COMPUTER SIMULATION OF TRACER INPUT EXPERIMENTS

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Incorporating a personal computer in the classroom has brought a new quality to the study of chemical engineering that was unthinkable just a few years ago. Due to its mathematical calculation powers, the computer allows us to study complicated experimental systems that are unapproachable from an analytical point of view, and its rapid results are especially beneficial in chemical engineering. Experimental results of several systems can be simulated by using the theoretical equations that govern the process. This methodology permits us to quickly analyze the influence of different variables that affect the system behavior without the long and hard experimental testing that can distract a student.

This paper proposes a practical class for undergraduates in their first year of chemical engineering where the characterization of a reactor is achieved through recording the residence time distribution (experimental response obtained from a typical test stimulus-response for the hydrodynamic characterization). The distributions are simulated with a theoretical model allowing the quantification of the parameters characterizing the reactor behavior as a function of the operating conditions (flow rate).

Specifically, this paper provides the particulars for a practical session using a versatile computer program that simulates the tracer input experiments. It is necessary, however, that the students have prior knowledge of reactor design theory. This background material should have included the ideal reactor models and the solutions to problems where several curves were analyzed using the momentum method or through simple iteration.[1]

The practical lesson described here has been designed for two students working together for a period of approximately four hours. The students should have some knowledge of how to program in the BASIC language, but it is not absolutely necessary. The experimental work is short and repetitive, so the students can center their attention more completely on the simulation. This obliges the students to make decisions concerning the design and plan of the lesson in the following ways:

- ▶ *They must elect the number of flow rates to study.*
- ▶ *They must perform small modifications in the BASIC program in order to extend its application (with instructor supervision).*

Finally, a student must hand in a report detailing the experimental procedure, its results, the conclusions gained, and the important and useful modifications that were made to the BASIC program. Once the reports have been presented and the program modifications detailed, the instructor can propose extending the program to the study of even

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more complicated systems.

The main objective is to demonstrate how the personal computer can help in the quantitative characterization, design, and scale of a reactor—a matter of some difficulty when other techniques are used. From the technical point of view, it is interesting to identify the flow pattern, to analyze the reactor behavior when the flow rate is varied, and to classify the reactor when the objective is to optimize the chemical process inside the reactor.

BACKGROUND AND THEORY

There are two standard models for the ideal behavior of a continuous chemical reactor: the piston plug flow and the backmixed reactor (widely studied in specialized literature[2,3]). Nevertheless, real reactors show deviations from this ideal behavior for a wide number of situations. A variety of theoretical approaches have developed in the literature for this non-ideality.

The dispersion model for a tubular reactor is presented by Levenspiel[2] in considering a hydrodynamical behavior with plug flow and axial dispersion:

$$\frac{\partial C}{\partial t} = D_{ax}\frac{\partial^2 C}{\partial z^2} - v\frac{\partial C}{\partial z} \tag{1}$$

where

C	concentration of property measured
t	time
z	length in the flow direction
v	linear flow rate
D_{ax}	axial dispersion coefficient

Equation (1) is usually expressed in dimensionless form as

$$\frac{\partial C}{\partial \theta} = -\frac{\partial C}{\partial Z} + \frac{1}{Pe}\frac{\partial^2 C}{\partial Z^2} \tag{2}$$

using the Peclet number, $Pe = vL/D_{ax}$, where L is the total length of the reactor in the flow direction. The dimensionless time, θ, is defined as

$$\theta = \frac{t}{\tau} \tag{3}$$

τ, the mean residence time, is

$$\tau = \frac{L}{v} \tag{4}$$

Z is the dimensionless length (=z/L), and C is the normalized concentration of the measured property (C is the conductivity in this paper).

The solution of Eq. (2) depends on the boundary conditions for the input and the output.[4] Some of these solutions are analytical. For the resolution of Eq. (2) it is interesting to consider whether or not the dispersion degree is high. On the other hand, the boundary conditions must be kept in mind, *i.e.,* if the system can be considered open or closed. Table 1 shows a scheme of the different cases that can be presented.

This dispersion model defines with great exactness several practical situations, especially when the shape of the experimental curves is Gaussian with a high symmetry (if the dispersion degree is low) or with a tail (if the dispersion degree is high).[5]

The model presented in this paper considers a more global non-ideal situation: coexistence of a dead volume inside the reactor and two flow paths, each modeled with an axially dispersed plug-flow model. Thus, Eq. (2) is the differential equation defining the dispersion model for each flow. In this paper, we considered the most common situation: open-open flow (inlet and outlet do not change the flow pattern) and a high axial dispersion degree. Equation (2) has, in this case, an analytical solution. The solution for the residence time distribution is, for each flow,

$$C(\theta) = \frac{1}{2}\sqrt{\frac{Pe_i}{\pi\theta_i}}\exp\left[-\frac{Pe_i}{4\theta_i}(1-\theta_i)\right]^2 \tag{5}$$

Other models are available in the literature as the simple tanks in series model[5] with just one parameter, or more complex multi-parameter differential models.[6] The former is not attractive from the didactic point of view, since it is less intuitive. The latter allows a better explanation of the mass transport phenomena, but it must be used only when there is evidence of such phenomena, or when the simpler models are not able to reproduce the experimental data.

