CHEMISTRY IN THE DEVELOPMENT OF THE COTTONSEED INDUSTRY*

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THE COTTONSEED industry has developed from the disposal of a waste product under compulsion of law to one turning out useful products, mainly for food purposes, having a value of \$150-300,000,000 annually. This development has been greatly fostered by the help of chemistry, and it should be of interest to trace briefly this connection. Chemists have directed their efforts partly to improving processes and making new products and partly to the analytical control of operations leading to improved and more profitable results.

The application of chemical control came first and may be divided into about three periods: first, the early history or the dark ages prior to about 1880; second, 1880 to about 1905 in which chemistry made its first appearance in the industry, demonstrated its value, and became an important and indispensable factor in the industry; and third, the modern period of close control of operations up to the present time.

In the early days no control whatever was exercised in milling and refining, but whatever came out of the press in the form of oil or cake was taken for granted and what came out of the crude oil in the form of refined oil in the refinery was likewise taken for granted. In 1880 there were some 45 mills, four refineries, and one chemist in the industry in the United States. About 1900 there were probably about 600 mills, with about half a dozen laboratories devoted to the testing of mill products, and possibly some 30 refineries, large and small, with perhaps half a dozen laboratories devoted entirely to refining and lard compound testing. All of these laboratories were operated by the larger producing companies.

The first chemist to enter the industry, as far as known, was Mr. W. B. Albright who entered the employ of the N. K. Fairbank Co. at Chicago in 1879. This company was engaged in lard refining, and at that time was the largest buyer of refined cottonseed oil, which they used in making so-called "refined lard," or lard compound, and about 1879 or 1880 they installed their own refinery. They were pioneers

There in many developments. were, as far as known, only three earlier refineries in the United States located respectively at New Orleans, Cincinnati, and Providence.

Two outstanding contributions seem to have been made by Mr. Albright, first the chilling roll for the rapid solidification of lard or lard compound, with Mr. O. G. Burnham, and second the use of fullers earth for bleaching, both of which were pioneer developments and became of enormous value to the in-dustry. Both of these processes were duly protected by patents, which of course have long since expired.

In 1883 Dr. David Wesson entered the cottonseed industry, and to him is largely due the introduction of proper control methods both in the mills and refineries. He has stated that he established the first laboratory in the United States for testing mill products at Chicago in 1887, when in the employ of the N. K. Fairbank Co. He afterwards left this company and went to the Southern Cotton Oil Company where he spent most of his active years, and here also he was instrumental in introducing methods for control of both mill and refinery operations, and in the improvement of these operations.

Mr. James Boyce followed Dr. Wesson in the 1890's, and afterwards became Chief Chemist of the N. K. Fairbank Co. and the American Cotton Oil Company. He likewise exerted a strong influence in the adoption and extension of laboratory control of mill and refinery operations in this early period, and in the improvement of these operations. All three of these men deserve a great deal of credit, as pioneers, not only for improvements in processing which they introduced. but also for initiating chemical control of operations and selling to the managements of their companies which were at that time the principal concerns in the industry, the idea of the benefits which could be derived from chemical analysis of the products of their operations. Up to the late 1890's, however, little more than a fair start had been made in this direction.

The improvements that have come about in the industry and the connection between these improvements and chemical control can be better understood if we first look at the status of the industry as it existed in the late 1890's. Cottonseed was stored with little regard to its quality or condition and it was a very common experience to have wet seed heat and spoil in storage causing a loss of many thousands of dollars to the owners. There was no way then known to avoid this when the seed could not be worked through the mill immediately, and these losses were considered unavoidable.

In the mill the separation of meats from hulls was very imperfect, oftentimes as much as 5% of whole meats being left in the hulls and 3 to 4% of oil absorbed in the hulls. The pressed cake very commonly ran as high as 12% in oil content and anything as low as 8 or 9% was considered good.

