(—29, —18, 22, and 38°C.). A modification of the procedure was necessary for butterfat. In order to obtain a red color it was necessary to heat the samples before applying the TBA extraction procedure. The results shown in Table I were obtained from samples preheated in a boiling water bath for 60 minutes. The 530 mμ absorbancy value of the 100°F.(38°C.) sample increased 560% after such treatment whereas the corresponding carbonyl value increased 11%. The high free fatty acid content of this sample is apparently not responsible for its high TBA value since tests on a sample of butyric acid (Technical) were negative. Iodometric peroxide and free fatty acid determinations (1) were also made on these samples.

TABLE I

TBA Values, Peroxide Values, and Free Fatty Acid Values of Butterfat, Stored Approximately 2 Years at —20, 0, 72, and 100°F. (—29, —18, 22, and 38°C.)

Storage temperature of sample (°F.)	TBA values[a]	Peroxide values	Free fatty acid values as oleic
		m.e./kg.	%
—20	0.29	2.00	0.67
0	0.30	1.33	0.60
72	0.51	4.32	0.69
100	0.87	3.33	1.42

[a] Samples were preheated in a boiling water bath for 60 min. before applying the TBA test.

It may be seen that the TBA values were more directly related to storage temperatures than peroxide values. Sensory evaluations made by an expert panel of laboratory personnel showed a direct relationship between TBA values and off-tastes and odors.

Butterfat Oxidized by Ultraviolet Irradiation

Butterfat was irradiated by exposure to the ultraviolet rays of a Mineralight lamp. In contrast to the stored butterfat samples no preheating of the fat was necessary to obtain a red color upon subsequent application of the TBA extraction procedures. Although the test solutions were turbid, they were easily clarified by centrifugation. Values thus obtained were directly related to iodometric peroxide values.

When the butyric acid sample, which had given a negative TBA test, was exposed to ultra-violet irradiation for a 19-hr. period, it also reacted positively to TBA. The absorption spectrum was similar to that obtained from oxidized fat samples. The oxidation of butyric acid by photochemical means has been reported (5, 12).

Summary

An evaluation of thiobarbituric acid (TBA) as an agent for the measurement of fat oxidation was made by the application of several empirical procedures to animal and vegetable fats. An extraction procedure was used for removing the products of oxidation. The reaction with TBA was conducted in a boiling water bath to produce a red color, which was then estimated spectrophotometrically.

Fats stored at —20, 0, 72, and 100°F.(—29, —18, 22, and 38°C.) and fats oxidized by the active oxygen method (A.O.M.) and by ultraviolet irradiation were examined. It was found that the TBA test might be of value in following the course of oxidation of cottonseed oil and soybean oil in the A.O.M. apparatus. Higher TBA values were obtained for soybean oil than cottonseed oil at comparable peroxide values. This is of interest because of the greater tendency of soybean oil to develop oxidized flavors. The volatile reaction products of oxidation were collected in toluene, and a comparison of the TBA values at comparable peroxide values of lard, cottonseed and soybean oils showed that the soybean oil volatiles developed the greatest intensity of color.

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Pilot-Plant Application of Filtration-Extraction to Soybeans[1]

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PILOT-PLANT development of a new solvent process called filtration-extraction for extracting cottonseed and rice bran has been reported in previous papers (1, 5) by this laboratory. Extension of the process on a pilot-plant scale to the processing of soybeans is warranted by the fact that during the 1951-52 crushing season there were 58 mills outside the North Central States that processed soybeans and other oilseeds (9) and most of these were cottonseed oil mills that crushed soybeans after their supply of cottonseed had been exhausted. Since practically all of these were small to medium-sized mills, application of the process to soybeans is further warranted because the new process holds definite promise of cost feasibility (6) for low capacity installations, together with versatility, and may for the first time bring direct solvent extraction within the economic reach of the small processor who can predicate his installation on a longer crushing season by processing rice bran,

[1] Presented at the 44th Annual Meeting of the American Oil Chemists' Society, New Orleans, La., May 4-6, 1953.
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peanuts, flaxseed, and other available oleaginous crops in various combinations or sequences.

The filtration-extraction process differs from current direct extraction systems in that the oil extraction step employs a soaking vessel and a horizontal vacuum filter in place of the conventional immersion or percolation-type of extractor, and in that the material processed is usually subjected to a mild cooking and crisping operation prior to extraction.

This report presents data on application of filtration-extraction to soybeans.

Data are reported on a series of four pilot-plant runs, Nos. 1 through 4, on cooked flakes, using conditions found satisfactory in preliminary bench-scale tests similar to those previously reported for cottonseed (8). The bench-scale tests on soybeans indicated: rapid filtration rates, good oil extractability, high capacity, relatively low solvent ratio, low fines content in the miscella, and oil and meal products of acceptable quality. Data are also presented on three additional runs, Nos. 5, 6, and 7, with uncooked flakes to show that soybeans are an exception in that the flakes where properly prepared can be processed efficiently in the raw state. The data reported here supplement those presented in a previous paper (4).

The over-all process as applied either to cooked or to raw flakes is illustrated in the flowsheet, Figure 1.

