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 critèria.

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.

Acknowledgment

Grateful appreciation is expressed by the authors to Mississippi Cottonseed Products Company and Central Soya Company Inc. for furnishing the soybean raw materials for this investigation; to N. H. Kruse and N. H. Witte of Central Soya Company Inc. for assistance in evaluation of the oil and meal products; to A. F. Kurtz, Lukenweld Division of Lukens Steel Company, for consultative advice; to E. A. Gastrock, Head, Engineering and Development Section of this laboratory, for his helpful suggestions.

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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 $17\frac{1}{2}$ in. in diameter with $1\frac{9}{16}$-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 $24\frac{3}{4}$ ft. of standard No. 35 roller chain with semi-circular flights, $1\frac{15}{16}$ 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 $2\frac{1}{2}$-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 $7\frac{1}{4}$-ampere, 115-volt

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[1] Presented at the spring meeting of the American Oil Chemists' Society, San Antonio, Tex., Apr. 12-14, 1954.

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variable transformers, which enabled isothermal extractions to be carried out. The lower horizontal Pyrex glass section shown in Figure 1 was replaced for these experiments with a standard steel pipe section which permitted installation of thermocouples along the extractor section from the miscella inlet to the liquid level on the solvent inlet end. A general view of the extractor is given by Figure 1, a section of the chain is shown in Figure 2, and the instrumentation of the unit is shown in Figure 3.

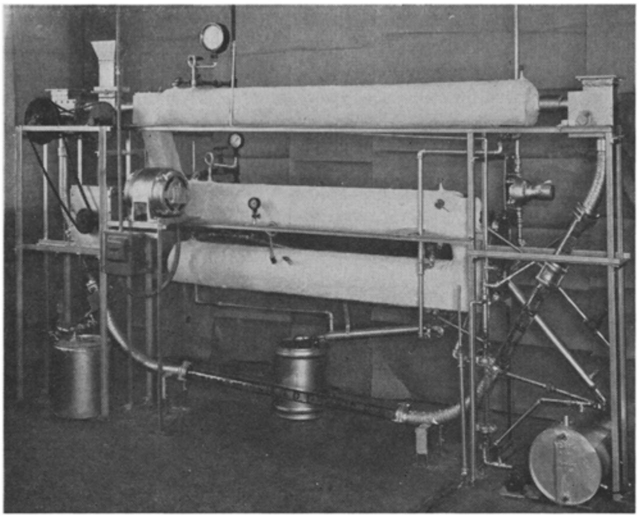

FIG. 1. Pilot plant extractor.

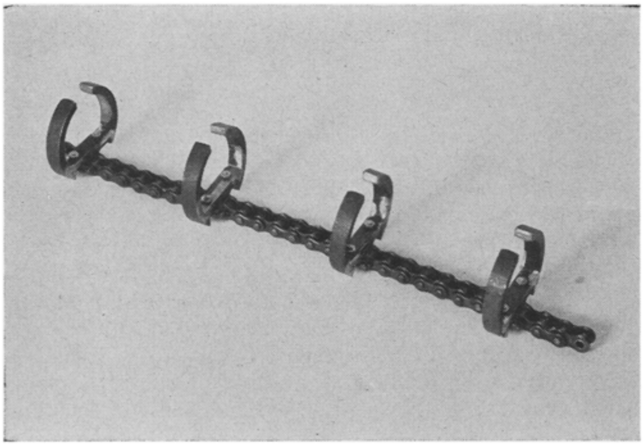

FIG. 2. A short section of the extractor chain.

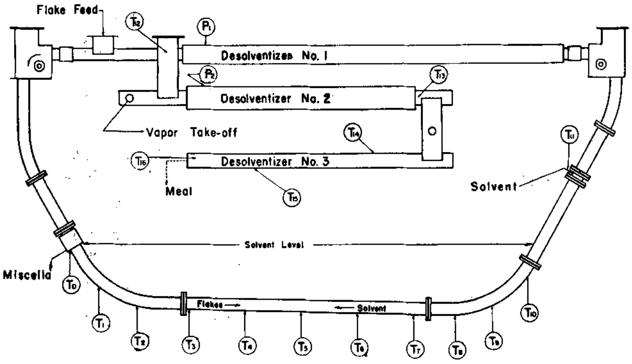

FIG. 3. Diagram of extraction unit showing instrumentation.

