LOW-COST EXPERIMENTS IN MASS TRANSFER
Part 4. Measuring Axial Dispersion in a Bubble Column

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The bubble column is one of the most efficient methods of gas-liquid contacting, and it is simple, comprising merely a vertical column and a means of distributing gas into the liquid at the base of the column. Deckwer[1] has given a detailed account of bubble columns and their usefulness as gas-liquid reactors as well as mass transfer devices. Bubble columns are often used in organic gas-liquid reactions such as hydrogenation, chlorination, and alkylation. An important application is in the "liquefaction" (hydrogenation) of suspended coal particles at high temperature and pressure.

Bubble columns are often operated countercurrently, with the liquid fed to the top and the gas distributed to the base of the column. The full benefits of countercurrent operation can only be realized if both phases move in plug flow,[2] but there is evidence of considerable axial mixing in the liquid phase of bubble columns.[1] With axial mixing, the mean driving force for mass transfer is less than it would be in plug flow.[2]

The effect of liquid-phase axial mixing on countercurrent mass transfer can be expressed in terms of the Peclet number

$$Pe = UL / E \qquad (1)$$

where U is the superficial velocity of the liquid phase, L is the active height of the gas-liquid dispersion, and E is the axial dispersion coefficient. If the Peclet number is greater than 20, plug flow of the liquid can be assumed for engineering design purposes.[3] A Peclet number less than about 0.05 will correspond to well-mixed liquid behavior; that is to say the solute concentration is essentially uniform throughout the column. The terms U and L are well defined, but the

axial dispersion coefficient E must often be measured before Pe can be estimated. Therefore, the measurement or prediction of E is an important step in the design of a bubble column contactor or reactor.

The axial dispersion coefficient E is defined in terms of the axial dispersive flux (N) of a solute:

$$N = -E \frac{\partial c}{\partial z} \qquad (2)$$

Although in this equation E is analogous to the molecular diffusion coefficient, it is determined by bulk liquid motion such as turbulence, circulation, or motion of bubble wakes. Its value is therefore independent of the molecular structure of the solute. Because of its dependence on bulk fluid motion, E can be many orders of magnitude greater than molecular diffusion coefficients. Typically, E is in the range of 1 to 100 cm^2/s, compared with about 10^{-5} cm^2/s for molecular diffusion coefficients in liquids at ambient temperatures.

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The objective of this experiment is to provide quantitative data on unsteady axial dispersion as well as a visual observation of the turbulent mixing phenomenon in a small laboratory bubble column. The experiment uses an acid-base-indicator system that adds some complexity to the data analysis but eliminates the need for sampling and analysis of liquid at different times and locations.

MEASUREMENT OF E

Figure 1 shows the experimental bubble column schematically. Axial mixing can be measured by the well-known pulse tracer addition method[3] in which the spread of the initial pulse of tracer is determined by the unsteady diffusion equation, assuming that no bulk flow is present:

$$\frac{\partial c}{\partial t} = E \frac{\partial^2 c}{\partial z^2} \tag{3}$$

The conventional tracer method requires sampling the liquid at one point (preferably more than one point) in the column, followed by analysis of the samples. This is too time consuming for most undergraduate experiments. In this experi-
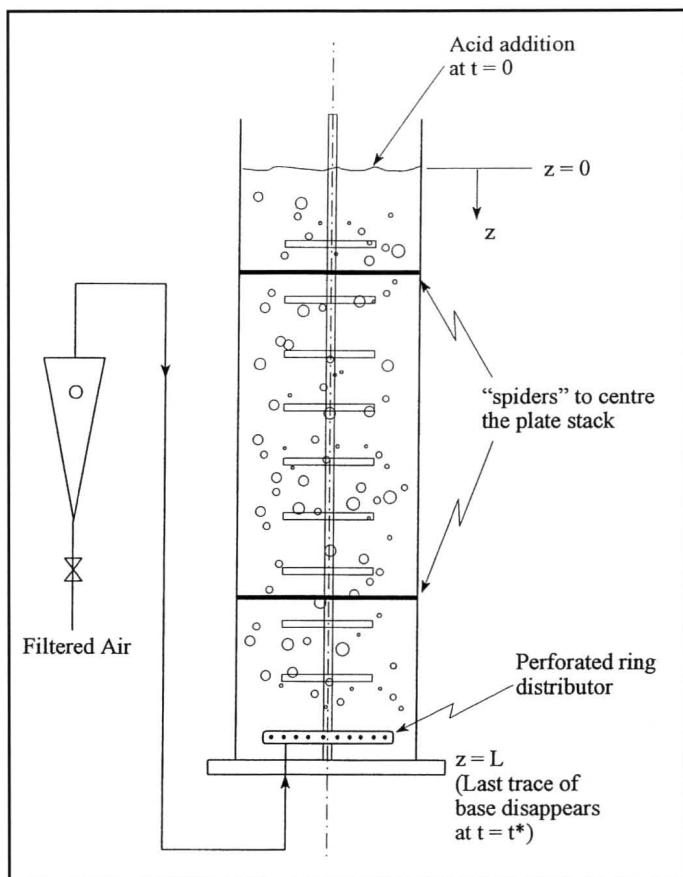
Figure 1. *Schematic diagram of bubble column showing coordinate system for equations. The baffle stack can be removed for "open column" experiments.*

ment, the tracer added is an acid that reacts very rapidly with a base, which is initially uniformly distributed through the liquid in the presence of a suitable indicator. The change in color of the aqueous solution as the pH passes through the neutral point is easily observable, and the movement of the color change through the column can be timed and related to the solution of Eq. (3) so as to give calculated values of E at various conditions.

METHOD AND THEORY

The column in Figure 1 is initially filled with a dilute alkaline solution at a molar concentration c_{b_1}. We recommend diluting exactly 50 mL of 1.0 mol/L NaOH into distilled water in the column (volume about 40 L), with a few mL of phenolphthalein solution to indicate alkalinity. The concentrated "tracer," added to the top of the column at t=0, is a measured volume of 1.0 mol/L hydrochloric acid, which is *more than sufficient* to neutralize the sodium hydroxide. Observation of the column after the addition of acid shows that the red color of the alkaline phenolphthalein starts to disappear at the top of the column; the reaction zone (color change) then moves downward until the last trace of red finally disappears at the base of the column.

The free acid and base mix according to Eq. (3), which is written for each solute, is

Where free acid is present

$$\frac{E}{(1-\varepsilon)} \frac{\partial^2 c_a}{\partial z^2} = \frac{\partial c_a}{\partial t} \tag{4}$$

Where free base is present

$$\frac{E}{(1-\varepsilon)} \frac{\partial^2 c_b}{\partial z^2} = \frac{\partial c_b}{\partial t} \tag{5}$$

The term in holdup (ε) is a correction factor for the volume of gas in the dispersion.

Note that E is the same for acid and base. The acid-base reaction is extremely fast and takes place on a 1:1 mole ratio at a zone where c_a and c_b are both very small (pH about 7). The reaction (color change) zone is assumed to be a very thin boundary at which

$$z = z_r \tag{6}$$

$$c_a, \ c_b \to 0 \tag{7}$$

$$N_a = -N_b \quad \text{or} \quad -\frac{\partial c_a}{\partial z} = \frac{\partial c_b}{\partial z} \tag{8}$$

The chemical reaction is confined to a very thin zone ($z \cong z_r$ and $c_a \cong 0 \cong c_b$). At other parts of the column there is no chemical reaction, and therefore Eqs. (4) and (5) do not contain a reaction term. This type of assumption is well

known in the analysis of gas absorption with instantaneous irreversible reaction.[4]

Equations (4) and (5) can be combined in a way consistent with Eqs. (6-8) by creating a concentration variable c' as follows:

$$\text{For} \quad c_a > 0 \qquad c' = c_a + c_{b_1} \qquad (9)$$

$$\text{For} \quad c_b > 0 \qquad c' = c_{b_1} - c_b \qquad (10)$$

In this way, an equation of the same form as Eq. (3) can be obtained, with $c'=0$ at $t=0$ and $c'_\infty = \left(c_{a\infty} + c_{b_1}\right)$ at $t = \infty$.