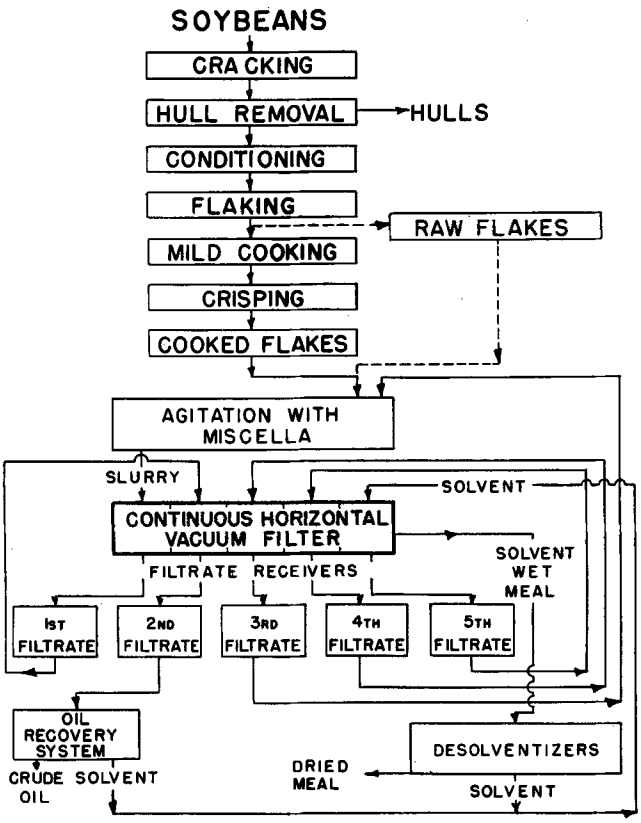

FIG. 1. Flow diagram of filtration-extraction process for cooked or raw soybean flakes.

For cooked flakes the sequence of operations consists of conventional cracking, dehulling, and tempering, followed by moist cooking, crisping, slurrying with miscella (oil-hexane mixture), filtering, and countercurrent washing of the drained cake three times on an enclosed rotary horizontal vacuum filter, and conventional recovery of the solvent from the oil and meal

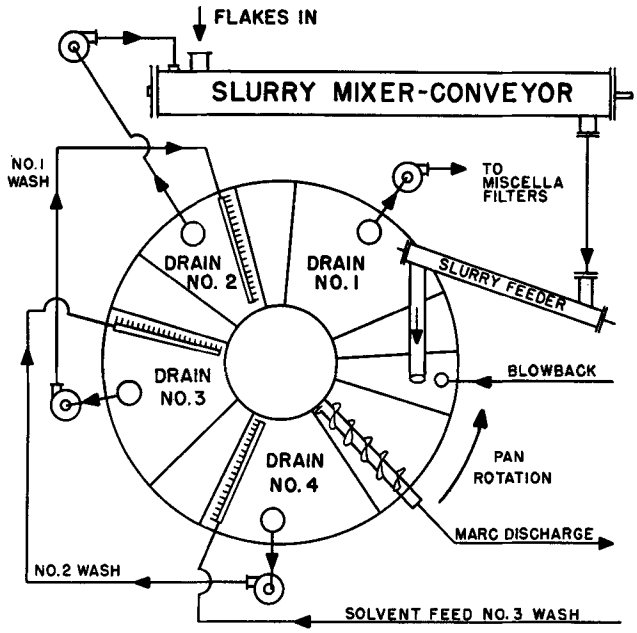

FIG. 2. Flow diagram of solvent-extraction phase of process.

products. For processing raw flakes the cooking and crisping steps are by-passed.

Cooking was the first approach tried since it has been a requisite for the successful operation of the process with all other oil-bearing materials processed to date. Cooking is utilized to impart the desired characteristics which result in improved extractability, relative incompressibility of material during filtration, and a particle size distribution that favors rapid and efficient washing and drainage. Cooking also reduces the need for careful flake preparation so important to the successful operation of immersion and percolation type extractors. Also in the case of soybeans the need for conventional toasting of the extracted meal after desolventization is eliminated since the required effects of toasting can be accomplished during the cooking operation.

Experimental Results

Material. Five different lots of beans, identified in Table VI as A through E, were processed in the seven runs reported. Lots A and B were reasonably free of trash and small green beans. Lots C and D contained foreign material in excess of 3%, along with numerous small green beans, and were thoroughly cleaned to remove the bulk of these components. For Runs 1 to 4 where the flakes were cooked, a different lot of current crop beans obtained from Mississippi was used in each. For Runs 5 to 7 where the flakes were not cooked, the material (Lot E) for Run 5 was flakes received from a commercial solvent extraction processor in Illinois and produced under normal operating conditions at his mill. The flakes were from 1-year-old beans and had been in transit and in 60°F. storage for five weeks before they were processed. For Runs 6 and 7, the same lot of beans (Mississippi) was used. The fact that this lot was also used for Run 4 afforded some measure of comparison of the two types of processes reported.

Equipment. Material preparation equipment used in this investigation included a Carver[3] 3-tray pneumatic-mechanical purifier for cleaning the beans and for hull removal after cracking; Allis-Chalmers[3] sin-

gle pass cracking and flaking rolls; French[3] 5-high stand of cracking and flaking rolls, top two rolls corrugated; French[3] 5-high jacketed stack cooker. All have been described in previous publications (1, 7). The cooker was equipped with atomizing sprays and steam ejectors for flake moistening, vents, sampling ports, and indicating and recording instruments to control as closely as possible the moisture and temperature of the flakes in each cooker ring.

A 6-in. open top screw conveyor, 7 feet long, was used to receive the material discharged from the cooker. An 8-mesh shaker screen was located at the discharge end of the conveyor to screen the material.

