Extraction of the component parts of the triticale with hexane yielded lipids having a substantially different fatty acid pattern from that obtained with 80% 1-butanol. The hexane extracted only ca. 74% of the lipids obtained by extraction with 80% 1-butanol followed by extraction of the butanol water solubles with chloroform. The hexane extractables are less tightly bound in the tissues and are called "free lipids" by some investigators. The hexane extractables were essentially free of compounds containing odd-carbon fatty acids, such as C_{15} and C_{17}. Others have demonstrated that hexane extracts a considerably lower proportion of polar lipids than does water-saturated 1-butanol (10).

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Quality of Oil from Acid Delintered Cottonseed

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ABSTRACT

Oils were extracted experimentally by direct solvent and prepress solvent processes from saw delintered cottonseed as well as from some of the same seed delintered with dilute sulfuric acid. Oils were refined, bleached and deodorized. Deodorized oils were stored in an air oven at 60 C for 30-35 days. Approximately twice a week, stored samples were analyzed for peroxide value. No significant differences in rate of increase of peroxide value were found among the oils.

INTRODUCTION

Delintering of cottonseed with saw or abrasive delintering machines is a standard procedure in cottonseed oil mills. Delintering is done primarily to decrease losses of oil and kernels in hulls and to decrease operating problems associated with linters on seed (1). However, the linters have values of their own (2).

In recent years, some segments of the cottonseed industry have been considering alternatives to mechanical delintering. One is acid delintering, such as that applied to cotton planting seed. A previous paper described economic evaluation of gaseous HCl and dilute sulfuric acid delintering applied to oil mill use (3). Preliminary investigations of qualitites of oil and meal from the dilute sulfuric acid process were made. This paper reports the results of those studies.

Cotton Inc. developed the dilute sulfuric acid process for planting seed after several years of work and publicized it during 1975-77 (4-8). The process is now in use by several commercial seed companies (J.K. Jones, personal communication).

In this process, seed is wetted with sulfuric acid (10% concentration) with a pickup of ca. 10% of the weight of seed (6). Wetted seed is passed through a rotary dryer which removes the water from the acid, leaving concentrated acid on the linter fibers. Under the influence of acid and dryer heat of ca. 52 C maximum (125 F), the linters become brittle. Tumbling action of the dryer and a subsequent rotary abrader remove embrittled linters and suspend loose linters in the drying air stream. Lesser degrees of delintering than are required for planting seed may be suitable for oil mill purposes. These would be achieved by lower amounts and/or concentrations of acid.

Iron salts are known to increase the rate of oxidative deterioration of soybean oils (9) and of other unsaturated vegetable oils (10). Because of the contact between sulfuric acid and iron in the acid delintering machinery, presence of iron salts in the seed, which might find their way into oil, seemed likely. Therefore, measurement of the oxidative stability of the oils was desired.

Oven storage, in combination with organoleptic evaluations and peroxide determinations may be employed to measure oxidative rancidity tendencies of oils (11), and this method was selected for use.

MATERIALS AND METHODS

This study compared oils extracted from the same lot of seed, half of it saw delintered and the other half acid delintered. Seed was collected in an oil mill after cleaning and first cut saw delintering. Half of it was given a second delintering cut. The other half was transported to a commercial planting seed plant where it was acid delintered. After delintering, the seed was treated with anhydrous ammonia to neutralize residual acid. Both the oil mill and the seed plant were in Mississippi.

Both types of seed were hulled and separated in pilot plant machinery in College Station, TX. Material balances were made on this operation with accumulation and weighing of 9 standard fractions from separating machinery (Table I). The fractions were placed in a cold room (4 C) until they were mixed for further processing by prepress-solvent and direct solvent extraction processes.

For oil extraction, the fractions were removed from storage and portions were weighed out in proportions to the material balance percentages to make 32 kg (70 lb) batches of meats. Separator fractions combined were the first 7 in each list in Table I. Batches were mixed and moistened with water during mixing to ca. 10% moisture.

