A New Solvent Extraction Process for Soybean Oil

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EFFICIENCY, safety, simplicity, and low cost of operation were goals in the development at Iowa State College of a new soybean oil extraction process designed specifically for small scale operations. When the work originated about 10 years ago, small plants were visualized as very desirable as part of the program to decentralize industry and to bring it to the rural middle west. Soybean oil meal, the main product of soybean processing from a tonnage standpoint, is a desirable item for the livestock feeder. It was believed that small plants strategically located could best serve agriculture since they would simplify and reduce costs of moving beans into the plants and meal back to the farms. It was believed that any lowering of processing costs which might accrue to the large plants because of their size would be offset by the lower freight costs on the beans and meal and by lower labor costs prevalent in smaller communities.

High efficiency in oil removal from soybeans may be attained by continuous solvent extraction methods. While "hexane" has been used mainly for solvent extraction of oil seeds for many years, several considerations indicated the desirability of non-flammable solvents. Safety construction and equipment for use with flammable solvents are expensive. Safety men in a small plant add heavily to overhead. Insurance—fire, explosion, and liability—is more expensive when flammable solvents are used. Labor for operation with flammable solvents should be more highly skilled and therefore more expensive. Plants using hazardous materials are limited in their location in respect to other occupancies.

The Solvent

Of the non-flammable solvents the chlorinated hydrocarbons appeared to offer the most promise. The characteristics of these were carefully studied, resulting in the adoption of trichloroethylene. Trichloroethylene offered certain advantages. It met the requirement of non-flammability, not forming flammable mixtures with air at ordinary temperatures and pressures. It was an excellent solvent for soybean oil. It was readily available, being produced in quantity for degreasing of metal parts. When properly inhibited, it was practically non-corrosive and could be used with ordinary construction materials. From the standpoint of removal from the oil and meal as well as recovery the characteristics of trichloroethylene were attractive. The boiling point of 86.7°C. (188.1°F.) was low enough without introducing problems of excessive volatility. Being a single chemical rather than a mixture, there were no heavy ends to be removed from either oil or meal. A heat of vaporization of 103.1 B.t.u. per pound indicated low steam and cooling water costs. A solubility of 0.11 parts per 100 in water insured low losses in solvent-water separation. A specific gravity of 1.464 at $\frac{20^\circ\text{C.}}{4}$ indicated ready separation from water with the water as the upper layer.

One disadvantage of trichloroethylene was the high cost. The initial cost of the solvent represents a capital investment but, unless excessive, has little effect on process costs. The cost of solvent lost per unit of beans processed however is a definite part of the processing cost, and it was early recognized that low solvent loss was one of the necessary goals in the new process. Any cost of solvent above that for a cheaper solvent would have to be balanced against such advantages as lower building and equipment cost, lower labor costs, and lower insurance.

A disadvantage more apparent than real is the toxicity of trichloroethylene. All volatile solvents when inhaled in any appreciable quantities produce abnormal physiological effects. However in a properly operated extraction system, the amount of trichloroethylene getting into the air would be negligible. According to Lehman and Flury (1), the highest safe concentration of trichloroethylene in a work room is 190 parts per million by volume. As much as 13,500 parts are required to be serious for a 30-minute exposure. The immediate effect of high concentrations of trichloroethylene in the air is to produce symptoms similar to intoxication from alcohol. The patient recovers rapidly in fresh air. The toxic effects of trichloroethylene are apparently not cumulative.

Pilot Plant Studies

Continuous solvent extraction involves the movement of both the flaked soybeans and solvent continuously through an extraction chamber. For the most efficient extraction the movement of the solvent should be counter-current to that of the beans. In the early pilot plant studies at Iowa State College the beans were moved through the extractor by means of screw conveyors. This extractor consisted of two connected tubes; one at an angle of about 16° from the horizontal, the other extending upward from the lower end of the first tube at an angle of 14° from the vertical towards the first tube. The flakes entered the

