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An Evaluation of Continuous Degumming of Cottonseed Oil

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In previous papers we have given the mechanics of degumming, performance data (13), technical data, and our ideas on methods of handling degummed cottonseed oil in the laboratory (11). There remain certain other considerations which we would like to present. One of these is the economics of degumming. The other is an attempt at evaluating the effects of adding gums to meal *versus* the effects of the addition of soapstocks. The effects will be presented in the light of their possible relation to animal feeding. All evidence is the result of chemical tests only. Space does not permit us to present data on actual feeding tests which have been carried out by ourselves and other agencies.[1]

With regard to the plant economics of continuous degumming, too much emphasis cannot be placed on the necessity for good operational practices and close quality control. No other two factors play a more vital role in the economic success or failure of our degumming process, since, as has been pointed out before, the mechanical problems have been overcome through the use of modern efficient, hermetically sealed centrifuges.

It has long been recognized that excess heat and moisture have damaging effects on the quality of crude oil produced (7). This is especially true of degummed oil. Figure 1 shows how quality was improved by correcting these two deleterious effects.

Figure 1 may seem a bit complex at first glance, but since it is designed to show inter-relation of effects, it could hardly be simplified and still show these inter-relations properly. For convenience the chart is divided into three parts by two heavy vertical lines. The sections are also numbered at the top. Section 1 is from January 1 to January 9, section 2 from January 10 to January 18, and section 3 from January 19 through January 31. The month of January, 1957, was chosen because all three types of degumming took place in that month, thus indicating more clearly the effect each change in process had on the results.

In the first portion may be seen the effects of excess moisture as well as heat. In the second portion the excess heat has been removed, and an improvement in quality can be noted. In the third and final section of the chart can be seen the very marked improvement due to two causes: the removal of excess moisture and the lessening of heat damage because of continuous degumming. Whereas heretofore we had an eight-hour holding period of our crude oil before degumming, we now pump crude oil directly from the final still to the degumming process. Crude oil is degummed and pumped to storage in a matter of a very few minutes. The results of this type of operation can be observed in Figure 1, section 3. In section 1 can be seen the effects of the old eight-hour holding period method of degumming. In section 2 there is an in-between type. It is continuous degumming, but quality was impaired by a faulty evaporator on the crude oil side of our solvent-extraction plant. Oil was being pocketed in a trap, laden with moisture, and subjected to rather high temperature, causing some quality damage. The former eight-hour holding period had caused some color "set" and a slight rise in refining loss that the degumming process failed to correct before the advent of continuous degumming, which has eliminated the necessity of keeping warm, moist oil stored for eight or more hours.

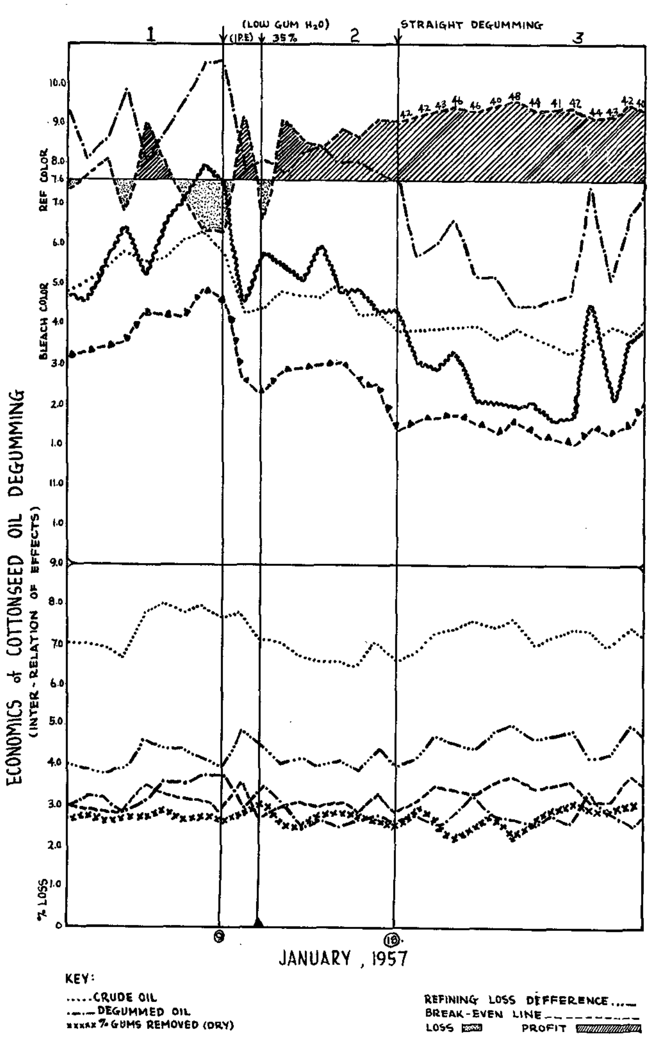

FIG. 1.

