

dence was obtained that cholesterol ingestion increased the ester fraction of the blood cholesterol, an observation in line with the view that the esters aid in fat transport. A study of the lipid composition of the fatty livers reveals a large increase in neutral fat and a marked increase in cholesterol esters; on the other hand, the phospholipid content appears unchanged. Iodine values of the different lipid fractions show that cholesterol probably exerts a saturating effect upon the fatty acids. The only apparent conclusion from these data, at present, is that cholesterol is in some way concerned with fat metabolism, especially where the liver is involved.

In summary, we can still state that the role of cholesterol in the vital process remains a mystery which thirty years of research have failed to solve. That it has a function in cell life can hardly be denied. It confronts us from the epidermis to

the inmost bodily protoplasm; from the cerebral cortex to the pedal capillaries. The renewed activity in studying this problem at the present time is a reasonable indication that new light will soon be shed on the function of this important biochemical compound.

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## THE METHOD OF

## SOYBEAN OIL EXTRACTION

AS DEVELOPED AT THE EDISON INSTITUTE OF TECHNOLOGY\*

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The Edison Institute of Technology was founded in 1929, by Mr. Ford in honor of Thomas A. Edison. The purpose of the Institute is to train students along the lines that Edison pursued in his work and to give them an opportunity to study farm products for industrial possibilities.

In Mr. Ford's Early American Village, in Dearborn, the Edison Institute has its research laboratory which is large enough for semi-plant production; the raw materials for carrying out experiments come from an experimental farm. The students deal with the practical problems and some of the products of the Institute have been used in the Ford car.

After working on practically every principal farm crop grown in the United States we finally chose the soybean for complete development because of its versatility. It not only has a wide use in industry but pos-

sesses a high nutritional value. It is complete enough to sustain life for a good length of time. The soybean is a summer leguminous annual, the beans resembling navy beans in size. The Orient and the United States produce 94 per cent of the world's crop, 14 per cent being grown in the United States. The bean contains 17 to 20 per cent oil, 35 to 40 per cent protein and from 33 to 35 per cent carbohydrates which are digestible. It is rich in minerals and contains vitamin A, B, and D and has an alkaline ash. The soybean may be grown wherever corn or cotton is grown, taking from 70 to 120 days to mature.

After a series of experiments with the soybeans it was found that the meal could be utilized in the manufacture of plastic material and the oil in making enamels for the car. The meal, to be satisfactory for use, in the plastic, however, had to have an oil content of 2 per cent

or lower. We decided to develop an extractor because the previous methods of extraction did not remove the oil to this extent with sufficient speed and economy. The batch system of extracting the oil is not continuous, and the time required for extraction is excessive. The pressing method is not continuous and the pressed meal contains too high an oil content to warrant its use. The expeller method is continuous but the meal also contained too high an oil content to be used.

We wanted to build an extractor suitable for our needs and one that the farmer could afford to buy and operate. It must be compact, operate continuously, and would remove 95 to 98 per cent of the oil.

The work was started in the laboratory using a small Soxhlet apparatus and, after observing its action, a large apparatus was built that would handle 20 pounds of meal. This method of extraction

\*A paper presented at the eighth fall meeting of the American Oil Chemists Society in Chicago, October 11, 1934.

proved satisfactory except for difficulty in recovering the solvent from the meal and the time required.

Experiments showed us that some chlorine solvents would hydrolyse into HCl and have an injurious effect on the meal and parts of the extractor. Some petroleum solvents had too high a boiling range and some were too high in specific gravity. Aviation gasoline, light petroleum naphtha and Hexane can be successfully used because of a satisfactory boiling range and specific gravity.

Then a study was made on how to apply the above method to a continuous apparatus. Finally the idea was conceived of using some of the principles embodied in an Archimedes' water screw by conveying the meal through a stream of solvent flowing counter current to the meal.

After considerable experimentation our present extractor was built, consisting of a flaker, percolator, steamer, filter, preheater and still.

In this type of apparatus the preparation of the beans for extraction required some study. First, if the meal is ground too coarse all of the oil cells are not broken up, making it difficult for the solvent to get at the oil and consequently slowing up operation. Second, if the meal is ground too fine it will cake around the screw conveyor and a high percentage of the meal will not be touched by the solvent; also too many fines will be carried over with the solution of oil and gasoline to the filter. We tried flaking the beans on a large metal roll, and finally developed a flake which proved very satisfactory. It was only necessary to use beans with the proper moisture content to prevent powdering.

