

trol this through organoleptic means; however a more objective means, based upon detection of offending compounds, might be more useful.

### Stability of Fried Product

In the case of yeast raised and cake doughnuts we are concerned with retention of moistness in the product. It has been observed that there is variation in effect of fat on this property. While this has not been studied carefully, it appears that fats with low melt point fractions, such as lard, do exert a greater effect upon moisture retention than other fats. This, while not a real problem, is an element to consider in frying fat investigations.

### Summary

The user of frying fats must consider a number of elements in choosing the fat to be utilized. The relationships between fat composition and these factors appear not to be clearly known. Much can be con-

tributed to frying technology through work in the field of fat composition and its relationship to quality frying factors.

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## Effect of Heat on Solvent-Extracted Soybean Oil Meal

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ALTHOUGH all commercial methods of extracting oil from soybeans involve the application of some heat, the effect of this heat on the soybean oil meal protein is not well known. Since over 98% of the soybean oil meal produced in this country in the last three years was used as a protein supplement in livestock and poultry feeds (14), it is important to both the processors and consumers that it be given the proper heat treatment to obtain maximum nutritional value. Just what constitutes this optimum heat treatment is not known, but reports of feeding studies indicate that certain chemical characteristics of soybean oil meal may be correlated with its nutritional value when used in feeding chicks. While there is no indication that these correlations apply to other animals than chickens, it has seemed desirable to study the effect of heat on these chemical characteristics.

### Commercial Processing

While in general the heat treatment received by the meal in the various solvent extraction processes is similar, the commercial processing data considered in this paper are specifically those from a 25-ton per day plant (Figure 1) of the type described by Sweeney and Arnold (13). All of the experimental results were obtained with trichloroethylene-extracted meal.

The first application of heat was the "tempering" treatment given to the cracked soybeans to render them plastic for flaking. Since the beans were not heated over 160°F. and for not more than 5 to 10 minutes, this tempering had little effect on the final characteristics of the meal. The same could be said for the slight heating the flaked beans underwent during the extraction where the retention time was 30 minutes and the highest temperature 135°F. The third heat treatment occurred in the desolventizers or dry-

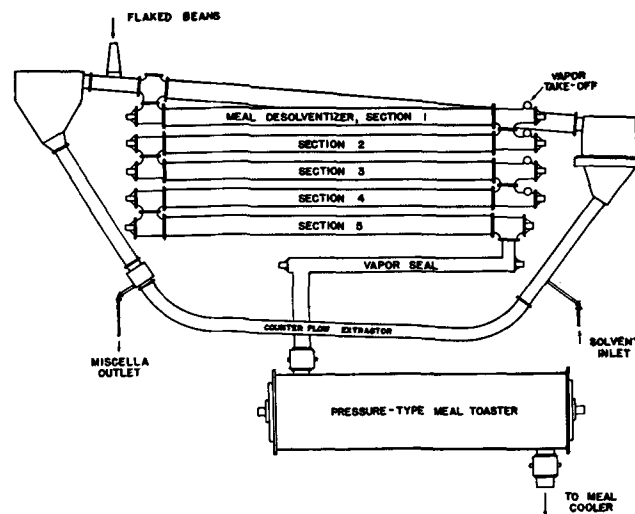


FIG. 1. Schematic flowsheet of commercial unit.

ers, which together with the extracting and toasting portions of the plant are shown in Figure 1.

The extractor was essentially a loop of 12-inch pipe about 20 feet high and 45 feet long through which the flaked beans were slowly conveyed by means of a continuous chain conveyor. The solvent entered the extractor at the opposite end to the flakes and flowed through the flakes counter-currently, emerging as a miscella at a point in the extractor loop below the flake entrance. The extracted flakes were carried up and out of the solvent into the horizontal top run of the loop, which was steam-jacketed to preheat the extracted flakes before they dropped into the desolventizer.

