TABLE VI

Polyester				Polyepoxide				
Species	Iodine value	Olefin equivalent weight	Acid value	% Resin isolated	% Oxirane oxygen	Epoxide equivalent	% Conversion to epoxide	Acid value
THPA[a]-1,5-pentanediol	98.1	258.8	3.4	89.6	4.82	333.8	80.4	2.3
THPA-1,4-butanediol	93.0	273.0	8.6	89.5	5.27	303.3	95.3	10.7
THPA-ethylene glycol	101.5	250.1	3.9	90.0	5.96	268.6	99.1	6.2
THPA-diethylene glycol	104.6	242.3	10.1	93.2	4.82	331.6	77.9	13.2
Unsaturated alkyd	78.9	321.2	10.5	96.1	3.36	474.8	71.0	11.5

[a] THPA—Tetrahydrophthalic acid.

epoxides are unique in that they may be formulated to have a large number of epoxide groups per molecule; 10 or more may be practically obtained.

An *in situ* process for the formation of peracetic acid, using dehydrated cation exchange resin of the styrene-divinyl benzene sulfonic acid type, has been described for the efficient epoxidation of the unsaturated polyesters reported.

A dehydrated strong base anion exchange resin will remove free acid, the major impurity resulting from the peracid epoxidation reaction, without contaminating the polyepoxide polyester with appreciable water and without appreciable loss in epoxide content during the purification. Alternatively co-distillation of the free acid with aromatic solvent *in vacuo* may be used as a method of purification but with some loss in epoxide content. These techniques are desirable in preparations of compositions of the type described wherein the usual washing techniques cannot be employed.

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Pilot Plant Development of the Alkali Cooking Process for Cottonseed Meats. II. Effect of Additional Comminution

NESTOR B. KNOEPFLER, W. H. KING, M. F. STANSBURY, and E. J. McCOURTNEY,
Southern Regional Research Laboratory,[1] New Orleans, Louisiana

COTTONSEED MEALS of low free-gossypol content, high nitrogen solubility, and high nutritive value can be produced by processing the flaked meats at high moisture levels in the presence of alkali (3, 4). Under these conditions the pigment glands are ruptured and the gossypol inactivated without adversely affecting the nitrogen solubility. At the same time, semi-refined crude oils, depending upon the amount of alkali used, are produced. The preparation procedure consists of conventional hulling and rolling, followed by the addition of sufficient sodium hydroxide, in the moisture adjustment phase of the operation, to bring the pH to 8.2, mixing or prestirring at moisture levels of 25 to 40%, and then cooking at low temperatures under conditions which effect evaporative dehydration. The usual range of free-gossypol content of meals produced by the above method is from 0.02 to 0.04% and is considered low enough for unlimited use of the meal in growing chick and broiler rations but not low enough to prevent discoloration of eggs when fed to laying hens (2).

Earlier work disclosed the tendency of increased cooking temperatures and time in the presence of high moisture content to reduce the free-gossypol content of the meal. Concurrently, however, such conditions resulted in decreased nitrogen solubility of the meals produced. The only variable studied which showed a tendency to reduce the free-gossypol content of the meal without adversely affecting the nitrogen solubility was the fineness of comminution (flake thickness) of the raw cottonseed meats. This indicated that finer comminution than had previously been used might result in a further reduction of the free-gossypol content of the meal without a corresponding reduction in the soluble nitrogen content. Experiments were designed to determine the effects of additional comminution by grinding after rolling (or flaking), of temperature and moisture during rolling, and of the addition of alkali upon the characteristics of the meals produced.

The close relationship between the extent of comminution and the mixing operation, which is usually performed after the moisture and alkali are added to the comminuted raw meats, indicated that some additional reduction in the free-gossypol content of cottonseed meals could be expected during mixing. The present study therefore includes experiments designed to determine the optimum conditions for mixing, which would accomplish reduction in free-gossypol content of the meal without effecting a reduction in nitrogen solubility.

Tables I, II, and III outline the experimental plan for the evaluation of the effects of the variables under consideration.