The standard solution to Eq. (3) in terms of c' can be shown to be

$$c' = c'_\infty \left[1 + 2\sum_{n=1}^{\infty} e^{-\alpha n^2 \pi^2} \cos\left(\frac{n\pi z}{L}\right)\right] \qquad (11)$$

where, in this case

$$\alpha = \frac{Et}{(1-\varepsilon)L^2} \qquad (12)$$

If the elapsed time is long enough for $\alpha > 0.1$, all terms but the first in the series can be neglected, and Eq. (11) becomes

$$c' = c'_\infty \left[1 + 2 e^{-\alpha \pi^2} \cos\left(\frac{\pi z}{L}\right)\right] \qquad (13)$$

Making the transformation from c'_∞ by Eq. (9), we find

$$c' = \left(c_{a\infty} + c_{b_1}\right)\left[1 + 2 e^{-\alpha \pi^2} \cos\left(\frac{\pi z}{L}\right)\right] \qquad (14)$$

where $c_{a\infty}$ is the final concentration of acid remaining in the column after mixing has been completed ($\alpha \to \infty$).

From the point of view of quantitative measurement, the most accurate quantity is the time t^* it takes for the last trace of red (alkalinity) to vanish at the base of the column. The neutral zone at $z_r = L$ corresponds to $c' = c_{b_1}$, and Eq. (14) becomes

$$c_{b_1} = \left(c_{a\infty} + c_{b_1}\right)\left(1 - 2 e^{-\alpha \pi^2}\right) \qquad (15)$$

$$e^{-\alpha \pi^2} = \frac{c_{a\infty}}{2\left(c_{a\infty} + c_{b_1}\right)} \qquad (16)$$

This equation is more usefully expressed in terms of R, the overall acid/base ratio,

$$R = \frac{c_{a\infty} + c_{b_1}}{c_{b_1}} = 1 + \frac{c_{a\infty}}{c_{b_1}} \qquad (17)$$

Hence,

$$e^{-\alpha \pi^2} = \frac{R-1}{2R} \qquad (18)$$

Taking logarithms and rearranging gives

$$\alpha = \frac{1}{\pi^2} \ell n\left(\frac{2R}{R-1}\right) \qquad (19)$$

Hence

$$t^* = \frac{(1-\varepsilon)L^2}{\pi^2 E} \ell n\left(\frac{2R}{R-1}\right) \qquad (20)$$

If t^* and ε are measured and R and L are known, E can be calculated in a given case. Better still, several experiments can be done under the same conditions of gas flow rate (i.e., same E) but with different values of R. Then the measured values of t^* can be plotted against $\ell n(2R/(R-1))$ to check for linearity. The slope of the plot will provide an average value of E.

APPARATUS

The bubble column used in this project (see Figure 1) is vertical, cylindrical, open at the top, and closed at the bottom. Internal diameter is 8.9 cm and height is 65 cm. The material of construction is clear (methacrylate) plastic. Air is supplied from the 20 psig (230 kPa) line via a rotameter and needle valve. The metered flow of air enters the base of the column via a bubble distributor. A full list of dimensions and operating conditions is given in Table 1. Experiments can be carried out in the absence of column internals, or a stack of baffles (Figure 1) can be placed in the column.

PROCEDURE AND MEASUREMENTS

Before experiments are begun, a check should be made to ensure that the column is mounted vertically, using a spirit level. Mixing can be affected if the column is tilted away from the vertical.

At the beginning of each experiment, the column is filled to within about 10 cm of the top with distilled water. Then, exactly 50 mL of 1.0 mol/L sodium hydroxide is added. The

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TABLE 1
Summary of Experimental Conditions

Column diameter (d)	8.9 cm
Column height	65 cm
Height of gassed dispersion (L)	60 cm (typical)
Number of centrally mounted disk baffles	9
Baffle thickness	0.75 cm
Baffle diameter	5.08 cm
Spacing between baffles	5.08 cm
Values of R studied	1.05, 1.10, 1.40, 2.00
Values of u_g studied	0.83, 1.96, 3.11 cm/s
Gas holdups measured ( )	0.03 to 0.12

alkaline solution is thoroughly mixed by bubbling air for 5 minutes. During this process, about 5 mL of phenolphthalein solution is added to color the solution red. The air flow is started at a desired rate. A volume of stock 1.0 mol/L hydrochloric acid, equal to R times the volume of 1.0 mol/L sodium hydroxide added earlier, is measured out. It is necessary that R>1 in order to completely decolorize the solution.

