

cedure for crisping; selection of best conveyor type for conveying cooked material over relatively long distances without objectionable comminution; selection of filter medium combining the desired properties of durability, non-fouling, and low fines retention; determination of optimum filter-cake thickness; and development of a method for clarification of the product miscella by continuous recycling through the formed cake on the filter. Also discussed is the quality and color stability of the oil produced from comparable lots of cottonseed by the filtration-extraction process, as compared with that by hydraulic pressing, and by solvent extraction of uncooked flakes.

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## Solvent Extraction of Fish Offal<sup>1</sup>

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SOLVENT EXTRACTION, which has been applied extensively to certain oilseeds, such as soybeans, has had a relatively limited application to fish and fish products. Most of these applications have been to fish livers or whole fish (1, 7, 8). The large amounts of fish offal and surplus fish which are wasted or inefficiently processed have aroused an interest in the industry in the application of continuous countercurrent solvent extraction to these materials.

This investigation is concerned primarily with the solvent extraction with trichloroethylene of fish meal produced by the wet cooking of fish wastes. The material used in this study contained from 13 to 19% oil and 20 to 45% moisture and was prepared for extraction in the laboratory static-bed rate extractions by passing it through a Universal No. 2 household food chopper with a coarse cutter. The material used in the pilot plant model of the Iowa State College extractor was prepared for extraction by passing the fish meal through a John Deere No. 10-A hammer mill operating at 3,150 r.p.m. with a screen having 1/2-in. square openings.

### Laboratory Studies

The laboratory data were obtained by means of normal static-bed rate extractions. The apparatus (4) for this investigation consisted of a 1-in. jacketed glass tube with the necessary auxiliaries to permit the bed of fish meal and the incoming solvent to be maintained at a constant temperature. The overflow miscella rate was 10 ml. per minute.

The screen analysis of the fish meal after passage through the food chopper is given in Table I. The

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TABLE I  
Screen Analysis of Fish Meal Used in Laboratory and Pilot Plant Studies

U. S. Screen Size	Screen opening	Percentage passing screen	
	Inches	Laboratory	Pilot plant
3	0.371	98.3	94.5
4	0.263	90.7	87.2
6	0.185	72.4	81.8
8	0.131	58.4	77.4
12	0.093	42.1	70.5
	0.065	27.7	60.8

variables investigated in the laboratory were temperature, particle size, moisture, and wetting agents. The results of these experiments for the first three variables are given in Figures 1, 2, and 3.

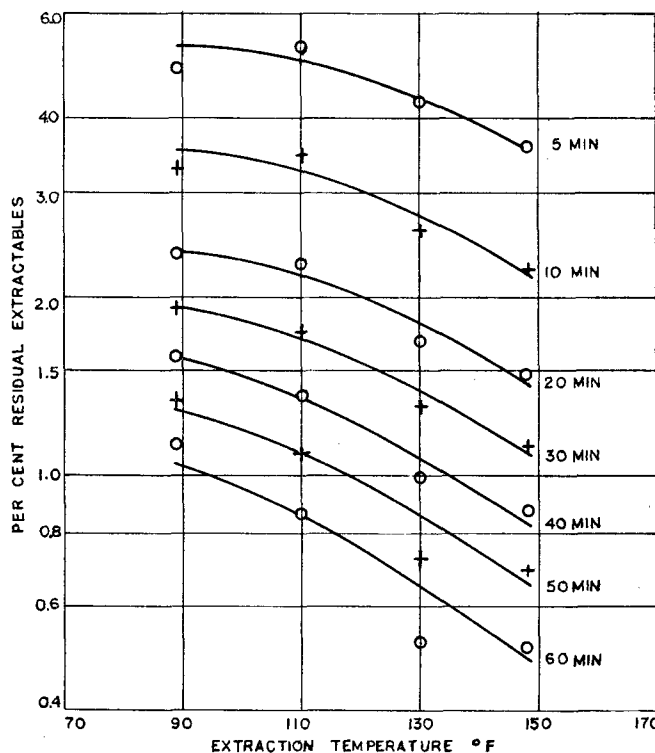


FIG. 1. Variation of residual extractables with temperature in laboratory studies.

Moisture content of feed: 25.4%  
Residual extractables on moisture-free basis.

The fish meal used in the study of the effect of temperature on the extraction had a moisture content of 25.4%, and it will be shown later that moisture content influences the results appreciably. The effect of temperature on the residual extractables<sup>2</sup> is not great because the extracting solution is almost pure

<sup>2</sup> Residual extractables are determined by means of a Soxhlet extraction using trichloroethylene as a solvent. The ratio of hexane extractables to trichloroethylene extractables has been found to be 0.86.

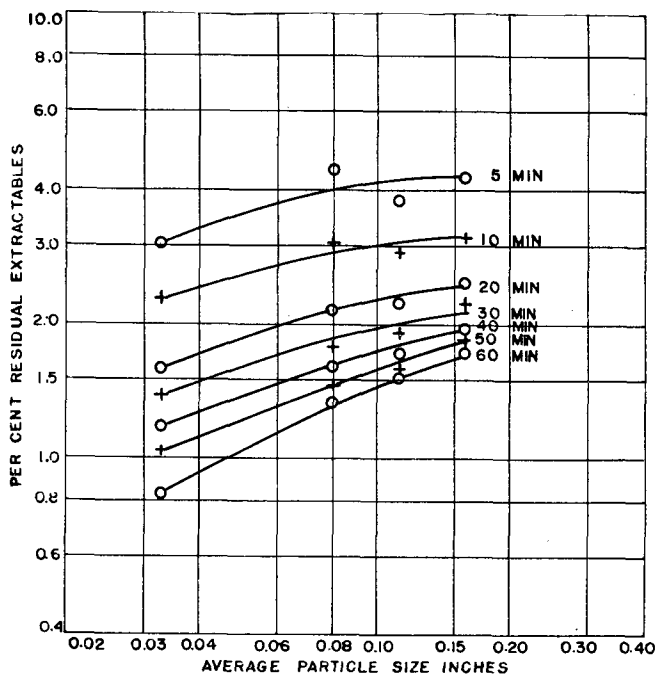


Fig. 2. Variation of residual extractables with particle size in laboratory studies.

Moisture content of feed: 25.4%  
Residual extractables on moisture-free basis.

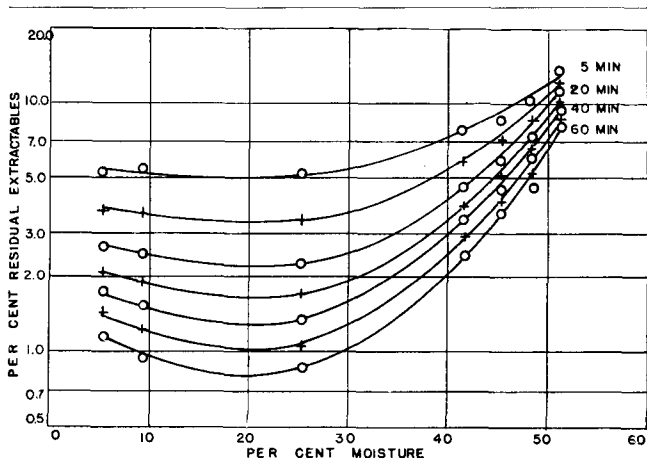


Fig. 3. Effect of moisture on residual extractables in rate extractions.

Extraction temperature: 110°F.  
Residual extractables on moisture-free basis.

solvent, and the physical properties of this are not affected greatly by temperature.

The particle size data give the expected result, and the effect of particle size variation is not very great when the range of particle sizes tested is considered. Fish meal, in general, gives smaller particles than does material of land animal origin (2) which has been processed in a somewhat similar manner.

Moisture contents above 20% have a profound effect upon the residual extractables. This effect is due to the failure of the organic solvent, trichloroethylene, to wet the fish meal readily. At moistures below 20% the increase in residual extractables, particularly in the diffusion portion of the extraction, probably results from a physical shrinkage taking place in the particle during the air-drying operation which retards the entrance of the solvent into the solid. These results duplicate the trend as to the effect of moisture

as reported by Arnold and Patel (4) for soybeans and cottonseed and by Arnold and Arvidson (2) for meat offal.

Because of the high moisture of the fish meal as it was received (45%) it was considered of interest to ascertain the improvement possible with some of the commercially available wetting agents. In all, 11 wetting agents of differing compositions were tested, and it was found that some improved the oil removal and others tended to retard the extraction. The best wetting agent, an alkyl aryl sulfonate, was found to improve the extraction efficiency from 76.4 to 91.6%.

