	D	Flake	2.6	15	16.3	1.17	27.7	30.3	55.0	3.3	30	0.022	79.71
12	2700	A	1.460	6	D	Flake	2.6	15	16.3	1.17	27.7	31.2	55.0	1.8	30	0.016	71.72
13	7200	A	1.460	6	D	Flake	2.6	15	16.3	1.17	27.7	30.2	55.0	1.4	30	0.059	92.45
14	6000	A	1.450	6	D	Flake	0.3	15	16.7	0.135	27.7	30.6	55.0	1.4	30	0.051	93.22
15	6000	B	0.672	6	D	Flake	3.8	15	22.8	2.44	38.8	46.5	36.0	2.2	30	0.039	87.89
16	7200	A	1.455	5	D	Pulv.[4]	3.6	20	20.3	2.04	34.5	37.5	43.2	3.3	30	0.052	86.52

[1] 4" impeller, 1" from bottom of container. Speed in ft./min.

[2] Type A—Mixture C_2Cl_4 and commercial hexane. Type B—Commercial hexane.

[3] Lot C—Prime lot, 1945 Arkansas. Lot D—Prime lot, 1946 Alabama.

[4] Screened through-30-mesh.

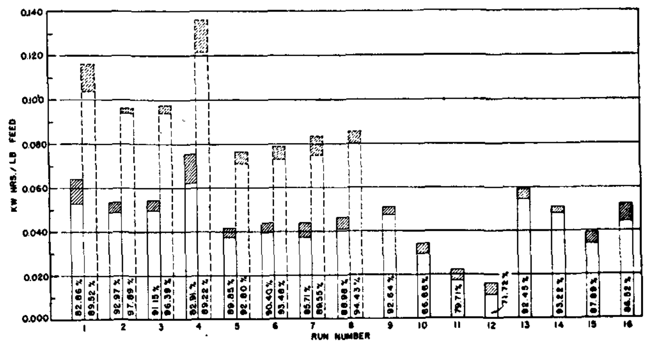

FIG. 3. Energy required per pound of cottonseed meats to produce indicated disintegration.

Disintegration runs 1-16.

1. Runs 1 to 8 were for 60 min. duration. Runs 9 to 16 were for 30 min. duration.
2. Solid lines represent data taken at 30 min. Dashed lines represent data taken at 60 min.
3. Figure in unshaded portion is percentage of 80-mesh material produced. Shaded portion represents material over 80-mesh.

seed meats to produce the disintegration indicated. For runs of 30 minutes' duration this varied from a low of 0.016 (run No. 12) where 71.72% of through-80-mesh material was produced to a high of 0.136 (run No. 4) where 89.22% of through-80-mesh material was produced. On the same basis this varied in the best six runs from a low of 0.051 (run No. 14) where 93.22% of through-80-mesh material was produced to a high of 0.059 (run No. 13) where 92.45% of through-80-mesh material was produced. Increasing the duration of the runs from 30 to 60 minutes increased the amount of through-80-mesh material produced by 2.95% (run No. 5) to 6.66% (run No. 1) and increased the energy consumption by 0.021 to 0.061 kw. hrs./lb. feed.

