

Residence Times in Mixed Tanks and a Tubular Reactor

Introduction

In a continuous fiber dyeing process for blue jeans, a reactive and unstable dye has to be produced, diluted, and used on a continuous basis. Since the demand for the diluted dye varies, as does the required dye concentration, your plant has used a continuous diluter, comprised of three vessels in series, for many years. In this diluter an operator samples the effluent dye concentration every five minutes, analyzes for dye concentration, and then adjusts the dye feed rate. This has proven unsatisfactory in that the effluent dye concentration varies too much, leading to excessive off-spec fiber that has to be discarded or sold at a discount. The recent advent of inexpensive microcomputers and A/D boards with digital output has convinced our technical director that a computer-based control system should be designed and installed. Such a system has been purchased from an outside group, Aginous Associates Inc.

Many questions about the system have arisen, however. For example, it is not known if the three vessels are well mixed at some or all flow rates, or if perhaps it might be better to use a plug flow or tubular reactor to attain better mixing. Certainly at very low flow rates, perfect mixing is not to be expected.) Data on which a mathematical model of the flow system can be based are not available. It is not known how well a PID controller can hold the effluent dye concentration at a desired level, and what controller settings gives the best, or even satisfactory, control.

Before we can proceed with the design of a control system, your group has been selected to test the dye mixing system without the controller to determine the mixing characteristics of the system.

In this experiment you will examine the mixing behavior of a single vessel, three vessels in series and a plug flow reactor using a residence time distribution (RTD) analysis.

Objectives

To investigate the concentration dynamics and residence times related to mixed tanks using impulse and step inputs as tracers and hence obtain best mixing conditions.

To gain further experience using the microcomputer as a tool for data acquisition.

Theory

Figure 1 shows a flow arrangement for several tanks in series (or a tubular reactor which replaces the tanks). These tanks are essentially CSTR's (continuous stirred tank reactors) except that we are not using them to carry out chemical reactions in this experiment. The tanks are all of the same volume (approximately 250ml) and a constant flow rate, v , of water and/or dye solution is maintained. The TFR (Tube flow reactor) is simulated by the use of a horizontal cylinder with a volume of about 150 ml. . Suppose that each reactor initially contains pure water, so that the concentrations of dye in the three vessels are zero. The initial conditions are therefore:

$$c_1(0) = c_2(0) = c_3(0) = 0 \quad (1)$$

DYNAMICS AND CONTROL OF DYE MIXING

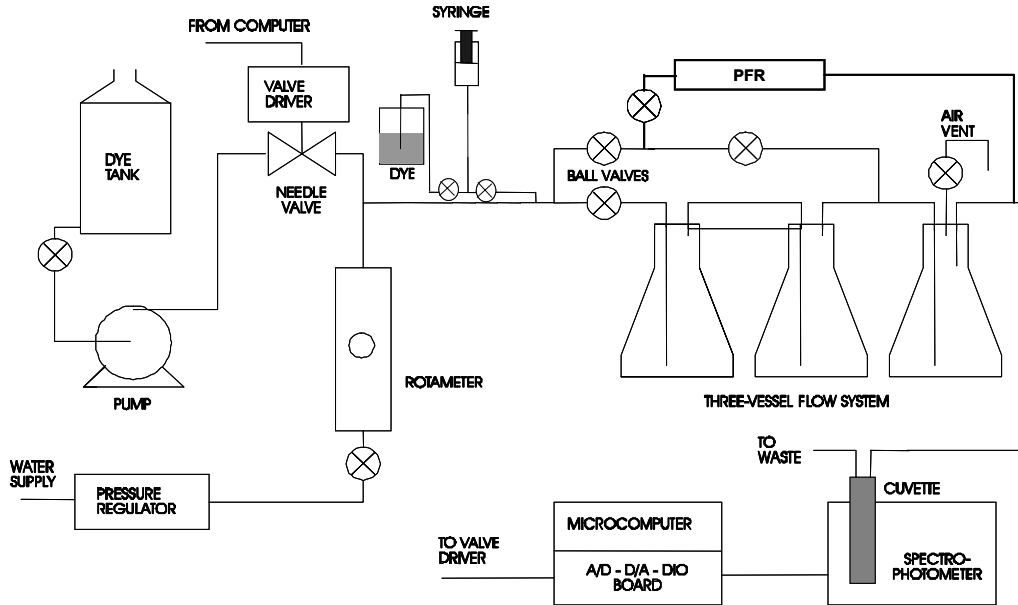


Figure 1 – Mixed Tanks in Series

During the test runs, the inlet dye concentration to the first tank, c_0 , will be changed (impulse) and the dye concentrations of all the tanks will subsequently change in a characteristic manner.