Equipment, Material, and Methods of Analysis

Pilot-plant scale Carver cleaning, hulling, and purification equipment[2] was used to prepare the essentially hull-free meats used for these experiments. Flaking was performed on a pilot-plant stand of five-high rolls (7). A Bauer peanut butter mill and a

[1] One of the laboratories of the Southern Utilization Research Branch, Agricultural Research Service, U. S. Department of Agriculture.

[2] The use of trade names does not constitute an endorsement by the Department of Agriculture of those products over similar products of other manufacturers.

Bauer high speed disc mill No. 148 were used in the experiment, which called for additional comminution subsequent to flaking. Mixing and cooking were conducted in the Loomis mixer (3).

One lot of 1953 crop prime cottonseed was used for this work. This same lot of seed was used in previously reported work (3) and had been stored for eight months.

Samples were analyzed for moisture and free-gossypol content by the official methods of the American Oil Chemists' Society (1), for total gossypol by the method of Pons, Hoffpauir, and O'Connor (6), and for nitrogen solubility in 0.02 *N* alkali by the method of Lyman *et al.* (5).

Experimental Procedures and Results

The Effects of Grinding in a Disc Mill at Different Moisture Levels at pH 8.2

The meats were adjusted to 9% moisture content and then flaked in the five-high rolls to an average thickness of 0.008 in. The flakes were then placed in the Loomis mixer and simultaneously adjusted to the pH and moisture content required by the plan (Table I). This was accomplished by the addition of a solution of sodium hydroxide of appropriate concentration (3) while constantly mixing. Mixing was continued for five minutes after the addition of the alkali water to obtain reasonable uniformity. The material was then force-fed through the peanut butter mill, using 0.020-in. clearance between grinding discs. Multiple repassing of the material, where required, was carried out as rapidly as possible so that temperature changes occurring would be cumulative and the moisture loss would be held to a minimum. In each instance the grinding was accompanied by a rise in temperature of the meats from 90°F. to 140–160°F.

TABLE I

Experimental Plan for the Investigation of the Effects of Grinding, in a Peanut Butter Mill at Various Moisture Levels, After Rolling

Moisture content at grinding %	Passes through mill						Cooking 45 min. 180°F. maximum
	1	2	3	4	5	6	
12[a]	X	X					
18	X						X
24	X						X
31	X	X	X	X	X	X	X[b]

[a] No pH adjustment made at this moisture level. All other experiments made at pH 8.2.
[b] Material from the sixth pass at 31% moisture content.

Certain of the materials prepared in these experiments were evaluated by cooking for 45 min. at 180°F. (Table I). The results of the cooking phase will be discussed in connection with the effects of time, temperature, and moisture content during cooking.

The ground meats, after cooling, were partially extracted with hexane and allowed to air-dry for 16 to 48 hrs. at room temperature. The air-dried material was then ground and re-extracted with hexane and air-dried as before. This procedure was followed for all experiments to provide uniform treatment of materials for subsequent chemical analysis.

Some of the characteristics of meals from materials treated in this manner are given in Table IV. The data indicate that the free-gossypol content of flaked cottonseed meats can be reduced by comminution. The magnitude of the reduction is influenced by the amount of moisture present during the grinding step. A very sharp decrease in the free-gossypol content of the ground meats was observed as the moisture content was increased from 12 to 18%, with a small additional decrease as the moisture content was brought up to 24%, and a still smaller additional decrease as the moisture content was brought up to 31%. To a lesser extent the reduction is also related to the number of times the material is passed through the mill. The rate of reduction decreases with each successive passage through the mill. For example: at 31% moisture content, the free-gossypol content was reduced from 0.930 to 0.071% in one pass through the mill. A further reduction to 0.057% resulted from a second pass through the mill, and a still further reduction to 0.047% resulted from four additional passes through the mill. Power requirements were high, and the materials were difficult to handle under the conditions studied. The grinding operation in all experiments was accompanied by a rise in temperature, which probably accounts for at least a part of the reduction in nitrogen solubility that is shown at all moisture levels of grinding above 12%.

One batch of flakes was cooled to —10°F. and ground, with concurrent addition of pellets of dry ice, through the high speed Bauer mill. The grinding discs were set at the closest spacing which would allow passage of the material without heating and gumming. The grinding operation reduced the free-gossypol content of the material from 1.08 to 0.78% without affecting the nitrogen solubility. Some difficulty was encountered in this experiment due to the condensation of moisture on the mechanical equipment.

