Low Backmixing in Multistage Agitated Contactors Used as Reactors

The objective of the study was to test the validity of an available backmix correlation in the low backmix regime required by multistage agitated contactors used as reactors. The correlation was found to be inapplicable, and new equation forms were developed to fit the new experimental data. Keys to low backmix rates are a small annular area around the shaft compared to the column cross-sectional area and an extended annular path length.

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SCOPE

Multistage agitated contactors have potential use as reactors if the interstage baffles are properly designed to minimize interstage mixing. Improper design can lead to low conversions or inordinately long residence times, perhaps impairing product quality. The purpose of this study was to investigate the reliability of a backmix correlation summarizing data available in the open literature and to establish the dependence of backmixing on various design parameters and operating variables.

Tracer pulse testing was used on a five-stage, 15.2 cm dia. × 22.9 cm/stage glass-walled prototype contactor. A ten-stage pilot reactor was designed using data obtained from the prototype. Pulse testing was also performed on the reactor, which has 20.3 cm dia. × 30.8 cm stages. Agitation levels studied were from 3.33 to 13.3 rps. The annular openings around the shaft were varied at annular area to column cross-sectional area ratios of 0.00391 to 0.0143. These ratios are much lower than those found in liquid-liquid extractors. Feed rates developed 0.85 to 9.3 cm/s velocity in the annuli. Annular path lengths were varied from 0.48 to 4.6 cm by using sleeves above each interstage baffle. The viscosity of the liquid in the reactor was varied from 0.52 to 46 centipoise (mPa/s).

CONCLUSIONS AND SIGNIFICANCE

Proper design of the interstage baffling in multistage contactors used in reactors is very critical. A 10-stage reactor deteriorates to the equivalent of a five-stage reactor in performance if the backmix ratio α (backmix rate/feed rate) is 0.59. With α equal to 1.57, performance is equivalent to a three-stage reactor.

The correlation of Haug (1971) developed primarily from liquid-liquid contactor data was found to be inapplicable in the low backmix, high residence time regime. The correlation predicted backmix ratios that were from 4 to 67 times higher than were obtained from a specially-designed glass prototype, and 35 times higher than data obtained from a pilot reactor. The disparity arises because the useful results of the current work were obtained by the use of small annular openings around the shaft and the use of extended annular path lengths.

Experimental data for the contactors having small annular openings and extended annular path lengths were well fit by an equation of the form:

$$\alpha = k_1 \left(\frac{1}{U_a} - k_2 \right) (\text{rps})^2.$$

The variable U_a is the annular velocity and k_2 is the reciprocal of the minimum annular velocity which gives zero backmix for all agitation levels at a fixed annular path length. Similarly a relationship involving annular path length is:

$$\alpha = k_3 \left(\frac{1}{t} - k_4 \right) \frac{(\text{rps})^2}{U_a}.$$

Here t is the annular path length and k_4 is the reciprocal minimum annular path length that yields zero backmix for all agitation levels at a constant ratio a/A of annular area to column cross-sectional area. Additional experimental data is required to unite and generalize the above equations. One possible combinatory form is:

$$\alpha = k_5 \left(\frac{1}{U_a} - k_6 \right) \left(\frac{1}{t} - k_7 \right) (\text{rps})^2$$

where k_6 would vary with annular path length and k_7 would vary with the ratio of annular area to cross-sectional area.

A very limited number of data points suggest that in the regime studied, the backmix ratio is inversely proportional to the square root of the viscosity.

This work presents correlational forms and guidelines for the physical design of multistage contactors operating in the low backmix regime. Development of a general design correlation will require additional building and testing of prototype and pilot units in which the annular areas and annular path lengths are varied systematically. