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Selective Fixation of Deleterious Phosphatidic and Pigment Materials in Commercial Processing to Improve Quality of Cottonseed Oil and Meal[1]

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IN A PREVIOUS REPORT (4) on a commercially operating, integrated cottonseed oil mill and salad oil plant a process was described for producing a quality, dust-free, solvent meal and a salad oil with excellent keeping quality, flavor, and chill test (5). Since these data were presented, additional work has been done on evaluating the specific effect each step of processing has on the meal and on the oil. These data comprise the subject matter for the present report.

As a basis for comparing quality of meal and oil in various stages of processing the following criteria were arbitrarily set up:

Meal

1. Free gossypol (1)
2. Nitrogen solubility in 0.02N NaOH (10)
3. Available gossypol units—A.G.U. (7).

In addition to the analyses enumerated above, all meal samples were analyzed for moisture, oil, crude protein, and pH. The latter measurement was determined by stirring 20 g. of meal for 10 seconds in a Waring Blendor with 200 ml. of distilled water. pH was read on slurry with a Beckman pH meter.

The criteria for evaluating the quality of the crude oil through the various stages of processing and also for determining the effect of exposure to sunlight and air on crude cottonseed oil were:

Oil

1. Moisture and volatile matter
2. Free fatty acids
3. Lovibond color of 1-in. column of crude oil
4. Chromatographic loss.

Analysis of the source material and processing conditions under which the meal and oil described in this report were produced follows:

Seed Variety: Acala 4-42
Seed Analysis

	%		%
Moisture	8.2	F.F.A.	0.5
Oil	19.8	Total lint	12.2
Ammonia	4.16	Grade	110.0
Free Gossypol	0.97 (moisture- and oil-free basis).		

The cottonseed meats with added hulls to control the protein to 41% were flaked in five-high rolls to 0.016 in. and cooked in five-high stack cookers. Moisture content of cooked meats at the cooker discharge was 12.6%, temperature of meats at cooker discharge, 208°F.(98°C.). Granular soda ash was added to the cooked meats at the rate of 0.4% of the weight of the cooked meats prior to prepressing.

The prepressed flake averaged 13.5% residual oil, and the temperature of prepressed flake at screw-press discharge was 207°F.(97°C.).

The highest temperature to which the crude oil was exposed was 210°F.(99°C.) in the cooking and prepress stages, the highest temperature to which the meal was exposed, 235°F.(113°C.) at the final, meal-dryer discharge.

The data in Table I show the effect each stage of processing has on the meal. Commercially processed, cottonseed meat-hull mixtures normally have an oil content of approximately 30%. Cooked meats before and after addition of soda ash and prepressed flakes were extracted in the laboratory with n hexane to obtain the meals shown in the first three columns in Table I. The meals from the extractor, tube dryer, and dryer and toaster were used as they came from the plant except that the extractor meal was air-dried to remove the hexane.

Table I shows a normal and reasonable reduction of gossypol in cooking, in the prepressing step after the addition of solid soda ash, and in the solvent-extraction and meal-drying stages (11). This is especially noteworthy because miscella-refined soapstock is added to the second of five tube-dryers in series. This soapstock has a slight excess of caustic soda and supposedly contains considerable amounts of gossypol (2) which has been removed from the oil by the alkali-refining processes (6). Yet the data show a normal decrease in free gossypol and also a decrease in pH in spite of the addition of the alkaline soapstock from miscella refining. This decrease in pH is attributed to the presence of residual soda ash from the initial treatment of the meats subsequent to cooking.

A.G.U., or Available Gossypol Units, is a combined biological and chemical assay of the gossypol present in cottonseed meal, which will combine with cephalin in egg yolks to form a cephalin-gossypol complex that is related to egg-yolk discoloration (7).

Dr. Grau and co-workers at the University of California are responsible for developing this new tool for evaluating a cottonseed meal with respect to pos-

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TABLE I

Comparative Analysis of Meats and Meal Through Various Stages of Plant Processing

	Extracted in laboratory with normal hexane			Meal from extractor	Meal from tube dryer after addition of soapstock	Commercial meal from dryer and toaster
	Cooked meats	Cooked meats + soda ash	Prepressed flakes			
Moisture	8.9%	7.5%	9.3%	9.3%	8.5%	7.3%
Oil	1.0%	1.0%	1.7%	0.6%	1.2%	0.9%
Protein	38.98%	39.41%	40.13%	38.92%	42.09%	40.50%
Free gossypol	0.225%	0.229%	0.077%	0.064%	0.058%	0.044%
Nitrogen solubility[a]	65.2%	61.6%	76.2%	80.9%	67.7%	78.9%
pH[b]	6.7	7.1	6.9	6.9	6.6	6.6
A.G.U.[c]	....	....	....	0.58[d]	0.46[d]	0.23[d]

