

rated acid fraction in order to determine the quantity of this acid it contained, owing to the solubility of lead myrstate in cold ether. Fifty-nine grams of the ethyl esters of the unsaturated acid fraction were prepared and distilled under one millimeter pressure. As was expected, all of the myristic acid was found in the first fraction collected. In this connection, it should be mentioned that no measurable quantities of other saturated acids of higher molecular weight were found in these distilled ester fractions.

The final results which were calculated from the various analytical data obtained are given in Table III.

Table III — Saturated Acids

Acids	Per Cent	Per Cent In Oil
Myristic	6.85	1.53
Palmitic	16.03	3.59
Stearic	48.42	10.84
Behenic	28.10	6.29
Lignoceric	0.60	0.13
	100.00	23.38

It may be of some interest to mention that the behenic acid finally obtained by repeated recrystallization from ethyl alcohol melted at 79.5° C. Also, in so far as could be determined, all of the lignoceric acid which was found, was in the small residue of undistilled ethyl esters.

The composition of the oil in terms of glycerides is given in Table IV.

Table IV — Glycerides of	
Acids	Per Cent in Oil
Myristic	1.6
Palmitic	3.8
Stearic	11.3
Behenic	6.5
Lignoceric	0.14
Oleic	71.1
Linoleic	3.9

SUMMARY

A brief description of the tree *Moringa oleifera*, flowers and fruits has been given. The oil expressed from seed produced in Haiti, the subject of this investigation, gave the following characteristics:

Refractive index at 25° C. 1.4671
Iodine number (Hanus) 68
Saponification value 186.4
Acid value 0.74
Unsaponifiable matter 1.5 per cent.

The results indicated that the oil contained 68.9% oleic acid, 3.8% of linoleic acid, 1.5% myristic acid, 3.6 % palmitic acid, 10.8% stearic acid, 6.3% behenic acid, and 0.13% lignoceric acid.

An Hypothesis Concerning The Role of The Enzymes in the Relative Value of Cottonseed

By G. S. MELOY

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THOSE of you who analyzed cottonseed grown in the Mississippi Valley during the season of 1937-38, as well as those of you who came into contact with the refining of the oil produced in that section of the Cotton Belt, will not have to be told that the crop of cottonseed produced in that section that season was probably without precedent not only because of the intensity of deterioration that had taken place but also because of the extensive territory in which deterioration appeared in the seed. In Mississippi, for instance, 46 percent of the shipments during October had excesses of free fatty acids; during November, 76 percent; during December, 85 percent; January, 91 percent; and during February, 94 percent of the shipments contained excesses of f. f. a. The maximum and average f. f. a. con-

tents during these months were 21.5 percent and 3.29 percent respectively, in October; 17.5 percent and 4.42 percent in November; 20.5 percent and 5.29 percent in December; 35.0 percent and 8.30 percent in January; and in February, 2449 shipments with a maximum of 38.0 percent and an average f. f. a. content of 12.40 percent, were received at the oil mills.

While we have no facilities for a direct study of the question of the generation of f. f. a., it occurred to me that something might be discovered by an analysis of the seed analysis records.

For the purpose of studying the question of the generation of f. f. a. I first examined the analyses of Mississippi Delta seed that were graded during December 1936. Among these there were 386 analyses in which the moisture content

ranged from 12.2 to 19.7 and averaged 13.08; but in all of the 386 analyses not a single report showed more than 1.8 percent f. f. a.

Then thinking that possibly the f. f. a. might be related to the degree of maturity or development of the seed as indicated by the oil content, I took some 23,671 grade reports for Mississippi-grown seed which were analyzed during October, November, and December, 1937, and January and February, 1938, and broke them down into groups of the various oil contents; but found that whether the seed had matured only 14 percent of oil or 22 percent, the reversed enzymic activity seemed to be equally as active, for just as high f. f. a. was found whether the seed contained little or much oil. The tabulations for January and February would

TABLE 1.—COMPOSITION AND GRADE OF COTTONSEED SOLD IN MISSISSIPPI, DURING MONTHS OF OCTOBER—NOVEMBER—DECEMBER 1937, AND JANUARY—FEBRUARY 1938, ARRANGED BY AVERAGE OIL CONTENTS.