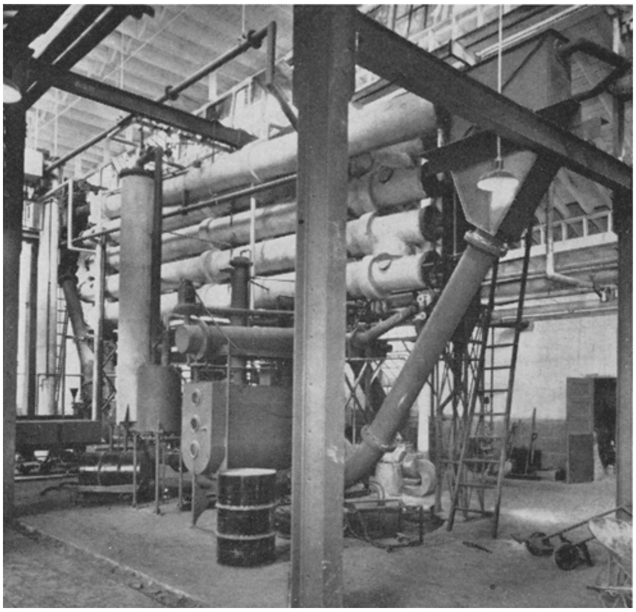
FIG. 1. Twenty-five ton extraction plant.

upper end of the first tube and were carried down through the solvent to the second tube. In the second tube they were carried upward out of the solvent and into a drier. The solvent entered the second tube moving downward to the first tube and flowing upward to a point just below the entering point of the flakes. Here the miscella flowed out of the extractor to the stripper. While this extractor gave good results, it was decided that it had certain inherent disadvantages due to the use of the screws as conveyors. For example, since the screws did not run full, there was by-passing of solvent, thus reducing the extraction efficiency. Screws also tend to produce some fines due to breakage of the flakes.

Following the study of the screw type extractor, a pilot plant extractor with a capacity of 1.5 tons per 24 hours, using a continuous chain conveyor for moving the flakes, was built and studied. The chain travelled through a casing which formed a rectangular loop about 12 feet long and 6 feet high, set at a slight angle with the horizontal. The bottom part of this casing formed the extractor while the upper horizontal run was steam jacketed to act as a drier. The extractor was completely filled with the flaked beans, which were moved along at a rate of about one foot a minute by the chain without agitation. Because of the skeleton-like nature of the conveying links very little obstruction was offered to the solvent flow. The uniform counter-current flow of solvent through the uniform mass of flakes resulted in excellent extraction. For example, the flakes were reduced to 1% or less oil content in 17 minutes of extraction, producing a miscella containing 20% oil. Since there was no agitation in the extractor, very little breaking of the flakes occurred, resulting in a minimum of fines to be removed from the miscella.

Various methods of recovering the solvent from the oil were studied, including both falling and rising film evaporators. One of the former consisted of two vertical steam-heated tube bundles, one above the other and connected by a short spool from which the vapor was removed. Miscella containing approximately 20% oil was fed in at the top and stripping steam blown in at the bottom through a pan of oil acting as a seal. Oil was drawn off continuously through the bottom pan. While oil substantially free from solvent was obtained, this evaporator was not considered satisfactory for commercial use. A rising film evaporator consisting of a miscella tank, vertical steam jacketed tubes, and a flash chamber proved excellent for concentrating the miscella from the extractor to approximately 80% oil. After filtering, the concentrated miscella was stripped free of solvent in a packed column, using Berl saddles. This produced a satisfactory oil.

Commercial Plants

A plant having a capacity of 10 tons per day, using this process, was designed for J. Roach Sons inc., Plainfield, Iowa, where it was put into operation with the technical assistance of the Iowa Engineering Experiment Station staff. After the usual difficulties common to new processes were ironed out, it was put into successful commercial operation. Patent rights on the process have been assigned by the Station to the Iowa State College Research Foundation inc. A license has been issued by the Foundation for the manufacture and sale of the equipment for the process. The equipment at present is being produced in 25-ton units (Figs. 1 and 2).

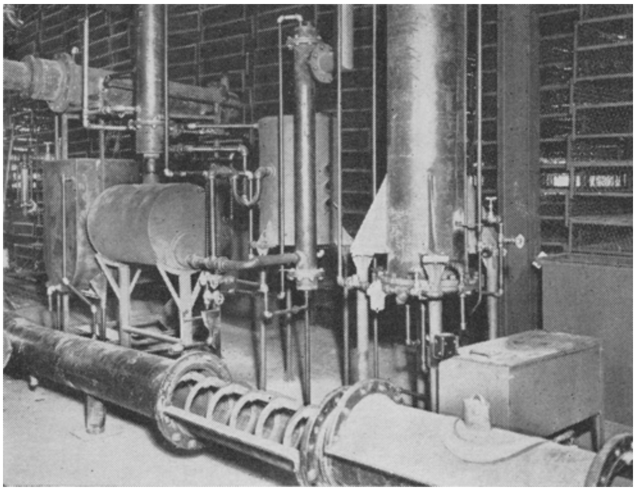

FIG. 2. Twenty-five ton plant. Extractor chain in foreground.

