Date: Tuesday, September 17 and 24

Topic(s): Characterization and modeling of reactors

Report: ***Group*** lab assignment due, Tuesday, October 1, 2019

Preparation: 1. Read lab handout

Objectives:
1. Apply the concept of a standard curve in experimental research.
2. Describe the physical differences between common reactors.
2. Characterize hydrodynamics of a non-ideal reactor and compare to ideal behavior.
3. Derive a CMFR mass balance and apply non-linear regression to quantitative reactor analysis.
4. Account for non-ideal behavior in environmental analysis.

As we will discuss in class, natural and engineered systems are often studied and represented by ideal engineering reactor configurations. Three common ideal reactors are batch reactors, completely-mixed flow-through reactors (CMFRs), and plug flow reactors (PFRs). The decision of which type of reactor to use in your model greatly influences performance or behavior of the overall system, whether physical, chemical or biological. In reality, ideal CMFRs and PFRs do not exist. However, the goal in reactor design or modeling is often to achieve either CMFR or PFR conditions as close to ideal as possible.

To observe and quantify reactor characteristics in this lab session, each group will input a spike input, or "slug" dose, of a conservative tracer, which will be a chemical dye called methylene blue (MB). The hydrodynamics (flow behavior) of each reactor will be determined based on observation of the behavior of MB. This lab will be performed over the course of two weeks.

Procedures
Wear safety glasses and gloves for this experiment.
Each group will work with a different reactor, but there will be two identical CMFRs.

All groups are required to observe and analyze data for all three reactors.

Week 1 – All groups:

1. Prepare a standard curve for the MB tracer. Using the 1,000 mg/L stock solution, prepare five diluted standards of MB using volumetric flasks, a pipettor, graduate cylinders, and deionized water. The concentrations of the standards should range from zero to 10 mg/L. Mix each dilution thoroughly. GOOD LAB TECHNIQUE IS CRITICAL FOR OBTAINING AN ACCURATE STANDARD CURVE FOR YOUR GROUP.
2. Determine the optimum wavelength at which to measure your tracer chemical:
    a. Find a square cuvette and fill it (up to the 10 mL line) with deionized water from the wash bottle on the lab bench. This will be your blank.
    b. Turn on the spectrophotometer and allow it to go through the self-test (the lid needs to be over the light cell opening).
    c. Select 'Wavelength Scan'.
    d. Insert the blank and press the zero button to zero the spec.
    e. Pick one standard (4 mg/L or 8 mg/L, for instance) and pour some into a clean square cuvette. Insert the square cuvette into the spec and press 'Read' to read the absorbance scan of the standard.

f. Write down the optimum wavelength (highest absorbance) for measuring MB, and use that wavelength for all subsequent steps this week and next week.

3. Read absorbance at optimum wavelength for all standards
    a. Select "main menu" → "single wavelength", and enter the wavelength from g.
    b. Zero the spec using DI water in a clean test tube ("cuvette").
    c. Transfer a portion of each dilution to a clean cuvette. Wipe the cuvette completely to remove fingerprints, etc.
    d. Measure and record absorbance of each standard using the spectrophotometer, set to the optimum wavelength determined above.
    e. Prepare a scatter plot (absorbance vs. concentration) in Excel or Kaleidegraph and fit a linear equation to the data showing the equation of the line and coefficient of determination (R^2). **Save this file for your use next week!** You'll need it to back calculate from absorbance to concentration.
4. Before the end of lab, post the Excel spreadsheet or Kaleidegraph plot with the equation information shown on the plot with this standard curve data and plot to the Lab 4 and 5 Google Drive Folder. This file should have a descriptive file name, using the following format "LastName1_LastName2_LastName3_Lab4_CEEG340".

Week 2: Each group will have one reactor:

Procedure for PFRs:

1. Before you start working with the reactors, zero the spectrophotometer at the same wavelength you used last week for the standard curve.
2. The volume of PFRs are ~ 2.0 L (without packing material) and ~0.95 L (with packing material).
3. **For the 8am Section**: The pump is pre-set to a flow rate of either 150 mL/min (no packing material) or ~70 mL/min (with packing material). Measure and record the flowrate (with a graduated cylinder and timer).
4. **For the 10 am Section**: Set the flow rate to 300 mL/min (no packing material) or ~150 mL/min (with packing material). Measure and record the flowrate (with a graduated cylinder and timer).
5. Turn pump off and inject 0. 5 mL of 10,000 mg/L MB solution into the injection port.
6. Close the valve and prepare 5 mL of water to inject.
7. Open the valve and inject 5 mL of water, close the valve.
8. Turn the pump on and start the stopwatch.
9. Take samples every minute and measure and record absorbance on the spectrophotometer. Record your observations of the tracer's behavior. Use your judgment to lengthen the sampling interval according to the rate of change of color intensity.
10. Stop sampling once the effluent concentration effectively reaches zero (~35-40 minutes).
11. Measure and record the volume of the PFR before you leave.
12. Using your standard curve from last week, calculate the concentration of MB for each sample.