[1] Collaborative nutritional studies have been carried out by Texas Technological College, Texas A. & M. College, and Spur Experiment Station.

The chart (Figure 1) also has another function, that of showing the inter-relation of effects in the total process and also of presenting an economic picture of the process by equating economic gain or loss to some common variable—in this case, color. Since by definition in Rule 201 of the N.C.P.A. Trading Rules (10) color is related to loss differences, this method of presentation was chosen to give the greatest amount of information with the least amount of data in order to keep the chart as simple as possible and yet reveal the inter-relation of all effects.

THE CHART is set up in this manner. The dash-dot lines represent degummed oil. The dotted lines represent crude oil. The heavy wave line represents degummed oil bleach color, and the dash-triangle line represents crude oil bleach color. The line of crosses represents the percentage of gums removed from the crude oil. This is, of course, an actual weight loss and simply means there is that much less crude oil to sell. The dash-three-dot line represents the percentage of the refining loss difference between crude and degummed oil. According to Rule 201 of the N.C.P.A. Trading Rules, premiums are figured on losses below 9.0% by multplying the difference of actual loss from 9.0% by 7.5. For ease in calculating, whole integers are used for the difference figures and the answer is in market points. This is the figure shown on the graph by the break-even line. By this token then, only 75% of the refining loss difference is available for an apparent economic gain. If from this 75% the percentage of gums removed is subtracted, the final figure represents an actual gain. If the percentage of gums figure is greater than 75% of the loss difference figure, this represents a loss. The dash lines represent the economic break-even point, or the point of perfect balance, where gain exactly offsets loss. In the lower or refining-loss portion of the chart, this break-even figure is plotted simply as the point where 75% of the refining loss difference exactly equals a theoretical gum loss. If therefore the actual gum loss is less, a gain results. Conversely,

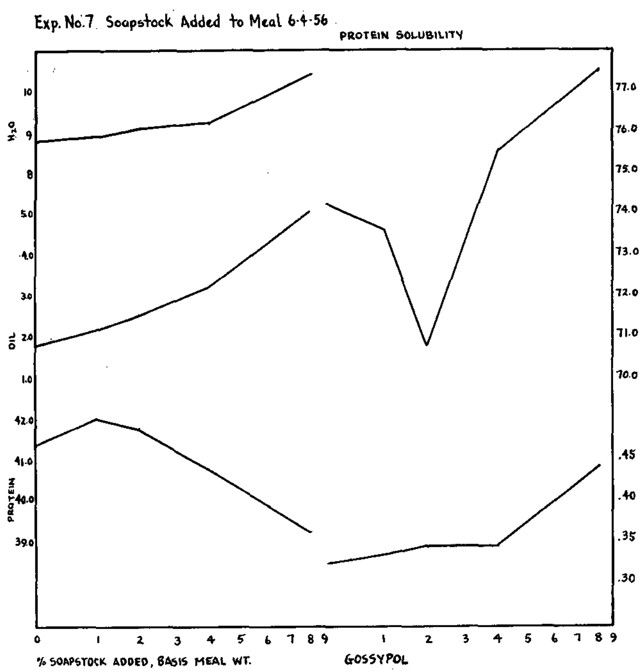

FIG. 2.

