Using the data of Figure 7 for ion retardation and the most satisfactory conditions for ion exclusion cited by Prielipp and Keller (2), parallel preliminary economic calculations were made for commercial-size plants. (For these calculations the authors are indebted to James W. Churchman, Technical Service and Development Department, The Dow Chemical Company, Midland, Mich.) The calculations were made basically by method 2 of Aries and Newton (8), in which fixed capital costs are estimated as a percentage of purchased-equipment cost. Equipment costs were derived, after appropriate cost-index corrections, from "Cost of Cation Exchange Equipment" by Peak and David (9), and evaporation costs were derived from an article by D.M. Stromquist and A.C. Reents on ion-exchange purification of glycerine (10).

For a 5-million-pound-a-year plant the total capital investment was $25–30,000 for ion retardation, and the operating costs (including polishing ion-exchange) were about 0.9¢/lb. of the glycerine produced. The comparative costs for ion exclusion were a total capital investment of $60,000–65,000, and operating costs of 0.9^{+}¢/lb. of glycerine produced. For a 40-million-pound-a-year plant the total capital investment for ion retardation was about $100,000; for ion exclusion, about $350,000. Again operating costs were somewhat less for ion retardation. These calculations, though only preliminary in nature, point out again that ion retardation may offer important capital investment savings in plants for the purification of glycerine. Alternately an ion-exclusion plant could increase capacity several-fold simply by shifting to ion-retardation operation.

The methods illustrated in this paper also should be applicable to the determination of optimum conditions for ion-retardation separations of other water-soluble materials, at least one of which is ionic and can be absorbed by the resin. The economic feasibility, which is illustrated for ion-retardation purification of glycerine, indicates that this novel separation method can have commercial utility in a variety of separation problems.

Nomenclature

C_f = Concentration of solute in solution fed to column
C_e = Concentration of solute in eulate from the column
V_e = Volume of eluate from column, starting with addition of feed solution to top of the fluid-filled column as zero
V_b = Total bulk volume of resin column (bed)

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Bland Undenatured Soybean Flakes by Alcohol Washing and Flash Desolventizing[1]

G.C. MUSTAKAS, L.D. KIRK, and E.L. GRIFFIN JR., Northern Regional Research Laboratory,[2] Peoria, Illinois

The conditions for removing the beany and bitter flavor from defatted soybean meal with ethanol and isopropyl alcohol were investigated. In the presence of alcohols, soybean protein is extremely sensitive to denaturation when temperature, moisture, and residence time are increased. If protein is to be isolated in good yield and quality, retention of the original, high water-solubility is important, and denaturation must be kept to a minimum.

Defatted soybean flakes were successfully debittered by countercurrent washing with aqueous alcohols on a pilot-plant scale, and the entrained solvent was recovered by flash desolventizing without excessive denaturation of protein. Effective debittering was obtained with 95 volume percentage ethanol and 91 volume percentage isopropyl alcohol whereas satisfactory flavor was not obtained with absolute ethanol. The solubility of the nitrogenous compounds in the meal product (Nitrogen Solubility Index—NSI = water-soluble nitrogen × 100 ÷ total nitrogen) was maintained at 68 NSI, or higher, regardless of the solvent system or conditions used when starting with 80 NSI defatted flakes. Residual alcohol in the desolventized products was reduced to 1–2% with the aqueous alcohol system and to less than 1% for the absolute alcohol system. Lower residual values can be obtained by recycling the material through the desolventizing unit. The desolventizing system described is simple, low in cost, and should be useful in any process requiring the rapid removal of solvent from residual solids where heat-sensitive constituents are present.

INCREASING industrial interest is being shown in the use of alcohols for treating defatted soybean flakes to obtain a bland soybean meal. Gelsoy (1–4), a research product developed at the Northern Regional Research Laboratory, is a water-soluble product derived either from soybeans that have been solvent-extracted with ethanol or from defatted soybean flakes that have been washed with ethanol. In this process, alcohol treatment is the factor responsible for debittering to give a bland product with high water-soluble, gelling, and whipping properties.