MONTHS	NUMBER OF SAMPLES	OIL AVERAGE CONTENT	AMMONIA AVERAGE CONTENT	QUANTITY INDEX AVERAGE	LOW QUALITY BY CAUSES									BELOW GRADE	GRADE			
					NUMBER OF SAMPLES LOW QUALITY	FREE FATTY ACIDS			MOISTURE			FOREIGN MATTER			MIN.	MAX.	AVER.	
						NUMBER OF SAMPLES	MAX.	AVERAGE FREE FATTY ACIDS	NUMBER OF SAMPLES	MAX.	AVERAGE MOIS. TURE	NUMBER OF SAMPLES	MAX.					AVERAGE FOREIGN MATTER
OCTOBER	1 6 408 895 2,147 2,991 2,000 255 5	14.90 15.65 16.65 17.59 18.48 19.49 20.33 21.25 22.08	3.28 3.65 3.87 3.85 3.79 3.68 3.68 3.56 3.59	82.18 88.15 94.47 98.46 101.66 105.04 108.04 111.36 114.46	1 6 96 624 1,247 1,741 2,026 128 3	1 4 89 576 845 1,292 938 122 3	12.5 7.0 10.0 21.5 12.0 10.0 9.0 7.0 3.0	12.50 5.15 4.56 4.19 3.23 3.15 2.95 2.65 2.57	1 5 60 328 670 1,019 296 25 —	23.0 22.5 16.0 19.6 18.8 17.0 14.8 14.0 10.9	23.0 17.0 12.9 12.9 12.9 12.7 12.5 12.6 —	1 2 9 70 55 36 12 1 —	3.5 12.9 6.9 8.4 8.6 8.9 6.0 4.0 0.8	3.5 12.8 4.5 4.8 4.1 4.1 4.0 4.0 —	— — — — — — — — —	31.5 40.0 52.5 49.5 60.0 63.0 30.0 107.0	31.5 86.0 98.0 102.0 106.0 109.0 112.5 116.0	31.5 71.9 82.9 90.4 98.3 104.7 104.9 112.3
NOVEMBER	3 65 514 1,217 1,830 1,608 181 4	15.73 16.63 17.56 18.48 19.51 20.34 21.23 22.35	3.79 3.92 3.50 3.83 3.72 3.67 3.60 3.45	89.39 94.67 98.64 101.90 105.36 108.38 111.52 115.10	3 60 445 809 1,442 1,405 159 3	3 58 409 747 1,352 1,392 159 3	8.5 12.0 17.5 16.5 15.5 12.0 9.5 4.4	6.47 5.24 4.87 4.00 4.56 4.33 4.38 3.03	2 35 176 323 419 143 4 —	14.5 22.4 25.5 20.6 18.5 19.7 15.1 12.7	14.0 13.1 13.0 12.8 12.7 12.6 12.8 —	1 8 61 72 89 62 6 —	3.4 19.8 9.3 14.9 13.1 7.3 5.4 1.5	3.4 6.0 4.5 4.4 4.2 3.9 3.8 —	— — — — — — — —	58.0 44.5 40.5 27.5 32.0 53.0 68.5 101.5	84.0 101.0 101.0 105.5 108.5 112.5 113.5 114.5	67.2 78.5 86.6 94.7 94.4 96.4 98.8 109.7
DECEMBER	9 81 545 1,235 1,726 611 64	15.62 16.65 17.57 18.47 19.43 20.27 21.18	3.87 3.87 3.83 3.77 3.62 3.63 3.61	89.32 94.47 98.26 101.50 104.74 107.86 111.38	9 73 488 1,115 1,647 598 59	7 65 398 944 1,573 594 59	16.0 18.0 16.5 20.5 19.5 16.0 10.5	2.40 5.60 4.52 5.19 6.22 5.85 5.34	8 39 244 636 1,078 216 6	14.3 17.5 20.3 20.0 19.0 15.1 13.7	13.3 13.6 13.5 13.4 13.1 12.7 12.7	4 17 45 127 321 86 4	20.9 9.4 14.6 15.0 27.5 10.3 3.7	2.6 4.6 4.6 4.7 4.8 4.4 3.5	1 1 1 7 6 — —	B.G. B.G. B.G. B.G. B.G. B.G. B.G.	89.0 96.5 101.0 105.0 107.5 110.0 114.0	67.8 80.3 87.9 87.9 86.5 82.9 92.4
