ChE laboratory

AN IMPROVED DESIGN OF A SIMPLE TUBULAR REACTOR EXPERIMENT

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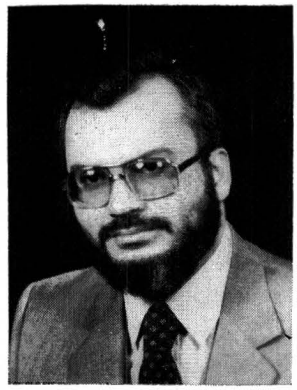

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DESIGNING A TUBULAR FLOW reactor experiment for an undergraduate laboratory is not a simple task. This is because the experiment will have to meet certain criteria, viz

- **It is safe**
- **It is simple and cost effective**
- **It is instructive**
- **Its analytical needs must be simple and easy, to meet the time constraints of an undergraduate laboratory**

Anderson [1] developed a tubular flow reactor experiment for an undergraduate laboratory at Princeton that utilized the system acetic anhydride-water. This particular experiment requires relatively elaborate safety precautions. Moreover, since the reaction is exothermic, rather expensive temperature control equipment is required. Samples taken at the reactor inlet and outlet are analyzed by the aniline-water method which is relatively lengthy and subject to errors.

Hudgins and Cayrol [2] utilized the basic design of Anderson in developing a simple and interesting experiment. They utilized the classical reaction system of crystal violet dye neutralization with sodium hydroxide. This system was studied earlier by other investigators, mainly in a batch reactor (Carsaro [3]). The two novel aspects of the Hudgins-Cayrol experiment compared with that of Anderson are

- **A colour change can be seen between the inlet and outlet of the reactor**
- **The temperature constraint is removed. This makes the experiment operable at room temperature**

Also, from the safety standpoint, a relatively dilute sodium hydroxide solution (0.04 N according to Hudgins and Cayrol) is used.

However, the experimental set-up design given by Hudgins and Cayrol can be significantly improved. The design improvements suggested in this article should make the experiment easier to run and control, significantly improve the reproducibility of results, and expedite the process of data collection within the time constraints of an undergraduate laboratory.

The main objectives of this experiment are

- **To study the effect of residence time on conversion in a tubular flow reactor**
- **To compare the experimental conversions with those obtained from plug-flow and laminar-flow reactor models**

THEORY

As it was established by Corsaro [3], the reaction between crystal violet dye and sodium hydroxide is of the first order in the concentration of each of the reactants, i.e., the reaction is of the second order. However, the reaction can be made pseudo-first order if sodium hydroxide is used in great excess with respect to crystal violet dye. In other words

$$-r_{dye} = k' [dye] \qquad (1)$$

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The value of the rate constant, k′, is needed for this experiment. Students are requested to run a batch experiment to determine the value of k′ at the same temperature of the flow experiment . . . (and) to prepare their own calibration curve of the dye concentration versus absorbance.

For the purpose of this experiment, 0.02 N sodium hydroxide solution is used with 6.86×10^{-4} M dye solution, i.e., the sodium hydroxide concentration would be about 282 times that of the dye, if equal volumes of reactants are used.

Experimental conversions are calculated, as will be described later, and compared with theoretical conversions predicted from the plug-flow model and the laminar-flow model.

For a first order reaction in a plug-flow reactor, the following equation applies assuming constant density of reaction mixture:

$$\tau = \frac{V}{v_o} = -\frac{1}{k'} ln(1-x) \qquad (2)$$

$$= -\frac{1}{k'} ln \frac{C_A}{C_{Ao}} \qquad (3)$$

If C_A is taken as $[dye]_e$, i.e., the dye concentration at reactor exit and C_{Ao} as $[dye]_i$, i.e., the dye concentration at reactor inlet, then one can rewrite Eq. (3) as follows:

$$\tau = \frac{V}{v_o} = \frac{1}{k'} ln \frac{[dye]_i}{[dye]_e} \qquad (4)$$

For a first order reaction in a laminar-flow reactor, the following equation applies assuming no change in volume by reaction as well as no mixing in both radial and axial directions (4)

$$x = 1 - \left(\frac{N_R}{2}\right)^2 E\left(\frac{N_R}{2}\right) + \left(\frac{N_R}{2} - 1\right) \exp(-N_R/2) \qquad (5)$$

where

$$N_R = k'\tau$$

$$\tau = \frac{V}{v_o} = \frac{L\pi r^2}{v_o}$$

The function E(y) is defined by:

$$E(y) = \int_y^\infty \frac{\exp(-\phi)}{\phi} d\phi$$

The function E(y) is tabulated in standard tables as $-E_i(-X)$.