The mixing process is started by quickly adding the acid (within less than one second) to the gas-liquid dispersion. We suggest that a small deflector (*e.g.*, an inverted, 3-cm-diameter, plastic filter funnel) be used so that the acid can be tipped into the column without creating a strong vertical eddy. As the acid is added, a stopwatch is started. The spread of the neutral zone down the column can be observed. It may be possible to measure zone position as a function of time, but the most important measurement is t^*, the time it takes for the last trace of red color (alkalinity) to disappear from the base of the column. An estimate of error in the t^* measurement should be made.

A measurement of the gas holdup ε is required for each gas flow rate. This is done by comparing the height of the gassed dispersion (L) with the height of the ungassed liquid (L_0),

$$\varepsilon = (L - L_0)/L \qquad (21)$$

The baffles (5.08-cm diameter) were mounted in a stack on a central support rod that was also equipped with two "spiders" to ensure centering in the column. The baffle stack could be lowered into the column from the open top. The baffle stack is shown in place in Figure 1.

SAFETY

The student will be handling 1.0 mol/L solutions of hydrochloric acid and sodium hydroxide. The main hazards are possible ingestion or eye damage from splashes. Skin contact is to be avoided, although at the 1.0 mol/L concentration, the skin hazard is moderate. Goggles should be worn; if any acid/base gets on the skin or clothing, wash exposed area with plenty of water immediately after contact. Rinse the apparatus thoroughly with water after each lab day to prevent corrosion damage. It is particularly important to rinse any metal parts that have been in contact with acid or base.

DATA COLLECTION

On the first day, the column can be operated as an open bubble column without the baffle stack. For a given air flow rate and distributor, the mixing time t^* should be measured for four values of "R." We suggest values of 1.05, 1.10, 1.4, and 2.0. Immediately after this, the four data points should
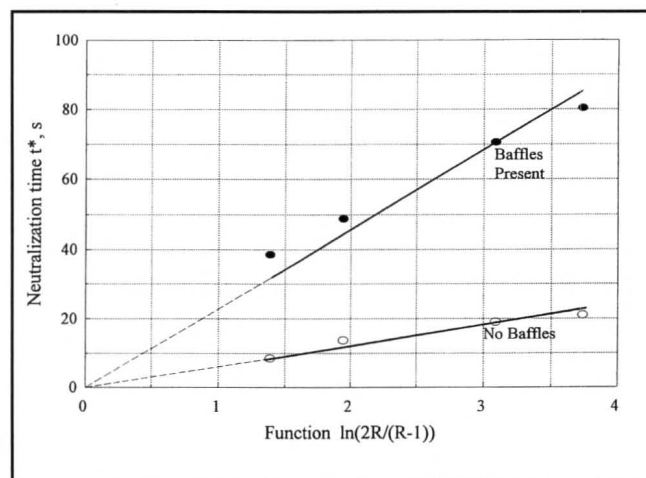
Figure 2. *Typical data plot of neutralization time t^* versus function of R according to Eq. (20). For these data, u_g=0.83 cm/s.*

be roughly plotted as t^* versus the log function of R (Eq. 20). If any of the points appear inconsistent, that particular experiment should be repeated.

Then the air flow rate is changed to a new set value, and four more experiments (varying R) are done. The sequence is continued until three air flow rates have been covered. A total of twelve experiments is expected, allowing approximately 10 to 15 minutes per experiment. Gas holdup is measured for each gas flow rate.

Then, on the second day, another series of measurements is performed, varying R and the air flow rate, with the baffle stack in place. This can be shown to give much less axial mixing than in the open column case.

RESULTS

Figure 2 shows typical data[5] on the effects of the chemical ratio R on the measured neutralization time t^*. Complete neutralization takes longer as the value of R approaches unity (*i.e.*, the abscissa function approaches infinity). The data points at the smallest value of R (typically 1.05) are sensitive to small errors in setting the ratio R, but it can be seen that the data lie reasonably close to a straight line through the origin. This confirms the assumptions leading to Eq. (20). For a given value of R the values of t^* are much greater in the presence of baffles than in the unbaffled condition, which is also to be expected. The slopes of the data plots in Figure 2 are found by linear regression and the value of E can then be calculated from Eq. (20).