The Effects of the Conditions of Rolling

Moisture. To determine the effects of moisture content during rolling upon the properties of the resulting meals, a series of experiments were conducted using meats containing 4, 8, 10, and 13% initial moisture content (Table II). The 4% moisture content meats were obtained by drying over-night in a forced-draft oven at a maximum temperature of 125°F. The 8, 10, and 13% initial moisture content meats were obtained by the direct addition of water to the meats and equilibrating for one hour before rolling.

TABLE II

Experimental Plan to Investigate the Effects of Moisture Content During Rolling Upon the Characteristics of Cottonseed Meals

Initial moisture content at rolling %	One pass through rolls	Three passes through rolls	One pass through rolls followed by grinding in Bauer mill (Number 148) with dry ice
4		X	
8	X[a]	X	
10			X
13		X	

[a] Separate batches mixed for 120 min. at 90°F., pH 8.2, and 18, 24 and 31% moisture content. All other flaked meats mixed for 120 min. at 31% moisture content, pH 8.2, and 90°F.

The results (Figure 1) showed that the free-gossypol content of the material was inversely related to the moisture content during rolling. The values for moisture content plotted in this figure, as well as in Figure 4, are the actual analytically determined

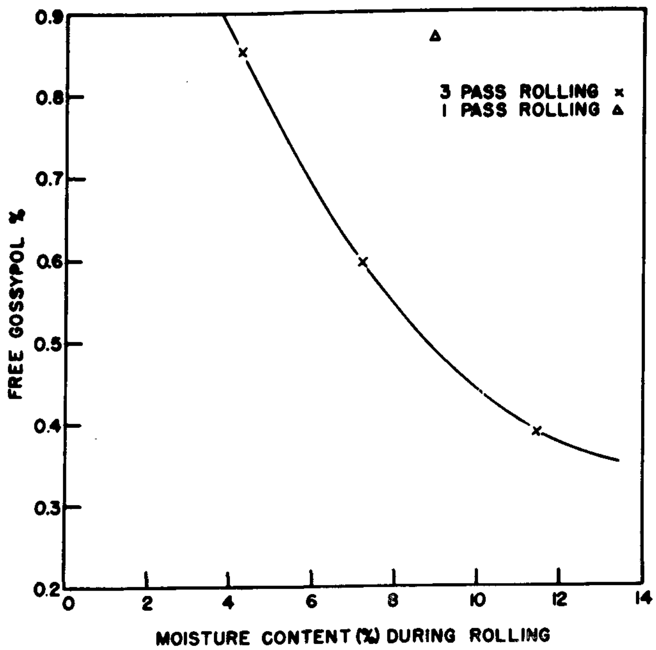

FIG. 1. The effect of moisture content and different rolling conditions upon the free gossypol content of cottonseed meats.

values and are lower in some cases than the initial values (8, 10, and 13%) called for in the plan for the work. These differences are due to losses in moisture in handling. The greatest reduction in free gossypol, from 1.08 to 0.386%, was shown at 13% initial moisture content, and the smallest reduction, from 1.08 to 0.854%, was shown at the 4% initial moisture content. These materials had been passed through the rolls three times.

For comparison with the multiple rolling one test was made at 8% initial moisture content, using only one pass through the rolls. This material showed a reduction in free-gossypol from 1.08 to 0.863% (Figure 1).

All of these flaked meats were subsequently treated with alkali and mixed for extended periods of time. The results are further discussed in connection with the effects of temperature, time, and moisture content during mixing.

Temperature. To study the effect of temperatures of rolling, meats equilibrated to 13% moisture content were heated in a closed rotating drum to 90, 120, or 150°F. as required by the plan shown in Table III. From 30 to 120 min. were required to reach the operating temperatures. The rolls were heated to the temperature of rolling during the time that the meats were being brought up to temperature. The meats were fed to the rolls at the rate of 5 lbs. per minute and flaked to 0.007 in. in average thickness.

