Processing Methods to Preserve Quality and Color of Cottonseed Flakes

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Abstract

Hull-free cottonseed flakes were prepared for direct solvent extraction by two passes through a pilot plant size, hot air drier set at 200 F. Moisture content was reduced to 6.5% without evidence of gossypol binding. Improved drainage characteristics resulted in better extraction. Glanded and glandless flakes which had been extracted and partially desolventized at low temperatures were heated indirectly in a batch desolventizer under vacuum to temperatures above the point of steam condensation and solvent stripped with superheated steam. Resulting available lysine and protein solubility values on the meals and flours were desirably high. The desolventization was accomplished without significant color darkening.

Introduction

Cottonseed protein is being viewed more intently today than ever before as a potentially important source of protein for feeding both humans and monogastric animals. Rapidly expanding populations have focused world attention on existing and impending needs for additional supplies of protein for food uses. Cottonseed is one of the principal oilseeds of the world. Its protein represents roughly 6% of the world supply (1). Thus, forward-looking cottonseed processors conscious of these needs and of the potential markets resulting from them are seeking processing methods that minimize damage to the nutritional and functional properties of their protein product.

The advent of the new glandless varieties of cottonseed has greatly reduced the problems associated with the production of cottonseed protein suitable for food use. Prior to the genetic development of gland-free, gossypol-free varieties, it was necessary in processing either to remove the gossypol present in pigment glands, or to render it non-toxic. When gossypol is not present to react with the protein and lessen its nutritive value, heat and moisture become the most important processing variables. These factors cause reduction in protein quality and darkening of meal color during desolventization operations as presently practiced (2).

The investigations reported herein were begun in search of processing methods which would substantially reduce protein quality losses occurring during meal desolventization and enhance the color of the finished products from both glanded and glandless seed.

Experimental Procedures

Preparation of Flakes for Extraction

A method of preparing flakes for direct solvent extraction different from methods now in commercial use was devised at the Oilseed Products Research Center (OPRC). In one series of tests, hull-free glanded cottonseed kernels were moistened to 11.5% moisture for flaking with smooth, one pair high flaking rolls, to a thickness of 0.010 in. Small lots of these flakes were then dried in a laboratory oven at a constant temperature of 180 F for different time intervals. Samples were taken at the end of each interval and analyzed for moisture and for free and total gossypol.

In a second series of tests, flakes containing 11.5% moisture were oven dried for a 1 hr period at each of several temperatures ranging from 180 to 220 F. Samples of dried flakes were again analyzed for moisture and for free and total gossypol. None of the samples analyzed showed evidence of gossypol binding as a result of the oven heating.

Drying tests were next conducted on larger scale equipment in the OPRC pilot plant. A rotary drier was utilized which employed a stream of hot air flowing countercurrently to wet flakes moving through it. The drier consisted of a cylindrical chamber 2.5 ft in diameter and 11 ft long rotating on its horizontal axis with the discharge end lower than the feed end. The stream of air was heated to 200 F by a gas flame as it was pulled into conducting pipe leading to the suction of a centrifugal fan which forced the hot air through the revolving cylindrical chamber and out the feed end. Lifting plates on the inside of the cylinder continually elevated the flakes and dropped them through the current of hot air. The charge was moved downward to the discharge end by the inclination of the chamber. Two passes of the flakes through the drier proved to be sufficient to reduce the moisture content from 11.1% to the target moisture of 6.5%. Flakes were fed into the drier at the rate of 90 lb/7 min.

Glanded flakes, thus dried, were direct-extracted in four, 5-gal batch extractors with the solvent mixture of acetone, cyclohexane and water (A:C:W) developed at the OPRC for removing gossypol and oil simultaneously (2). The extraction procedure followed was designed to simulate a countercurrent-type extractor. Flakes were extracted over a 120 min period with solvent temperatures maintained between 120 and 125 F. A solvent to flakes weight ratio of 7 to 1 was used.

Modification of Conventional Desolventization Procedures

A 5 gal extractor load of A:C:W-extracted glanded flakes was desolventized without heat to a residual moisture and volatile content of 9.94%. Samples of the incompletely desolventized flakes were then placed in hot, dry air circulating through a forced-draft laboratory oven. Excessively high temperatures of 240, 260, 280 and 300 F were maintained on these samples, which were tested in triplicate, for periods of 1 hr. At the start of heating, the protein solubility was 85.40%. The effects of treating these low moisture flakes to abnormally high temperatures were studied by observing any changes in protein solubility or color that resulted.