TABLE 1

Different Models for Non-Ideal Flow

	High Dispersion Degree	*Low Dispersion Degree*
Open/Open	$C(\theta) = \frac{1}{2\sqrt{\pi\theta / Pe}}\exp\left(-\frac{(1-\theta)^2}{4\theta / Pe}\right)$	
Closed/Closed	Numeric integration. Boundary condition equations: $Z = 0 \quad \text{then} \quad \delta(\theta) = C - \frac{1}{Pe}\frac{dC}{dZ}$	$C(\theta) = \frac{1}{2\sqrt{\pi / Pe}}\exp\left(-\frac{(1-\theta)^2}{4/ Pe}\right)$

EXPERIMENTAL DESIGN AND RESULTS

In the proposed simulation model (see Figure 1), there are two flows and a dead volume, with five fitting parameters to optimize. The five parameters are: two mean residence times, two Peclet numbers, and the ratio between the dead volume and the total volume (V_d/V). From these parameters the ratio between the volume used by flow 1 (flow rate = Q_1) and the total volume (V_1/V) can be calculated by using

$$V = V_1 + V_2 + V_d \tag{6}$$

$$Q = Q_1 + Q_2 \tag{7}$$

$$\tau_i = \frac{V_i}{Q_i} \tag{8}$$

The optimization method used is the Flexible Simplex method[7] and the objective function (O.F.) is the sum of the square of the differences between experimental and calculated values of the residence-time distribution E(t)

$$\text{O.F.} = \sum \left[E_{calc}(t) - E_{exp}(t) \right]^2 \tag{9}$$

The computer program estimates the best parameters to fit the experimental data, provides them, calculates the E(t) curve, and uses the value of the objective function.

DESCRIPTION OF THE EXPERIMENTAL SYSTEM

Figure 2 shows a schematic diagram of the experimental system used to measure and analyze the stimulus-response curve. The procedure is based on the instantaneous modification of a property of the fluid in the reactor inlet and recording the variation of this property with time at the outlet of the reactor. In this case, the electric conductivity of the fluid is modified by the injection of a small volume of saturated solution of the tracer. This response will be the data file for beginning the parameter-estimate calculation. Of course, the data acquisition could be computerized and the data analyzed on the same computer.

Any kind of reactor could undergo this analysis. The sole condition for the reactor is to know the total volume. In the practical example that follows, a built-in-house filter-press electrochemical reactor (UA200.08) is used. The compartment for the fluid is a drum of dimensions 18x12x0.8 cm. A more detailed description of this reactor can be found elsewhere.[8]

The design of the hydraulic part of the system is a typical configuration: a deposit for the fluid (water), pumps, and flow-rate measurement units controlled by valves permitting flow-rate adjustment in each experiment. More details can be found in the literature.[9]

EXPERIMENTAL DEVELOPMENT

Previous to the Experiment • Before experimental recording of the curves, it is important to calibrate the measurement apparatus. The conductivity probe and the flow-measurement units must be calibrated for the fluid and temperature of the experiment. An erroneous measurement of the flow rate would lead to an overflow situation. In this particular case the students must calibrate the probe, so the flow rate calibration is given to them. It is important to keep the solution temperature constant during the experiment because conductivity depends on the temperature. This is attained with a thermostat heat exchanger in the reservoir.

Curve Response Measurement • Once the flow rate, the temperature, and the other conditions are stabilized, the pulse of tracer can be rapidly injected, collecting the conductivity-time data shown in Figure 2. The injection of the tracer must be done as close as possible to the reactor inlet. In this case, the tracer was 2 mL of a saturated KCl solution (4.3 M). A conductivity probe (Ingold®) measures the conductivity of the outflow stream reactor. This probe is connected to a conductivity meter with an analog output of 0 to 10 V (error