The crude oil was refined by rule of thumb methods as to choice of lye, temperatures, time and rate of agitation, etc., with no attempt at studying conditions to see if one set of conditions would give better results than another, and indeed there was no standard for comparison such as our present standard refining test. A great deal of secrecy existed in all branches of the industry and a refiner, for example, would never think of discussing his procedure with another refiner, but each assumed that his work was better than that of anybody else.

When samples of mill products were subjected to analysis it was often found that the pressed cake from two different mills would vary greatly in oil content, and then some alert mill manager began to wonder why his mill produced cake with 12% oil while another produced cake with only 8 or 9% oil. This led to more chemical tests and then it would be found that the 12% mill sometimes made cake with only perhaps 9% oil. It did not take long for the mill manager then to realize that every pound of oil left in the cake was sold at the price of cake instead of at the much more favorable price of crude oil and that his men must get the oil down to 9%

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all the time. Likewise, the ammonia tests on the cake or meal were found to vary greatly and needed standardizing, and losses of meats or oil in hulls were often serious. The manager could only know what his mill was doing by calling on the chemist to aid him.

These developments quickly led to employment of analytical chemists by the leading companies, so that about 1900 the American Cotton Oil Company had four laboratories devoted exclusively to testing the products of approximately fifty-four mills, and the Southern Cotton Oil Company had about two laboratories, as far as can be determined now, for testing the products of some seventy mills. Usually only two or three samples of cake and other products from each mill were tested each week. The chemical analyses made by these laboratories as a guide to the managers in controlling and improving the work of the mills soon showed that profits could be greatly increased by first knowing what mills were doing in their press rooms, and secondly applying corrective measures where necessary. Cake with 12% oil very quickly became a thing of the past and the average oil content was soon reduced to 7-8%, and in modern times to 5-6%.

These developments occurred first only in the larger companies, but their importance led to the establishment of numerous independent chemical laboratories in the period from about 1900 to 1910 for bringing the benefits of chemical analysis of mill products to the numerous cottonseed oil mills outside the big companies. The first of these, as far as known, was established by Mr. Lehman Johnson at Memphis some time prior to 1900. Other laboratories were started by Dr. Paquin at Memphis and by Mr. Tilson at Houston about 1900, Mr. Law about 1901. Mr. Battle soon afterwards, and Mr. Barrow in 1905. Other laboratories followed. These chemical laboratories were certainly of great value to the industry in enabling the mills, both the independent ones and those operated by large companies, to know from frequent tests of their products how efficient or otherwise their operations were, and on the basis of these tests to gradually improve their press room work and thereby greatly increase their yield of profitable products and their profits. The separation of meats from hulls is now almost ideal and the pressed cake rarely contains more than about 6% oil. and oftentimes 5% or less, and while this result has of course been achieved largely by improved mechanical appliances and mechanical operation, it is a fact nevertheless, that it could never have been obtained if the way had not been pointed out by the chemists, nor can it be maintained today without chemical aid. When we remember that the average profits of all the cottonseed oil mills in the United States for the past eight years were only \$.17 per ton of seed crushed, and that a difference of less than .2% of oil in cake would have wiped out even this small profit, the importance of close control of mill operations and the importance of the analytical chemist in making such control possible become readily apparent.

This improvement of mill operations did not proceed spontaneously and many years elapsed before some of the old timers learned to appreciate the help that they could derive from laboratory tests. Mr. Boyce, on the other hand, has said that the crude oil mills of that time were "crude" in more ways than one.

Methods of analysis were imperfect, and while the larger companies attempted to perfect methods for their own laboratories, true cooperation was lacking between them and the independent laboratories, and this led to organization in 1910 of the Society of Cotton Products Analysts, the predecessor of the present American Oil Chemists' Society, which has done a wonderful work in improving and standardizing methods of analysis, and in maintaining a high degree of accuracy in laboratory work.