### Pilot Plant Studies

The pilot plant model of the Iowa State College extractor that was used in these studies was also used in the work reported by Arnold and P'Pool (5) and Arnold and Juhl (3). This extractor has a capacity of 5.44 lbs. of fish meal per hour at an extraction time of 51.2 min. and one of 14.43 lbs. per hour at an extraction time of 18.1 min.

The fish meal used in this phase of the investigation differed physically from that used in the laboratory phase. In the laboratory rate extractions the fish meal was composed mainly of fish flesh with some bone and shell material. However, in the pilot plant work, the fish meal consisted of fish fins, fish scales, and fish flesh. This difference in physical composition precluded the possibility of obtaining a correlation between laboratory and pilot plant results. It also indicates the difficulty of securing precise data representative of fish meal in general. The screen analysis of the fish meal used in the pilot plant extractor is given in Table I.

Data were collected in the pilot plant model of the Iowa State College extractor to show the effects of miscella concentration, extraction time, extraction temperature, and moisture, when using trichloroethylene as a solvent. Information was also collected on the extraction of fish meal with commercial hexane (Skellysolve B).

Effects of the four important extraction variables on the extraction of fish meal with trichloroethylene are given in Figures 4, 5, 6, and 7. The effect of miscella concentration upon the residual extractables can be represented as a straight line, and since this variable is not controllable to exact concentrations, it was necessary to correct the data from other studies to a constant miscella concentration as has been done in previous studies with cottonseed (3) and meat scrap (2). The selected miscella concentration for comparative purposes was 15%. This was considered the best value since it was easily obtained, it did not introduce variations in the results as would attempts to use a higher concentration with the accompanying increased pumping,<sup>3</sup> and it did not result in a fluctuating miscella overflow from the extractor such as might occur with a low solvent rate. The data were corrected to the 15% miscella concentration by multiplying the actual residual extractables by the ratio of the residual extractables at 15% to the value for the residual extractables at the experimentally determined miscella concentration. Both values for the ratio were taken from Figure 4.

The effect of extraction time, shown in Figure 5, indicates that the extraction process is one of surface-

<sup>3</sup> Pumping is the increase in the solvent level at the solvent inlet side of the extractor.

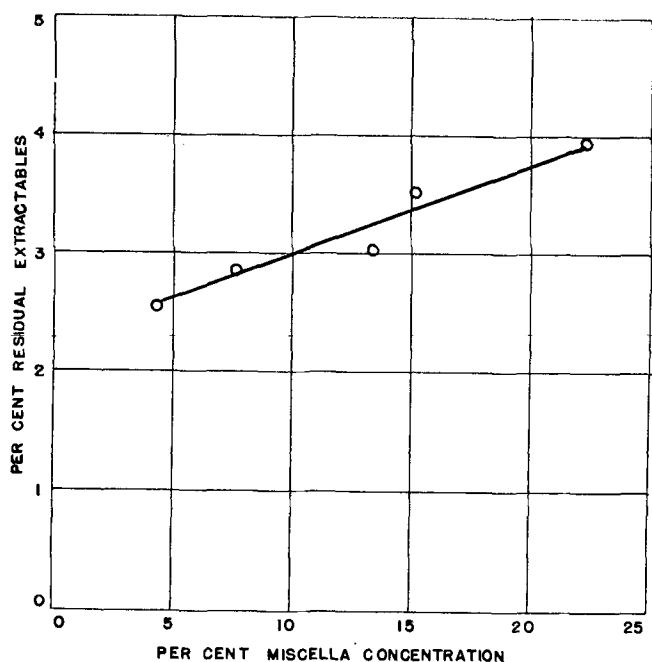


Fig. 4. Variation of residual extractables with miscella concentration in pilot plant.

Extraction time: 26 min.  
Extraction temperature: 110°F.  
Average feed moisture: 21.2%  
Residual extractables on moisture-free basis.