The desolventizer consisted of five steam-jacketed, 12-inch tubes through which the meal was conveyed and agitated by modified ribbon-type screw convey-

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ors. The five sections were arranged in a vertical bank, the meal progressing the length of each section before dropping into the section below. A small amount of stripping steam was introduced at the discharge end of the fifth section. The essentially trichloroethylene-free meal was discharged from the lower section through a special vapor seal directly to the toaster.

The pressure-type of toaster consisted of a steam-jacketed cylindrical vessel, approximately 15 feet long and 3 feet in diameter. The flakes were continuously added through a barrel valve and were agitated and gradually moved through the vessel by a paddle-type conveyor. From 5 to 10 pounds per square inch direct steam pressure were maintained in the chamber to cook or "toast" the meal.

The meal left the toaster through another barrel valve, was cooled in an open conveyor, was ground in a hammer mill, and was elevated to a hopper for bagging and shipping. The final product was a golden brown, granular, high-protein content meal, containing less than 0.5% residual oil.

### Optimum Heat Treatment

Heating soybean oil meal, particularly in the presence of moisture, results in a partial denaturation of the protein, the amount depending upon the temperature, time of exposure, and the amount of moisture present. In addition, certain undesirable enzyme and anti-enzymatic systems, naturally present in the meal and themselves of a protein nature, are inactivated. As a result, heating up to a certain point increases the nutritional value of the meal as a feed for chickens. Another desirable result is said to be the effective debittering of the meal, making it more palatable (11).

If the heating is continued beyond this optimum point, coagulation occurs, resulting in the protein becoming less available and thus producing a meal of lesser nutritional value (4, 7). The problem facing the soybean processor, then, is one of giving sufficient heat treatment to the meal to insure desirable feed characteristics, while avoiding undesirable overheating.

Numerous tests and criteria have been applied to determine when a meal possesses its optimum nutritional value. These may be classified as either animal feeding tests or analytical chemical tests. Feeding tests on soybean oil meal relating heat treatment to nutritional value, for the most part carried out using either chicks or rats, have not, as pointed out by Bird (2), provided conclusive information regarding the ideal conditions for the commercial processing of soybean oil meal. Animal feeding tests, while fundamental in determining the relative efficiency of soybean oil meal feeds, are of limited value to the processor in controlling his plant operating conditions. It was therefore considered desirable to study analytical tests which could be used in the evaluation of soybean oil meal.

### Protein Fractionation

Since it is the protein portion of soybean oil meal that is primarily responsible for its value as a feed, analytical tests used as a measure of nutritional value have been based on protein chemistry. The problem has been essentially one of measuring the nature and degree of protein denaturation existing in the meal and has normally been done using a system of protein

fractionation according to the classical scheme first proposed by Osborne (8). The protein was separated into different fractions through the use of different solvents and the principle of selective solubility. The percentage of each fraction varies with the degree of protein denaturation or, in this case, heat treatment.

One of the recent and more comprehensive investigations carried out on this basis was that of Evans and St. John (5), who separated the protein in soybean oil meal into five different fractions. They applied their method to several commercially prepared meals and to a limited number of meals prepared in the laboratory and subjected to different degrees of heat treatment. A coefficient of correlation of  $+0.928$  was observed between the weight gain figures for chicks fed these meals and the percentage of the protein appearing in the glutelin fraction (soluble in 0.2% KOH). This suggested that here was a basis for analytically relating nutritional value of soybean oil meal for chick feeding to processing conditions.

In this investigation the general method of Evans and St. John was applied to a large number of trichloroethylene-extracted soybean oil meal samples prepared both in the laboratory and the commercial plant in order to complete the relationship between the glutelin protein fraction and processing conditions. In the laboratory the effect of dry and moist heat and the solvent itself were considered.

