Cottonseed Oil

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ABSTRACT

Research on the effects of genetics and growing location on cottonseed has shown that oil and fatty acid composition could be improved if geneticists and agronomists would strive for improved seed quality as vigorously as they do for improved fiber quality. Breeding of glandless or gossypol-free cottonseed was a genetic breakthrough. Glandless varieties are now available that produce yields having the quality of fiber and seed equivalent to those of glanded cultivars. Oil, food-grade lecithin and meal byproducts are readily processed from glandless cottonseeds because of the absence of gossypol. Major research programs on cottonseed processing include: (a) testing alternative screw-press and extrusion operations for efficient direct solvent oil extraction; (b) developing alternative solvent extraction systems with ethanol, isopropanol and supercritical fluids; (c) using gas chromatographic/mass spectrophotometric techniques to characterize enzymatic and nonenzymatic mechanisms that produce secondary oxidation off-flavor products; and (d) controlling hexane losses in solvent extraction systems.

INTRODUCTION

The cottonseed industry is old and respected and has for many years enjoyed the luxury of research support from government, university and industry (1-6). Considerable technological progress has been made which has contributed materially to the overall efficiency of processing and improved quality of cottonseed products. Numerous primary and secondary products are now available from cottonseeds and their uses (3,5). Cottonseed kernels or meats are processed to crude oil, from which refined edible cooking oil and foots are processed. The foots are processed to soap, glycerine, fatty acids, or added to the meal for livestock feed. Many highly refined products are then manufactured from these basic materials.

COTTONSEED PRODUCTION

Cottonseed will yield ca. 16% crude oil, 45% meal, 9% linters and 26% hull (7). The losses during handling and processing amount to 4%. Oil and meal together will account for ca. 90% of the total income from cottonseed products available from an oilseed crushing plant. Relative values of the two may vary, with oil generally providing ca. 60% of the total return. In recent years, production of oil and meal are of approximately equal value.

In 1982, world production of the five major oilseeds, including soybeans, cottonseed, sunflower seed, peanuts and rapeseed, is expected to increase by ca. 8% (1976-81) to 154 million tons (8). This total will come close to the record level of almost 157 million tons for the year 1979/80. Cottonseed ranked second in world production between 1976 and 1981 ranging between 22.5 and 24.9 million metric tons, or 14.2-17.3% of world oilseed production (9). During this same time interval, the United States produced between 3.7 and 5.2 million metric tons of cottonseed compared to the rest of the world's production of between 18.8 and 20.9 million metric tons; the best crop-year for the United States growers was 1979/80.

The United States, Soviet Union, Mainland China, India and Pakistan produce most, or ca. 75.3% of the 27.8 million metric tons of the world cottonseed supply (Table I). World production of oil and meal (or 44% protein meal) for 1981/82 is predicted to be 3.5 and 10.1 (or 8.2) million metric tons, respectively. In 1981/82, the United States is expected to produce 5.8 million metric tons of cottonseed, from which 937,000 and 2.6 (2.1) million metric tons of oil and meal (44% protein meal), respectively, should be processed.

TABLE I

Cottonseed Production by Main Producers (9)

	1981/82 (preliminary) (1000 metric tons)	% of total production
Soviet Union	4,950	17.8
China, mainland	5,936	21.4
United States	5,803	20.9
India	2,750	9.9
Pakistan	1,470	5.3
Total	20,909	75.3
Other	6,857	24.7

COTTONSEED QUALITY

The breeding and production of cotton have traditionally been guided by considerations of fiber quality and yield. Until recently, cottonseed characteristics, except for viability and vigor, have generally been ignored. Competition from other seed sources in the oil and feed industry, and the even greater prospect for using cottonseed as a food, have increased the awareness of the importance of cottonseed to food and feed reserves of the world. As a result, the National Cottonseed Products Association, Inc., Memphis, TN, has developed a list of nine goals (5) relating to cottonseed quality which could lead the way toward improved products for the benefit of cottonseed products and the crushing industry (10).

These goals include the need to increase oil percentages and reduce cyclopropene fatty acids and gossypol compositions. Reduction, or removal, of gossypol will reduce the need for expensive refining and bleaching methods in cottonseed oil processing, and improve the nutritional value of the meal for expanded use in feeds and foods to nonruminant species. As research on cottonseed expands, it is becoming clear that many features affect their composition. All efforts to improve cottonseed composition are being accomplished without sacrificing lint yields, fiber quality, or planting performance. Efforts to further improve the food value of cottonseeds to the industry and to the consumer are long overdue.

During the past 80 years, intermittent periods of increased research on cottonseed composition have improved understanding of the influence genetics and growing location have on seed quality, and the quantity of such seed storage constitutuents as oil, protein and gossypol (11). These

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periods of increased interest, however, have been short lived, and little progress has been made toward the stated objective.

The past six years, however, have produced a concerted effort to develop a data base on the physical and chemical composition of cottonseeds from selected cultivars grown in different locations of the U.S. cottonbelt (10,11). These studies will aid in efforts to understand the amount and source of variability in cottonseed composition. In California, the data base study includes 4 Acala cultivars grown during 3 crop seasons at 4 locations in the San Joaquin Valley. In the Texas segment of the investigations, the seed of 4 glanded cotton varieties was grown at 8 locations for one year and then at 4 locations for 3 years. Also included in this study were 12 glandless and 3 glanded cultivars grown at one location.