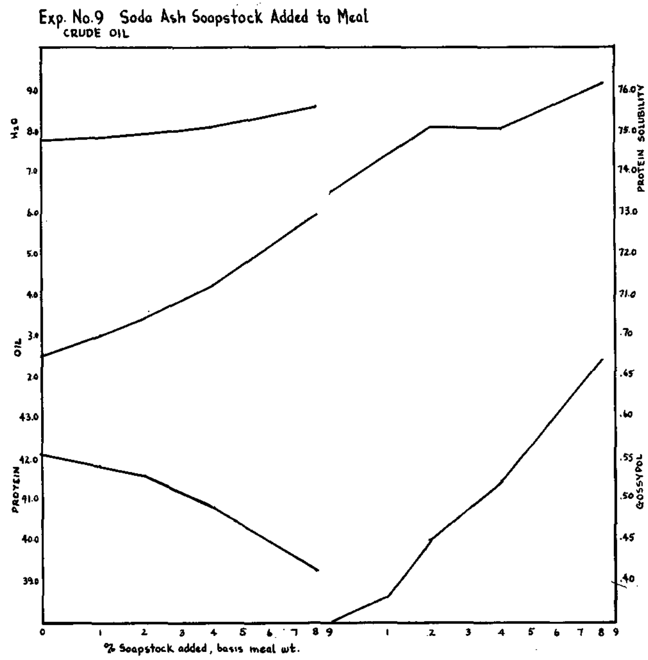

FIG. 3.

where the percentage of gums line crosses above the dash line, this represents a loss.

Since it is entirely possible to offset any apparent gain because of refining loss difference by high color discount, the upper portion of the chart is designed to show this relationship. Since color is related to loss in calculating discount points to be subtracted from the prime crude price, then by solving the equation backwards, we can arrive at a color figure that will show us what color we would have to make in order exactly to offset any gain or loss resulting from the refining loss calculation. Naturally, in a situation where it has already been ascertained that an economic loss exists because of the refining loss-gum relationship, to say that we have to make good any color below 7.6 Lovibond red is a ridiculous absurdity as it cannot be hoped to make up for an economic loss on account of refining loss differences by making any color below 7.6 because there is no premium offered for color below 7.6. Any calculated color falling under the 7.6 color line is stippled, representing real economic loss.

On the other hand, in a situation where a real gain in the refining loss-gum relationship exists, then we may calculate what color we would have to make before color discounts would offset our refining loss premium. This is shown in shaded black, representing profit in terms of Lovibond red color, not market points. It can be seen that the greater the actual refining loss, the worse the color has to be that would offset that increase. If the color in any case is below 7.6, then all of the portion above the 7.6 color line represents profit. As can be seen in the third section of the chart, this is the case in every instance since continuous degumming was started. We have consistently made a degummed oil with a loss of 2.0 to 3.0%, a color of 4.2 to 5.0 Lovibond red, and a bleach color of 1.8 to 2.4 red. These figures are based on results of approximately 400 loading samples. Destination analyses bear out these figures and give some indication of the stability of degummed oil produced by the continuous process.

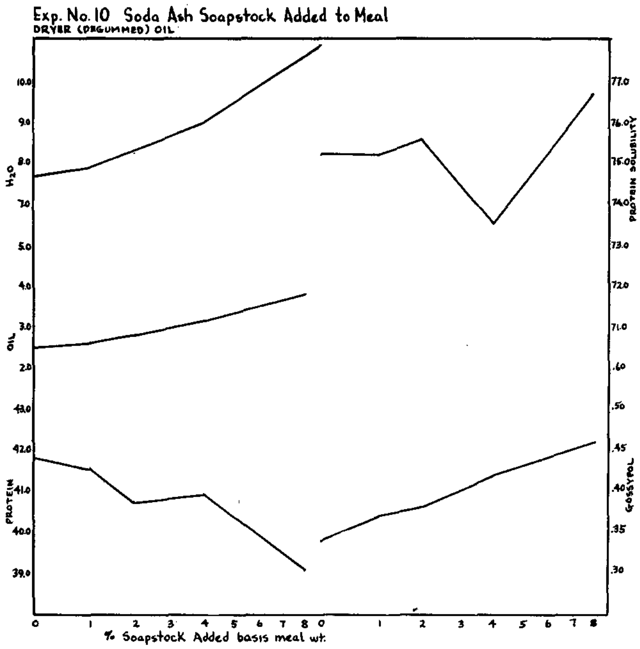

FIG. 4.

THE SMALL FIGURES at the crest of the upper break-even line represent the percentage of moisture left in gums. Since water is added in the degumming process but is immediately removed with the gums, it represents no weight gain or loss in the oil. The figure is used to show the efficiency of the degumming process since the amount of water added to the oil to facilitate degumming is critical. The amount of moisture left in the gums should be very close to 45.0% if efficient degumming is to result. Insufficient water causes gums to be left in the oil and thus increases the refining loss in the partially degummed oil. Too much water is wasteful and may create a color-set condition and a higher refining loss as well as a sloppy gum which is hard to handle and ferments rapidly. The correct amount of water is 2.5% of the oil by weight (12).