To recover these proteins in good yield and quality, it is essential that they remain highly dispersible in water. Hence denaturation, which results in diminishing solubility of the protein in aqueous solvents, must be avoided. Denaturation of protein in the presence

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[2] This is a laboratory of the Northern Utilization Research and Development Division, Agricultural Research Service, U.S. Department of Agriculture.

of aqueous alcohols was found by A.K. Smith *et al.* (5) to increase rapidly with water concentration and temperature. When soybean flakes are desolventized in the presence of alcohols, denaturation of the protein is extremely sensitive to increases in temperature, moisture, and residence time.

In most commercial desolventizing operations, soybean flakes are processed in either compartmented cookers or various types of jacketed conveyors where long retention-times, coupled with high temperatures and moisture, result in denaturation of the protein. This denaturation is done purposely in order to make the product suitable for cattle feed. Although this equipment is desirable for processing commercial feeds, it cannot be used to produce high NSI meal for soybean food products, such as Gelsoy. A new type of hexane desolventizer, using the vapor-phase principle for soybean meals, was patented by Leslie (6) in 1951. This equipment produced soybean flakes with an NSI approaching that of the original soybeans. A retention time of 5 min. or longer however was required. Although this time interval is not detrimental with hydrocarbon solvents, the degree of denaturation obtained with alcohol solvents is significantly high.

The present investigation was undertaken to develop a process for producing a bland-flavored soybean meal with high NSI value by countercurrent washing with alcohol and for recovering the wash solvent by flash desolventization. The new flash-desolventizing process under investigation preserves the original undenaturated state of the protein to a high degree. Although similar in principle to what is known as flash drying, flash desolventizing differs in that superheated vapors of the solvents being removed are used as the heat-transfer vapor rather than air. Flash desolventizing may be defined as a process for removing solvents from solids in a closed vapor system that essentially excludes air. Details of the experimental unit and its use for desolventizing hexane-extracted soybean flakes have been reported previously (7).

Materials and Equipment

Hexane-extracted soybean flakes with a water-soluble protein of 75–80 NSI were purchased from commercial sources.

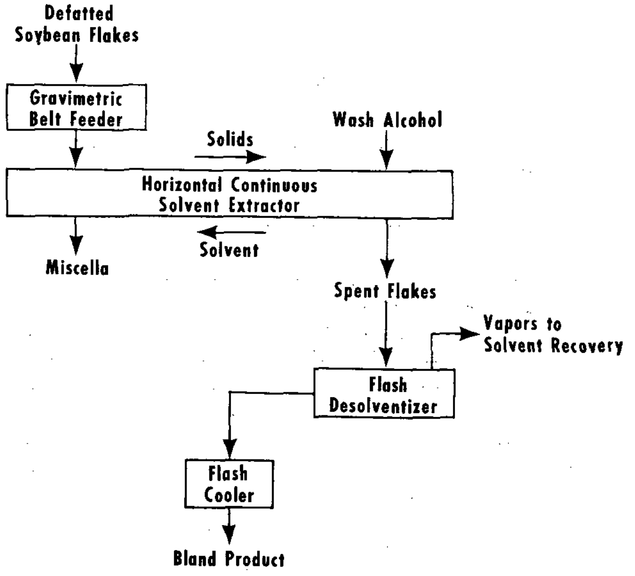


FIG. 1. Pilot-plant preparation of debittered undenatured soybean flakes.

Countercurrent washing was conducted in a Kennedy continuous horizontal extractor approximately 20 ft. in length. In a typical run soybean flakes were fed to the unit by a gravimetric belt feeder and were conveyed through 20 mixing and separating stages by revolving paddlewheels countercurrent to the flow of solvent. The paddlewheels, 12 in. in diameter by 10 in. wide, consisted of three stainless-steel flat blades attached 120 degrees apart to a common hub and shaft. They were slotted to permit drainage of the solvent. Flakes leaving the last mixing stage were lifted out of the solvent, drained on an inclined drag-link conveyor, and moved by transverse screw conveyor with choke feed to the desolventizer.

A simplified flowsheet of the pilot-plant processing is shown in Figure 1.

Figure 2 gives a diagrammatic sketch of the flash desolventizer.

