(stearic acid), $\dot{S}_3 = 296$, $I_3 = 85.8$ (oleic acid), we find

 $w_1 = p_1 + q_1 = 2873.6 \text{ S}^{-1} - 9.643 - 0.00076 \text{ I}$ $w_2 = p_2 + q_2 = -2873.6 \, \mathrm{S}^{-1} + 10.643 - 0.01090 \, \mathrm{I}$ $\mathbf{w}_3 = -(\mathbf{q}_1 + \mathbf{q}_2) = 0.01166 \, \mathrm{I}$

Summary

The calculations involved in ester fractionation analysis require the solution of sets of three simultaneous equations. Repeated solution of these equations is avoided by obtaining expressions for the proportions of the components. Frequent analysis

of similar fractions is further simplified by the preparation of tables, slide rules, or nomograms.

Acknowledgment

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REFERENCES

¹ Hilditch, T. R., "The Chemical Constitution of Natural Fats," Chapman and Hall, London (1947). ² Rapson, W. S., Schwartz, H. M., Stoy, R. H., and van Rensburg, N. J., J. Am. Oil Chemists' Soc., 24, 84 (1947).

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Solvent Extraction of Granular Cottonseed Cake

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URING the past several years a process for extracting granular presscake which has many novel features has been developed and put into successful commercial operation in several plants. Although some of these features have been described piece-meal in the past, an integrated picture of this solvent extraction plant has not before been presented.

Considerable experience in the extraction of cottonseed and cottonseed cake has been gained in recent years. In some plants cottonseed meats are flaked and then extracted in equipment essentially similar to that used for soybeans. A more popular approach has been to extract presscake. In the 1951-52 period 14 solvent plants were put into operation. Eleven of these were designed to operate on prepressed cottonseed meats or presscake, and three employ direct extraction of meats. The popularity of extraction of prepressed meats is due in part to lower residual oil in finished meal, simpler operation, and ease of conversion from an existing screw-press to a prepress extraction operation.

All cottonseed solvent extraction processes must overcome certain fundamental hurdles. First, the economics of the cottonseed industry do not justify a large investment for solvent-extraction equipment, especially where there is an existing screw-press plant. Second, solvent-extracted cottonseed is dusty and difficult to pelletize. Third, cottonseed is difficult to handle mechanically through a solvent-extraction plant. In devising this process, the designer took all of these factors into account.

This new process takes presscake prepared by conventional screw-pressing, except that it may contain 10% oil, prepares it for extraction by simple granulation to a coarse size, and extracts it with the Rotocel system which will be described. This method results in an improved granular meal prepared in compact, easily operated equipment that can be installed at an economical price.

Preparation of Presscake

Presscake for extraction is prepared in essentially the same fashion as regular low-oil cake. The solvent extraction process is surprisingly flexible, and it will accept a wide variety of presscake including hydraulic cake. Generally speaking, the only change in the pressing process that is required is new shafts for the screw-presses, by which their capacity is about doubled for prepressing compared with low-oil meal operation. The oil content of the cake may be between 6.5 and 11%, and the moisture content may be as low as 6%.

The rate of feed to the plant is controlled by the feeder under the presscake surge bin as shown on the flow diagram. Cake directly from the screw presses, normally in the form of pieces $\frac{1}{4}$ to $\frac{1}{2}$ in. thick and 1 to 3 in. in length and width, is first broken in a double roll sawtooth horizontal crusher to make particles $\frac{1}{4}$ to $\frac{1}{2}$ in. in size with a minimum amount of fines. Broken hydraulic presscake may be mixed with or substituted for the screw press cake fed to the crusher. The broken cake is granulated in a three-high corrugated rolling mill.

This is the entire preparation system. It may be housed in a small building immediately adjacent to the solvent extraction plant. The operator need only make occasional inspection visits to this building. The total connected horse power in this area is 40 h.p. for 100 tons per day of cake; the actual power requirement is about 21 h.p.