The laboratory samples were prepared, using soybeans of the Hawkeye variety which were cracked, flaked, and extracted at room temperature in such a manner as to duplicate commercial processing conditions. The wet flakes were air-dried at room temperature to remove all the solvent and then subjected to different degrees of heat treatment, using the specially built heating apparatus shown in Figure 2. When used as a toaster, the apparatus had steam supplied to both the jacket and the chamber. When dry heat was desired, only jacket steam was employed. In use, the apparatus was brought up to operating temperature and the meal sample then introduced through the

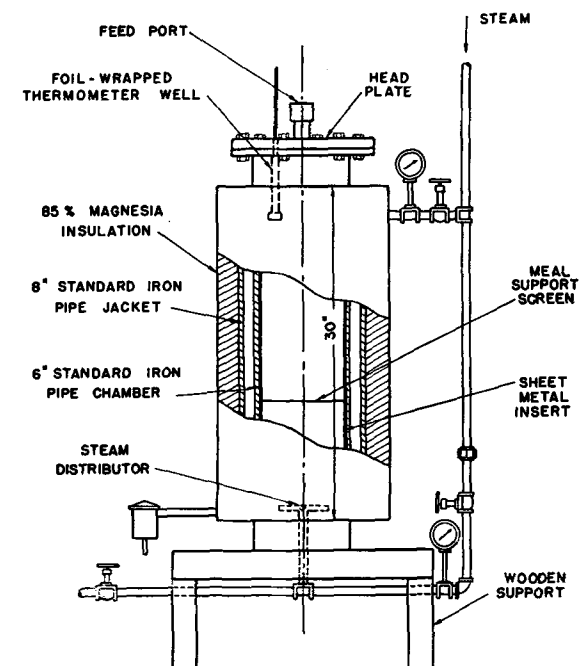


FIG. 2. Laboratory heat treating equipment.

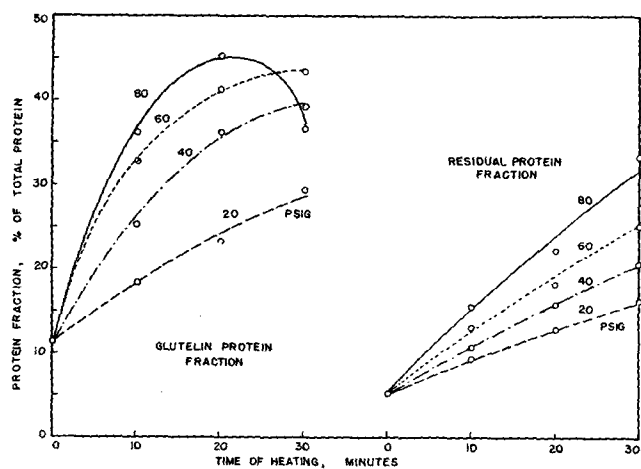


Fig. 3. Effect of dry heat on the glutelin and residual protein fractions in trichloroethylene-extracted soybean oil meal.

feed port and allowed to fall down onto the fine mesh support screen. At the end of the heating period the head plate was quickly removed and the sheet metal insert containing the sample on the support screen lifted out. The sample was immediately dumped out and allowed to cool before being ground and analyzed. The moisture content of the toasted samples as they were removed from the apparatus varied from the 13 to 18% of the dry meal weight, again corresponding to commercial practice. The final moisture content of the samples subjected to dry heat varied from 6 to 10% of the dry meal weight, depending upon the length of the heating period. The original moisture content averaged 11.30%.

The glutelin and the residual protein fractions were separated by a modification of the method used by Evans and St. John (5) and suggested by Sealock (9). The portion of the ground sample passing a 40-mesh screen and being retained on a 60-mesh screen was used for the analysis. Three- to four-gram samples were extracted in 50-ml. centrifuge tubes, using 40 ml. of solvent for each extraction. Following a 30-minute wetting period in which the samples were allowed to stand in contact with distilled water, the samples were extracted twice using distilled water, three times using a 5% KCL solution, twice more using 70% ethyl alcohol, and then five times using 0.2% KOH solution. Each time the meal and solvent were thoroughly stirred together, centrifuged, and the supernatant liquid decanted off. The five KOH extracts, containing the glutelin protein fraction, were made up to 250 ml. and a 50-ml. aliquot used for a conventional Kjeldahl analysis for organic nitrogen which was converted to protein, using a factor of 6.25.