Selected data on cottonseeds from Acala SJ-2 and the recently released new cultivar, Acala SJ-5 (T5690), are presented in Tables II and III. Statistically significant improvements in cottonseed quality which coincide with the goals set forth include: (a) a reduced portion of the seeds as hull and linters and an increase in the percentage of kernel; (b) decreased amounts of gossypol and cyclopropene fatty acids; (c) improved quantities of oil and protein; and (d) higher levels of select fatty acids, in particular, a significant decrease in palmitic acid and an increase in oleic acid.

An important finding of these studies was that during the past ten years since the development of the Acala SJ-2 variety, the breeding program in California has selectively increased oil and protein content in Acala cottonseeds (SJ-series), and reduced gossypol (10,11, Table II). These observations, and the fact that oil, protein and gossypol composition vary in Acala from different California and Texas locations was clearly shown (10,11). The variation in oil composition is shown in Table III.

The one crop year study to evaluate composition of cottonseed from 4 cultivars grown at 8 Texas locations produced similar observations as those with California-grown cottonseeds (12,13). Data to show variation in composition

TABLE II

Mean Values of Cottonseed Quality Traits of "Acala" Cultivars Grown at 4 California Locations, 1975-1977[a] (10,11)

Quality factors[b]	"Acala" cultivars		Covariance	Lowest standard deviation
	"SJ-2"	"SJ-5"		
Hull	41.60a	36.89b	5.2	1.50
Kernel	45.37a	51.63b	3.5	0.96
Lint	19.03a	21.81b	4.9	1.01
Quantity index	97.29a	109.59b	1.0	3.00
Grade	96.73a	109.29b	1.3	3.30
Oil	19.03a	21.81b	1.2	0.64
Protein	22.25a	23.44b	1.8	0.11
Free fatty acids	1.20a	0.86b	2.4	0.44
Free gossypol	1.03a	0.73b	6.4	0.05
Total gossypol	1.09a	0.80b	5.7	0.04
Phosphorus	0.94a	0.88b	7.0	0.05
Differential settling overflow:				
Free gossypol	0.03a	0.02b	6.4	0.05
Total gossypol	0.06a	0.04b	5.7	0.04

[a]Means among cultivars having the same letter are not significantly different to the Newman-Keuls multiple range test. Values for seed index (SJ-2, 11.68; SJ-5, 11.16), crude fiber (2.20; 2.09), ash (5.07; 5.15), total sugars (6.65; 6.70), ε-free amino lysine (3.87; 3.85), N-solubility (97.46; 97.90), quality index (99.32; 99.74), and differential settling overflow protein (61.44; 61.06) were not significantly different for the two cultivars.

[b]Hull, kernel, and lint are presented as % of seed; oil and protein are % of linted seed; free fatty acid is % of oil; free and total gossypol, phosphorus, crude fiber, ash, and total sugars are % of kernels; ε-free amino lysine is g/100 g flour; N-solubility is % of total protein; and differential settling overflow protein and free and total gossypol is % of flour. All of these values are presented on an "as is" moisture value which was 9.35 and 9.08 for the "Acala SJ-2" and "Acala SJ-5" cottonseed, respectively; values that were not significantly different.

TABLE III

Mean Values of Fatty Acids of Oil from Cottonseed of "Acala" Cultivars Grown at 4 California Locations, 1975-1977[a] (10,11)

Fatty acid (%)[b]	"Acala" cultivars		Covariance	Lowest standard deviation
	"SJ-2"	"SJ-5"		
Palmitic (C16:0)	23.32a	22.69b	0.57	0.26
Palmitoleic (C16:1)	0.72a	0.64b	7.4	0.02
Stearic (C18:0)	2.17a	2.29b	3.2	0.07
Oleic (C18:1)	16.63a	17.26b	0.98	0.20
Cyclopropene fatty acids	0.90a	0.84b	2.7	0.04

[a]Means among cultivars having the same letter are significantly different according to the Newman-Keuls multiple range test.

[b]Values for myristic (C14:0) ("SJ-2", 0.75; "SJ-5", 0.74), linoleic (C18:2) (55.80; 55.84) and linolenic (C18:3) (0.35; 0.34) acids were not significantly different for the two cultivars.

of oil are compared in Table IV. The analysis of variance showed that cultivar and growing location influence cottonseed composition (12,13). Statistically significant location × cultivar interactions for most quality factors suggested that all cultivars do not respond the same across locations. In addition to variability in percentage of oil, for example, for Texas growing locations and cultivars, Table V shows that some variability exists in and fatty acid composition of seeds collected from various crop years.

Geneticists, agronomists, chemists and process engineers have layed the groundwork for finding answers to many of the needs confronting the cottonseed industry. Their studies show that seed storage constituents can be influenced by cultivar and growing location. Careful selection of cultivars and growing locations should yield optimum quality cottonseed products that can continue to compete favorably in the world market, without affecting the fiber industry.

Harvest/Post-Harvest Factors Affecting Cottonseed Quality

In addition to cultivar and growing location factors, the quality of cottonseeds can be affected by harvesting procedures and all subsequent operations involved in handling, ginning, storing and preparing the seed for marketing (14). Reduction in seed quality during various operations is usually associated with mechanical damage, or physiological deterioration resulting from high temperature and seed moisture levels, and their interactions. Research has shown that these losses in quality can be minimized by proper selection and adjustment of equipment, better design of facilities, improvements in operational management, and rigorous quality assurance programs.

A major breakthrough that is revolutionizing the cotton and cottonseed industries has been the module builder which allows field storage of seed cotton. The module builder breaks the connection between harvesting and processing, allowing each operation to proceed at its own pace (5,10,15). Research has shown that the germinability and free fatty acid content of high quality cottonseeds are not affected significantly during module storage of seed cotton as long as seed moisture remains below 12%. Low quality seed deteriorates regardless of moisture level. The moisture in such trash as leaves, soil and branches cause "hot spots," localized temperature rises in the module which transfer to the seed. Temperatures that exceed 50 C during module storage indicate that seeds are deteriorating and that it should be ginned immediately. Good management during harvesting and close monitoring of the conditions of the seed cotton during storage are necessary for module storage.

GLANDLESS COTTONSEED

McMichael (16,17) generated considerable excitement in the cottonseed industry when he published data indicating that varieties with gossypol-free cottonseeds could be developed simply by selecting for the alleles at two genetic loci. Seeds from plants that are homozygous recessive $gl_2gl_2gl_3gl_3$ (gl = glandless) are essentially free of gossypol and related substances. Reviews on the genetic development of glandless cottons were presented by Hess (18,19).

Progress has been made in developing glandless cottons that outyield glanded cottons in lint and seed (20). The protein and oil compositions of some varieties of glandless seeds are comparable to or better than those of glanded varieties, and are affected by genetic and agronomic factors (10,21). Table VI shows the type of variability that exists for oil and fatty acid composition of seeds from 12 glandless cotton varieties and 3 glanded cottons grown at Lubbock, Texas, during 3 crop years. There obviously was little difference in oil and fatty acid composition of seed from these glandless and glanded varieties.

As with glanded varieties of cottonseed, careful selection of glandless cultivars should yield optimum quality cottonseed products that can compete favorably in the world market, without affecting the fiber industry.

Glandless Cottonseed Processing

Ginned glandless or gossypol-free cottonseed can be processed much more efficiently than glanded seed to high quality kernel, oil and meal products (23; Fig. 1). Because of the absence of gossypol, glandless kernels can be prepared for direct consumption or for use in foods as nut replacements. Kernels and meat fines can be flaked and ex-

TABLE IV

Fatty Acid Composition (%) of Oil from Glanded Cottonseed Kernels of Cultivars Grown in Various Texas Locations During Crop Year 1974 (12,13)

Fatty acid	Range of fatty acids		
	Low	High	Mean
Myristic (C14:0)	0.68 Co-Lu[a]	1.16 Lo-CC	0.82
Palmitic (C16:0)	21.63 A-Lu	26.18 Lo-CC	23.68
Palmitoleic (C16:1)	0.56 D-CS	0.82 A-CC	0.65
Stearic (C18:0)	2.27 Lo-Ch	2.88 C-W	2.55
Oleic (C18:1)	15.17 D-CS	19.94 Lo-CC	17.41
Linoleic (C18:2)	49.07 A-CC	57.64 Co-Lu	54.54

[a]Cultivars: A = Acala 1517-70; Co = Coker 310; D = Deltapine 16; Lo = Lockett 4789A. Locations: CS = College Station; EP = El Paso; CC = Corpus Christi; Ch = Chillicothe; W = Weslaco; La = Lamesa; Lu = Lubbock; P = Pecos.

TABLE V

Fatty Acid Composition (%) of Oil from Glanded Cottonseed Kernels of Cultivars Grown in Various Texas Locations During Crop Years 1975-77

Fatty acid	Range of fatty acid		
	Low	High	Mean
Myristic (C14:0)	0.64 Co-CS-'75[a]	1.30 A-CC-'77 P-Lu-'77	0.90
Palmitic (C16:0)	22.18 Co-EP-'75	27.76 P-CC-'77	25.24
Palmitoleic (C16:1)	0.66 D-Lu-'75	1.30 A-CS-'77	0.80
Stearic (C18:0)	2.14 A-CC-'76	3.23 P-EP-'77	2.69
Oleic (C18:1)	13.95 D-CS-'76	21.16 P-CS-'77	17.53
Linoleic (C18:2)	45.84 P-CS-'77	57.83 D-Lu-'76	52.55

[a]Cultivars: A = Acala 1517-70; Co = Coker 310; D = Deltapine 16; P = Paymaster 909. Locations: CC = Corpus Christi; CS = College Station; EP = El Paso; Lu = Lubbock.

TABLE VI

Percentages[a] of Lipid (Moisture-, Lint-Free Seed Basis) and Fatty Acids (% of Lipid) of Glandless and Glanded Cottonseeds Grown During Crop Years 1975-77 at Lubbock, Texas[b]

Varieties	Fatty acids in lipids[c]						
	Lipid	Myristic	Palmitic	Palmitoleic	Stearic	Oleic	Linoleic
Glandless:							
Gregg 35-W	25.3	0.78	21.5abc	1.00	2.5ab	17.4abc	56.2ab
Gregg 45-E	23.9	0.68	20.5ab	0.76	2.6ab	18.2bc	56.7ab
Lambright GI-4	25.5	0.80	24.3c	0.86	2.8b	17.5abc	53.7a
Lambright GI-5	26.1	0.80	23.6bc	0.87	2.6ab	17.2ab	54.1a
Lambright GI-N	25.2	0.82	22.6bc	0.80	2.5ab	17.6abc	55.1ab
Lockett 22-1	24.8	0.89	23.0bc	0.82	2.6ab	18.0abc	54.0a
Lockett 22-6	25.3	0.84	22.6bc	0.64	2.6ab	18.6bc	54.0a
Lockett 22-9	25.6	0.84	23.6bc	0.85	2.6ab	18.2bc	53.0a
Lyman BR-S	25.1	0.79	22.6bc	0.70	2.5ab	17.5abc	55.2ab
GSA 71017	26.1	0.92	23.0bc	0.67	2.4ab	16.5a	55.8ab
Dunn 95	27.2	0.75	21.3abc	0.64	2.5ab	19.0c	55.2ab
Paymaster 7922	26.2	0.56	18.4a	0.92	2.2a	18.2bc	59.1b
Glanded:							
Tamcot 788	25.8	0.75	21.8bc	0.72	2.5ab	17.0ab	56.6ab
Paymaster 468	26.0	0.78	22.7bc	0.79	2.5ab	18.4bc	54.2a
Paymaster 111A	25.4	0.74	21.7abc	0.62	2.5ab	18.3bc	55.5ab
Error	1.243	0.396	1.030	0.019	0.017	0.243	1.796

[a]Means among cultivars (glandless and glanded) having the same letter are not statistically different.
[b]Varieties from performance tests of Ray and Supak (22).
[c]Linolenic acid present in trace amounts.

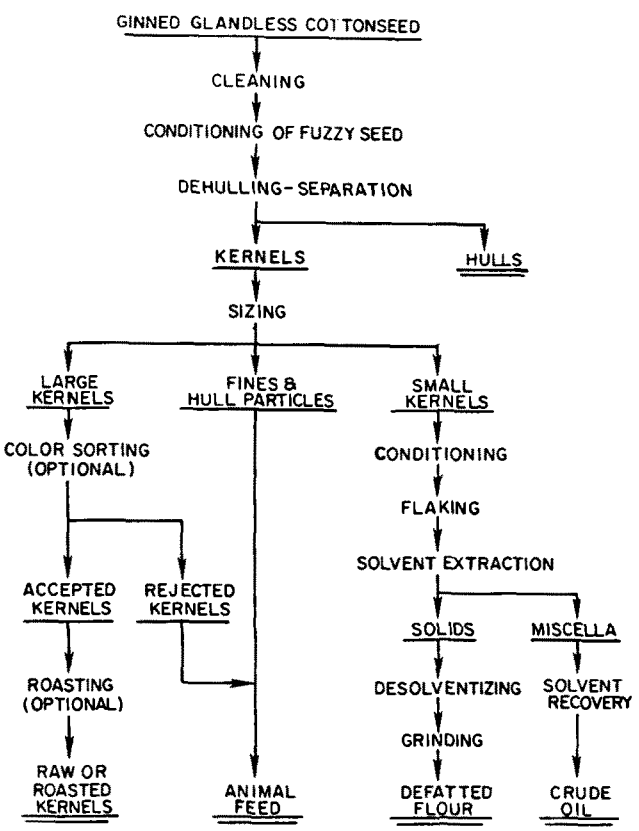

FIG. 1. Flowchart for production of glandless cottonseed products (23).

tracted with hexane to produce flour. To improve oil removal, kernels are conditioned to a moisture content of 8-10%, heated to 71-82 C, and rolled into flakes. Percolation of solvent through the flakes yields high quality oil and defatted meal. With glandless cottonseeds there is no need for extensive heat treatments in processing to bind gossypol in the meal to reduce color reversion in crude oil. Binding of gossypol with proteins reduces their nutritional value, a problem not present with glandless products. Also, to produce a refined oil from glandless cottonseeds requires much less purification processing and loss of neutral oil.

Glandless Cottonseed Phospholipids

Cottonseed phospholipids were marketed only to a small extent in the past (24). The heat and moisture of the old hydraulic press method of extracting oil from glanded cottonseed caused gossypol to bind to constituents of the meal. The screw-press, prepress-solvent and direct-solvent extraction methods result in the binding of some gossypol to the phospholipids (25). The advent of glandless or gossypol-free cottonseed provides an opportunity to produce a food-grade phospholipids fraction as a byproduct of edible oil production. Glandless cottonseed oil thus becomes more economically attractive by increasing revenues, decreasing waste-disposal costs, and reducing emulsion problems that occur during processing.

The phospholipid and fatty acid composition of the lecithin fraction from hexane-extracted glandless cottonseed oil are summarized in Tables VII and VIII. Cottonseed phospholipids are superior to those of other oilseeds, especially soybeans, which presently are the main source of commercial lecithin. Since cottonseed oil contains only trace amounts of fatty acids with more than two double bonds, it is more stable to oxidation and rancidity processes (Table VIII). Soybean phospholipids contain high amounts of linolenic acids that cause flavor, color and odor problems, that will not occur with phospholipids from glandless cotton-

TABLE VII

Composition of Phospholipid Fraction from Glandless Cottonseed Oil

Phospholipid[a]	Composition (% of total phosphorus)
Origin	4.12
Lysophosphatidylcholine	2.56
Phosphatidylinositol	13.41
Phosphatidylserine	2.38
Phosphatidic acid	8.76
Phosphatidylcholine	23.16
Phosphatidylethanolamine	13.46
Phosphatidylglycerol	7.62
Lysophosphatidylserine	ND[b]
Lysophosphatidylethanolamine	ND
Unknown (sum: 6 TLC spots)	25.30

[a]Water (2-4%) was added to hexane-extracted glandless cottonseed oil, stirred 30 min at 70 C and centrifuged to separate the oil and phospholipid-containing fraction (26). The phospholipids were separated by 2-dimensional thin-layer chromatography (TLC) on silica gel-60 plates. Dimension I = chloroform:methanol:7N NH_4OH (65:30:1); Dimension II = chloroform:methanol:acetic acid:water (170:25:25:4). Quantitation of the phospholipids was according to El-Sebajy et al. (27).

[b]ND = not detected.

TABLE VIII

Fatty Acid Composition of Phospholipid Fraction from Glandless Cottonseed Oil

Fatty acid	Composition (% of total fatty acids)
Myristic (C14:0)	0.72
Palmitic (C16:0)	24.19
Palmitoleic (C16:1)	0.72
Stearic (C18:0)	2.96
Oleic (C18:1)	16.94
Linoleic (C18:2)	53.58
Linolenic (C18:3)	0.23
Arachidic (C20:0)	0.41
Gadoleic (C20:1)	0.08
Lignoceric (C24:0)	0.18
Percentage of fatty acids recovered	49.90

seed. Cottonseed phospholipids also contain more lysolecithins which improve their functionality as food emulsifiers.

ADVANCES IN GLANDED COTTONSEED PROCESSING

Cottonseed processing involves the following steps: (a) eliminate leaves, twigs, pieces of bolls and sand; (b) remove linters either once as "mill-run" (7% of total) linters, or twice as "first-cut" (26%) and "second-cut" (67%) linters, the "mill-run" and "first-cut" linters are long, resilient fibers; (c) dehull; (d) screw-press, solvent extraction or prepress-solvent extraction; and (e) desolventize, toast and grind or pelletize (28). The preparation and separation processes necessary to achieve maximum extraction of oils from various seeds are summarized in Figure 2 (29). The crude oil is warmed and treated with sodium hydroxide to enable removal of the soapstock or foots. This refining process also removes the darker colored materials such as gossypol when crushing glanded cottonseeds, leaving a clear yellow-colored oil. Bleaching clay is used to remove any remaining colored substances. A winterization step at 3-4 C removes stearine; materials that turn the oil cloudy at 4-10 C.

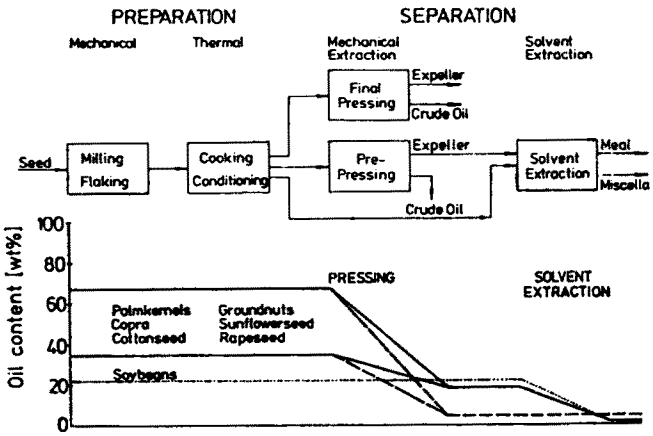

FIG. 2. Oil recovery processes (29).

Economic and regulatory factors have stimulated consideration of alternative methods of processing cottonseeds which would eliminate or reduce the mechanical, i.e., saw or abrasive, delintering step. This has produced information on the effects of residual linters on oil and protein recovery, from cottonseeds during processing (30-32). Observations on the effects of residual cottonseed linters on direct solvent oil extraction showed that (a) linters level (2.2-11.2%), percolation level depth (2-6 ft) and amount of hull removal (41 vs 51-58% meal protein) did not affect oil recovery during extraction – oil recovery ranged from 93.5 to 96.4%; and (b) the efficiency of oil extraction from flakes with maximum hull removal was no different than the oil recovery for the control, which represented current industrial practices. Hulling-separating and preparation for solvent extraction experiments indicated that any linters level within the range of 2.2-11.2% could be successfully hulled and separated. However, higher levels (11.2%) did cause operating problems (plugging of spouts and choking of the hull and seed separator), and oil losses tended to rise moderately with higher linters content when employing universal hulling. The biggest problem in preparation of linters was the tendency of hulls to segregate from meats. This tendency increased with increasing linter levels. High protein meats (51-58%) did not segregate and operation with these meats was suggested as a way of solving this latter problem.

Glanded cottonseeds require moist-heat conditioning for oil removal during processing by the screw-press and direct and prepress solvent extraction methods. The moist-heat treatment step causes excessive pigment gland breakage and gossypol-protein interactions occur which make the defatted material darker in color and reduces protein nutritional value. Ziegler et al. (33) showed that direct hexane extraction at a percolation rate of 2000 lb/hr/ft^2 or higher, at ambient temperature, could extract 92% plus oil from uncooked glanded flakes (0.015 in. thick) made from cottonseed meats with moisture content of less than 9%. A modified filter apparatus was developed to determine mass flow velocities of hexane for extraction of uncooked glanded cottonseed flakes (Fig. 3). A solvent-to-flake ratio of 1.2:1 to 3:1, hexane feed rates of 90 and 140 lb/hr, and percolation rates of 9,157 and 5,378 lb/hr/ft^2 appeared to be critical in the attainment of residual lipid percentages between 0.9 and 1.6% in a continuous pilot plant extractor with this method. Most of the gossypol remained in the flakes formed during processing. Values as low as 0.013% total gossypol were noted in the crude oil.

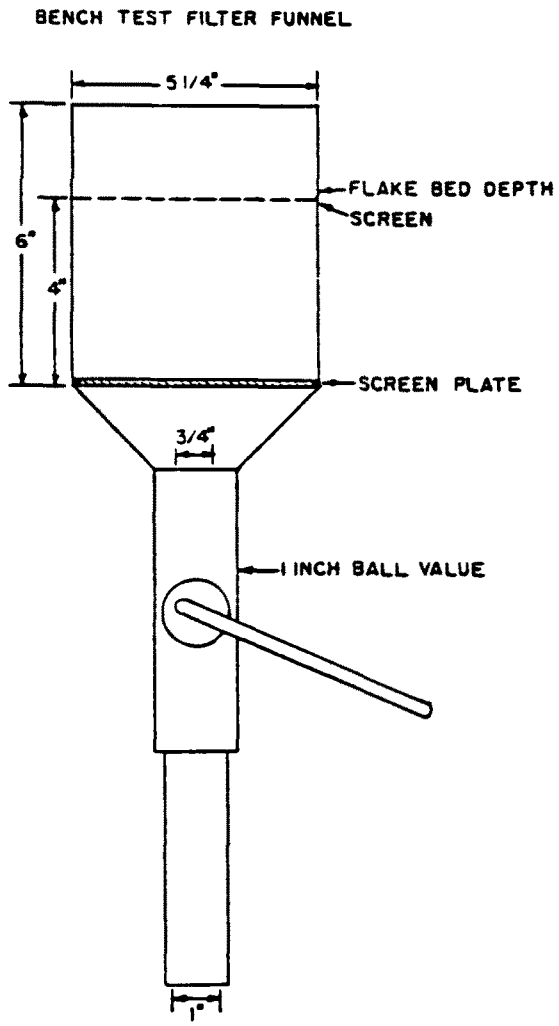

FIG. 3. Bench test filter funnel (33).

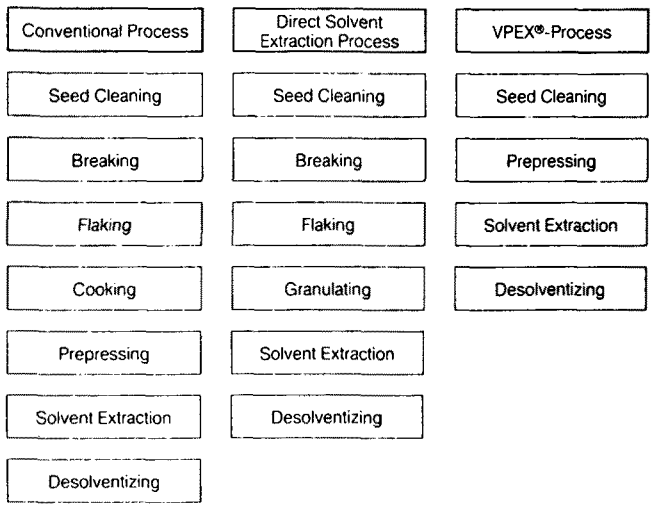

FIG. 4. Comparison of processing procedures showing the potential for reducing the number of steps (29).

Milling and separation processes for oil extraction function efficiently only when individual cell walls are thoroughly disrupted within seeds (29). This concept has led to the development of an oil extraction machine having the ability to perform both operations simultaneously when operating on whole and cold seeds. The possibility of extracting oil with a reduced number of operations exists (Fig. 4). Preparation and extraction is performed simultaneously by a specially modified slow-running screw press (29,34). In this process (named "VPEX®"; 29,34), shearing forces are achieved when the whole material flux is forced through a narrow circular gap formed by the screw-shaft itself and a throttle ring, or deep necking in the free cross section of the press. In these throttles, which are continuously adjustable during operation, the material is extensively sheared; i.e., nearly all of the oil cells are disrupted. For this reason, prepressing can be done without conditioning the material mechanically and thermally, as is usually necessary. Optimum operation depends on there being high pressure in front of the gap due to flow resistance and passage of intact individual seed particles through the gap must be prevented. Several throttle rings within the press cage allow a multistage milling and pressing operation; i.e., as the material moves through the system, it is first loosely stacked and then pressurized within each throttle making new channels available for oil transportation through the press cake and collection. By setting the gap width of the throttles during operation, the press can be easily adapted to various types of seeds, in particular, cottonseeds that have differing moisture content, size and ripeness.

The use of ethanol and isopropanol to extract oil from full-fat cottonseed has been demonstrated by a number of investigators (35-40). These procedures were reexamined and resulted in the development of an extraction process by which full-fat cottonseed flakes are sequentially extracted, first with a relatively dilute alcohol to extract aflatoxins and/or gossypol, fatty acids and non-oil lipids, then with concentrated alcohol to extract semirefined oil (35-38). The new procedure includes four sequential countercurrent extraction steps through which flakes move in series from left to right (Fig. 5). Concentrated alcohol (either ethanol or isopropanol) enters the process at the right and moves countercurrent to the flakes through steps IV, III and II and is diluted before entering step I. Because of the cost, the most concentrated ethanol and isopropanol solutions are 92 and 87.7 weight %, respectively. The solubility of cottonseed oil at 77 C in 87.7% isopropanol is 16%; at 43 C, the solubility is 4%. In 92% ethanol, solubilities at these same temperatures are 4.4 and 1.3%, respectively.

The oil produced by this process was shown to be semirefined, being almost free of fatty acids (0.015-0.3%), gossypol (trace) and phospholipids (trace). Residual oil in the flakes was shown to be as low as 0.5%. An optimum run produced flakes with protein solubility, and free and total gossypol compositions of 70.5%, 0.019% and 0.29%, respectively.

Considerable attention has been given to the use of supercritical gases for the extraction of oil from seeds (40,41). Basically, the process involves compression of a gas such as carbon dioxide to its critical temperature (in this case, 27C) to form a liquid. When the liquified gas is heated and allowed to exceed its critical temperature, it will revert to a gas and no amount of additional pressure will reliquify it. The gas is now "supercritical" and has the properties and extraction capacity of a liquid.

Preliminary studies with supercritical carbon dioxide (SC-CO_2) extraction of cottonseed (conducted at 8,000 psi,

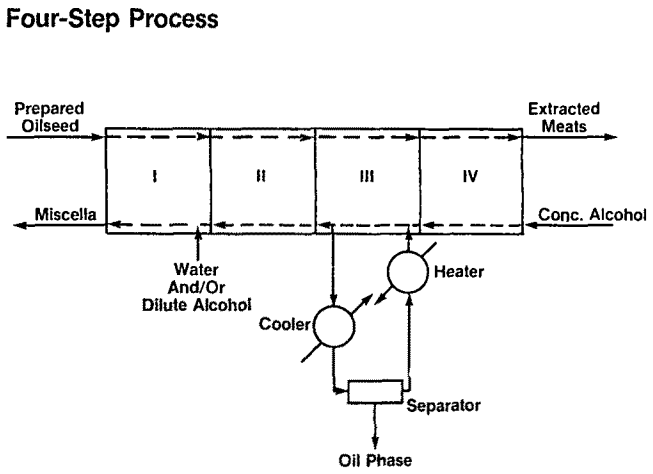

FIG. 5. Four-step alcohol extraction process (38).

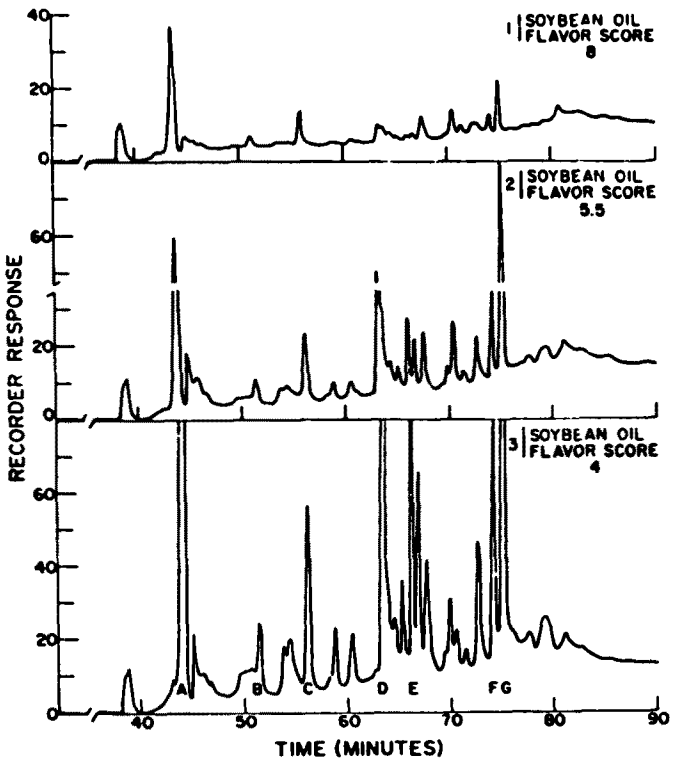

FIG. 6. Profiles of volatiles for three flavor-scored soybean oils: (A) pentane; (B) pentanal; (C) hexanal; (D) *trans*-2-heptenal; (E) *trans*-2, *trans*-4-heptadional; (F) *trans*-2, *cis*-4-decadienal; (G) *trans*-2, *trans*-4-decadienal (42).

50 C) have produced similar results as those obtained with soybeans (J. Pominski, private communication). Compared to the hexane-extraction process, there is less refining loss with the SC-CO_2 technique. The SC-CO_2 extracted oil is light-colored and seems to be at a semirefined stage. In addition, it contains less free fatty acids and phospholipids than hexane-extracted oils from direct extractions of cottonseeds. Crude oils from prepress hexane extractions of cottonseeds contain larger amounts of gossypol. No differences were noted in the gossypol and cyclopropene fatty acid compositions of SC-CO_2 and hexane extracted oils.

