Experiment 4
Effect of throughput on conversion using tubular reactor

CHEN3010/ CHEN5040 - Chemical Reaction Engineering - S1 2024

> ❗ Prelab Module
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> Prelab module for experiment 4 Can be accessed here.

1 Objective

To investigate the effect of throughput on conversion

The saponification reaction of sodium hydroxide and ethyl acetate

$$\mathrm{NaOH} + \mathrm{CH_3COOC_2H_5} \longrightarrow \mathrm{CH_3COONa} + \mathrm{C_2H_5OH} \tag{1}$$

$$\text{Sodium Hydroxide} + \text{Ethyl Acetate} \longrightarrow \text{Sodium Acetate} + \text{Ethyl Alcohol}$$

is elementary as written. The rate equation is given by $-r_A = kC_AC_B$, Where, A is NaOH, and B is $CH_3COOC_2H_5$. We would like to determine the rate constant k using a continuous plug flow reactor system.

2 Experimental procedure

The experiment will be carried out in a tubular flow reactor (Armfield Limited, model: CET-MKII). The Armfield Continuous Tubular Flow Reactor is designed to demonstrate the mechanism of a chemical reaction in such a reactor as well as the effects of varying the process conditions such as reaction temperature, reactant concentration, feed rate etc. Refer to the instruction manual for the reactor setup to understand more about the experimental setup. A virtual tour is also provided that describes the setup in detail.

For this experiment, Reactions are monitored by conductivity probe as the conductivity of the solution changes with conversion of the reactants to product. The reaction eventually reaches steady state when a certain amount of conversion of the starting reagents has taken place. This experiment will be repeated at various reactant flow rates to investigate the relationship between the specific rate constant and the residence time. Please refer to equipment manual exercise C for further details.

2.1 Steps

1. Make up 2.5 litre batches of 0.1M sodium hydroxide and 2.5 L of 0.1M ethyl acetate.
2. Remove the lids of the feed bottles and carefully fill with the reagents. Refit the lids and fit the silicone pipe from the pumps.
3. The experiments involve the collection and storage of conductivity data. The USB port located at the front of the Service Unit must be connected to the computer. This will enable data logging of the conductivity, flow rates and temperature sensors at selected time intervals over a selected period of time.
4. Ensure the conductivity probe and temperature sensor has been installed in accordance with the Installation section.
5. Start the software using the option of the experiment with heater.
6. It has been determined that the degree of conversion of the reagents affects the conductivity of the reactor contents so that recording the conductivity with respect to time using the Armfield data logger can be used to calculate the amount of conversion.
7. Prior to priming the hot water circulating system, fill the reactor with water. Fill the vessel to a level above the overflow (return to the circulator), just below the reactor lid, using a suitable hose from a domestic supply through the temperature sensor gland in the lid. A non-return valve prevents water flowing out of the reactor via the inlet. Ensure the thermocouple is re-fitted and the gland tightened securely by hand before releasing the outlet tubing.
8. Set PID controller loop according to the settings for an Experiment with Heater described in operation section.
9. Adjust the set point of the PID to 30 $^\circ$C.
10. Change PID 'mode of operation' to 'Automatic'.
11. Switch on the Hot Water Circulator by clicking 'Hot Water Circulator' and then 'Power On'. The temperature of the water in the reactor vessel will begin to rise and within 10-15 min will be automatically maintained at the desired set-point (30 $^\circ$C in this instance).
12. When temperature reactor is steady type in the value of the concentration of both solutions on the software.
13. Switch on the pumps by typing the flow rate in the software and instigate the data logger program (or begin taking readings if no computer is being used).
14. Reactants will flow from both feed bottles and enter the reactor through the connections in the lid. Each reactant passes through pre-heat coils submerged in the water in which they are individually brought up to the reaction temperature. At the base of the tubular reactor coil, the reactants are mixed together in a "T" connection and begin to pass through the coil. The reacting solution will emerge from the coil through connector in the lid where a probe senses continuously the conductivity which is related to degree of conversion. For an accurate conductivity reading, no bubbles are allowed in the reactant pipe.

15. Collection of data will be until a steady state condition is reached in the reactor and this takes approximately 30 minutes. It is advisable to set the data collection period to 45 minutes.

3 Data analysis

Having recorded the conductivity of the contents of the reactor over the period of the reaction, the conductivity measurements must now be translated into degree of conversion of the constituents. Both sodium hydroxide and sodium acetate contribute conductance to the reaction solution whilst ethyl acetate and ethyl alcohol do not. The conductivity of a sodium hydroxide solution at a given concentration and temperature however, is not the same as that of a sodium acetate solution at the same molarity and temperature and a relationship has been established allowing conversion to be inferred from conductivity.

On conclusion of the experiment, the set of readings of conductivity with time are obtained. The calculations required for converting the conductivity data into concentration are given in the instruction manual "CET-MKII Issue 21 Instruction Manual.pdf"

4 Tasks

Prepare a report based on your interpretation of experimental data. The report should consider the following:

1. Starting from first principles, derive an expression for the conversion in PFR in terms of residence time. List the assumptions made
2. **Data analysis**: For the experimental data obtained, perform data analysis in Excel to analyze the data for three different reactant flow rates. Based on the analysis, report the reaction rate constants at three different reactant flow rates, and the activation energy for the reaction. Present relevant graphs for all the data sets.