Experimental Procedure

Experimental countercurrent extractions were carried out to study the effect of various extraction variables on the residual trichloroethylene extractable material remaining in the extracted flakes. These experiments were divided into "series," in each of which the effect of one of the extraction variables was studied. The variables considered were: oil concentration in the miscella, extraction temperature, flake thickness, extraction time, meat diameter, and moisture content of the flakes. The general operational procedure for all runs was essentially the same, consisting of three steps: preparation of the flakes, extraction of the flakes, and analysis of the extracted flakes.

After the dehulling and the separation of the hulls, the meats were tempered for five minutes and flaked. Although every effort was made to produce flakes of the same thickness for all series some variation, largely the result of variation in meat size between series, resulted.

Extraction. Prior to the start of the actual extraction, a warm-up period was required during which the desolventizers and extractor loop were brought up to the desired operating temperatures. After this warm-up period the extractor conveyor was started. The starting times for feeding the flakes and solvent were adjusted so that miscella started to flow from the unit at the same time the first extracted flakes were being conveyed out of the solvent. The feed rate of the flakes was measured by recording the time required to feed 4 lbs. of flakes. Approximately 2 hours after the feeding of flakes began, the unit was brought to steady state conditions of temperature and miscella concentration, the latter being determined by specific gravity readings on the overflow miscella. Data were now recorded and samples taken every half hour. The length of individual runs varied, the minimum being the time necessary to produce the 2 gal. of miscella needed to yield an oil sample large enough for complete analysis. The average operating time at steady state conditions was 2½ hours, during which time four sets of data were taken and four sets of samples collected. The samples obtained from a given point were mixed to give a composite sample for the run.

Samples taken were: flakes as fed to the unit, extracted meal entering the No. 1 desolventizer, meal discharged from the No. 1 desolventizer, meal discharged from the No. 2 desolventizer, meal discharged from the No. 3 desolventizer, and overflow miscella from the extractor.

At the end of one run in a particular series the conditions of the next run were set, and the entire unit was again allowed to come to a steady state condition, after which the sampling and data collection processes were repeated. The total time required for a series of runs varied with the number of individual runs from 10 to 16 hours. After each series of runs was completed, the unit was emptied of solvent and solids and cleaned externally and internally in preparation for the next series of runs.

Analytical Methods. Standard methods of analysis (6) were used, with the exception of the determination of trichloroethylene "residual extractables." The procedure used for this determination was similar to that specified for the determination of oil in cottonseed materials except that the solvent used was trichloroethylene.

Results: Trichloroethylene as a Solvent

The feed material to the extractor averaged 38.3% trichloroethylene extractable material and 36.5% commercial hexane extractable material.

Preliminary experimental extractions indicated that while the concentration of oil in the overflow miscella would have considerable effect on the extraction efficiency, the time required to adjust the oil content of the miscella to a constant value in all runs would be excessively long. Therefore the experiments were carried out without attempting an exact control of the miscella concentration, and the results corrected to a standard miscella concentration of 20% by the relation developed from the data obtained in those experiments in which only the miscella concentration varied.

Effect of Miscella Concentration. The effect of the concentration of the oil in the overflow miscella was studied at two extraction temperatures, 103°F. and 122°F., and is shown graphically in Figure 4. Since the viscosity of the extracting miscella increases with an increase in the oil content, the rate of extraction can be expected to decrease, due to the decrease in the over-all driving force. This will result in a higher residual oil or extractable content in the extracted flakes, which is apparent from inspection of Figure 4.

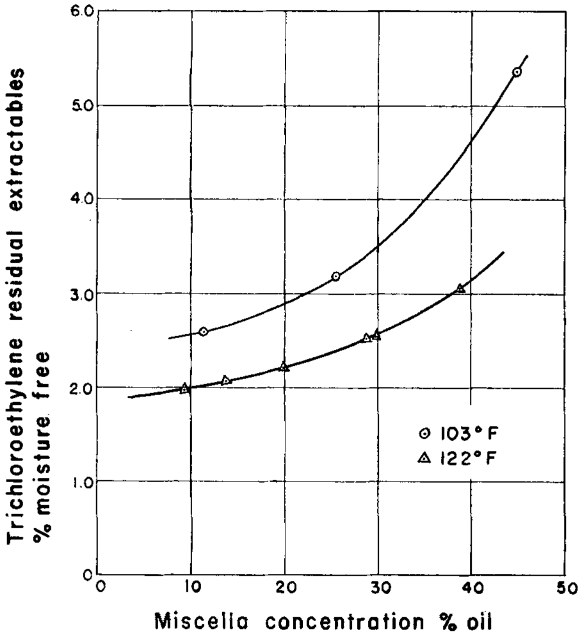

FIG. 4. Relation of residual extractables to miscella concentration at two temperatures.