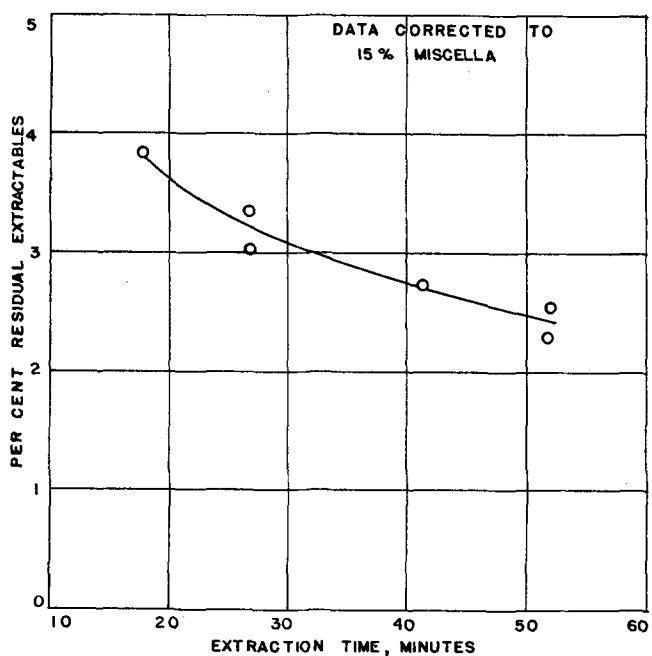


Fig. 5. Variation of residual extractables with extraction time in pilot plant.

Average feed moisture: 21.2%  
Extraction temperature: 110°F.  
Residual extractables on moisture-free basis.

washing for short extractions and one of washing and diffusion for longer extractions. The reasons for this conclusion are:

1. The fish meal has been cooked, and in this operation the protein of the fish has been coagulated and sufficient time has elapsed for a good portion of the oil to diffuse to the surface of the meal particles.
2. If the data had been plotted on semi-logarithmic paper, the data for extractions of more than 28 min. in duration would lie on a straight line. This straight line means that

diffusion is controlling the reduction in residual extractables with increased extraction time.

Figure 6 shows that a proportional decrease in residual extractables is obtained with increasing extraction temperature. Moisture (Figure 7) plays a more important role in extraction in the pilot plant than in the laboratory. This greater effect of moisture is due to the change in the effect of the wetting of the fish meal by the extracting miscella. Nevertheless the effect of moisture in the range of contents covered is not great.

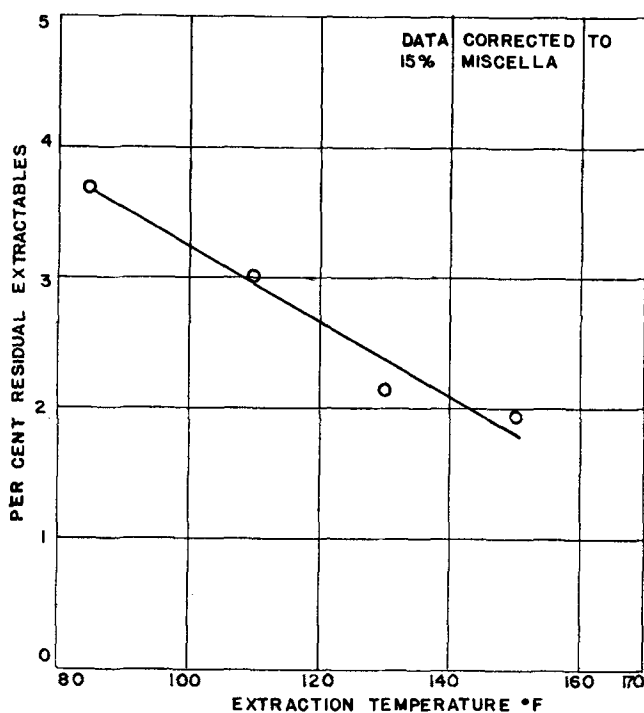


Fig. 6. Variation of residual extractables with extraction temperature in pilot plant.

Average feed moisture: 21.2%  
Extraction time: 26 min.  
Residual extractables on moisture-free basis.

Following the promising results in the laboratory with wetting agents, an extraction in the pilot plant was carried out, using the best wetting agent tested in the laboratory. The results showed that the extraction is not improved to any appreciable extent by the wetting agent. The fish meal used in this experiment was the same as that used in the laboratory.

The conditions that were found to be best for the extraction of fish meal with trichloroethylene were 130°F., extraction time of 28 min., a moisture content of the feed of 6.5%, and a miscella concentration of 15%. The residual extractables under these conditions were 1.27%. The results of the extraction run with commercial hexane are given in Table II.

TABLE II  
Extraction with Commercial Hexane

Extraction temperature.....	130°F.
Extraction time.....	26.0 min.
Miscella concentration.....	4.04 %
Moisture in feed.....	23.09%
Residual extractables, hexane soxhlet.....	3.18%
Residual extractables, trichloroethylene soxhlet.....	3.71%
Residual extractables (trichloroethylene extraction), trichloroethylene soxhlet <sup>a</sup> .....	1.62%

<sup>a</sup> Data corrected to 4.04% miscella so that a more realistic comparison could be obtained. Residual extractables on moisture-free basis.