A flow sheet of the commercial process is shown in Fig. 3. All of the equipment is normally installed on one floor of a single building. The extractor is essentially a continuous loop of circular tubing or pipe 12 inches in diameter as shown in Fig. 3. A continuous conveyor chain having circular open top links (Fig. 2) travels in the tube and is driven by means of a sprocket.

Operation of the plant is as follows: The beans from storage pass over a rotary screen and a permanent type magnetic separator to remove foreign matter. They are then weighed by automatic dump scales dropping into a small bin below. From the bin they are fed into a two-pair high set of cracking rolls. Several types of feeders were tried for this purpose, but an electrically operated vibrating feeder gave the best control. The cracked beans are then passed through a steam-jacketed tempering tube, where they heated to about 150°F. From the tempering tube the hot cracked beans drop into flaking rolls. A single pair of smooth rolls produce flakes about 0.010 of an inch thick. The flakes are conveyed by a screw conveyor to a drag type of vertical elevator, where they are elevated to the top of the extractor. This type of elevator is used in preference to a bucket elevator since it handles the flakes with little breakage. The flakes enter the extractor just to the right of the drive sprocket, feeding in by gravity. A sufficient head of flakes is maintained in the feed spout above the extractor to prevent the loss of any solvent vapor. Experimental work has shown that the unextracted flakes absorb the trichloroethylene vapors but allow any air to pass through. The proper flake level is secured by maintaining a slight overflow which accumulates in a bin from which it may be fed back into the feed conveyor when necessary. An "electric eye" actuating an electric light or bell may be installed in the feed spout to give warning if the level of flakes drops too low.

The solvent is pumped through a rotameter into the extractor tube at the opposite end from the flake entrance. The miscella flows out through slotted screens in the side of the extractor tube below the point where the flakes enter. The flakes are carried down through the solvent and up out of it into the upper steam-jacketed preheating section. The

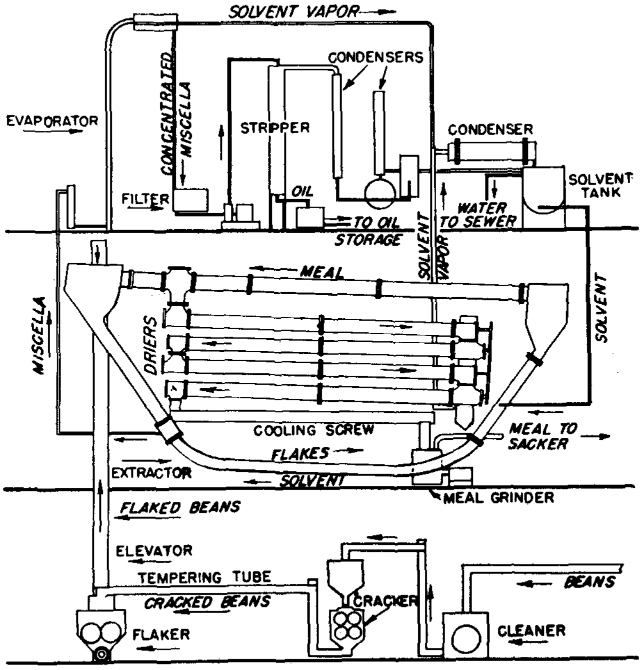

FIG. 3. Flow sheet of extraction process.

hot solvent-saturated extracted flakes or meal drop into the upper drier tube where volatilization of the solvent begins. The hot chain passes down into the extractor preheating the incoming flakes and the outgoing miscella. Proper heating of the flakes prevents condensation of moisture on their surface, which might result in clogging of the miscella screens. Heating the miscella at this point assists extraction, and since it lowers the viscosity, it also insures better flow of the miscella out through the screens.

The regular drier sections are steam-jacketed tubes, through which the meal is carried by ribbon-type screws equipped with special agitators. The solvent and water vapor are taken off at one end of the drier, passing under a water spray to remove any meal dust and to a water-cooled condenser. The condenser is set below the level of the lower drier to facilitate the flow of the heavy solvent vapors into it. The condenser is vented into the incoming flakes above the extractor to allow the non-condensable gases to escape to the atmosphere without loss of the accompanying solvent vapor.

The condensed solvent and water run into a tank where the solvent separates as the lower layer. The solvent is pumped from the bottom tank back into the extractor. A layer of water is maintained over the solvent to act as a seal to prevent loss by volatilization. The excess water overflows to the sewer.

The meal leaves the lower drier section through a special vapor seal. The plug of meal which forms the seal is being continuously formed of the discharging meal at the drier end and continuously breaking up as it emerges to drop into the moistening and cooling trough. Here water is sprayed on to adjust the moisture content. From here it drops into a hammer mill, where it is ground to proper size to meet trade requirements. From the mill the meal goes directly to the bagging bin for bagging.

The miscella from the extractor contains about 20% oil and 80% solvent by weight. It passes into a climbing film type of evaporator consisting of a series of vertical steam jacketed tubes discharging into a rectangular flash chamber. The miscella from the flash chamber contains about 80% oil and 20% solvent. It now passes down through a leaf type of filter, where the fine meal is removed. Since there is practically no agitation to break the flakes in the extractor, the percentage of fines produced is small—about one-half pound per ton of beans processed.

The filtered concentrated miscella passes through a preheater to a packed column stripper in which the remainder of the solvent is removed. This stripper consists of a vertical steam jacketed tube filled with Berl saddles over which the miscella film moves downward. Steam blown in at the bottom strips the solvent from the oil. The solvent and water vapor are condensed in a double condenser system, which not only condenses the two vapors but breaks up any emulsion which may form as the result of colloidal oil droplets carrying over with the vapor. The solvent goes to the solvent tank for reuse and the water to the sewer. The oil collects in a small tank below the stripper, from which it is pumped to an outside storage tank.

The operation and control of the plant are simple. The rate of feed of the beans is set by the feeder at the cracking rolls so as to maintain a slight overflow of flakes at the extractor. The excess flakes accumulate in a small bin, from which they are fed back by gravity into the horizontal screw flake conveyor. They may be used to compensate for the lag between the cracker and the extractor after the feed at the cracker has been increased, or they may be fed into the extractor with the feed cut back for that purpose. The flake thickness is checked by micrometer calipers and the rolls adjusted accordingly.

The solvent flow is adjusted by a needle valve or by a variable speed drive on the pump and metered by the rotameter. The proper rate of solvent flow for a given rate of processing is learned by experience and checked by determining the concentration of oil in the miscella from the specific gravity. When operating with flakes not over 0.010 inch in thickness and a miscella containing not over 20% by weight of the oil, the meal should contain less than one per cent oil. The concentration of the miscella going from the evaporator to the stripper may also be checked from the specific gravity. The presence of solvent in the oil may be determined by the plant operator by a simple colorimetric test (2), using pyridine and sodium hydroxide as reagents. This method as originally developed would determine amounts of trichloroethylene in soybean oil as low as 0.02%. A modification of this method which may be used to detect amounts as low as 10 parts per million (0.001%) is now in use.

The oil produced by this process is substantially free of solvent; grab samples taken during normal operation usually show about 10 to 15 parts per million. Tank car shipments show less than 50 parts per million. A typical sample showed free fatty acids 0.40%, moisture and volatile 0.20%, and refining loss 5.0%. No taste or odor of solvent is noticeable in the meal. The heat treatment of the meal in the driers may be varied, depending upon the use for the meal. Studies under way on the use of the meal as an adhesive indicate that it is suitable for this purpose. It has also been used experimentally in the production of plastics (3). While meal with a urease content suitable for feed purposes has been made directly in the driers in the pilot plant, it is considered more practical in the commercial installation to use a "toaster" (not shown in the photograph) recently designed. Feeding tests have shown the meal to be free of any toxic effects and to be entirely satisfactory as a protein feed.