An objective measurement of lightness of color in desolventized meal was achieved by using a Gardner

[1] Operated by the Texas Engineering Experiment Station for the Cotton Research Committee of Texas.

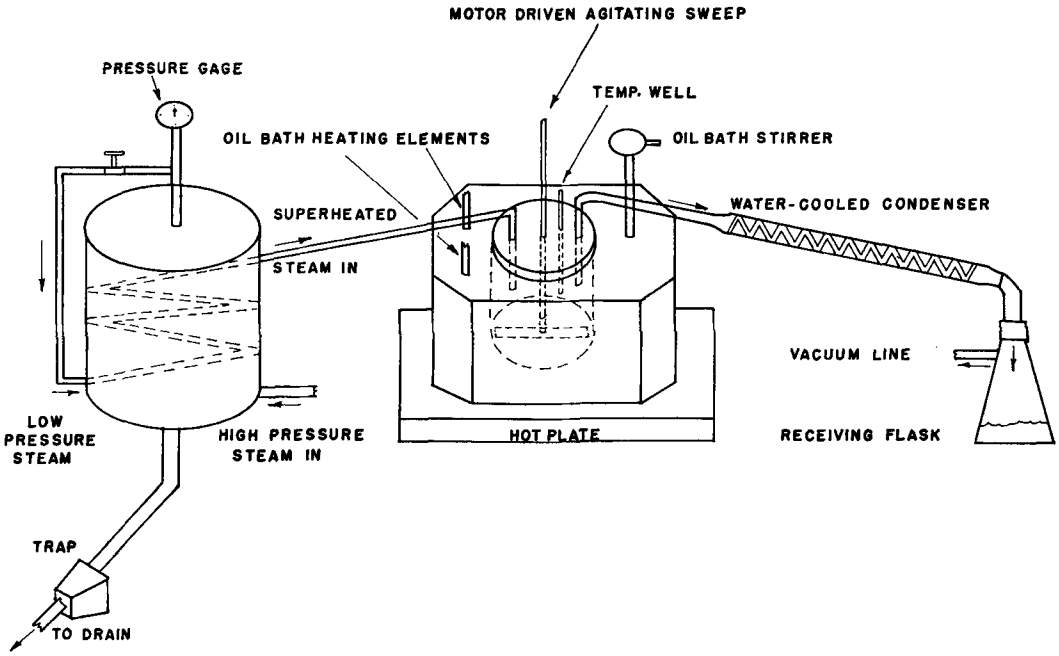

FIG. 1. Seven inch diameter batch desolventizer with steam superheater.

Automatic Color-Difference Meter. This instrument compares a test specimen with standards of predetermined color characteristics. Its instrumental precision is comparable to the smallest color difference discernible to the trained human eye. The standard deviation of readings obtainable by one operator using one instrument in a particular laboratory is 0.20, according to manufacturer's claims. Readings were taken using the R_d and L scales of the meter. Color values chosen to represent the different samples were a numerical average of the readings obtained using these two scales.

In a second phase of the investigations, superheated steam was employed as a medium for solvent stripping cottonseed flakes from which most of the solvent had already been removed at low temperatures. Experimental runs were made using a laboratory scale 7 in. diameter batch desolventizer. The desolventizer was equipped with a superheater positioned to supply superheated steam to material in the kettle (Fig. 1).

In the cycle of operation, a charge of A:C:W-extracted glanded flakes containing 22.45% moisture and volatile matter was placed in the kettle. A period of indirect heating followed in which heat was applied to the kettle from an electrically heated oil bath and an electric hot plate. Heating continued for approximately 10 min until the inside temperature was elevated to a point at which steam would not condense under the average vacuum of 15 in. Hg imposed on the system. Superheated steam was then turned into the kettle for a specified time interval and throttled to achieve a particular maximum inside temperature, in each run. The indirect heat sources were also regulated to assist in controlling material temperatures. Temperature levels were established in a pre-planned experimental design.

Solvent vapors driven off during the indirect heating period were pulled from the desolventizer through a water-cooled glass condenser and collected for measurement. Similarly, the dry, superheated steam that passed through the material during steaming was pulled from the kettle and condensed along with the remaining solvent stripped out. A rotating sweep agitated material in the desolventizer.

The first series of runs with the desolventizer and superheated steam was carried out in random order using a simple statistical design with two-way classification of experimental units. The moisture and solvent content of the material charged to the kettle was held constant and inside temperature and steaming time were varied. The response variables measured in the treated charges of material were protein solubility and color. An analysis of variance was performed on the solubility and color data to determine if any observed differences between treatments were significant.