The development of the check meal analyses is of special interest. In the period of the early 1900's the two leading companies each required their mill laboratories to analyze check samples of meal and other products to check up on their accuracy. This led to great improvements in the accuracy of the work at all laboratories. The Southern Cotton Oil Company developed this work to a high degree under the leadership of Dr. F. N. Smalley, then Chief Chemist of this Company, and he also extended it to state chemists and some others who desired to participate in these cooperative analyses for their own benefit. This work, as is well known, was later taken over by the American Oil Chemists' Society and has continued to the present day under the name of the Smalley Foundation. The value of this work to the industry from year to year cannot be overestimated. Practically all the laboratories in the country specializing in this kind of work participate, and it has led to an extremely high degree of accuracy and reliability, particularly as to oil, ammonia, and moisture in meal. This affects not only the current operations of the mills themselves, but the buying and selling of their products as based on tests in commercial laboratories, and the control of their products by state chemists when sold under the various state laws. I do not believe there is any other system of cooperative analyses in any industry that has brought about as high a state of perfection as the check meal work conducted by the Smalley Foundation, and our industry could ill afford to see this work given up.

In the period since about 1905 the milling industry has learned to utilize to the best advantage the scientific knowledge acquired from chemists and others. Seed is handled and stored more intelligently, with drying and cooling when necessary, so that losses commonly experienced in the early days from heating and rapid deterioration of wet seed have been largely eliminated. Losses of oil in hulls and pressed cake are consistently kept at a minimum. Protein content of the meal is controlled largely by regulating the proportion of hulls, so that the product is uniform. This is better for the buyer, and avoids giving away excess protein on the one hand, and violation of law by shipping meal with low protein content, on the other hand. For these and other purposes chemical laboratories are today recognized as necessary for the proper and economical conduct of milling operations.

With regard to the refining of crude oil, chemists were employed in some of the larger refineries in the late 1890's, but true chemical control of refining with resulting reduction of refining losses to a minimum did not occur until many years thereafter. There was no satisfactory standard method of testing oils for refining loss and color. but about 1902 a committee of three was appointed by the Interstate Cottonseed Crushers Association to develop such a laboratory test, principally for the benefit of refinery laboratories, and the committee was instructed to impart the information to those who in their opinion should have it. This committee consisted of Messrs. Wesson, Boyce, and Hulme. A method was then drawn up, and a typed copy kept in the possession of each member of this committee, but not published. They were very secretive about it, however, and I believe gave out information only to a few others and then only verbally. It was considered unwise to put it into the hands of those who were not already expert in refining, because of the variable results obtainable from it. The need of a laboratory refining method that could be used as a basis of settling trades as well as for comparison with plant results later led to the compiling and publishing of our first official refining test about 1910. While this proved of great use to the industry with the improvements that were made in it from year to year, yet it was not sufficiently exact and reproducible in its results, so that about 1927 the whole problem was carefully investigated and a revised method adopted which assured more closely agreeing results in the hands of different operators. This method with slight modifications is still in use and has largely eliminated disputes and differences between chemists which oftentimes cast discredit on the chemists themselves, and it has also provided a standard basis for comparison of plant results which has enabled improvements to be made in plant operations, equipment, and personnel, so that where formerly it was common to find factory losses as much as 50% higher than the laboratory test, they now are usually less than 10% above the standard laboratory test. Here again the setting up of a standard by the chemists has been an invaluable aid to the industry.

The modern improvement in refining by the use of a continuous and rapid centrifugal process on 2^{-1} large scale has further improved the work of refineries, but here again there would be no way of knowing the possibilities, or any standard of comparison between new and old procedures, if we did not have the standard laboratory test as a basis for comparisons.

Improvements in processing and development of new processes and products have enabled the industry to grow and greatly extend its field of usefulness.