In this extraction apparatus the beans are first fed through a set of five high rolls (B) which reduces them to a flake with a thickness of approximately .010. A screw conveyor (C) then conveys the flakes into the neck of the percolator (D) where they fall through gasoline onto a revolving screw in the percolator (E). This screw conveys the meal upward against the gasoline. The flakes, after being in contact with the gasoline for approximately 12 minutes, continue on the same screw into the preheater (F). This part of the extractor is six feet long and has a steam jacket around the conveyor tube. The heat from this jacket not only vaporizes the solvent, but also brings the meal to a high enough temperature to prevent the sparger steam in the steamer from condensing upon hitting the

meal. As the meal passes from the preheater into the steamer chamber (G), low pressure steam which has been superheated to 260° F. is sprayed upon the meal, forcing the vapors into the condenser (H) from which the gasoline and water flow into the decanter tank (P). The meal comes out of the sparger section dry, free from solvent and containing an average of 1 per cent oil.

The gasoline fed in at the top center of the percolator tube (E) works its way down counter current through the meal, washing out the oil. This solution overflows at the neck (D) and is pumped through the filter (K) that removes the fine particles of meal.

The filter consists of a chamber approximately 24 inches in diameter containing a circular drum, made of perforated sheet metal with a canvas bag covering it. After the chamber has accumulated a sufficient amount of meal the meal is mixed with the gasoline by the revolving drum and the solution forced into the percolator through a valve between the filter and the percolator tube. Originally a Cuno type filter was used, but it was necessary to supplement it with a canvas bag filter. Later a single, simple working filter was designed to take its place.

The solution of gasoline and oil from the filter passes through a heat exchanger (L) into the top of the still; the heat exchanger utilizes the hot vapors passing out of the still to preheat the gasoline and oil solution. The still consists of three sections, the first section housing coke which is used to break up the stream of solution and present a greater surface to the sparger steam entering at the bottom of the coke section. The second section, which is three feet high, has a reservoir 12 inches deep with a steam coil in it to volatilize off any solvent which remains in the oil that the sparger failed to remove. The oil and water emulsion flows into the bottom section, which is a settling tank for separation of oil and water. The water overflows to the sewer and the oil to the storage tank (N). The vapors from the still pass through the heat exchanger and into the condenser (O), the water and gasoline flowing to the decanter tank (P). The gasoline passes from this separator to the storage tank (R) from which it is again pumped to the percolator. Both the oil and the meal have been heated sufficiently to remove all traces of the solvent.

The advantages of this extraction apparatus are that it is simple to

build and operate, and will remove 95 per cent of the oil. The required horsepower for running is 11.

The original still consisted of a column of coke with a sparger line at the bottom and a kettle below the coke column equipped with a steam coil to volatilize off the water. This type of still was not continuous and the oil was burnt. This still was replaced with a fractionating column which was previously used in the fractionation of alcohol solutions. This worked satisfactorily for awhile but was too expensive to build and too complicated to run. Therefore the present still was built with a reservoir to separate the water and oil by gravity rather than by heat.

The extractor tube is made from standard 8-inch water pipe and is 22 feet long. The solvent inlet into the tube is two feet below the upper end. A 6-inch diameter screw, 37 feet long with a lead of 6 inches, is used to convey the meal through the entire apparatus.

A standard 8-inch pipe, 6 feet long, is used in the preheater. For the steamer an 8-inch pipe with a 12-inch pipe for a jacket is used. The steamer has an 8-inch vapor line welded in at the top at a 10° angle. The difference between the split halves of the 12-inch pipe used for the jacket is filled in with  $\frac{3}{8}$  boiler plate.

The oil produced in this extractor is generally lighter than pressed oil and is suitable for some paints and enamels without further refining.

Our present solvent is aviation gasoline with a boiling point of 116° and an end point of 260° F. The specific gravity is .7054.

This extractor was in the Industrialized Barn at "A Century of Progress" at the opening of the Fair. The barn was displayed to show a closer relationship between agriculture and industry, which Mr. Ford believes is essential to help heal the ailments of both. The object is to have the farmer grow raw materials for industry and do the initial processing himself, thereby getting a greater return for his product.

Recently, however, we have removed this extractor from the Barn and one with a change in design, and a new heat supply method has been installed. This is to show the possibilities of how a simple working plant could be afforded by the farmer and run without any great mechanical knowledge. This new apparatus utilizes the gases from a 4-cylinder engine in place of steam, and the water circulates through

the water jacket on the motor to supply heat for the evaporator. A new type flaking machine which extrudes the flakes was supplemented for the rolls.

Although the extractor removed is not obsolete, this new extractor is more simple in design, the cost is very low and a steam plant and steam engine are unnecessary. The

new extractor will handle 100 bushels a day. We are still experimenting with this type of extractor and hope to have it for distribution by the first of the year.

## PHOSPHATIDES IN AMERICAN SOY BEANS AND OIL

By George S. Jamieson and Robert S. McKinney

CONTRIBUTION FROM THE OIL, FAT AND WAX LABORATORY, BUREAU OF CHEMISTRY AND SOILS, U. S. DEPARTMENT OF AGRICULTURE.