A simple one-sheet daily supplemental report will help in accounting procedures to show how much gain or loss is incurred each day. Surprisingly accurate figures can be obtained as to tonnage of degummed oil produced if an appreciation of the difference in specific gravity between crude and degummed oil is recognized. Also the effect of temperature on specific gravity must be realized. In this way oil can be measured in volume without the use of scale tanks.

The second consideration is an evaluation of the effects of adding gums to cottonseed meal *versus* the effects of adding soapstock to the meal. Four charts depict these findings. Figure 2 shows the over-all effect of adding raw caustic soapstock to meal, Figure 3 the effects of adding soda ash soapstock made from refining crude oil, Figure 4 the effects of soda ash soapstock made from refining degummed oil, and Figure 5 the effects of adding gums made from crude oil.

All soapstocks were prepared in the laboratory by using the standard cup test refining and A.O.C.S. lye tables for caustic (5) and a calculated equivalent of soda ash for the soda ash soapstocks. The gums came from regular production in the plant. The soapstocks assayed as follows: caustic soapstock (Figure 2) moisture 30%, free oil 6.5%. Soda ash soapstock made from crude oil (Figure 3) moisture 20.0%, free oil 8.0%. Soda ash soapstock made from degummed oil (Figure 4) moisture 45.0%, free oil 2.0%. Gums (Figure 5) moisture 42.0%, free oil 12.5%. Soapstocks were not acidulated.

In Figure 2 a geometric progression was used in the amount of soapstock or gums added to meal. Five tests were run on each set, namely, moisture (1), oil (2), protein (3), protein solubility (8), and free gossypol (4). By adding caustic soapstock to meal, we see that moisture content rises, oil content rises, but not pound for pound. Addition of soapstock to meal increases oil content only about 10 to 30%, depending on the source of the stock. Stock from centrifuge-refining is the lowest, as would be expected, Because it is freer of entrained oil. This ratio of only 10 to 30% rise holds true whether the test is for oil using petroleum ether (2) or for fat, using anhydrous diethyl ether (6). The protein figure is inversely proportional to stock additions. This is to be expected since stock has little or no crude protein content. The protein solubility picture is quite a puzzle. Presumably because sodium hydroxide solution is used in the protein solubility test, some interference as to proper dilution ratio was encountered on account of the caustic present in the stock. Results are not presented as being too reliable or even indicative, but it may be noted that the general tendency is downward. The free gossypol picture is certainly to be expected. Increase is directly proportional to the amount of stock added since stock is high in gossypol content.

Examination of Figures 3 and 4 does not offer a much better picture than that presented in Figure 2. The protein solubility in Figure 3 is encouraging, presumably because this soda ash soapstock was made from crude oil and would therefore contain gums as well as neutralized fatty acids while the stock in Figure 4 contains no gums.

Finally we see the picture produced by adding

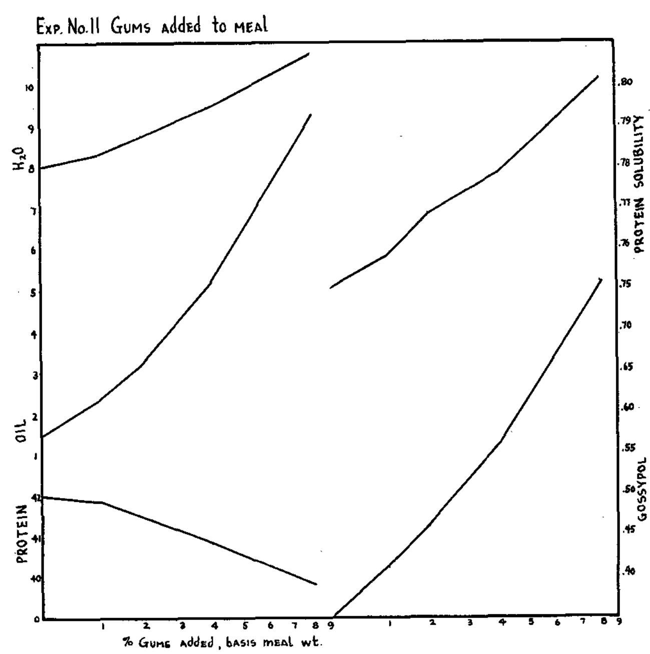

FIG. 5.

