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Pilot Plant Desolventization of Fine Cottonseed Meal[1,2]

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IN the course of developing the fractionation process (5, 10, 11, 12) at the Southern Regional Research Laboratory, relatively large quantities of fine cottonseed meal were produced. This solvent-damp, fine cottonseed meal, mostly 300-mesh, was desolventized satisfactorily in a continuous pilot-plant meal dryer of the screw conveyor type.

In addition to accomplishing the primary objective of desolventizing this fine meal on a continuous pilot plant scale, the investigation showed that a reduction in free gossypol of up to 69% could be obtained during the drying operation and also that a desirable granulated product, 80% of which was coarser than 20-mesh, could be produced.

A brief resumé of the fractionation process follows since the characteristics of the meal produced in fractionation were not only responsible for the drying problem but also had some effect on the gossypol reduction and on the type of dried material obtained. In fractionation, defatted or undefatted flakes are disintegrated (10) by liquid shear to separate the pigment glands from the meal and then screened. After screening, the material is differentially settled (12), and the fine suspended meal essentially free of glands is decanted. The bulk of the solvent is removed by either centrifugation or pressure filtration. The final step in the process is desolventization, or drying of the fine meal.

In the early pilot-plant-scale fractionation runs, where the quantities of the fine meal recovered were fairly small, the meal was merely spread on trays and air-dried. This method afforded little or no control over the desolventization operation. The prevailing temperature and relative humidity determined the time required for complete desolventization, also the final moisture content of the meal. The solvent obviously was not recoverable. In the further development of the fractionation process therefore a procedure for the continuous desolventization of larger amounts of fine meal under controlled conditions was needed.

Five pilot-plant-scale fractionation runs were then planned and conducted to determine the possibility of continuously desolventizing the fine meal without clogging the vapor system and without seriously affecting the quality of the meal produced.

As an approach to the problem, a 4-stage, jacketed meal dryer, which was available as part of the Southern Laboratory's continuous solvent-extraction plant (4), was modified so that it would be suitable for fine meal desolventization. In ordinary continuous solvent-extraction runs the extracted meal was fed to this dryer by gravity from the discharge end of the inclined extractor, and the material was then taken through the dryer by screw-conveyors. Sections 1 and 2 contain ribbon-type conveyors 10 inches in diameter which rotate at 2.18 r.p.m., and sections 3 and 4 have standard solid flight conveyors, 16 and

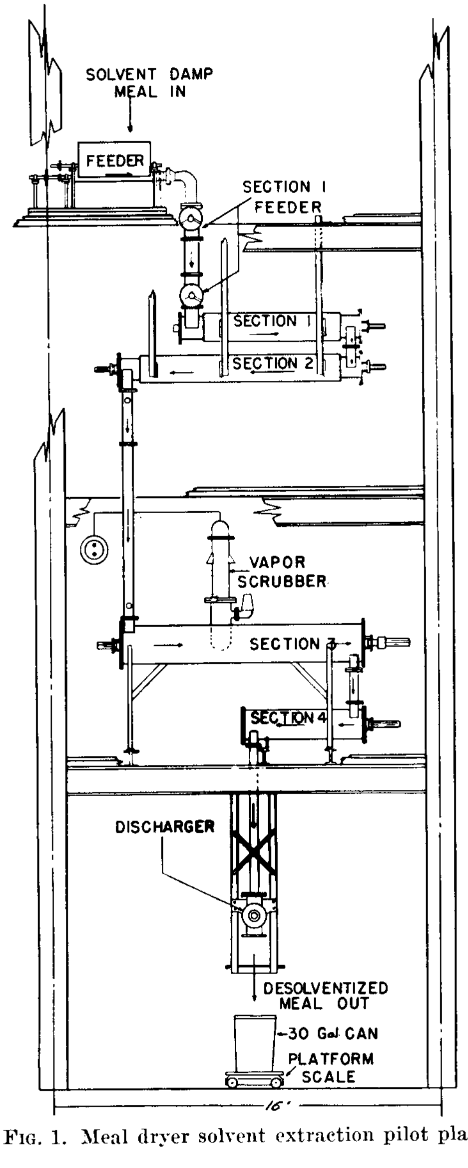

FIG. 1. Meal dryer solvent extraction pilot plant.

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[1] Report of a study made under the Research and Marketing Act of 1946.

[2] Presented at the 42nd Annual Meeting of the American Oil Chemists' Society, New Orleans, La., May 1-3, 1951.