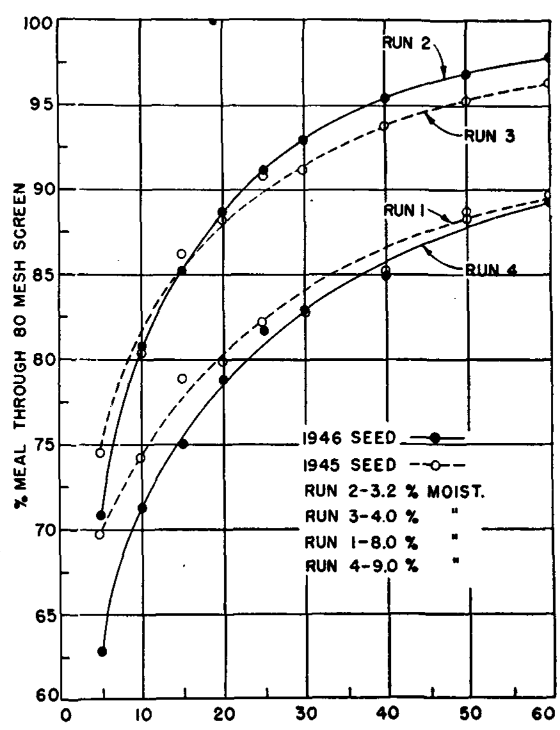

FIG. 4. Effect of moisture upon disintegration of cottonseed meats in solvent slurries.

Disintegration runs 1-4.

The detailed studies of the three factors considered are outlined below.

Disintegration. Figure 4 depicts the effect of moisture upon the disintegration of cottonseed meats. It clearly shows that increasing the moisture content of the meats decreased the production of meal of through-80-mesh particle size — low moisture (3.2-4.0%) meats produced 97% while high moisture (8-9%) meats produced 89.5%.

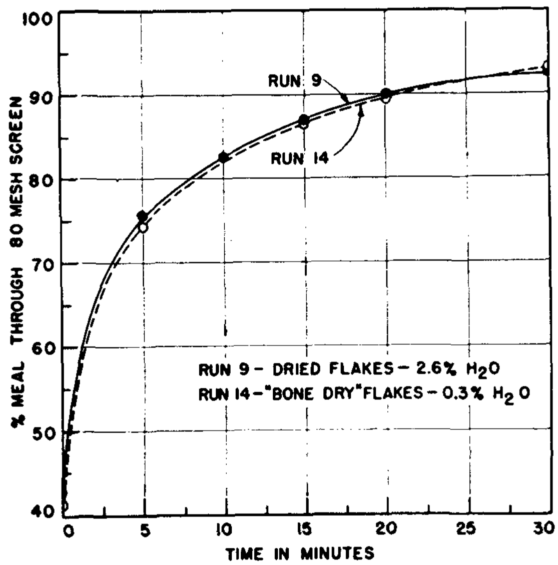

FIG. 5. Comparison of disintegrating characteristics of dried and bone-dry cottonseed flakes in solvent slurries.

Disintegration runs 9 and 14.

Figure 5 illustrates the comparison of disintegrating characteristics of dried and bone dry cottonseed flakes in solvent slurries. Reducing the moisture content below 2.6% had little effect upon the amount of through-80-mesh material produced.

Figure 6 shows the effect of using whole cottonseed flakes on pulverized meal as feed. Dry-pulverizing

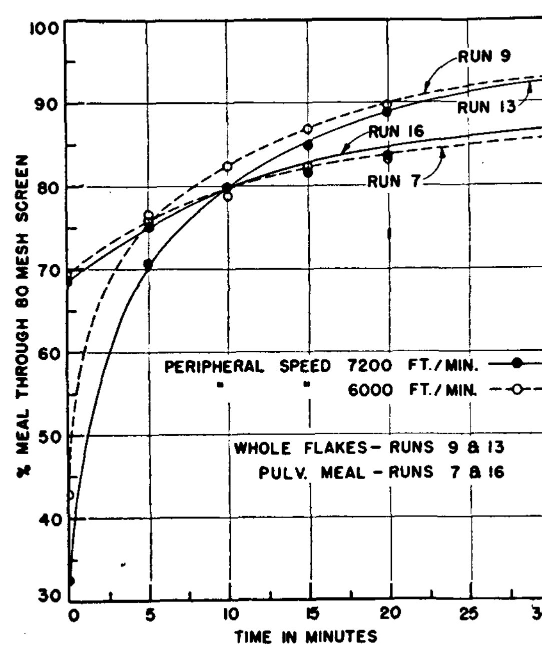

FIG. 6. Effect of using whole cottonseed flakes or pulverized cottonseed meal as feed.

Disintegration runs 7, 9, 13, and 16.

the flakes prior to disintegrating in solvent slurry reduced the amount of through-80-mesh material finally produced.

Figure 7, depicting the effect of hulls upon disintegration of cottonseed meats in solvent slurries, shows that the removal of hulls before disintegrating pulverized meats in solvent slurry slightly decreased the amount of through-80-mesh material produced.

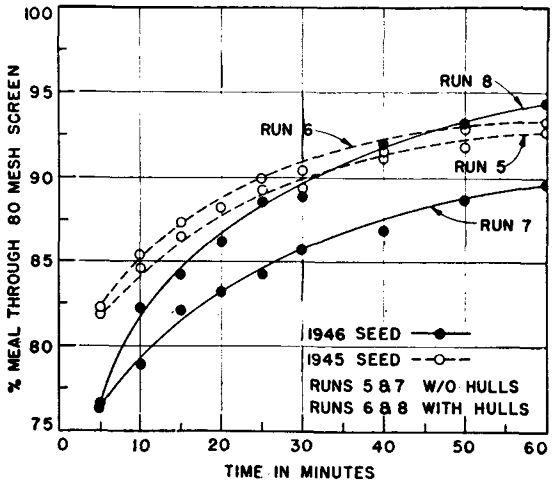

FIG. 7. Effect of hulls upon disintegration of cottonseed meats in solvent slurries.
Disintegration runs 5-8.

Figure 8 illustrates the effect of solvent upon disintegration of cottonseed meats in solvent slurries.

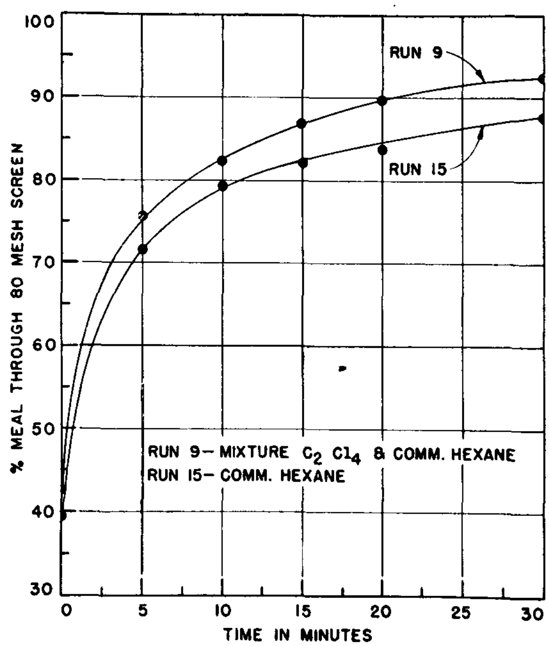

FIG. 8. Effect of solvent upon disintegration of cottonseed meats in solvent slurries.
Disintegration runs *9* and *15*.

With the heavier solvent (a mixture of perchlorethylene and commercial hexane) a slightly larger ($4\frac{1}{2}\%$) amount of through-80-mesh material was produced than with the lighter solvent (commercial hexane).

Figure 9 illustrates the effect of varying peripheral speed upon disintegration of cottonseed meats in solvent slurries. The amount of through-80-mesh material produced increased with increase in peripheral

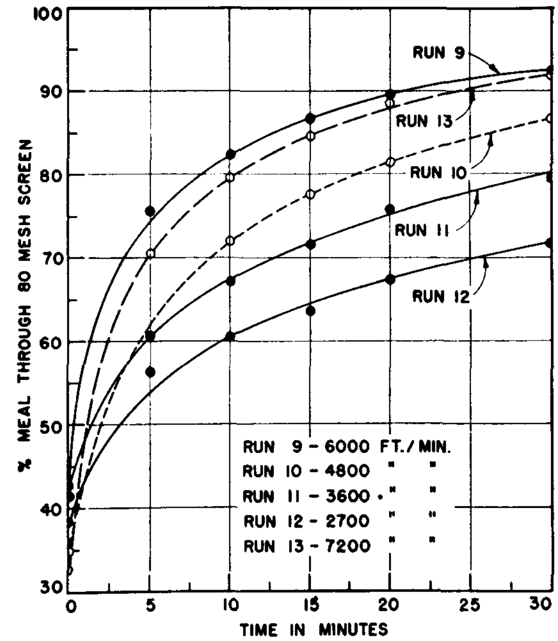

FIG. 9. Effect of peripheral speed upon disintegration of cottonseed meats in solvent slurries.
Disintegration runs 9-13.