Inside temperatures and steaming times followed in the design appear in Tables IV, V and VI along with data taken on the charges of desolventized flakes.

In addition to the runs using A:C:W-extracted glanded flakes, a second series of three runs using hexane-extracted glandless flakes was made. These flakes also were partially desolventized at low temperatures before being charged to the desolventizer. Each charge was steamed for 20 min at one of the three temperatures previously used. Tables IV, V and VI contain data on the desolventized glandless flakes as well.

Available lysine determinations were performed on three glandless and three glanded meal samples desolventized with superheated steam. The six samples selected were all steamed for 20 min periods. Available lysine was also determined on the partially desolventized glandless material and on a sample of glanded flakes heated to 220 F for a 20 min period without steaming. Available and unavailable lysine values on the six steamed samples and partially desolventized glandless material are reported in Tables IV, V and VI. Available and unavailable lysine values for the heated but unsteamed sample were 4.10 g/ 16 g N and 0.33 g/ 16 g N, respectively.

Available lysine was determined by reaction of the sample with fluorodinitrobenzene and column chromatography on a Beckman amino acid analyzer (Lyman and Cater, manuscript in preparation). Protein determinations were made using the Kjeldahl method. Soluble protein percentages reflect the portion of the protein that was soluble in 0.02 N NaOH.

TABLE I

Test Data on Cottonseed Flakes After Hot Air Drying and Direct Solvent Extraction: Operational Data

Sample treatment	Temperature air stream F	Flakes temperature F	Flakes moisture F
Flakes entering drier	200	amb.	11.12
After 1st pass through drier	199	147	8.48
After 2nd pass through drier	198	160	6.54
After 3rd pass through drier	196	164	4.94

Results and Discussion

Preparation of Flakes for Extraction

Two possible applications are envisioned for the hot air drying method of flakes preparation: in direct extraction of glandless or glanded hull-free flakes and in direct extraction of glanded flakes with water miscible solvent mixtures.

To consider the first of these applications, cottonseed protein products for food use must be essentially free of hulls and lint fiber. Currently, the preferred way to assure products desirably low in fiber content is to remove all lint and hulls from kernels prior to rolling. However, direct extraction of hull-free flakes may produce operating problems if conventional conditioning methods are used. The absence of hulls results in a flakes bed of lower permeability creating solvent drainage problems in some types of extractors (3). This is true for both glandless and glanded flakes.

Hot air drying as used in these investigations yielded flakes that were more resistant to breakage. The toughened character and lower moisture content of the flakes resulted in increased permeability and better drainage in batch extractions at the OPRC. Swelling within stationary beds of flakes, ordinarily troublesome with polar solvents, was greatly reduced. Data in Table II show the nitrogen solubility of flakes after drying and extraction to be desirably high.

Hot air drying was first incorporated in research at the OPRC involving the second application suggested. The A:C:W solvent mixture employed to degossypolize glanded flakes has acetone, a polar solvent, as its major component. Solvent systems containing such a water miscible solvent tend to remove water from flakes during extraction. This produces an imbalance in the solvent mixture, and necessitates removal of the excess water before the mixture can be readied for reuse. Experimentation has shown that, by drying flakes ahead of extraction, water can be induced to flow from the solvent into the flakes (2). A moisture content of 6.5% proved to be sufficiently low to achieve this using a water component of 1.8% by weight in the starting A:C:W mixture.

TABLE II

Test Data on Cottonseed Flakes After Hot Air Drying and Direct Solvent Extraction: Analytical Data

Sample description	Moisture and volatile %	Protein %	Protein solubility %	Gossypol[a] free	Gossypol[a] total
Flakes entering drier					
Run A	10.92	34.81	.....	0.462	0.487
Run B	11.30	.....	.....	0.427	0.502
Flakes after drying					
Run A	6.73	37.08	.....	0.490	0.515
Run B	5.70	37.69	.....	0.509	0.593
Dried flakes after extraction (desolventized without heat)					
Run A	2.20	54.44	91.9	0.058	0.232
Run B	3.60	58.88	96.1	0.069	0.225

[a] Fifty per cent protein basis.

TABLE III

The Effect of Desolventization With Dry Oven Heat at High Temperatures on Protein Solubility and Meal Color

Sample treatments	Moisture and volatile %	Protein solubility %	Color reading
Unheated starting material	9.94	85.40	73.5
1 hr in oven at 240 F	1.69	85.09	71.4
1 hr in oven at 260 F	1.37	84.65	70.0
1 hr in oven at 280 F	1.59	82.34	69.5
1 hr in oven at 300 F	1.13	79.58	67.3

Thus, hot air drying was used to lower the moisture in glanded flakes to this target moisture for A:C:W extractions.

