United States Patent

[11] **3,615,657**

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[21] Appl. No. **759,647**
[22] Filed **Sept. 13, 1968**
[45] Patented **Oct. 26, 1971**
[73] Assignee **The United States of America as represented by the Secretary of Agriculture**

[54] **PROCESS FOR PRODUCING COTTONSEED PROTEIN CONCENTRATE**
4 Claims, 4 Drawing Figs.

[52] **U.S. Cl.** .... **99/17,** 99/2O.E.
[51] **Int. Cl.** **A23j 1/14**
[50] **Field of Search** .... 99/17, 98, 2 E

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ABSTRACT: A process for producing a high protein cottonseed concentrate from cottonseed meats which process is characterized by an integrated sequence of drying, flaking, disintegrating, screen separating and gravity separating steps. The process accomplishes the substantially complete removal of intact cottonseed pigment glands and as a consequence thereof, the isolation of gland-free material, which material can be exalted to exhibit a protein content as high as 73 percent by weight on an oil and moisture free basis.

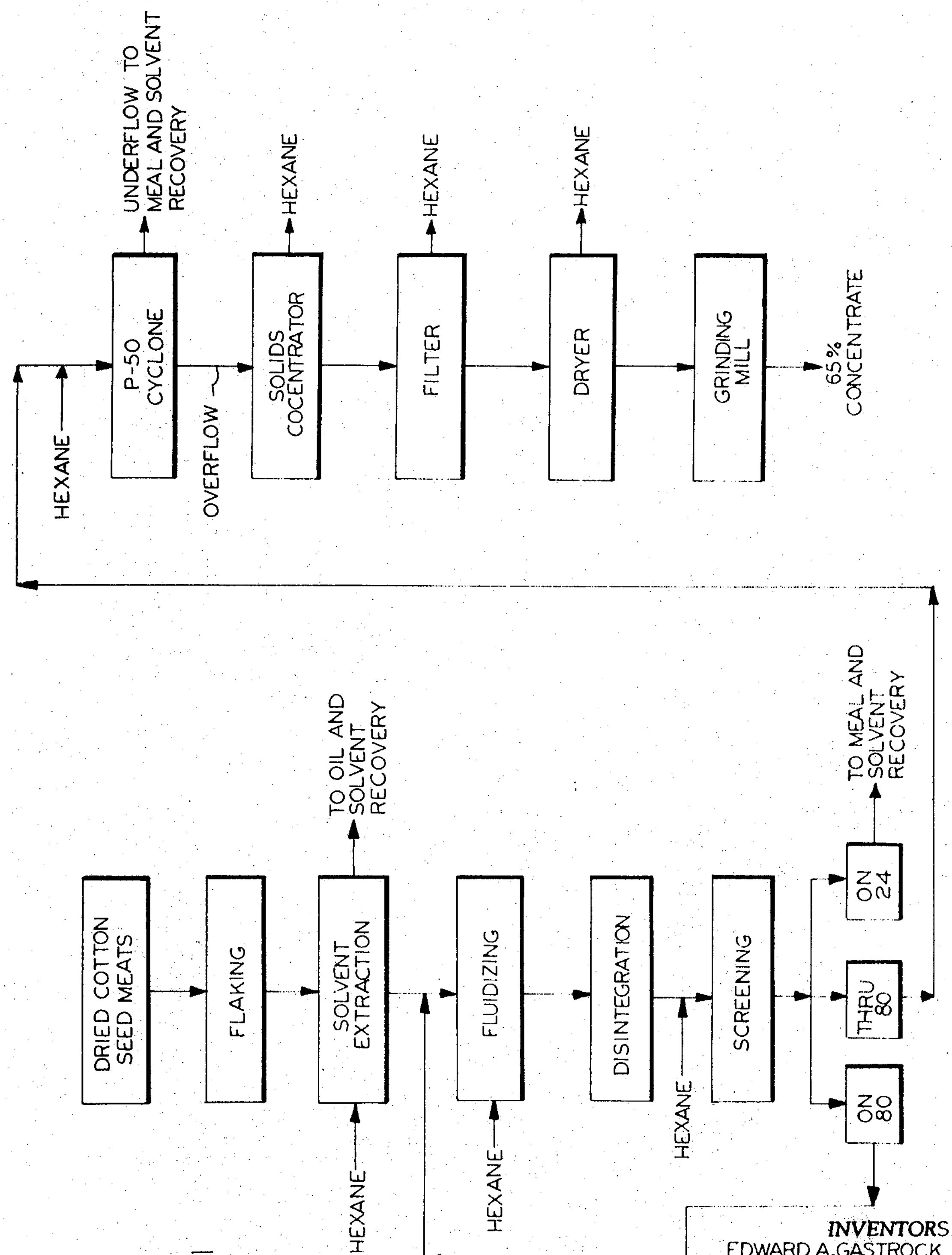

FIG. 1

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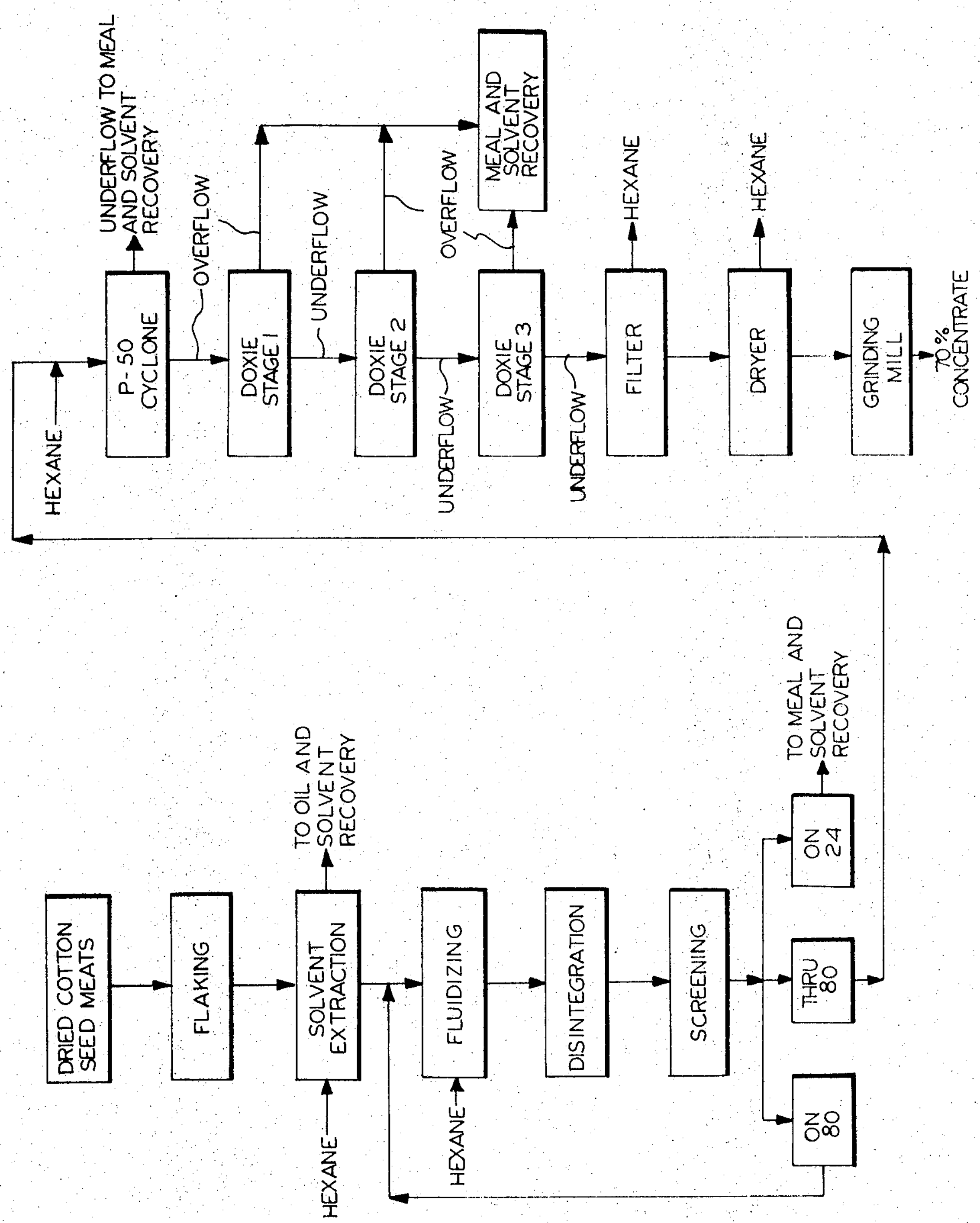

FIG. 2

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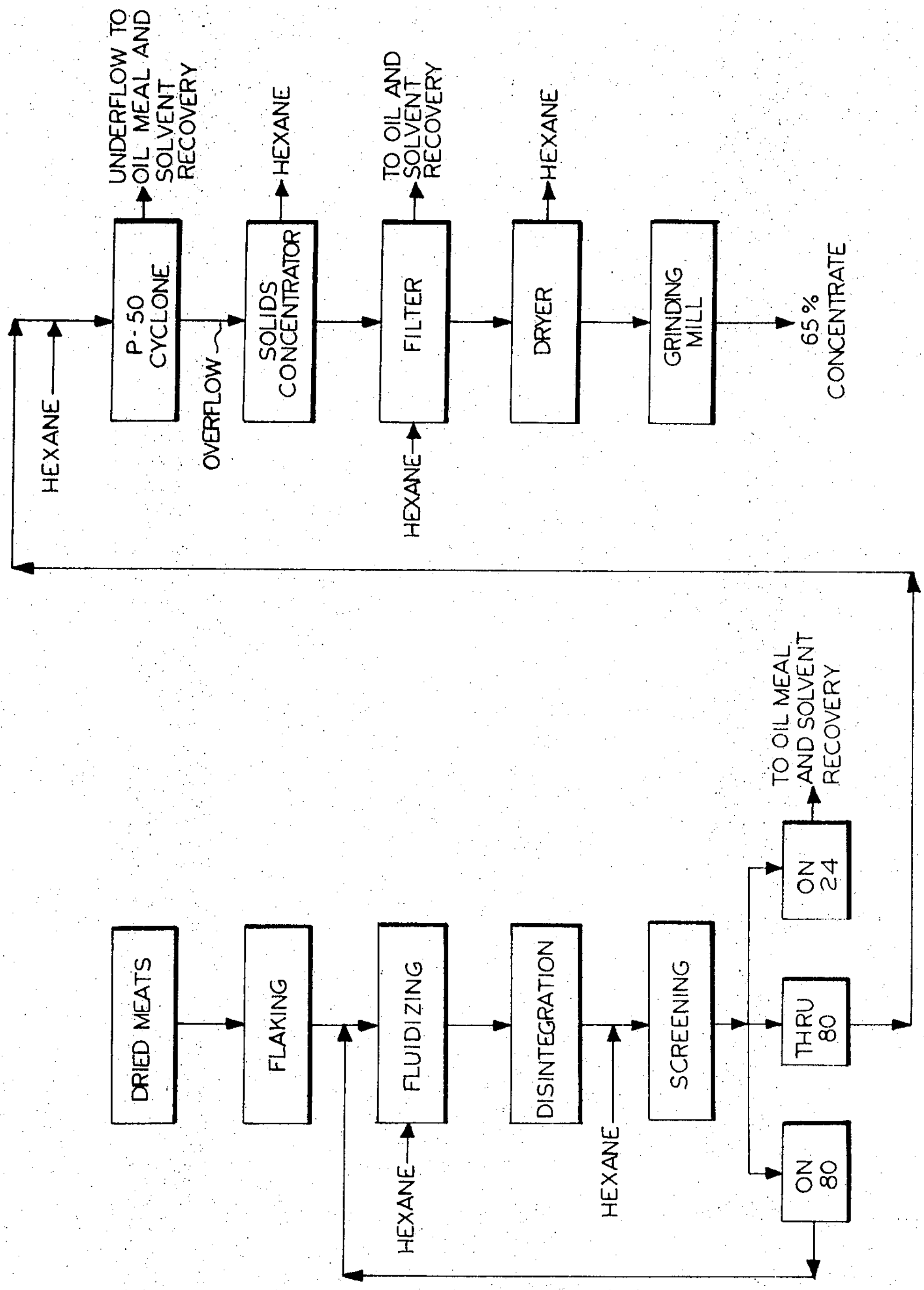

FIG. 3

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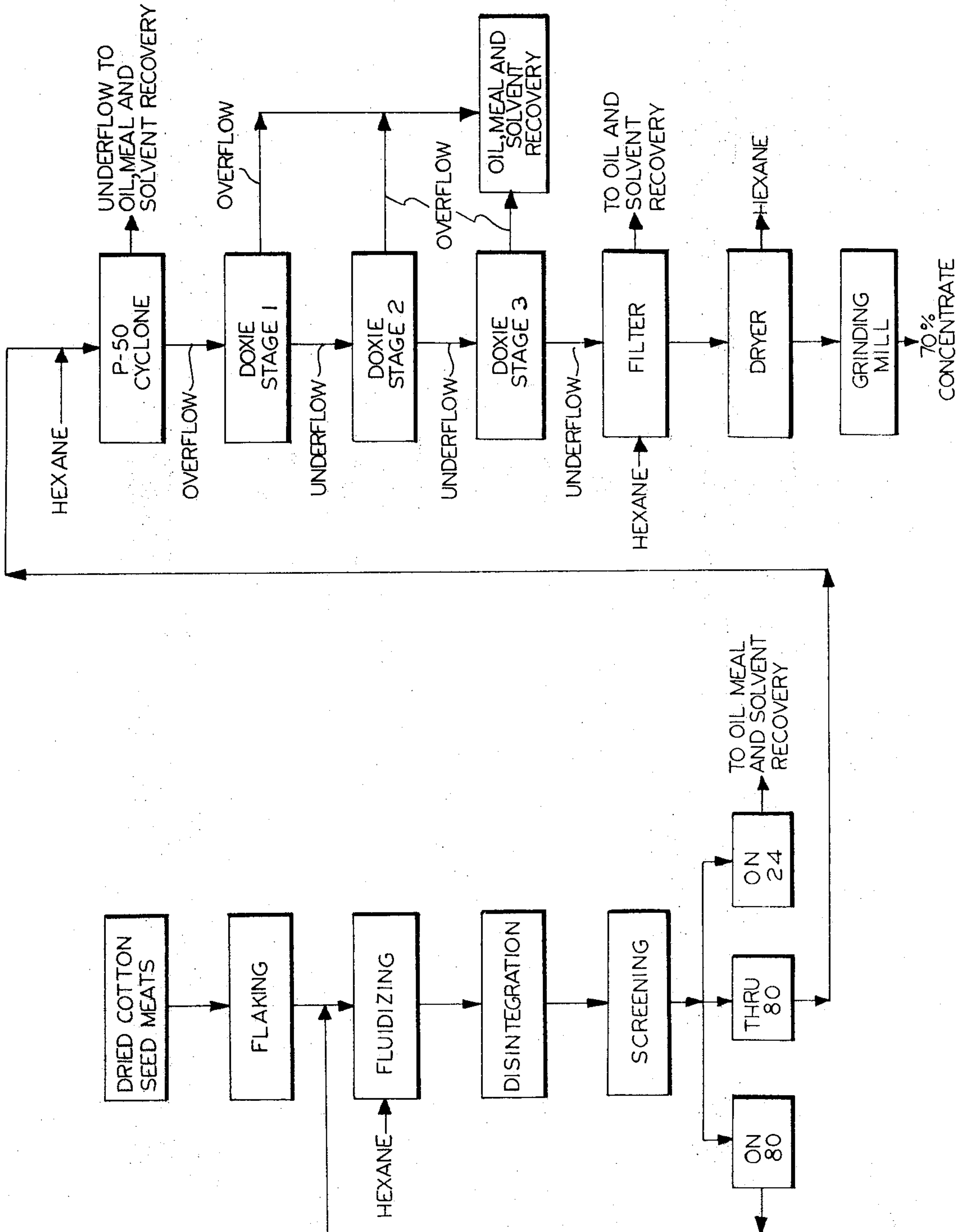

FIG. 4

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PROCESS FOR PRODUCING COTTONSEED PROTEIN CONCENTRATE

The salient features of the process, which process is applicable to either defatted or undefatted cottonseed prime in quality and free of deleterious contaminants as a starting material, comprise: rigorous control of moisture in the starting material (meats) at moisture levels well below those levels previously considered feasible in conventional oilseed milling practice; precisely controlled, practically instantaneous disintegration of the material being processed while maintaining the integrity of the gland structure to avoid dispersal of gland contents in the processed material; disintegration in a nonaqueous, nonpolar, fluid medium by use of a high-speed stone mill; and the use of vibrating screens and liquid cyclones in series.

