Experiments With Solvent Extraction of Glandless Cottonseed and Glanded Cottonseed

D. E. CROSS,[1] D. T. HOPKINS,[2] E. L. D'AQUIN, E. A. GASTROCK,
Southern Regional Research Laboratory,[3] New Orleans, Louisiana 70119

Abstract

Preliminary bench scale tests indicated that the rate of extraction of oil from glandless cottonseed flakes is about the same as for glanded flakes. Pilot plant experiments, using 20 lb. batches of flakes in baskets 8 × 8 in. in cross section, showed that the usual percolation rates can be used and will produce the same results with glandless flakes as with glanded. Solvents used were commercial hexane, nearly pure normal hexane, and a mixed solvent of acetone, hexane and water. In some runs raw flakes were extracted; in others, the meats were tempered by heating to various temperatures. Refine and bleach tests were run on the resulting oil. Colors were much lower than generally obtained with oil from glanded seed, most samples testing below one red Lovibond on a spectrophotometer. The meals from the extractions were used in rat feeding tests. The mixed solvent meal seemed to be a cut above the hexane-extracted meals in protein quality, showing up equivalent to a casein based diet. The meal from meats which had been heated to 230 F seemed very slightly inferior to those which had undergone less drastic heating. All glandless meals were much superior to a commercial prepress solvent meal which was run for the purpose of making comparisons.

Introduction

One of the principal drawbacks of conventional (glanded) cottonseed is the presence of gossypol and related substances. These materials, which can be described chemically as polyphenolic compounds, constitute 1% to 2% by weight of the hulled seed. They are concentrated in so-called pigment glands which appear as black specks scattered throughout the kernel. The contents of the glands impart to crude cottonseed oil the dark color which is difficult to remove, especially if the seed has suffered any deterioration or if the oil has been stored for some time under unfavorable conditions. The gland materials also severely limit the outlets for the meal. Although suitable for cattle feed, the quantity of cottonseed meal must be restricted in swine or poultry rations because of unfavorable reactions due to the presence of the gossypol-like substances.

To eliminate the problems caused by gossypol, geneticists have developed glandless varieties of cottonseed (1,2). Chemical and physical analyses of oil obtained from glandless cottonseed show it to be essentially identical to that obtained from conventional cottonseed except for the difference in content of pigments (3).

Although studies have been made of the storage characteristics of glandless cottonseed (4), commercial oil mills have had little experience with processing this type of seed. For this reason, it was decided to investigate solvent extraction of glandless cottonseed under various processing conditions on a pilot plant scale to determine whether the quality of the oil and meal products would be sensitive to process changes and what special problems or advantages could be expected in processing glandless cottonseed in commercial direct extraction plants. Cooking temperature of meats was varied from ambient (raw meats) to 228 F. Both basket extraction and filtration extraction procedures were used. Three solvents were employed: standard hexane, a mixed solvent containing acetone, hexane and water, and a special hexane containing a very high percentage of *n*-hexane. In some cases, for purposes of comparison, parallel runs were made with glanded seed. Refining loss and refined and bleached color tests were run on the oil samples. Chemical analyses and feeding tests were run on the glandless meals produced.

Preliminary Observations

A small quantity of glandless seed was hulled carefully by hand and compared with glanded seed similarly hulled, as to weight and specific gravity of kernel. Average weights ranged from 0.055 to 0.072 g per kernel, while specific gravity ranged from 1.14 to 1.18. There was no significant difference between glanded and glandless meats.

Laboratory scale extraction experiments were made on both glanded and glandless flakes using a glass Buchner type funnel (approximately 2⅞ diameter × 5⅝ in. deep) with no filter disc, and lined with a thin tissue paper. A suitable cork stopper was pressed into the stem of the funnel before the tissue was positioned. The end of the stem was inserted into the neck of an Erlenmeyer flask from which a glass rod extended up into the stem, nearly touching the bottom of the stopper. By lowering the stem into the flask slightly the glass rod could be made to push the stopper up out of the stem. Tests were conducted on 100 g samples of flakes (thickness 0.008 in.) made from coarse meats and from fines. A sample was charged into the pocket formed by the tissue in the funnel. Two hundred grams of hexane at ambient temperature was quickly poured into the funnel, completely immersing the flakes. After a given time interval the stopper was pushed up out of the stem allowing the miscella to drain into the flask. The miscella was then evaporated to find the lipids content. The data, plotted in Figure 1, show that the rate of transfer of oil into solvent for glandless seed was about the same as for glanded seed.

Pilot Plant Experiments

A series of basket extraction runs was made on glandless cottonseed in which the meats were cooked or tempered at various temperatures (including ambient) before flaking. Two runs were also made in which the flakes were extracted by the filtration extraction process (5).