speed. In run No. 13 vortexing occurred, changing conditions in the disintegration vessel and lowering the amount of through-80-mesh material produced.

Power Consumption. Figure 10 shows the effect of moisture upon power consumption. Power consumption increased substantially with increase in moisture content of the meats.

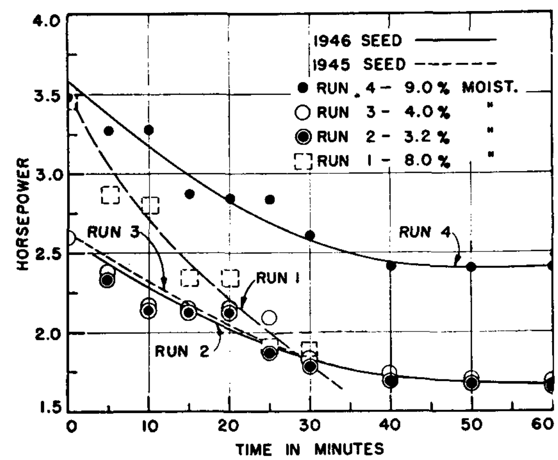

FIG. 10. Effect of moisture upon power consumption.
Disintegration runs 1-4.

Figure 11 illustrates the comparison of power consumption of runs made with bone dry and dried cottonseed flakes. It shows slight increase in power consumption using the bone dry flakes.

Figure 12 depicts the effect upon power consumption of using whole cottonseed flakes or pulverized meal as feed. Under comparable conditions the disintegration of the whole flakes required more power.

Figure 13 shows the effect of hulls upon power consumption. Under comparable conditions the presence of hulls slightly increases the power consumption.

Figure 14 depicts the effect of solvent upon power consumption. The heavier solvent (a mixture of per-

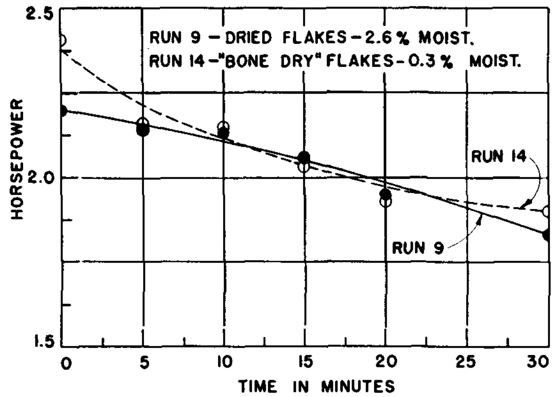

FIG. 11. Comparison of power consumption of runs made with bone-dry and dried cottonseed flakes.
Disintegration runs 9 and 14.

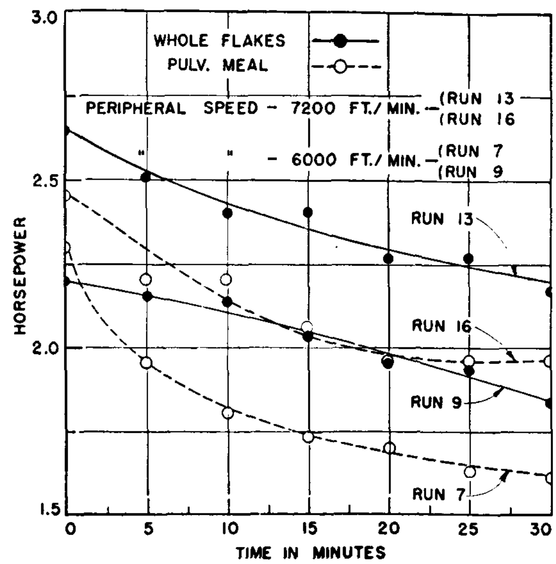

FIG. 12. Effect upon power consumption of using whole cottonseed flakes or pulverized cottonseed meal as feed.
Disintegration runs 7, 9, 13, and 16.

chlorethylene and commercial hexane) required considerably more power than the lighter solvent (commercial hexane).

Figure 15, depicting the effect upon power consumption of varying peripheral speed, shows that power consumption increased with increase in peripheral speed.

Viscosity of Slurry. Figure 16, illustrating the effect of moisture upon viscosity of cottonseed slurries, shows that high moisture content greatly increased the initial viscosity of the slurry. The viscosity decreased during the run as the oil was extracted from the meats and as the meats were disintegrated finally reaching a constant value. For runs made with seed of low moisture content (3-4%) the initial viscosity was lower and the final constant viscosity reached early in the run.

Figure 17 depicts the comparison of viscosities of slurries from runs made with bone dry and dried cottonseed flakes. The viscosity of the "bone dry" run had a constant value from the first (five minute) sample and the viscosity of the "dried" run was initially higher but quickly became the same as the viscosity of the "bone dry" run.

Figure 18 shows the effect of hulls upon viscosity

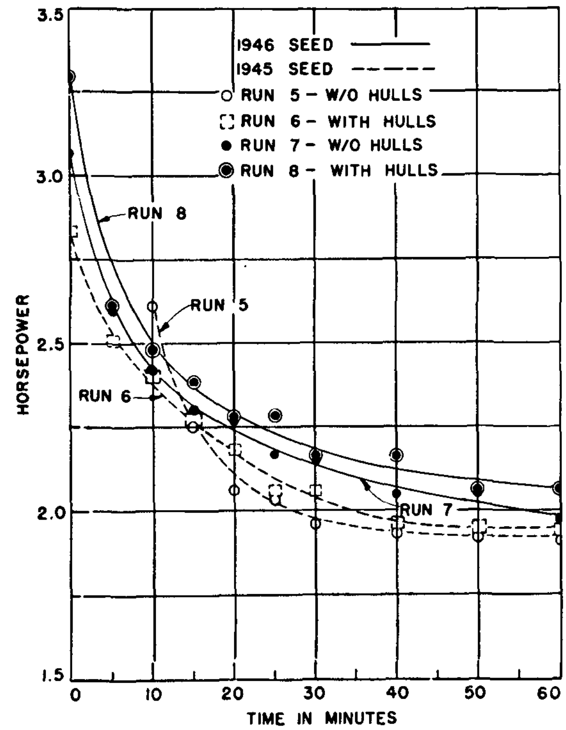

FIG. 13. Effect of hulls upon power consumption.
Disintegration runs 5-8.

of cottonseed slurries. The presence of hulls increased the viscosity of the slurry.

Figure 19 illustrates the effect of solvent upon viscosity of cottonseed slurry. The viscosity of the slurry from the run using the lighter solvent (commercial hexane) was less than that using the heavier solvent (mixture of perchorethylene and commercial hexane).

Figure 20, illustrating the effect of peripheral speed upon viscosity of cottonseed slurry, shows that the higher the peripheral speed the sooner the constant limiting viscosity is reached for the particular slurry.