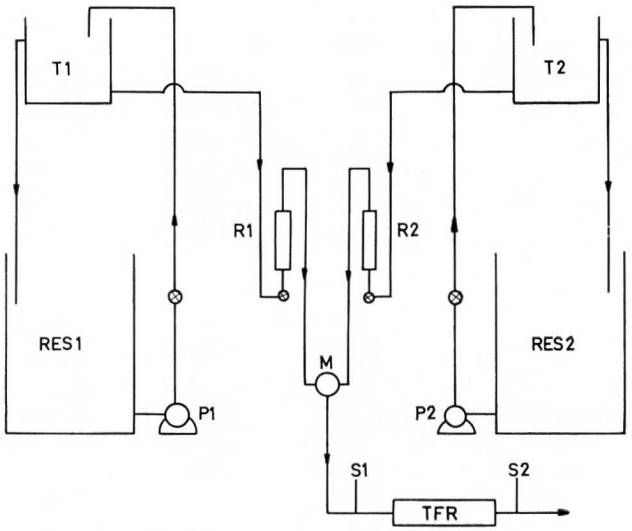

FIGURE 1. Schematic diagram for the experimental set-up. M: mixer, P: pump, R: rotameter, RES: reservoir, S: sampling point, T: constant head tank, TFR: tubular flow reactor.

EXPERIMENTAL

A schematic diagram of the proposed experimental set-up is given in Fig. 1. The experimental apparatus is comprised of the following components.

Reservoirs : (RES 1) 200-L polyethylene tank for the sodium hydroxide solution

(RES 2) 20-L polyethylene tank for the dye solution

Constant Head Tanks : (T1) 20-L polyethylene tank for the sodium hydroxide solution

(T2) 4-L polyethylene tank for the dye solution

Pumps : (P1) Magnet drive gear pump; Model P/N 81152 manufactured by Micropump Corp., Conford, California. Purchased from Cole Parmer Co.

(P2) Centrifugal pump. Cole Parmer catalogue No. K-7004-30

Mixer : (M) Little Giant Pump. Model 2E-38NT. Purchased from Can Lab

Rotameters : (R1) Size R-6-15-A rotameter. Max flow 450 ml/min with SS-float. Purchased from Brooks Instrument Co.

(R2) Size R-6-15-B rotameter. Max flow 1300 ml/min with SS-float.

		Purchased from Brooks Instrument Co.
Reactor	:	40 meters of 3/8 in. I.D. Tygon tubing, wound on spool (made of lexan), 28 cm in diameter and 55 cm in length*
Valves	:	needle valves to adjust flow
Spectrometers	:	Spectronic 20 (Bausch & Lomb) modified, as will be described later, to provide continuous measurements.

Two polyethylene tanks (RES 1 and RES 2) of capacity 20 liters and 200 liters serve as reservoirs for the crystal violet dye and sodium hydroxide solutions, respectively. Two pumps (P1 and P2) are employed to pump the reactants to two constant head tanks (T1 and T2). The overflows from the constant head tanks are returned to their respective reservoirs. The underflows from the constant head tanks go via rotameters (R1 and R2) to a small pump that acts as a mixer (M). The reactant streams are mixed in the mixer, M, and are pumped through the reactor. The tubular reactor is in the form of a helical coil wound on support. Connections are provided at the inlet and outlet of the reactor to the flow-through curvettes of the spectrometers.

The main advantages of the proposed experimental set-up over that suggested by Hudgins and Cayrol are

- **Reservoirs and constant head tanks are used. This arrangement provides more stable rotameter operation, especially at low flow rates.**
- **A flow-through accessory which is simpler in design and operation than that suggested by Hudgins and Cayrol has been used. The flow-through accessory shown in Fig. 2 allows one to use Spectronic 20 for continuous measurements.**

PROCEDURE

Due to the limitation of the headroom in most undergraduate laboratories, the constant head tanks (T1 and T2) are placed about 3 meters above the rotameters' level. This limitation makes it only possible to attain maximum flow rate of 1300 ml/min of NaOH. The maximum flow rate of the dye is set at about 135 ml/min.

*One of the reviewers suggested the use of polyethylene instead of Tygon tubing, which discolors to a deep purple making it difficult to observe gradual colour-change along the reactor. It is believed that polyethylene is more resistant to the dye than Tygon tubing.

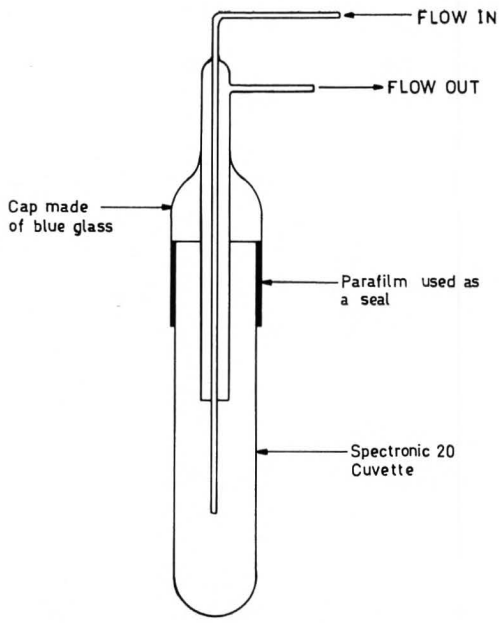

FIGURE 2. Flow-through cuvette for the Spectronic 20.

The flow rates of sodium hydroxide and the dye solution are set such that the ratio is 9:1. One should start at the highest possible flow rate to expel all air bubbles from the reactor.