TABLE III

Experimental Plan to Investigate the Effects of Temperature of Rolling and Temperature of Mixing Upon the Characteristics of Cottonseed Meals

Temperature of rolling °F.	Temperature of mixing, °F.			Cooking 45 min. 150°F. maximum
	90	120	150	
90	X			X
120	X[a]	X		X
150	X		X	X

[a]This experiment duplicated after adding part of the alkali before rolling.

TABLE IV

Effects of Moisture Content During Grinding Upon the Characteristics of Cottonseed Meals

Moisture content of flakes during grinding	Number of passes through grinder	Nitrogen solubility	Gossypol [a]	
			Free	Total
%		%	%	%
Unground flakes	0	96.0	0.930	1.12
12	1	95.5	0.702	0.91
12	2	95.5	0.591	0.86
18	1	80.3	0.090	0.89
24	1	86.5	0.077	0.80
31	1	91.6	0.071	0.84
31	2	82.6	0.057	......
31	3	82.6	0.056	......
31	4	80.0	0.053	......
31	5	80.0	0.049	......
31	6	82.2	0.047	0.74

[a] Calculated to a 41% protein basis.

TABLE V

The Effects of Temperature of Flaking, Mixing, and Cooking Upon the Characteristics of Cottonseed Meals

Flaking temperature	Mixing[a] temperature	Cooking[b] temperature	Sample description	Gossypol[c]		Nitrogen solubility
				Free	Total	
°F.	°F.	°F.		%	%	%
90	....	....	Flaked	0.674	0.89	93.3
90	90	....	Mixed	0.056	1.02	95.6
90	90	150	Cooked	0.032	0.96	68.7
120	....	....	Flaked	0.535	0.76	87.9
120	90	....	Mixed	0.058	0.97	89.6
120	90	150	Cooked	0.033	0.97	70.1
120	....	....	Flaked	0.493	0.78	85.0
120	120	....	Mixed	0.049	0.90	78.7
150	....	....	Flaked	0.524	0.87	86.5
150	90	....	Mixed	0.071	0.93	91.1
150	90	150	Cooked	0.032	0.93	68.0
150	....	....	Flaked	0.454	0.73	85.0
150	150	....	Mixed	0.035	0.86	64.7
150	150	150	Cooked	0.027	0.88	63.1
120[d]	....	....	Flaked	0.530	0.83	93.4
120	90	....	Mixed	0.092	1.02	96.3
120	90	150	Cooked	0.035	0.99	73.6

[a] Mixing time 120 min. at 31% H_2O, pH 8.2.
[b] Cooking time 45 min.
[c] Calculated to a 41% protein basis.
[d] Part of alkali added before flaking.

The results, given in Table V, show that an increase in the temperature of rolling caused a slight decrease in the free-gossypol content of the materials produced. It will also be noted that rolling at elevated temperatures was accompanied by some reduction in the nitrogen solubility. However this reduction in solubility and in free-gossypol content cannot be completely attributed to rolling. It is quite possible that some reduction took place during the heating of the meats to the temperature of rolling, which in the case of the 150°F. experiment required 120 min. and is comparable to mild tempering conditions.

The Effects of Temperature, Time, and Moisture Content During Mixing

As shown in Tables II and III, certain materials from the comminution studies were further investigated to ascertain the effects of prolonged mixing in the Loomis mixer. The procedure for this operation consisted of moisture content and pH regulation (simultaneously), followed by mixing for periods of up to 120 min. at temperatures of 90 to 150°F.

The length of mixing time, up to 120 min., in the presence of 24% moisture content at pH 8.2 and 90°F., had no appreciable effect upon nitrogen solubility of flakes (13% H_2O) which had been rolled a number of times, while progressively reducing the free-gossypol content from 1.20 to 0.100% (Figure 2).