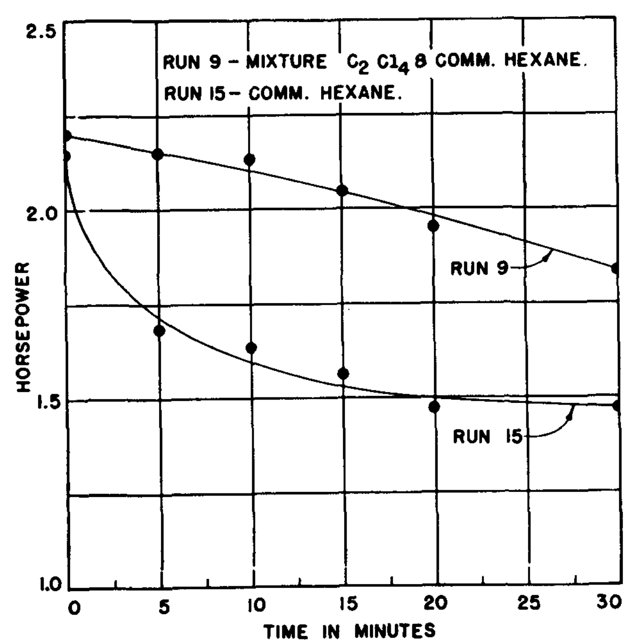

FIG. 14. Effect of solvent upon power consumption.
Disintegration runs 9 and 15.

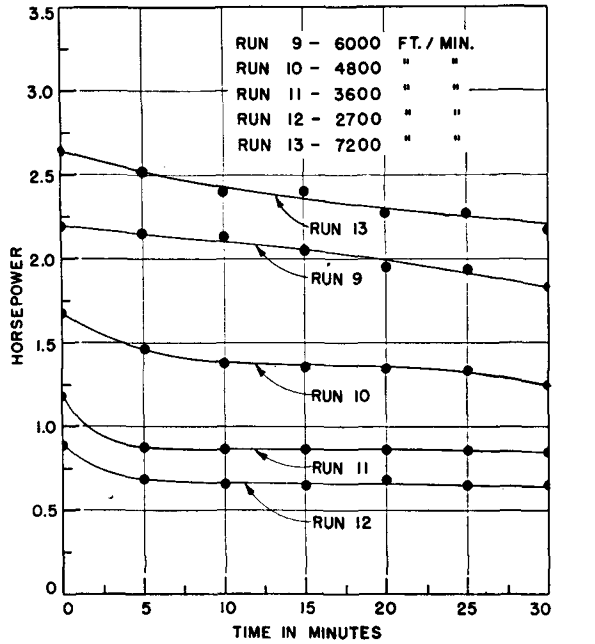

FIG. 15. Effect upon power consumption of peripheral speed. Disintegration runs 9-13.

Conclusions

1. The type of disintegration unit described, utilizing the liquid shear produced by a high-speed impeller, is satisfactory for the disintegration of cottonseed meats in solvent slurries.

2. The moisture content of the cottonseed meats was found to be very important. Moisture contents ranging above 5% materially reduced the efficiency of disintegration and increased the power consumption and viscosity of the slurry. However, bone dry

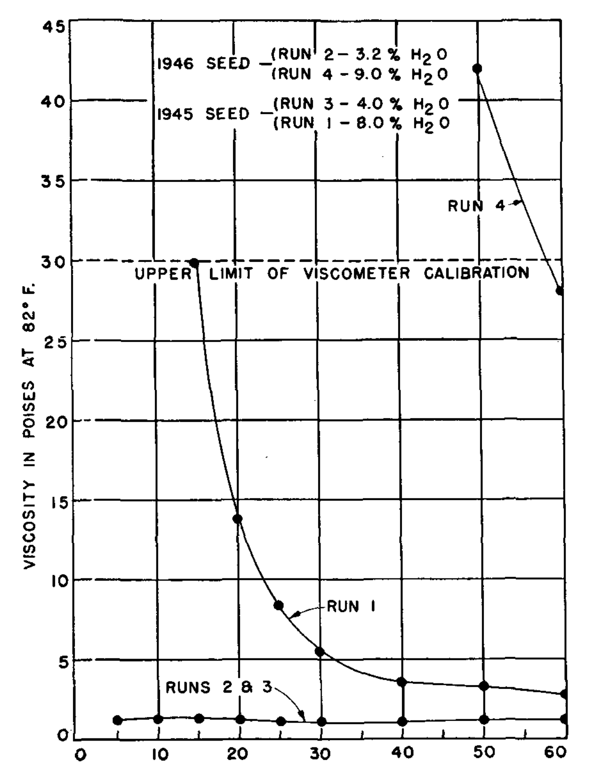

FIG. 16. Effect of moisture upon viscosity of cottonseed slurries.
Disintegration runs 1-4.