The main element of the system consisted of 3-in. i.d. galvanized steel tubing that formed a closed circuit with a superheater, turboblower, and a cyclone collector. The superheater was a single pass shell and tube heat-exchanger with 21 sq. ft. of heat-transfer area. It contained 10 stainless steel tubes 1-in. o.d. x 8 ft. long. The 250 c.f.m. turboblower was constructed of aluminum with a high-strength radial type of aluminum impeller. Duct work, blower, and cyclone separator were wrapped with steam tracer copper tubing and covered with magnesia insulation. The desolventizing zone is designated by the dotted line. In this system, extracted solids were suspended in a superheated vapor stream and heated by indirect steam at 115–120 p.s.i.g. to temperatures above the condensation point. The vapor stream consisted of the same solvent associated with the solids. The solid material was maintained in suspension for only a few seconds, during which time it was desolventized. It was then passed to a hot cyclone separator for separation of the solvent vapor. Hot flakes were discharged at the cyclone base by a rotary valve into a pneumatic air-cooling system consisting of a blower, 38 ft. of conveying duct, and a cyclone collector. Cool air drawn into this duct reduced the flakes essentially to room temperature. Solvent vapors were bled out of the system at the blower discharge and passed through a

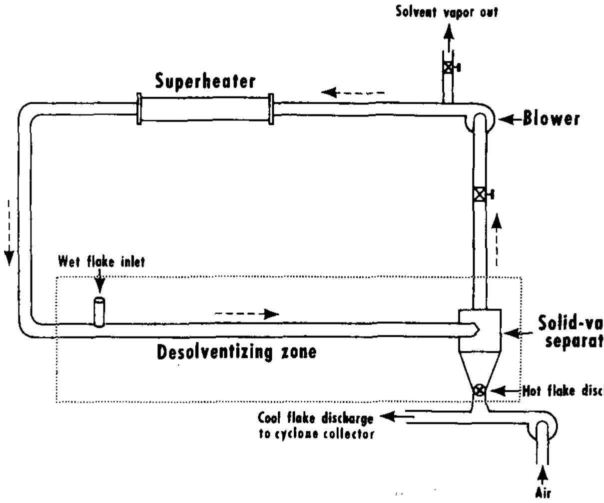


FIG. 2. Flow diagram of flash desolventizer.

cloth dust filter before condensation and collection in a receiver.

Experimental

NSI for the measurement of water-soluble protein was obtained by modifying the method of Smith and Circle (8). The aqueous solid suspension was agitated for 2 hr. at 25°C. with a flat-blade paddle that revolved at 125 r.p.m. The decrease in NSI during processing indicated the degree of protein denaturation.

Small quantities of residual alcohol in the desolventized flakes were analyzed by adding n-heptane to a weighed sample, then distilling to remove the alcohol as a heptane azeotrope. The condensate was analyzed by a known esterification procedure for primary alcohols (9).

Moisture in the spent flakes was determined by the Karl Fischer method. Alcohol content in spent flakes was taken as the difference between the percentage total volatiles and the percentage moisture. Total volatiles were determined by oven drying in a Brabender moisture tester for 2 hr. at 120°C.

Soybean flakes either were used with the original moisture content of 8–10% or were predried to approximately 3–5%.

To limit denaturation, aqueous alcohols with a minimum alcohol content of 91 volume percentage were used. Since a minimum NSI value (1) of 68 is required for meal suitable for Gelsoy, this value was considered as a minimum standard.

Solids feed rates to the extractor were 25 lb./hr. except for capacity studies where feed rates were increased to 75 lb./hr.; solvent ratio was 1.5 to 1. Temperatures in the extractor were controlled at either 75 or 100°F., and retention times at these temperatures were 36 and 18 min., respectively.

Drainage time of the spent flakes was varied by setting the speed of the drag-link conveyor. This variable was not studied separately but was regulated to obtain a minimum solvent load to the desolventizer. Other means of reducing volatiles in spent flakes, such as use of presses or of centrifuges, were not investigated although these would be effective for decreasing the desolventizer load.

Vapor velocity measurements in the desolventizer were made by a Pitot tube, located between the superheater and solids inlet. Composition of the vapors was determined by sampling the vapor at the blower discharge, separating the condensable components, and analyzing the noncondensables by Orsat gas analysis.

Temperatures and static pressures were measured at selected points by thermocouples and water manometers, respectively. Temperature of the hot desolventized flakes was measured directly below the rotary discharge valve by collecting a sample in an enclosed cup in which a thermocouple was inserted.