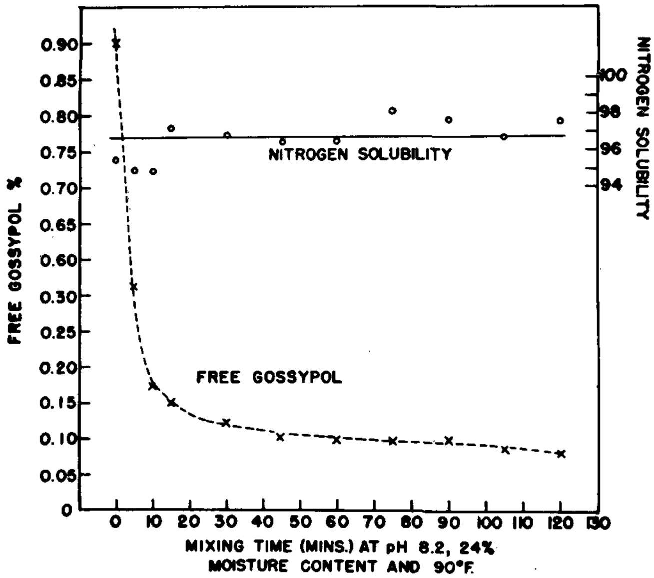

FIG. 2. The effect of mixing time upon free gossypol content and nitrogen solubility of cottonseed meats.

The free-gossypol content of cottonseed meats flaked at 4, 8, 10, and 13% initial moisture content was further reduced by mixing for up to 120 min. at pH 8.2 in the presence of 31% moisture content (Figure 3). This figure also shows that the free-gossypol content of these mixes is in the same relative order at all stages of the mixing operation indicating that the effect of moisture content during rolling cannot be overshadowed by the duration of mixing. The driest meats at rolling gave the highest final free-gossypol content (0.072%) after mixing, and the meats having the highest moisture content when rolled gave the lowest final free-gossypol content after mixing (0.052%). The effect of multiple repassing of the material through the rolls can be overcome by mixing (Figures 1 and 4). The free-gossypol content of the material which had been rolled only once was considerably higher than that

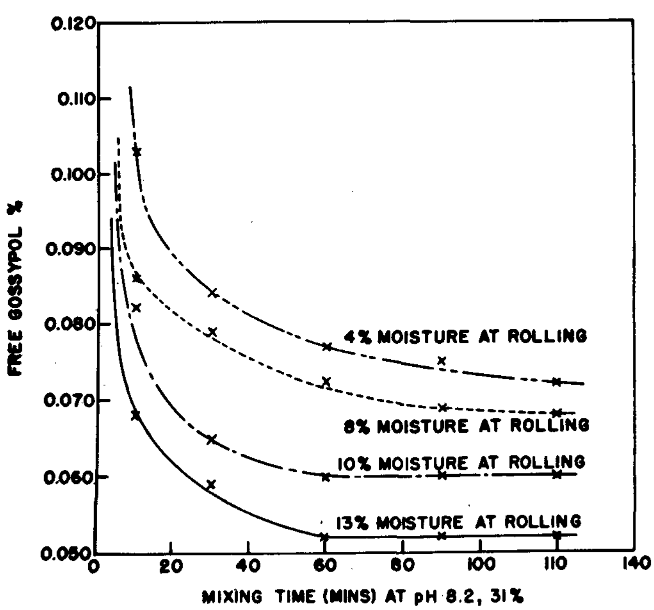

FIG. 3. The effect of mixing time and moisture content during rolling upon the free gossypol content of cottonseed meats.

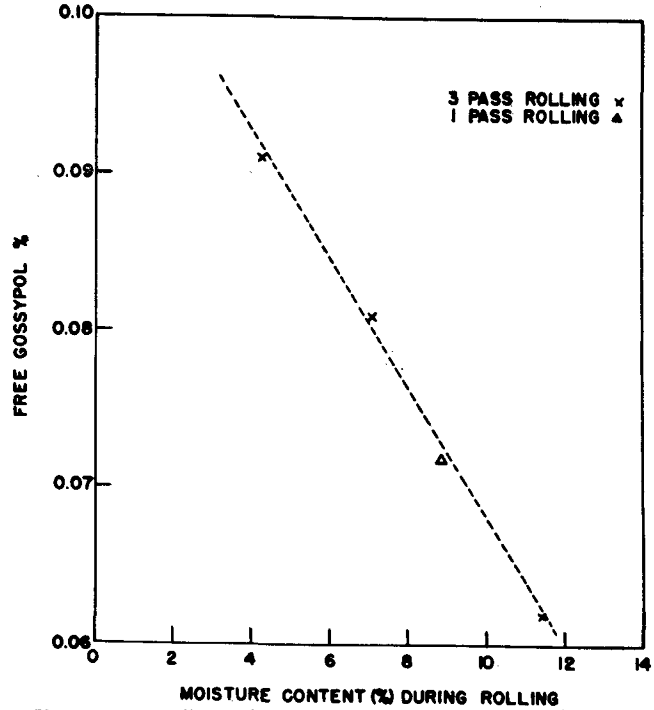

FIG. 4. The effect of moisture content during rolling upon the free gossypol content of cottonseed meats mixed for 120 min. at 90°F., pH 8.2, and 31% moisture content.