The first deodorizing process for cottonseed oil was devised and installed by Mr. Henry Eckstein about 1891 for the N. K. Fairbank

Company, at Chicago. It consisted in heating the oil with boiler steam to about 340°F. and blowing live steam through it without superheating. A closed tank was used with an outlet to the atmosphere for the steam and vapors, but without vacuum. The improvement in the oil over the undeodorized product was of course very great, although far from equaling the quality of modern deodorized products, and greatly extended the field of use of cottonseed oil for edible products. This procedure was improved upon by Mr. Boyce, particularly with regard to condensing the vapors instead of letting them go free into the atmosphere, and also later on by the introduction of superheated steam.

The Wesson deodorizing process was introduced about 1900 and was the first to combine the use of superheated steam and a vacuum, thus greatly improving the quality of the product.

The outstanding new chemical development was probably the application of the catalytic hydrogenation process to edible oils, especially cottonseed oil, about 1910. This was soon adopted by all the leading oil refiners. It led to the production of a new all-vegetable compound in which the hard animal fat previously used was displaced by a hard vegetable fat made by hydro-genating cotonseed oil at a cost which was usually much lower. It also led to the introduction of an entirely new type of shortening consisting of cottonseed oil hydrogenated to about the consistency of lard. These chemical developments greatly extended the use of cottonseed oil with consequent benefit to the industry.

A recent development in connection with milling which should be mentioned is the seed grading system which offers many advantages to the industry and which again requires extensive use of chemical laboratories.

The keeping quality of cottonseed oil products has been improved to a remarkable degree by the use of antioxidant substances discovered by chemical research. These ensure a better quality of products over a long period of time and should result in increased usage of cotton oil products.

Modifications of oil molecules have been developed, especially in the form of mono- and diglycerides which, even in small proportions, impart new and valuable properties to shortenings. This is another chemical development that should extend the use of cottonseed oil.

Numerous other new products and new fields of use for cottonseed products have been developed with the aid of chemistry, some of which may be briefly mentioned. From lint we now obtain nitrocellulose, cellulose acetate lacquers, photograph films, rayon and a high grade of paper. From cottonseed meal is made a non-toxic, wholesome flour suitable for use as a human food. The oil finds extensive use in new forms of shortenings and margarine. From foots are obtained soap, glycerin, nitroglycerin, distilled fatty acids, candles, pitch, and paint.

The question next arises: What may chemistry be expected to do for the industry in the future?

While, of course, nobody can predict what the future holds, some things that might reasonably be looked forward to may be mentioned.

The mills will be operated with the highest type of mechanical equipment and under constant chemical and mechanical control. The increased overhead thus involved will be offset by lower operating expenses and better yields of desired products, and by running substantially twelve months every year instead of about four to eight months as many have done in the past. They will handle peanuts, soya beans, and perhaps other oil seeds, as well as cotton seed.

It is likely that the extraction process will eventually displace the expression process at least partly, with the result of yielding some 50 pounds additional oil per ton of First class edible oil and seed. cake can now be made by extraction. This process, on the basis of a pound of oil being worth \$0.06 more than a pound of meal, would yield about \$3.00 per ton greater gross return, or in 1934 about \$34,-000,000 additional to the industry. Compare this with the average net profit of only \$0.17 per ton made by the mills of the country during the past eight years.

Crude cottonseed oil will be stored indefinitely under properly controlled conditions with little or no deterioration, just as linseed and other oils are stored today, and with the modern continuous centrifugal equipment a refinery will be able to operate with a much smaller equipment and take care of its requirements from day to day throughout the year instead of having to install sufficient equipment to refine a flood of crude oil as fast as received in the producing season and having the equipment standing idle for about half the year.

Refining processes should be improved so as to remove nothing but the unwanted ingredients of the crude oil, that is, the free fatty acids and the non-fatty organic matter. An oil containing one per cent of free fatty acids and one per cent of organic matter, which refines according to the present kettle process with perhaps 6% loss, should be refined with a loss of but little more than 2%.

Bleaching methods will be improved with possibly the substitution of chemical agents for fullers earth, with reduced loss of oil in the operation, and obtaining lighter colors. which should eventually approximate water-whiteness.