Processing costs are low. Since the solvent is nonflammable, all of the processing equipment can be installed on one floor in a single building. As a result of the compact arrangement and automatic operation two men can operate a 25-ton plant including the sacking of the meal. The greatest power consumption is by the cracker and flaker. Total power consumption is about 32-kilowatt hours per ton of beans. The process steam required for the driers, evaporator, and stripper is about 1,300 pounds per ton. The water required will vary with the temperature but is about 200 cubic feet per ton. Solvent loss is low; about one-half gallon per ton of soybeans processed. Total processing costs are estimated at about 20 cents per bushel.

Extraction research is being continued by the Iowa Engineering Experiment Station along two broad lines. In the first series are studies on possible improvements of the present process. For example recent research indicates that it may be possible to reduce the amount of stripping steam required to remove the residual solvent from the oil to one-fourth that now used and still obtain the same high grade oil. In the second series are studies on the application of the process to the extraction of other oil-bearing materials than soybeans, such as corn germs, wheat germs, flax seed, and cotton seed.

REFERENCES

1. Lehman, K. F., and Flury, F. Translated by E. King and H. F. Smyth Jr. "Toxicology and Hygiene of Industrial Solvents." Williams and Wilkins Co., 1943.

2. Arnold, L. K., and Hollowell, A. G. "The Determination of Small Amounts of Trichloroethylene." Iowa Academy of Science *54*, 181-183 (1947).

3. Arnold, L. K., and Quackenbush, A. D. Jr. "Plastics from Soybean Meal and Furfural." Iowa Academy of Science *47*, 231-234 (1940).

Polyunsaturated Fatty Acid Retarders in the Emulsion Polymerization of GR-S Synthetic Rubber

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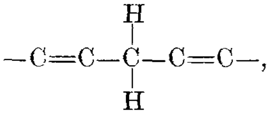
THE importance of scientific research in retaining existing markets for fats and oils, while at the same time extending their uses to new fields, has been well illustrated by the progress made during prosecution of the government's synthetic rubber program. At the beginning of the program tallow soaps were adopted as emulsifiers in the emulsion polymerization process for manufacture of butadiene-styrene synthetic rubber, GR-S. The standard recipe called for 5 parts of soap per 100 parts of monomers, and with the eventual need for at least 800,000 tons of rubber annually, the quantity of suitable soap required would exceed 100 million pounds annually. The early soap specifications included use of relatively high grade tallow, with maximum iodine number of 52, a difference between iodine number and thiocyanogen number not to exceed 6, and a negative test for insoluble bromides.

Two serious difficulties involving emulsifiers arose early in the program. Erratic rates of polymerization were observed with different batches of soap which met the specifications in use then. These variations prevented establishment of a uniform cycle of plant operations. The second difficulty was a critical shortage of high grade tallow. Increasing diversion of edible grade tallow became necessary as production of rubber increased.

To solve these problems the Rubber Reserve Company in 1943 initiated a soap development program for government synthetic rubber. Some of the results of this program were reported before the American Oil Chemists' Society by Burke (7) and have appeared in various publications by Semon (16), Dunbrook (8), and Wilson and Pfau (20). Research at this Laboratory in which various rubber companies, soap manufacturers and universities have collaborated by contributing specialized techniques established the fact that polyunsaturated fatty acid components of the tallow soap emulsifiers acted as retarders in the emulsion polymerization; that variations in the content of polyunsaturated acids undetected by iodine value minus thiocyanogen value (I.V.—T.V.) differences resulted in variable conversion rates; and that selective hydrogenation of the fat stock was a successful remedy for the difficulties encountered.

Although many possible theories were advanced to explain the erratic behavior of the tallow soaps as emulsifiers, the supposition that varying amounts of unidentified "retarders" were present in the soaps seemed most plausible. From a consideration of the composition of tallow, it seemed probable that such retarders if present would be found in one or more of the following groups: a) naturally occurring antioxidants and other congeners, b) polyunsaturated acids present in tallow in small amounts, or, c) metallic impurities in the soap. Substantial basis existed for suspecting these substances of retarding action. For example, synthetic antioxidants exert a great retarding influence. Also diene structures of the type  $—C{=}C—\overset{\displaystyle H}{\underset{\displaystyle H}{C}}—C{=}C—$, found in polyunsaturated fatty acids and certain hydrocarbons were very damaging (9a) if present in significant amounts.

To discover whether any of the naturally occurring antioxidants affected the polymerization we ob-

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