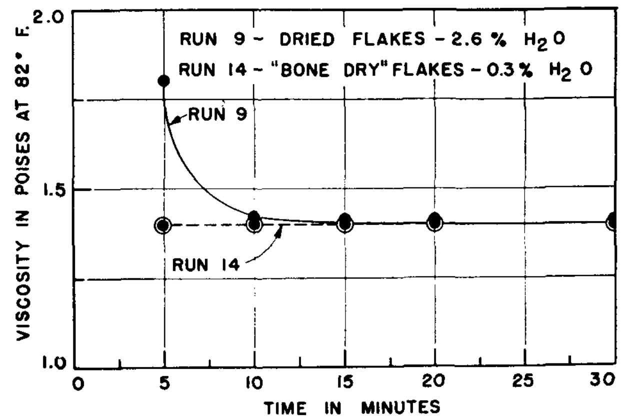

FIG. 17. Comparison of viscosities of slurries from runs made with bone-dry and dried cottonseed flakes.
Disintegration runs 9 and 14.

flakes (moisture content less than 1%) had little or no advantage over normally dried flakes (moisture content 2½ to 4%).

3. Whole flakes gave a finer end product when disintegrated in solvent slurry than meal which had been pre-pulverized in the dry state. This was probably

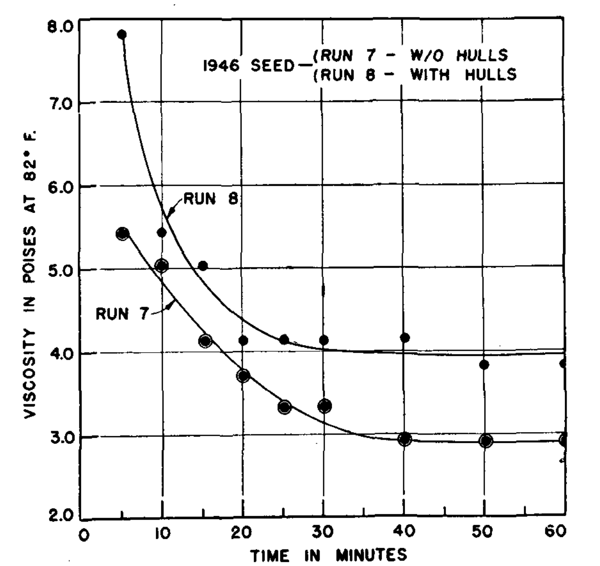

FIG. 18. Effect of hulls upon viscosity of cottonseed slurries.
Disintegration runs 7 and 8.

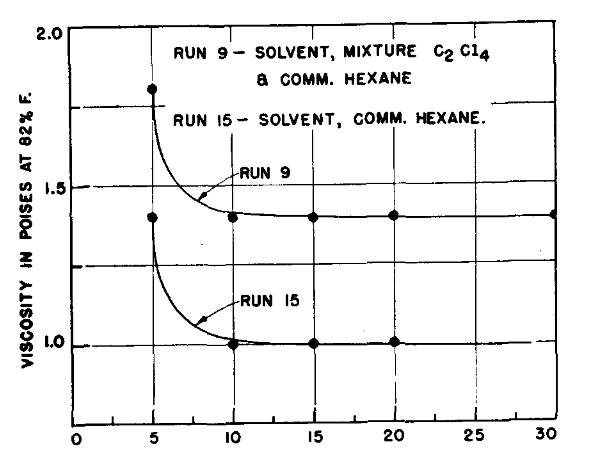

FIG. 19. Effect of solvent upon viscosity of cottonseed slurry.
Disintegration runs 9 and 15.