Appropriate techniques were employed to minimize protein denaturation during processing. Static pressure at the desolventizer feed inlet was controlled at either atmospheric or very slight vacuum to prevent superheated vapors from flowing back into the transverse feed conveyor. Increased contact of solids in the conveyor in the presence of superheated vapors created a condition for rapid denaturation. For safety and for maintaining a high solvent concentration in the recycle vapor, air leakage into the system was undesirable. Carbon dioxide was used both at the feed inlet to minimize condensation of hot recirculating vapors on the feed flakes and at the cyclone discharge valve to minimize air leakage into the system.

Vapor velocity could be varied by either altering the blower speed or throttling a gate valve that was installed in the duct between the cyclone separator and blower.

Steam pressure to the superheater was regulated to study hot discharge flake temperatures in the range of 140–210°F.

Temperature, retention time, alcohol concentration, feed rate, and feed moisture were variables studied in the wash step to determine their effect on flavor quality and denaturation. For the desolventizing step, feed rate, temperature, vapor velocity, and vapor composition were evaluated.

Results and Discussion

Flavor Quality. Defatted flakes washed with 95 volume percentage ethanol or 91 volume percentage isopropyl alcohol were completely debittered and light-colored whereas the bitter and beany principle was not extractable with absolute ethanol. These results show that moisture is required in the solvent to achieve a bland product. This requirement was further demonstrated by the fact that predrying of the original flakes had an adverse effect on flavor, particularly with the 95% ethanol system. Flavor quality of the product after various alcohol wash treatments is given in Table I. Higher temperatures in the extractor improved flavor slightly for the 95% ethanol wash. However, with isopropyl alcohol, an equally bland product was obtained at either 75 or 100°F., but denaturation was increased slightly.

The alcohol-washing step removed a small portion of the flake solids in the form of a dark yellow waxy

TABLE I

Flavor Quality and NSI Values Obtained with Use of Various Alcohol Washes

Process stream	Wash Alcohol System					
	Ethyl alcohol				Isopropyl alcohol	
	Absolute		95 Volume %		91 Volume %	
	Predried flakes [a]	As received flakes [b]	Predried flakes [a]	As received flakes [b]	As received flakes [b]	
Flavor quality of product						
Cool desolventized flakes	Bitter	Beany	Slightly beany	Bland	Bland	Bland
NSI value						
Unwashed feed flakes	75.7	78.5	80.8	81.9	79.2	79.2
Washed flakes	73.8 [c]	79.5 [c]	77.0 [c]	79.7 [d]	76.1 [c]	74.3[c]
Hot desolventized flakes	77.2	76.7	72.3	69.9	72.0[e]	68.2 [f]
Cool desolventized flakes	74.9	79.4	72.8	70.4	72.1	68.0

[a] Moisture, 3–5%.
[b] Moisture, 8–10%.
[c] Temperature of wash step = 100°F.
[d] Temperature of wash step = 75°F.
[e] Hot flake discharge temperature = 160°F.
[f] Hot flake discharge temperature = 180°F.

TABLE II

Typical Miscella Compositions of Various Alcohol Wash Systems

Components	Wash Alcohol		
	Absolute ethanol [a]	95 Volume % ethanol [b]	91 Volume % isopropyl alcohol [c]
	Weight %	Weight %	Weight %
Water	1.08	10.20	8.69
Alcohol	97.74	86.52	88.90
Solubles	0.90	1.82	1.22
Fines	0.28	1.46	1.19
Ash	0.01	0.09	0.01

[a] Wash temperature 100°F., feed flakes = 4.0% moisture.
[b] Wash temperature 75°F., feed flakes = 8.6% moisture.
[c] Wash temperature 75°F., feed flakes = 7.9% moisture.

material. Miscella compositions are given in Table II for the various solvent systems. The slightly higher solubles obtained with the aqueous alcohols probably indicate higher extraction of ash, carbohydrate, and phospholipid materials.