System Dynamics

First, consider the concentrations of dye in each of the four tanks as functions of time. Mass balances for the dye in each tank, accounting for the dye that enters, leaves and accumulates, yield the following three first order differential equations, if all the tanks are perfectly mixed:

$$vc_0 - vc_1 = V_1 \frac{dc_1}{dt} \quad (2)$$

$$vc_1 - vc_2 = V_2 \frac{dc_2}{dt} \quad (3)$$

$$vc_2 - vc_3 = V_3 \frac{dc_3}{dt} \quad (4)$$

Residence Times

Not all the liquid exiting the tank spends the same amount of time before it is discharged through the exit. Because of the turbulent mixing, some of the entering molecules will find their way rapidly to the exit, while others will linger much longer in the tank before they, too, pass through the exit. Indeed, there is a whole range of possible residence times, a representative statistical distribution for which is shown in Figure 2.

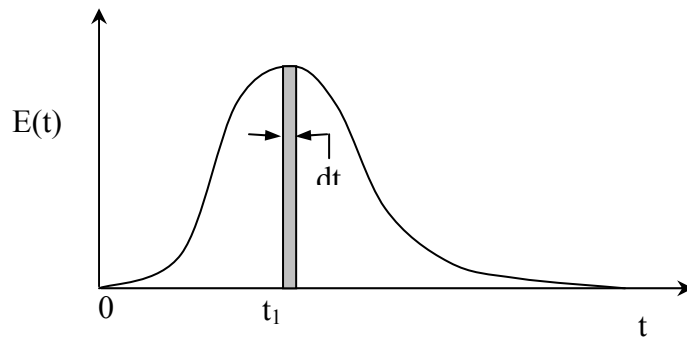


Figure 2 Residence Time Distribution Function (The "E" Curve)

The residence time distribution function is defined such that $E(t)dt$ is the fraction of the total molecules that reside in the tank for a time between t and $t+dt$. Clearly, since all molecules must eventually leave the tank if we wait an infinitely long time:

$$\int_0^{\infty} E(t)dt = 1 \quad (6)$$

Knowledge of residence times is important because it bears directly on the mixing performance of a given reactor. The longer time a molecule has to remain in the reactor, the better chance it will have to react.

It may be shown (Fogler, p. 814) that the "E" curve can be generated from *the impulse tracer data* using the following expression:

$$E(t) = \frac{C(t)}{\int_0^{\infty} C(t)dt} \quad (7)$$

Another useful characteristic of the "E" curve is that it can be used to determine the fraction of the molecules leaving the vessel that have resided in it for a certain amount of time:

$$\int_0^t E(t)dt = \left\{ \begin{array}{l} \text{the fraction of molecules in the effluent that} \\ \text{have resided in the reactor for less than time, } t \end{array} \right\} = F(t) \quad (8)$$

This function, based on the "E" curve, is called the "F" curve. It is called the cumulative distribution function. $F(t)$ always lies between 0 and 1.

The *step tracer data* directly provides the “F” curve (see, p. 818, Fogler 3rd edition):

$$\left[\frac{C_{out}}{C_0} \right]_{step} = \int_0^t E(t) dt = F(t) \quad (9)$$

By differentiating the above expression, one obtains the RTD function $E(t)$:

$$E(t) = \frac{d}{dt} \left[\frac{C(t)}{C_0} \right]_{step} \quad (10)$$

We can also calculate the mean residence time, τ , for the tanks from the "E" curve:

$$\tau = \frac{\int_0^{\infty} tE(t) dt}{\int_0^{\infty} E(t) dt} = \int_0^{\infty} tE(t) dt \quad (11)$$

Levenspiel (1972, p.291) gives the following theoretical expression for the "E" curve for identically sized vessels:

$$\tau E(t) = \left(\frac{t}{\tau} \right)^{N-1} \frac{1}{(N-1)!} e^{-\frac{t}{\tau}} \quad (12)$$

where N is the number of identically sized vessels in series

$E(t)$ for a tubular reactor in laminar flow

$$E(t) = \begin{cases} 0 & t < \frac{\tau}{2} \\ \frac{\tau^2}{2t^3} & t \geq \frac{\tau}{2} \end{cases} \quad (13)$$

The mean residence time t_m is

$$t_m = \frac{\tau^2}{2} \left[-\frac{1}{t} \right]_{\tau/2}^{\infty} \quad (14)$$

Apparatus

The continuous flow dye mixing system was shown in Figure 1. The major components are as follows:

Dye Feed Tank

This is a 20 L polyethylene tank holding a weak solution of methylene blue, approximately 20 mg/L.