[3] One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

TABLE I

Data Showing Gossypol Content and Protein Solubility of Meals Before and After the Desolventization Operation

Run Number	Feed to Dryer				Desolventized Product				Free Gossypol Reduction %
	Solvent (Average) %	Gossypol		Protein Solubility %	Gossypol		Protein Solubility %	Thiamine Content p.p.m.	
		Free %	Total %		Free %	Total %			
1	20	0.45	0.75	75.0	0.21	0.85	65.0	29.73	53.4
2	20	0.48	0.48	82.5	0.15	0.48	74.0	34.82	68.8
3	20	0.22	0.23	85.0	0.07	0.22	81.0	38.81	68.2
3-A[a]	20	0.29	0.36	83.0	0.09	0.35	76.0	38.36	69.0
4	20	0.29	0.39	83.0	0.12	0.41	76.0	37.96	58.6
5	50	0.13	0.24	86.6	0.06	0.19	84.7	33.70	53.8
5-A[a]	50	0.17	0.40	83.7	0.06	0.33	84.5	....	64.8
5-B[a]	50	0.29	0.45	83.9	0.10	0.39	85.3	33.67	65.6

[a] Refers to the fine meal recovered by reprocessing the coarse meal fraction produced in differential settling.

10 inches in diameter, respectively, and rotate at 0.37 r.p.m. The discharge end of the dryer was equipped with a quick-opening slide gate which was manually operated at predetermined intervals. This method of discharging would have caused fluctuation in vacuum and consequently would have induced fine meal to enter the vapor system.

To modify this dryer for fine meal desolventization a plug-type screw feeder and a plug-type screw discharger were added (Figure 1), and the chutes between the sections were provided with heating coils and insulated. The partial sealing, effected by the feeder and discharger, permitted operation of the dryer system under a vacuum of 0.3 to 0.5 inches of water without an appreciable fluctuation. This slightly reduced pressure and heating of the chutes promoted the transfer of solvent vapors from the dryer sections to the vapor scrubbers and solvent condensers without the condensation of solvent and moisture in the discharge chutes and seal arrangements.

The feed rate to the dryer was 84 to 100 pounds of fines per hour on a solvent-free basis. Meal retention time in the dryer was about 45 minutes. In each of the 5 runs 150 to 450 pounds of meal were desolventized. A steam pressure of 25 pounds per square inch in the dryer jackets resulted in vapor temperatures of 180°F. in the first section, 170° in the second section, and 125° in the last two sections. The apparent drop in the vapor temperature in the last two dryer sections indicated that the bulk of the solvent was removed in the first two sections. Water vapor, removed in increasing amounts as the solvent content was reduced, exerted a stripping effect in removing the final solvent traces in the last sections. Although the vapor temperature in the last dryer section was 125°F., the exit meal temperature was 150 to 155°F.

Under these conditions it was found that the fine meal, as well as marc (solvent damp, extracted flakes) from the solvent extraction plant, could be continuously desolventized in this modified dryer without clogging of the vapor system.

Table I shows the results of analyses of meal samples taken before and after desolventization. In the first 4 runs the fine meal was recovered by centrifugation whereas the meal for the fifth run was recovered by pressure filtration. The main differences in these two meals were that the centrifuged meal contained 20% solvent and had a bulk density of 15 pounds per cubic foot while the meal recovered by filtration contained 50% solvent and had a bulk density of 95 pounds per cubic foot.

The percentage reduction in free gossypol due to the desolventization operation ranged from 53 to 69%, based on the free gossypol content before desolventization. This reduction on a pilot-plant scale is comparable to results obtained in a series of laboratory experiments (6) in which the free gossypol was reduced by 50 to 55% under controlled conditions of moisture and heat. The reduction in per cent protein solubility (2) ranged from 4 to 10% for the first 4 runs whereas for the fifth run the protein solubility was, for all practical purposes, unchanged. The thiamine contents ranged from 29.7 to 38.8 p.p.m. which is considered very high. For hydraulic- and screw-pressed meals the thiamine contents range between 10 and 15 p.p.m. The trend of decreased gossypol content and increased protein solubility in the feed to the dryer for succeeding runs was due to variations in the fractionation process operating conditions. Not shown in the table are the moisture contents which averaged 8% before, and 3% after drying. The free and total gossypol determinations were made according to the *p*-anisidine colorimetric method (8, 9) developed at the Southern Regional Research Laboratory. Protein solubility was determined by peptization with 0.5 N sodium chloride solution. The thiamine content was determined by a published method (3, 7), modified at the Southern Regional Research Laboratory. The desolventized meal from the fifth fractionation run, unlike that from the other 4 runs, was a granulated, pellet-like, material. Figure 2 shows several screened fractions of the granulated meal. A screen analysis was made of the granulated material using