Effect of Extraction Temperature. The effect of extraction temperature was the second variable studied, since the relation of this variable and the residual extractables would afford a means of checking the miscella concentration correction procedure. The method used in correcting the residual extractables for varying miscella concentration to a standard value, 20%, was to multiply the actual residual extractables by the ratio of residual extractables at 20% miscella to the residual extractables at the actual miscella concentration taken from the 122°F. curve of Figure 4.

A plot of the extraction temperature *vs.* the corrected residual extractables is given in Figure 5. If we assume these data can be represented by the straight line drawn on this figure, the equation representing the temperature effect is: $R = e^{(2.623 - 0.0137t)}$ where, R is per cent residual extractables, and t is extraction temperature, °F.

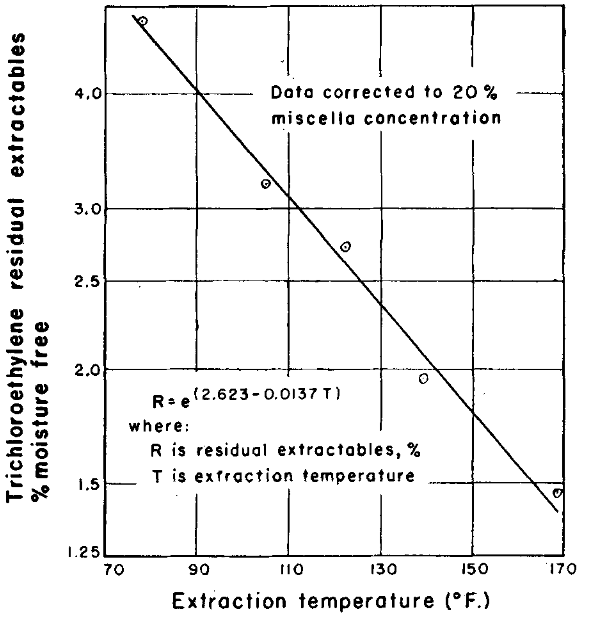

FIG. 5. Effect of extraction temperature upon the residual extractables.

Effect of Flake Thickness and Meat Diameter. Batches of flakes with different average thicknesses were prepared by flaking batches of meats of different diameters. These were extracted, and the residual extractables corrected to a 20% miscella basis. The effect of average flake thickness on the residual extractables is given in Figure 6. The equation of the line drawn through the data is: $R = 0.0132\ b^2$ where, b is average flake thickness, in. $\times 10^3$.

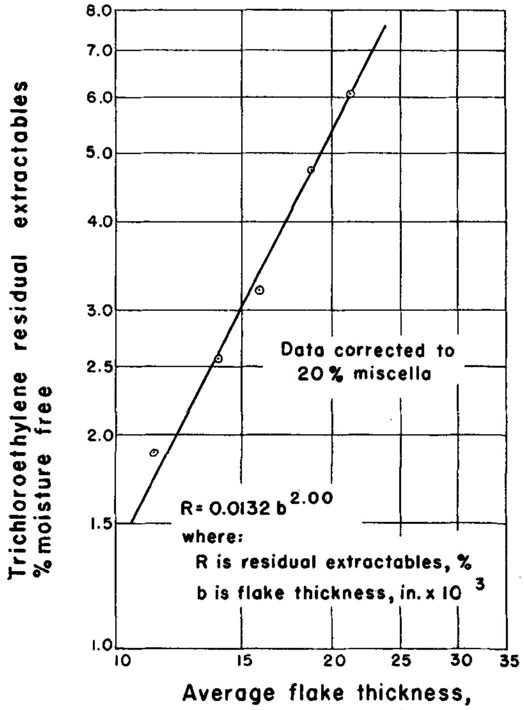

FIG. 6. Effect of flake thickness on residual extractables.

