ChE laboratory

CHEMICAL REACTION EXPERIMENT FOR THE UNDERGRADUATE LABORATORY

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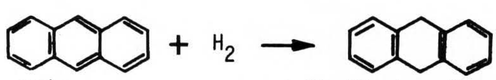
TUSKEGEE'S CHEMICAL ENGINEERING Department was founded in 1977 and was accredited by the EAC/ABET in 1983. There are approximately eighty students presently enrolled. Three chemical engineering laboratory classes are taught; one for junior students and two for senior students. The first laboratory class consists mainly of fluid mechanics and heat transfer experiments. The second laboratory consists mainly of mass transfer, thermodynamics and chemical reaction experiments, as shown in Table 1. Approximately twelve of the experiments are done in any one semester with the choice being made by the instructor. The third chemical engineering laboratory consists of process control experiments. The laboratory classes are offered twice a year with an average class size of ten students, usually divided into three groups. Each student must analyze the data, make the necessary calculations and submit a written report conforming to acceptable standards.

CHEMICAL REACTION EXPERIMENT

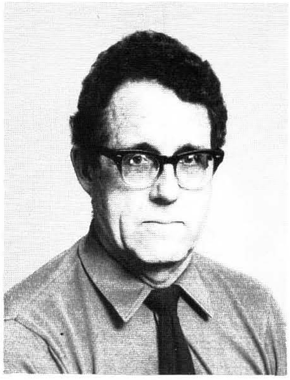
For Experiment 16, anthracene is hydrogenated with molecular hydrogen in the absence of catalyst in a batch-type microreactor to identify the reaction order, the reaction rate constant, the frequency factor and the activation energy for the anthracene-hydrogen reaction system shown in Eq. 1.



anthracene 9,10-dihydroanthracene (1)

EQUIPMENT DESCRIPTION

The 316 stainless steel microreactor assembly consists of a 1/2 inch tee, an 11-inch piece of high pressure 3/8″ O.D. tubing and a shut-off valve. The tee is the



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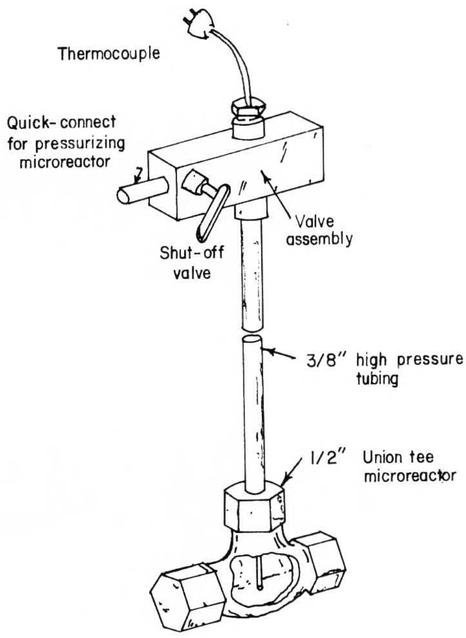

FIGURE 1. Microreactor Assembly

actual microreactor and is connected to the shut-off valve by the 3/8" tubing (see Figure 1). A thermocouple extends through the tubing and into the microreactor, allowing temperature monitoring of the reactants throughout the experiment. A quick-connect is attached to the shut-off valve in order to introduce hydrogen into the microreactor assembly during charging and to release excess hydrogen from the microreactor after an experimental run is completed. The total internal volume of the microreactor is roughly 13 cc. The microreactor assembly is submerged and heated in a fluidized sand bath (see Figure 2) and is shaken throughout the experimental run in order to eliminate the mass transfer effects. The sand bath temperature is adjusted using a thermocouple and temperature controller.

TABLE 1
List of Experiments for the ChE LAB II

EXP. NO.	DESCRIPTION
1	Continuous Distillation with Total Reflux
2	Continuous Distillation with Feed at Bubble Point
3	Batch Distillation in a Packed Column
4	Fluid Flow Through a Packed Column
5	Flow Through a Fluidized Bed
6	Filtration
7	Gas Chromatograph
8	Evaporation
9	Vapor-Liquid Equilibria
10	Liquid-Liquid Equilibria
11	Liquid Extraction
12	Hydrodynamics of a Packed Column
13	Absorption of CO_2 in Water/Analysis of Gas Streams
14	Absorption of CO_2 in Water/Analysis of Liquid Solutions
15	Heats of Solution
16	Reaction Kinetics of the Anthracene-Hydrogen System
17	Spray Drying

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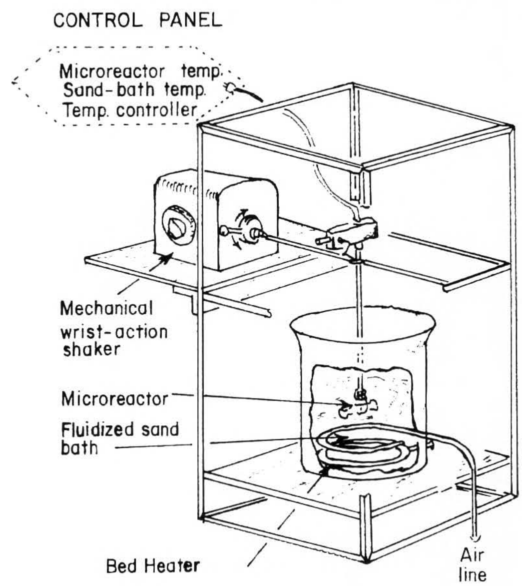

FIGURE 2. Fluidized Sand Bath

EXPERIMENT DETAILS

A series of anthracene hydrogenation experiments is conducted at 375°C, 400°C, and 425°C. The microreactor is charged with 0.1 g anthracene, 2.0 g 1-methylnaphthalene as a physical solvent and 1200 psig hydrogen at room temperature. After being charged with the reactants, the reactor is attached to the shaker mechanism and is submerged in the preheated fluidized sand bath.