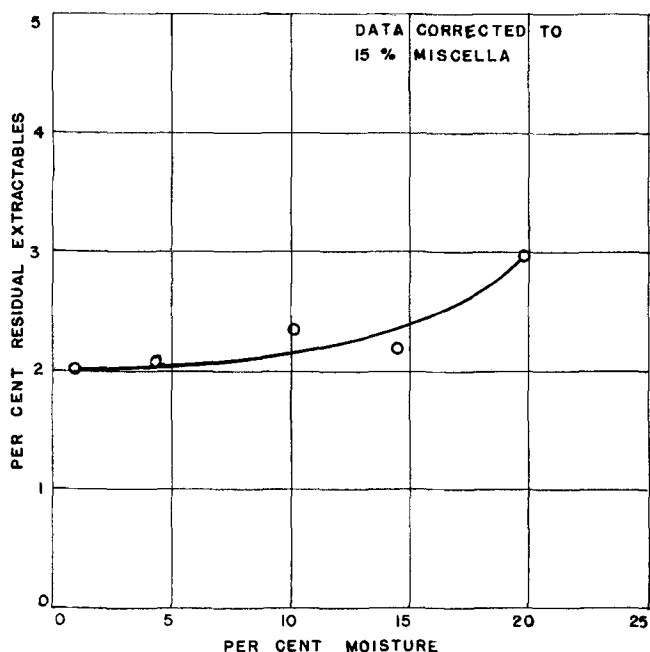


Fig. 7. Effect of moisture on residual extractables in pilot plant.

Extraction time: 26 min.  
Extraction temperature: 110°F.  
Residual extractables on moisture-free basis.

The extraction with hexane shows conclusively that extraction of fish meal is best carried out with trichloroethylene if as great an oil recovery as possible is to be obtained under a given set of extracting conditions. The actual difference in the results is due to the difference in the rate of solution of the oil com-

ponents in the solvents, as has been suggested by Karnofsky (6).

The toxicity to cattle of certain batches of trichloroethylene-extracted soybean oil meal has raised the question of possible toxicity of other products extracted by trichloroethylene. Since the work presented in this paper was a study in extraction only, the use of trichloroethylene as an experimental solvent should not be construed as a recommendation by the authors that the product resulting from this extraction is or is not suitable as a feed.

### Summary

Data are given which show the effect of the various extraction variables on the extraction of fish meal in both the laboratory and pilot plant. The use of wetting agents, although promising in the laboratory, does not appear to be of much value for pilot plant operation. A higher percentage of the oil components is removed by trichloroethylene under a given set of extracting conditions than with hexane. The mechanism of the extraction is believed to be one of surface-washing for short extractions, and for longer extractions this washing process is supplemented by diffusion.

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## Adsorption of Synthetic Detergents Affects Use and Waste Disposal<sup>1</sup>

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**D**ETERGENTS are members of a broad class of compounds known as "surface-active agents," so-called because they are withdrawn from water solution by surfaces of all types. The detergent molecules, attracted from the solution to the surface, orient themselves with their water-solubilizing groups directed toward the solution and the water-repellent groups toward the surface. In this way an entirely new surface is created. This surface effect, known as "adsorption," gives rise to the washing, wetting, sudsing, dispersing, and other properties associated with detergents.

In ordinary use the amount of detergent which migrates to the surface is no inconsiderable part of that which is present. It may in fact involve most of the detergent present in solution. Furthermore the detergent which becomes associated with the surface may be fixed there in a way that serves to alter the properties

of the treated material even after it is removed from detergent solution.

### Adsorption Needs to Be Understood

This discussion is concerned with a description of the magnitude of these surface effects and with the manner in which they can be promoted or retarded, according to the particular effect which the user wishes to achieve.

The economical and effective use of detergents, especially in industry, requires a clear understanding of the effect which the detergent is intended to achieve and whether the effect is to be temporary, as in washing where it must last only until the dirt is floated away, or whether it is to be more permanent, as in textile finishes or in processes for sterilizing and mothproofing. It also requires a knowledge of the bond between the surface and the detergent. The detergent may be loosely bound so that the bulk of it can be rinsed away by fresh water; or it may be firmly bound

<sup>1</sup> Presented at the fall meeting, American Oil Chemists' Society, Minneapolis, Minn., Oct. 11-13, 1954.