A nonexclusive, irrevocable, royalty-free license in the invention herein described throughout the world for all purposes of the United States Government, with the power to grant sublicenses for such purposes, is hereby granted to the Government of the United States of America.

This invention relates to an industrially practical, continuous method of processing cottonseed to produce as a major end product, an edible grade of cottonseed concentrate that is high in protein content and essentially free of gossypol, oil, and hulls. The said end product is eminently suited for use as a high protein dietary supplement for human nutrition, and is of a quality and purity, with respect to its protein content, that have not hitherto been possible of attainment by contemporary processing methods.

Cottonseed is unique among oilseeds in that distributed throughout the oil and protein bearing kernel are numerous small ovoid sacs, commonly known as pigment glands. These pigment glands contain about 35 percent to 45 percent by weight of gossypol and gossypollike compounds.

By chemical analysis whole mill run cottonseed, with linters removed, contain up to about 1.5 percent of gossypol. Since the hulls contain little or no gossypol, the gossypol content of dehulled kernels is higher. If the protein content, only, of the cottonseed kernel is considered, its content of gossypol may be as high as 3 percent. This is an important consideration because as the protein content of any cottonseed product is increased by the removal of hulls, oil and other nonprotein constituents, the gossypol content will rise proportionally unless concurrent steps are taken to remove gossypol.

Gossypol is a highly reactive material, and under the processing conditions normally used including, but not limited to, moisture, heat, pressure and time, the pigment glands of cottonseed are ruptured, the gossypol is discharged and some or most of it combines with various constituents of the meal. The most usual combination appears to be with lysine, one of the essential amino acids present in cottonseed. When combined with gossypol, this essential amino acid is rendered nutritionally unavailable. Two methods of gossypol analysis are presently in use and these methods permit the determination of gossypol with a high degree of accuracy. One method determines the "free" or uncombined gossypol present. The other method determines the "total" gossypol content. The difference between the two values is referred to as the "bound" gossypol.

Cottonseed pigment glands normally are mechanically strong and resistant to rupture; however, in the presence of moisture, and particularly moisture in combination with heat, and pressure, pigment glands readily rupture and discharge their gossypol content which material is thereby brought into intimate contact with the protein, oil, and other constituents making up the kernel.

Currently, cottonseed is processed by mechanical pressing (screw pressing or hydraulic pressing), by solvent extraction with a commercial grade of *n* hexane, or by prepress solvent extraction in which a major part of the oil is first removed by screw-pressing followed by solvent extraction of the resulting press cake with commercial *n* hexane. The meal or cake produced by any of these three processes is typically adjusted to contain 41 percent protein (nitrogen × 6.25) by incorporation of cottonseed hulls that contain little or no protein. Some few commercial cottonseed crushing mills produce a meal with about 50 percent protein. The cake or meals just described (41 percent to 50 percent protein) are destined for use as animal feed. Processing conditions vary considerably in the different mills and can affect, in a significant manner, sometimes adversely, the quality and nutritive value of the cottonseed meal being produced, especially if use as a feed for nonruminants is intended.

The preparation and processing conditions employed in the aforementioned methods all employ in some degree the addition of moisture to either the kernels or the flaked meats, together with heating or cooking and the application of pressure where screw pressing, prepressing, or hydraulic pressing steps are employed. These conditions of processing are in general ideally suited to rupture the pigment glands, liberate the gossypol contained therein into intimate contact with the other kernel components, and promote the reaction of gossypol with the protein constituents of the kernel.

The presence of cottonseed pigments together with attendant processing conditions often cause the crude cottonseed oil produced conventionally to have a color so dark that the normal alkali refining and bleaching will not yield an oil prime in color. Such colored oils must be sold under a price penalty.

There is limited production at one mill in the United States of a cottonseed flour intended for human consumption. By dint of careful selection of prime cottonseed kernels low in gossypol content from an adjacent production line (producing oil and feed grade meals), by the elimination of as many hulls as possible, and by diversion of the fine meats fraction (containing much hull material) back to the adjacent production line, and by careful screw pressing of the selected kernels followed by fine grinding, a flour product is produced which is higher in quality than the feed grade meal collaterally produced. The flour product is nevertheless much higher in gossypol content and much lower in protein content, in protein quality, and is much darker in color than the cottonseed flour produced by the process of this invention.

The high grade protein concentrate produced by the process of this invention has approximately the following representative chemical analysis:

COMPOSITION

Moisture, %	4.0
Protein (Nitrogen × 6.25) %	68.0
Nitrogen, %	10.8
*Nitrogen solubility, %	98.0
Total gossypol, %	0.25
Free gossypol, %	0.05
Lipids, %	1.0
Crude Fiber, %	2.6
Ash, %	8.0
E.A.F. Lysine, g./16 g. N	3.85

*in 0.02 N NaOH

The above-listed compositional analyses are possible of achievement by reason of new discoveries that relate to the handling, preparation, and drying, of the cottonseed kernals and extraction of the rolled flakes; by the discovery of new continuous techniques in the application of equipment for disintegrating the cottonseed whereby the pigment glands are freed from their enrobing tissue, and the meal particles are finely comminuted without rupturing the pigment glands; by the discovery of a continuous screening technique whereby hulls can be removed and incompletely disintegrated meat particles can be separated for additional disintegration treatment, screening, and recycling to increase the yield of final high protein product, if desired, and to reduce the loss of protein in any diverted hull or concentrated pigment gland fractions; by the discovery of a highly efficient, rapid, and relatively inexpensive continuous process using 50 mm. and 10 mm. liquid cyclones whereby a concentrated pigment gland fraction for diversion from the process is instantly obtained from the underflow of the 50 mm. liquid cyclone, the overflow from the 50 mm. liquid cyclone being employed directly to produce a product of 65 percent or higher protein contents or if a higher protein content product is desired it may be passed through one or more stages of 10 mm. liquid cyclones whereby the slurry is concentrated or thickened to a total solids content of up to 30 percent or more and the protein content of the solids is increased to 70 percent or more; by the discovery that a slurry so thickened can be filtered on a vacuum filter at capacity rates as high as or higher than 50 pounds of solids per hour per square foot of filter area, which rate is particularly suitable for continuous vacuum filtration on a commercial-type rotary drum filter to produce a cake containing 50 percent of solvent or less which material is suitable for the final removal of solvent in commercial-type continuous or batch meal desolventizing equipment.

As an alternative to the use of the smaller, but higher gravity, liquid cyclones for increasing the solids content of the process stream prior to filtration and drying, it is possible to feed the overflow stream at the 50 mm. liquid cyclone for thickening to a filter, to an evaporator or to a settling chamber.

Since the smaller, but higher gravity liquid cyclones produce some exaltation of the process stream with respect to protein content, substitution of other methods of concentration for the function of these cyclones is made at the expense of ultimate protein content (about 65 percent vs. more than 70 percent) but the total yield of concentrate is enhanced.

