Michigan Technological University
Department of Chemical Engineering
CM 4110 Unit Operations Laboratory

Continuous Stirred Tank Reactor Experimental Notes
Phenolphthalein Fading Reaction

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In this experiment, you will determine the kinetics of the phenolphthalein fading reaction in a batch reactor at room temperature and then apply your findings to continuous reactors. See the handout for information about the theory of this reaction in a batch and in continuous reactors. The reaction will be monitored by reading the absorbance at 550 microns using the Spectronic 20 Series Spectrophotometer.

1. To do this experiment, you will first do batch scale reactions. You are to use the following set of solutions, which you will also dilute with distilled water to perform additional studies.

0.15M NaCl (0.8775 g table salt dissolved in of 100 ml distilled water)
0.000375M Phenolphthalein (0.1195g solid Phenolphthalein which is first dissolved in some 190 proof ethanol and then distilled water is added to make 1 liter)
0.75M NaOH (3.0 g NaOH dissolved in 100 ml distilled water)

2. You will add to a glass test tube (for example), 2 ml of 0.15 M NaCl, then 2 ml of 0.75 M NaOH, and LAST 2 ml of 0.000375M Phenolphthalein. Then start the timer (watch) and put the test tube in the sample compartment (in the spectrophotometer) and take an absorbance value at 0 sec, 30 sec, 60 sec, etc. until the absorbance value does not change anymore. After 20 minutes you should stop recording data and just wait until the number does not change every few minutes.

At t = 0 seconds (when all 3 reactants are added), the absorbance (A) will typically be the highest value (at 2 = dark pink color, for the 'best case concentrations' you want the initial and maximum absorbance to be about 1.0). As time passes, A decreases. Note that A = 0 for a clear solution (no light is absorbed). Realize for this example that if you added 2 ml of 0.15 M NaCl, 2 ml of 0.75M NaOH, and LAST 2 ml of 0.000375M Phenolphthalein, then in your solution in your batch reactor you now have 0.05 M NaCl, 0.25 M NaOH, and 0.000125 M Phenolphthalein.

Note that you may have the absorbance meter 'stuck' at 2 for over 2 minutes. When A = 2, the reaction is happening but we can't measure it (this is bad).

3. To run the Spec 20, you must first calibrate the device. For more information see the manual.

a. Turn on the power switch (On/Off main power switch - operated by Zero Control knob). Set this to read 0% T (transmittance) when the sample compartment is empty and the cover is closed.

b. Set the Wavelength Control to 550 microns (displayed on left side of LED display).

c. Put distilled water in a clean test tube (6 ml: want test tube about 70% full so light passes thru the solution to be tested) and put it in the sample compartment. Be sure you always close the cover of the device to get an accurate reading. Adjust the Transmittance/Absorbance Control knob (right side) to get 100% T reading = none is absorbed.

d. Use the Mode button on the top of the instrument to switch between transmittance and absorbance. Use the absorbance mode for your tests.

e. When you are finished with the Spec 20, turn it off by turning the Power Switch counterclockwise until it clicks.

4. You will study some batch reactor cases. Run one batch reactor test and determine the rate constants for each case.

Case 1) Use the pure solutions as provided.
Case 2) Dilute the NaOH solution by a factor of two.
Case 3) Dilute the Phenolphthalein solution by a factor of four.

Hint – for case 2 and 3 mix up in a separate beaker the correct ratios of the solution required. Then add the correct volume to the test tube using a buret.

5. Determine which of the cases is most appropriate to study in a CSTR. You should pick a case where (during batch reactor studies) there was an initial absorbance close to 1.0 and a decay to equilibrium after about ten minutes. Repeat two more batch reactor tests for this case.

6. You will run the CSTR experiment at a total flow rate of 0.45 liters per minute.

a. The volume of the 1st CSTR (top one) at steady state is 2.0 liters. The volume of the 2nd CSTR (bottom one) at steady state is 3.0 liters.

b. Use distilled water to clean out all the CSTR lines BEFORE you run the CSTR. One or two people in your group can do this while the others work on the batch reactor part of this experiment. Running with distilled water will also enable you to calibrate the flow rates that you will need when you have reacting solutions in the supply tanks.

c. When you turn on the CSTR pumps, open the bleed valve to get air out of the lines so the pump can operate properly. If you do not get any flow ask Tim Gasperich, the TA, or Jason Keith for help.

d. Determine the rotameter setting required for each pump to give one-third of the total flow rate. Keep checking the rotameter settings as they may change slightly. Use the attached calibration curves as a guide to help you. Let the system run at a constant flow rate using distilled water for at least half an hour.

e. Mix up 5 liters of each solution in the big plastic jugs.

f. Start all 3 streams (chemicals: NaOH, Ph, NaCl) at once. Let the system run for at least five minutes at the desired flow rate values.

g. Use a buret to take a sample out of each CSTR (note that you only need to get a sample when at steady state (see the handout to learn CSTR theory) using the 'probe' hole. Then quickly put the sample from the buret into the test tube and record the 1^{st} good reading on the spectrophotometer. You only want a 2-3 seconds to elapse between taking the sample out of the CSTR and getting a good absorbance value.

h. Wait 5 minutes and repeat step g. until you have the same absorbance values (this means the reactor should be at steady state). You should have at least three absorbance data points for each CSTR to determine error.

i. You may wish to compute the volumetric flow rate again as the rotameters may have drifted during operation.

j. When you are finished, wash out all of the jugs that you used, and flush the system with distilled water at a high flow rate (about 15 on each rotameter) for at least 30 minutes.