The extraction equipment included as principal continuous operating units a variable discharge flake feeder (2), a paddle type mixer-conveyor, a variable-speed slurry feeder, and an enclosed horizontal, rotary, vacuum, Oliver[3] filter of 3-foot diameter (3.5 sq. ft. screen area). All have been described in previous reports (1, 5). The assembly is pictured in Figure 3.

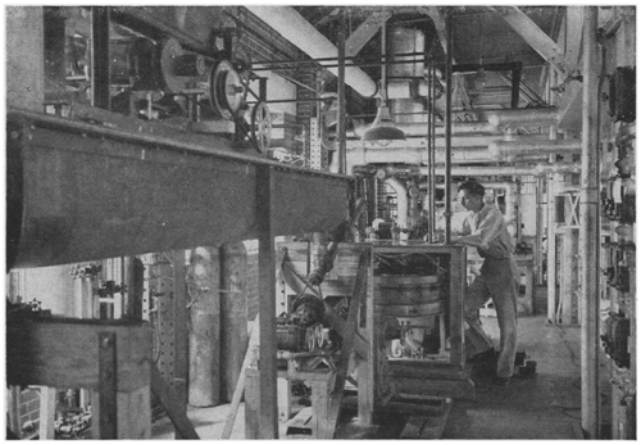

FIG. 3. Slurry mixer and its feeder at left, 3-in. slurry feed conveyor and drive in lower left center, and 3-ft. diameter rotary vacuum horizontal filter at right.

The filter, a close-up view of which is shown in Figure 4, is equipped with wash weirs for flowing the wash liquids onto the bed and with rakes to disturb the bed surface between washes.

The filter operates with a gas blowback system to keep the filter medium clean, and a vacuum system for drainage of the bed. For blowback, nitrogen gas is introduced continuously (rotameter) at a point directly beneath the screen section onto which the slurry is deposited. Vacuum is applied under the filter screens by means of a Nash[3] wet-seal pump, using hexane as the sealing liquid. This vacuum is supplied through a common header, connecting the five liquid receivers that collect the separate flows of filtrate from the multi-port filter valve. A separate centrifugal pump handles the filtrate from each receiver.

The pressure filters for polishing the concentrate or full miscella, the conventional type continuous evaporators and steam stripping column for oil recovery, and the continuous desolventizers for meal recovery have also been previously described (3).

Material Preparation. Five different methods of material preparation, using the five different lots of

[3] The mention of the names of firms or trade products does not imply that they are endorsed or recommended by the U. S. Department of Agriculture over that of other firms or similar products not mentioned.

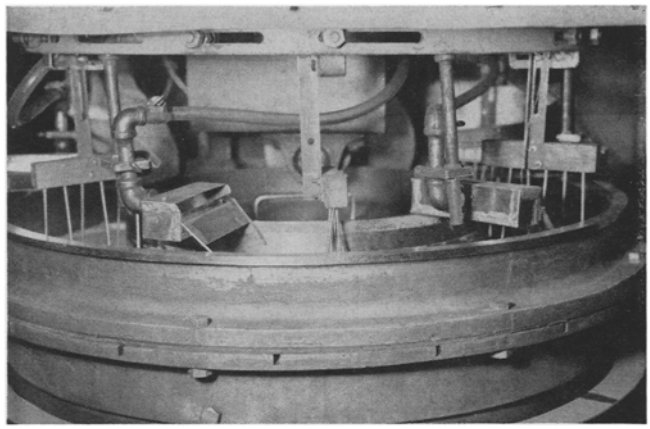

FIG. 4. Close-up view of filter showing wash weirs, rakes, and discharge scroll.

soybeans were evaluated for filtration-extraction in the seven pilot plant runs. Selection of these methods was based on preliminary pilot-scale processing information and on bench-scale tests. The principal differences in the preparation treatments for the runs are given in Tables I to IV and are summarized below:

Run 1 — Beans cracked (not dehulled or conditioned), flaked, cooked as for hydraulic pressing, rerolled.

Run 2—Beans cracked (not dehulled), conditioned, flaked, mildly cooked, screen overs rerolled.

Runs 3 and 4 — Beans cracked, dehulled, conditioned, flaked, mildly cooked, screen overs rerolled.

Run 5—Beans cracked, dehulled, conditioned, flaked by commercial processor.

Runs 6 and 7 — Beans cracked, dehulled, conditioned, flaked.

Cracking, Hulling. The beans were cracked in one pass through the one-pair-high corrugated rolls set at .080" for Runs 1, 2, 3, and at .060" for Runs 4, 6, and 7. The hulls were removed on the purifier and the "overs" were recracked and recycled (Table I).

Conditioning. The cracked beans for all runs except Run 1 were tempered. This was done in the top two rings of the cooker under conditions shown in Table II, using continuous feed and discharge. The beans for Run 5 had been tempered and flaked in the processor's plant. Conditioning differed for Run 2 in that only 8 minutes were used and a higher temperature (214°F.) was reached through the use of direct steam.

Flaking. Conditions for flaking are listed in Table I. The cracked meats were flaked at temperatures ranging from 80 to 180°F. Lowest, 80°F. (room temperature), was in Run 1 where no conditioning was done. In Runs 2 to 7, where the meats were conditioned, they were flaked hot. Temperatures used were in the range of 125 to 141°F., except for Run 2 where it was 180°F. Flake thickness was about the same, 0.0105 in. (average), for Runs 1 to 6 but was somewhat lower for Run 7. Flake moisture content ranged between 8.0% and 9.8% except for Run 4, where it was 12.6%. Bulk density data are given in Table IV.