Procedure for CMFR

1. Before you start working with the reactors, zero the spectrophotometer at the same wavelength you used last week for the standard curve.
2. The volume of the CMFR is approximately 10 L.
3. **For the 8 am section:** Set the mixer speed to 100 rpm.
4. **For the 10 am section:** Set the mixer speed to 200 rpm.
5. The pump is pre-set to a flow rate of 1,000 mL/min. Measure and record the flowrate (with a graduated cylinder and timer).
6. With the pump running and effluent flowing out of the reactor, use a 5mL pipettor to inject 5mL of 10,000 mg/L MB solution right in front of the reactor inlet. Start the stopwatch.

7. Take effluent samples as rapidly as possible at the start (e.g., at 5s, 10s, 15s, 30s, 1 minute) and then at one-minute samples thereafter. Record your observations of the tracer's behavior. Use your judgment to lengthen the sampling interval according to the rate of change of color intensity. Measure and record absorbance for all samples on the spectrophotometer.
8. Continue sampling until the dye has essentially been cleared from the reactor (~35 minutes).
9. Repeat the procedure at a mixer speed of 200 rpm, or possibly 0 rpm.
10. Using your standard curve from last week, calculate the concentration of MB for each sample.

Week 2 Pre-deliverable: *By the 11:59pm on Wednesday, 9/19/19, upload a spreadsheet (to the appropriate Lab 6 google drive folder) that includes:*

a. Reactor type
b. Reactor volume
c. Flow rate(s) and/or mixing rates
d. Your concentration vs. time data
e. This file should have a descriptive file name, using the following format "LastName1_LastName2_LastName3_Lab5_CEEG340".

Final Deliverable – Due at the beginning of your next lab period – this is a group deliverable

For this lab, you team is to hand in one HW style response to the questions below. This is not a full lab report write-up, but rather an analysis and interpretation of the data through the response to the questions below. Although not a full lab report, this is a formal write-up. All your responses to the questions must be typed, your figures and tables should have captions and numbers and in the text when you discuss them, refer to the figures and tables by their figure and table numbers. Each figure should be referred to in the text before it is presented. Label all your responses according to the question numbers below. This is a group assignment. Each group will turn in one assignment.

1. Create and plot a detailed, dimensioned, scaled AutoCAD drawing for the CMFR, PFR and PFR with packing materials. Draw both the front and top view of each reactor. Be sure each drawing is labeled appropriately and the flows are shown on the drawing

2. Create *three* separate figures:

    a. C vs t data for the PFR at two flowrates– show both data sets on the same graph, without the packing. On the graph, show what the data would look like for an ideal PFR. This is a thought experiment, as you can't really derive an equation for this case, but you can show conceptually what the graph should look like for ideal behavior.

    b. C vs t data for the PFR for two flowrates– show both data sets on the same graph, with the packing. On the graph, show what the data would look like for an ideal PFR. This is a thought experiment, as you can't really derive an equation for this case, but you can show conceptually what the graph should look like for ideal behavior

    c. C vs t data for the CMFR reactors at both mixing speeds – show both data sets on the same graph (one at each mixing speed). Also plot the curve for an ideal CMFR. The equation of the ideal CMFR plot is determined from reactor volume, flow, initial concentration and a mass balance with simplifying assumptions (see next item below).

3. For the CMFR, derive the equation of a mass balance on MB. Make appropriate assumptions (state them) and solve the equation for effluent concentration of MB as a function of time. Be sure to show all your work. Plot this line on the figure as instructed above for the theoretical hydraulic retention time (HRT=V/Q) based on flow and volume of the CMFR.

4. For the PFR reactors, estimate the actual HRT and show where this would occur on the PFR graphs. For the PFR, you can do this visually using your graphs, the average time a fluid element remained in the reactor is the centroid of the area of the C vs t graph, you can determine this by visually estimating this point on the graph

5. For the CMFR, estimate the actual HRT using regression through the data. You can use the equation derived in #3 above, and Kaleidegraph, and 'fit' through the tracer data using HRT as the variable. Show this fit through the data using a separate graph.

6. Based on your analysis for the previous questions, comment on how closely each reactor achieved "ideal" characteristics.

    a. PFR – how did the shape of the curve compare to the ideal situation? Did the packing materials for the PFR improve or worsen the hydraulic behavior relative to the ideal expectations? Why do you think this occurred?

    b. CMFR – how did the shape of the curve compare to the ideal curve? Explain possible reasons for differences in the actual versus ideal.

7. For each reactor test, calculate the mass of the tracer injected and the cumulative mass of tracer that passed out in the effluent. This is a mass balance on tracer. You can do this by estimating the area under the curve of C vs t and knowing the flowrate. Calculate, as a percentage, how much of the injected tracer was recovered in the effluent? Summarize your results for all reactors and all tests in a table (Min, Mout, Mass Recovered). Do you think your results are acceptable and why do you think there are differences between the mass in and mass out? Be sure to show all your work and how you determined the different parameters.

8. How should non-ideal behavior be accounted for when we design and model environmental systems?