Rotocel Extraction System

The cake extraction system consists of a Rotocel extractor, a vapor-type meal desolventizer, and miscella desolventizing system combined in an unconventional manner. A description of construction and operation of the Rotocel is necessary to understand this system.

In the extractor a rotor approximately 6 ft. high and 13 ft. in diameter, divided into 18 sector-shaped cells, rotates slowly about a vertical axis inside a vapor-tight tank. Only a 1/4 h.p. motor is required to turn the rotor at about one revolution per hour.

Cake granules are fed continuously through a seal conveyor, where they are slurried with miscella, into each cell. There the granules form beds which rest on hinged, screened doors, supported by rollers on a track. As the cells move around the circular path,

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TABLE I Screen Analysis of Finished Extracted Cottonseed Cake

U. S. Standard Mesh Screen	Percentage by weight
Through No. 8 on No. 10 screen	
Through No. 8 on No. 20 screen	
Through No. 8 on No. 40 screen	
Through No. 40 screen	

TABLE II Moisture and Oil Content of Cake

	Percentage by Weight	
-	Moisture	Oil
Prepress Cake		
Plant A.	6.2	10.7
Plant B	7.0	8.5
Plant C	11.5	7.2
Extracted Granular Cake		
Plant A.	7.9	0.32
Plant B	7.7	0.39
Plant C.	9.7	0.40

TABLE III Oil Quality

On Quanty			
	Plant A Unfiltered Oil	Plant B Filtered Oil	Plant C Filtered Oil
Free fatty acids Refining loss	1.6% 9.7% with 8.5% of 12° Bé caus- tic soda	1.0% 6.6% with 5.5% of 14° Bé caus- tic soda	1.3% 7.8% with 6.7% of 14° Bé caus- tic soda
Refined oil color	35 yellow 5.2 red		5.9 Lovibond R
Flavor Moisture and volatile	Prime	Prime 0.09%	Prime 0.2%

the beds are flooded by successive miscella washes of gradually decreasing oil concentration. Liquid drains from the cells into stage compartments under the rotor. From each stage compartment liquid is advanced in a direction countercurrent to the rotation by pumping it to a manifold above the rotor. Finally, the granules are sprayed with fresh solvent, then permitted to drain completely before they are discharged. At the proper location each door leaves the supporting track, falls open, and the extracted cake discharges.

In its elements the Rotocel is a compact, mechanically simple countercurrent percolation extractor. In more detail this extractor incorporates several features worthy of mention. Before leaving the extractor, full miscella is pumped over an established bed of cake granules, which acts as a filter, for removal of fines. As a further means of clarifying full miscella, a tent screen is placed over the stage compartment in which full miscella is collected. This screen is so designed that it is cleaned by the flow of miscella itself. These means of clarification are so effective that filtration of miscella prior to distillation is no longer necessary.

The Rotocel operates at a low solvent ratio. This results from adequate drainage of the extracted granules, by the provision of drainage between extraction stages and by controlled flooding under each manifold.

The extractor is only a part of the complete extraction system. This system differs from others in that solvent vapors from the meal and miscella desolventizers are returned to the extractor and are condensed in a reflux condenser mounted on the extractor. As a consequence of this arrangement several items of equipment which are usually found in extraction plants have been eliminated. These are the solvent tank, pump and meter, solvent heater, desolventizer scrubber, and much interconnecting piping. Since the extractor is at the boiling point of the solvent, maximum extraction efficiency is realized. In addition to the elimination of these items of equipment, the meal and oil desolventizing systems have been simplified. Meal desolventizing and steam stripping are accomplished in a single unit. Oil is freed of solvent by evaporation, followed by vacuum steam stripping. These operations require only two columns.

Miscella Distillation System

Clarified full miscella is pumped from the extractor to the miscella tank. This tank provides surge capacity between the extraction and distillation steps and is large enough to hold the entire liquid volume of the system at shut-down. Make-up solvent is added to the miscella tank as necessary.