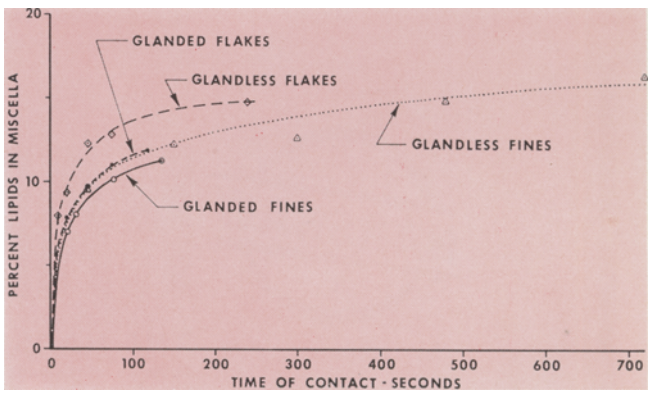

FIG. 1.

[1] Deceased.
[2] Ralston Purina Company, St. Louis, Missouri.
[3] So. Utiliz. Res. Dev. Div., ARS, USDA.

TABLE I

Operating Conditions for Cooking and Extraction

Run No.	Cooking					Extraction		
	Cooker type	Max. cook. temp., F	Cook. time, min.	Flake thickness, in.	Flake moisture, %	Extractor type	Solvent type	Solv. temp., F
1	None	79	....	0.008	8.5	8 × 8 in. Basket	Std. hexane	120
2	None	81	....	0.008	8.5	8 × 8 in. Basket	Std. hexane	120
3	Kneader	200	40	0.008		8 × 8 in. Basket	Std. hexane	120
4	Kneader	200	40	0.009	4.5	8 × 8 in. Basket	Std. hexane	120
5	Kneader	170	44	0.009	5.5	8 × 8 in. Basket	Std. hexane	120
6	Kneader	170	40	0.009	5.6	8 × 8 in. Basket	Std. hexane	120
7	Kneader	148	40	0.009	6.4	8 × 8 in. Basket	Std. hexane	120
8	Kneader	140	42	0.009	5.9	8 × 8 in. Basket	Std. hexane	120
9	Kneader	120	40	0.009	7.8	8 × 8 in. Basket	AHW[a]	105
10	Kneader	120	40	0.009	8.4	8 × 8 in. Basket	AHW	105
11	None	78	....	0.009	8.5	8 × 8 in. Basket	Std. hexane	78
12	None	79	....	0.009	8.5	8 × 8 in. Basket	Std. hexane	78
13[b]	Kneader	228	53	0.009	6.8[b]	10½ in. Buch. funnel	Std. hexane	120
14[b]	Kneader	220	67	0.009	6.9[b]	10½ in. Buch. funnel	Std. hexane	120
15	Kneader	188	36	0.009	5.4	8 × 8 in. Basket	Std. hexane	120
16	Kneader	175	36	0.009	6.5	8 × 8 in. Basket	*n*-Hexane	120
17	Laboratory	220	40	0.012	7.7	7 in. Buch. funnel	Std. hexane	100
18	Laboratory	220	40	0.015	7.8	7 in. Buch. funnel	*n*-Hexane	100
19	Laboratory	165	16	0.007	9.0	7 in. Buch. funnel	Std. hexane	100
20	Laboratory	165	20	0.008	8.9	7 in. Buch. funnel	*n*-Hexane	100
21	Kneader	164	29	0.009	8.2	8 × 8 in. Basket	Std. hexane	120
22	Kneader	163	29	0.009	7.8	8 × 8 in. Basket	*n*-hexane	120

[a] 39% acetone, 60% std. hexane, 1% water (w/w).
[b] Filtration extraction run. Meats were flaked raw and flakes were cooked. Moisture is that of granules after cooking.

All were carried out using warmed standard commercial hexane containing approximately 85% normal hexane, except for runs No. 11 and 12 in which the hexane was not warmed. The other basket extraction runs were made using a mixed solvent (AHW) composed of 39% acetone, 60% hexane and 1% water (w/w) (6). In addition, a series of runs was made on both the glandless and glanded seed using a solvent said to analyze 95–98% normal hexane. The flakes preparation and extraction procedures are tabulated in Table I.

A quantity of delinted glandless cottonseed was thoroughly mixed to assure uniformity, then hulled and purified to produce a supply of relatively hull-free meats. The moisture content of the seed was 9.1%, the oil content 22.3%. The meats were weighed out into 20 lb. aliquots. Each was poured into a polyethylene bag which was in turn placed in a lard can, sealed and stored at 35 F until used. For a given run one bag of meats was charged into a steam jacketed kneader-type mixer for heat treatment. Steam pressure in the jacket and in the hollow sigma-shaped blades was adjusted so that the desired temperature was reached in about 40 min. At the completion of the cooking cycle the meats were discharged and flaked immediately to 0.008–0.009 in. thickness. Eighteen pounds of the flakes were then loaded into a galvanized sheet metal basket 8 × 8 in. in cross section by 23 in. high. The resulting bed of flakes averaged 17 in. in height. The flakes were supported by a wire screen below which was a valved drain connection. The drain valve was closed and hexane, warmed to 120 F (except as noted in Table I) was pumped over the flakes as a coarse spray. When the flakes were completely immersed the pumping was temporarily suspended during a 5 min steeping period. The drain valve was then partly opened so as to control the flow rate from the bottom at about 0.5 gpm and pumping was resumed at a rate sufficient to keep the flakes immersed. This set the extraction cycle for approximately 1 hr at an 8:1 solvent to flakes ratio. In a few cases the percolation rate was too slow to complete the extraction cycle in 1 hr. This was especially true with the mixed solvent runs which exhibited exceedingly slow percolation rates. The bed of flakes was allowed to drain for 10 min after completion of hexane addition. In mixed solvent runs the drain time was 1 hr. These runs are numbered from 1 through 12.

In the filtration-extraction runs (No. 13 and 14), 15 lb. aliquots of raw meats were flaked and the flakes were cooked in the kneader, reaching a temperature of 220 F in approximately 60 min. About 0.75 pounds of water was sprayed into the flakes during the cooking to maintain the moisture content. The granular material discharged from the cooker was passed through an 8 mesh

(Continued on page 30A)

TABLE II

Results of Refining and Bleaching Tests on Extracted Oils

Run No.	Seed	Crude FFA %	Crude color[a]	Refining loss		Refined color		Bleached color	
				Cup 1 %	Cup 2 %	Cup 1[a]	Cup 2[a]	Cup 1[a]	Cup 2[a]
1	Glandless	.814	9.24	4.77	3.53	1.78	1.76	1.65	.82
2	Glandless	.927	7.42	5.1	4.9	1.89	1.77	.71	.60
3	Glandless	1.107	6.08	5.58	5.39	1.15	1.25	1.135	.82
4	Glandless	1.054	6.8	6.76	5.52	1.44	1.32	1.01	.6
5	Glandless	1.374	7.62	6.28	5.91	1.87	1.65	.72	.72
6	Glandless	1.518	6.82	6.47	9.69	2.29	2.3	.39	.8
7	Glandless	.708	7.01	2.74	1.23	2.07	1.85	.49	1.21
8	Glandless	.7	6.81	3.59	1.51	1.89	2.07	.39	1.0
9	Glandless	1.367	48.46	7.93	7.81	2.21	2.38	.94	.85
10	Glandless	1.394	50.88	8.24	9.52	2.62	2.31	.94	1.23
11	Glandless	.83	8.83	2.73	2.77	1.12	1.52	1.53	.5
12	Glandless	.658	4.46	2.4	2.5	1.04	1.24	.71	.62
13a[b]	Glandless	.902	2.92	2.72		1.26		.93	
13b[b]	Glandless	1.002	5.9	[c]	4.6	[c]	1.55	[c]	.83
14a[b]	Glandless	1.077	5.61	4.38		1.06		.73	
14b[b]	Glandless	1.073	4.25	[c]	4.33	[c]	1.16	[c]	.83
15	Glandless	.941	10.69	6.65	6.18	1.95	2.04	.88	.47
16	Glandless	.96	11.91	6.4	5.25	2.05	2.14	.27	.06
17	Glanded	1.25	[d]	7.36	7.5	5.93	5.67	3.8	3.08
18	Glanded	1.303	[d]	6.7	7.16	4.4	3.99	2.02	1.49
19	Glanded	1.29	[d]	[c]	5.08	[c]	8.0	[c]	3.46
20	Glanded	1.4	[d]	[c]	5.29	[c]	3.56	[c]	1.6
21	Glanded	3.49	[d]	10.01	9.97	30.61	28.58	28.76	24.89
22	Glanded	4.15	[d]	11.81	10.9	29.0	29.31	14.39	

[a] All colors were read with Photovolt Corp. Model 140IM spectrophotometer.
[b] Granules from cook divided into two equal portions for extraction. Resulting miscellas and oils not recombined but treated separately.
[c] Weaker alkali denoted by cup 1 in all cases. Not used in this run.
[d] Too dark to read.