Cooking and Crisping. Detailed cooking data for Runs Nos. 1 to 4, in which the flakes were cooked, are given in Table III.

Flakes were fed semi-continuously into the top ring of the cooker in batches of about 62 pounds. For Run 1, cooking conditions were similar to those used for conventional hydraulic pressing; whereas for the

TABLE I

Data on Cracking and Flaking of Soybeans

Run No.	1	2	3	4	5[a]	6	7
Roll, cracking type	1-Pr. high	1-Pr. high	1-Pr. high	1-Pr. high	.....	1-Pr. high	1-Pr. high
Roll, flaking type	1-Pr. high	5-High	5-High	1-Pr. high	.....	1-Pr. high	1-Pr. high
Hull removal, %	0	0	80	85	85	85	85
Conditioned meats temp., °F.	80	180	125	140	.....	141	137
Flake thickness, in.	.010	.011	.011	.008 to .011	.010	.007 to .011	.007 to .010
Flake moisture, %	.....	9.6	9.1	12.6	8.6	8.0	9.8

[a] Flakes obtained from commercial solvent extraction plant. Detail data not available.

TABLE II

Data on Conditioning of Cracked Soybeans

Run No.[a]	2	3		4		6		7	
Rings Nos.	1	1	2	1	2	1	2	1	2
Feed rate, lbs./min.	11.0	8.5	....	8.0	....	8.0	....	7.6	....
Jacket steam, psig.	10	60	6-15	62	6	62	6	60	6
Direct steam, psig.	12	0	0	0	0	0	0	0	0
Water added, lbs./min.	....[b]	0	0	0	0.2	0	0	0.1	0
Temperature mat'l reached, °F.	214	130	186	130	182	128	180	129	185
Retention time, min.	8	8	7	8	8	8	8	8	8
Temp. meats dischg., °F.	180	....	147	....	147	....	148	....	144

[a] Run No. 1, meats not conditioned; Run No. 5, dehulled meats conditioned but data not available.
[b] Sufficient water added to increase moisture content from 7.0 to 11.8%.

other three runs milder conditions were used in that the total cooking time was one-half or less and the temperatures were lower. The moisture content of the flakes was increased in the first ring to a relatively high level, as noted, to obtain the desired moist cooking at temperatures above 200°F. Vents were kept partially open in the succeeding rings and the steam jacket pressures controlled so as to decrease the moisture content to about 12 to 15%.

The hot cooked and partially dried flakes as discharged from the cooker were then subjected to an evaporative cooling step in order to impart the desired crispness to the material. For Run 1 this was done by spreading the hot cooked flakes on trays for cooling in the open air. After cooling, the material was rerolled in one-pair high rolls to break up any large agglomerates of water or oil balls that formed in the cooker and to roll out any hulls that may have curled around meat particles. For Runs 2, 3, and 4 crisping was accomplished by first passing the hot cooked material through the 7-ft. open conveyor and then screening it on the 8-mesh shaker screen (see Table IV for bulk densities of the cooked and crisped material). Large soft agglomerates were reduced in size to the extent that about 95% of the total material passed through the screen. The screen overs were then rerolled and recycled to the screen. During the evaporative cooling steps the temperatures of the materials decreased to about 130°F., and the moisture contents decreased 2 to 3%. This operation has been termed "crisping" since it produces particles which are characteristically crisp, granular, porous, and relatively incompressible. Such properties are essential for efficient removal of oil by filtration-extraction.

The cooking and crisping operations impart to the material still another characteristic important to filtration-extraction—proper particle size distribution.

TABLE III

Data on Cooking of Soybean Flakes

Run No.	Ring No.	Feed rate, lbs.[a]	Jacket steam, psig.	Retention time, min.[b]	Temperature, °F.[c]	Moisture, %[c]
1	1	60.0	13	12	205	16.0
	2	....	27	12	216	....
	3	....	24	12	217	....
	4	....	40	12	223	....
	5	....	12	12	228	13.0
2	1	63.5	0	12	216	17.7
	2	....	15	12	208	16.2
	3	....	21	5	212	15.5
3	1	62.5	0	12	219	16.0
	2	....	15	12	206	13.2
	3	....	21	4.5	212	12.6
4	1	62.5	0	12	185	17.5
	2	....	15	12	207	15.8
	3	....	22	5	213	15.9

[a] In pounds per 7 minutes for every 14-minute interval except Run No. 4 which was pounds per 8 minutes.
[b] Excluding 2 minutes to drop to next ring.
[c] Sufficient steam and atomized water added in first cooker ring to attain the given temperature and moisture contents.

TABLE IV

Bulk Density Data[a] on Raw and Cooked Soybean Flakes

Run No.	2	3	4	6	7
Raw					
Unpacked	25.0	25.0	31.5	24.1	24.1
Packed	32.0	32.0	41.7	32.7	30.8
Cooked					
Unpacked	31.5	33.5	31.0	....	....
Packed	38.9	42.9	36.0	....	....

[a] Pounds per cubic foot, using 1 cu. ft. container.

Table V compares the particle sizes of the flakes before and after they are cooked and crisped, as determined by wet screen analysis (2). Cooked material, which has the type of particle size distribution pattern as shown, tends to trap a large portion of the fines throughout the 2-inch cake bed on the filter whereas the size distribution shown for the uncooked flakes is conducive to plugging of the cake and filter screen during the draining and washing operations. In the latter case fines are readily washed through the relatively porous bed of flakes (about 60% larger than 8-mesh), gravitating to the bottom of the bed where they tend to form an impervious layer immediately above the screen. This retards the filtration rate objectionably unless a relatively open screen is used as discussed later.