We have found that a meats stream from the cottonseed hulling equipment composed of the whole meats, cracked meats, and the bulk of the fine meats can be used. This meats stream represents up to 90 percent of the protein originally present in the cottonseed. Excess hulls present are removed in subsequent screening and liquid cyclone operations.

We have discovered that when disintegrating either defatted or undefatted material in order to prevent or minimize the concurrent rupture of pigment glands it is essential first to dry the meats below 4.0 percent by weight of moisture and then to feed a very thick but flowable slurry of the cottonseed material and solvent to the stone mill. The solids content of this slurry is preferred to be from about 40 percent to 50 percent but is not limited to this precise range. Before the slurry is fed to the stone mill, it must be fluidized by vigorous agitation for about 5 to 15 minutes or more. We have discovered that the high solids content is particularly important when disintegrating defatted cottonseed material in contrast to undefatted material. We have used n-hexane successfully as a solvent but other nonpolar hydrocarbon solvents are obviously operable.

The disintegration step is carried out with a high speed (3500 r.p.m.) stone mill wherein a rolling, torsional, or rubbing action is applied to a thick n-hexane solvent slurry of the cottonseed. Clearance between the stones may be as little as 0.002 in., which clearance can be attained by precise adjustment of the spacing between the stones. Surprisingly, this action is more gentle and controllable relative to the effect on pigment glands than impact or liquid shear as has previously been proposed for the disintegration of cottonseed in solvent slurry. In addition, previously investigated modes of disintegration using liquid shear or impact (i.e., a hihh-speed blender) required 60 to 90 minutes, batchwise, when operated to disintegrate 100 pounds of flakes. Lengthy batchwise blending generates sufficient heat to cause evaporation of solvent, and also results in appreciable rupture of gossypol pigment glands with attendant binding of the gossypol so released to protein components of the meal.

We have discovered that the proper degree of disintegration, without gland rupture can be accomplished in a fraction of a second with a high-speed (3500 r.p.m.) stone mill, using a continuous feed and using discovered control techniques. In spite of the high speed, the disintegration is gentle, due to the rubbing, rolling and torsional action that takes place between the stones. It is essential that the meats be dried to less than about 4.0 percent moisture content, but they may be unextracted or extracted prior to the disintegration step.

The presently accepted method of preparing cottonseed for conventional commercial solvent extraction involves moistening the cottonseed meats to about 10 to 12 percent H_2O, drying or tempering these meats at temperatures of from 180° to 210° F. for up to 60 minutes to obtain a moisture content of about 10 percent and then flaking to a thickness of 0.007 in. to 0.012 in.

This preparation is held to be essential to the production of a flake sturdy enough to permit satisfactory percolation in a basket type of extractor and thin enough to extract satisfactorily. However, this procedure is known to rupture pigment glands thereby releasing gossypol which then combines with meal constituents to produce bound gossypol. Ruptured glands are not detectable visually. The gossypol released from the ruptured glands combines chemically and preferentially with the essential amino acid lysine thereby rendering it unavailable nutritionally. Contrary to the above-described time-honored and industry-accepted concept, we have discovered that drying the meats first to about 4.0 percent H_2O by weight or less (which drying is essential to prevent pigment gland rupture) at a temperature below 180° F. and then flaking the dried meats to a thickness of about from 0.008 in. to 0.012 in. will produce a low moisture flake that crumbles, but surprisingly results in a solvent percolation rate that is comparable to the percolation rates obtained in conventional commercial extractors.

The slurry from the stone mill is diluted to a consistency of between 10 and 15 percent total solids to aid in the screening operation. Dilution may be accomplished by the use of recycle solvent streams of lower total solids content, by use of fresh hexane, or by use of both.

Screening is accomplished with conventional, commercially available continuous screening apparatus. Three fractions are separated in the screening operation: (1) a coarse fraction consisting mainly of hulls: (2) an intermediate fraction containing a few smaller hull particles mixed with particles of the flakes that have not been sufficiently disintegrated; and (3) a fine fraction composed largely of small meats particles which have a large surface area relative to their mass, disengaged but intact pigment glands, a few smaller particles of insufficiently disintegrated flakes, and a few fine hull particles.

A **24** mesh screen is used to collect the coarse fraction. The mesh opening may be varied somewhat under different operating conditions, with different varieties of cottonseed, etc.

An **80** mesh screen is used to collect the intermediate fraction. Here, also, the mesh opening may be varied somewhat under different operating conditions with different varieties of cottonseed, etc.

The fine fraction is the material passing through the **80** mesh screen. The principal purpose of the screening operation is to obtain a slurry of fine meal particles mixed with pigment glands, which glands have been freed from enrobing tissue, and which glands are capable of separation from the fine gland-free meal particles as separate entities under the intense centrifugal action in the 50 mm. liquid cyclone.

An ideal slurry of fine meal particles would have no protein particles coarse enough or heavy enough to be removed along with the freed pigment glands. An approach to such an ideal slurry is obtained by careful attention to the disintegrating procedure.

The slurry feed is pumped at a pressure of at least 15 pounds per square inch into the tangential feed port of the liquid cyclone at its largest diameter. The resulting centrifugal action whirls the feed stream around the periphery of the interior of the bowl and exerts a centrifugal force of 5000 to 7000 times the force of gravity, depending on the pressure and rate of feed of the slurry material. This centrifugal action causes the larger, heavier, and more compact particles having the lowest ratio of surface area to mass (as typified by the ovoid-shaped pigment glands and the larger particles of meats tissue) to travel rapidly to the outer periphery of the liquid cyclone bowl. Thus these particles, which include the bulk of the pigment glands, the larger meats particles, and hull particles, are forced by the moving liquid down the tapered sides of the lower portion of the liquid cyclone to the constructed tip, or "apex," of the cyclone where they are discharged, together with a minor portion of the solvent, as underflow. The finer meal particles, which are essentially free of pigment glands and are of lower effective specific gravity than the pigment glands and coarse meal particles due to their relatively high ratio of surface area to mass, move much more slowly towards the periphery of the bowl of the liquid centrifugal and are forced upwards by the moving liquid through the vortex finder at the center of the bowl and are discharged through the vortex finder as overflow.

We have found that the underflow stream ranges in solids content from about 25 percent to about 40 percent by weight while the overflow stream ranges in solids content from about 3 percent to about 7 percent by weight, with the overflow stream amounting to from about 80 percent to about 96 percent and higher by weight of the feed stream, while the underflow stream amounts to from about 4 percent to about 20 percent of the weight of the feed stream. We have also found that the ratio by weight of the overflow stream to that of the underflow stream and the solids content of the respective streams is controlled by the rate and pressure at which the feed stream enters the tangential feed port of the liquid cyclone, the cross-sectional area of the "apex" orifice through which the underflow discharges, the makeup of the solids of the feed stream with respect to particle size, and the solids content of the feed stream. The larger, and more compact particles of the slurry which include the bulk of the pigment glands, are forced by the moving liquid down the tapered sides to the "apex," or small lower end where they are discharged as underflow (UF). The finer meal particles, practically free of pigment glands, are forced to the center of the device and are discharged upward through the vortex finder as the overflow (OF). The "apex" orifice may be varied to adjust the ratio of the weight of OF to the weight of UF. This ratio is called the "split." The underflow "apex" orifice of the **P50** liquid cyclone may be adjusted to the point where a "split" (ratio of overflow slurry, lbs., to underflow slurry, lbs.) from approximately 4 to 1 to approximately 30 to 1 is maintained. Under certain conditions smaller or larger splits may be desirable.

Liquid cyclones are available in many sizes, usually designated by the maximum inside diameter of the bowl, expressed in millimeters (mm.). Two sizes of liquid cyclones have been used in this invention, namely 50 mm. (**P50**) and 10 mm. (Doxie) cyclones. This invention is not limited to the use of these two sizes because other liquid cyclones, larger and smaller and also of intermediate size, can be used.

