Catalytic Hydrogenation

Using Teledyne Isco Syringe Pumps

Syringe Pump Application Note
AN19

Overview

Hydrogenation is a chemical reaction of great importance to the petrochemical and fine chemical industries. In its most elementary sense, the term hydrogenation refers to the addition reaction of molecular hydrogen with an unsaturated carbon-carbon double bond as illustrated:

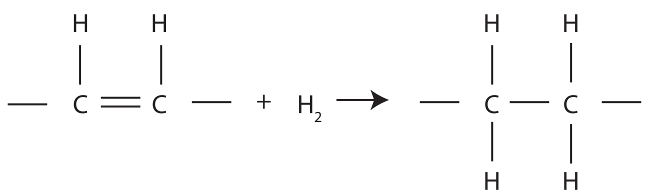

Figure 1: Typical hydrogen reaction

The first compound, called an alkene, is converted into the corresponding alkane.

In addition to the example above, there are other ways in which molecular hydrogen can be reacted with other types of molecules. These include the incorporation of hydrogen accompanied by cleavage of the starting molecule as in hydrodesulfurization and reactions in which the starting molecule undergoes rearrangement such as isomerization.

Hydrogenation in Practical Use

For the petrochemical industry, many of the compounds found in crude oil are of little use since they contain multiple double bonds; they must be first converted to saturated compounds before use as commodities such as gasoline. In addition, countless other molecules are produced from petroleum; hydrogenation is most often the first step their production.

In the fine chemical and active pharmaceutical ingredient industries, the hydrogenation reaction is often an important step in producing the end product.

The food industry uses hydrogenation to completely or partially saturate the unsaturated fatty acids in vegetable oils to convert them into solid or semi-solid fats (e.g. margarine). These latter compounds offer different cooking or taste characteristics that are more satisfactory for consumers.

Hydrogenation will be a critical step in the upgrading of biocrude to usable, renewable biofuels. Biocrude has been hydrogenated to minimize its negative aspects. The instability of oil is reduced by reaction of the most unstable functional groups. Concurrently, the oxygenated component of the oil was also reduced, resulting in an improved energy density. Hydrotreated biocrude is also more miscible with refined petroleum products.

Methodology

Molecular hydrogen does not readily react with organic molecules; a catalyst is always required. A catalyst is a substance that controls a chemical reaction, but is not consumed or part of the final product. Catalysts work by lowering the activation energy needed for colliding molecules to reach the transition state. Therefore, catalysts can allow reactions to take place that would otherwise not be possible or allow them to happen at a much faster rate.

A comparison of the effect catalysts can have in the energy required can be seen in Figure 2:

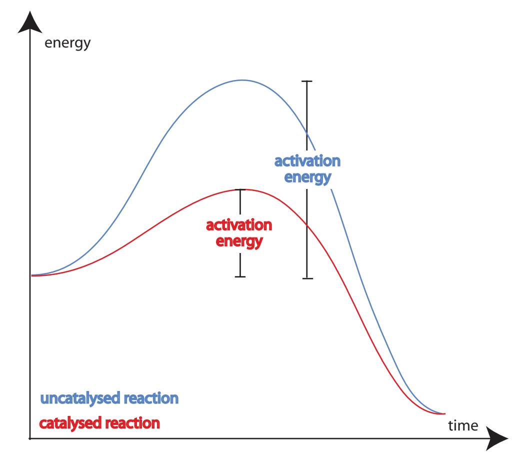

Figure 2: Boltzman Energy Diagram for reaction pathway

Catalysts have no effect on the chemical equilibrium or overall energy change; they cannot make the impossible possible. A catalyst simply:

- Enables reactions
- Improves efficiency
- Increases selectivity

Hydrogenation reactions use a catalyst such as palladium, platinum, rhodium, ruthenium, or Raney nickel, and are performed at elevated temperature and pressure. Temperatures range from 70 to several hundred °C, and at pressures ranging from 12 to 2000 bar or higher.