gums to meal in Figure 5. Moisture in meal rises rapidly with the addition of gums because of their high moisture content. Oil rises in direct proportion to the amount of gums added as gums are 100% soluble in both petroleum ether and anhydrous diethyl ether even though they contain only 12 to 13% free oil in themselves. A pound of gums added to 100 pounds of meal will increase the oil content of the meal 1%. Protein content of the meal does not decrease as rapidly as with soapstock because gums contain 12% crude protein themselves. There is a definite increase in protein solubility. Gums themselves are 100% soluble in .02N·NaOH. Of course, the gossypol picture is an adverse one for gums contain about half the gossypol normally present in crude oil. Hence the gossypol rise is identical with the soapstock picture, except that it is slightly higher. This results from the fact that much of the gossypol in soapstock has been either converted or decomposed in the harsh refining process.

FROM THE FOREGOING it appears that if an oil miller were faced with a choice of degumming his own oil or buying soapstock to add to meal, he would do well to consider degumming. Economically it is sound. Addition of gums to meal results in a) reducing of dustiness formerly inherent in solvent-process meals, b) raising of the fat content of meal pound for pound, c) weight gain in meal, d) increased protein solubility, e) no sacrifice of crude protein when added in normal amounts, *i.e.*, 1 to 3%, and f) increased nutritive value. By way of explaining this last statement, cottonseed oil gums contain about 65% crude lecithin, which is a phosphoprotein and of proven benefit in animal as well as human nutrition (9). The high gossypol content of meals with gums added should present no problem to cattle feeders, who are the prime outlet of our meal. Space does not permit a thorough study of these premises and their ramifications. but suffice it to mention that the addition of gums to meal is to be preferred over the addition of soapstock from whatever source, as viewed soley from results of chemical tests.

No mention has been made of acidulated soapstock. No thorough investigation was made, but skeletal analyses pointed in the direction we might well expect they would. Since acidulated stock is more concentrated and freer of oil, we get all the same effects we would from raw stock only more exaggerated with the exception of the rise in moisture content of the meal. Acidulated stock is drier. Its gossypol content is higher percentagewise because of the dryness. Protein solubility is inversely affected but in a more orderly fashion than is the case with raw stock.

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Problems Arising in Connection with the Use of Antioxidants in the Food Industry[1]

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RESEARCH on antioxidants started in our institute in November 1945 as a result of the growing interest in greater stability of oils, fats, and fatty products. First the antioxidative effects of oats and oat extracts were studied, but the difficulties encountered soon directed our attention to synthetic antioxidants. We were particularly interested in the work done by Boehm and Sabalitschka (3) on the lower gallic acid esters, especially propyl gallate as an effective compound of low toxicity.

Various considerations prompted us to concentrate our attention on the higher gallic acid esters, which had better fat-solubility and perhaps better carry-over properties. One of the problems however was to obtain these higher esters, not described in the literature prior to 1946, in a reasonably pure state. The direct esterification, which yielded good results in the lower esters, was attended by certain difficulties (2). Later on several syntheses were published (1, 10). The Institute for Organic Chemistry T.N.O. succeeded in developing two syntheses for the higher esters (9), the result of which was the manufacturing of octyl and dodecyl gallate on a technical scale in The Netherlands.

Coinciding with the development of the synthesis, a study was also made of the toxicity and potential applications of octyl and dodecyl gallate. The acute oral toxicity (LD_{50}) as determined with albino rats, was 4.5 g./kg. for octyl gallate and 6½ g./kg. for dodecyl gallate. Dodecyl gallate, in a concentration of 0.2% calculated on the fat fraction of the diet, did not show harmful effects for three generations (5). The harmlessness of the higher gallates has been confirmed by Van Esch (6).

As to the antioxidant properties of the gallates, it was found that they were highly effective in animal fats in concentrations of 0.005–0.01% but that in vegetable oils and margarine scarcely any effect was achieved. Good results were obtained with whole-milk powder, employing tetradecyl and dodecyl gallate in a concentration of 0.01%, calculated on the powder (12). In bakery products the results varied considerably although in some cases the incorporation of 0.05% dodecyl gallate, calculated on the fat, was

[1] Lecture delivered at the symposium on antioxidants, organized by the Low Temperature Research Station, Cambridge, April, 1957.