which had been rolled three times at comparable moisture content (Figure 1). However, after two hours of mixing at the 31% moisture level and pH 8.2, the free-gossypol content values fell on a smooth curve (Figure 4) independently of whether the materials had been rolled once or three times.

When materials which had been rolled at 4, 8, and 10% initial moisture content were mixed for 120 min. at 31% moisture content and pH 8.2, a reduction in nitrogen solubility of 4 to 7% was noted. There was an additional sharp reduction in nitrogen solubility during the initial 10 to 25 min. of mixing, but when mixing was continued, much of this initial reduction was recovered (Figure 5) and the materials had nitrogen solubilities in excess of 92% at the end of the two hours of mixing. All of these materials had been solvent-extracted, air-desolventized, and moisture-equilibrated without heating, ground, then re-extracted, and air-desolventized before nitrogen-solubility determinations were made.

The data in Table V demonstrate that increasing the temperature of mixing from 90 to 150°F., when carried out at 31% moisture and pH 8.2, resulted in lowered free-gossypol contents in the final meals. At 90°F. mixing temperature there was no significant reduction in nitrogen solubility. At the 150°F. mixing temperature there was a large reduction in nitrogen solubility, down to 65% from the initial of 90%, after two hours of mixing.

Figures 2, 3, and 5 and Table VI show that the moisture content during mixing at 90°F. has a pronounced effect upon the free-gossypol content of cottonseed meals without significantly affecting the nitrogen solubilities of these meals. This effect on the free-gossypol is apparent after only 10 minutes of mixing. At the 31% moisture content level the free-gossypol content was reduced by 90% in that length of time (from 0.80 to 0.08%). The rate of

TABLE VI
Effects of Mixing in the Presence of Alkali at pH 8.2 Upon the Free Gossypol Content and Nitrogen Solubilities of Cottonseed Meals When Carried out at Various Moisture Levels

Moisture content during mixing	Flakes before mixing [a]		Flakes after mixing for 10 min.	
	Free gossypol[b]	Nitrogen solubility	Free gossypol[b]	Nitrogen solubility
%	%	%	%	%
18	0.77	93.5	0.364	90.4
24	0.76	95.6	0.174	94.9
31	0.76	96.0	0.084	93.2

[a] Flaked at 8% moisture content to 0.007" thickness.
[b] Calculated to a 41% protein basis.

reduction in free-gossypol content diminishes with time, and after 60 min. of mixing has almost leveled off.

The Effects of Moisture and Temperature During Cooking

Selected materials from the grinding experiments (Table I) were cooked at a maximum temperature of 180°F. in the Loomis mixer to determine the effects of cooking upon the nitrogen solubilities of the final meals and to determine the amount of free-gossypol reduction which was accomplished by the cooking operation. For this purpose ground, flaked meats were placed in the mixer at 18, 24, and 31% moisture content levels and pH 8.2, and the temperature was brought to 180°F. within 8 to 10 min. This temperature was maintained for the balance of the cooking time (a total of 45 min. from the start of heating). For the final 15 min. of the cooking period a stream of air was passed through the cooker to dehydrate the meats to a moisture content suitable for oil extraction. The properties of the meals derived from these cooking experiments are given in Table VII. A significant reduction in nitrogen solubility resulted when cooking was carried out at 180°F. in the presence of 18 to 31% moisture content. This reduction was apparent in samples taken as early as 20 min. after heating was commenced. There are indications that the reduction had taken place even earlier. There was no further significant reduction in the nitrogen solubility after the 20-minute cooking sample was taken. The data also show that the free-gossypol content of these materials is inversely related to the moisture content during mixing. The lowest free-gossypol content achieved was 0.025% at the 31% maximum moisture content during cooking. This material had a nitrogen solubility of 64%.