Boucher and co-workers (4) found that the rate of extraction varied inversely with the square of the thickness of the particles being extracted, providing all particles in a given batch or lot had the same thickness. Although the thicknesses used in developing the second equation were average values, there appears to be some theoretical significance to the equation based upon the data of these investigators.

According to the reports of various investigators, there is very little rupturing of the oil cells during the flaking operation, thus any increase in rate of extraction due to flaking of larger-sized meats is probably caused by the greater increase in surface area. The meats may be assumed to be spherical, and, if they are not broken during flaking, will produce flakes which are circular plates with negligible edge area. It has been shown that the residual extractables vary as a power, such as the square, of the flake thickness. It is also obvious that the residual extractables are an inverse function of the surface area. The surface area, in turn, is a direct function of the meat diameter and an inverse function of the flake thickness. Thus we can assume the following relationship where *Dm* is the average meat diameter in inches and *b* is the average flake thickness in inches: $R = c\,b^n/D_m$.

Using the data from those experiments in which the variables were miscella concentration, flake thickness, and meat diameter, and correcting the residual extractables for varying miscella concentration to a value of 20% oil, the assumed relation was checked by plotting the product of the residual extractables and meat diameter against the flake thickness on logarithmic graph paper, as given in Figure 7. The slope of the line drawn through these data is 2.00 and the intercept 0.00153 so the equation can be written as: $R = 0.00153\,b^2/D_m$.

Effect of Extraction Time. The extraction time was varied by changing the speed of the conveyor by changing the size of sprocket used on the main drive. The effect of extraction time on the residual extractables is shown in Figure 8, the equation of the line being: $R = 70.5\ \theta^{-0.995}$, where θ is the extraction time in minutes.

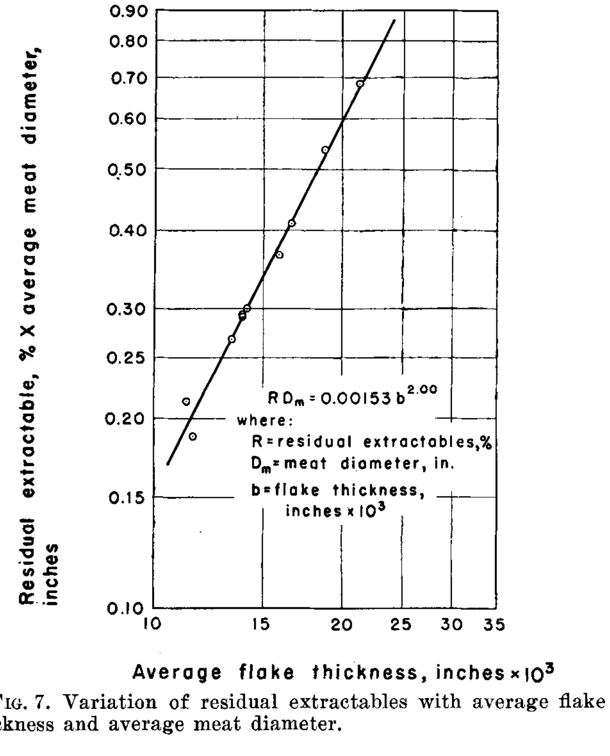

FIG. 7. Variation of residual extractables with average flake thickness and average meat diameter.

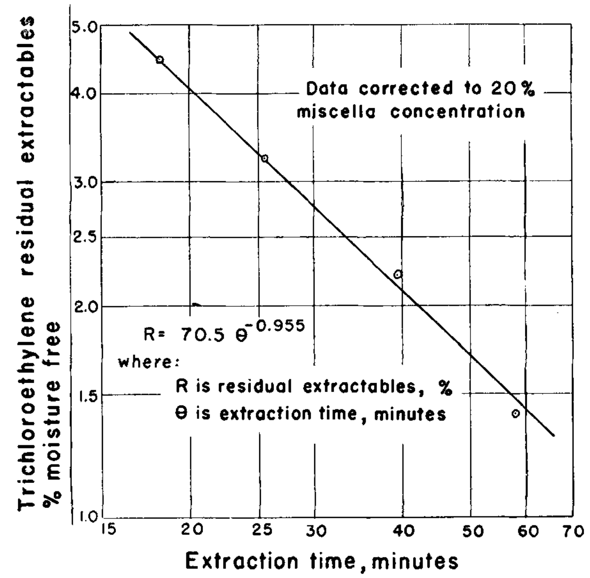

FIG. 8. Effect of extraction time upon residual extractables.

Effect of Flake Moisture Content. When the moisture content of the flakes was varied and the results were calculated to 20% miscella concentration and a flake thickness of 0.011 in., the residual extractables increased from 1.70% at 8.64% moisture to 4.21% at 20.13% moisture. The extraction time was 25.5 minutes and the temperature 122°F. Although carried out with lower solvent to flake ratios and much higher oil content in the miscellas than the rate extraction studies of Arnold and Patel (2), the results show the same general trend over the moisture range studied.

Correlation of Extraction Variables. An equation expressing the residual extractables as a function of the extraction variables is desirable for estimation of the extraction efficiency under various processing conditions and to check the consistency of the data obtained for the previously discussed experiments. It is very possible that variations in the growing conditions and storage conditions of the cottonseed will affect the physical properties of the seed, which in turn will affect the relations of the residual extractables with the extraction variables.

A correlation of the data from the experiments in which miscella concentration and extraction temperature were the main variables was carried out first; the values of the residual extractables in the temperature experiments were corrected for different flake thickness and meat diameter by applying the equation: $R = 0.00153\,b^2/D_m$. Expressing the miscella concentration in terms of the kinematic viscosity and plotting the residual extractables in this function gave a satisfactory correlation. The data used to develop this correlation are given in Table I.

A semi-logarithmic plot showing the variation of residual extractables with the kinematic viscosity of the overflow miscella is given on Figure 9. The data from the experiments in which the extraction temperature was constant gave parallel lines with a slope of 7.15; therefore parallel lines were drawn through the single points for the other temperature, and the

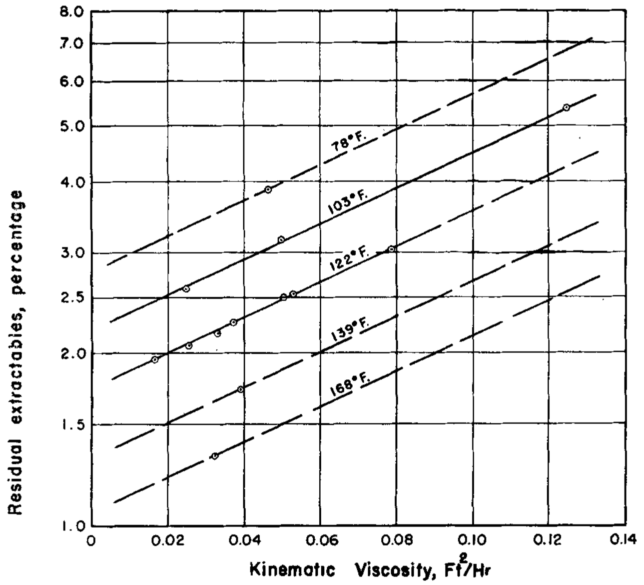

FIG. 9. Variation of residual extractables with the kinematic viscosity of the miscella.

slope is independent so the equation representing the data can be written as:

$$\text{Ln R} = 1.969 - 0.0117t + 7.15\,\mu/\rho$$
$$\text{or R} = 7.16\, e^{(7.15\mu/\rho - 0.0117t)}.$$

By assuming that the combined effect for the variables would be the product of their individual effects, the following equation was proposed and tested for consistency with the data:

$$R = m + n\left(\frac{b^2}{D_m\,\theta^{.995}}\right) e^{(7.15\mu/\rho - 0.0117t)}.$$

Values of *R* and the right hand expression of the equation were plotted to give a straight line. By using the method of least squares, the values of *n* and *m* in equation 8 were calculated and found to be 26,000 and 0.0039, respectively. The coefficient of correlation of the line represented by these constants is 0.988.

Since the value of *m*, 0.0039, is small in comparison to the values of *R*, it can be neglected, and this will result in the following equation:

$$R = 26{,}000\left(\frac{b^2}{D_m\,\theta^{.995}}\right) e^{(7.15\mu/\rho - 0.0117t)}$$

where *R* is residual extractables, % moisture free basis; *b* is flake thickness, ft.; D_m is meat diameter, ft.; θ is extraction time, hours; μ is viscosity, lb./ft.-hr.; ρ is density, lb./ft. (3); and *t* is extraction temperature, °F.