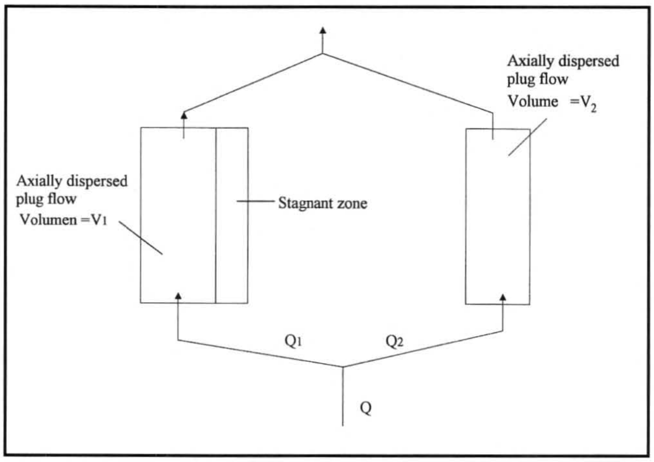

Figure 1. *Sketch of the model for flow characterization.*

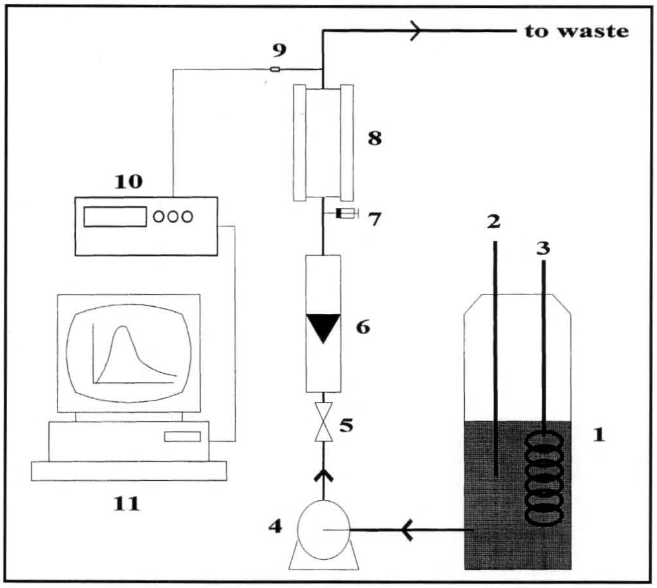

Figure 2. *Diagram of the experimental set up: 1. reservoir; 2. thermometer; 3. heat exchanger; 4. pump; 5. valve; 6. flowmeter; 7. injection of tracer; 8. reactor; 9. conductivity probe; 10. conductimeter; 11. computer.*

less than 1%). The conductivity meter, through a data acquisition card (12 bits, monopolar channel, input free tension 0-10V), permits saving the data in an ASCII file in the computer (PC-compatible system, at least 80386 processor, 2 MB RAM memory). Recording the response must be done until the value of the conductivity signal reaches the initial value. This simple procedure is repeated for every flow rate studied.

Analysis of the Response • The computer program proposed in this paper needs the data collected in an ASCII file with names RV**.GEX, where the flow rate must be included. If the experimental system is similar to the one in this paper (with the data acquisition card), the conductivity-time curve will be recorded automatically by the computer. The experiments can also be done by simply recording the analog X-t curve and digitizing it.

The computer program is written in BASIC; a copy of it can be requested by e-mailing JA.CONESA@UA.ES. The program needs the reactor dimensions, the name of the data file, the flow rate, and the initial value of the parameters to be fitted (in part II of the program).

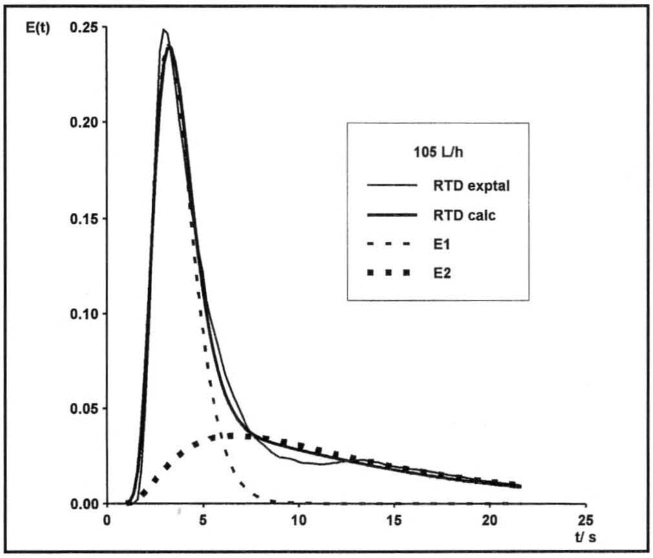

Figure 3. *Superposition of experimental and calculated curves. Volumetric flow 105 L/h.*

TABLE 2
Estimating Model Parameters for Different Volumetric Flow Rates

$Q_V/L\ h^{-1}$	τ_1 / s	Pe_1	τ_2 / s	Pe_2	V_1/V	V_D/V	O.F.
66	3.84	15.46	12.72	2.76	0.33	0.42	$1.5\ 10^{-3}$
105	3.38	20.11	8.74	3.58	0.43	0.21	$5.0\ 10^{-3}$
155	2.41	22.38	6.65	3.39	0.43	0.10	$3.3\ 10^{-3}$

The solution is not strongly dependent on the initial values of the parameters. Thus the initial values for the mean residence time could be selected as the ratio between the total volume and the flow rate (the same value for both ways). On the other hand, typical values of the Peclet number are between 1 and 50, and the initial value of V_d/V could be selected as 0.5.