A typical hydrogenation setup using a semi-batch or continuous CSTR reactor is shown in Figure 3.

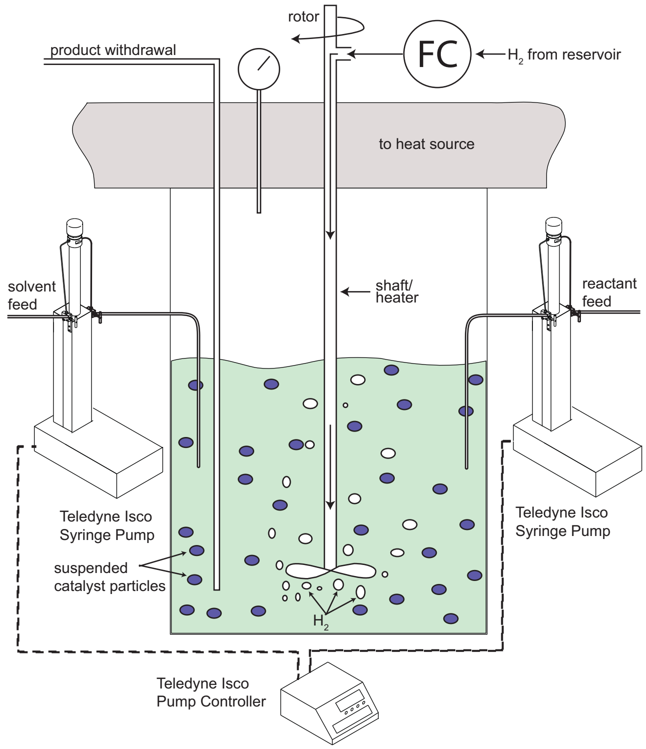

Figure 3: Typical Laboratory scale hydrogenation reactor system with two Isco pumps operating independently from one controller, in constant flow mode

The stirred reactor is a 100 milliliter to 300 milliliter pressure vessel with controlled heating and cooling on both the input and output. Hydrogen is metered and its consumption measured using a mass flow control system.

Teledyne Isco Syringe Pumps

This type of reaction requires pumps for liquid reactant and solvent at precise flow rates over a wide pressure range. The Teledyne Isco syringe pumps provide precise and accurate flow rates at a precision of 0.5% or better of setpoint over the reaction pressures of 50 to 2000 psi or higher. Pump calibration is not required. Mass loss from a balance of calculations and feedback control to the pump are not required. Furthermore, if the reactant is a highly viscous liquid of low melting solid, the entire syringe pump cylinder can be easily heated to provide easy liquid flow control.

The syringe pumps provide truly pulseless flow; the feed rates of the liquid reactant are constant and show no sinusoidal flow typical of other pump types, such as reciprocating pumps. Thus, the kinetics of formation of the depletion of the reactant, and the formation of the desired product, are kept absolutely constant at any specified H_2 pressure or reaction temperature. Unwanted or side reactions due to varying concentrations are suppressed.

The syringe pumps will be set to the selected flow rate, and that flow rate will be kept constant should the reactor pressure vary. In the reactor system discussed here, the control of the H_2 flow with a mass flow controller, and the use of a back pressure regulator, will cause some minor swings and variations in the reactor pressure. The syringe pumps are being operated in constant flow mode. In this mode, the preprogrammed flow rate will remain absolutely constant at the set rate, regardless of any pressure swings. This feature will again minimize the occurrence of undesired reactions.

Table 1: Commonly Recommended Pumps

	1000D	500D	260D	100DM	65D
Flow Range (ml/min)	0.100 - 408	0.001 - 204	0.001 - 107	0.00001 - 30	0.00001 - 25
Pressure Range (psi)	0 - 2,000	0 - 3,750	0 - 7,500	0 - 10,000	0 - 20,000

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