Microanalyses for chlorine and sulfur were performed by Mrs. Ruth B. Kelly.

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The Reduction of Free Gossypol in Cottonseed Flakes During Solvent Extraction

LIONEL K. ARNOLD, Professor of Chemical Engineering, and WILLIAM G. JUHL,[1]
Formerly Graduate Assistant, Iowa Engineering Experiment Station,
Iowa State College, Ames, Iowa

AS IS COMMON to most vegetable oil seeds, cottonseed contains small amounts of pigments such as carotenoids and chlorophyll. However, from the standpoint of oil extraction, the most important cottonseed pigment is a yellow solid called gossypol, which is unique to cottonseed. Because of its toxic properties the removal or destruction of gossypol in processing cottonseed meal is important. The pigments are located throughout the tissue of the meats in glands, which are strong, semi-rigid, ovoid-shaped cells about 100 microns long (3). If the gland wall holding the gossypol can be ruptured, the gossypol is converted to a non-toxic form, called "bound" gossypol. In contrast, the "unbound" or toxic gossypol is called "free" gossypol. Because of their small size the cells are generally not ruptured in the various mechanical operations carried out on the seed during the oil removal. Heating, in the presence of moisture, will effectively rupture the pigment glands, but the degree of heating required to accomplish this also denatures the proteins (12, 13), reducing their nutritional value (10). This denaturing is indicated by a reduced solubility in both 3% sodium chloride solutions and water. The solubilities of the gland walls in various dehydrated solvents has been studied by Dechary and co-workers (6), who report that the alcohols and ketones are the most effective solvents. Olcott (9) reports that gossypol is almost completely extracted by chlorinated hydrocarbon solvents, but Boatner (2) found that pigment glands in sliced sections of cottonseeds were unaffected by exposure to moisture-free trichloroethylene for 24 hrs. Commercial solvent extraction with hexane removes the gossypol to some extent, but the amount removed is variable. Wetting the flaked cottonseed with water causes immediate rupture of the pigment glands (4).

Experimental

Preliminary, small-scale experiments in this laboratory in glassware indicated that extraction of cottonseed flakes with extraction grade trichloroethylene would greatly reduce the free gossypol content of the flakes. The resulting meal could then be desolventized at a fairly low temperature with minimum damage to the proteins. In order to check these preliminary experiments cottonseed meats were next extracted in the continuous countercurrent pilot-plant extractor described by Arnold and P'Pool (1). The analysis of the cottonseed products for free gossypol was carried out according to the method of Pons and Guthrie (11). The methods used for moistures and oil were those recommended by the National Cottonseed Products Association Inc. (8). The determination of the water-soluble protein was by a modification of the method employed by Burnet and Arnold (5), using semimicro digestion followed by distillation in a Pregl apparatus.

Results

The effect of extraction temperature in reducing the free gossypol and the protein solubility of the cottonseed oil meal is shown in Figure 1. Extraction

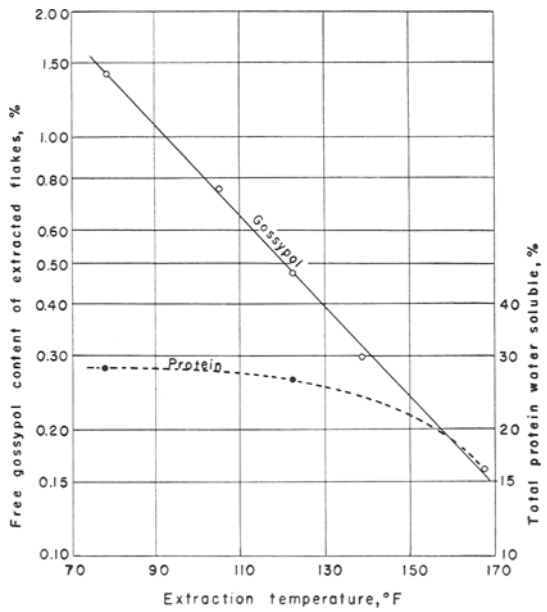

FIG. 1. Reduction of free gossypol and water-soluble protein with increase in temperature.

temperatures up to 122°F., while effecting a considerable reduction in gossypol content, produced only a small reduction in the water-soluble content of the extracted meal.

The most commonly used commercial method of reducing the free gossypol content of the flakes is to

[1] Present address: Lion Oil Company, El Dorado, Ark.

"cook" the cracked or flaked meats. Gibbons (7) reports that the reduction in free gossypol of the meats during the cooking process can be expressed mathematically as:

$$1/X - 1/X_o = M\theta 10^{(0.0296t - 10.02)}$$

where

X is free gossypol in uncooked meats, percentage moisture- and oil-free.

X_o is free gossypol in cooked meats, percentage moisture- and oil-free.

θ is cooking time, minutes.

t is cooking temperature, °F.

M is initial moisture in meats, percentage.

A comparison of the cooking method and extraction with trichloroethylene for reduction in the free gossypol content in the processed cottonseed flakes is given in Table I; the cooking times are calculated by Gibbon's equation. These data indicate that extraction with trichloroethylene is a more effective process for reduction of the free gossypol than the meat cooking process when both the extraction process and the cooking are carried out at a moisture content of 6.55%. Since the extraction does not require the high temperature heating or the excessively long processing times of the meat cooking process, the gossypol reduction is accomplished with only a small reduction in the soluble protein content of the meal.

Under conditions of constant extraction temperature the reduction in free gossypol is apparently a function of only the residual extractables in the meal as shown in Figure 2. The form of the relation for extraction temperatures other than 122°F. is difficult to predict since the reduction in free gossypol content is probably accomplished both by actual extraction and by oxidation of the free gossypol to the bound form.

TABLE I

Comparison of Meat Cooking and Trichloroethylene Extraction on the Reduction of Free Gossypol in Cottonseed [a]

	Reduction in free gossypol, %				
	20	40	60	80	90
Cooking temp., °F.	Time required, in minutes				
220	74	210	462	1,096	2,840
250	9.6	27	60	141	366
280	1.3	3.6	7.9	19	49
Extraction temperature [b] required to give equivalent reduction °F.	82	93	109	134	166

[a] Initial moisture of 6.55% and free gossypol of 1.69%, moisture- and oil-free.

[b] Extraction time of 25.5 min.

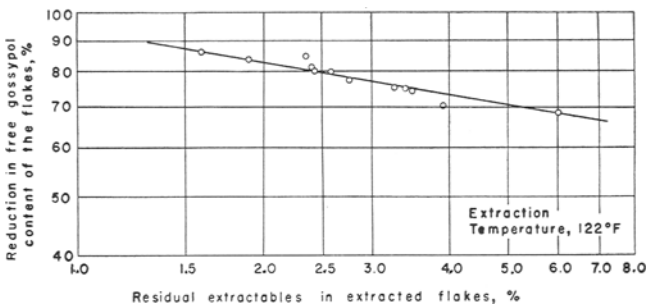

FIG. 2. Variation of the reduction in free gossypol during extraction with the residual extractables.

The toxicity to cattle of certain batches of trichloroethylene-extracted soybean oil meal has raised the question of possible toxicity of other products extracted by trichloroethylene. Since the work presented in this paper was a study in extraction only, the use of trichloroethylene as an experimental solvent should not be construed as a recommendation by the authors that the product resulting from this extraction is or is not suitable as a feed.

Conclusions

Extraction with trichloroethylene was found to be an effective method of reducing the free gossypol content of flaked cottonseed meats. The reduction was found to be a function of both extraction temperature and residual extractables in the extracted meal. Because of the low temperatures involved the meal produced by this method has a higher soluble protein content than a meal in which the free gossypol is reduced by heat treatment.

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