SOME STUDIES ON

COTTONSEED

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In preparing this paper, I decided to change the subject and present to you a resume of some of the work which we have been conducting at the A. & M. College of Texas. I believe that the paper will be more interesting than if I took the "Chemistry of Seed" and presented a large amount of detailed tabular matter. The paper I had intended presenting will be offered for publication with little or no discussion for your perusal and study.

In presenting to you this resume of some of the studies on cottonseed, I will treat the various phases in a utilitarian rather than a theoretical manner. I do this because I am more interested in the practical side of the industry and most of our studies have had a distinctly utilitarian end.

The oil miller is charged with the responsibility of getting from the cotton seed the maximum yield of each of the various products of the highest grade compatible with economic conditions.

There are many factors which influence his reaching this goal. Some of these factors are human reactions and limitations while others are due to natural causes and economic conditions. As a result of the various evils which beset him, the oil mill operators have developed a philosophy which is unique and a technique of operation which will meet a maximum number of these troubles in the most economic way. Research and experimentation will justify some of these practices while others can not be justified.

Some of the difficulties from natural causes are to be found in the coloring matter, yield, hardness of cake, extracted solids, and acidity. Each of these affect the value of the products to a marked degree.

One of the first experiments conducted was on the possibility of the oil extracting the brownish coloring matter from the hulls.

The brown pigment in the sound hull is not soluble in the oil if no heating or oxidation has taken place. If the hulls have been ground to a meal or the cells have been ruptured by either heat or oxidation, it is probable that some of this will be extracted.

The influence of hulls upon refined oil is shown by Table I.

TABLE I

Influence of Hulls Upon Color of Refined Oil

	% Hulls	Condition	F.F.A.	Refining Loss	Color of Refined Oil
Rolled Meats	None		1.2	5.1	5.8 r
"	4	Sound	1.3	5.1	5.7 r
"	8	"	1.2	5.4	5.8 r
"	12	"	1.1	4.9	5.9 r
"	16	"	1.1	5.2	5.9 r
"	4	Sour	1.3	5.7	7.9 r
"	8	"	1.6	7.2	11.2 r
"	12	"	2.1	9.8	13.0 r
"	16	"	2.5	11.1	16.0 r

From a study of this table it is evident that little color comes from sound, coarse hulls. It is also seen that decomposed hulls have a decided darkening effect on the oil. This is of little direct interest itself to the oil miller, since the meats are usually sour when the hulls have been decomposed. However, it may be inferred from these experiments that finely ground hulls will have some similar effect, as the hull structure is destroyed and the pigment cells are more exposed to leaching by the oil present.

The coloring matter carried by the oil expressed from prime meats and hulls is therefore derived largely from the kernels. This coloring matter may come from three sources—the resin glands, the chloroplasts, and decomposition of these substances.

The composition of this coloring matter has not been definitely determined although numerous investigators have advanced several theories as to its structure.

As previously mentioned the sound hull does not impart any color to the oil, yet we find that the commercial oil is loaded with coloring matter. This must be extracted from some source since the oil as it exists in the cell is seen under the microscope to be colorless or nearly so. This coloring matter apparently is extracted to some extent from the resin ducts (Stanford and Viehoever, Journal Industrial Agricultural Research—13—419 (1918), but the quantity carried is too great to be entirely dependent upon this source. Experiments conducted indicate that this coloring is largely formed from the Leuco-Chlorophyll both unchanged and altered by the action of heat and oxidation (Willistater, Uber der Chlorophyll). Experiments were conducted to determine the extent to which oxidation influences the color. The results are as follows:

TABLE II

Influence of Oxidation on Color of Refined Cottonseed Oil.

Refined oil from meats cooked in atmosphere of methane	3.2 Red
Refined oil from meats cooked in air	6.2 Red
Refined oil from meats cooked in presence of copper sulphate (an oxidation catalyst)	11.6 Red

The oil in each case had approximately the same refining loss and temperature the same.

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From the above results, we are justified in concluding that heat plays a considerable part in the formation of soluble color from the meats. To study the influence that heat has, several experiments were made.

TABLE III
Influence of Heat on Color of Refined Oil

Cooking Meats at	215° F	4.8 Red
	225° F	5.4 Red
	235° F	6.8 Red

Plotting the color against temperature shows that to remove the influence of temperature on the color would necessitate lowering the temperature below that which may be economically used. However, these figures show conclusively that as the temperature increases, the color rises rapidly, which is to be expected.

If we may be justified in taking the figures from Table II as absolute, then the influence of atmospheric oxygen is found to be $6.2\ R - 3.2\ R = 3\ R$. By subtracting this figure from those in Table III, the influence of heat will stand out more clearly and these become 1.6, 2.2 and 3.6 Red, respectively, as indicating the influence of heat. Of course, we are assuming that at each temperature the amount of oxidation in each case will be the same, which is probably not strictly true. However, it is seen that oxidation plays a large part in the formation of color in the refined oil.

In addition to the coloring derived from the chlorophyl in sound seed, it is found that in those seed which have been damaged by frost in the field show an abnormal amount of red. It is believed that this is due to the change in the chloroplasts similar to that occurring in leaves whereby the red pigment predominates and its subsequent solution in the oil. It may be stated, however, that the microscope does not reveal any such phenomenon.