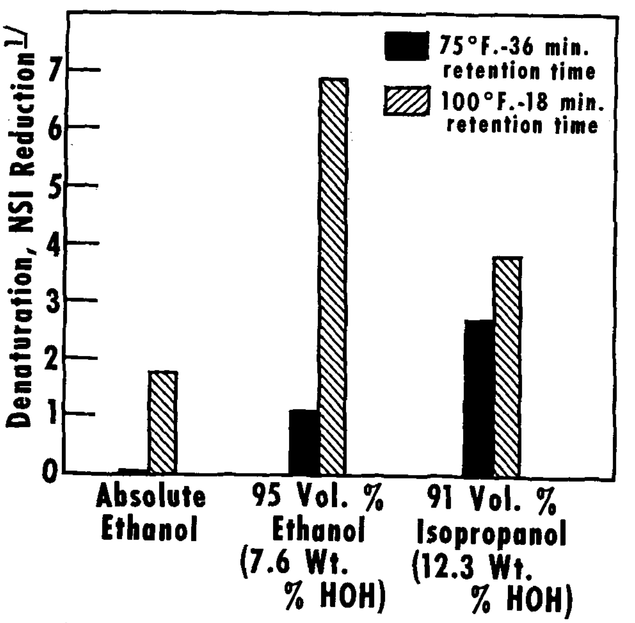
Denaturation of Protein. Denaturation in the process is a function of temperature, time, and moisture content. Essentially no denaturation was obtained with absolute ethanol whereas some did occur with aqueous alcohol. Total denaturation however was restricted to approximately 12 NSI units, or less, with 95% ethanol or 91% isopropyl alcohol.

Bar graphs in Figure 3 show the effect of alcohol concentration in the extractor on denaturation at two washing temperatures, 75 and 100°F. At the lower temperature, denaturation was proportional to water content of the alcohols. At 100°F. 91% isopropyl alcohol gave less denaturation than did 95% ethanol.

Typical NSI values for the process streams and the relative denaturation obtained with various alcohol systems are given in Table I.

Since moisture was a significant variable in denaturation, moisture equilibrium values for the various process streams were obtained and plotted as a function of alcohol concentration and predrying treatment of the flakes (Figure 4). The curves show that a) regardless of alcohol concentration, low and essentially equal moisture levels were reached in the desolventizer, b) absolute ethanol had a pronounced dehydrating effect on "as is" feed flakes, and c) flakes washed with 95% ethanol reached similarly high moisture levels in the extractor whether or not the original flakes were predried.

Moisture content of the flakes that passed through the extractor increased with retention time and lower temperature. Moisture content of the spent flakes increased directly with their total volatiles content (Figure 5). Because higher volatile content of the material that entered the desolventizer decreased desolventizer capacity and increased denaturation, maximum drainage time must be allowed for spent flakes.



1/ Average experimental values used

Fig. 3. Effect of alcohol concentration and temperature on denaturation of soybean flakes in the extractor.

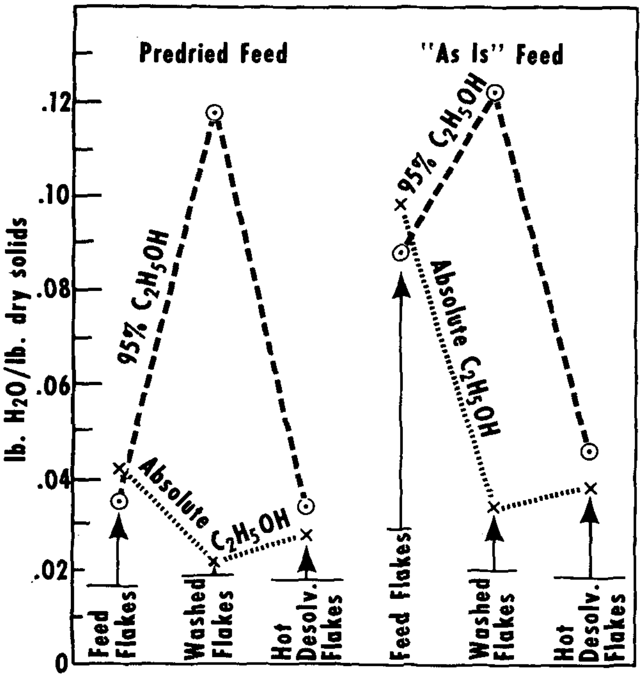


Fig. 4. Moisture content in process streams as a function of alcohol concentration and pretreatment of soybean flakes.