During 1932, our attention was first called to the trouble experienced with soy bean oil expressed from seed grown in North Carolina. Shortly after expression the clarified crude oil became turbid and gradually a sizable precipitate separated. Obviously, this precipitate had to be removed before the oil would be acceptable to the purchaser. On account of its gelatinous character the removal of the precipitate by filtration was extremely difficult. Also, after filtration the precipitate retained a large quantity of oil. It was found preferable, therefore, to hold the crude oil in settling tanks at the mill until the precipitate had settled, and then carefully withdraw as much as possible of the clarified oil. In so far as could be ascertained, practically no trouble of this character was experienced by the soy bean oil mills in the Middle West States.

To determine the composition of this precipitate, we examined a gallon sample of the clarified soy bean oil<sup>1</sup>, freshly expressed from a mixture of Mammoth Yellow, Tokio Green, Hollybrook, Mammoth Brown and Tar Heel beans. The Mammoth Yellow and Tokio Green varieties predominated. Several days after expression it was observed that the oil became turbid. After standing for several weeks a brownish-yellow gelatinous precipitate amounting to 2.1 per cent of the weight of the original oil had settled to the bottom of the bottle. At first, it was believed that the precipitate separating from the oil was a stearine because upon warming it dissolved in the oil. Upon cooling to room temperature the precipitate gradually reappeared. Consequently the oil was examined to see whether or not it contained an abnormal quantity of saturated acids. It was found to contain 11.12 per cent of saturated acids, which is within the

usual range, indicating that the precipitate in question was not stearine. A test for phosphorus indicated that it consisted largely of phosphatides.

The precipitate was collected by centrifuging the oil and transferred to a Buchner funnel fitted with a filter paper. After three days no more oil could be separated by filtration. The precipitate was then removed from the funnel to a flask and dissolved in a small quantity of chloroform, and about 20 volumes of acetone was added to the solution. The precipitated phosphatides were easily filtered on the Buchner funnel and washed with acetone until all the oil was removed. After the phosphatides were dried for several weeks over calcium chloride in a desiccator they were readily reduced to a powder. Analysis showed that the substance contained 3.9 per cent of volatile matter, 1.15 per cent of nitrogen and 3.20 per cent of phosphorus (the average of four closely agreeing determinations). The phosphatides calculated in terms of lecithin, using the average figure for phosphorus, amounted to 81.6 per cent.

Tests made for sulphur and carbohydrates were both negative. The fact that the precipitated substances separated from the oil were completely soluble in chloroform precluded the presence of peptones and pentoses, as well as carbohydrates, such as were found in cottonseed oil settlings\*. After the oil from the precipitate had stood about eighteen months, analysis showed that it still contained 0.016 per cent of phosphorus, which is equivalent to 2.70 per cent of lecithin.

In attempting to discover the cause for the different behavior of the oils from soy beans grown in the Eastern and Middle States, samples of the more important varieties were examined with regard to their phosphatide content. After experimenting with the various

methods proposed for the extraction of phosphatides from seeds, it was found that a single treatment with boiling 95 per cent alcohol was satisfactory. The method used is as follows:

Accurately weigh one-gram portions of finely ground samples of beans. Spread each weighed portion in a thin layer on a 150-mm. filter paper (Reeve-Angel No. 211 or equivalent grade); fold paper at a point about one-quarter of the distance from each of two opposite sides over the sample to the center of the paper; wrap into a cylinder by coiling the paper from one of the unfolded sides and fold a second filter paper around this (to prevent the escape of fine meal), leaving one end open like an extraction thimble. Place a piece of absorbent cotton lint in the opening to facilitate the even distribution of the solvent, and transfer the cartridge or thimble to a Butt type extraction tube and connect it with a reflux condenser. Add 40 cc. of 95 per cent alcohol to a 150-cc. wide mouth extraction flask, connect it with the extraction tube and extract for four hours, preferably by heating the flask in a sand bath. The alcohol should drop from the condenser on the center of the cotton plug at the rate of about 150 drops per minute. After the extraction is completed, remove the flask, evaporate the alcohol as completely as possible on a steam bath and heat it in an oven at 100 to 105° for one hour. In addition to the phosphatides this residue contains some other alcohol extractable substances, including a portion of the oil.

Add 4 cc. of sulphuric acid and about 0.2 gram of potassium nitrate to the flask containing the residue. Hold the flask at about a 45° angle and rotate it over a small gas flame until the mixture thickens and chars. After it has stood for 5 or 10 minutes cautiously add 3 to 4 cc. of nitric acid, invert over the neck of the flask a porcelain crucible

<sup>1</sup>Sent by the Eastern Cotton Oil Company from their Elizabeth City, N. C., expeller mill.

\**Jr. Oil and Fat Ind.* 3, p. 352, 1926.