Filtration-Extraction. Figure 1 is a flow diagram of the over-all process, including both the preparation and the extraction phases of processing cooked or raw flakes. The filtration-extraction phase is more specifically illustrated in Figure 2.

The cooked, crisped materials or the uncooked flakes were fed to the continuous mixing-conveyor (Figure

TABLE V

Wet Screen Analyses[a]

Material	Raw Flakes						Cooked Flakes		
Run No.	2	3	4	5[b]	6[b]	7[b]	2	3	4
Mesh	% on	% on	% on	% on	% on	% on	% on	% on	% on
5	33.6	10.6	47.9	9.5	40.6	57.1	0.0	0.0	0.0
8	25.7	32.7	25.9	46.2	34.5	22.7	0.5	0.7	2.3
14	21.4	24.4	11.4	19.0	13.4	12.6	14.2	14.9	22.0
20	11.8	16.5	7.1	14.9	7.2	7.6	21.1	25.6	32.8
40	4.1	9.0	3.9	6.1	2.2	3.2	34.2	28.0	24.4
60	1.2	3.3	1.4	2.2	0.7	0.9	14.9	14.4	10.0
80	0.7	0.9	0.5	0.7	0.3	0.4	7.8	6.1	2.6
120	0.5	0.8	0.3	0.5	0.3	0.4	2.1	4.5	2.6
170	0.4	0.5	0.3	0.2	0.3	0.4	3.1	1.9	1.7
200	0.1	0.1	0.1	0.1	0.1	0.1	0.8	1.1	0.5
300	0.1	0.2	0.1	0.1	0.1	0.1	0.8	1.6	0.5
Bottom	0.4	1.0	1.1	0.5	0.3	0.5	0.5	1.2	0.5

[a] Wet screening method developed at Southern Regional Research Laboratory (2).
[b] Runs in which raw flakes (uncooked) were fed to the slurry mixer. Data not available for Run No. 1.

3) and gently agitated with the second most concentrated filtrate (No. 3 filtrate) for 20 to 30 minutes. During this soaking period most of the oil goes into solution to form a concentrated miscella. A 3-in. inclined screw conveyor deposits the resultant slurry continuously onto the revolving annular pan of the filter (Figure 4). In 1 to 3 minutes, the time for one revolution of the filter pan, the bulk of the concentrated miscella is filtered from the slurry and the resultant cake countercurrently washed 3 times. The first two washes are with progressively weaker miscellas and the third with solvent (hexane). The solvent-damp extracted meal (marc) is discharged through a chute to the desolventizers below. The first and most concentrated filtrate miscella which contains some fines is usually pumped to a small polishing filter. In Runs 4, 6, and 7 however this filtrate was recycled to the filter pan as shown in Figure 1 for refiltration through the freshly formed cake bed in order to reduce the content of fines. The resulting filtrate is pumped to the polishing filter prior to oil recovery, or directly to the evaporator, when further polishing is not required.

Operating conditions for the runs are given in Table VI. The quantity of material extracted for each run, excepting Run 5, where the overall operation was unsatisfactory, as explained later, ranged from about 400 to 1,600 lbs., feed rate was 400-600 lbs. per hour, duration was 2-3 hours.

Retention times in the slurry-mixer ranged from around 30 minutes for the lower material feed rates to around 20 minutes for the higher rates. Slurry temperature reached was 80°F. (room temperature) for Runs 1 and 5, where the solvent was not preheated but was 110-120°F. for the other runs. Solvent-to-feed material ratio was varied from 1.0 to 1.3.

Cake thickness was controlled at around 2 in. (1.6-2.5), based on previous experience which indicates this thickness will probably prove most practical on a commercial scale.

As for filter medium, 24 x 110 mesh plain Dutch weave wire cloth (stainless steel), which works very satisfactorily with cooked cottonseed, proved satisfactory for Run 1 and was tried for raw flakes in Run 5, but excessive flooding occurred and it was replaced by one of more open construction, a 60 x 60 mesh stainless steel screen, which proved excellent in the remaining runs on both cooked and raw flakes.

Wash weirs were used to apply the washes on the filter cake in place of the nozzles used previously. Multi-toothed rakes (Figure 4), placed immediately downstream of each wash weir, were used to scratch the surface of the cake.

Representative samples were taken of the material as fed to slurry mixer, five filtrates, solvent-damp cake after each wash, final desolventized meal, and oil.

Results and Discussion

The data in Tables V to IX indicate that the filtration-extraction process, as has been previously demonstrated for cottonseed and rice bran, is likewise applicable for processing either raw or cooked soy-