The material remaining in the centrifuge tube at the end of the final KOH extraction was considered as containing the residual protein fraction and was washed into a Kjeldahl flask and also analyzed for protein. The residual protein fraction was selected in place of the more popular water-soluble fraction because of its more uniform change with heat treatment. The first three fractions were discarded without analysis.

The results of these tests are presented in Figures 3, 4, and 5. In the dry heat tests, Figure 3, jacket steam pressures of 20, 40, 60, and 80 psig. were combined with heating periods of 10, 20, and 30 minutes to give 12 different processing conditions. Both the

glutelin and residual protein fractions initially increased with heat treatment. The glutelin fraction tended to reach a maximum value however, beyond which additional heating caused it to decrease. These results are similar to those reported by Evans and St. John (5), using moist heat, and if the analogy is complete, the maximum glutelin value corresponds to the point of maximum nutritional value when the meal is fed to poultry. The type of heat treatment to which these meals were subjected is similar to that received in commercial desolventizing equipment once the meal has entered the falling-rate portion of the drying operation.

In the moist heat or toasting tests a uniform jacket steam pressure of 30 psig. was employed and different direct steam pressures ranging from 5 to 20 psig. Heating periods varied up to 60 minutes in length, depending upon the severity of the toasting conditions. Referring to Figure 4, it is seen that regardless of the steam pressure employed in the chamber, a constant maximum glutelin protein percentage of approximately 53% of the total protein present was eventually attained. The heating time required to reach this value varied from 5 minutes at 20 psig. to 45 minutes at 5 psig. direct steam pressure. It is also of interest that the maximum glutelin percentage in the case of dry heat was only 45%, a value significantly lower than the 53% figure found in the toasting tests. On the basis of the correlation of Evans and St. John (5) between glutelin and nutritional value, we can assume that, within the processing ranges investigated, meal of maximum feed value for chicks can be produced only through the application of moist heat. In addition, if one assumes that the findings of Evans and St. John are valid at direct steam pressures other than the one investigated by them (15 psig.), it can be concluded that meal of equivalent nutritional value can be produced at any of a large number of combinations of direct steam pressure and toaster retention time. For each direct steam pressure however there is only one corresponding time which will produce a meal at its maximum nutritional value, assuming a constant jacket steam pressure.

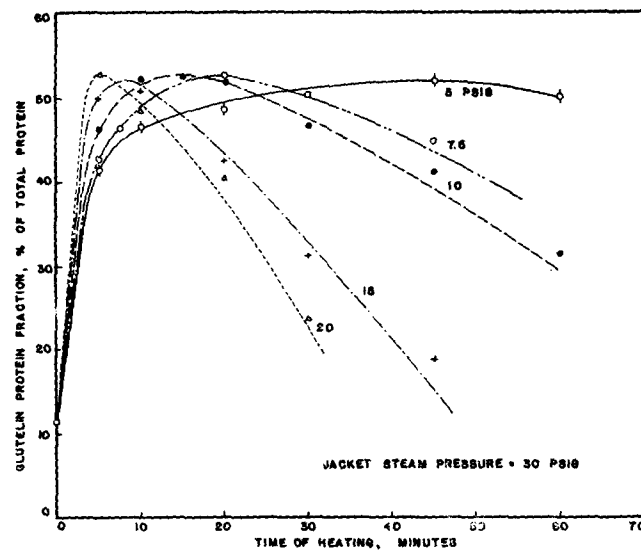


Fig. 4. Effect of moist heat on glutelin fraction of soybean oil meal.