JANUARY	1 5 73 547 1,021 1,030 259 23	14.80 15.72 16.66 17.55 18.47 19.40 20.29 21.18	3.99 3.78 3.80 3.82 3.71 3.65 3.61 3.61	85.94 89.28 94.10 98.12 101.14 104.50 107.82 111.38	1 5 68 515 968 999 250 23	1 5 62 477 894 980 283 23	10.5 11.0 28.0 35.0 27.5 28.0 22.0 17.5	10.50 9.10 7.44 5.97 7.84 9.58 9.67 7.94	1 4 19 295 612 775 169 10	14.7 19.2 20.2 21.7 23.4 17.6 15.7 14.3	14.7 16.1 14.3 13.8 18.8 13.4 13.1 13.0	— — — — — — — —	1.7 4.3 5.4 4.8 6.1 5.9 5.6 9.4	— — — — — — — —	46.0 42.5 B.G. B.G. B.G. B.G. B.G. B.G.	46.0 63.5 97.5 100.5 105.5 107.5 110.5 108.5	46.0 53.6 75.2 83.6 81.2 71.0 70.1 77.6	
FEBRUARY	3 53 369 879 998 288 10	15.63 16.66 17.53 18.50 19.38 20.29 21.19	3.80 3.92 3.84 3.71 3.64 3.58 3.62	88.95 94.82 98.16 101.26 104.36 107.64 111.48	3 51 360 845 959 280 10	3 50 350 813 946 277 10	12.5 36.0 38.0 37.0 33.0 38.0 22.5	8.00 8.75 8.11 12.25 14.07 13.30 11.32	1 28 155 165 733 150 4	15.7 18.0 19.2 20.1 17.1 15.3 12.6	15.7 13.4 13.7 13.7 13.2 12.9 12.4	1 18 34 373 547 144 4	22.0 13.5 19.8 45.8 28.6 23.1 5.2	22.0 6.1 6.5 7.3 6.5 6.1 4.2	1 4 44 287 348 98 3	B.G. B.G. B.G. B.G. B.G. B.G. B.G.	70.0 95.0 103.5 104.5 108.0 110.0 108.0	69.8 70.2 79.3 78.9 64.2 67.8 79.8
MONTHLY SUMMARIES																		
OCTOBER	8,405	19.24	3.71	104.22	(58.7%) 4,932	(46.0%) 3,870	24.5	3.29	(28.6%) 2,404	23.0	12.78	(2.2%) 186	11.9	4.47	—	31.5	116.0	105.4
NOVEMBER	5,422	19.36	3.74	104.88	(81.4%) 4,416	(76.0%) 4,123	17.5	4.42	(20.3%) 4,102	25.8	12.91	(5.5%) 299	19.8	4.28	4	B.G.	114.5	94.3
DECEMBER	4,285	19.00	3.72	103.37	(93.1%) 3,989	(84.9%) 3,637	20.5	5.29	(46.2%) 2,227	20.3	13.20	(14.1%) 604	27.5	4.72	(0.4%) 16	B.G.	114.0	85.5
JANUARY	2,959	18.75	3.70	102.20	(95.6%) 2,829	(90.7%) 2,685	35.0	8.30	(66.9%) 4,980	23.4	13.60	(23.4%) 842	33.4	5.82	(9.0%) 291	B.G.	110.5	76.9
FEBRUARY	2,600	18.27	3.69	102.62	(96.5%) 2,508	(94.2%) 2,449	38.0	12.40	(65.4%) 1,700	20.1	13.40	(45.0%) 1,171	45.8	6.70	(30.2) 785	B.G.	110.0	72.3

