Mixing Effects in Dead-End Hydrogenation

Hydrogenation of vegetable oils and animal fats involves not only several consecutive and simultaneous reactions but also physical steps of mass transfer to and from the solid catalyst surface. In addition to saturation of the double bond, geometrical and positional isomerization also take place. Selectivity and isomerization are affected by the operating variables, and their true effect can be evaluated only when mass transfer resistances are eliminated. Only four investigations are known (1,2,3,4) in which these resistances were substantially eliminated, and their conclusions should be important for evaluating the true effect of operating variables.

Most of the recent papers on hydrogenation have pointed out that agitation rates play an important role in determining whether or not mass transfer resistances have been eliminated. Unfortunately an indication of the rpm used is not quantitative information of the degree of turbulence achieved in the reaction mixture and of the possibility of absorbing hydrogen at the appropriate rate. The reported experimental data are characteristic of the particular piece of equipment in use and not of the chemical reaction per se.

Industrial hydrogenators are chiefly of the dead-end type where the gas is bubbled at the bottom of the apparatus. Hydrogen comes partly from the fraction of the bubbles which is absorbed and partly from the gas space. Agitation is usually provided by one or more turbines located on a central shaft. Often the design of the mixing system is based on the recommended characteristics for an open gas-liquid contractor, which indicate that, for the most favorable operation with one turbine, the liquid height inside the apparatus must be equivalent to one autoclave diameter and that the turbine be located at about one-third of the liquid level.

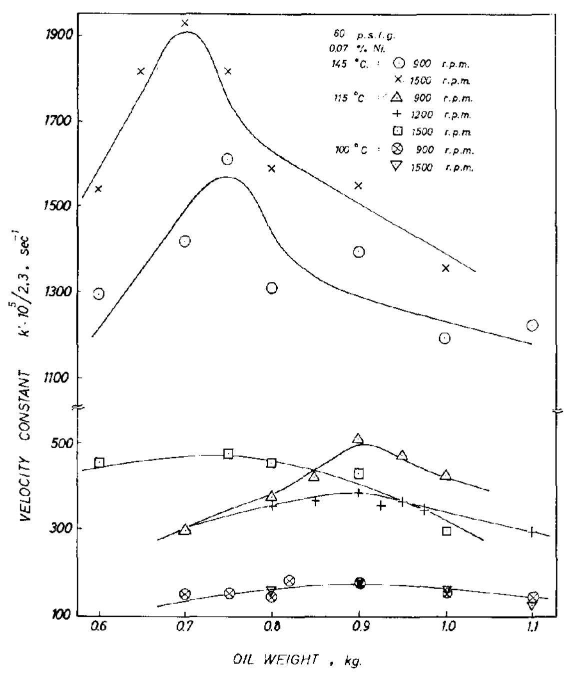

Fig. 1.

It is the purpose of this communication to show that, for dead-end equipment, these considerations are not valid and that higher rates of reaction can be attained when the impeller is located in the upper half of the liquid. This fact has been indicated qualitatively by Wisniak and Albright (4), who pointed out that it would seem advisable to locate the turbine at about two-thirds of the liquid level.

Hydrogenation runs were made in a dead-end batch autoclave, Model Magnedrive. (Autoclave Engineers Inc.). The equipment and its operation were similar to those used previously (4). Agitation was provided by a Dispersimax turbine, with a diameter 40% that of the autoclave, which was driven by a variable-speed motor.

In all of the runs, after an initial induction period, the data could be correlated by a pseudo-first-order reaction. For this reason all of the kinetic data were expressed as the over-all reaction rate constant, k′ sec-1.

Figure 1 clearly indicates that the rate of reaction goes through a maximum when the specific rate constant k′ is plotted against the oil weight which is being hydrogenated. From this figure it can be inferred that, for the same rpm and under conditions by which the process is controlled by chemical resistances, namely, 100C and 900 rpm, the effect of the oil level is smaller; the maximum reaction rate corresponds to a level equivalent to about 0.9–1.0 kg of oil or to the turbine in a position 50.5 to 57% that of the liquid level. Conditions that favor the chemical reaction, such as pressure and temperature increases, enhance the level effect by shifting the maximum reaction rate to higher levels. Thus, when the temperature is increased to 145C, the maximum rate corresponds to the turbine location at 75.5% of the oil level. Similar results were obtained when the pressure or catalyst concentration were increased.

The results obtained at 115C are surprising since they seem to indicate that higher rates can be attained at lower rpm. Although no exact answer can be given to this problem it must be pointed out that experimental evidence shows that considerable foaming occurs with Dispersimax mixers, so that good contact of the gas and the liquid is not realized. It may be that the combination of agitation rate and temperature produces a reaction mixture considerably different from that of a solution of hydrogen in oil.

The results described thus far demonstrate that it is possible to increase the rate of reaction by simply changing the position of the turbine with respect to the liquid surface. In addition, it has been shown that there exists a position for which the rate of hydrogenation is maximum; lower rates occur below or above this critical level. In all cases the optimum position of the turbine is at more than 50% of the liquid height, a figure that is radically different from that which is recommended for open systems in which a gas is bubbled through a liquid.

For hydrogenations that are to be performed under conditions similar to those used in this work, the agitation level must comply with two basic conditions. First, it must provide a translational motion sufficiently intense to create in every point of the liquid a velocity higher than that of sedimentation of the catalyst and originate a mixing action that will keep the mixture uniform. Second, a turbulence regime must be established that will provide appropriate mass transfer for the over-all chemical reaction. These two fundamental conditions affect to a different degree the various steps that constitute the process and are dependent upon the disposition of the equipment, its dimensions, the liquid level, and the rpm. The interrelations among these variables will determine the type of agitation obtained and how it will satisfy the process requirements.

For every set of conditions it was possible to demonstrate the existence of a maximum in the curves of liquid level vs. rate constant. Considering that this effect was observed for two different oils and that it has already been reported in a previous work with a third oil (4), it should be accepted and used for the proper design of hydrogenators.

With dead-end hydrogenation the need for agitation requires internal agitation of the liquid for an appropriate transport of reagents and products to and from the reaction zone; provision of an appropriate suspension of catalyst; and inclusion of hydrogen from the zones where it is available. Internal mixing, at a level above the optimum, will mean that the same power must be distributed in a larger volume, thus diminishing the intensity of agitation. If the liquid level is continuously decreased, agitation efficiency diminishes until the impeller is practically at the liquid surface and becomes inoperative. Similar arguments can be used with respect to the suspension of the catalyst.

It is inferred that a certain impeller position will provide the best conditions for gas-liquid contact and the improvement in the rate of solution of hydrogen. This can be seen upon inspection of the two ways in which hydrogen may enter the oil. First, as to the feed at the bottom of the autoclave, the emerging bubbles are carried away by the flow created by the turbine. They are finely subdivided with the corresponding increase in contact area and retention time. An impeller placed low within the liquid and/or at a high level will favor this manner of gas dissolution. Second, the space above the liquid is occupied by hydrogen that has travelled through the oil without being absorbed. When the impeller approaches the liquid surface, its capacity of hydrogen suction will vary according to the rpm, relative height, and geometrical dimensions.

If the process is controlled by the chemical resistance, the hydrogenation process requires a certain minimum residence time of the elements in order that it may take effect. The hydrogen fed by bubbling is enough, and a maximum is attained in which a low agitation is sufficient. The impeller is located at about 55% of the liquid height. Increasing the liquid level decreases the available hydrogen, and lowering the level augments the agitation per unit volume with a corresponding decrease in the reaction rate.

For fast reaction rates, that is, for conditions of low chemical resistance, there is a need for large amounts of hydrogen and the transport of the gas from above the liquid surface predominates. This causes a change of the relative position of the impeller toward the liquid surface. The turbine is now located at about 75% of the liquid height.

It is also assumed that, at low rpm (900) and intermediate liquid levels (0.9 kg), translational and distributional movements are favored above those of local turbulence. The bubbling hydrogen is absorbed with ease while the upper volume does not contribute in a significant degree to the hydrogen availability.

The experimental results indicate that at 145C the large inflection in the curves of oil weight vs. k′ occurs for turbine positions between 65 to 90% of the liquid height. These data indicate that, by comparison with general practice, the turbine is located near the surface.

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A Compact, Convenient Stand and Heater for Soxhlet Extractors

FAT EXTRACTIONS have been carried out in this laboratory by using Soxhlet extractors. Use was made of a hot plate with a support rod, but this was considered unsatisfactory. Consideration was given to the purchase of a six-unit commercial heater, but this type was bulky and heavy. Since we were not using the extractors routinely, it was necessary that the heater be readily movable to and from valuable bench space. Also, the considerable problem of the numerous condenser hoses would still be present, plus the need for the necessary plumbing connections. After consideration of the above problems and our particular needs, the device described herein was designed and constructed. It has been in use in our laboratory for more than a year to our complete satisfaction.

To eliminate the usual jumble of condenser hoses, all the water connections are in the center of the unit, permitting completely free access to each extractor. Cold water is delivered through the center tube, which also serves as the support rod, and the condenser drains are individual tubes ringed around it.

Throughout this description, reference is made to Figure 1 as each part is considered. For clarity, only the basic parts are shown in the drawing. The description for each, plus the photographs of the complete unit in Figure 2, should be sufficient to permit construction.

Item A. This is a laboratory triangular base standard with 6½-in. legs, which has been drilled and