TABLE VII
Effects of Cooking After Grinding Upon the Characteristics of Cottonseed Meals

Moisture content of flakes during grinding	Passes through grinder	Sample description	Moisture when sampled	Nitrogen solubility	Gossypol[a]	
					Free	Total
%			%	%	%	%
18	1	Feed to cooker (after grinding)	18	80.3	0.091	0.86
18	1	After cooking (45 min.)	12	71.8	0.055	0.87
18	1	Screened and cooled	10	70.4	0.055	0.88
24	1	Feed to cooker (after grinding)	24	86.5	0.077	0.80
24	1	After 30 min. cooking	22	71.5	0.050	0.91
24	1	After 45 min. cooking	18	67.9	0.044	0.91
24	1	Screened and cooled	16	68.6	0.045	0.92
31	6	Feed to cooker (after grinding)	31	82.2	0.047	0.74
31	6	After 20 min. cooking	22	65.0	0.029	0.75
31	6	After 40 min. cooking	21	63.6	0.029	0.75
31	6	After 45 min. cooking	16	63.9	0.027	0.76
31	6	Screened and cooled	15	64.0	0.025	0.75

[a] Calculated to a 41% protein basis.

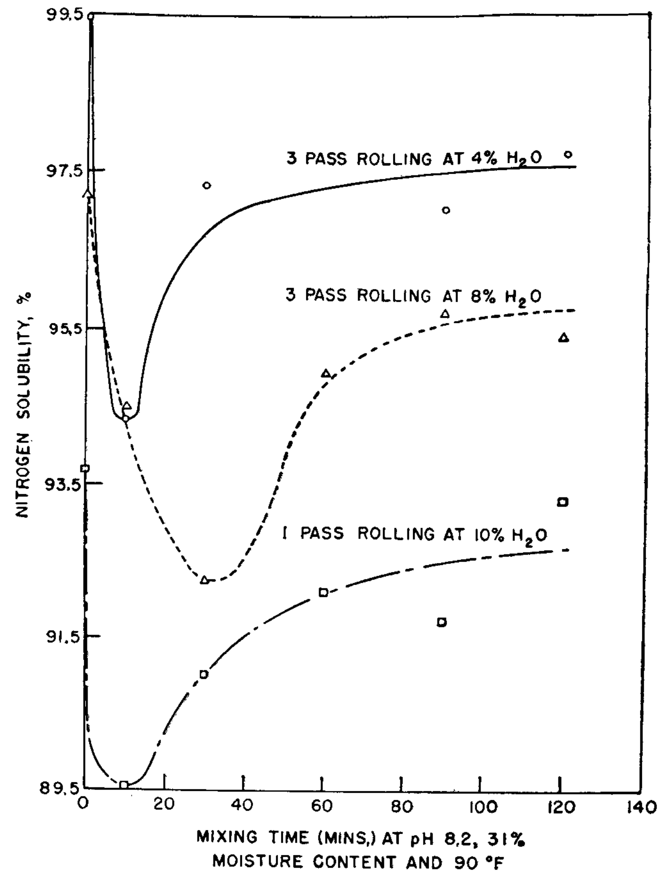

FIG. 5. Variation of nitrogen solubility of cottonseed meats with mixing time for different rolling conditions.

The materials from the studies on rolling and mixing at various temperatures were evaluated by cooking at 150°F. (Table III). After the completion of 120 min. of mixing the temperature of the meats was brought to 150°F. over a 5- to 8-minute period and maintained at that temperature for the balance of the 45-minute cooking period. A stream of air was passed continuously through the cooker during this time to effect dehydration to 8–10% for subsequent solvent extraction. All of the meals produced in this series of experiments had free-gossypol contents between 0.027 and 0.035% (Table V). The nitrogen solubilities of these materials were considered low. They varied from 63 to 70% after cooking, indicating that considerable reduction had taken place at this low (150°F.) temperature. The apparently low, total gossypol content values shown for flaked meats in Table V is explained by the fact that these materials were solvent-extracted immediately; consequently some of the gossypol was removed with the oil before it could be bound to the meal.