TABLE VI

Extraction Data

Material	Cooked Flakes				Raw Flakes		
Run No.	1	2	3	4	5	6	7
Soybeans, Lot No.	A	B	C	D	E	D	D
Total wt., lbs.	1236	1560	1485	767	430	723	1147
Oil, %[b]	20.2	19.6	21.2	19.7	19.2	21.2	20.8
Moisture, %[b]	11.3	12.4	10.4	13.7	8.6	8.0	9.8
Feed rate, lbs./hr.	480	600	450	438	300	300-485	495
Solvent/meats ratio, lb./lb.	1.0/1.0	1.2/1.0	1.3/1.0	1.3/1.0	1.3/1.0	1.0/1.0	1.2/1.0
Slurry temp. °F.	80	108	108	115	80	115	120
Retention time, min.	25	20	25	26	30	28-20	21
Filter:							
Medium, mesh	24 x 110	60 x 60	60 x 60	60 x 60	24 x 110	60 x 60	60 x 60
Pan speed, min./rev.	1.5	1.5	2.5	2.5	1.6	2.25	2.0
Cake thick, in.	1.9	2.0	2.0	2.0	1.6	1.7-2.5	2.0-2.5
Vacuum, in. hg.	2.0	1.1	1.4	0.7	8.0	0.5	1.0
N_2 blowback, cu. ft./hr.	80	79	96	54	157	48	60
N_2 blowback, cu. ft./hr./sq. ft.	23	23	27	15	45	14	17
Cake on filter, % lipides[a]:							
Before 1st wash	14.08	11.33	9.11	....	....	....	22.8
Before 2nd wash	4.75	4.25	1.98	....	....	....	9.5
Before 3rd wash	2.04	2.21	1.04	....	....	....	3.9
Solvent in marc, %[b]	30.0	30.3	28.1	31.7	40.0	37.1	43.5
Moisture, desolventized meal, %[b]	8.2	8.7	7.8	9.1	4.5	6.6	6.8
Lipides, desolventized meal, %[b]	1.21	1.19	0.57	1.07	3.29	3.11	1.23

[a] Solvent-free basis. [b] "As is" basis.

TABLE VII

Lipides and "Fines" Contents of Filtrates

Material	Cooked Flakes				Raw Flakes		
Run No.	1	2	3	4	5	6	7
Lipides in filtrate, %:							
First	23.0	20.3	19.0	15.2	19.0	23.6	23.8
Second[a]	....	....	....	15.5	....	21.6	24.1
Third	13.0	9.1	7.6	5.5	14.8	12.8	13.8
Fourth	4.7	2.5	1.7	1.3	9.8	6.3	4.5
Fifth	1.6	0.9	0.5	0.5	3.6	2.6	1.2
"Fines" in filtrate, %:							
First	0.08	0.41	0.33	0.29	0.76	1.63	0.78
Second[a]	....	....	....	0.06	....	0.66	0.12
Third	....	0.16	0.08	0.05	0.15	0.36	0.09
Fourth	....	0.05	0.04	0.05	0.04	0.15	0.08
Fifth	....	0.02	0.01	0.01	0.02	0.10	0.04

[a] First filtrate recycled through filter bed prior to first wash.

beans. Extraction down to about 1.0% was obtained at a satisfactory handling capacity rate per square foot of filter screen area, and vacuum, blowback gas, and solvent requirements were in the desirable low range. In analysis and treatment of the data reported it is pointed out that since the seven runs were spread over five different lots of soybeans, and in addition, operating conditions were varied in the filtration-extraction step, strictly valid comparison of any one run with any other is precluded, except for Runs 4, 6, and 7 where the same lot of beans was used. However a number of significant observations can be made, as noted below.

Residual Lipides in Extracted Meal. The percentage of lipides in the product meal for Runs 1 to 4, where the flakes were cooked, ranged between 0.57 and 1.21% and averaged 1.01%. Lowest was for Run 3 in which the beans were dehulled and tempered, followed by mild cooking and 5-high rolling. This small difference cannot necessarily be attributed to the particular preparation any more than to other factors such as the particular soybean used, the use of 5-high instead of 1-pair high flaking rolls, and to the smaller average particle size of the meal as noted in Tables V and VIII.

Residual lipides for Runs 5 to 7, where the dehulled meats were only tempered and flaked, were 3.29, 3.11, and 1.23%, respectively. Run 7 was a repeat of Run 6 to attempt to further reduce the residual lipides. The same beans and bean preparation were used, but the flakes were somewhat thinner (which yielded a smaller average filter cake particle size), and the solvent-to-meal ratio and slurry temperature were higher. This combination of factors in the correct direction was effective in reducing the residual lipides from 3.11 to 1.23%. Results for Run 5 are disregarded as not comparative because of excessive flooding conditions on the filter (see under Filter Medium below).

It is recognized that considerable latitude exists for further reduction of residual lipides, such as by further increasing solvent ratio, solvent temperature, and slurrying time; use of additional washes and recirculation of filtrates through the filter bed; use of thinner flakes, and of multiple in place of single-pass rolling to reduce particle size; and improved tempering and cooking procedures.

Solvent-to-Meats Ratio. Ratios used in this investigation are somewhat higher than that used in commercial soybean extraction plants but can be reduced where means as mentioned under heading "Residual Lipides in Extracted Meal" are employed to obtain maximum oil extraction efficiency, and should result in increased product miscella concentration and filter capacity. The results as shown in Tables VI and VII, although limited, indicate that because of the lower extractability and the poorer drainage characteristics of raw as compared to cooked flakes, a somewhat higher solvent-to-meats ratio may be required for raw flakes, resulting in lower concentration of the product miscella.

Solvent Temperature. Extraction temperatures used in these runs were considerably lower than those used commercially. Temperatures up to 130°F. have been used successfully in runs with other oilseeds.

Miscella Concentration. Data in Tables VI and VII show the effect of countercurrent washing on the oil content in the filtrates and in the cake at the various stages on the filter pan and also show that most of the oil is removed during the initial draining of the slurry, and by the first wash. This is true to a significantly greater extent for cooked as against raw material because of the superior draining characteristics of the former as is further exemplified in Table VI. Concentration of product miscella ranged between 15.2 and 23.8%, depending upon the solvent ratio used, oil content of beans, and amount of oil extracted. The higher concentrations shown for the raw flake runs can be attributed mainly to the higher static holdup.