[a] Nitrogen solubility calculated by method of Chang, Lyman, and Couch, Texas A&M.
[b] 20 g. meal stirred 10 sec. in Waring Blendor with 200 ml. water. Read pH on slurry.
[c] A.G.U. available gossypol units by method of Grau, Univ. California.
[d] Tentative data subject to confirmation by independent laboratory.

sible egg-yolk discoloration. This method of analysis is so sensitive that 1% or less or an expeller type of meal can easily be detected in a laying-hen ration by analysis of the egg yolks. This combined biological and chemical analysis is fully described in the reference (7).

AGU Calculation:

$$\text{AGU} = \frac{\text{Ab400–Ab445 diet egg} - \text{Ab400–Ab445 basal egg}}{\%\ \text{CSM in diet}} \times 100$$

Using data and analyses obtained over a three-year period, Dr. Grau and co-workers state that a cottonseed meal with A.G.U. of 0.25 or less (8) can be safely fed to laying hens in amounts up to 10% of the total weight of the ration without egg-yolk discoloration.

The last three columns of Table I show that, in lowering the free gossypol 0.006% between the extractor and the tube dryer, the A.G.U. were lowered by 0.12. In lowering the free gossypol 0.014% between the tube dryers and the dryer and toaster, the A.G.U. were decreased by 0.23.[2] No claim for any correlation between free gossypol and A.G.U. is indicated or implied by the relationship shown in these particular meals. Other meals tested show a lower A.G.U. with 0.04% free gossypol than was obtained with a meal analyzing 0.035% free gossypol.

Over 200 A.G.U. determinations have been made in our laboratory on eight different lots of meal produced commercially, as described earlier in this paper (4). Three of these meals have been tested by independent laboratories or universities; others are presently being tested. A.G.U. values on these commercial meals have all been in the range of 0.16 to 0.33, and a very close correlation exists between different collaborators on the same meal. Commercial feeding trials on 300-bird lots of laying hens are planned at three different locations to evaluate further this type of cottonseed meal and the analytical method. To date none of this meal has been sold in commercial channels for laying-hen rations, nor is any sale contemplated for this purpose until additional data are obtained.

In addition to the A.G.U. work, eight-week feeding trials were completed March 27, 1957 with 128 Vantress Cornish New Hampshire Crosses, which were separated into cages of 16 birds each. The pullets were kept in cages separate from the cockerels. Soybean meal, screw-press-type of cottonseed meal and prepressed solvent-meal produced by the method (4) described earlier in this paper were obtained from mixed feed manufacturers by an independent chemical laboratory, where the feeding trials were conducted. The birds were fed under the direction and supervision of Hobart Halloran, a nutritionist. The basal diet containing soybean meal was an exact duplication of the A.N.R.C. (Animal Nutritional Research Council) basal or equivalent. The results of this feeding trial showed a significant increase in feed efficiency of the soda ash soapstock-treated, prepressed solvent-meal over the expeller type of meal.

The feed efficiency of this prepressed, solvent-meal ration was as high as or fractionally higher than the basal soybean diet. Feeding trials of the same number, breed, and diets were duplicated. The results substantiated the original work.

From data accumulated to date it would appear that, by careful supervision and control of the various processing steps, commercial meals of quality at least comparable to that described above can be consistently produced (9).

The crude oils from the laboratory-extracted meats and prepressed flakes were recovered from the solvent for the analysis shown in Table II by removing the n hexane under vacuum in the dark at temperatures below 150°F.(65°C.). Some difficulty was experienced in completely removing the solvent from the oil even at temperatures of 150°F.(65°C.) under 0.3-mm. Hg vacuum. The incomplete removal of n hexane from samples 1, 7, and 16 in Table II may have had a significant adverse effect on the chromatographic losses on these samples under the various conditions of exposure in the test.

Normally an increase in F.F.A. of 0.1% to 0.5% in the oil from the seed during cooking and processing is expected. This may be caused by enzymic breakdown of the triglycerides as a result of processing temperature and pressure or by unknown causes. As F.F.A. increase represents a loss during the refining step of some factor multiplied by the actual increase in F.F.A. (12, 3), it is apparent that controlling the F.F.A. increase during processing is important in obtaining the greatest possible yield of refined oil per ton of source material. The addition of granular soda ash to the cooked meats not only neutralizes some of the F.F.A. naturally present but retards F.F.A. increase during processing. This conclusion is based on three years of actual plant operation data, comparing daily seed analysis, plant refining losses, and actual weighed yields of refined oil.

The chromatographic losses shown in Table II are a bit erratic, possibly because of the reasons previously mentioned, but certainly exhibit a consistent trend of increasing with increasing exposure to sunlight and air. The color data on the crude oil follow the same pattern in general as the chromatographic losses with respect to exposure to sunlight and air. We have found this to be more pronounced and more

[2] Tentative data subject to confirmation.

TABLE II

Comparative Analysis of Oil Through Various Stages of Plant Processing

	Moisture and volatile	F.F.A.	Loss data chromatographic	Lovibond color 1-in. column crude oil
	%	%	%	
1. Laboratory-extracted oil from cooked meats	0.69	0.53	2.88	35/11.3
2. Same as No. 1 plus 15 min. sunlight			3.60	35/11.3
3. Same as No. 1 plus sun and air			5.08	35/13.6
4. Same as No. 1 plus granular soda ash	0.09	0.43	2.19	35/10.6
5. Same as No. 4 plus 15 min. sunlight			3.10	35/11.2
6. Same as No. 4 plus sun and air			2.48	35/13.6
7. Laboratory-extracted oil from prepressed flake	0.80	0.51	3.11	35/13.7
8. Same as No. 7 plus 15 min. sunlight		0.50	5.14	35/14.1
9. Same as No. 7 plus sun and air		0.56	4.06	35/15.7
10. Plant-prepressed oil low-pressure	0.10	0.70	1.33	35/13.3
11. Same as No. 10 plus 15 min. sunlight		0.67	3.78	35/15.1
12. Same as No. 10 plus sun and air		0.61	4.25	35/18.5
13. Plant-prepressed oil high pressure	0.06	0.64	1.34	35/15.7
14. Same as No. 13 plus 15 min. sunlight		0.59	4.24	35/22.2
15. Same as No. 13 plus sun and air		0.50	2.49	35/26.0
16. Plant solvent-extracted oil	0.12	0.53	2.97	35/11.9
17. Same as No. 16 plus 15 min. sunlight		0.62	4.63	35/14.9
18. Same as No. 16 plus sun and air		0.50	6.36	35/16.6

apparent on oils produced from seed with over 1.0% F.F.A. than is indicated on the 0.5% F.F.A. seed processed during this test period.

A more complete appraisal of cup loss and color data correlated with the rest of the oil analysis shown in Table II is contemplated in the future. We propose to continue this work in our plant along comparably comprehensive lines. Periodic progress reports will be made to the American Oil Chemists' Society when justified by plant and laboratory data obtained.

Summary

Plant operating-procedures and laboratory controls were set up to evaluate the quality of meal and oil which could be produced through each stage of commercial, prepress-solvent-extraction processing. By altering conventional, prepress-solvent-processing conditions and by increasing moisture during cooking and adding granular soda ash after cooking meats, cottonseed meal rations can be produced which are comparable in feed efficiency to soybean meal rations and satisfactory for feeding laying hens in amounts up to 10% of the total weight of the ration with no egg-yolk discoloration and crude cottonseed oils with low F.F.A. and light color can be produced which refined to low Lovibond colors and with refining losses approximating the chromatographic loss when miscella refined within minutes after separation from the source material with the exclusion of air and light.

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Composition of Acidulated Cottonseed Soapstocks as Influenced by Commercial Methods of Processing Seed and Oil[1]

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SOAPSTOCKS containing approximately 100 million pounds of anhydrous fatty material are produced as a by-product in refining the annual domestic production of cottonseed oil. The major outlets for this material are as a source of fatty acids and pitch. In recent years increasing amounts of soapstocks have been used as a plasticizer in pelleting oilseed meals and as a source of fat in mixed feeds.

No systematic study of the composition of acidulated soapstocks as related to processing conditions and refining methods has been reported. In fact, relatively little information on their composition is available (3, 8, 9, 13, 20). The trend toward replacement of hydraulic pressing methods with screw-pressing,

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[2] One of the laboratories of the Southern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture.