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Flash Desolventizing[1]

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DESOLVENTIZING, that is, the removal and recovery of solvent adhering to the residual solids, is an economic necessity in any extraction process employing volatile organic solvents. Also of importance to the vegetable-oil-extraction industry, but less understood, is the associated problem of producing undenatured proteinaceous residues from which specialty products can be manufactured. The role of the factors which cause denaturation of the protein fraction of soybean oil meal has been previously discussed in the literature (1, 2). For optimum results the factors of time, temperature, and moisture content must be considered in the design of desolventizing equipment as well as those that affect mechanical performance. The purpose of this publication is to describe a new, rapid desolventizing process based on fluidization principles, to evaluate its performance on a pilot-plant scale, and to discuss its commercial possibilities.

Existing Commercial Units

In the majority of existing desolventizing units the solvent is evaporated by the application of heat through the walls of jacketed vessels, followed by the use of open steam and indirect heat to remove the last traces of the solvent. Specialty products have been produced by removing the solvent under vacuum. The first of these two methods tends to discolor and denature the protein of the residual meal and often is nonuniform in operation. The equipment needed is cumbersome, difficult to maintain, and high in initial cost.

A decided advance was made with introduction of the first vapor phase desolventizer, which employs a stream of superheated vapors for volatilizing the solvent. This type of equipment has been described by Leslie (5), and its performance with respect to denaturation reported by Belter and Smith (2). Leslie's vapor phase desolventizer made possible, with certain processing limitations, the commercial production of soybean flakes with a nitrogen solubility approaching that of the original beans. These limitations are the result of the physical arrangement of the unit. In order to minimize entrainment of finely divided material in the vapor stream, it is necessary to limit the linear vapor velocity within the equipment. At permissible velocities, the rate of vaporization of the solvent is sufficiently decreased so that a retention time of 5 minutes or longer is required to remove the solvent satisfactorily. Such a time-interval has not proved to be particularly detrimental to the protein during the removal of the commercial hydrocarbon solvent, providing the temperature of the residual flakes does not go above 170°F. However for other solvents, like ethyl alcohol, such a time-interval at this temperature partially denatures the protein fractions of soybean oil meal (7). Also, when a hydrocarbon solvent is employed, and soybean oil meal having a high-protein solubility is desired, the operating conditions must be carefully watched to maintain satisfactory results. For example, any reduction in the flow of material from the extractor rapidly unbalances the heat equilibrium within the desolventizing unit and consequently results in an increased discharge temperature for the flakes. This small increase of temperature substantially lowers the solubility of the protein, which is extremely sensitive in this temperature range.

Experimental Unit

The desolventizing system studied in this investigation is illustrated in Figure 1. The simplicity of the unit is readily apparent. It consists essentially of a conveying and desolventizing tube into which solvent-saturated flakes are fed, and through which superheated solvent vapors simultaneously desolventize and convey the flakes to a cyclone separator. In the cyclone the vapor stream and the desolventized flakes are separated, the flakes are discharged from the system through a barrel valve, and the vapors are recirculated by a blower through the superheater and back into the conveying tube. A quantity of solvent vapors equal to that vaporized from the flakes is continuously withdrawn to a condenser. The type of marc-feeding mechanism and vapor-solids separator may be altered to fit the requirements of the application. Such a unit combines the desirable features of

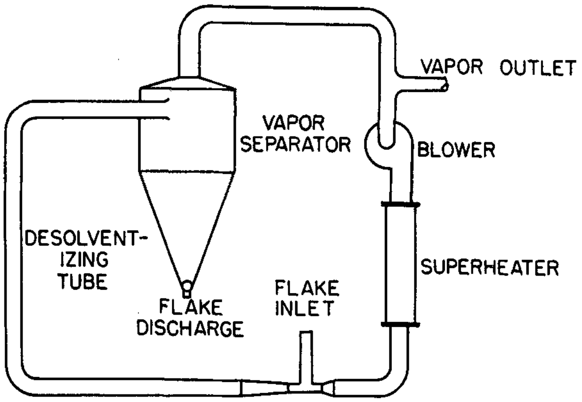

FIG. 1. Diagram of flash desolventizer.

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vapor desolventizing and flash drying to produce a compact and efficient desolventizing unit.

A desolventizing system as described has been installed in the oil-extraction pilot plant in the laboratory of the Northern Utilization Research Branch. This unit was designed with the intent of being of general utility for all solvents and various types of extraction residues.

The main element of the system, the desolventizing tube, was fabricated from a section of steel tubing with an inside diameter of 3 in. The length of the desolventizing zone, measured from the point of entry of the "wet" flakes to their entry into the cyclone, is 24 ft. A low-velocity cyclone, 20 in. in diameter, is used for the solids-vapor separator.

The solvent-saturated flakes are transported from the drag-link conveyor of the pilot-plant extractor, which has been previously described by Lerman, Kennedy, and Loshin (4), by means of a 4-inch transverse conveyor of the screw type. The flakes are fed by gravity into a venturi section inserted in the desolventizing tube. The venturi-feed section serves a dual purpose: a) prevents the flow of superheated vapors into the transverse conveyor, and b) aids the suspension of the "wet" flakes into the vapor stream. Its use however is not essential for satisfactory operation of the system. The desolventized flakes are discharged from the cyclone by means of a barrel valve driven with a variable-speed motor and reduction unit.

The energy necessary to propel the vapor through the system is supplied by a blower capable of delivering 50 c.f.m. of standard air against a static pressure of 21 in. of water. The linear velocity of the vapors within the desolventizing duct is calculated to be approximately 1,500 ft. per minute, depending somewhat on the solvent used in the system. The retention time of the flakes in the system is estimated to be approximately 2-3 seconds. The superheater is of sufficient size to give a maximum vapor-inlet temperature of 330°F. when supplied with 125 p.s.i.g. steam. The capacity of the installation as described and operated at the stated conditions is approximately 13 pounds of hexane per hour.

Cold spots were eliminated in the equipment by tracing all vapor-transporting lines, the cyclone, and the blower with copper tubing supplied with steam at 125 p.s.i.g. The tracings were in turn covered with 2 in. of magnesia insulation. This precaution, which is considered essential, maintains the wall temperature of the equipment above the saturation temperature of the vapors and prevents the accumulation of solid material in the unit. Thermometers were placed in strategic positions throughout the system so that its performance could be analyzed.

A steam-sparging line was connected into the base of the cyclone so that the composition of the desolventizing atmosphere could be altered.

Materials and Methods

Although the data reported will be for the recovery of the commercial hydrocarbon solvent, hexane, from extracted soybean flakes, other materials and solvents have been used in this system with sufficient success to justify further investigation.

Cleaned soybeans, No. 2 grade, were cracked, dehulled, conditioned, and flaked in the pilot-plant preparation equipment. The resultant flakes averaged from 8- to 12-thousandths of an inch in thickness in the various experiments with a standard deviation of approximately 2-thousandths. These flakes, which compare in size to those produced on commercial equipment, were subsequently extracted with the commercial hexane (boiling point range 150-160°F.) to a residual oil content of under 1%.

Because of the importance of knowing the extent of removal of the solvent and the amount of injury to the protein encountered during this operation, tests for determining these data were routine for the runs. The total-volatiles determinations, which include both moisture and solvent, were conducted by the A.O.C.S. Official Method BA-2-38, which employs 2 hours drying at 130°C. The residual-solvent values were obtained as the difference between the total volatiles and the moisture values determined by the Bidwell-Sterling procedure (9). The NSI values (Nitrogen Solubility Index) were determined by the method originally developed by Smith and Circle (6). The reported values are the averages of two or more determinations.

The temperature of the desolventized flakes was measured by filling a specially designed scoop, containing a Weston dial thermometer, with discharged flakes. This procedure was repeated until a constant temperature was obtained.

Results and Discussion

The residual-volatiles contents of soybean flakes after flash desolventizing are reported in Table I and the corresponding NSI values in Table II. The results reported are the averages of numerous values obtained during operation at the stated conditions. It will be observed that the extracted flakes fed to the desolventizer have a lower NSI value than normally obtained in commercial practice as previously reported (2). The conditioning procedure necessary to produce satisfactory flakes on a small scale is responsible for this slight amount of denaturation prior to desolventizing. With the exception of the flakes for run 6 which had a NSI value of 63.8%, these materials would be acceptable for most specialty uses.

TABLE I

Residual-Volatiles Content of Soybean Flakes After Flash Desolventizing [a]

Run No.	Temperatures, °F.			Volatile content of desolventized flakes, %		
	Vapor		Desolventized flakes			
	Inlet	Discharge		Total	Moisture	Solvent
5	290	260	183	4.21	......	......
6	329	281	191	7.32	6.96	0.36
7b	301	264	210	4.02	3.27	0.75
7c	322	286	221	3.06	2.32	0.74
8	322	290	215	4.20	4.00	0.20

[a] The solvent-saturated flakes fed to the desolventizer averaged 35.4% total volatiles and 9.00% moisture.