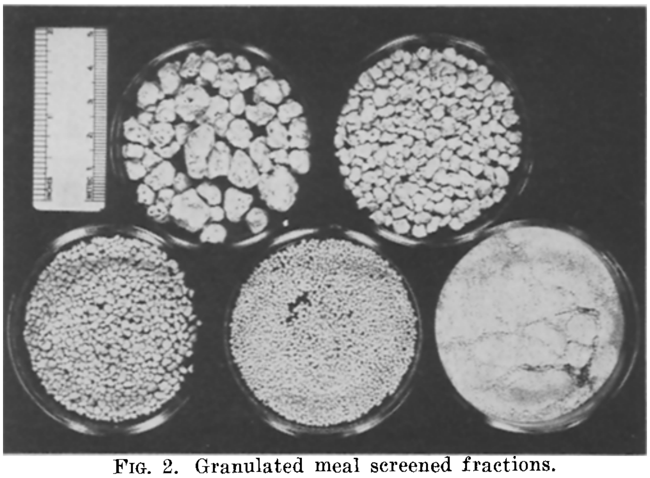

FIG. 2. Granulated meal screened fractions.

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[1] The use of trade names does not constitute an endorsement by the U. S. Department of Agriculture of their product over similar products not mentioned.

a Rotap[4] for a 10-minute period. The weight percentage of the screened fractions were 37.3% on 5-mesh, 21.4% on 8-mesh, 16.3% on 16-mesh, 5.5% on 20-mesh, 9.2% on 40-mesh, and 10.3% through 40-mesh. This gives a total of 80.3% of the material coarser than 20-mesh.

Since operating conditions were the same in all the desolventization runs, the physical characteristics of the meal fed to the dryer was the principal cause of obtaining the different product although the different degree of compression obtained with the heavier material in the plug-type screw feeder may have contributed.

Summary

1. The continuous desolventization of fine cottonseed meal has been successfully accomplished in a modified pilot-plant meal dryer of the screw-conveyor type under controlled conditions. The work should also have application in commercial solvent-extraction where a general fines problem (1) exists.

2. Free gossypol content has been reduced as much as 69% during the desolventization operation without appreciably affecting the protein solubility of the meal.

3. A meal in granular form can be obtained, provided the feed material has certain physical characteristics. This granulation, or essentially pelleting, effect should have application particularly in nutritional work where the feeding of meal in pellet form of various sizes is required. It is believed that a number of conditions contributed to the granulation. The most important was probably the high density of the material which was fed in a compressed or extruded form. It is likely that the fineness of the material, the moisture content, and the solvent content contributed to this consolidation during desolventization, but the quantitative effect of these variables has not been determined. It is thought that any material that can be made to hold together during the initial part of the desolventization will tend to produce a granulated product at the end of the drying period.

Acknowledgment

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A Note on A Sensitive Detector for Trichloroethylene

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THERE are on the market leak detectors that are very sensitive to trichloroethylene. The instrument is designed to detect leaks in distillation equipment. One method is to spray a halogenated hydrocarbon around the suspected spot, withdraw vapors from the outlet of the still, and pass these through the detector. The vapors pass by a red hot surface, near another (cool) surface, several hundred volts D.C. being impressed across the gap. The hot surface ionizes the halogenated hydrocarbon, and the voltage drives the ions across. The current is amplified and shows on a meter.

The work reported here was done with the leak detector described in Electronics, March, 1948, page 100. A similar device is described in Chemical Processing, April, 1951, page 64.

A series of known samples containing 1,000, 100, 10, and 1 p.p.m. of trichloroethylene in oil was prepared. The samples were 250 grams and were mixed well in 500 ml. Erlenmeyer flasks, stoppered, and allowed to stand at room temperature overnight.

A test was made by lowering the instrument probe vertically within the flask to about one-half inch from the surface of the oil. The meter showed a definite deflection with the 0.01% sample and none with 0.001% at room temperature. At 85°C. this limit of detection is lowered to between 0.001 and 0.0001%. The practical limit of the modified Beilstein test now in use is 0.01% (100 p.p.m.).

The leak detector is simple and rapid in operation. A standard method of using it could be devised if the increased sensitivity obtained can justify the cost. Roughly quantitative results are possible in the range 10-100 p.p.m. trichloroethylene and perhaps lower. The instrument costs about $500.

The work was done by E. M. Sallee, who is submitting a paper on the modified Beilstein test for the same purpose.

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