For direct solvent extraction, moistened meats were heated in a single-kettle steam jacketed cooker to ca. 71 C (160 F) and then flaked with single-pass flaking rolls. Flakes were divided among 3 batch extractors and oil was extracted by percolating heated hexane through the beds of flakes. Oil from each extractor was recovered separately from miscella and carried forward as a replicate in all subsequent processing.

Solvent was recovered from miscella on the same day produced, in a vacuum evaporator without exceeding 74 C (165 F) to levels of ca. 95% oil and 5% hexane. Recovered oil was placed in cold storage until it was refined without further stripping of solvent from it. Solids from extraction were desolventized in ambient air.

For prepress oil extraction, batches of meats moistened to 10% moisture were flaked with flaking rolls. Flakes were placed into the preheated steam jacketed cooker and heated to ca. 88 C (190 F). Then water was sprayed onto the flakes (with mixing) sufficient to raise moisture to 13%. Heating continued for total times of 42-65 min and final temperatures of 103-108 C (217-227 F). Immediately after cooking, cooked materials were pressed with a pilot-plant-size Chuo Giken Ozawa (Japanese made) screw press. Two batches of cooked flakes constituted a run. Press oil was saved for further processing. Press cake was broken and extracted in batch extractors with heated solvent, followed by oil-solvent separation as already described. Prepressing and solvent extraction of cake were performed without replication.

TABLE I

Data from Hulling-Separating Tests

Fraction	Material balance (%)	Composition	
		Kernels (%, mfb)	Oil (%)
Saw delintered seed			
Coarse meats	57.7	76.2	
Fine meats	12.5	79.2	
Purifier top	0.2		
Purifier middle	0.0		
Purifier pan	0.1		
Hullbeater fines	2.2	21.5	
Tailings beater fines	2.4	4.5	
Hullbeater coarse	16.9	1.7	0.78
Tailings beater coarse	2.0	1.2	0.67
Total	100.0		
Acid delintered seed			
Coarse meats	47.7	83.5	
Fine meats	21.5	84.0	
Purifier top	0.0		
Purifier middle	0.0		
Purifier pan	0.0		
Hullbeater fines	2.0	22.0	
Tailings beater fines	3.7	9.0	
Hullbeater coarse	19.8	0.0	0.53
Tailings beater coarse	5.3	0.0	0.62
Total	100.0		

TABLE II

Analytical Data on Seed Used (%)

	Delintered by:	
	Saw	Acid
Moisture	10.8	7.2
Free fatty acid in oil	1.6	2.0
Linters	5.3	4.9
Loose kernels	1.1	0.3
Residual acidity, as H_2SO_4	—	0.038

Analyses were performed according to AOCS methods (12). Oils from all treatments were refined by the standard AOCS cup method, Ca 9a-52, using screw press modification for press oil and solvent method for solvent extracted oils, including oil from press cake.

One sample of each kind of oil was analyzed for gossypol (Ca 13-56). Oils were bleached with standard bleaching earth (Cc 8a-52).

In order to accumulate enough oil (ca. 800 g/batch) for deodorization, refined and bleached oils from refinings with different strengths and quantities of lye were combined. Oils were deodorized in a home-made glass laboratory deodorizer at ca. 737 mm (29 in.) of mercury vacuum and 220 C.

Oil colors were measured by a Wesson-type colorimeter using Lovibond glasses (Cc 13b-45).

Samples of deodorized oil from each treatment and replicate were placed in 20 carefully cleaned and rinsed 4-oz bottles with screw caps. Bottles were placed in an air oven with forced circulation, set at 60 C (140 F). Bottle covers were then unscrewed and set loosely on top of the bottles to exclude dust but not air. Periodically (about twice a week) 3 bottles from each replicate were removed from the oven, lids were screwed on and bottles and contents were allowed to cool to room temperature. The oils were sniffed and tasted by 3 persons and then were submitted to analysis for color and peroxide value (Cd 8-53).

RESULTS AND DISCUSSION

Table II shows the analytical data on acid and saw delintered seed before hulling. Acid delintered seed contained lumps of black, sticky material which broke off from deposits in the acid delintering machinery. It was not removed before hulling, and might have been the source of oil quality problems if any had appeared. Linters contents for both types of seed were similar and were higher than normally expected for saw delintered seed in an oil mill.