TABLE I  
Characteristics of Soybeans and Meal at Various Stages of Commercial Processing

Sample	Moisture % of Dry Meal	Protein % <sup>a</sup>			Enzyme Activity	
		Water- Dispersible	Glutelin	Residual	Urease Change in pH	Lipoxidase % of Original Activity
Cracked whole beans.....	16.80	89.90	11.63	3.46	2.11	100.0
Whole beans, cracked and tempered.....	15.02	84.60	16.88	4.11	2.10	87.5
Whole beans, cracked, tempered and flaked.....	15.10	84.30	16.70	4.07	2.20	90.1
Extracted flakes, leaving extractor section.....	14.90	80.60	18.44	10.30	2.17	68.2
Extracted flakes, entering desolventizer section No. 1.....	14.80	69.20	20.02	14.62	2.18	36.0
Extracted flakes, entering desolventizer section No. 2.....	12.61	69.0	17.43	13.60	2.17	62.1
Extracted flakes, entering desolventizer section No. 3.....	8.25	72.8	17.62	14.38	2.12	63.1
Extracted flakes, entering desolventizer section No. 4.....	7.52	74.40	22.02	10.57	2.11	66.6
Extracted flakes, entering desolventizer section No. 5.....	9.34	70.60	25.02	10.90	1.88	3.6
Desolventized flakes, leaving desolventizer section No. 5.....	6.38	62.80	35.25	14.90	1.25 <sup>b</sup>	0.0
Toasted meal, leaving toaster.....	13.46	8.64	50.50	40.60	0.13	0.0

<sup>a</sup> Expressed as a percentage of the total protein present, 0.54 gram protein per gram of oil-free, moisture-free solids. Because of the fact that the three classifications are not mutually exclusive, the sums of the percentages in some cases are greater than 100%.

<sup>b</sup> Values from 0.81 to 1.90 found.

The relative values for the different fractions in the protein solubility curves depend upon the analytical method used in their separation. In the glutelin fraction even with certain variations in the analytical procedure the maximum was attained in approximately the same place, but the entire curve was offset from the corresponding curves in Figures 3 and 4. It should also be noted that the type of equipment used determines to a great extent the amount of protein degradation occurring in a specified length of time and under the same conditions of temperature and moisture content. The curves shown in Figure 4, for example, could not be applied directly to a piece of commercial toasting equipment because of such differences between it and the laboratory equipment as the amount of radiation and heating surface contact for unit weight of meal. They show however the importance of proper relation of time and pressure to obtain the maximum glutelin value. Exact values for these can be determined for specific equipment by the use of the analytical procedure outlined.

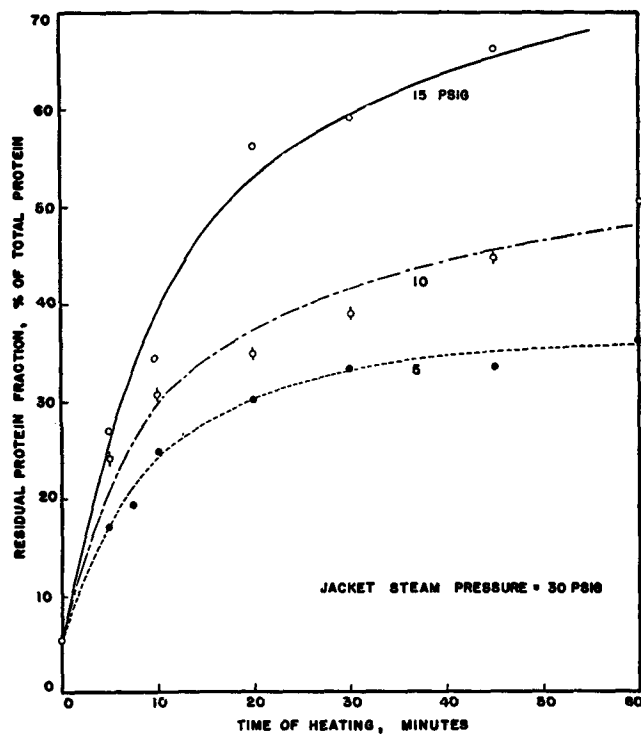


FIG. 5. Effect of moist heat on the residual protein fraction in soybean oil meal.

