tration no further nucleation or crystal growth occurs, the time required to approach the saturation concentration from a supersaturated solution in the liquid oil determines the time for crystal growth.

Bailey's Industrial Oils and Fat Products recommends a crystal growth time of 48-60 hr. In certain well designed systems, the crystal growth time for the same oil can be reduced to 6-10 hr.

From this discussion it is clear that, for the design of a winterization process, the rate of cooling of the oil, the temperature of crystallization and the agitation of the oil are crucial and play a significant role both in separation of solid fats as distinct crystals and in helping separate them from the liquid oil. Thus, under the general objective of obtaining data to develop a more efficient winterization process, the main emphasis should be in studying the effect of these variables on the performance of the winterization process.

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[Received April 10, 1980]

Hydrogenation of Oils and Fats[1]

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ABSTRACT

Hydrogenation is a complex 3-phase reaction, and the end result depends largely on transport and chemical resistances. Control of these factors is of critical importance to a predictable end product and this control is possible if a fundamental understanding of the process is achieved. The basic transport steps involved to bring the hydrogen and oil together in the presence of catalyst and the effect of different process variables on the course of the chemical reaction will be the major subjects of discussion in this presentation.

NOMENCLATURE

a = Bubble area/unit volume of reactor, cm^2/cm^3; A_P = particle surface/unit volume of catalyst, cm^2/cm^3; C^* = saturation concentration of hydrogen in oil, mol/l; C_L = hydrogen concentration in oil at any time, mol/l; C_P = hydrogen concentration on catalyst surface, mol/l; C_r = reactant concentration on catalyst; mol/l; C_{r_1} = concentration of fatty acids with one double bond, mol/l; $C_{r>_1}$ = concentration of fatty acids with more than one double bond, mol/l; H = Henry's law constant, mol/1-atm.; k_L = mass transfer coefficient, cm/min; k_P = oil-catalyst mass transfer coefficient, cm/min; k_r = kinetic rate constant, 1-cm/mol/min; k'_r = pseudo kinetic rate constant; k'_{r_1} = pseudo rate constant for fatty acids containing one double bond; $k'_{r>_1}$ = pseudo rate constant for fatty acids containing more than one double bond; P_{H_2} = hydrogen partial pressure, atm.; R = reaction rate, mol/min-liter; S_p = particle active surface/unit volume of catalyst, cm^2/cm^3.

INTRODUCTION

Hydrogenation is a unit process whereby hydrogen is added directly to unsaturated oils in order to increase their stability to oxidative changes (rancidity). Also, it is widely used to convert liquid oils to semisolid fats for their use in margarines and shortenings. Hydrogenation processes in combination with interesterification and/or fractionation processes are used to make confectionery fats. In today's world of increasing population and limited resources, hydrogenation processes play a vital role in providing food products both in quality and quantity. Soybean oil, e.g., which is inherently vulnerable to oxidative changes, can attain a desired stability by partial hydrogenation.

In hydrogenation, edible oil is reacted with hydrogen gas in the presence of an active metal catalyst. The hydrogenation reaction is exothermic with heat of reaction of ca. 25 Kcal/mol/double bond of hydrogenation, in the usual range of commercial conditions of 130-200 C and 5-20 psig pressure (1). The degree of hydrogenation of an oil is directly related to iodine value (IV), the addition of 1 mol of hydrogen corresponding to the absorption of 1 mol of iodine. It can be calculated that ca. 14.1 cu ft (at 0 C and 760 mm Hg) of hydrogen is required to reduce the IV of 1000 lb of oil by 1 unit. The heat generated during this reduction (by 1 unit IV) is enough to raise the oil temperature by 1.6-1.7 C. Commercially, hydrogenation reaction is conducted in stirred batch reactors, although some installations use continuous processes as well. After the reaction has occurred to a desired extent, the metal catalyst is separated from the oil by filtration. At some installations, oil is posttreated to reduce the residual metal content in oil to <0.5 ppm.

GENERAL NATURE OF THE REACTION

Oils or fats are triglycerides of fatty acids; when they are hydrogenated, the hydrogenation process reduces the number of double bonds on the various unsaturated fatty acid molecules. Also, hydrogenation alters the location of double bonds (positional isomerization) by migration along the fatty acid chain (2-4) and transforms some of the naturally occurring *cis* bonds into *trans* (geometric isomerization) configuration (5). All these changes alter the physical and chemical properties of the fat.

Since fats and oils contain a spectrum of fatty acids with different numbers of double bonds, for a given set of reaction conditions, they react differently. This preferential nature of reaction for one kind of fatty acid group over the

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[1] Presented at the AOCS short course, April, 1980.

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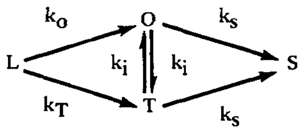
other is, in general, expressed as "selectivity" of the reaction. Classically, the term selectivity for hydrogenation of fats and oils is defined as the preferential hydrogenation of polyunsaturated acid groups such as linoleic and linolenic acids, as compared to monounsaturated groups such as oleic acid. Numerically, it is expressed as the ratio of the pseudo-first-order rate constants of polyenoic and monoenoic fatty acid hydrogenation (1,6). A high magnitude of selectivity indicates that the formation of saturated fatty acids will not start until most of the polyunsaturated fatty acids have hydrogenated to monoenoic stage. With the increasing competition of the hydrogenated products in the market place, a need for precision in the physical and chemical properties of the oils was realized. Thus, several other definitions of selectivity have emerged (6). One selectivity concept describes the preferential hydrogenation of fatty acids with more than 2 double bonds, leaving fatty acids with 2 or one double bond unattacked. This concept is of special importance with oils such as soybean which have higher contents of linolenic acid. To quantify the extent of geometric isomerization occurring during a partial hydrogenation, a further selectivity concept, specific isomerization, as the number of *trans*-double bonds formed per double bond eliminated by hydrogenation, has been introduced (3,7-9).

MECHANISM OF HYDROGENATION

Several mechanisms of hydrogenation reaction of edible oils have been proposed by different investigators (9-14). In general, it is believed that the sequence of reactions occurring during the hydrogenation reaction may be represented by the following scheme (14):



$$L \xrightarrow{k_T + k_o} (O + T) \xrightarrow{k_s} S,$$

where: L = polyunsaturated fatty acid, linolenic or linoleic; O = monounsaturated fatty acid, oleic (*cis*); T = monounsaturated fatty acid, oleic (*trans*); S = saturated fatty acid, stearic; k_o, k_T, k_s, etc. = rate constants for different reactions.

The hydrogenation reaction is believed to occur via formation of intermediate compounds of adsorbed hydrogen and unsaturated molecule on the catalyst (15). The formation of the intermediate state, commonly referred to as half hydrogenated state, is governed by the Horiuti and Polanyi mechanism (16). If there is plenty of active hydrogen available on the catalyst, then the half hydrogenated state takes a second hydrogen atom to complete hydrogenation. In hydrogen deficient conditions, it gives back the hydrogen atom it had taken. In doing so, the possibility occurs for the isomerization reaction which results in formation of *trans* isomers. Also, under low hydrogen concentration situations, the residence time of the molecule on the catalyst surface will increase. The adsorbed or half hydrogenated molecule may undergo a large number of alternating steps of taking and giving up hydrogen. As a result, the double bond may wander far from its original position resulting in the formation of positional isomers (6).

Since different unsaturated fatty acids have different reactivities, they tend to compete for the active sites on the catalyst (17); the most unsaturated is most preferably adsorbed and reacted. This is the reason that different fatty acids react at different rates.