Dye Feed Pump

This is a magnetically-driven centrifugal pump that provides a relatively constant pressure feed of dye solution to the automatic control valve. This pump should not be run when the impulse injection runs are being made.

Dye Control Valve

This is a needle valve, of range 8 turns, driven by a 400 step per revolution Arrick stepper motor. Thus the full range of the valve is 3200 steps. This range can be covered in about 10 seconds. For the RTD experiments, the valve has been placed in the wide open position. You will not adjust the valve position during the experiment.

Water Pressure Regulator

The regulator is connected to the water feed line. It is used mainly to reduce the effect of lab water pressure variations on the water feed rate to the experiment. A pressure at the inlet to the water flow control valve of say 30 psig is sufficient to cover the desired flow rate range.

Water Control Valve and Rotameter

The rotameter (Fisher Scientific) has an upper limit of about 1400 ml/min, and is connected in series with a valve used to set the flow rate. A glass float (black) is used for low flows, and a stainless float is used for higher flows. It is recommended that you calibrate the flow meter with a graduated cylinder and stop watch prior to starting the experiment.

Impulse Injector (note this device does not function well and will not be used)

This device consists of a U-shaped aluminum block in which slides an I-shaped acrylic block containing two passages (o-ring sealed) which align with passages in the aluminum block. Initially the upper passage is filled with a relatively concentrated dye solution by using a syringe. At time zero of an impulse run the block is snapped sharply downward by finger pressure, inserting the dye filled passage into the water feed stream. O-rings seal the ends of the passages in the acrylic block. A removable acrylic retainer allows removal of the acrylic block for o-ring replacement, if needed. Use of a light lubricant such as Vaseline prevents binding of the sliding block. If the impulse injector has to be removed for lubrication or cleaning, it can be replaced by a 6" length of 1/4" tubing provided with the experiment. In this case the 5 ml syringe can be used for impulse injection, connecting it to the tubing and ball valve assembly located upstream of the impulse injector.

Syringe Injector for Impulse Injections

A 50ml syringe will be used for the impulse runs. With the valve connecting the syringe to the process closed, the valve to the small dye tank is opened. The syringe is filled with dye from the reservoir. Be careful not to get too much air in the syringe during this process. Close the valve to

the reservoir. Impulse injections of about 5ml can now easily be made by opening the valve to the process, injecting the dye quickly and then closing the valve.

Flow System

The flow system consists of three borosilicate Erlenmeyer flasks of nominal volume 250 ml and a cylindrical tube of 150 ml. Each flask is equipped with an o-ring sealed Teflon plug. Ball valves allow the flow to (a) pass through the three vessels in series, (b) pass through only the last vessel, or (c) pass through the tube flow reactor. Each configuration has its own residence time distribution, which of course is a function of flow rate. The flask inlet and outlet lines are designed so that air bubbles will pass through the first two flasks and collect in the third flask, which is equipped with an air vent line. If the Tygon outlet line is pinched and the ball valve in the vent line is opened, air in the top of the last flask can be removed before a run. This flask then acts as a bubble trap during the run and prevents air bubbles from entering the spectrophotometer cuvette. A valve is provided to vent air from the flask. Note: the friction of the two o-rings is sufficient to prevent the flasks from disengaging from the plugs. If more friction is needed, either or both o-ring may be replaced by o-rings of the same (1/16") size but larger diameter.

Spectrophotometer and Cuvette

The concentration of dye in the effluent from the flow system is determined by a Spectronic Model 20 spectrophotometer (Spec 20). The Spec 20 is equipped with a specially designed long-path flow-through cuvette. A light beam passes through the cuvette, to a grating that selects the wavelength (normally 640 nm), and then to a photodetector. The photodetector signal is displayed as transmittance on an analog meter on the Spec 20, and also converted to a voltage which is sampled and digitized by the A/D board in the computer. In the computer the dye concentration (dimensionless) is computed using the equation

$$C = 1000 \ln\left(\frac{890}{V}\right) \quad (15)$$

where C is concentration and V is voltage (millivolts) generated by the Spec 20.

PROCEDURE

Inspection and Initialization

The apparatus should be examined carefully. Check that the effluent water line from the cuvette is connected to the drain and that the cable to the Spec 20 is plugged into the Jones connector on the bottom of the Spec 20. Turn on the power strip, make sure the pump is off until a step change run is to be made. Turn on the computer. Open the folder **Dynamics and Control of Dye Mixing** located on the desktop. Double click the Lab View software icon in it, to launch the software for the experiment.