It should be noted in the case of the **P50** liquid cyclone and also in the case of the 10 mm. Doxies the capacity of the system may be greatly expanded by the use of multiple liquid cyclones in parallel in any of the stages. The cyclones in any of the stages will be served by a single pumping unit and supply tank.

The overflow from the **P50** liquid cyclones is pumped under a pressure of 30 p.s.i., or more, to two or more stages of 10 mm. Doxies in series. The underflow from any one stage becomes the feed for the next stage. The centrifugal action (up to 10,000 G) in the smaller diameter 10 mm. Doxies is more intense than in the larger diameter 50 mm. **P50** liquid cyclones. Most of the protein particles found in the overflow of the **P50** liquid cyclones are now found in the underflow of the Doxies. The total solids content of the underflow is considerably greater than that of the feed. The total solids content of the overflow is considerably less than that of the feed.

We have discovered two other unexpected and surprising facts in the operation of the Doxies:

1. the protein content of the solids in the underflow of each Doxie stage is greater than that of the solids in the underflow of the preceding Doxie stage and is greater than that of the solids in the feed to the first Doxie stage; also, the protein content of the solids in the corresponding overflows is reduced;
2. the total gossypol content of the solids in the underflow of each Doxie stage is less than that in the solids of the preceding Doxie stage and of the solids in the feed of the first Doxie stage; also the total gossypol content of the corresponding solids in the overflows is increased. By the use of two or more stages of Doxies the total solids content of the underflow may be progressively increased to a value as high as 30 percent.

An underflow slurry from the final Doxie stage having a total solids content of about 30 percent is suitable as feed to a continuous, vacuum drum filter. Filter tests have yielded rates as high as 50 pounds of solids per square foot of filter area per hour producing a filter cake with a solvent content of about 50 percent.

The Doxie overflows of lower total solids content may be recycled to the system in whole or in part at appropriate points, to enhance the yield of protein concentrate.

It will be obvious to those skilled in the art of oilseed processing that the protein exalting operations of the process of this invention should be applicable to other materials such as soybeans, peanuts, sunflower seed, castorbeans, and rice.

LIQUID CYCLONE PROCESS FOR HIGH PROTEIN COTTONSEED CONCENTRATE

The more detailed description of the various steø of the process that follow are more easily related one to the other by recourse to the several flow diagrams (**FIGS. 1–4** inc.) which of themselves are self-explanatory.

We distinguish four embodiments of the process. A first process embodiment wherein the cottonseed meats are dried, flaked, and extracted for removal of the oil prior to the steps of disintegrating the extracted material in a solvent, separating and concentrating the protein fraction. This embodiment yields a concentrate that exhibits a protein content of about 65 percent by weight (see FIG. **1**).

A second process embodiment parallels the several steps of said first embodiment but incorporates the additional step of passing the process stream of the protein concentrate through a series of liquid cyclones for the purpose of exalting the protein content of the finished concentrate product to at least about 70 percent by weight (see FIG. **2**).

A third process embodiment involves the steps of drying and flaking the cottonseed meats but immediately thereafter the process stream enters the phases of fluidization, disintegration in a solvent, protein separation, and concentration, with oil enriched solvent (miscella) being withdrawn from the process stream at several appropriate steps. This embodiment like that of said first embodiment is designed to yield a product concentrate of about 65 percent protein by weight (see FIG. **3**).

A fourth embodiment parallels the several steps of said third embodiment but like said second embodiment incorporates the additional step of passing the process stream of the protein concentrate through a series of liquid cyclones for the purpose of exalting the protein content of the finished concentrate product to at least about 70 percent by weight (see FIG. **4**).

DRYING MEATS (ALL EMBODIMENTS)

Meats are dried preferably to 2–4 percent moisture content at a temperature not exceeding about 180° F. Drying meats prior to extraction prevents the increase in moisture of meats tissue resulting from removal of oil, i.e., meats at 8 percent initial moisture and 33.3 percent oil, when extracted "as is," would yield oil free marc having a moisture content of about 12 percent. At this high level of moisture, pigment glands are weakened and ruptured simply by transfer of moisture to the gland walls. If the same meats are dried to a moisture content of 3 percent before extraction the moisture content of the oil free marc (on a solids basis) produced is only about 4.6 percent, a concentration insufficient to affect the pigment glands. It also appears that drying the meats tends to toughen the pigment gland walls and to loosen the attachment of the pigment glands to the enrobing meats tissue.

FLAKING (ALL EMBODIMENTS)

Meats are flaked preferably to a thickness of 0.008–0.012 inch while still warm from the drying operation. Flaking the meats while they are still warm mitigates pickup of moisture following drying. Flake thickness is controlled to prevent crushing or rupture of the pigment glands in the flaking operation while still giving a flake thin enough to expedite oil removal by extraction. The flakes produced differ from those employed in conventional extraction, due to their lower moisture content and are sandy and granular in texture.

EXTRACTING OIL (FIRST AND SECOND EMBODIMENTS)

The oil is extracted from the flakes with hexane in a conventional type of extractor to a residual lipids content of about 2 percent or less. The miscella containing the oil is routed to a conventional oil and solvent recovery system. The solvent damp extracted marc is routed to a feeder which feeds the wet marc to the liquid cyclone system through a fluidizer.

FLUIDIZING (ALL EMBODIMENTS)

Fluidizing of the wet marc to convert it into a thick, but free flowing slurry, is necessary to obtain a material of the proper consistency to feed evenly and smoothly to the stone mill and to provide a material of the proper viscosity for maximum disintegration in the mill without rupture of the pigment glands.

Fluidization is accomplished by passage of the wet marc through a pug type, baffled mixer which provides vigorous nonimpact agitation. Best results have been obtained with wet marc containing preferably about 45 percent solids and 55 percent hexane. Wet marc from the extractor may contain less than 55 percent hexane, in which case the requisite amount of additional hexane is added to the marc at the point of entry into the pug mill mixer.

Once "on stream" conditions are reached shortly after startup, the on-**80** mesh material coming from the vibrating screen is also fed to the pug mill mixer at the point of entry of the wet marc. The on-**80** mesh material consists of a slurry of about 50 percent solids, mostly of insufficiently disintegrated meats particles, and about 50 percent hexane. The solids in the on-**80** mesh stream amounts to about 15 percent of the total solids fed to the system initially in the form of wet marc.

DISINTEGRATION (ALL EMBODIMENTS)

Disintegration of the meats into ultrafine particles of meats tissue and intact glands, most of which are entirely free of adhering meats particles, without rupturing the glands, is accomplished by passing the fluidized marc from the fluidizer through a high-speed stone mill. This mill consists of two horizontally mounted coarse grit carborundum stones about 4¾-inches in diameter. The upper stone is stationary and has a center hole about 2⅛-inches in diameter through which the fluidized marc is fed. The feed opening is in the form of an inverted cone with the large end about 3 inches in diameter and terminating in a flat horizontal surface seven-eighths of an inch across. The lower stone is of the same diameter as the upper with the center portion in the form of a cone, which fits into the cone of the upper stone, and terminates in a horizontal flat peripheral surface seven-eighths of an inch across. The lower stone is mounted on an adjustable spindle which permits adjustment of the clearance between the stones from contact of the horizontal plane surfaces to 0.25 of an inch. The lower stone revolves at 3600 r.p.m.

For this operation the stones are set for a clearance of from 0.002 to 0.015 inch (preferably 0.006 to 0.008 inch) so that there is no actual contact between the stones and there is no grinding action as such. The force exerted on the material passing between the stones is a torsional, rolling, fluid shearing action which has been found to effectively disrupt the meats tissue into micron size particles and to separate the glands cleanly from the enrobing meats tissue with essentially no breakage or permanent deformation of the glands. For best results the fluidized marc should be of the maximum solids content compatible with free flow.