Table I shows weight and composition of fractions from hulling-separating seed. Differences in distribution of kernels between coarse and fine meats for the 2 types of seed can be attributed to differences in moisture content of the seed. Drier seed (acid delintered) tended to shatter more during hulling.

Table III presents data on oils during preparation of them for oven stability tests. All oils except direct solvent extracted oil from saw delintered seed had similar free fatty acid levels. The exceptional oil had levels approximately twice as great. The reason is unknown, but presumably some unrecognized situation occurred during processing which allowed enzymatic hydrolysis to occur. This caused refining losses and deodorized oil colors to be higher than in similar oil from acid delintered seed. The other 2 pairs of oils, from prepressing and from press cake, were quite similar to each other for both types of seed.

The persons who sniffed and tasted the oils from oven storage were untrained, and consequently results of these evaluations were inconclusive. Colors of stored oils did not change during approximately 30 days of storage.

Data on peroxide values are summarized on the plot in Figure 1. Data on the 3 replicates for each direct solvent extracted oil were averaged and mean values are shown.

The storage data were analyzed by regression analyses using replicates of direct solvent oils in one analysis, as well as means for these oils in a second analysis, compared with the other 4 oils. These analyses tested the hypothesis that a single regression line would fit the data as well as separate lines. The test compared the sum of the residual sums of squares from separate lines to the residual sum of squares of a single line as follows:

	SS	df	ms	F	
Residual for one line	4121.4	46			
Sum of residuals, 6 lines	2873.8	36	79.8		
Difference (lack of fit)	1247.5	10	124.8	1.56	N/S

TABLE III

Data on Oils during Preparation of Composite Oils for Stability Tests

		Refining			Wesson color—red		
FFA (%)	Gossypol (%)	Lye °B	Amt. lye	Loss	Refined	Bleached	Deodorized
Saw delintered seed; direct solvent extracted oil							
4.6		16	max.	15.4	6.8	1.7^a	
		20	max.	15.2	4.8	1.1^a	
Composite oil for stability test							1.2^c
4.0		16	max.	15.3	4.8	2.1^a	
		20	max.	12.2	12.7	2.5^a	
Composite oil for stability test							1.7^b
4.6	0.72	16	max.	15.2	4.9	1.2^a	
		20	max.	15.1	6.8	1.3^a	
Composite oil for stability test							1.5^b
Acid delintered seed; direct solvent extracted oil							
2.4	0.54	12	80%	10.6	4.5	1.0^c	
		16	80%	7.8	3.8	0.9^c	
		16	max.	11.0	3.7	1.0^c	
Composite oil for stability test							0.5^d
		12	80%	9.4	4.1	1.0^c	
		16	80%	7.3	3.9	1.0^c	
		16	max.	6.9	3.9	1.0^c	
Composite oil for stability test							0.6^d
		12	80%	8.5	4.1	1.0^c	
		16	80%	7.6	4.4	1.2^c	
		16	max.	9.6	3.9	0.9^c	
Composite oil for stability test							0.7^d
Saw delintered seed; prepress oil							
2.1	0.48	16	80%	11.5	4.8	1.4^c	
		20	80%	12.5	4.8	0.9^c	
		20	max.	11.9	4.1	1.1^c	
Composite oil for stability test							0.8^c
Acid delintered seed; prepress oil							
2.2	0.58	16	80%	9.3	4.4	1.5^b	
		20	80%	8.3	4.4	1.9^b	
		20	max.	10.7	4.4	1.5^b	
Composite oil for stability test							0.8^c
Saw delintered seed; oil solvent extracted from cake							
2.3	0.30	12	80%	10.0	6.8	3.0^a	
		16	80%	8.8	8.0	3.0^a	
		16	max.	13.6	4.8	2.3^a	
Composite oil for stability test							1.5^c
Acid delintered seed; oil solvent extracted from cake							
2.9	0.37	12	80%	10.7	7.0	1.0^c	
		16	80%	9.4	7.0	3.0^a	
		16	max.	11.4	6.4	1.9^a	
Composite oil for stability test							1.5^b

[a] Red color measured at 35 yellow.