Calibration of the Water Flow Rotameter

With the ball valves set to bypass the first two vessels, set the water rotameter to 30 (steel ball, read the center of the ball), and record the time needed to collect 250 ml of effluent water in a graduated cylinder. Then increase the water flow rate in increments of 20 to 150 and measure the water flow rate (ml/min) at each setting. The water rotameter is now calibrated.

Spectrophotometer Calibration

Set the Spec 20 to 640 nm, a good wavelength for detecting methylene blue. When all traces of blue dye and all bubbles have been washed from the cuvette (running at a low flow rate will remove even small bubbles), and with the light shield in place, use the lefthand black knob to turn on the Spec 20. Lift the cuvette until a shutter inside the Spec 20 falls, and set the needle of the meter to 0% transmittance. Replace the cuvette, and use the black knob on the lower right to set the transmittance to 100%. The Spec 20 is now calibrated.

Tracer Runs

Select the desired flow path, one tank or three. For an impulse run, make sure the dye pump is off and the ball valve on the pump inlet is closed. Load the syringe injector as described above. For a step test, turn the pump on, but leave the ball valve on the pump inlet closed. For step runs, make sure the valves on either side of the syringe are closed. Adjust the flow rate to a specified value using the rotameter control valve. Access the LABVIEW software on the computer interfaced to the spectrophotometer by clicking on the appropriate icon on the desktop of the computer monitor. Enter the number of tanks used and the rotameter reading in the designated cells on the display screen, and hit the “start (Arrow button)” *right after* injecting the dye. Save the excel file by the group name with a file extension *.xls. For outlet concentration to reach zero, use sufficient number of data points and a suitable sample interval. The sample interval parameter is typically selected as 0.2 to 0.5 seconds. For runs at higher flow rates, say 1000 ml/min, using only one vessel, 0.2 seconds is good value. For runs at lower flow rates using three vessels, values of 0.5 to 1.0 seconds may be needed. When you hit enter, you will see the effluent concentration plotted on the screen. The program terminates after specified time intervals. If the program terminates before the effluent dye level has returned to zero, the interval should be increased. After the program terminates, the file will be saved as an excel sheet in the **Dynamics and Control of Dye Mixing** folder located on the desktop. The excel sheet will have the information of concentration of dye at various time and will also provide values of the

integrals $\int_0^{\infty} c(t)dt$ and $\int_0^{\infty} tc(t)dt$ also.

A series of runs should be made, at rotameter readings of say 30, 60, 100, and 120. This series should be repeated with the ball valves set to send flow through three vessels in series and then for flow through the tubular reactor. (At high flows with three vessels in series, a more concentrated solution of dye may be used to fill the injector.) Since each run should take no more than 10 minutes, about 90 minutes should suffice for say 8 to 10 runs. You may want to repeat a run several times to test reproducibility. You should perform runs for both step and impulse tracers.

SAFETY

Guard against electrical hazards by making sure that all equipment is well grounded using three-wire plugs and other means. All components should be plugged into the power strip attached to the experiment, since this is equipped ground-fault current interrupter. **THINK FIRST OF SAFETY IN ANY ACTION YOU TAKE.** If not certain, ask the TA or a faculty member before you act. Note that methylene blue dye is relatively innocuous, although it does stain the skin. Soap and hot water remove the blue color from the skin.

DATA ANALYSIS

1. For the single tank impulse runs, calculate and plot the $E(t)$ curves using Equation (7). Compare the impulse run results to the theoretical $E(t)$ curve that you would predict from theory using Equation (10) for $N=1$.
2. Estimate the mean residence time for the one tank system using the RTD data and Equation (9) for your impulse runs. How do these results compare with the residence time predicted based on the system flow rate and the volume of the tank?
3. Repeat 1 and 2 for the three-tank system.
4. For the PFR, plot the $E(t)$ curve using Equation (7), and compare it with Equation (11).
5. Derive $E(t)$ and t_m given in Equations (11) and (12) for a tubular reactor.
6. Estimate the mean residence time for the TFR using the RTD data and Equation (12) for your impulse runs. How do these results compare with the residence time predicted based on the system for rate and the volume of the tanks?
7. Explain why the Equations (11) and (12) describe the experimental data better at lower flow rates, and how to improve them for higher flow rates.
8. Describe detailed observations of mixing patterns in the reactors, and compare the patterns. What are the unique differences of mixing pattern between the CSTR and TFR, between one and three tank CSTRs, and between the horizontal and vertical TFRs. What do you learn from the observation?
9. Based on your data analysis and comparisons, suggest how you improve the experiments.
10. Based on your observation and data analysis, suggest how you improve the mixing. What are the important things you must consider to obtain good mixing?