The calculated values of the residual extractables for the conditions of the various runs had an average error of 3% when compared with the experimental values. As stated previously, the equation is probably valid only for the cottonseed meats used in these experiments. However the fact that such an equation could be developed from the data and that it has the accuracy that it does indicates that the data are fairly consistent.

TABLE I

Viscosity Data Used in Developing Equations

Main Variable	Residual Extractables, % R	Miscella Concentration, % X	Extraction Temp., °F. t	Kinematic Viscosity, Ft²/hr[a] u/p
Miscella concentration	2.60	11.28	103	0.0249
	3.16	25.65	103	0.0497
	5.36	44.90	103	0.1251
Miscella concentration	1.95	9.48	122	0.0166
	2.07	13.64	122	0.0252
	2.16	19.70	122	0.0330
	2.51	28.92	122	0.0505
	2.55	29.91	122	0.0529
	3.05	38.76	122	0.0789
Extraction temperature[b]	3.94	20.85	78	0.0461
	2.67	22.05	105	0.0418
	2.30	22.35	122	0.0374
	1.76	25.90	139	0.0390
	1.35	26.20	168	0.0326

[a] Calculated by equations presented in reference 1.

[b] Corrected by the extraction time equation to give a feed material consistent with that used for obtaining the data from the experiments in which the miscella concentration was the main variable.

Results: Hexane as a Solvent

The results secured with trichloroethylene as a solvent can be expected to apply with certain limitations to hexane. The rate of extraction of cottonseed oil by hexane, as shown by Karnofsky (5) and others, becomes, like that by trichloroethylene, more rapid with increasing temperature. The experimental extraction with hexane was therefore carried at 130°F., which was considered the highest practical operating temperature under the experimental conditions. Flake thickness likewise may be considered to affect extraction with hexane much the same as that by trichloroethylene. The thickness used was 0.011 in. The time, 25.5 minutes, used for all but one extraction was that used for most of the extractions with trichloroethylene. The moisture content of the flakes was 6.49%.

Effect of Miscella Concentration. The solvent-feed ratio was varied and the time, flake thickness, flake moisture, and extraction temperature held constant to give the miscella concentration-residual oil relationship shown in Figure 10. Data from a similar extraction with trichloroethylene at 122°F. are also shown for comparison. These data were determined by the official A.O.C.S. method for residual oil, rather than by the method previously described for determining "residual extractables" with trichloroethylene.

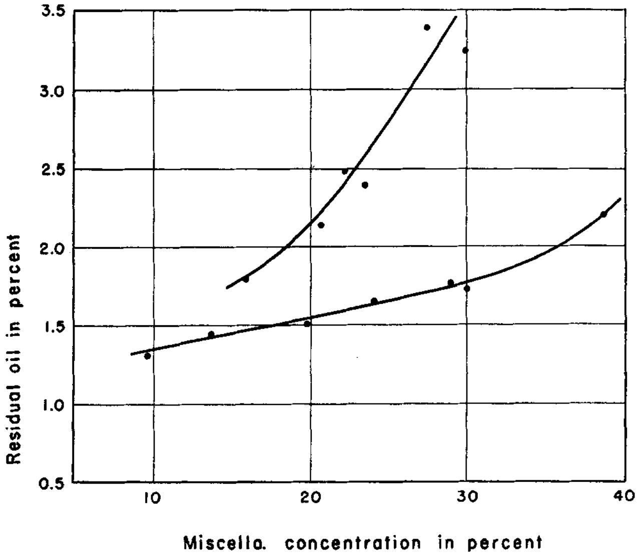

FIG. 10. Relation of miscella concentration and residual oil. Solvent in upper curve: hexane; lower curve: trichloroethylene.

Discussion. While the equipment used in this extraction work was designed specifically for trichloroethylene as a solvent, it worked well with hexane. While the equation developed with suitable constants would probably apply to hexane as well as trichloroethylene extraction, sufficient data are not available to demonstrate this. In general, it may be observed that hexane is inferior to trichloroethylene as a solvent for cottonseed oil. The effect of change of miscella concentration on the amount of residual oil in the meal is greater in hexane extraction than in trichloroethylene extraction.

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

In the experimental countercurrent extraction of flaked cottonseed meats by trichloroethylene the residual oil content of the extracted flakes decreased with: first, a decrease in the final oil content of the final miscella; second, decrease in the flake moisture down to 8.64%; third, decrease in flake thickness; fourth, increase in temperature; and fifth, increase in extraction time. For the batch of cottonseed meats used the following equation was developed:

$$R = 26{,}000\ (\frac{b^2}{D_m\,\theta^{.995}})\ e^{(7.15\,\mu/\rho - 0.0117t)}$$

where *R* is percent residual extractables, *b* is flake thickness in feet, *D* is meat diameter in feet, θ is extraction time in hours, μ is viscosity, lb. per ft. hr., ρ is density, lb. per cu. ft., and *t* is extraction temperature in degrees F.

Not enough data were secured by extraction with hexane to check the equation developed for trichloroethylene extraction. Hexane is a poorer solvent for cottonseed oil than trichloroethylene. The amount of oil remaining in the meal is affected to a greater extent by the miscella concentration in hexane extraction than in trichloroethylene extraction.

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[Received April 20, 1954]

Summary of Study of Economies of Cottonseed Oil Mills[1]

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IN 1948 the Secretary of Agriculture's Cotton and Cottonseed Advisory Committee recommended that a study be undertaken to determine what types and sizes of cottonseed oil mills would constitute the most economical industry.[2] It also desired a determination of the extent to which industry-wide shifts to more economical processes might affect the supply and price of oil and returns to growers of cottonseed.

Four types of processes were involved: the hydraulic, screw-press, direct-solvent, and prepress-solvent. High-speed Expellers were not included as the study was too far under way when this development came into the picture. The same was true of the filtration-extraction process.

Mills of the four processes were nowhere in operation under the same conditions; therefore their comparative economies could be determined only by designing model mills and checking their elements against the experience of well-operated mills.[3] These mills were assumed as operating at their normal rates.

It was necessary to compare the net revenue yields of the four processes at specified crushes under comparable operating conditions. The comparisons were made in terms of 1949-50 cost-price relationships, the most recent period for which all needed data were available.[4]

The study led to six main findings. 1. Cost of diverting seed from competitors usually puts a relatively low limit on the sizes of mills, whatever the type. 2. In general, for widely different specified volumes of seed at uniform seed prices, the higher oil-extraction types of processes yielded greater net revenue per ton of seed. 3. Either of the solvent processes can handle a smaller crush without losing money than can the hydraulic process. 4. With each process operating at its normal rate on 1949-50 average quality seed, an industry-wide shift from the hydraulic process to the prepress-solvent process would increase the supply of cottonseed oil by approximately 10.8% and of all edible oils (exclusive of butter and lard) by 5.4%. Similar shifts to the direct-solvent and screw-press processes, respectively, would increase cottonseed oil by 9% and 2.1% and all edible oils by 4.5 and 1.1 %. 5. As a consequence, industry-wide shift from the hydraulic to the prepress-solvent process would reduce the price of cottonseed oil by approximately 8.9% as compared with reductions of 7.6% and 1.8% for the direct-solvent and screw-press processes, respectively. 6. As measured by change in size and type of the industry's marginal mill (or mills), industry-wide shift to more efficient mills of whatever type could benefit growers in the form of higher seed prices. But shifts to a more efficient hydraulic industry could benefit growers more than similar shifts to either of the solvent industries although the latter would be more beneficial to consumers.

[Received April 21, 1954]

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[1] Presented at annual meeting of American Oil Chemists' Society, Apr. 12-14, 1954, San Antonio, Tex.

[2] John M. Brewster, "Comparative Efficiencies of Different Types of Cottonseed Oil Mills and Their Effects on Oil Supplies, Prices, and Returns to Growers," U. S. Department of Agriculture, Agricultural Marketing Service, Washington, D. C., Marketing Report No. 54, 1954.

[3] Data for this phase of the study were developed by A. Cecil Wamble and S. P. Clark of the Cottonseed and Cottonseed Products Research Laboratory of the Texas College of Agriculture.

[4] These prices contributed to conservative conclusions because the price of oil (approximately 11.5 cents per pound for prime crude) in that year, as compared with other years, was low in relation to the prices of other cottonseed products and also the costs of most production factors.