TRANSPORT MECHANISM IN A HYDROGENATED REACTOR

We shall consider the case of hydrogenation of soybean oil. Because the reaction might be conducted in an autoclave, the hydrogen is dispersed throughout the liquid oil within which, say, a nickel catalyst is suspended. Vigorous agitation, induced via gas flow and mechanical means, assures fairly uniform composition and temperature throughout the geometric confines of the autoclave at any time during the reaction cycle. The transport-reaction event for this case may be represented by the following steps: (a) hydrogen from the gas phase dissolves in the oil; (b) dissolved hydrogen in the gas-liquid interface diffuses to the bulk liquid; (c) dissolved hydrogen from the bulk liquid diffuses to the catalyst surface and to the interstices of the catalyst; (d) diffused hydrogen to the catalyst surface gets adsorbed on the surface; (e) oil from the bulk liquid diffuses to the catalyst particle; (f) diffused oil to the particle adsorbs on the surface; (g) adsorbed oil and adsorbed hydrogen react to form the saturated product; (h) saturated products diffuse back from the surface of catalyst to the bulk.

These 8 steps occur in order to bring about a hydrogenation reaction. One or more steps can be rate-controlling. Except for the reaction and adsorption steps, all other steps are physical and can be varied by changing the physical conditions of the reaction. Since, in the oil hydrogenation, the quantity of unsaturated fatty acids is very high in contact with the catalyst, it is usually found that the reaction is hydrogen-diffusion-controlled (11). Thus in order to have a simple transport model for a hydrogenation reaction, the following steps are considered:

(i) Transfer of hydrogen from gas phase to the liquid phase

$$R = k_L a\,(C^* - C_L) \qquad [I]$$

(ii) Diffusion of hydrogen dissolved in the liquid to the catalyst surface

$$R = k_p A_p\,(C_L - C_P) \qquad [II]$$

(iii) Reaction rate at the surface (assuming first order reaction)

$$R = k_r S_p\,(C_P C_r) \qquad [III]$$

At equilibrium all these rates must be equal, therefore:

$$k_L a\,(C^* - C_L) = \dot{k}_p a_p\,(C_L - C_P) = k_r S_p\,(C_P C_r) \qquad [IV]$$

Furthermore,

$$\text{Rate} = \frac{\Sigma \text{ driving forces}}{\Sigma \text{ resistances}}$$

Therefore from I to III,

$$R = \frac{C^* - C_L + C_L - C_P + C_P}{\frac{1}{k_L a} + \frac{1}{k_p A_p} + \frac{1}{k_r S_p C_r C}} \qquad [V]$$

or,

$$R = \frac{C^*}{\frac{1}{k_L a} + \frac{1}{k_p A_p} + \frac{1}{k_r S_p C_r}} \qquad [VI]$$

If we call the overall resistance as:

$$\frac{1}{K} = \frac{1}{k_L a} + \frac{1}{k_p A_p} + \frac{1}{k_r S_p C_r} \qquad [VII]$$

Then Equation VI becomes

$$R = KC^* \qquad [VIII]$$

Note that the rate of reaction is always proportional to the hydrogen concentration at the gas-liquid interface.

By Henry's law,

$$C^* = H P_{H_2} \qquad [IX]$$

Substituting IX in VIII:

$$R = KH P_{H_2} = K' P_{H_2} \qquad [X]$$

If the reaction is conducted under such conditions that the gas-liquid and liquid-solid mass transfer resistances are low, i.e., k_La and k_pa_p are high, then Equation VI reduces to the intrinsic chemical rate equation as:

$$\begin{aligned} R &= k_r S_p C_r C^* \qquad [XI] \\ &= k_r S_p H C_r P_{H_2} \\ &= k'_r C_r P_{H_2} \end{aligned}$$

On the other hand, if the reaction conditions are such that the major contributor to the resistance is the gas-liquid mass transfer, then the rate equation becomes:

$$\begin{aligned} R &= k_L a C^* \qquad [XII] \\ &= k_L a H P_{H_2} \\ &= k' P_{H_2} \end{aligned}$$

Using Equation X, a more general equation for selectivity can be derived. Selectivity by definition was the ratio of the pseudo-first-order rate constants, mathematically,

$$S = \frac{K'_{>1}}{K'_1} = \frac{1/K'_1}{1/K'_{>1}} = \frac{\dfrac{1}{k_La} + \dfrac{1}{k_pa_p} + \dfrac{1}{k_r S_p C_{r_1}}}{\dfrac{1}{k_La} + \dfrac{1}{k_pa_p} + \dfrac{1}{k_{r>1} S_p C_{r>1}}} \qquad [XIII]$$

The variation in the operating conditions will vary both the mass transfer and kinetic resistances and hence it becomes very difficult to predict the effects of process variables on the selectivity of reaction. If the hydrogenation reaction was kinetically controlled, then selectivity will be given by

$$S = \frac{K'_{>1}}{K'_1} = \frac{k_{r>1}\, C_{r>1}}{k_{r_1}\, C_{r_1}}$$

On the other hand, if the rate of reaction was gas-liquid mass transfer controlled, as may be the case in several commercial units, the selectivity will be unity.

Since most of the commercial processes operate in the intermediate zone where the total selectivity is partly contributed both by the mass transfer and kinetic resistances, the effect of process variables on the selectivity become hard to predict.

EFFECT OF PROCESS VARIABLES ON HYDROGENATION REACTION

It is evident from the discussion just given that the effect of process variables on the hydrogenation reaction cannot be determined unless transport resistances are minimized. Except for a few studies in the literature (6,9,10) virtually all other hydrogenation studies were made under unknown (probably dominant) mass transfer effects. Thus, making any conclusions from the published work could be a futile effort.

In general, the effect of process variables for commercial hydrogenations will be determined by how they affect the rate of hydrogen transfer and rate of hydrogen consumption. These effects are summarized in Table I.

COMMERCIAL HYDROGENATION PRACTICES

For most of the commercial hydrogenations of edible oils, the oil most be of a certain quality in order to hydrogenate effectively. In general, the requirements are that the oil contain $<0.1\%$ free fatty acids, <1.5 ppm soap and $<0.1\%$ moisture. In addition to this it should have a low color ($<6R$) and peroxide value (<10 meq/kg) because polar pigments and oxidized ions can act as catalyst poisons. The hydrogen gas used for hydrogenation preferably should be 99+% pure. The gaseous impurities, especially the sulfur compounds (e.g., H_2S, CS_2 and SO_2) are very injurious to the catalyst. Impurities such as CO, CO_2, CH_4 and N_2 don't affect the catalyst activity.

COMMERCIAL CATALYSTS

The most widely used commercial catalyst for edible oil hydrogenation is active nickel supported on an inert substance (16,19). Other less commonly used metal catalysts are Cu-Cr (20,21), and precious metals like Pt and Pd (22,23). In general, the particle size of catalysts varies between 1-50 μm and major concentration is in the 1-10 μM size range. On the average, the catalyst porosity is ca. 0.75. The activity of a catalyst depends on the number of active sites available on it for hydrogenation. These active sites may be located on the surface of the catalyst or deep inside the pores. The typical pore length and pore diameters are ca. 1.66×10^{-6} m and 1.2×10^{-7} m, respectively (14). It is thus evident that the transportation of the unsaturated fat and hydrogen will not be as free to the active sites in the pores as it is to the outer catalyst surface. Very little is known about the influence of the pore diffusion in fat hydrogenations. Coenen et al. (24), however, have clearly demonstrated experimentally that the pore dimensions of catalyst influence the selectivity of fat hydrogenation.

The application of Pd and Pt catalysts is not very popular in the edible oil industry. These catalysts are exceed-

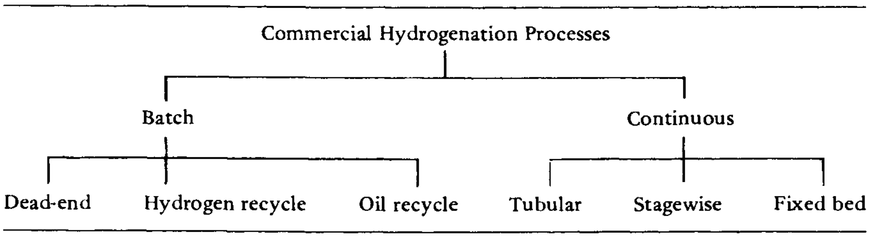
TABLE I

Effect of Process Variables on Hydrogenation

Process variable	Effect on rate of hydrogen		Effect on selectivity	
	Transfer	Consumption	S	*Trans* isomer formation
Agitation	+	0	-	-
Design	-/+	0	+/-	+/-
Pressure	+	+	-	-
Temperature	$+^1$	+	+	+
Amount of catalyst	0	$+^2$	+	+
Type of catalyst (nature)	0	+/-	-/+	-/+

+ = Increases; - = decreases; 0 = unaffected; 1 = slight increase; 2 = up to a certain limit.

TABLE II



ingly active, and usually form more *trans*-isomers than nickel catalysts. The hydrogenation reaction with these catalysts can be carried out at moderate temperature and low pressure conditions. The hydrogenated product is totally free of these precious metals because neither colloidal metal nor metal soaps form during the reaction (22,23).

Copper-chromite catalysts (20,21) were recently introduced for selective hydrogenation of linolenic acid to linoleic acid in soybean oil. The selectivity offered by these catalysts is excellent but activity is not very good.

COMMERCIAL HYDROGENATORS

An excellent review of commercial hydrogenation processes was recently published by Hastert (25). In general, commercial processes can be classified as shown in Table II.

BATCH HYDROGENATORS

Batch hydrogenations are most commonly used in the edible oil industry primarily because of their simplicity and flexibility for use with different oils. The most commonly used reactors are known as dead-end and hydrogen recirculation types. Recently an oil circulation process using a Ventury nozzle has been introduced (26).

In a dead-end hydrogenator, hydrogen gas is bubbled in a well mixed hot oil and catalyst dispersion. A part of the gas is dissolved and reacted with the oil. The remainder is collected in the headspace of the converter and reacts with the oil by transferring to it from the turbulent oil surface. In the hydrogen recirculation system, a fairly large headspace is provided because hydrogen gas is recirculated by a compressor. The gas enters the reactor through a sparging ring located near the bottom of the reactor. A small portion of the gas is absorbed by the oil and is reacted; the remainder provides agitation as it bubbles upward.

From the operating standpoint, the 2 reactors do not differ very much. In general, the dead-end type is preferred by many processors because it requires less energy, offers more versatility of the type of raw material handled and finished product formed, requires less capital and operating cost and is safer than the hydrogenation recirculation system.

CONTINUOUS HYDROGENATION

The continuous processes have been around for more than 3 decades. Their commercial usage is very limited because continuous flow patterns with minimum backmixing are not easily achieved. Because of this, the continuous processes give lower reaction selectivities. Other reasons for the unpopularity of continuous processes were a large variety of oils handled, small quantities of various oils involved and lack of end-point control. All these factors made the batch process more attractive. With the recent emergence of soybean oil as the single most used oil, interest in continuous processes has been regenerated.

Several continuous hydrogenation processes are available in the patent literature (27,28,29). In some, partial success has been achieved in eliminating backmixing of oil (30). In others, the mass transfer resistances have been minimized (29). It is interesting to note that the reaction mechanism followed by hydrogenation in certain systems is entirely different from that observed in batch processes (29).

One of the simplest concepts in continuous reactors is the tubular reactor, which provides sufficient time for the reactants to result in the desired degree of conversion. A lab scale reactor using this concept has been used by the researchers at the U.S. Department of Agriculture (31). A patented commercial process (27) on similar principles currently is being used at 2 commercial plants in the United States. This process needs low capital and operating cost, but its disadvantages are poor controls and selectivity.

Another commercial process is based on the inventions of Mills et al. (29) about 30 years ago. In this process, oil, hydrogen and catalyst all flow concurrently through the reactor from one stage to the other. Analysis of this reactor and probable flow patterns therein indicate that the rate-controlling step is probably different from that observed in conventional batch reactors.

Other patented, published process using packed beds (32-34) and plate columns (28,30,35) are not widely adopted as yet.

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[Received April 10, 1980]

Computer Control in the Extraction and Edible Oil Industries

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ABSTRACT

The cost of computers is coming down, and the availability of programming languages and application packages for process control is increasing. These facts indicate that computers should find more application to extraction and edible oil plants. Some of the potential applications for these industries, with examples, are discussed.

Process control computers have not found wide application in the vegetable oilseed extraction or edible oil processing industries. Two reasons for this might be: (a) plant capital costs for typical-size plants in the industry are an order of magnitude less than costs for typical plants in the petroleum, petrochemical, or chemical process industries where such computers have found wide application. As a result, the cost of computer control in the vegetable oil industry may represent a higher fraction of the total plant cost than it does in the larger industries; (b) application of computers to plants requires more sophisticated engineering support for both application and maintenance than is available at many plants.

Computer technology (hardware and software) is advancing rapidly, and the capital cost for computers is coming down whereas the capital cost for the process equipment in the plant continues to rise with inflation. Thus, the economic barrier to computer application is becoming less significant. The remaining barrier is largely one of communication between the process engineer and the computer and its hardware and software manufacturers. This paper will develop some background information on computers, from the process engineer's viewpoint, and show some examples of computer applications in the extraction, vegetable oil processing and allied industries.

Most of the discussion in this paper will be directed toward the computer size defined for the past few years as a "mini." Computer sizes may be broadly defined as the micros, the minis and the large main frame machines.

The mini-size computer has been available much longer than the more recent microcomputer and has been applied to most process control functions. It is characterized by processing speeds and memory capacity intermediate between the micros and the large machines, and has sufficient capacity to handle almost any individual plant to which it might be applied.

HARDWARE

Process control computer hardware has developed during the past decade at a much faster rate than has process control software. Computer speed is doubling every one and one-half years, while costs are coming down at a similarly rapid rate. Hardware is currently available in sufficient processing speed and memory capacity to do anything we might imagine in the process. One problem in using this new capability is understanding and defining the process so that the computer program can anticipate all of the variations which may happen in the process and direct the appropriate action.

Process control computer systems have many similarities to data processing computers, but there are some significant differences about which the process engineer should be knowledgeable. Figure 1 shows a very simplified block diagram of a process control computer system. The center portion of this figure shows the hardware which we would generally call a computer. From a hardware standpoint this unit can be electronically very similar to a computer used for data processing. On the right side of the figure are keyboards, cathode ray tubes (CRT or television screens), and printers, which are again very similar to the types of units found in data processing systems.

The unique difference between the hardware in process control systems as compared to data processing systems is the input and output hardware shown on the left side of the figure. The data processing computer system ultimately receives its inputs and outputs only from human beings via its keyboards and printers. The process control computer must receive process inputs and transmit outputs directly without human intervention.

The basic function of the input equipment is to tell the computer what the process is doing in terms that the computer understands. This basically means that the input equipment must present to the computer the state of the process variable in the form of a binary digital signal.

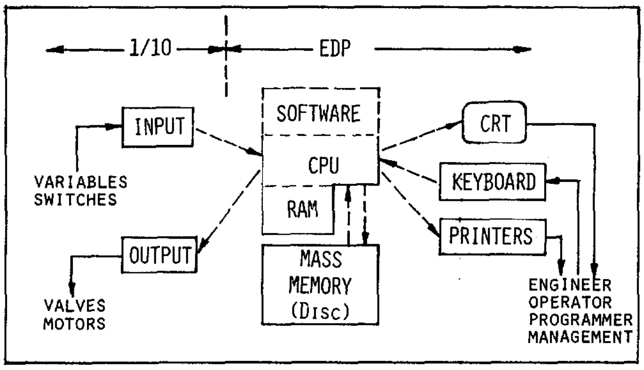

FIG. 1. Process control computer.