Glanded cottonseed meals and flours extracted with solvent mixtures having acetone as a component characteristically possess objectionable odors and flavors. While such off-flavors were not a primary subject of study in these investigations, samples of the finished glanded products were, nevertheless, tasted. Off-flavors were found to be present in the glanded meals and flours.

Desolventization of Extracted Flakes

Conventional desolventization practices in which extracted flakes are subjected to moisture and heat in combination result in meals that are darker in color and lower in nitrogen solubility and available lysine. Historically, processors have observed this with glanded flakes and were less concerned about it because of the need to inactivate gossypol in the meal and because the end use of the meal was for animal feeds. However, in processing glandless seed for use in human foods, it is imperative that its excellent nutritive and functional properties be preserved.

In the most significant commercial processing trials to date with glandless seed conventional desolventization practices were basically followed. The production meal was described as having a grainy texture and darkened color by one observer (3) and as being light brown by another (4). The protein fraction which did not pass through the desolventizer was reported to be white in color. Soluble nitrogen values in production meal from these trials ranged from 74.2% to 81.4%. In the work reported herein an alternative to the use of live steam, which in conventional practice condenses on the material being desolventized, was investigated.

As shown in Table III, extracted glanded flakes containing low moisture experienced a relatively small reduction in nitrogen solubility with little color darkening when subjected to dry air at excessively high temperatures. Analytical and color values shown in Table III are a numerical average of values for three samples.

Results from experimental runs using dry, superheated steam under vacuum to strip solvent from partially desolventized flakes were decidedly favorable. As demonstrated by the data in Tables V and VI, the protein solubility and available lysine

TABLE IV

Experimental Treatments Applied in Desolventization and Analyses of Resulting Products: Materials Before Desolventization

Seed type	Moisture and volatile %	Protein %	Color	Protein solubility %	Lysine available	Lysine unavailable
Glanded	22.45	57.19	73.4	93.40	.....	.....
Glandless	24.06	53.06	83.5	96.93	4.16	0.22

TABLE V

Experimental Treatments Applied in Desolventization and Analyses of Resulting Products: Glanded Meals

Inside temperature F	Time steamed in desolventizer							
	10 min		20 min				30 min	
	Color	Protein solubility, %	Color	Protein solubility, %	Lysine		Color	Protein solubility, %
					available	unavailable		
200	67.8	91.32	65.7	90.61	3.92	0.33	66.3	89.81
220	65.6	91.51	64.4	91.33	3.90	0.36	64.0	87.56
240	64.0	91.48	65.4	89.10	4.04	0.35	65.3	88.17

values in the finished products were desirably high and the desolventized meals remained light in color.

An analysis of variance was performed for each of the variables, protein solubility and color using the data obtained for glanded flakes. The differences between temperatures and between steaming times for both variables were found to be nonsignificant at the 5% level.

By desolventizing under vacuum, less heat had to be added to flakes containing solvent to elevate the temperature above the point of steam condensation. Solvent could thus be vaporized and removed without subjecting the flakes to the higher temperatures that would otherwise have been necessary. Also, a shorter heating time prior to steaming was needed to achieve the required inside temperatures. No runs were made without vacuum. However, data obtained indicate similar results may be expected when vacuum is not used.

TABLE VI

Experimental Treatments Applied in Desolventization and Analyses of Resulting Products: Glandless Meals

Inside temperature F	Time steamed in desolventizer, 20 min.				
	Moisture and volatile %	Color	Protein solubility %	Lysine	
				available	unavailable
200	6.20	79.2	97.40	4.13	0.15
220	4.00	80.3	95.14	3.87	0.15
240	3.23	79.6	94.74	3.94	0.18

Indirect heat from an oil bath was utilized to elevate material temperatures to the steaming point in these experiments. In commercial desolventization equipment indirect steam would instead be used and could be supplemented if necessary with heat from superheated solvent vapors pulled through a stage of the desolventizer as is done in conventional vapor desolventization.

These experiments indicate desolventization of low moisture, extracted cottonseed flakes with superheated steam to prevent moisture addition holds promise of measurably improving protein quality and color in meals and flours.

ACKNOWLEDGMENTS

C. M. Cater of the Oilseed Products Research Center furnished available and unavailable lysine analyses on the extracted flakes used in the desolventization study.

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[Received February 3, 1969]