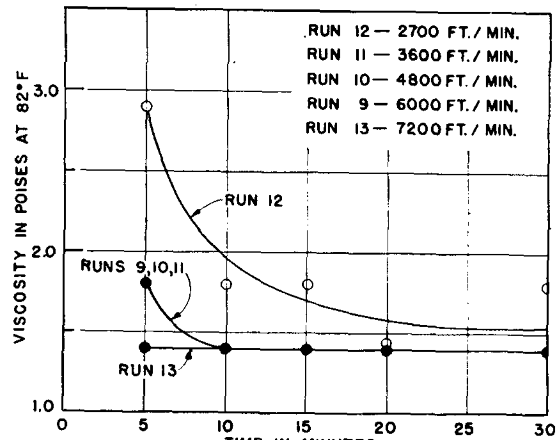

FIG. 20. Effect of peripheral speed upon viscosity of cottonseed slurry.
Disintegration runs 9-13.

due to hardening of the meal particles during prepulverizing.

4. The presence of hulls slightly increased the disintegration, power consumption, and viscosity.

5. The effect of solvents was small; a slightly greater disintegration, power consumption and viscosity being obtained with the heavier solvent.

6. The higher peripheral speeds were the more efficient, the disintegration and power consumption increasing with increase in speed. The higher speeds also caused the limiting value of the slurry viscosity to be reached more quickly owing to the quicker extraction of the oil.

7. In disintegrating the cottonseed meats to less than 80-mesh-particle size, two-thirds of the meats were reduced to less than 300-mesh particle size.

8. The high mechanical strength of pigment glands was confirmed.

Acknowledgment

The authors wish to express their appreciation to V. E. Maurer of the Cowles Company for his cooperation in the pilot-plant disintegration runs.

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Corrosion Tests in Organic Sulfations and Sulfonations

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SULFATION and sulfonation are among the processes used to change the properties of organic materials such as to introduce greater solubility or to make a hydrocarbon more reactive for further synthesis. These processes are used particularly in the preparation of surface-active materials including detergents, emulsifiers, wetting agents, and penetrants from animal and vegetable oils and from fatty alcohols, aromatics, and other hydrocarbons. The agents commonly employed for sulfation or sulfonation are various strengths of SO_3 in water, from 66° Bé sulfuric acid, or even weaker, to strong oleums. The strength of acid used and temperature will depend usually upon the degree of saturation of the hydrocarbon, the location to which the attachment is to be directed and other factors. In some cases chlorosulfonic acid and even bisulfite solutions are used as sulfonating agents.

The selection of corrosion-resisting materials for construction of reactors and for washing and neutralization equipment depends chiefly upon the temperature used and upon the amount of dilution of sulfuric acid which occurs during processing. The purpose of this paper is to present the results of some plant and laboratory corrosion tests in organic sulfations and sulfonations under a variety of operating conditions. This will serve as an indication of the metals and alloys which should be considered for the construction of processing equipment. The information given concerning the test conditions in the plant corrosion tests is that provided by the plant where the tests were made, or as much of it as can be published. Because of the scope and complexity of this field no attempt has been made to deal with the chemistry of the processes referred to except in a general way.

The corrosion tests reported here were made with the spool-type specimen holder illustrated in Fig. 1. This method of testing is substantially in accord with A.S.T.M. Recommended Practice for Conducting Plant Corrosion Tests, A224-41.[1] Briefly, the assembly consists of previously cleaned and weighed specimens of the several metals and alloys to be tested, mounted on the spool-type holder with nonmetallic parts of porcelain to separate and insulate the specimens from each other and from the metallic parts of the holder. Two similar specimens of each material were included on each spool. The complete test assemblies were fastened firmly in place in the desired test locations in operating plant equipment and allowed to remain for sufficient lengths of time to give reliable indications of corrosion behavior. Each of the test specimens used had an exposed area of 0.5 sq. dm.

[1]A.S.T.M. Standards, Part I, p. 522, 1944.