Miscella is pumped through the preheater into the vertical long tube, rising film type evaporator, operating at atmospheric pressure. The miscella is heated to 220°F., equivalent to approximately 90% oil concentration. Rich miscella collecting in the boot of the

TABLE IV Miscellaneous Operating Data

Α.	Utilities	
	Steam.	
	Cake desolventizing	675 lbs. per hr.
	Oil desolventizing	1,815 lbs. per hr.
		2.490 lbs. per hr.
	598 1	os. per ton of cake
	Electric nower	~
	Cake preparation	23.1 KWH
	Cake preparation Cake extraction	94.9 KWH
		118.0 KWH
	27.3 K	W per ton of cake
	Water	IT per ton or cune
	Boiler water and cooling tower make-up	
	72 g	al, per ton of cake
В.	. Labor	
	Two men per shift for complete cake	
	preparation and extraction	8 man-hours/day
	0.48 man	-hour/ton of cake
C.	Solvent Usage	
	Commercial hexane	1,400 lbs./day
	1	4 lbs./ton of cake
D.	. Solvent to Cake Ratio	0.75:1.00
Ε.	. Solvent to Extracted Solvent-Free Cake Ratio	0.37 : 1.00
F.	. Full Miscella Concentration	

TABLE V

Unit	Processing	Costs
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A. Basis	
Operating rate 4.17 tons per hour	
B. Unit Costs	\$/hour
Steam at \$0.75/1,000 lbs	
Electric power at \$0.02/KWH	
Water at \$0.10/1,000 gal	
Solvent at \$0.03/lb	
Labor at \$1.50/man-hour	3.00
Maintenance	1.77
	10.75
C. Cost Per Ton of Cake	
Processing cost	\$ 2.58

evaporator separator is drawn into a vacuum-operated oil stripper. As the oil flows down the stripping column, a counter-flow of steam effectively removes the last traces of solvent. Finished oil is pumped from the stripper to the holding tank and from the holding tank is transferred to the unfiltered oil tank in the press room.

The hexane evaporated in the rising film evaporator flows back to the extractor. Hexane and steam from the oil stripper pass through a water-cooled and a refrigerated condenser in series. The non-condensed refrigerated vapors are compressed to atmospheric pressure by the steam-operated ejector. The exhaust from the ejector furnishes steam to the water stripper to vaporize any hexane accidentally entering the water stripper from the water separator.

Extracted Cake Desolventizing

Extracted cake is conveyed from the extractor to the vapor desolventizer. Solvent is vaporized from the granules with heat supplied directly by a recirculating stream of solvent vapor which is continuously super-heated. The solvent vapor so generated flows to the extractor.

The desolventizer is a horizontal vessel approximately 5 ft. in diameter and 22 ft. long. Rotating in it is a ribbon conveyor modified with lifting flights to shower the meal. Super-heated vapor entering the vessel at about the mid-point of its length flows countercurrent to the meal and is recirculated to the vapor heater by the recycle vapor blower. The recycle vapor heater is a shell and tube heat exchanger with the solvent vapor flowing through the tubes. Solvent vapor evaporated from the cake leaves the desolventizer at the point of entry of wet cake.

In the second half of the desolventizer, cake is effectively freed of last traces of solvent by countercurrent steam stripping. The heat input to the recycle vapor heater and the flow of sparge steam are so controlled that most of the steam condenses on the cake in the desolventizer. The additional moisture in the desolventized cake is utilized in the next step where the cake is cooled.

After passing through a rotary discharge lock, the cake from the desolventizer spouts to the cake cooler, which is a rotating drum containing lifting flights welded to the drum. Air flowing countercurrently effectively cools the cake to within 20°F. of the entering air temperature, largely by evaporation of water. Air leaving the cooler contains a small amount of fines and lint particles. This air is blown through cyclone and bag type of collectors in series. The fines recovered from the air stream recombine with the main flow of extracted cake granules leaving the extraction plant.

Solvent vapors from the cake and oil desolventizing flow through the extractor to the reflux condenser, which is mounted directly above the zone of fresh solvent wash. As the vapors flow across the extractor, they are effectively scrubbed free of fines. The condensate from the reflux condenser returns by gravity to the solvent decanter where water is separated and solvent over-flows to the extractor. Water from the decanter flows to the water stripper, where it is heated to boiling before discharging to the drain.

Operating Data

The three-Rotocel system plants serving as a basis for this paper have a nominal operating capacity of 100 tons of press cake per 24-hr. day. All the data presented are based on processing that quantity of cake.

Summary

Advantages of the Rotocel system for cottonseed cake extraction may be summarized in the following manner:

- 1. Economical conversion to solvent extraction is possible by utilizing existing or reconditioned screw-press equipment.
- 2. Conditioner and flaking rolls are not required for this process.
- 3. The electrical power requirements for the cake preparation and extraction are less than the power reduction when converting from straight pressing to a prepress operation. This results in a net savings in power consumption.
- 4. Vapor scrubbers used in conventional plants are not required in this process.
- 5. Efficient extraction at low solvent ratios keeps the steam requirement of this process to a minimum.
- 6. Low residual oil in extracted meal accomplished by this process yields a maximum product value per ton of seed.
- 7. The low temperature vapor desolventizer eliminates discolorization and protein degradation of the cake granules during the desolventizing step.

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Oils and Fats

Ralph W. Planck, Abstractor Dorothy M. Rathmann, Abstractor Sin'itiro Kawamura, Abstractor

Antioxidants in edible fats. Jouko Valpola. Teknillisen Kemian Aikakausilehti 10, 461-2(1953). Eleven antioxidants at 0.05% concentration and 6 edible fats were studied at room temperature and boiling-water temperature. The course of autoxidation of fat was measured by the peroxide no. At 100° pyrogallol inhibited oxidation of rapeseed oil. At the same temperature L-ascorbic acid was an excellent inhibitor but not so at room temperature. Although at 100° rapeseed oil was more stable than soybean oil, the reverse was true at room temperature. (C. A. 48, 4143)

Investigations on the unsaturated acids of the liver oil of Galeocedro tigrinus. R. Sen Gupta, A. Grollman, and S. C. Niyogy. *Proc. nat. Inst. Sci. India* 19, 527-39(1953). A sample of liver oil from *Galeocedro tigrinus* which lowered the blood pressure of animals with experimentally induced hypertension was analyzed. Some of the constituent unsaturated acids were identified by preparation of the *p*-phenyl phenacyl esters, and the constitution of these acids was determined by oxidation with potassium permanganate. These were palmitic, oleic, linoleic, linolenic, gadoleic and tetracos 12:16 dienoic acids. The physiologically active component was not identified. (*Food Sci. Abs.* **26**[3], 278)

The growth and optical properties of stearic acid crystals. Ajit Ram Verma and P. M. Reynolds (Univ. London). Proc. Phys. Soc. (London) 66B, 989 (1953). A discussion of 2 types of stearic acid crystals, identified as the B and C polymorphs is given. (C. A. 48, 6769)

The safety of mono- and diglycerides for use as intentional additives in foods. H. E. Longenecker, et al. Natl. Research Council—Natl. Acad. Sci. (U.S.), No. 251, 14 pp.(1952). Evidence concerning the safety of certain emulsifying agents in processed foods is reviewed. Reference is made to the natural occurrence of mono- and diglycerides in food fats and oils, the formation of monoglycerides during food preparation, and of triglycerides during digestion. Information is presented as to the amount of monoglycerides and diglycerides intentionally added to foods. Reference is made to the nutritive value and also to the surface activity of mono- and diglycerides. The authors find no evidence to question the safety of these as food additives. Extensive bibliography. (C. A. 48, 7213)

The safety of polyoxyethylene stearates for use as intentional additives in foods. H. E. Longenecker, et al. Natl. Research