*THE AVERAGES GIVEN ARE OF THE GRADED LOTS. BELOW GRADE LOTS ARE NOT INCLUDED.

almost indicate that the higher the oil content the greater the deterioration (table I).

I then arranged the reports for January in an ascending scale of f. f. a. contents and in columns according to the oil content, and determined the average moisture content for each f. f. a. — oil group. The moisture content of these samples ranged from 9.2 percent to 19.8 percent. The average moisture content for each group is given in table 2. The average oil content of the samples listed in the column under 18.0 percent — 18.9 percent oil is 18.47 percent; and the average moisture, 13.89. In the 19.0 percent group the average oil

content is 19.40 percent, and the average moisture, 13.25 percent; and in the 20.0 percent group the average oil content is 20.29 percent and the average moisture, 12.69 percent. From these figures it is apparent that there is little or no relation between f. f. a. and moisture (table 2). This lack of relation is further shown by the scatter diagram, Figure 1, in which the moisture content is plotted for each oil group used in the study, against the percentages of free fatty acids in the oils.

I am more and more impressed with the idea that the generation of f. f. a. is more directly related to general atmospheric conditions

than to the actual moisture content of the seed. A comparison of the weather reports for the two seasons does not show significant differences in the actual precipitation. The harvest season of 1936-37 was slightly colder and less humid than the harvest season of 1937-38; but the general atmospheric condition of 1937-38 was characterized by high relative humidity. This humid condition probably resulted in a stagnation of the normal transpiration of the seed.

It may be that the enzymes do not become dormant unless or until there has been an initial or partial dehydration of the plant juice. If this is so, then the enzymes doubt-

less are in an active state so long as the plant juice remains unchanged through dehydration.

Now if we suppose that in the growth and development of the seed the usual order of enzymic activity is in three stages, first, the constructive stage in which the enzymes are bathed in plant juices, which are made up of substances dissolved in water and of a definite consistency, in which habitat the enzymes have the ability to convert carbohydrates into either oil or protein through a series of divergent steps. But if at any time the density of these juices is increased through dehydration, the enzymes lose their constructive power and become dormant. During severe droughts we see this effect in the arresting of the elaboration of oil and protein. During normal seasons we see the same result when the bolls burst open and the seeds dry out. The second stage is the dormant stage. The third stage is the resuscitation of the enzymes through the reabsorption of moisture; and in this stage the enzymes are no longer constructive agents, but are now active as destructive agents, breaking down the oil and protein, through a reversal of the stages of construction, back to carbohydrates or to even less complex substances.

It is evident that dehydration of the plant juice is necessary to dormancy of the enzymes. But it is also evident that dormancy is not necessary to a reversal of their activity.

For instance, we now know that at the elapse of approximately 45 days after flowering, cotton bolls have fully developed, and that they normally open at approximately 10 days after this period of maturation. We also know that at the end of this normal course the enzymes are still constructively active so that oil is elaborated for a few days after boll opening and during the normal dehydration of plant juices.

But now let us take a case in which we have what is commonly known as delayed opening. The enzymes apparently having reached their constructive zenith, reverse themselves, and start destructive action. Seed produced under such conditions, for want of a better term, are said to be field damaged, since at the time of boll opening the oil in them contains from 2 to 3 percent f. f. a.; and if on opening, dehydration does take place,

TABLE 2. — AVERAGE MOISTURE CONTENT OF COTTONSEED CONTAINING VARIOUS PERCENTAGES OF FREE FATTY ACIDS AND OIL

FREE FATTY ACIDS	OIL		OIL	
	18.0% TO 18.9%	19.0% TO 19.9%	20.0% TO 20.9%	20.0% TO 20.9%
5.0	12.53	12.89	12.33	
5.5	13.03	12.59	11.73	
6.0	13.00	13.07	12.42	
6.5	13.30	13.52	12.23	
7.0	13.45	13.04	12.56	
7.5	13.46	13.03	12.44	
8.0	13.69	12.74	13.05	
8.5	13.23	13.04	12.22	
9.0	13.23	13.01	12.50	
9.5	13.72	13.09	12.82	
10.0	13.55	12.97	12.00	
10.5	14.46	12.96	12.34	
11.0	13.64	13.09	13.32	
11.5	14.32	13.22	12.65	
12.0	13.70	13.19	12.37	
12.5	13.55	13.37	13.28	
13.0	14.46	13.69	12.32	
13.5	13.36	13.27	13.40	
14.0	13.62	13.28	13.17	
14.5	14.16	13.48	13.72	
15.0	14.25	13.81	12.56	
15.5	14.55	12.93	13.12	
16.0	13.65	13.34	13.18	
16.5	14.81	13.26	12.50	
17.0	13.95	13.11	13.50	
17.5	14.50	12.66	12.65	
18.0	14.10	13.09		
18.5	14.40	13.55	13.30	
19.0	13.92	13.48	11.60	
19.5		13.22	13.13	
20.0	14.60	12.90	13.20	
20.5	13.76	13.12		
21.0	14.51	13.30		
21.5	14.20	13.35		
22.0	13.95	13.40		
22.5	14.08	13.77		
23.0	13.80	13.33		
23.5	13.60			
24.0	14.30	15.00		
24.5	14.57	13.15		

no higher acidity develops. But if, on opening, atmospheric humidity prevents any dehydration of the plant juice in the seed, enzymic activity continues, and the acidity goes up and up.

It is also evident that once the enzymes have become dormant, two factors — moisture and warmth — in proper combination are necessary to revive them.

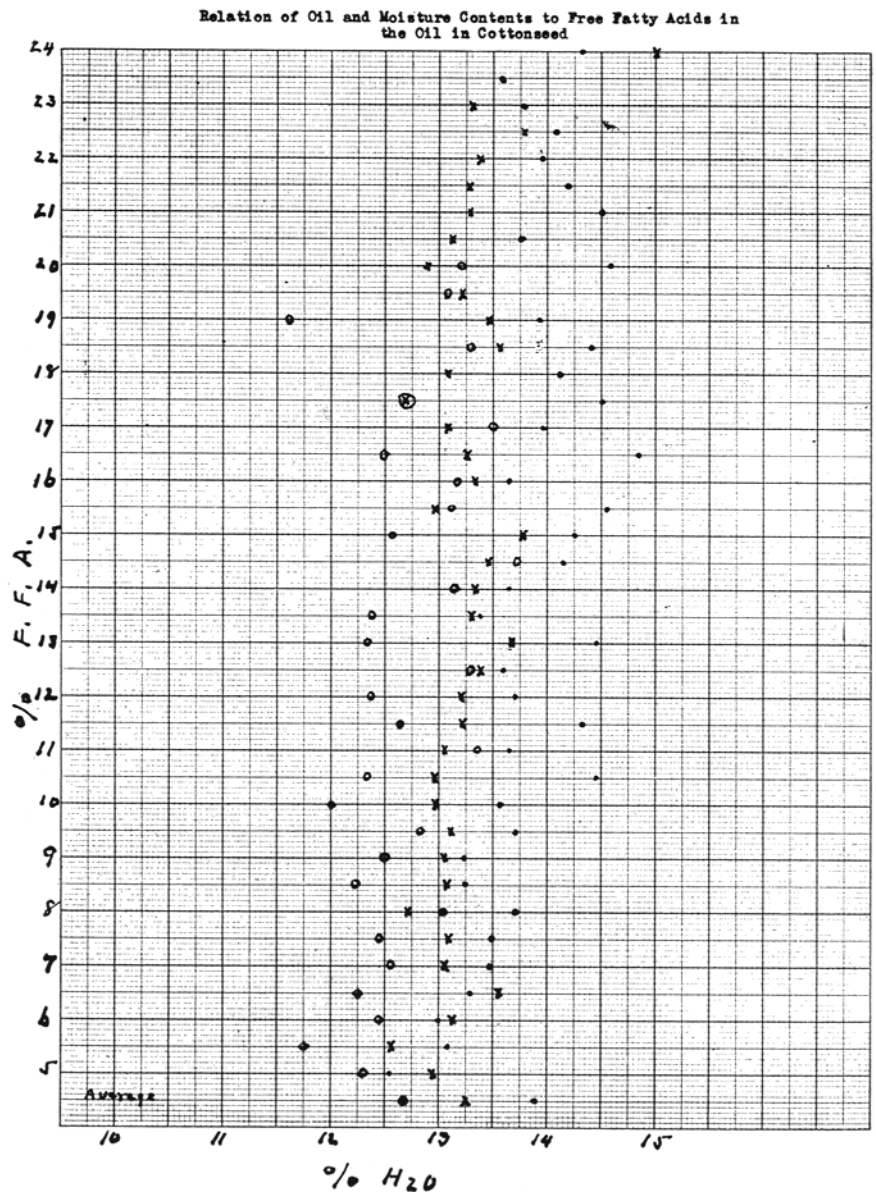
These observations suggest that the sine qua non of low f. f. a. in the oil in cottonseed is an initial dehydration of the plant juices in the seeds within a period of possibly 15 days after the maturation of the bolls. That prolongation of conditions that keeps the plant juices in their original and natural composition fosters the generation of f. f. a.

If this theory is correct we can explain not only the f. f. a. content of so-called field-damaged seed, but also can explain why seed, after having once been dried either while hanging from the bolls or through aeration of the seed cotton and later reabsorbing moisture from rains, may contain considerable and excessive moisture without developing f. f. a. in their oil; and also explain the excessive f. f. a. content of practically all seed in some seasons.

You will note that I said practically all seed. I used that word for the reason that in the worst season of which we have record, there were occasional exceptions. Individual lots bobbed up here and there that contained only normal amounts of acids. These exceptional individuals stood out so conspicuously from all the others as to arouse my curiosity, and I have been able in some cases to secure their history. In every instance I found that the cotton had been picked from bolls that had matured during the few brief dry spells of the season, so that the seed juices had had that initial dehydration essential to putting the enzymes into the dormant state. And in spite of the fact that some of the lots were later exposed to rain so that they contained as much as 14.4 percent moisture, the f. f. a. content did not rise above 0.9 percent.

The obvious conclusion to be reached from these findings is that if in such seasons as that of 1937-38 we wish to secure prime, if not premium oil, and if the farmers are not to lose two-thirds to three-quarters of the base value of cottonseed, then we will have to reverse our former ideas regarding the harvesting of cotton. Heretofore during wet seasons it has been recommended that picking be postponed in the hope that a dry spell would come along. But if this theory is correct, it may be best, from the cottonseed standpoint, to pick as soon as the cotton opens, promptly dry the seed cotton either through driers at the gins or through aeration of the seed cotton in drying sheds.

In the absence of farm curing of the seed cotton the procedure should be prompt and in the following order: First, prompt picking; second, quick drying of the lint at the gin; third, prompt removal of the seed from the gin to the oil mill; and fourth, quick dry-



ing of the seed at the mill before storing.

May I make a suggestion about seed-house driers. Seed-house driers, it seems to me, are designed not for drying seed to cause dormancy of the enzymes, but solely for the alleviation of heating in the seed pile. In my opinion seed drying should be a part of the conveyor system between the receiving platform and the seed house. Such a drier would be no more complicated nor difficult to operate than the seed-house coolers, and would, without doubt, arrest the development of f. f. a.

Ordinarily it probably would not be necessary to remove more than 3, possibly 4, percent of the moisture content to bring about dormancy of the enzymes. Putting it

in other language we might find that reducing the seed moisture to not to exceed 12 percent will accomplish the purpose. The exact procedure of course must be determined from actual tests. It is my purpose if possible to arrange some experiments this coming season, to prove or disprove this theory.

There is yet another angle on the subject of f. f. a. generation that might be gathered from a comparison of the quality of the cottonseed produced in the 4th Crop Reporting District of Mississippi with the quality of the seed produced in Poinsett and Mississippi Counties in Arkansas during this 1937-38 season. Poinsett and Mississippi Counties are in northeastern Arkansas and are the heaviest cotton-producing counties in the State.

The 4th Crop Reporting District of Mississippi is the south half of the Delta and includes the Counties of Humphreys, Issaquena, Leflore, Sharkey, Sunflower, Washington, and Yazoo. During the season of 1937-38 these counties averaged 450 pounds of lint per acre compared with an average of 433 pounds produced in Poinsett and Mississippi Counties, Arkansas.

The moisture content of only about 13 percent of the seed in the

4th District exceeded 12 percent, but the average f. f. a. content of about 95 percent of the seed was over 10 percent. Compare this with conditions in Poinsett and Mississippi Counties where more than 51 percent of the seed contained an average of 14.3 percent moisture but only 13 percent of the seed exceeded 1.8 percent f. f. a. and this averaged only 3.0 percent. The difference between these two sections is about 2 degrees of lati-

tude, so that f. f. a. control may be a matter of temperature at the time of maturity of the seed. It may be that if cotton is picked promptly after the bolls open, and the seed moved quickly from the field to the oil mill and there chilled before storage, f. f. a. generation will be arrested.

It seems to me that both of these angles of approach to better quality cottonseed are worthy of special study.

Hydrolytic Treatment Of Cottonseed Hulls†

By W. H. BALDWIN* and J. A. LeCLERC**

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THE annual production of cottonseed hulls in the United States amounts to more than one million tons. Some of this material is used for cattle feed, a portion being used directly for roughage, and a portion as a filler in order to control the protein content of cottonseed meal sold for feed.

Since the digestibility of cottonseed hulls is quite low (Henry and Morrison, 1936, show 43.7 percent), any treatment which would increase this factor would enhance the value of such feed as is prepared from the hulls. The present work was undertaken to follow the changes produced in cottonseed hulls by various treatments with acids and alkalies.

Alkaline digestions (1), varying in kind of alkali and conditions of treatment, have been made in order to increase the feed-value of straw and wood. This treatment washes out silica and lignin which presumably act as incrusting materials in the untreated hulls. Archibald (2) extracted cottonseed hulls with cold 1.5 percent sodium hydroxide, but concluded from feeding tests that the results "... were in the main negative."

Honcamp and Hilgert (3) found

† Food Research Division Contribution No. 430.

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that by simply steaming straw a product of improved value for use as a feedstuff was obtained.

The hydrolysis of cottonseed hulls with acids has resulted in the formation of reducing sugars. Hudson and Harding (4) and Hall, Slater and Acree (5) have described methods for the preparation of xylose from cottonseed hulls. Sherrard and Blanco (6) used high temperatures and pressure to form reducing sugars from cottonseed hulls; these sugars, they found, could be only partially fermented. Ivannova and Kuronova (7) reported improvement in the feeding value of cottonseed hulls after acid hydrolysis.

Experimental

After hydrolysis with either acid or alkali and subsequent neutralization or washing, the wet hulls were dried in a steam-heated oven at a temperature below 30°C. The oven dried samples were spread in a thin layer to establish moisture equilibrium with the air of the laboratory and allowed to remain overnight. One hundred grams of this material were ground to pass through a one mm. sieve and subsequently used for analysis.

The Methods of Analysis — A.O.A.C. — Fourth Edition — was used as a source of methods for determining (a) moisture by 135° oven; (b) ash; (c) pentosans; (d) crude fiber; (e) ether extract; (f) nitrogen; and (g)

lignin (without correction for protein).

Cross and Bevan cellulose was determined by the method of Phillips (8) with the exception that no permanganate bleach was used at the end. The method described by Shaffer and Hartmann (9) was used for the determination of reducing sugar which was calculated as xylose.

Methods of Digestion

A. With alkali: — Three one-hundred-gram samples of hulls were weighed into two-liter Erlenmeyer flasks. Eight hundred c.c. of 1.5 percent suspension of calcium oxide was added to each. One flask was stoppered and allowed to remain at room temperature overnight (18 hours); another flask was connected to a reflux condenser and the contents were boiled for one-half hour; the third flask was placed in an aluminum pressure cooker and the pressure was held at 25 pounds for one-half hour. Hulls were treated in a similar manner with 1.3 percent sodium hydroxide and with 1.3 percent sodium carbonate, except that the sodium carbonate treatment was not made under pressure. After cooling, each sample was filtered and washed until the washings were colorless.

The hulls which had been treated with sodium hydroxide were given a more thorough washing in order to reduce the amount of ash in the residue. After filtering, the hulls