[b] Red color measured at 15 yellow.

[c] Red color at 10 yellow.

[d] Red color at 5 yellow.

Unless otherwise noted, all refined colors were as in a.

To have been significant at the 5% level, F would have been 2.69. Therefore, the 6 oils were concluded to be no different from each other in storage stability.

Table IV gives analytical values on press cakes, solvent extracted cakes and direct solvent extracted meats. Protein in extracted meats from saw delintered seed should have been ca. 41%. The low value is considered to have been caused by an error in weighing out the separation fractions before mixing and oil extraction. The data on soluble nitrogen and gossypol indicate that meals from saw and acid delintered seed are not different from each other.

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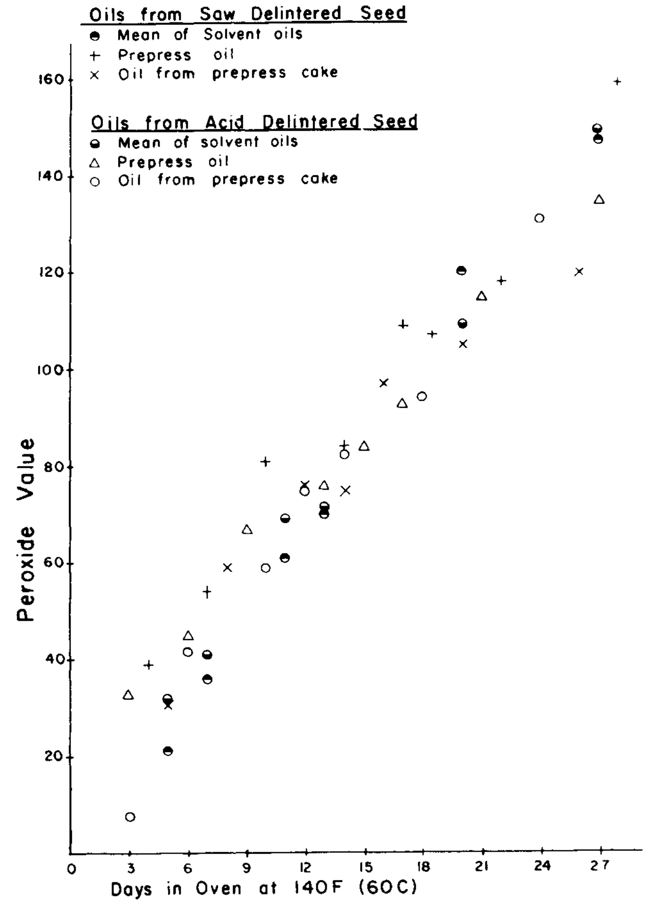

FIG. 1. Peroxide value vs days of oven storage.

TABLE IV

Analytical Values for Solvent Extracted Press Cake and Solvent Extracted Meats

Seed treatment, cake or meal	Replicate	Moisture	Oil	Nitrogen	Protein	Soluble nitrogen	Free gossy.	Total gossy.
Saw delintered seed								
Press cake			7.6					
Solvent extracted cake		2.2	0.5	6.38	39.9			
Acid delintered seed								
Press cake			8.7 6.9					
Solvent extracted cake		2.9	0.7	6.74	42.1			
Saw delintered seed								
Extracted meats	A	8.8	1.2	5.14	32.13	86.5	0.89 (2.77)	1.30 (4.05)
	B	6.6	1.1	5.21	32.56	89.9	0.89 (2.73)	1.16 (3.56)
	C	9.5	0.9	5.16	32.25	90.1	0.86 (2.67)	1.20 (3.72)
Acid delintered seed								
Extracted meats	A	3.7	2.0	6.39	39.9	93.3	1.14 (2.86)	1.48 (3.71)
	B	3.9	2.7	6.20	38.8	90.7	1.18 (3.04)	1.52 (3.92)
	C	3.5	2.2	6.40	40.0	86.7	1.28 (3.20)	1.54 (3.85)

[a] Numbers in parentheses are gossypol values expressed as percentages of the protein in the material.