Following hydrogenation of anthracene at the desired reaction time and temperature, the reactor is quenched in cold water and the excess hydrogen is released. The liquid products, consisting of anthracene, 9-10 dihydroanthracene and 1-methylnaphthalene, are injected into a gas chromatograph,

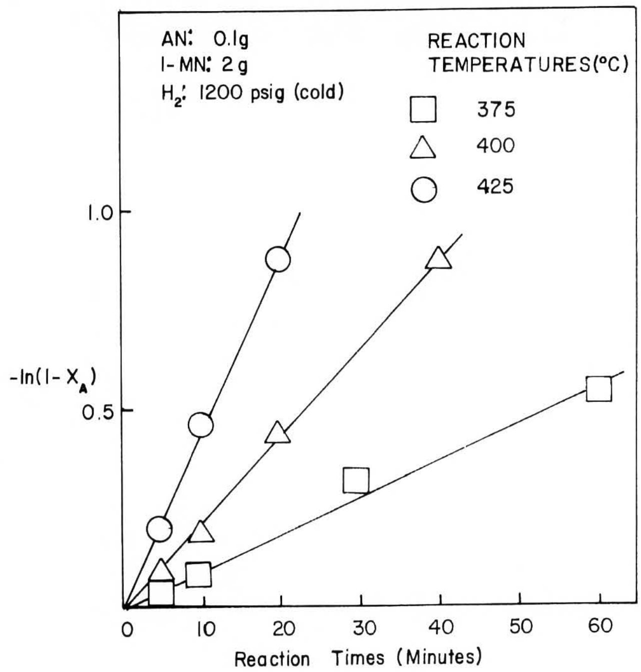

FIGURE 3. Conversions of Anthracene vs. Reaction Times

equipped with a flame-ionization detector, an integrator-plotter and an 8 ft. long, 1/8 inch O.D., SP 2100 packed column, to analyze conversions of anthracene to 9,10-dihydroanthracene.

DATA ANALYSIS

The reaction data, anthracene conversions *vs* reaction times, are plotted on semi-logarithmic paper to identify the reaction order for the anthracene-hydrogen system. A typical plot is shown in Figure 3 and produces a straight line through the origin, indicating that the anthracene-hydrogen reaction system is first order. Reaction rate constants are calculated by applying conversion *vs.* reaction time data to the first-order reaction equation, as shown in Eq. 2.

$$-\ln(1 - X_A) = kt \qquad (2)$$

where X_A = fractional conversion of anthracene
k = reaction rate constant, min^{-1}
t = reaction time, minutes

The activation energy and the frequency factor for the anthracene-hydrogen reaction system were found to be 2.699 x 10^7 cal/gmole and 1.215 x 10^4 min^{-1}, respectively, by applying reaction rate constant *vs* reaction temperature data to the Arrhenius' Law, as shown in Equation 3 and Figure 4.

$$k = k_o \exp(-\Delta E/RT) \qquad (3)$$

where k = reaction rate constant, min^{-1}
k_o = frequency factor, min^{-1}
ΔE = activation energy, cal/gmole
R = ideal gas constant, cal/gmole-K
T = reaction temperature, K

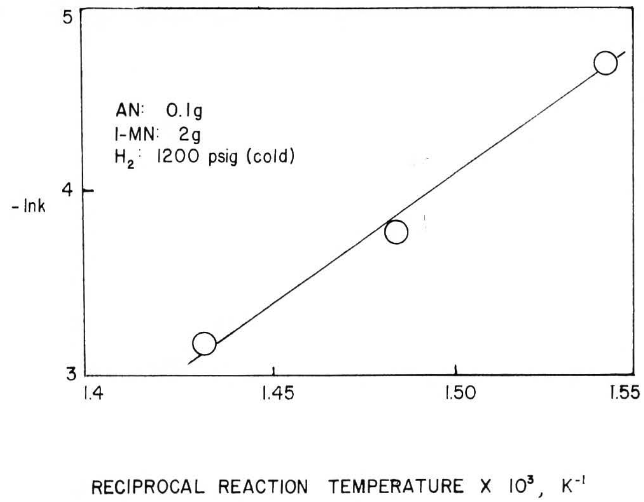

FIGURE 4. Reaction Rate Constants vs. Reaction Temperatures

CONCLUSION

A series of reaction samples is obtained by performing reaction runs at the desired hydrogenation temperatures and times. These samples are analyzed using a gas chromatograph.

This batch-type microreactor has several advantages over other type reactors in carrying out reaction experiments for undergraduate laboratory classes:

- It takes a short time (1 minute) to increase reactor temperatures from an ambient temperature to a desired reaction temperature in comparison with conventional autoclave reactors. Therefore, several experimental runs can be conducted during the 3-hour class.
- It is easy to clean a reactor after finishing a reaction experiment and then to prepare another experimental setup.
- Reactants such as anthracene, 1-methylnaphthalene and hydrogen are needed in small quantities, in comparison with other conventional autoclave reactors.
- There are fewer leakage problems with microreactors during reaction experiments at high temperatures and pressures, in comparison with conventional autoclave reactors which utilize stirring systems. □