When these initial parameters, experimental conditions, and response curve values are entered, the program can be run. Using the initial values of the parameters, the successive iterations will diminish the value of the objective function. After each iteration, the program shows the new value of the parameters, the current value of the objective function, and the minimum value O.F. achieved until this iteration.

The simulation ends when the value of the O.F. minimum reached is repeated and the variations of the parameter values do not change. The program generates an output data file (with ASCII format and name RV**.CAU) with five columns of data: time, experimental residence time distribution, calculated residence time distribution, calculated residence time distribution for flow through path 1, and calculated residence time distribution for flow through path 2. In addition, the optimized parameters are also saved at the end of the output file (Pe and average residence time for each path, and V_1/V), and with the value of the dead volume (V_d/V) and minimum O.F. reached.

ANALYSIS OF RESULTS

The experiments were carried out with three different flow rates (66, 105, and 155 l/h). Figure 3 shows the experimental and calculated E(t) curves, $E_1(t)$, and $E_2(t)$, respectively, for the flow rate 105 l/h. The optimized parameters for the three experiments are shown in Table 2. In the Table, "Q" is the flow rate, τ_i is the average residence time of the path "i", and V_d/V and O.F. were defined previously.

Analyzing the results, we can conclude:

- An increase in the flow rate produces a decrease of the residence time in both flow paths, favoring path 1 (of minor residence time and a higher Pe, *i.e.*, greater plug flow behavior) versus path 2, with low Pe. This conclusion is obtained from the value of the parameter V_1/V. The flow circulating through path 1 can be calculated using the relationship between the average residence time and the flow rate (Eq. 8).
- The flow rate also affects the percentage of dead volume, decreasing as the flow rate increases. This is a very important conclusion.

DISCUSSION

The practical lesson proposed in this paper allows the

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fall exactly on the line. If $R^2 = 0$, then there is no fit whatsoever; there is no correlation between X and Y.

Shady ethics enter when R^2 is used to determine the goodness of fit without also including the plotted data, or without using common sense. Figure 15 shows how statistical correlation can be subverted.[2] The four data sets can be plotted to yield exactly the same $R^2 = 0.67$. Each data set has the same mean and is described by the same least-squares equation. And yet the data, when plotted and viewed on the plots, represent four totally different populations. Only by plotting the data would the reader understand the actual relationship between the two variables.

CONCLUSION

Depicting the truth by avoiding both lies and deceptions in engineering graphics is invariably the safest course of action for engineers. Or, as Mark Twain suggested, "Always tell the truth. That way you don't have to remember what you said."

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Tracer Input Experiments

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student to understand and complete the theoretical lessons concerning chemical reactor design. The versatility and rapidity that comes with using a personal computer has excellent pedagogical aspects. It must be remembered, however, that it is necessary to refer constantly to the suitability of using more simple models (lower number of parameters), making some of the variables in the program constant. In this way, we avoid the problem of using models with more parameters than degrees of freedom of the system.

The time necessary for recording the responses and simulation of one curve of residence time distribution, once the preliminary steps are finished, is estimated to be between 10 and 15 minutes for a reactor of dimensions similar to the one described in this paper. This allows the student to perform a series of eight different flow rates in two hours, using the first hour to prepare and calibrate the system as well as to prepare the tracer solution. The last hour can be used to discuss different aspects with the teacher, proposal of program modifications, and other applications.

CONCLUSIONS

This class was designed for students to familiarize them with the concepts of reactor design and characterization. The reasonably good agreement between experimental and calculated values of the RTD makes them feel confident about applying engineering concepts.

The students find the experimental procedure relatively uncomplicated and possible to complete within the laboratory period. Using personal computers to study an electrochemical reactor rather than simply studying the theoretical concepts provides better comprehension of the reactor flow pattern and the model development.

It is important that the theoretical concepts be explained in class before the students attempt the laboratory exercises. Operational problems also become clear while the students are performing the experiments. For example, the importance of rapid injection of the tracer was discovered by several students who found that the response was "abnormal" in the sense that many peaks were found when non-instantaneous modification of the conductivity was achieved in the input.

Another important concept involved in this practice is the optimization method and its structure in the BASIC program. Students appreciate when someone explains how to run an optimization method such as the Simplex used in this lab session.

Students find the session interesting and enjoyable, and they relate well to the engineering principles involved. The lesson allows them to perform and validate what they have learned in class.

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