reinforced cloth and on the outside of the spirals of the reinforced cloth. Thus in the former case the stream of liquid was in a relatively thick film; and because of this and its position inside the spiral the film presented a smaller surface to the stripping steam.

A comparison of column capacities when stripping to less than 0.02% trichloroethylene shows an increase from 45 pounds per hour of stripped oil, when using center feed with 1-inch Berl saddle packing, to 101 pounds per hour using the same packing but distributor feed. The substitution of the reinforced spiral weave metallic cloth spaced with ½-inch Berl saddles increased the column capacity to 221 pounds per hour and reduced injected steam requirements to 0.21 pounds of steam per pound of oil. This improved behavior was obtained in a column with a spaced cloth packing of only 78 inches in height compared with a Berl saddle height of 96 inches.

Summary

A study of the effect of liquid distribution on the performance of a packed column resulted in the development of a distributor head for feeding which not only improved the distribution but decreased the steam consumption by about 20% and increased the column capacity by about 30% when stripping soybean oil-trichloroethylene miscella from 20% to 0.02% residual solvent. Liquid distribution studies with rolled packing units composed of 10-mesh reinforced spiral weave stainless steel cloth with ½-inch Berl saddles as spacers between turns showed that these units maintained a very uniform distribution of liquid down the stripping column. A column 8 inches in diameter with packing units having a total height of 78 inches had a capacity, when operated with the distributor head on 20% soybean oil-trichloroethylene miscella, for stripping 229 pounds of soybean oil per hour to a residual solvent content of 0.02%, using 0.20 pound of steam per pound of oil.

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[Received August 2, 1951]

The Effect of Trichloroethylene in the Hydrogenation of Soybean and Cottonseed Oils

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DESPITE the widespread use of hydrocarbon solvents for the extraction of oil-seeds there has been an increasing desire on the part of many processors to change to an essentially non-inflammable and non-explosive solvent such as may be found among the chlorinated hydrocarbons. This is mainly in the interest of safety. One large British mill changed over to trichloroethylene during World War II because of the danger of bombing, and results were entirely satisfactory. In this country the use of trichloroethylene in oil-seed extractions has been limited to a few plants processing soybeans. Further development has probably been hampered by the comparatively high cost of the solvent and, at least in the case of cottonseed, by certain technological difficulties requiring considerable research.

In addition to the foregoing, there has been a certain amount of misapprehension with regard to trichloroethylene as a solvent. An example of this is the belief held in some quarters that it is impossible to hydrogenate trichloroethylene-extracted oils because of residual solvent not removed during processing. This subject has been investigated in our laboratory and pilot plant, and we are presenting our findings herewith.

Laboratory-scale Hydrogenation

In some early experiments cottonseed oils containing 0.0-0.1% trichloroethylene (extreme levels were deliberately selected for testing so as to show up any effects) were subjected to hydrogenation in a small glass laboratory reactor,[1] in which hydrogen at atmospheric pressure is blown through an oil-catalyst mixture agitated by a high speed stirrer and maintained at any desired temperature. In this and all subsequent hydrogenations a commercial, reduced nickel catalyst containing approximately 25% metallic nickel was used. The catalyst concentration employed in the present experiments was 0.12%, and the reaction temperature was maintained at 165°C. (329°F.). Oil samples were prepared by adding various amounts of trichloroethylene to refined and bleached oil, mechanically expressed. In each test 190 grams were hydrogenated. Results are shown in Figure 1.

It is seen that oils containing .007% trichloroethylene hydrogenated as well as the trichloroethylene-free oil used for a control. Also it is apparent that oils containing as much as 0.1% trichloroethylene still hydrogenated reasonably well. The explanation for this is probably that trichloroethylene is continuously distilled from the oil under the conditions employed in this type reactor. It evidently does not form an irreversible adsorption complex with the catalyst. Reasoning from this, one would expect that the effect of any concentration of trichloroethylene in oil would vary with the operating conditions employed in the hydrogenation and, at least theoretically, oil containing any quantity of trichloroethylene could be hydrogenated under suitable conditions.

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[1] Baker Instrument Company, Orange, N. J.

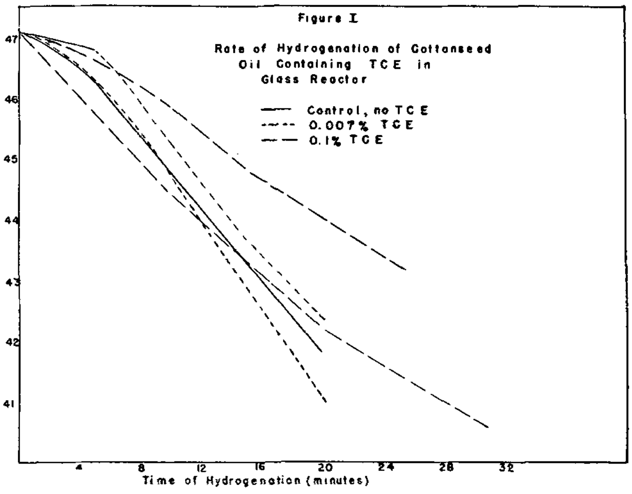

Figure I

Rate of Hydrogenation of Cottonseed Oil Containing TCE in Glass Reactor

Further laboratory experiments were conducted in a 3-liter metal converter (dead-end type) which, previous experience had shown, gives results comparable with those obtained in plant production. Varying percentages of trichloroethylene were added to refined and bleached, mechanically expressed, soybean oil, and the hydrogenations were carried out at 385°F., with 25 pounds of hydrogen pressure and 0.08% nickel catalyst. As routine practice the oils were heated, with vigorous agitation, to 385°F. under 25 inches of vacuum prior to the addition of the catalyst. During the course of the hydrogenation the reactor was evacuated at least once every 10 minutes.

It may be seen from the data in Figure 2 that in this dead-end type apparatus as little as 0.01% trichloroethylene exerted a marked inhibitory effect and 0.1% stopped hydrogenation entirely after a brief initial period. It was found with the oil containing 0.03% trichloroethylene that the addition of another 0.04% of catalyst greatly accelerated a lagging rate of hydrogenation.

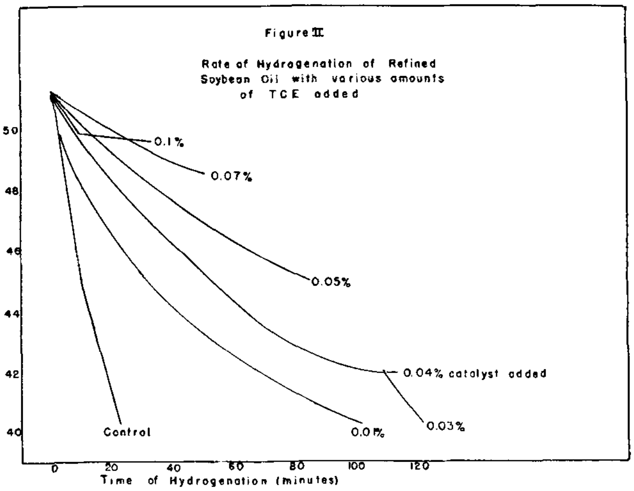

Figure II

Rate of Hydrogenation of Refined Soybean Oil with various amounts of TCE added

Somewhat similar results were obtained in the hydrogenation of refined and bleached, mechanically expressed, cottonseed oil to which trichloroethylene had been added (Figure 3). In this case less catalyst (0.05%) was used, and the batch was purged every three rather than every 10 minutes during the early stages of hydrogenation. Although 0.005% trichloroethylene was tolerated quite well by the cottonseed oil, 0.01% had a very serious retarding effect. It was found however that by using more catalyst (0.1%) oils containing 0.01% trichloroethylene hydrogenated much more rapidly. Also it was observed that the addition of the catalyst in two portions gave results superior to those obtained when the catalyst was all added at one time. Further reference will be made to this point later.

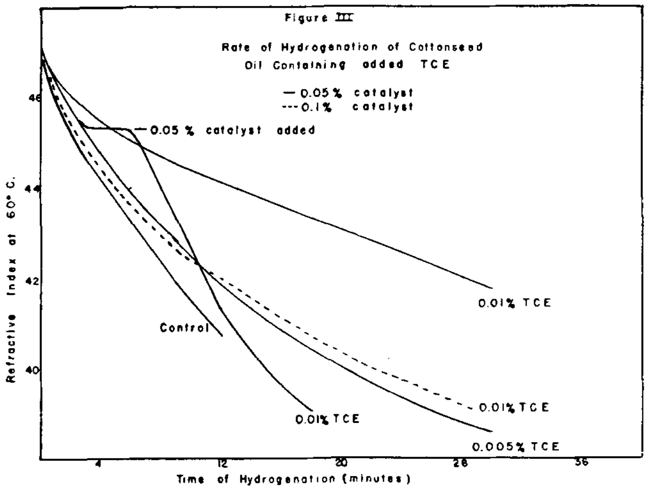

Figure III

Rate of Hydrogenation of Cottonseed Oil Containing added TCE

These data and the results obtained with the glass hydrogenator previously described indicated that it should be possible to hydrogenate cottonseed oil containing relatively large quantities of trichloroethylene in a dead-end type converter, provided proper conditions of hydrogenation were employed. For example, if an oil were known to contain excessive quantities of trichloroethylene, it should be helpful to hold the oil for some time under vacuum at hydrogenation temperature before adding the catalyst. That such is the case can be seen from the data in Figure 4. Holding the oil containing 0.1% trichloroethylene under vacuum at 385°F. apparently reduced the concentration to less than half that quantity, based on comparative rates of hydrogenation.

A series of experiments was conducted in order to compare the relative merits of the split catalyst addition technique with the improvement by prelim-

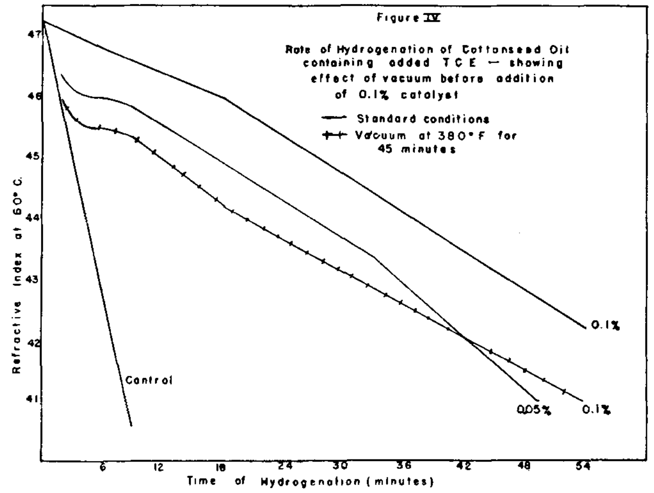

Figure IV

Rate of Hydrogenation of Cottonseed Oil containing added TCE — showing effect of vacuum before addition of 0.1% catalyst