It is also found that immature seed will also yield an oil which is excessively high in red pigment. It is difficult to adequately explain this and all theories propounded so far fail to stand up under a critical examination. It has been suggested that carotins are in predominance during the growing period, being relegated to the background by the chlorophyl upon maturing. This theory fails to explain how an immature seed yielding 90% of the oil of the mature seed will contain more than 10% greater red. The final answer to this must come from the plant physiologist and plant chemist, for it seems to be related to the process of formation just as the color from the frost-bitten seed appears to be connected to the natural processes of autumnal decay.

Seed which have been moistened and allowed to ferment yield an oil which is extremely dark colored. However, it seems probable that this color is derived from the decomposed chlorophyl rather than from the decomposition of the gums and proteins, although these substances do yield oil soluble products which color the crude oil. These latter are probably in a colloidal suspension and are readily removed during the refining process.

In addition to the coloring matter, the producer is concerned with yield of oil. The oil is contained within the numerous cells of the kernel, where it is stored as reserve food to the future plant. To get this oil out of these cells is the problem confronting the miller. To facilitate this, the kernels are rolled into thin sheets to break down the structure as completely as possible and to expose each individual cell. After rolling, the rolled kernels are put in a steam jacketed bottle fitted with stirrers and heated with steam in the presence of moisture. This heating must be accompanied by a movement of the mass during which the cell walls are worn down and the oil liberated. The cell walls are worn down by attrition, the steam and heat merely serve to soften the tissue permitting the attrition to rupture them. It is difficult to understand how the temperature and moisture used can materially influence the physical properties of cellulose and to the speaker it seems as if these cell walls must be of some cellulose product which has not been fully dehydrated and is closely akin to the Hemi-celluloses. In making this statement, the speaker is well aware of the micro-chemical tests that have been applied to these cell walls and the typical cellulose reactions that may be obtained, but in spite of these tests he must hold to his first statement which is based upon the action of the kernels in the mill. In this connection, however, seed that have been dried down to 2-3 per cent H_2O require much longer cooking and stirring in order to obtain a full yield of oil. This tends to strengthen the belief that the cell walls are not of cellulose but of some allied forms which may be hydrated and dehydrated.

It has been found that cottonseed grown under arid conditions have thickened cell walls and necessitate excessive cooking to obtain a full yield. (Latta, Thesis—A. & M. College of Texas). This is a characteristic of desert plants in general. This explains why the millers in West Texas and other parts of the cotton belt have to use excessive cooking methods to obtain the maximum yield of oil from seed produced under arid or semi-arid conditions. This moisture must be available for the growing seed after the boll forms and available until it opens to result in seed with thin cell walls.

One of the difficulties being encountered by the Oil Mill Operators in the southwest is the hardness of the cake. This cake is cracked and sold as "Cracked Cake" and used as cattle feed. Whenever it becomes necessary to use excessive heat and time in cooking to obtain the maximum oil yield, the cake becomes flint like and very hard. This hardness is more intimately related to temperature and moisture than any other factors in cooking. It appears that some of the constituents of the cake form hydratedes and fuse under the heat and upon cooling set to a concrete like mass. There has been no experimental evidence of this fusion so far, but it is being investigated and some evidence for or against this theory will be obtained soon.

When the crude oil is expressed from the kernels, large quantities of pectins, phosphatides, meal, and nitrogen bases are carried along. The meal is in mechanical suspension. The others are either in solution or colloidal suspension. The quantities of these substances carried is dependent upon the condition of the seed and the method of raising the temperature. If the seed are quite dry before milling, the phosphatides are lower in the oil than if the seed are moist. This would lead one to believe that the phosphatides are not present in the cells with the oil. Likewise, the pectins are lower than if the seed are moist. This of course would be expected. However, if the temperature is raised rapidly to 200° F. during cooking, the phosphatides and pectins are lower than in case the kernels are slowly heated. This gives rise to the theory that there is a range of temperatures through which these substances are rapidly peptized and pass into colloidal suspension in the oil. Above this range, the tendency is toward coalescence. Why this is so is not clearly visualized and no theory is advanced.

Since the high temperature tended toward coalescence of these impurities in the kernels, it was believed that the same phenomenon would be observed in settling the crude oil. To ascertain what effect temperature would have on sedimentation, several tanks were filled with crude oil from the presses at various temperatures and maintained at constant temperature as nearly as practicable. The temperatures used were 80° F., 105° F., 130° F., 155° F., and 180° F. The oil was examined every hour for 48 hours for subsidence. Those which were held at an elevated temperature showed no sediment at the expiration of the test, whereas those which were held at the low temperatures settled rapidly, indicating that the coalescence will take place more rapidly below 100° F. than at temperatures above. It is possible that cooling to 70° F. and washing with water will give oils far superior to those now produced. Experiments are now being conducted with this end in view. I might say that we have produced oils which after settling had a crude color of 14 r in a 5¼ inch cell. To produce such oils, the temperature of pulling was at 220-225° F. and the moisture in the cooked meats high.

The free acidity in the oil is closely related to the percentage of moisture present during ripening. It seems that as long as the moisture is below 10%, the oil will be neutral or nearly so. It the moisture is over 10% the oil will sour. Likewise, seed exposed to moist air absorbs water according to the well known laws of partial pressures up to 10 or 11%. At that percentage of moisture, the curve takes a sharp break upward. This leads to the conclusion that moisture less than 10% is not present in the seed as free moisture, but is present as a hydrate of some of the seed constituents. When we exceed this amount, the free moisture is floating around in the intercellular spaces and within the cells and is available for hydrolysis of the oil and heating.

It, therefore, is essential for the seed to be dried to less than 10% moisture before storing.