Solvent Content in Marc. Solvent content of the filter drained marc (Table VI) averaged about 30% for the cooked flakes compared to about 40% for the uncooked.

Fines in Miscella. Table VII gives the fines content of the five filtrates. The lower percentage of fines in the No. 1 and No. 2 filtrates for the cooked flake runs indicate the superior filtering characteristics of the filter bed over that for uncooked flakes, as indicated by the wet screen analysis data in Table V. The reduced fines content in the No. 2 filtrate shows the effectiveness of refiltration and suggests possible elimination of the polishing filter for clarification of the product miscella. It is also noted that, for the cooked material runs, a lower fines content was obtained with

Table VIII

Dry Screen Analysis of Pilot Plant Desolventized Meal

Material	Cooked Flakes			Raw Flakes		
Run No.	2	3	4	5	6	7[a]
Mesh	% on	% on	% on	% on	% on	% on
10	0.6	0.6	0.3	5.7	9.5	2.0
20	18.1	8.8	24.1	41.2	43.4	24.3
40	29.3	22.4	39.1	33.0	33.1	43.8
60	23.6	22.6	19.9	11.8	9.2	16.2
80	10.9	11.4	6.6	3.1	1.8	4.7
100	4.0	6.0	2.4	0.8	0.6	1.0
200	10.0	16.1	0.8	1.6	0.2	4.5
Bottom	3.5	12.1	6.7	2.8	2.2	3.5

[a] Dry screen analysis of first 80 lbs. of desolventized meal.

TABLE IX

Refining, Bleaching, and Analytical Data on Crude Oils

Run No.	Type flakes extr.	F.F.A., %	Refining loss, %	Color Refined and Bleached Oil[a]				Break[b] %	Phosphorus %	Lecithin (P x 30) %
				Natural earth		Activated earth				
				Y	R	Y	R			
1	Cooked	0.8	6.8[c]	....	....	35	2.3	....	....	....
2	Cooked	0.6	4.8	35	2.3	15	1.1	0.68	0.058	1.74
3	Cooked	0.5	4.1	35	2.3	20	1.2	1.30	0.134	4.02
4	Cooked	1.0	7.8	35	2.7	....	....	0.63	0.089	2.67
5	Raw	0.9	6.2	....	....	....	....	....	0.054	1.62
6	Raw	0.8	5.8	35	2.3	....	....	0.50	0.072	2.16
7	Raw	0.7	6.3	35	2.7	....	....	0.66	0.111	3.33

[a] Bleaching method A.O.C.S. Cc-8b-49; color readings by Wesson method A.O.C.S. Cc-13b-45 using Lovibond glasses. All crude oils tested No. 1 prime color grade. Activated earth bleaches shown for comparison only.
[b] Modified Gardner method.
[c] Refined by method A.O.C.S. Ca-9a-41 for hydraulic and expeller oils; all others by method Ca-9b-46 for solvent (hexane) extracted oils.

the finer (24 x 110) screen. For the raw flake runs the high fines content in Run 6 is attributed to the larger average particle as noted in Table VIII.

Filter Media. Of the two filter screens tested, the 60 x 60 mesh was found highly satisfactory for both materials. While passage of fines is greater because of the larger openings, this can be easily counteracted by utilizing the technique of refiltration through the cake bed. Not only is it less susceptible to clogging than the twill weave, but it is easier to clean and is more economical. Selection of this medium was also based on previous experience with cottonseed and other materials.

The 24 x 110 screen proved satisfactory for cooked cottonseed but when tried with raw soybean flakes in Run 5, over-all operation was poor due to retardation of mass velocity, which caused flooding and higher vacuum and blowback requirements.

Vacuum and Blowback. Vacuum as well as blowback gas requirements (volumetric rates and pressures) for all of the runs except Run 5 were in the desirably low range (Table VI). These requirements will vary mainly with the mass velocity of the material, the type and size of filter medium, and the cake thickness. Rate of blowback gas required is usually low at low vacuum conditions and will increase as the vacuum increases. Vacuum pump c.f.m. can be reduced by increasing flooding on the filter. Nitrogen gas was used as the blowback gas here, but in commercial installations it would be more practical to use hexane-saturated air from the filter hood.

Filter Capacity. The pilot-plant feed rates of around 500 pounds of soybean material per hour are equivalent to approximately 143 lbs. per sq. ft. of filter screen area per hour. By extrapolation, commercial size filters of 6 to 10-ft. diameter sizes, having 25 to 65 sq. ft. of filtering area, respectively, should therefore be expected to have capacities of around 43 and 112 tons of soybeans, respectively, per 24 hours.

In the runs reported considerable latitude existed for increasing the rate of feed to the filter because of the high mass velocities of the materials at low vacuum. Also it is recognized that further increase should be forthcoming from improvements in the material preparation.

In general, filter capacity can be increased by reducing the solvent ratio as far as is practicable, increasing the cake thickness, increasing pan speed, and use of higher vacuum and blowback. The extent to which the capacity of a given filter for any definite residual oil content in meal could be increased above that specified above would have to be determined in the field for the particular material and material preparation.