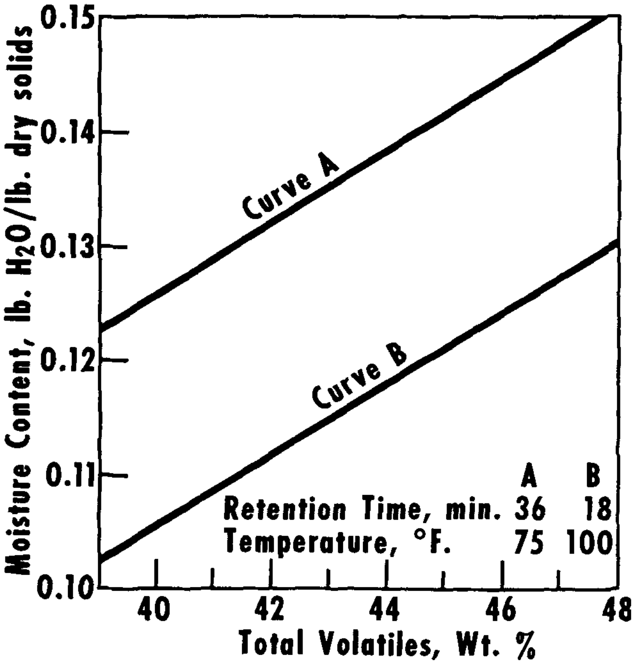


Fig. 5. Relationship between total volatiles and moisture in spent flakes for two extractor conditions using 95 volume percentage ethanol.

A high moisture level in spent flakes resulted in a correspondingly high moisture content in the desolventizer vapor stream. Also, when high blower speeds were used, vapor velocity increased and raised the moisture content of the vapor stream. This moisture increase resulted from an improved heat-transfer rate that favored a greater evaporation of water from the wet solids into the vapor. The direct effect of moisture content in the vapors on denaturation is shown in Figure 6. These data were taken with hot discharge flake temperatures that were controlled in the range of 160–190°F.

Temperature and moisture were interrelated in their denaturing effects so that, with low moisture values, the desolventized flakes could be heated to higher temperatures without appreciable denaturation. An attempt was made to correlate the temperature of the hot discharge flakes with denaturation, but only a rough relationship could be shown between high and low temperatures with a scattering of data in the intermediate range. With the isopropyl alcohol system NSI decreased approximately four units as the hot flake discharge temperature increased from 160 to 180°F. (Table I).

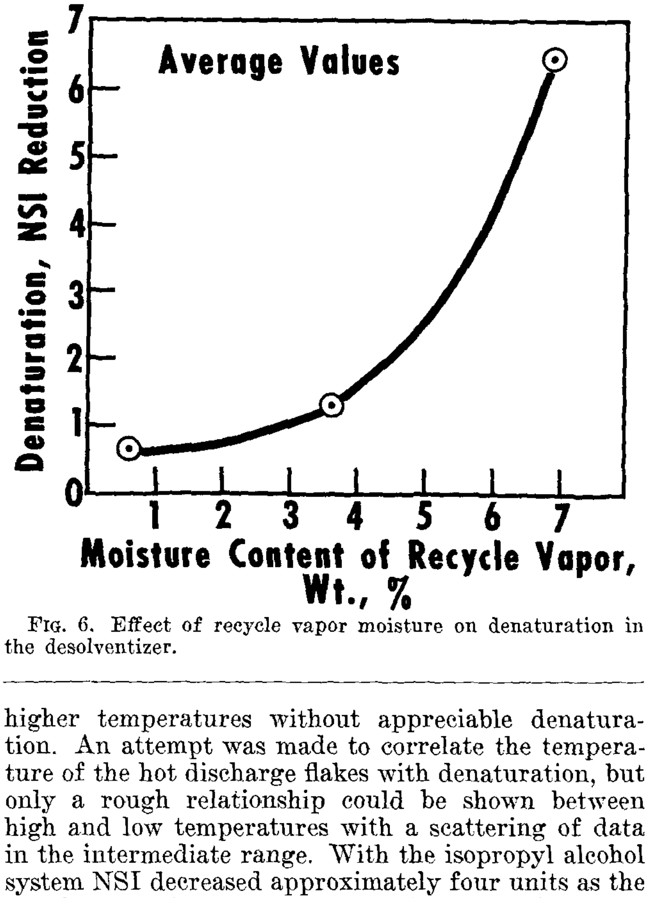


FIG. 6. Effect of recycle vapor moisture on denaturation in the desolventizer.

Desolventizing. Evaporative performance of the flash desolventizer was dependent on factors which controlled heat transfer rate and established the system's capacity. Controllable variables were the solids throughput, the recycle vapor temperature or heat input, and the recycle vapor velocity. Higher solids throughput increased the vapor load to the desolventizer. When the solids feed-rate to the extractor was varied from 25 to 75 lb. per hour, residual total volatiles in the desolventized flakes increased from 4.5 to 8.2%.

An improved heat-transfer rate was achieved from the increasing turbulence as vapor velocity was increased. Recycle vapor velocity was changed from 38 to 52 ft. per second by increasing the blower speed from 2,900 to 3,500 r.p.m. This change of blower speed decreased the total volatiles in the desolventized product from 7.6 to 4.5%.

Residual total volatiles and residual alcohols for various process streams are shown in Table III. Very low residual volatiles were achieved with anhydrous alcohol. A relationship between residual volatiles in the end product and hot discharge flake temperatures was obtained (Figure 7). Further relationships between residual total volatiles and residual alcohol for the three solvent systems are given in Figure 8.

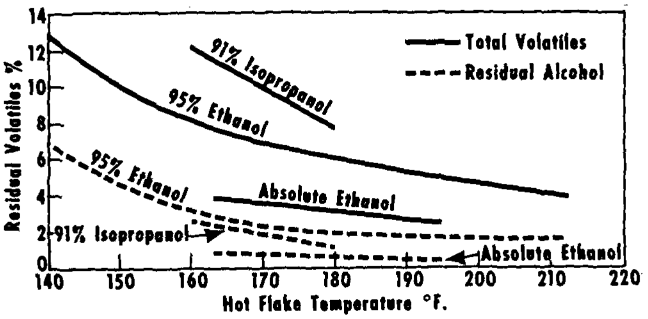


FIG. 7. Correlation between temperature and residual volatiles of desolventized flakes for three solvent systems.

Minimum values of residual alcohol content below 2% were obtained when total volatiles were reduced to approximately 8% for 91% isopropyl alcohol, 4% for 95% ethanol, and 3% for absolute ethanol. Lower residual values were obtained with 91% isopropyl alcohol than with 95% ethanol at similar discharge flake temperatures.

This work indicates that lower residual values can be achieved with a) higher solids-discharge tempera-

TABLE III

Total Volatiles and Residual Alcohol of Process Streams Obtained with Various Wash Alcohols

Process stream	Wash Alcohol System					
	Ethyl alcohol				Isopropyl alcohol	
	Absolute		95 Volume %		91 Volume %	
	Predried flakes	As received flakes	Predried flakes	As received flakes	As received flakes	
	Total volatiles, %					
Unwashed feed flakes	3.9	8.8	4.2	9.7	7.9	8.1
Washed flakes	36.5	39.2	40.8	40.8	48.0	46.0
Hot desolventized flakes	2.9	3.4	4.6	5.2	12.2 [a]	7.3 [b]
Cool desolventized flakes	4.2	4.0	4.9	5.8	9.9	5.8
	Residual alcohol, %					
Hot desolventized flakes	0.8	0.5	1.8	2.0	2.6 [a]	1.3[b]
Cool desolventized flakes	0.7	1.1	1.8	2.1	2.2	0.8

[a] Hot flake discharge temperature = 160°F.
[b] Hot flake discharge temperature = 180°F.

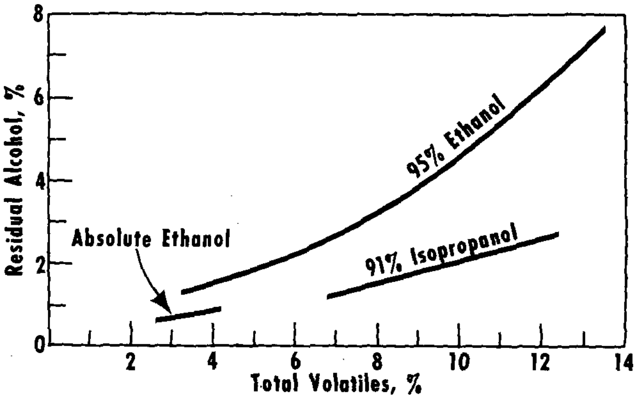


FIG. 8. Correlation between residual alcohol and total volatiles in desolventized solids for three solvent systems.

ture, b) longer residence time, or c) use of relatively anhydrous alcohols. However, since moisture is apparently a prerequisite for achieving good flavor and since temperature contributes to denaturation, some compromise in optimum conditions is necessary.