The milled marc is discharged directly from the mill into a tank provided with an agitator. Initially hexane is pumped to this tank at a rate such as will provide a slurry containing about 15 percent of total solids. When "on stream" conditions are attained a portion of the overflow from the second liquid cyclone (a battery of 10 mm. diameter cyclones known as the No. **1** Doxie) may be returned in whole or in part to this tank to provide a portion of the solvent for dilution and the hexane feed is correspondingly reduced.

SCREENING (ALL EMBODIMENTS)

The diluted milled marc (preferably 12–15 percent solids) is pumped from the feed tank to a vibrating screener fitted with **24** mesh and **80** mesh screens. The vibrating screener discharges three streams of slurry as follows:

A. On **24** mesh. The on-**24** mesh material contains about 1 percent of the solids in the feed to the screener and contains 60 percent to 70 percent solids. The solids of this material consist chiefly of flat hull particles, with a small amount of meats particles too large to pass through the **24** mesh screen. This on-**24** mesh material is combined with the underflow discharge from the first liquid cyclone and filtered. For embodiments one and two this cake is routed to dryers. For embodiments three and four it is washed free of oil on the filter and is then routed to dryers.

B. On **80** mesh. The on-**80** mesh solids amounts to 15 percent or less (depending on the efficiency of disintegration) of the total solids of the slurry fed to the screens and as discharged from the screen contains about 50 percent solids and 50 percent hexane. The solids consist of coarser particles of meats tissue containing embedded glands plus a small amount of hulls. This material is returned in toto to the system via the fluidizer for reworking as described under "Fluidizing."

C. Through **80** mesh. This stream contains 85 percent to 90 percent of the total solids of the input wet marc. Total solids content amounts to about 11 percent to 14 percent, with the solids being made up of the ultrafine meats particles which are free of pigment glands and constitute the desired end product, coarser meats particles containing some embedded pigment glands, pigment glands free of adhering meats particles, and some fine hull particles. This through **80** mesh slurry discharges from the screen directly into the feed tank for the first liquid cyclone, the 50 mm. diameter **P50**.

FIRST LIQUID CYCLONE P50, MILLIMETER (ALL EMBODIMENTS)

The through **80** mesh slurry from the screen, containing about 11 percent to 14 percent solids, is initially diluted with hexane in this tank to a solids content of about 7.5 percent. When "on stream" conditions are attained the hexane for dilution may be replaced in whole or in part by overflow feedback from the second liquid cyclone (the No. **1** Doxie), which contains about 1 percent solids.

The diluted slurry is maintained under vigorous agitation in the tank to keep all solids in suspension and is fed to the **P50** cyclone at 15–40 p.s.i. pressure (preferably 20–30 p.s.i.) by a pump. Classification and separation of the suspended particles in the slurry takes place in the liquid cyclone to deliver an underflow and an overflow stream. The underflow discharges from the lower tip, or "apex" of the liquid cyclone. The underflow preferably amounts to between 5 percent and 14 percent of the total slurry entering the feed aperture of the **P50** liquid cyclone and contains from about 25 percent to 45 percent of solids. The overflow discharges from the upper, or the vortex finder outlet, of the **P50** liquid cyclone. This overflow stream preferably amounts to 86 percent to 95 percent of the total slurry entering the feed aperture of the liquid cyclone and contains from about 3.5 percent to 7.0 percent of solids. The weight ratio of overflow to underflow is defined as the "split" and preferably ranges between from six parts of overflow to one part of underflow to 20 parts of overflow to one part of underflow.

The split ratio is controlled primarily by the relative cross-sectional areas of the "apex" and vortex finder orifices and the rate and pressure at which the slurry feed stream is pumped to the liquid cyclone. The solids contents of the overflow and underflow streams are also controlled by those factors but are also strongly affected by the percentage of solids in the feed stream and the degree of fineness of the solids.

The underflow contains essentially all of the intact pigment glands of the feed slurry, relatively coarse (but smaller than **80** mesh) particles of meats many of which contain embedded pigment glands, and hull particles. These solids range from 3 percent to as much as 8 percent in gossypol content and from 45 percent to 60 percent in protein.

The underflow stream is removed from the system and filtered. For embodiments one and two the cake is routed to dryers. For embodiments three and four it is washed free of oil on the filter and then routed to dryers.

The overflow stream discharges from the upper, the vortex finder outlet, of the **P50** liquid cyclone into an agitated feed tank. This overflow stream contains the extremely fine solids comprising the desired high protein, low gossypol portion of the feed stream.

SECOND LIQUID CYCLONE No. 1 DOXIE BATTERY of 10 MILLIMETER DIAMETER LIQUID CYCLONES (SECOND AND FOURTH EMBODIMENTS)

The overflow stream from the **P50** liquid cyclone is maintained under vigorous agitation in a tank and pumped from the tank through a battery of 10 mm. diameter liquid cyclones mounted in parallel at a pressure of at least 30 p.s.i. This battery of small liquid cyclones serves to concentrate the solids. Overflow from the No. **1** Doxie battery, liquid containing about 1 percent of solids, amounts to approximately 65 percent of the feed stream. The overflow stream may be exited from the system at this point or may be returned to the milled marc dilution tank, and/or the **P50** feed tank in whole or in part to serve as dilution solvent.

The underflow stream from No. **1** Doxie battery amounts to about 35 percent of the feed stream and contains about 10 percent of solids. This stream discharges into a tank and comprises the feed to the No. **2** Doxie battery.

THIRD LIQUID CYCLONE, NO. 2 DOXIE BATTERY OF 10 MILLIMETER LIQUID CYCLONES (SECOND AND FOURTH EMBODIMENTS)

The underflow discharge from the No. **1** Doxie battery is maintained under agitation in the receiving tank and is pumped to the third liquid cyclone, the No. **2** Doxie battery, at a pressure of at least 30 p.s.i. The overflow from the No. **2** Doxie battery, containing about 2 percent solids and amounting to about 60–65 percent of the feed stream may be exited from the system at this point or may be returned to the No. **1** Doxie battery feed tank.

The underflow from the No. **2** Doxie battery contains about 20 percent solids and amounts to about 40 percent of the input feed stream.

FOURTH LIQUID CYCLONE, NO 3 DOXIE BATTERY OF 10 MILLIMETER LIQUID CYCLONES (SECOND AND FOURTH EMBODIMENTS).

The underflow discharge from the No. **2** Doxie battery is maintained under agitation in the receiving tank and is pumped to the fourth liquid cyclone, the No. **3** Doxie battery, at a pressure of at least 30 p.s.i. The overflow from the No. **3** Doxie battery containing about 12 percent solids, and amounting to about 48 percent of the feed stream may be exited from the system at this point or may be returned, in whole or in part, to the No. **2** Doxie battery feed tank.

The underflow from the No. **3** Doxie battery contains about 30 percent or more of solids, and amounts to about 52 percent of the input feed stream. This high solids content stream is sent to a rotary vacuum filter.

FILTERING

The high solids stream containing the desired protein concentrate product is fed to a rotary vacuum filter which yields a cake containing about 50 percent solids. For embodiments one and two this cake is routed to the dryers. For the third and fourth embodiments the cake contains oil and is washed free of oil with solvent on the filter and is then routed to the dryers.

DRYING

The cake is heated in a suitable dryer to about 225° F. in 1 hour to coincidentally remove solvent and destroy micro-organisms.

GRINDING

After heat treatment as above, the cake is ground through a sanitary stud mill to a fine flour and packaged. The final product flour has a protein content on the order of 65 percent or higher for embodiments one and three, and 70 percent or higher for embodiments two and four, and for all embodiments, a total gossypol content of 0.30 percent or less.

The Tables 1 through 5 that follow present operational data for each of the several steps of the general process. The Tables are entirely self-explanatory.

TABLE I.—DRYING, FLAKING, AND SOLVENT EXTRACTION

Meats drying, flaking								Solvent extraction					
Meats				Flakes								Wet marc	
Lbs.	Before H_2O, percent	After H_2O, percent	Temp., °F.	Lbs.	Thick, in.	Oil, percent	Solids, lbs.[1]	Extr. time, mins.	Solvent	Temp., °F.	Resid. oil, percent[1]	Total, lbs.	Solids, lbs.[1]
110	8.5	3.6	150	100	.012	32.3	64.1	120	Hexane	80	1.4	120.5	64.1

[1] Volatiles- and oil-free basis.

TABLE II.—FLUIDIZATION AND DISINTEGRATION OF FLAKES

Fluidization								Disintegration		
Wet marc						Rate, No./min.			Rate, No./min.	
Total, lbs.	Solids, lbs.	Solvent lbs.	Solvent added, lbs.	Liquid to solids ratio	Time, mins.	Slurry	Solids	Mill setting, inch	Slurry	Solids
120.5	64.1	56.4	22.5	1.25	14.5	9.86	4.42	0.015	28.6	12.8

TABLE III.—SCREENING OF DISINTEGRATED FLAKES SLURRY

On-24			On-80			Through-80		
	Solids			Solids			Solids	
Wet, lbs.	Lbs.[1]	Percent[1]	Wet, lbs.	Lbs.[1]	Percent[1]	Wet, lbs.	Lbs.[1]	Percent[1]
20.09	9.96	49.53	29.69	13.17	44.60	259.03	40.97	15.30

[1] Volatiles-free basis.

TABLE IV.—FIFTY (50) MM. LIQUID CYCLONE—OPERATION AND RESULTS

Pumping			Slurry materials				Chemical analysis[1]		
							Gossypol, percent		
Pressure, p.s.i.	Rate, No./min.	Ratio, OF/UF[2]	Iden.	Lbs.	Solids content, percent	Solids yield, percent	Free	Total	Protein, percent
20	93.9	11.25	Feed	507.0	7.37		1.01	1.62	63.4
			OF	465.6	4.93	61.2	0.07	0.22	68.9
			UF	41.4	35.19	38.8	2.92	4.12	54.9

[1] Volatile-free basis.
[2] OF=Overflow; UF=Underflow.

TABLE V.—TEN (10) MM. LIQUID CYCLONES—OPERATION AND RESULTS

	Pumping			Slurry materials				Chemical analysis[2]		
								Gossypol, percent		
Stage No.	Pressure, p.s.i.	Rate, No. min.	Ratio OF/UF[1]	Iden.	Lbs.	Solids content, percent[2]	Solids yield, percent	Free	Total	Protein, percent
1	30	25.3	1.28	Feed	367.5	4.65		0.07	0.25	69.1
				OF	206.0	0.75	8.8	0.11	0.59	56.9
				UF	161.5	9.95	91.2	0.07	0.20	69.9
2	30	26.5	1.40	Feed	154.3	10.13		0.07	0.20	69.9
				OF	90.2	1.68	10.1	0.10	0.41	61.3
				UF	64.3	20.88	89.9	0.06	0.17	70.2
3	30	30.3	0.93	Feed	60.6	20.88		0.06	0.17	70.1
				OF	29.2	11.95	25.1	0.06	0.21	67.1
				UF	31.4	32.03	74.9	0.04	0.15	72.9

[1] OF=Overflow; UF=Underflow.
[2] Volatile-free basis.

We claim:

1. A process for producing from cottonseed meats a protein concentrate product having a low gossypol content and a protein content not less than about 65 percent, which process comprises the following steps carried out in sequence:

a. drying the cottonseed meats at a temperature below about 180° F. to a moisture content of not above about 4 percent by weight,

b. immediately flaking the still warm dried cottonseed meats to produce flakes with a thickness within the range of about 0.008 to about 0.012 inch,

c. extracting the flakes with a nonpolar hydrocarbon solvent, to obtain extracted flakes with a residual oil content below about 2 percent by weight,

d. adding solvent to the solvent-damp extracted flakes from step (c) and mixing to produce a pumpable free-flowing slurry,

e. disintegrating the solid particles in the slurry from step (d) by passing the slurry through a high speed rotary stone mill with opposed stone faces set at a clearance within the range 0.002 to 0.015 inch.

f. adding solvent to the slurry of disintegrated material from step (e) and mixing to produce a screenable slurry containing not more than about 15 percent by weight of solids,

g. screening the material from step (f) to obtain three separate streams of material segregated with respect to particle size as follows:

1. a first stream of material comprised of solvent-wet particles which remain on a screen of about **24** mesh, which material exits the process at this step,
2. a second stream of material comprised of solvent-wet particles which remain on a screen of about **80** mesh, which stream of material is returned to step (f) of the process,
3. a third stream of material comprised of solvent-wet particles which pass through a screen of about **80** mesh,

h. diluting the said third stream of material from the preceding step (g) with solvent to produce a slurry material containing from about 3 to about 10 percent of solids, and

i. feeding the diluted slurry material of step (h) at a pressure of at least about 15 pounds per square inch to a liquid cyclone, which liquid cyclone is adjusted to produce an overflow stream and an underflow stream in the ratio by weight range of about six parts of overflow to one part of underflow to 20 parts of overflow to one part of underflow, with the solids content of the overflow being in the range of 3 to 10 percent by weight and the solids content of the underflow being in the range of about 25 to about 45 percent by weight, which said underflow stream exits the process at this step for meal and solvent recovery,

j. feeding the overflow stream from the liquid cyclone of the preceding step (i) to a solids concentrator wherein the solids are partially freed from solvent, and then to a filter for further solvent removal to produce a solvent-damp material of a solids content of about 50 percent by weight,

k. removing the residual solvent from the solids of the preceding step (j) by volatilization to produce a protein concentrate having a protein content of at least about 65 percent by weight, a total gossypol content of less than about 0.30 percent by weight, and an oil content of less than about 2 percent by weight.

2. A process for producing from cottonseed meats a protein concentrate product having a low gossypol content and a protein content not less than about 70 percent, which process comprises the following steps carried out in sequence:

a. drying the cottonseed meats at a temperature below about 180° F. to a moisture content of not above about 4 percent by weight,

b. immediately flaking the still warm dried cottonseed meats to produce flakes with a thickness within the range of about from 0.008 to about 0.012 inch,

c. extracting the flakes with a nonpolar hydrocarbon solvent to obtain extracted flakes with a residual oil content below about 2 percent by weight,

d. adding solvent to the solvent damp extracted flakes from step (c) and mixing to produce a pumpable free-flowing slurry,

e. disintegrating the solid particles in the slurry from step (d) by passing said slurry through a high speed rotary stone mill with opposed stone faces set at a clearance within the range 0.002 to 0.015 inch,

f. adding solvent to the slurry of disintegrated material from step (e) and mixing to produce a screenable slurry containing not more than about 15 percent by weight of solids,

g. screening the material from step (f) to obtain three separate streams of material segregated with respect to particle size as follows:

1. a first stream of material comprised of solvent-wet particles which remain on a screen of about **24** mesh, which material exits the process at this step, for meal and solvent recovery,
2. a second stream of material comprised of solvent-wet particles which remain on a screen of about **80** mesh, which stream of material is returned to step (e) for further disintegration,
3. a third stream of material comprised of particles which pass through a screen of about **80** mesh,

h. diluting the said third stream of material from step (g) with solvent to produce a slurry material containing from about 3 to about 10 percent of solids,

i. feeding the diluted slurry material of step (h) at a pressure of at least about 15 pounds per square inch to a liquid cyclone, which liquid cyclone is adjusted to produce an overflow stream and an underflow stream in the ratio by weight range of about six parts of overflow to one part of underflow to 20 parts of overflow to one part of underflow, with the solids content of the overflow being in the range of 3 to 10 percent by weight and the solids content of the underflow being in the range of about 25 to about 45 percent by weight, which said underflow stream exits the process at this step for meal and solvent recovery,

j. feeding the overflow stream from the liquid cyclone of step (i) to the first stage of a series of stages of small diameter liquid cyclones at a pressure of at least about 30 pounds per square inch, which first stage of small diameter liquid cyclones produce an overflow stream and an underflow stream, the said overflow stream exiting the process at this step for meal and solvent recovery,

k. feeding the said underflow stream from step (j) at a pressure of at least about 30 pounds per square inch to the first stage of the remaining series of stages of small diameter liquid cyclones, which series of stages of small diameter liquid cyclones each produces an overflow stream and an underflow stream, and are so arranged that the underflow stream from each of the stages of small diameter liquid cyclones is fed to the next succeeding stage of small diameter liquid cyclones of the series at a pressure of at least about 30 pounds per square inch, while the overflow stream of each of the successive stages of small diameter liquid cyclones exits from the process for meal and solvent recovery, each of the stages in the series of small diameter liquid cyclones including that of step (j) being adapted to yield a weight ratio of overflow to underflow within the range of 1 to 1 to 1.5 to 1, with solids content of the respective underflow streams from each stage of small diameter liquid cyclones being successively exalted in each succeeding stage until the solids content of the underflow stream from the final stage of small diameter liquid cyclones of the series has reached at least about 30 percent by weight, and

l. feeding the underflow stream from the final stage of small diameter liquid cyclones of the preceding step, step (k), to a filter wherein the solids of the said underflow stream are partially freed from solvent to obtain a solvent-damp material of a solids content of about 50 percent by weight, and

m. removing the residual solvent from the filtered solids of the preceding step (step 1) by volatilization to produce a protein concentrate having a protein content of at least about 70 percent by weight, a total gossypol content of less than about 0.30 percent by weight, and an oil content, of less than about 2.0 percent by weight.

3. A process for producing from cottonseed meats a protein concentrate product having a low gossypol content and a protein content not less than about 65 percent, which process comprises the following steps carried out in sequence:

a. drying the cottonseed meats at a temperature below about 180° F. to a moisture content of not above about 4 percent by weight,

b. immediately flaking the still warm dried cottonseed meats to produce flakes with a thickness within the range of about 0.008 to 0.012 inch,

c. adding a nonpolar hydrocarbon solvent to the flakes from step (b) and mixing to produce a pumpable free-flowing slurry,

d. disintegrating the solid particles in the slurry from step (c) by passing said slurry through a high speed stone mill with opposed stone faces set at a clearance within the range of 0.002 to 0.015 inch,

e. adding solvent to the disintegrated material from step (d) and mixing to produce a screenable slurry containing not more than about 15 percent by weight of solids,

f. screening the material from step (e) to obtain three separate streams of material segregated with respect to particle size as follows:

1. a first stream of material comprised of miscella-wet particles of solids which remain on a screen of about **24** mesh, which material exits the process for oil, meal, and solvent recovery, and
2. a second stream of material comprised of miscella-wet particles of solids which remain on a screen of about **80** mesh, which stream of material is returned to the fourth step (step d) of the process for further disintegration.
3. a third stream of material comprised of the major portion of the solvent and oil (miscella) and the particles which pass through a screen of about **80** mesh, and

g. diluting the said third stream of material with solvent to produce a slurry material containing about 3 to about 10 percent of solids, and

h. feeding the diluted slurry material of the preceding step (g) at a pressure of at least about 15 pounds per square inch to a liquid cyclone, which liquid cyclone is adjusted to produce an overflow stream and an underflow stream in the ratio by weight range of about six parts of overflow to one part of underflow to 20 parts of overflow to one part of underflow, with the solids content of the overflow being in the range of about 3 to about 10 percent by weight, and the solids content of the underflow being in the range of about 25 to about 45 percent by weight, which said underflow stream exits the process for oil, meal, and solvent recovery, and

i. feeding the overflow stream from the liquid cyclone of the preceding step (h) to solids concentrator wherein the solids are partially freed of solvent and oil to produce a thick but flowable slurry comprised of solids and oil-rich miscella, which flowable slurry is filtered and washed essentially free of oil to yield a solvent-wet solids material of a solids content of about 50 percent by weight, and

j. removing the residual solvent from the solids of the preceding step (i) by volatilization in a dryer, to produce a product material having a protein content of about 65 percent or more by weight, a total gossypol content of less than about 0.30 percent by weight, and an oil content of less than about 2 percent by weight.

4. A process for producing from cottonseed meats a protein concentrate product having a low gossypol content, and a protein content not less than about 70 percent, which process comprises the following steps carried out in sequence:

a. drying the cottonseed meats at a temperature below about 180° F. to a moisture content of not above about 4 percent by weight,

b. immediately flaking the still warm dried cottonseed meats to produce flakes with a thickness within the range of about from 0.008 to about 0.012 inch,
c. adding a nonpolar hydrocarbon solvent, to the flakes from step (b) and mixing to produce a pumpable free-flowing slurry,
d. disintegrating the solid particles in the slurry from step (c) by passing said slurry through a high speed rotary stone mill with opposed stone faces set at a clearance within the range 0.002 to 0.015 inch,
e. adding solvent to the disintegrated material from step (d) and mixing to produce a screenable slurry containing not more than about 15 percent by weight of solids,
f. screening the material from step (e) to obtain three separate streams of material segregated with respect to particle size as follows:
  1. a first stream of material comprised of miscella-wet particles which remain on a screen of about 24 mesh, which material exits the process for oil, meal, and solvent recovery, and
  2. a second stream of material comprised of miscella-wet particles which remain on a screen of about 80 mesh, which stream of material is returned to the fourth step (d) of the process for further disintegration, and
  3. a third stream of material comprised of the major portion of the solvent and oil (miscella) and the particles of solids which pass through a screen of about 80 mesh, and
g. diluting the said third stream of material with solvent to produce a slurry material containing from about 3 to about 10 percent of solids, and
h. feeding the diluted slurry material of the preceding step (g) at a pressure of at least about 15 pounds per square inch to a liquid cyclone, which liquid cyclone is adjusted to produce an overflow stream and an underflow stream in the ratio by weight range of about six parts of overflow to one part of underflow to 20 parts of overflow to one part of underflow, with the solids content of the overflow being in the range of about 3 to about 10 percent by weight and the solids content of the underflow being in the range of about 25 to about 45 percent by weight, which said underflow stream exits the process for oil, meal and solvent recovery, and
i. feeding the overflow stream from the liquid cyclone of the preceding step (h) to the first stage of a series of liquid cyclones at a pressure of at least about 30 pounds per square inch, which first stage of liquid cyclones produces an overflow stream and an underflow stream, the said overflow stream exiting the process for oil, meal, and solvent recovery, and
j. feeding the said underflow stream from the preceding step (i) at a pressure of at least about 30 pounds per square inch to the first stage of the remaining series of stages of liquid cyclones, which series of stages of liquid cyclones each of which produces an overflow and an underflow stream and are so arranged that the underflow stream from each of the stages of liquid cyclones is fed to the next succeeding stage of liquid cyclones of the series at a pressure of at least about 30 pounds per square inch, while the overflow stream of each of the successive stages of liquid cyclones exits the process for oil, meal and solvent recovery, each of the stages in the series of liquid cyclones, including that of step (i), being adapted to yield a weight ratio of overflow to underflow within the range of 1 to 1 to 1.5 to 1, with the solids content of the respective underflow streams from each stage of liquid cyclones being successively exalted in each succeeding stage until the solids content of the underflow stream from the final stage of liquid cyclones of the series has reached at least about 30 percent by weight, and
k. feeding the underflow stream from the final stage of liquid cyclones of the preceding step (j) to a filter wherein the solids of the said underflow stream are washed essentially free of oil with solvent and partially freed from solvent to obtain a solvent-damp material of a solids content of about 50 percent by weight, and
l. removing the residual solvent from the solids from the preceding step (k) by volatilization in a dryer to produce a protein concentrate having a protein content of about 70 percent by weight, a total gossypol content of less than about 0.30 percent by weight, and an oil content of less than about 2 percent by weight.

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