As a check on the effectiveness of the commercial equipment in producing a properly heat-treated meal, a series of samples were taken for analysis at various stations in the commercial plant. These stations, indicated in Table I, were selected so that the samples would show the changes occurring in each step in the process. The samples were taken at time intervals so that one particular batch of beans was followed through the plant from raw beans to finished meal. The average operating conditions at the plant when the samples were taken are given in Table II.

TABLE II  
Average Operating Conditions at the Commercial Plant

Flake thickness.....	0.012 inches
Solvent rate.....	3.5 gpm.
Miscella flow rate.....	2.6 gpm.
Steam to tempering tubes.....	30.0 psig.
Steam to desolventizer.....	120.0 psig.
Desolventizer retention time.....	20 min.
Stripping steam rate to desolventizer.....	0.13 lb./min.
Steam to toaster jacket.....	32.0 psig.
Steam to toaster (internal).....	7.0 psig.
Water feed to toaster.....	20.0 gal./hr.
Retention time in toaster.....	22 min.

The results of the glutelin protein analyses indicate that the meal does not receive adequate heat treatment for maximum nutritional value in the desolventizers alone. The product from the toaster however had a glutelin value nearly as great as the best experimentally produced meal.

### Enzyme Activity

Two enzyme systems, urease and lipoxidase, are also significant in determining the nutritional value of soybean oil meal. Urease activity can be conveniently determined using the test developed by Caskey and Knapp (3), the chemical basis for which is an increase in alkalinity produced by the liberation of ammonia from a standard buffered urea substrate solution. The test was originally proposed as a measure of overall nutritional value on the premise that adequate heat treatment will always destroy the enzyme. In practical application however the test has come to be considered more a measure of the anti-tryptic factor present in soybeans. This latter viewpoint has been supported by numerous investigations such as that of Balloun, Johnson, and Arnold (1), which showed that heating soybean oil meal beyond the point where the urease became inactive still in-

creased the biological value of the meal when fed to poultry.

The presence of lipoxidase in soybean oil meal to be used for feed purposes is undesirable because of its vitamin A-suppressing activity. It is capable of destroying both vitamin A and provitamin A pigments by an oxidation coupled with a peroxidation of unsaturated fats (6). Like urease it is deactivated by heat. Its activity is conveniently measured using the method of Sumner (12), a colorimetric procedure utilizing the controlled peroxidation of a standard linolenic acid solution.

TABLE III  
Effect of Dry Heat on the Enzyme Activity of Trichloroethylene-  
Extracted Soybean Oil Meal

Jacket Steam Pressure, psig.	Time of Heating, min.	Urease Activity, Change in pH	Lipoxidase Activity, Colorimeter Reading <sup>a</sup>
0	0	2.24	124
20	10	2.20	66
	20	2.19	45
	30	2.10	0
40	10	2.18	24
	20	2.05	4
	30	1.92	0
60	10	2.08	2
	20	2.04	0
	30	1.61	0
80	10	2.07	0
	20	1.70	0
	30	0.74	0

<sup>a</sup> Determined using the Klett-Summerson colorimeter zeroed on distilled water at 25°C.

The effect of dry heat on the activity of these two enzyme systems is shown in Table III. From these values it is apparent that lipoxidase is by far more sensitive to dry heat than is urease. The lipoxidase activity dropped rapidly to zero whereas the pH change registered in the urease tests did not drop below the critical value of 1.0 set by Caskey and Knapp (3) until the most severe conditions investigated were tried, viz., 80 psig. jacket steam pressure and a heating period of 30 minutes.

Table IV shows the results obtained when moist heat was employed. These data emphasize the effect of moisture in increasing the degree of protein degradation during heating. Lipoxidase activity was very

TABLE IV  
Effect of Moist Heat on the Enzyme Activity of Trichloroethylene-  
Extracted Soybean Oil Meal

Direct Steam Pressure, <sup>a</sup> psig.	Time of Heating, min.	Urease Activity Change in pH	Lipoxidase Activity, <sup>b</sup> Colorimeter Reading
0	0	2.24	124
5	5	1.93	4
	7.5	0.10	2
	10	0.93	1
	20	0.00	0
7.5	5	0.71	2
	10	0.12	0
	20	0.02	0
10	5	0.07	0
	10	0.02	0
15	5	0.04	0
	10	0.00	0
20	5	0.00	0

<sup>a</sup> A jacket steam pressure of 30 psig. was used in all tests.

<sup>b</sup> Determined, using the Klett-Summerson colorimeter zeroed on distilled water at 25°C.

quickly destroyed by moist heat. The urease activity was destroyed rapidly at relatively low steam pressures and short periods of heating.

Urease and lipoxidase activity were also determined on the commercial plant samples as shown in Table I. Both enzymes were observed to decrease in activity during the various processing operations and to follow a definite pattern such as might have been predicted from the results of the laboratory tests. The introduction of moisture into the fourth and fifth sections of the desolventizer in the form of stripping steam, for example, greatly accelerated the breakdown of the two enzyme systems. The meal leaving the desolventizer was found to be urease positive, requiring additional heating in the toaster in order to be considered efficient for feeding poultry.

### Effect of the Solvent

To determine the effect of the solvent itself upon certain characteristics of soybean oil meal, samples of the same beans used in the laboratory tests were allowed to stand in contact with trichloroethylene for different periods of time at room temperature and at the boiling point (86.7°C.). For purposes of comparison identical tests were run, using methylene chloride (boiling pt. of 39.8°C.) and commercial hexane (boiling pt. of 70°C.). (See Table V.) The protein

TABLE V  
Effect of Solvents on Protein Solubility and Enzyme Activity in  
Soybean Oil Meal

Solvent	Sample					
	Raw Beans	Time in Solvent at Room Temp., hours			Heating Time, at Boil. Pt.	
		1	2	24	30	60
Percent Protein in Water-Soluble Fraction <sup>a</sup>						
Methylene Chloride.....	79.3	85.90	91.90	87.70	87.70	85.60
Trichloroethylene.....	79.3	84.00	88.90	86.40	80.40	77.80
Percent Protein in Glutelin Fraction <sup>a</sup>						
Methylene Chloride.....	4.26	2.80	1.43	1.89	2.89	3.62
Trichloroethylene.....	4.26	3.02	1.74	3.16	5.89	6.85
Percent Protein in Residual Fraction <sup>a</sup>						
Methylene Chloride.....	5.82	5.06	4.16	4.65	4.56	5.24
Trichloroethylene.....	5.82	5.61	5.21	5.49	5.39	6.65
Percent of Original Lipoxidase Activity Present						
Methylene Chloride.....	100.0	55.6	50.0	31.4	34.7	20.0
Trichloroethylene.....	100.0	72.5	62.0	58.9	30.6	26.6
Hexane <sup>b</sup> .....	100.0	84.1	81.3	69.8	70.9	59.0
Change in pH in the Urease Test						
Methylene Chloride.....	2.24	2.29	2.29	2.32	2.29	2.30
Trichloroethylene.....	2.24	2.28	2.21	2.23	2.23	2.22
Hexane <sup>b</sup> .....	2.24	2.28	2.30	2.24	2.24	2.24

<sup>a</sup> Expressed as a percentage of the total protein present, 0.512 gram protein per gram of oil-free, moisture-free solids.