TABLE II

Effect of Flake-Discharge Temperature on Protein Solubility (NSI)

Run No.	Temperature of desolventized flakes, °F.	NSI	
		Spent [a] flakes	Desolventized flakes
5	183	66.1	65.7
6	191	63.8	62.0
7b	210	70.7	72.1
7c	221	70.3	69.8
8	215	68.4	65.4

[a] Solvent-saturated flakes fed to the desolventizer.

Run 5 was conducted without the addition of steam to the desolventizing vapor stream. In the other runs, steam was added to the vapor stream to a level of approximately 15% by weight. Non-condensable gases were present in the vapor stream during all of the runs at a level of approximately 30-50% by weight. This amount of non-condensables was inherent with the operation of the pilot plant and could not be lowered. Their presence would have no known effect on the NSI results obtained, and present indications are that they may have only a minor effect on the residual-solvent values. Admittedly the presence of the non-condensables would lower the residual-solvent values at equilibrium. However, due to the short residence time of the flakes in the desolventizing zone, equilibrium undoubtedly is not reached. Also it has been reported by Chu, Lane, and Conklin (3) and Wenzel and White (8) that the rate of vaporization of a liquid into its superheated vapors is more rapid than into an atmosphere of air. The two opposing forces would tend to minimize the effect of the non-condensables on the resulting residual-solvent values. The final answer can only be obtained by additional experimentation.

The results given in Tables I and II show that soybean flakes can be desolventized in several seconds without excessive residual-solvent loss and without denaturing the protein fraction. It is also apparent that, because of the reduced retention time in the desolventizing zone, the flakes may be heated, even in the presence of steam, to temperatures considerably above the level previously thought to be the maximum permissible for the production of meal with a high NSI. Higher discharge temperatures for the desolventized flakes mean potentially lower residual-solvent contents compared to other systems. It is not implied that this unit will completely remove the solvent as do the combination processes of desolventizing and deodorizing as commercially practiced, but rather that it will reduce the solvent content to a lower value than existing desolventizing units. For safety in subsequent handling of the residual flakes, an additional step involving stripping probably will be necessary. It must be determined for each installation whether it is economical to recover the solvent removed in this final stage.

Beckel *et al.* (1) have estimated that the temperature coefficient of the reaction velocity for the denaturation of soybean protein is approximately 3.5 for a 10°C. rise in temperature. Using this value, which normally would be considered low for this type of reaction and the previously stated satisfactory conditions of 5 minutes at 170°F. as a basis, it may be calculated that an increase in the discharge temperature to 220°F. would require that the desolventizing operation be completed in 9 seconds or less if denaturation is to be minimized. The solubility results obtained in the operation of the flash desolventizer are not therefore totally unexpected.

Commercial Possibilities

The simplicity of the flash desolventizing system can best be appreciated by persons having had experience in the operation of both of the older systems described above. The equipment has fewer moving parts than the conventional desolventizing units, and consequently maintenance costs should be lower. This system will occupy less space than existing desolventizing equipment. Its initial cost of fabrication and installation in commercial sizes should not exceed that for existing systems and probably would be considerably less for both large and small installations.

Two applications of this unit in commercial processing are apparent. First, because of the negligible quantity of material retained in the system at any instant, this unit is especially useful in pilot-plant investigations or other installations operating intermittently. Second, this equipment will provide soybean and other solvent-extraction processors with a compact desolventizing unit operating in parallel with existing desolventizing equipment which normally produces denatured meals for use in animal feeds. The new unit could operate either intermittently or continuously to produce a supply of essentially undenatured soybean flakes consistent with the demand for specialty products. This procedure will eliminate excessive storage and reduce inventories of this material, whose protein solubility is known to decrease with time (6). Addition of an auxiliary desolventizer would minimize the production of improperly processed materials resulting from changes of operating conditions necessary under the present installations. The desolventizing system described should be useful in any process requiring the rapid removal of an organic solvent from a residual solid material for the protection of a heat-sensitive constituent of that solid.

Acknowledgment

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Correction

Norman O. V. Sonntag of Colgate-Palmolive Company, Jersey City, N. J., writes that several minor errors in the paper "Reactions of Fatty Acid Chlorides. I. Preparation of Fatty Acid Anhydrides," J. Am. Oil Chemists' Soc., *31*, 151 (1954), have been discovered:

p. 153. In Table II, under column labelled Anhydrides, last two entries should read Oleic.

p. 153. Line 11 should read 38.0-40.0°.

p. 154. Figure 1: ordinate of curves should read % Absorption.

p. 155. Table IV: under column labelled Method, delete the superscript a from the entries of B and I.