Residence time in the desolventizer was not varied in the work reported here. Newer studies indicate that increased residence time, achieved by recycling the solid material in a second pass, can reduce residual alcohol values to significantly lower levels than reported in Figures 7 and 8; no further denaturation of the desolventized flakes occurs.

An interesting correlation was obtained when hot flake discharge temperature was plotted against alcohol-to-water ratio in the residual volatiles retained by the discharged flakes (Figure 9). The 95% ethanol curve reached a minimum value at 180°F., then reversed its direction. The curve shows that, below 180°F., volatiles were being removed in favor of the alcohol component whereas, above 180°F., the water component was being removed more rapidly than the alcohol. This effect could indicate that absorption or hydrogen bonding of the alcohol component on the solids takes place. Residual alcohol values shown for the isopropyl alcohol system are significantly lower but represent data over a narrower temperature range. Alcohol-to-water ratios over the range of 160–180°F. were essentially constant, but probably above 180°F. the curve would follow a slope similar to that for the 95% ethanol.

In desolventizer operations a certain amount of air entry into the vapor stream was unavoidable. Vapor seals at the solids inlet and outlet for the desolventizer were ineffective and permitted air to enter from 5 to as high as 45% by weight of air in the recycle vapor stream. The noncondensables, air and carbon dioxide, however apparently did not affect desolventizing adversely; in fact they lowered the dew point to permit desolventizing below the actual boiling point of the entrained liquid. In a vapor system free from noncondensables the residual alcohol possibly will be higher for flake temperatures below the boiling point but will be approximately the same for flake temperatures where dew-point condensation is not a problem. Experiments where steam was injected into the vapor stream to reduce the concentration of noncondensables showed that the residual alcohol values were similar to those that used a vapor system containing noncondensables. Steam injection could not be used in the present experiments without considerable denaturation of the product.

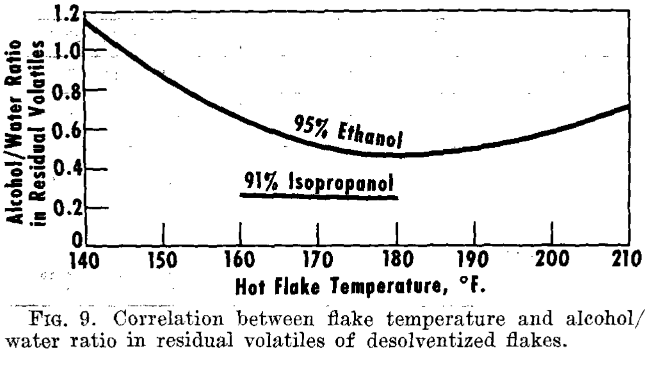


FIG. 9. Correlation between flake temperature and alcohol/water ratio in residual volatiles of desolventized flakes.

Solids Handling. Excessive moisture at any stage of soybean meal processing is objectionable because of its physical effect on the characteristics of the solid material, rendering it sticky or doughy rather than free-flowing and easily dispersed. The flash desolventizer however, because of low retention time and rapid heat-transfer, averts these difficulties and produces a free-flowing product.

Since soybean flakes are fragile, a certain amount of size reduction in handling is expected. Sieve analyses of the feed and final product streams showed that no significant attrition occurs when processing with 95% ethanol whereas significant attrition was obtained with absolute ethanol. Moisture in the aqueous alcohol wash may have an agglomerating effect on the "fines" to compensate for the breakdown of solids on account of mechanical attrition. Table IV presents the sieve analyses of feed, spent flakes, and desolventized product streams when processing is done with absolute ethanol and predried feed.

TABLE IV

Particle Size Comparison of Extractor Feed Flakes, Spent Flakes, and Desolventized Product Streams

U.S. Standard sieve number	Absolute Ethanol System Cumulative % retained		
	Extractor feed [a]	Spent flakes	Desolventized flakes
8	27.5	10.55	2.51
14	59.5	34.67	18.07
20	91.0	75.80	58.74
40	97.5	95.98	89.86
60	99.0	98.49	96.39
200	100.0	99.70	99.90
Pan	100.0	100.00	100.00

[a] Moisture content 4.1%.

Acknowledgment

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