Discussion and Summary

Pilot-plant experiments were conducted to determine the effects of several methods of comminution of raw cottonseed meats, with and without alkali, upon the properties of the resulting meals. The comminution methods studied included grinding at moisture contents from 12 to 31%, rolling and re-rolling at various moisture levels from 4 to 13%, and rolling at temperatures from 90 to 150°F.

Grinding of cottonseed flakes, at 18 to 31% moisture content and at pH 8.2, through a peanut butter mill reduces the free-gossypol content to between 0.047 and 0.090%, depending upon the moisture content and the number of times the material is passed through the mill. Concurrently the nitrogen solubilities of these materials were reduced from approximately 96% to approximately 80%.

The moisture content of cottonseed meats during rolling significantly affects the free-gossypol content of the meals. The free-gossypol contents of the flaked meats are inversely related to the moisture content during rolling. The free-gossypol contents of these flaked meats are in the same relative order during all stages of processing, indicating that moisture content during rolling is critical and its influence cannot be overcome during subsequent processing steps.

The effects of multiple re-rolling to reduce free-gossypol content can apparently be overcome by subsequent processing. The values for materials which had been rolled only once fell on the same smooth curve as those for materials which had been rolled three times, after all of these materials had been mixed for two hours at 31% moisture and pH 8.2 and 90°F.

The nitrogen solubility of cottonseed meals is slightly reduced by rolling at moisture contents in excess of 10%.

There is an indication that the nitrogen solubility of flaked cottonseed meats decreased during the initial phase of mixing at pH 8.2 and 31% moisture and then increases to approximately its original value.

Mixing in the presence of alkali at moisture contents of 24 to 31% and at a temperature of 90°F. gave a significant reduction in the free gossypol content of cottonseed meals without a corresponding decrease in nitrogen solubility.

Increased temperatures of flaking, up to 150° F., are accompanied by a reduction in the free-gossypol content of the flakes produced. At the higher temperatures there is some reduction in nitrogen solubility (to approximately 85%).

Prolonged mixing (for 120 min.) in the presence of 31% moisture and at pH 8.2 and 120 or 150°F. significantly reduced the nitrogen solubilities of cottonseed meals when compared to mixing at 90°F.

Cooking at temperatures as low as 150°F. at 31% moisture content and pH 8.2 significantly reduced the nitrogen solubilities of cottonseed meals (to approximately 70%). Most of these meals had free-gossypol contents of less than 0.04%.

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Some Properties of the Lipase Present In Germinating Rapeseed[1]

L. R. WETTER, Prairie Regional Laboratory, National Research Council of Canada, Saskatoon, Saskatchewan, Canada

A PRELIMINARY SURVEY has indicated that the various oilseeds grown in Western Canada have extremely low contents of lipase. According to the literature, the lipase activity of some oilseeds increase during germination. Ramakrishnan (13), for example, reported a qualitative increase in the lipase content of the groundnut when it was germinated. Gershtein (3) has reported that sprouting tung seed increases five-fold in lipase activity after 40 days and then there is a gradual decline; Johnston and Sell (7) found that the activity increased eight and a half times after 32 days. On the other hand, oats produce a lipase that reaches maximum activity after 8 hrs. of germination and then drops off rapidly (6). Germinating cottonseed produced a marked increase in lipase along with a decrease in total lipids (12).

One of the oilseeds grown in Western Canada, rapeseed, possesses very little lipase activity in the dormant seed. In common with the behavior of other oilseeds already cited however it exhibits a marked increase in lipase activity on germination. This paper deals with quantitative aspects of this increase and also discusses some of the properties of the enzyme.

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Materials and Methods

Materials. Polish rapeseed (*Brassica campestris* L.) was used throughout in this investigation unless otherwise designated. The seeds were germinated either in the dark or in the light at approximately 25°C. on white Ottawa sand in large Pyrex dishes (10 by 15 in.). A glass plate cover was employed to maintain the humidity near saturation. During germination no nutrients were added, and the moisture content was maintained in the dish with distilled water.

After the desired period of growth, distilled water was used to wash the seedlings free of sand. Then