Meal and Oil Quality. Analytical data on the final meals are contained in Table VI. Residual oil content averaged around 1.0% for the five principal runs. This has been discussed above, together with suggested means for reducing same. None of the meals were toasted during or after desolventization as is done commercially. However the meals from the four cooked-flake runs showed negative trypsin and urease tests, indicating no further toasting would be required, but they were somewhat lighter in color than commercially toasted meals. Biological tests to compare the meals in nutritional value have not been completed.

Dry screen analysis data (Table VIII) on the final meals indicate that in general the meals produced from the cooked flakes are somewhat finer, as would be expected, than those from the raw flakes in that both the average particle size is smaller and the fraction through 80-mesh is larger. Meal from Run 4 is an exception however in that it compares favorably in both respects with that from Run 7 produced from raw flakes prepared from the same lot of beans. However it should be possible to upgrade the meals in this respect both by improved cooking procedures and through the use of suitable techniques for agglomeration of fines with water in the desolventization step.

Table IX gives the refining, bleaching, and pertinent analytical data on the crude oils. All of the oils were prime as to refining loss except Run 4 (7.8%), and all refined normally and bleached with natural earth to prime colors. No important or conclusive differences are noticeable between the oils produced from the cooked and that from the raw flakes. Break tests were in the normal range for crude soybean oil, except for Run 3 (1.30%). Phosphorus contents were also within the normal range, except for Runs 3 (0.134%) and 7 (0.111%).

Summary and Conclusion

Filtration-extraction has been satisfactorily applied to the processing of soybeans in seven pilot plant runs in which several procedures of preparation for extraction were evaluated. In four of the runs the meats were flaked and the flakes were mildly cooked and crisped; in the other three the meats were tempered and then flaked. Successful operation with either preparation was demonstrated by the fact that extraction to about 1.0% residual lipides in meal was obtained at a satisfactorily high capacity rate per sq. ft. of filter screen area at relatively low solvent, vacuum, and blowback requirements.

The procedure recommended for the preparation of cooked flakes is as follows: coarse cracking of the beans; dehulling by aspiration; conditioning at 130-140°F. and 9-11% moisture content; flaking to about 0.010 in. thickness; cooking for 20 to 30 minutes at temperatures up to 225°F., with initial moisture content of 15 to 17%; and crisping by evaporative cooling to a temperature of about 140°F. and moisture content of 10 to 12%.

For preparation of the uncooked flakes, the recommended procedure is dehulling and coarse cracking followed by conditioning at 130-140°F., and 9-11% moisture content, and flaking to about .008 in. thickness.

The extracted meal product from the cooked flakes showed negative trypsin and urease tests without requiring subsequent toasting. It was somewhat finer than the meal product (untoasted) from the raw flakes. The crude oils obtained by the two preparation methods were comparable in quality.

As for choice of one preparation method over the other, this would depend upon the particular processor, the equipment on hand, and upon oil and meal quality considerations, among other criteria.

It is emphasized that at this stage of the development of filtration-extraction for cooked or raw soybean flakes, no particular claims are made for this new process over existing processes for direct extraction of raw flakes. However its application should be of particular interest to the small and medium-sized mills confronted with the problem of crushing in a single season soybeans and one or more oil-bearing materials.

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Solvent Extraction of Cottonseed Meats[1]

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PREDICTION of the operation of continuous countercurrent solvent extractors for vegetable oils by applying data from batch and rate extractors has proved to be impractical because of basic differences in the systems. To obtain information on the effects of the factors controlling the rate and completeness of extraction of cottonseed meats in a countercurrent system the meats were extracted in a laboratory pilot plant previously used for similar study on soybeans (3).

Materials

The cottonseed was prime cottonseed purchased in November and stored in an unheated building until used during the winter and the following spring. Two solvents were used: extraction grade trichloroethylene with a boiling point of 188°F. and a specific gravity of 1.464 at 20/4°C. and commercial hexane (Skellysolve "B") with a boiling range of 146° to 157°F. and a specific gravity at 60°F. of 0.686.

Equipment

An attrition mill and a seed-fanning mill were used to dehull the cottonseeds and separate the resulting hulls and meats. The meats were tempered in a steam-jacketed, screw-type conveyor prior to flaking. Flaking was done by a pair of adjustable spring-loaded, smooth rolls 17½ in. in diameter with 1 9/16-in. faces which were operated at 206 r.p.m. A set of divider plates centered under the rolls was used to separate from the flakes the hulls which had not been removed in the fanning mill.

The extractor proper, which has been described by Arnold and P'Pool (3) consisted of a 2-in. diameter loop conduit enclosing a special conveyor chain which moved the flaked meats through the unit. The loop was fabricated of 2-in. standard pipe, interspaced with four Pyrex glass pipe sections which permitted inspection of the extraction process. The conveyor chain consisted of 24¾ ft. of standard No. 35 roller chain with semi-circular flights, 1 15/16 in. in diameter, attached to the chain every 3 in. by means of K-1 attachment links. The upper horizontal section of the extractor loop was jacketed with a 7-ft. length of 3-in. pipe, forming the first of three meal desolventizer sections. The second and third meal desolventizers were made of 2½-in. standard pipe, fitted with special ribbon type of conveyors. The second desolventizer was heated by a steam-jacket, and the third was heated electrically with two Chromel A asbestos-covered resistance wire windings connected to a 220-volt circuit through a carbon pile rheostat. The lower section of the extractor loop was wrapped with three lengths of Chromel A resistance wire, each having a resistance of 30 ohms. The power input to these heating elements was controlled by 7¼-ampere, 115-volt

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