For each gas flow setting studied, and for the presence or absence of baffle plates, a plot of the form of Figure 2 is made, and values of E are calculated. Figure 3 shows typical

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final data[5] on the effect of u_g on E, with and without baffle plates. Also shown for comparison on Figure 3 is an equation that has been suggested in the literature[6] for axial dispersion in unbaffled bubble column,

$$E = 0.35(gu_g)^{1/3}d^{4/3} \qquad (22)$$

We can see that the unbaffled column data (open circles) are in the same order of magnitude as predicted by Eq. (22), showing the same trend of a slight increase in E with respect to gas velocity. In the presence of the baffles, E is greatly reduced and the values go through a shallow minimum with respect to u_g. The tendency of E to increase at the lowest values of gas velocity is thought to be due to the formation of trains of gas bubbles following a preferred path up one side of the baffles, resulting in a downflow of liquid on the other side.

GENERAL REMARKS

This experiment can be carried out in as little as one three-hour laboratory period, although two such periods are better for a thorough comparison between the results with and without baffles. The experimental work is not highly demanding, although students must be careful to measure solution volumes accurately, and necessary safety precautions must be followed. It is preferable that prepared standard 1.000 mol/L solutions of acid and base be used; these are available in 20 L quantities from most scientific suppliers.

Students should be encouraged to use computers to examine the time-dependent concentration profiles in the column by means of Eq. (11). Numerical examples can also be developed to confirm the stated assumption leading to Eq. (13), namely that only the first term in the series is important when $\alpha > 0.1$.

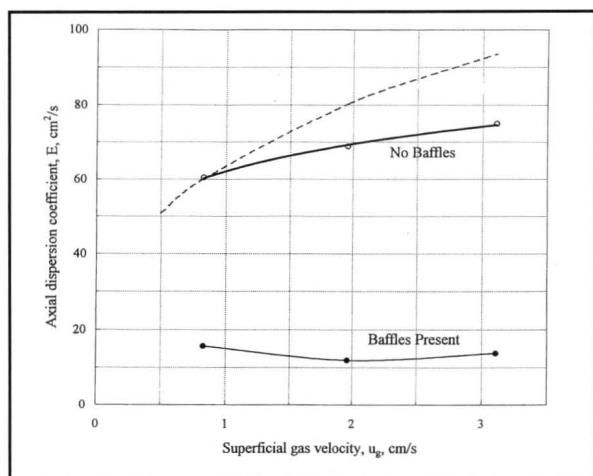As a further exercise, the eddies of color at the neutralization zone can be videotaped in close-up mode. Playing these back at slow speed provides a good visualization of the highly random nature of turbulent axial dispersion.

Our experience over a period of about eight years has shown that this experiment provides students with good insight into axial mixing. The present method offers two advantages over more classical "trace injection," such as salt or dye. First, the complexities of sampling/analysis of the tracer are diminished, allowing more data to be obtained in a given period, and second, the sharp color change boundary allows students to directly observe the mixing process.

ACKNOWLEDGMENTS

NOMENCLATURE

c   concentration, mol m^{-3} or mol/L
c′   concentration variable defined by Eqs (9) and (10), mol m^{-3} or mol/L
d   internal diameter of column, m
E   axial dispersion coefficient m^{2}s^{-1}
g   acceleration due to gravity, ms^{-2} (=9.81)
L   depth of gas/liquid mixture, m
L_0 depth of ungassed liquid, m
n   term in expansion series
N   flux, mol m^{-2}s^{-1}
Pe   Peclet number
R   ratio (mols acid added) to (mols base added)
t   time, s
t^* time for neutralization at z=L, s
U   superficial liquid velocity, m s^{-1}
u_g superficial gas velocity, m s^{-1}
z   axial distance, m (measured downward, see Figure 1)

Greek Symbols
α   dimensionless time, see Eq. (12)
ε   gas holdup (fraction)

Subscripts
a   free acid
b   free base
1   initial value
∞   as $t \to \infty$
r   reaction zone

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Figure 3. *Axial dispersion coefficients plotted versus u_g, with and without baffles. Dashed line denotes Eq. (22)*