<sup>b</sup> Skellysolve-B.

fractionations were accomplished using the same procedure described previously except that the number of distilled water extractions were increased from two to five to insure complete separation of the water soluble fraction.

The initial effect of the chlorinated solvents was one of increased water solubility at the expense of the other protein fractions. A similar effect due to hexane has been reported by Simon and Melnick (10). Prolonged contact for a 24-hour period reversed the initial pattern, resulting in a decrease in water solubility and an increase in the glutelin and residual protein fractions. Where heat was applied, the solu-

bilizing effect of the solvents was slowly overcome by the protein degradation caused by the heating, the net effect depending upon the length of the heating period.

In the case of lipoxidase all three solvents very effectively reduced the activity, methylene chloride showing the greatest effect. With urease the pattern was somewhat different. All three solvents caused an initial increase in activity probably due to an opening up of the protein molecules. Except in the case of the methylene chloride, this was then followed by a decrease in activity upon prolonged contact or heating. The effect of lipoxidase activity is reflected in the commercial plant data shown in Table II.

### Conclusions

As a result of this investigation it appears that if the relationship between the nutritional value of soybean oil meal and its protein glutelin fraction is confirmed for a wider range of processing conditions than those reported on by Evans and St. John (5), there then exists a simple, reliable, and reproducible analytical means of further relating nutritional value to plant processing conditions. For the range of processing conditions normally employed in plant practice, a maximum glutelin fraction percentage is consistently attained with the application of either

dry or moist heat, beyond which additional heat treatment results in a decrease, completing a pattern with heating similar to that normally followed by the nutritional value of the meal. In all cases where moist heat was employed, the activity of both the urease and lipoxidase enzyme systems was reduced to a level satisfactory for feeding prior to the point where the maximum glutelin protein fraction was attained. This is in agreement with numerous poultry feeding tests relating feed value to processing conditions.

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## Solvent Extraction of Oil From Cottonseed Prior to the Removal of Linters and Treatment of the Residue to Effect Separation of Meal, Hulls, and Linters<sup>1</sup>

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THE mechanical delinting of cottonseed is one of the most expensive operations in cottonseed processing. Even so, only about 80 to 90% of the linters on the seed are recovered. Linters are somewhat contaminated with hull particles, and some oil is lost with the hulls. With these apparent shortcomings of present processes as justification, research was conducted by the Texas Engineering Experiment Station to investigate the technical and economic feasibility of solvent extracting the oil from rolled whole seed and then separating the solids residue into meal, linters, and hulls.

### Process

A process was developed which was considered to be technically feasible and can be carried out in the following steps starting with seed from storage: cleaning; cracking of the hulls to facilitate subsequent conditioning and rolling; conditioning by heating and moisture adjustment; rolling or flaking; solvent extraction of the oil, desolventizing of the extracted solids and of the oil; cooling the extracted solids;

separating the solids into a protein or meal fraction with most of the lint still attached to the hulls; separating the hulls-lint fraction into hull bran and lint fiber.

A flow sheet of the process is shown in Figure 1 and is designated No. 1 Seed Process. A material balance for the process is given in Table I.

All of these operations can be carried out in standard equipment. Up to the stage of separation of the solvent-extracted solids into protein and hull fractions, the equipment is the same as that performing similar functions in commercial direct solvent or prepress-solvent extraction plants. Rolling is accomplished with standard one-pair-high flaking rolls. The separation of protein and hulls-lint is carried out by a combination of standard hull beaters, purifiers, and tailings beaters such as are used in the commercial separation of delinted cottonseed hulls and meats. The separation of the hulls-lint into lint fiber and hull bran is effected with commercial hull defibrating machines.

The last operation is the only one which was not investigated experimentally. No tests were made because the project was set up as only an exploratory investigation, and preparation of enough extracted seed for even one test on a commercial defibrating machine would have been costly with the small output

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