Deodorizing processes should be perfected to yield a more perfect product in a much shorter time. Besides being tasteless it should not give off any odor at any temperature used in frying. There seems no good reason why four to eight hours' treatment, for example, should be required to remove the minute percentage of odors from cottonseed oil as required by present processes.

Hydrogenation should be further perfected, especially in the direction of speed, selectivity, and flavor so that all the unsaturated constituents subject to rancidity may be readily hydrogenated with little if any action on the more stable constituents, and without introducing the odor and flavor caused by present hydrogenation procedures.

Many other possibilities exist. There is no reason to think that we have reached or even closely approached the limit of improvements which chemistry can offer to the cotton seed industry, and I have no doubt myself that the next 25 years will bring about changes just as important as those of the past 25 years.

Some Nutritive Developments in Soybean Products*

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S OYBEAN products have enjoyed a rapidly increasing importance in the United States during the past few years. According to the Bureau of Agricultural Economics soybean oil as recently as 1930 accounted for less than 1% of the production of vegetable oils from domestic materials in the United States. But in 1936 soybean oil had grown to 12% of the domestic vegetable oil production.

Soybean products during this same period have won for themselves increasing importance in the field of nutrition. In 1931 only $31\frac{1}{2}\%$ of the soybean oil produced found its way into edible products. According to figures just compiled by the Statistical Committee of the National Soybean Oil Manufacturers' Association 85.3% of the domestic soybean oil produced in 1936 was used in edible products. 95.5% of all soybean meal produced in 1936 was used in feed, or food, while only 1.8% was used for technical purposes, such as plastics, glues, or caseins.

It is the purpose of this discussion to review a few of the nutritive developments in soybean products which are partly the reason for the popularity for these products for edible uses.

One of the reasons for the rapidity with which soybean oil meal has won favor as an ingredient in livestock feeds is because of its high biological value. In a series of biological assays made in the Purina Mills Laboratories, on white rats as the test animal, expeller type soybean oil meal of high quality showed the highest biological value of protein of the ingredients tested and exceeded even dried skimmed milk and fish meal in growth factors. The relative biological values found are as follows:

Each sample tested represented a composite sample of a car of the ingredient.

Expeller type soybean meal	
	100
Dried skimmed milk(6 samples)	- 92
Menhaden fish meal(6 samples)	82
Milk albumen(1 sample)	82
Sardine meal	79
Liver meal	64
Corn gluten meal(1 sample)	49
Meat scrap	41

Unfortunately not all samples of soybean oil meal have the same high biological value. Some samples tested had only half the biological value of other samples. This same wide variation, however, is found with fish meals and meat scraps depending upon the source of supply of raw material and method of production.

When one remembers that the soybean is not one uniform product, but that there are literally hundreds of types and strains—that over twenty well known varieties are commercially available in quantity, it is easy to see why soybean oil meal and soybean oil may vary from week to week and from oil mill to oil mill. It is possible that certain varieties of soybeans or even soybeans raised in different localities vary considerably in the combination of amino acids which make up the protein. The United States Department of Agriculture have assayed a number of varieties of beans which showed considerable variation in their amino acid composition.

Hayward, Steenbock, and Bohsteadt in 1936 in studying the effect of heat as used in the production of soybean oil found that "raw soybeans contained protein of low nutritive value as determined by grams of growth per gram of protein eaten."

"Commercial soybean oil meals, such as the expeller meal processed at low temperatures, 105° C. for 2 minutes, or the hydraulic meal at 82° C. for 90 minutes, contained protein similar in nutritive value to raw soybeans. However, commercial soybean oil meals which had been prepared at medium and high temperatures, such as expeller meals processed at 112° to 150° C. for 2½ minutes, or hydraulic meals at 105° to 121° C. for 90 minutes contained protein of about twice the nutritive value of raw soybeans, or low temperature meals."

"The food intake of all rats which received either the raw or heated soybean diets adlibitum was found to be similar for the first few days of the feeding period. This sug-

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