One should wait for slightly longer than the residence time, for a particular flow rate, for steady-state to be reached. The reaction mixture is then allowed to flow through the Spectronic 20 flow-through cuvettes and the readings are recorded. Usually, one waits for two minutes and takes another reading as a duplicate. Experience has shown that the Spectronic 20 readings are highly reproducible. Other flow rates of NaOH and dye solution are chosen, keeping the flow rates ratio 9:1 as before, and the Spectronic 20 readings are recorded. The experiment usually lasts for one hour provided that the solutions are prepared prior to the laboratory period.

TABLE 1
Holding Time and Conversion Data

Holding Time min	Reynolds Number	PFRM conv.	LFRM conv.	Exp. Conversion x
6.42	791	71.7	63.3	71.5
4.3	1181	57.1	50.3	53.3
3.23	1651	47.1	41.6	44.5
2.54	2000	39.4	34.96	36.9
2.13	2385	34.3	30.7	33.85

The value of the rate constant, k′, is needed for this experiment. Students are requested to run a batch experiment to determine the value of k′ at the same temperature of the flow experiment. This has proven worthwhile, since temperature fluctuations in most undergraduate laboratories do not allow conducting a batch experiment at the beginning of the semester and giving the value of k to the students to perform the required calculations. Also, students are required to prepare their own calibration curve of the dye concentration versus absorbance. This leads to better results.

RESULTS AND DISCUSSION

Table 1 reports the residence time and the conversions from the plug-flow reactor model (PFRM), laminar-flow reactor model (LFRM) and the experimental conversions. Fig. 3, also, depicts the conversions against the residence time. The data reported in Table 1 and Fig. 3 were obtained from an experiment conducted on the set-up available in Windsor.

It is clear from Fig. 3 that, as expected, the experimental conversions fall between the conversions obtained from the two theoretical models, viz., the PFRM and LFRM.

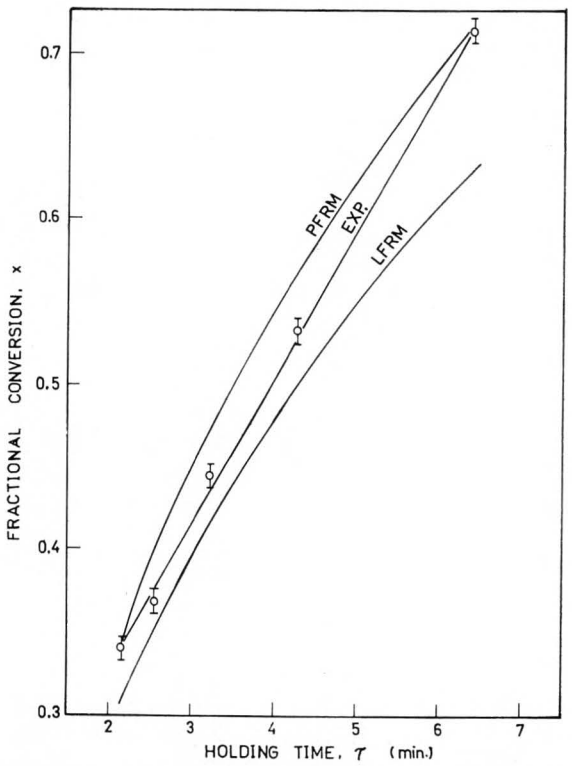

FIGURE 3. Comparison between conversion obtained from experiment and those obtained from LFRM and PFRM.

REQUEST FOR FALL ISSUE PAPERS

Each year CHEMICAL ENGINEERING EDUCATION publishes a special fall issue devoted to graduate education. This issue consists 1) of articles on graduate courses and research written by professors at various universities, and 2) of announcements placed by ChE departments describing their graduate programs. Anyone interested in contributing to the editorial content of the fall 1985 issue should write the editor, indicating the subject of the contribution and the tentative date it can be submitted. Deadline is June 15th.

It is worthwhile to note here that the data reported by Hudgins and Cayrol indicate that the experimental conversion curve crosses the LFRM curve at short holding times, i.e., experimental conversions are lower than those predicted by LFRM, which is not possible. Such results may be attributed to the obvious design flaws in the set-up reported by those authors.

The change of colour of the reaction mixture between the inlet and outlet of the reactor is due to the conversion along the reactor. Such a visual effect helps the students to integrate the laboratory experiment with what they learned in the lecture part of the course about conversion in tubular flow reactors. □

REFERENCES

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4. Holland, C. D. and R. G. Anthony, *Fundamentals of Chemical Reaction Engineering*, Prentice-Hall, Englewood Cliffs, N.J., 1979.

NOTATION

o	=	subscript symbol for initial
i,e	=	subscript symbols for reactor inlet and exit, respectively
C_A	=	concentration of component A, (mole/L)
k′	=	pseudo-first order rate constant, (min^{-1})
L	=	length of reactor tube, (m)
N_R	=	k′ = reaction number for a first order reaction
r	=	inside radius of reactor tube, (m)
(−r)	=	reaction rate, (mol/L·min)
v_o	=	volumetric flow rate, (L/min)
V	=	reactor volume, (m^3)
x	=	conversion
τ	=	V/v_o = space time (min)