Degossypolized Cottonseed Flour—The Liquid Cyclone Process[1]

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ABSTRACT

Predictions can be made safely that glanded cottonseed is likely to be with us for quite some time. Worldwide, 20 to 22 million metric tons of glanded cottonseed are produced annually. Hence a workable process for the removal of pigment glands is needed urgently if food-grade products are to be made from cottonseed. A brief history of the development of the Liquid Cyclone Process for the preparation of degossypolized cottonseed flour is outlined. Gossypol is removed in pigment glands via liquid cyclones, thus giving the development its name. The process consists of several unique operations including adequate drying of the meats prior to flaking, fluidizing of the flakes using commercial hexane, comminuting the fluidized slurry in a stone mill and adjusting the solids content of the milled slurry for proper separation of the fine flour from the glands, hulls and coarse meal in the cyclones. Finally, the flour is defatted and washed with hexane on a rotary vacuum filter, dried and desolventized under mild conditions to maintain protein quality. It is visualized that the above operations can be incorporated in a satellite plant operated in conjunction with a parent solvent extraction cottonseed oil mill. Sanitary conditions of the satellite plant will meet the exacting standards of the better food processing plants. Raw material specifications as well as type of plant needed and potential markets are discussed.

INTRODUCTION

Annually, the world produces over 22 million metric tons of glanded cottonseed and it is likely that production of this commodity will continue at approximately the same level (1). Potentially, this is equivalent to nearly 4.8 million metric tons of high quality edible protein flour and means the availability of some 10.6 billion pounds of nearly 70% protein flour for feeding the hungry. Cast in these figures, the big question is why isn't anyone able to get his hands on tons of such a protein product? The answer rests on the nature of the commodity—principally that glanded cottonseed cotyledons contain a collection of glands of various sizes full of pigments, predominantly gossypol. Hence, a workable process for the removal of pigment glands is urgently needed if food-grade products are to be made from such cottonseed.

DEVELOPMENT OF LIQUID CYCLONE PROCESS

This paper presents one workable method for the removal of glands from cottonseed to produce gland-free

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FIG. 1. Longitudinal and cross sections of glanded cottonseed showing pigment glands embedded in the kernel.

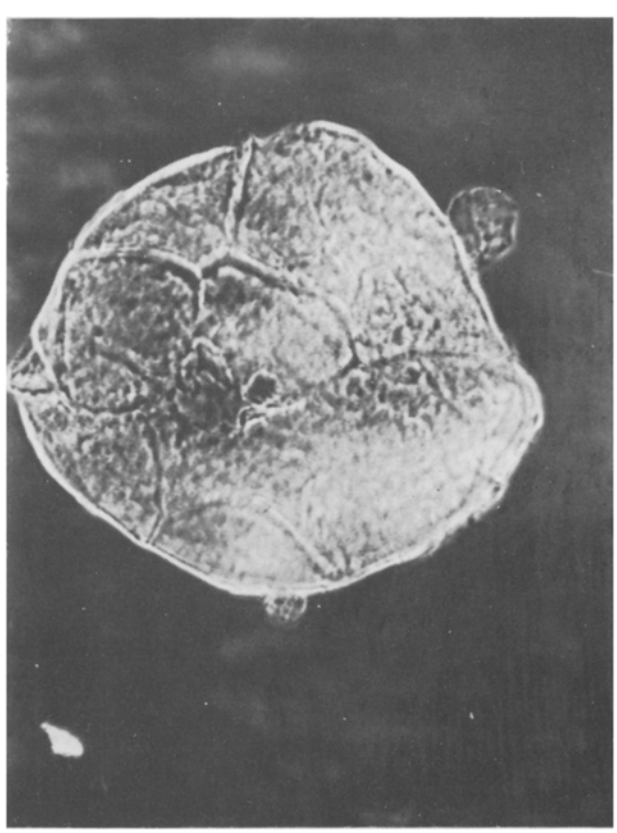

FIG. 2. An empty cottonseed gland (gossypol removed) showing platelets. Magnification 250 x, reduced approximately 50%.

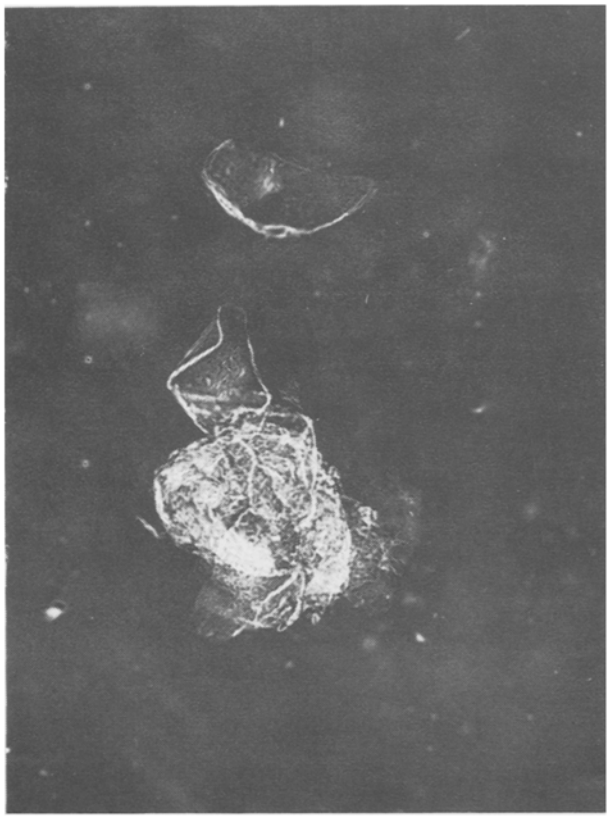

FIG. 3. Empty cottonseed gland showing separation of platelets. Magnification 250 x, reduced approximately 50%.

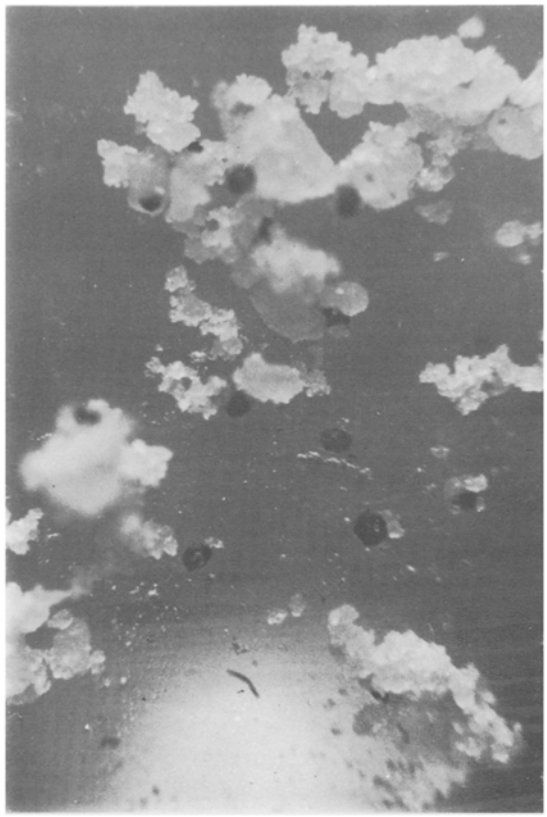

FIG. 5. Cottonseed tissue and pigment glands in hexane showing integrity of glands.

flour. A genuinely interesting facet of this presentation is that it exemplifies how utilization research can be guided to success by fundamental studies.

Nearly 25 years ago, Charlotte Boatner published a number of papers on the pigments of cottonseed (2-4). Some of this work showed that pigments were stored in the seed in separate glands (Fig. 1), that cottonseed glands were made of platelets (Fig. 2), and that the platelets can be separated from each other (Fig. 3). In the course of this work, it was discovered that polar solvents ruptured pigment glands immediately. Figure 4 illustrates pigments streaming from a gland as soon as water comes in contact with it (2). This is what happens to pigment glands in commercial processing of cottonseed, particularly during the cooking operation.

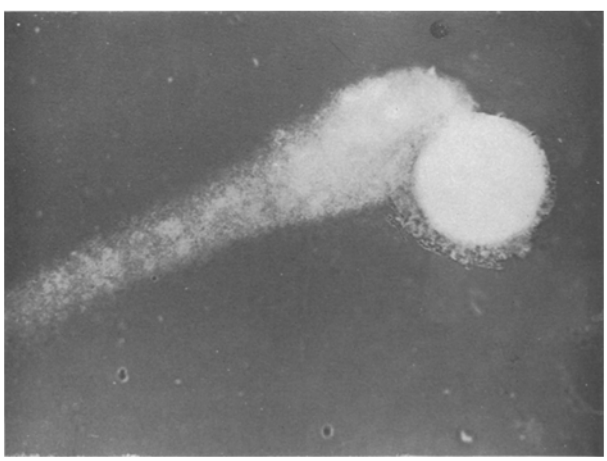

FIG. 4. A single cottonseed pigment gland showing pigment streaming out on contact with water. Magnification 250 x, reduced approximately 50%.

It was also discovered that nonpolar solvents such as hexane had seemingly no effect on the pigment glands (Fig. 5) (2). Historically, this information was translated into a gland flotation method (laboratory scale) by Dr. Boatner (5) and then upgraded to a pilot plant scale by the Engineering and Development Laboratory of the Southern Division (6). Pigment glands separated from cottonseed by the flotation process were found to suffer no detectable alteration during their isolation. The glands could be obtained almost entirely free of adhering tissue by prolonged agitation and comminution of the suspended flakes in commercial hexane. This work led to a pilot plant fractionation of cottonseed called "differential settling" (6). In this connection, Figure 6 shows an interesting scanning electron micrograph of a single pigment gland still embedded in and surrounded by cell tissue. Relative sizes of pigment glands compared to spongy mesophyll cells show why it is possible to make a separation. Comparatively speaking, a pigment gland is more than 12 times as large as one of the spongy cells. Glands range in size from 50 to 400 μ (7).

Figure 7 illustrates how the inside of a spongy mesophyll cell of a cotyledon of dry cottonseed embryo appears at a magnification of 7000 times. Each constituent is neatly packaged. The large dark gray bodies (A) containing small white globoids (G) are aleurone grains or protein bodies. Globoids (G) are sites of phytin storage (10). The elongated irregularly shaped light gray body is the nucleus (N). The small, white, round particles ringing the protein bodies are the spherosomes (S) or lipid particles, generally of the order of 0.3 to 3 μ in diameter (8,12). It is the natural packaging of the cell constituents coupled with a significant difference in size between gland and cell that is so important to processing of glanded cottonseed for edible products. Such information on the architecture of the cottonseed cotyle-

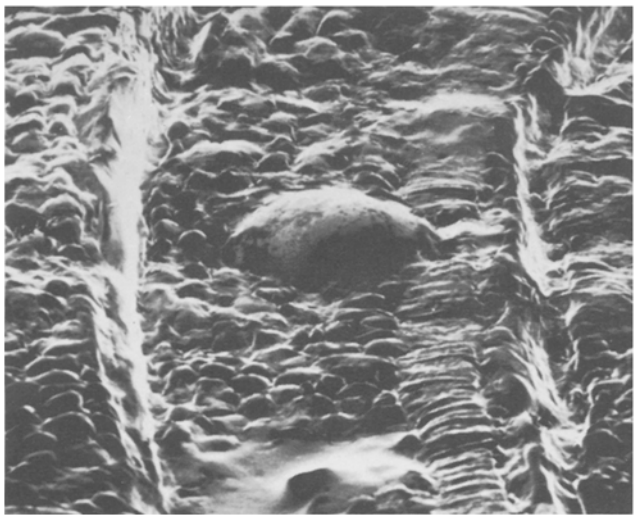

FIG. 6. Scanning electron micrograph of cross section of partially hydrated glanded cotton embryo. Large bulbous shape in center of figure is pigment gland. Small round cells are spongy tissue of the mesophyll. Courtesy of W.R. Goynes, Jr., Reduced approximately 50%.

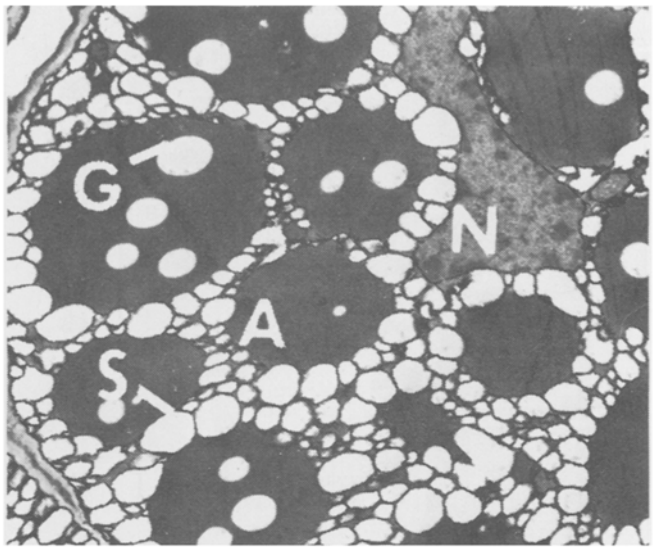

FIG. 7. Spongy mesophyll cell from cotyledon of dry cotton embryo showing aleurone grains or protein bodies (A), spherosomes (S), globoids (G) and nucleus (N). Lithium permanganate fixation. Magnification 7000 x, reduced approximately 50%. Courtesy of L.Y. Yatsu.

don originated in the basic research of Altschul and coworkers (9-12).

In a preliminary study of the characteristics of the solid components of cottonseed (meal, hulls and pigment glands) it was shown that the hulls were continuous solid particles with relatively smooth surfaces; pigment glands were compact, ovoid-shaped particles with a granular appearing surface; and, protein bodies had no definite shape, resembling a fluffy, feathery, amorphous material with a relatively large surface area per unit weight. The slow settling characteristics of the fine protein particles suggested a possible separation by frictional resistance of the liquid medium. This resistance was attributed to the texture of the fine protein bodies and their total area per unit weight. These differences formed the basis for development of differential settling of a hexane slurry of the cottonseed components. Figure 8 shows the appearance of the slurry prior to settling, after settling for a few minutes, and after settling for 12 hr (6). Note, in the middle graduate, a layer of pigment glands resting above the settled coarse meal fraction and the major fraction of fine protein particles still suspended in hexane.

It was the outgrowth of this early work based on differential settling of the components in the mixture using commercial hexane as the liquid medium that formed the foundation of the Liquid Cyclone Process (LCP) developed by Gastrock, D'Aquin, Eaves and Cross (13). Since then, the process has been refined by Eaves and Gardner (14) to yield a product which comes close to approximating the quality of flour made from glandless cottonseed. The LCP process simultaneously removes pigment glands and lipids and concentrates protein. Figure 9 is a flow diagram of the refined process upon which has been imposed sequential pictures of whole and cracked cottonseed meats, flakes, overs, unders and final flour product. Figure 10 shows one flake which displays clearly the retention of integrity of the pigment glands through the flaking process. The overs contain protein bodies relatively free of pigment glands and the unders include pigment glands with some large fragments of seed tissue as well as some small particles of hulls. The final product, the cottonseed flour, is a light cream color.

ENGINEERING SPECIFICATIONS

Engineering specifications for the LCP included preparing a whole and cracked meats fraction, essentially hull free, from prime quality, uncontaminated cottonseed; drying this fraction to a moisture content of 1.5% to 3%, flaking to 0.012 in; preparing a fluidized slurry in hexane containing about 45% solids; and passing the fluidized slurry through a horizontal stone mill to detach the slurry meat tissue from the pigment glands. The milled slurry is then diluted with additional hexane to a solids content in the range of 15-20% and pumped under optimum pressure through a 3 in diameter stainless steel Liquid Cyclone (Fig. 11) to effect separation of two fractions, an overflow containing finely divided, high protein flour es-

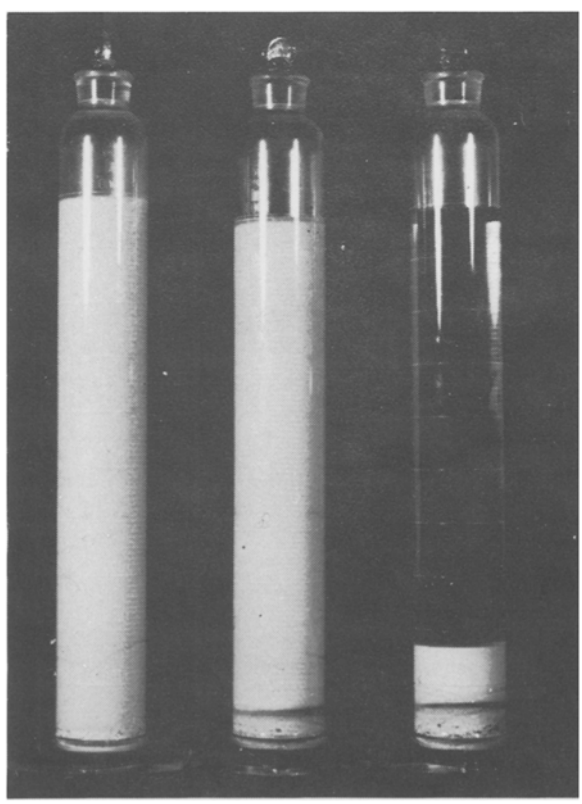

FIG. 8. Settling of hexane-slurried cottonseed flakes at different time intervals.

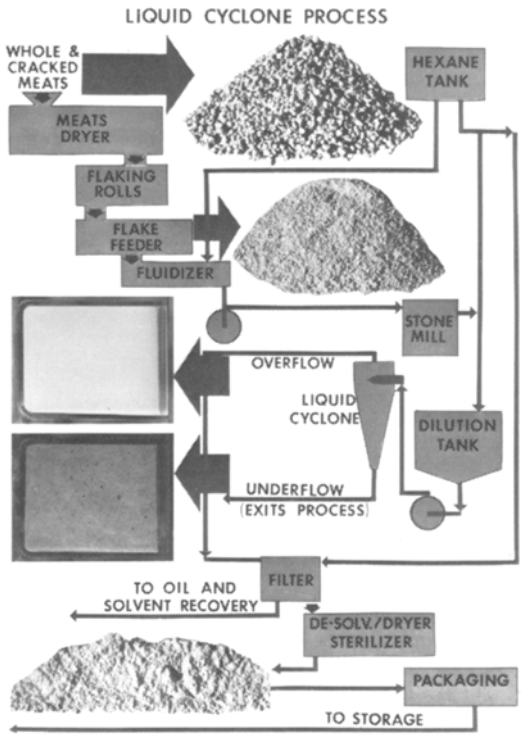

FIG. 9. Flow diagram of Liquid Cyclone Process for removal of pigment glands from glanded cottonseed.

sentially free of gossypol and an underflow containing principally pigment glands, coarse meat particles embedded with glands, and some hull particles. After filtration, washing to remove oil, desolventization, drying and sterilization, a high quality cottonseed flour is recovered. The unders may be recycled through the milling step to obtain an additional yield of fine flour product or they may be returned to the parent extraction plant for recovery as feed.

PRODUCT QUALITY AND YIELD

With the above unit operations at Southern Division, successful continuous pilot plant runs were made in which several thousands of pounds of cottonseed flakes were processed to produce over a ton of high quality, edible flour for use in evaluation studies. Composition of the product is shown in Table I. Free gossypol content is of the same low order of magnitude as that of commercially produced glandless cottonseed (0.02-0.04%) (15) and the protein content, epsilon-amino-free lysine and nitrogen solubility are equally high compared to that from glandless seed. The flour is bland in flavor and has an attractive light cream color. In fact, it approximates a concentrate in protein content, containing 68.5% protein on a moisture-free basis.

In order to retain the low free gossypol content in the final flour product, careful examination is being made of the comminution operation prior to passing the slurry through the cyclone to avoid rupturing glands. The cyclone operation is likewise being studied for the same reason. Desolventization of the flour product also offers a means of reducing the free gossypol content by causing protein and gossypol to combine.

Yields of 35-40% flour have been obtained based on the weight of solids input into the cyclone, these yields are somewhat low but high quality products have been obtained at their expense. Yields may be increased to enhance economic desirability of the process by multiple milling, remilling of the underflow, lowering the concentration of solids in the feed to the cyclone or use of two or more cyclones in series.

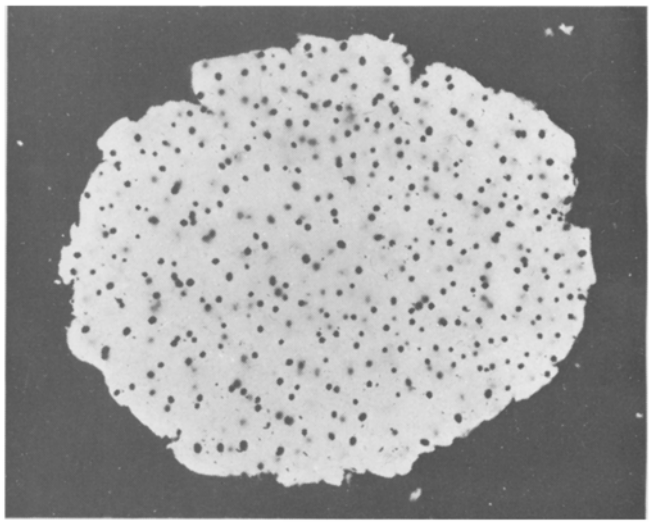

FIG. 10. Cottonseed flake showing retention of integrity of pigment glands during flaking process.

Capacity of a 3 in diameter stainless steel cyclone fed at 15% solids content-slurry ranged from 480 lb/hr of flour at 10 psig feed pressure up to 950 lb/hr of flour at 40 psig pressure. The latter figure is roughly equivalent to slightly over 10 tons/24 hr day. The lower the feed solids concentration to the liquid cyclone, the higher the yield of recovered flour. For instance, if the solids concentration of the feed to the cyclone is 10% and the underflow of the cyclone is regulated at 45% solids, the 46% of the input solids can be recovered as fine flour. However, when the solids-feed to the cyclone is increased to 25% with the same degree of milling (50% fines and 50% coarse disintegration of the flakes) then only 37% of the feed input is recovered as fine flour.

ADVANCES TOWARD COMMERCIALIZATION

There is considerable interest in the LCP flour product in the United States. A satellite plant is under consideration in one large cottonseed mill to produce flour from glanded seed. The product and process by which it is produced will probably have a significant impact on alleviating food shortages in those developing countries where cotton is grown. The process is also being evaluated successfully in India in a small-scale commercial operation by the Dorr-Oliver Company. With the advent of the LCP, there is no reason why the cottonseed industry cannot produce, under optimum sanitary conditions, a good edible flour product from glanded cottonseed. The industry already has the technology.

It is visualized that the above food raw material will be processed in a satellite plant operated in conjunction with the parent cottonseed oil mill. This satellite plant is one in which the highest sanitary conditions are maintained. The possibility of controlling bacterial count of the product during the desolventization operation is envisioned. The latter is a very important operation, not only because conditions can be maintained to make it serve as a sterilizing operation for the product, but also because it may seriously affect color of the product. Product handling after desolventization, such as grinding of the meal to produce a flour, should be carried out under conditions comparable to those used in the food industry in order to prevent recontamination.

MARKET POSSIBILITIES

It is anticipated that degossypolized cottonseed flour

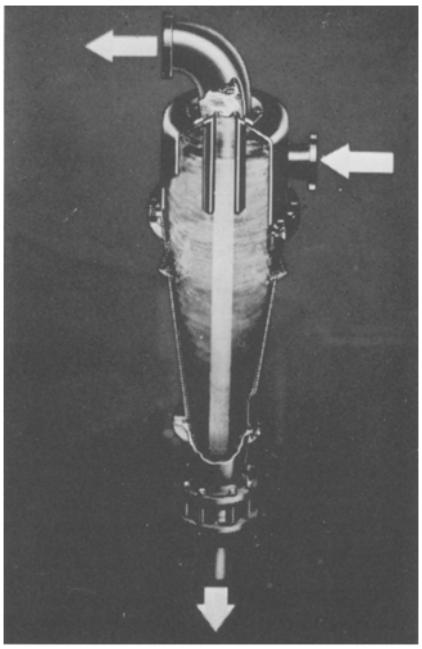

FIG. 11. Liquid Cyclone.

will be used not only in the preparation of interesting products by the food industry, but also as a basis for the preparation of other protein products such as isolates. All that is needed now is a dependable source of supply of this food-grade flour. Some work is being conducted by the meat-packing, baking and beverage industries to determine desirable functional properties. Many favorable letters have been received from food manufacturers who are currently exploring the possibility of using the LCP flour in their products. Considerable information is expected as this work develops. At the Southern Division, tasty biscuits have been prepared using the LCP four as a substitute for white flour at levels of 5%, 10% and 20%.

Present major markets for the cottonseed flour product, which closely approximates a concentrate and for isolates that may be made from it, appear to be in meat and bakery products, in textured food analogs, and in beverages. Small quantities or protein additives generally improve qualities important to the consumer such as texture, fat dispersibility and the like.

Preliminary estimates based on many experimental runs at the Southern Division lead to the expectation that costs for making the flour product will not exceed 8 to 10 cents per pound. Because the product has such a high protein content, (69% on a moisture-free basis) it is hoped that the selling price will be at least 15 to 18 cents per pound, that is, competitive with soybean protein concentrate. The high quality of the product promises to play an important part in meeting nutritional and functional requirements of food formulations desirable to many segments of the population in our country as well as that of developing nations.

TABLE I

Analysis of Flour by the Liquid Cyclone Process[a]

Components	Amount
Moisture	3.2%
Lipids	1.2%
Free gossypol	0.020%
Total gossypol	0.065%
Nitrogen	10.57%
Protein (N x 6.25)	66.1%
Protein MFB	68.5%
Nitrogen solubility (0.2N NaOH)	99.5%
EAF lysine	3.96 g/16g nitrogen
Fiber	2.3%
Ash	8.9%
(Flavor)	(Bland)
(Color)	(Light cream)

[a]Prepared at the Southern Division.

ACKNOWLEDGMENTS

Scanning electron micrograph of a cross section of partially hydrated glanded cotton embryo, by W.R. Goynes, Jr., picture of the contents of a cottonseed cell by L.Y. Yatsu; artwork by Nancy Meadows; and photography by A. Fayette and Jack Bergquist.

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