Supercritical fluids will probably not be competitive with traditional methods of processing oilseeds and oils (41). This process will most likely be used in a few special applications, such as the deoiling of crude lecithin. Yet if the price of hexane should increase dramatically and if large-scale equipment should become available at reasonable cost, the deodorization of oils, and eventually the extraction of oilseeds with supercritical fluids, may become competitive with steam deodorization and hexane extraction.

COTTONSEED OIL FLAVOR PROFILES

Much research has been completed on objective instrumental tests with gas chromatography (GC) and mass spectroscopy (MS) as a replacement to trained taste panelists to provide information on flavor quality and/or shelf-life stability of processed vegetable oils (42,43). The direct GC-MS method for examining volatiles in oils is a simplified procedure requiring no prior enrichment of the volatiles. The oil is positioned directly into the heated inlet of the GC through a liner tube packed with glass wool. The technique is sensitive to ca. 10 ppb of pentane, pentanal, heptanal, 2-pentyl furan and nonanal when these compounds were added to good quality oils. The identification of volatile components, the mechanisms for their formation (nonenzymatic and enzymatic) and/or their characteristic odors and flavors comprise the information developed for reliable GC-MS analysis and are useful in correlation studies with taste panel scores (Fig. 6; 43). For example, specific volatile components associated with deteriorating soybean oil include pentane, pentanal, hexanal, *trans*-2-heptanal, *trans*-2, *trans*-4-heptadienal, *trans*-2, *cis*-4-decadienal and *trans*-2, *trans*-4-decadienal (Fig. 6).

A plot of taste panel flavor scores against those predicted by pentanal/hexanal multiple regression tests for cottonseed, soybean and peanut oils is presented in Figure 7. Much of the research of this type and thus advances have been done with soybean oils. In studies such as these, accelerated storage tests were conducted by storing the oils in clear glass bottles, loosely stoppering them with cellophane-covered corks, and placing them in a forced-draft oven at 60 C for selected time intervals of 2-16 hr. Immediately after storage, the oils were evaluated for volatiles by GC-MS and flavor by a 20 member trained, experienced, oil taste panel using a scale of 10 to 1, with 10 as very good (bland) and 1 as very bad (strong) (43). The multiple regression coefficients were all significant at the 99% confidence level. These data demonstrated that instrumentation can be effectively utilized in the full flavor analyses of a vegetable oil such as that from cottonseeds.

RESIDUAL HEXANE DETECTION

The concern about environment, health, safety and cost in solvent extraction systems for oilseeds has focused attention on the need for simple direct methods of residual solvent analysis in the cottonseed industry. Dupuy and Vercellotti (44) have extended their GC-MS methods for hexane, alcohols, acetone detection, etc., to monitor cottonseed extraction plant processes and to assay solvent content of finished meals. In general, a 100-500 mg sample of oilseed meal is placed between plugs of glass wool in a 9 × 84 mm glass cartridge with two or three sandwich layers, depending on the coarseness of the meal. To the glass wool in the cartridge is added 50-100 mg of water, depending on the moisture content of the meal, to codistill the solvent of the matrix. The cartridge is heated in a specially designed

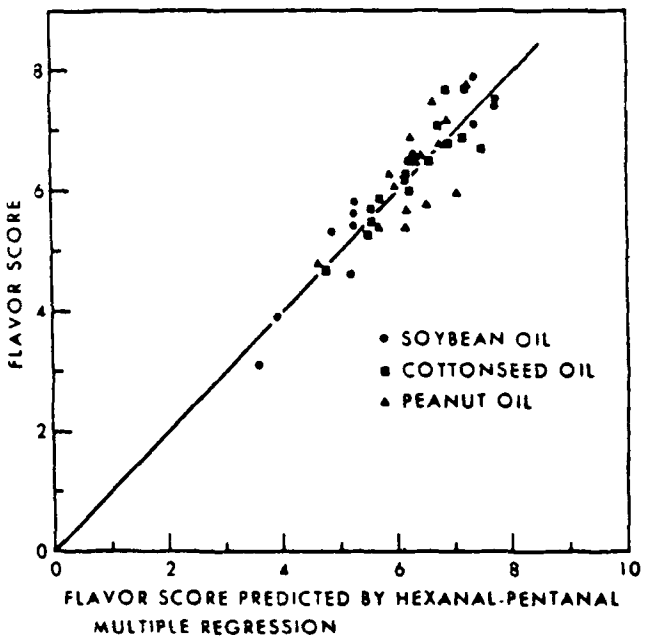

FIG. 7. Correlations of actual and calculated flavor scores for aged vegetable oils (43).

inlet for 20-25 min at 120 C to elute the hexane onto a GC trapping column at ambient temperature. The GC column oven, set at 90 C, is programmed to increase at 4 C/min until after the hexane peak emerges (retention time of about 8 min). Standard solvent response curves are set up by weighing known quantities of solvent into vegetable oil and running in the external inlet system. Detection of solvent in meals at ppm levels is routine for this system.

This direct inlet system has multiple uses such as the direct examination of waste water for solvent. To assess atmospheric solvent in an extraction plant, samples of air can be pulled through a glass cartridge packed with absorbent by using a gas syringe or air monitoring pump. The cartridge is then put into the direct inlet GC system.

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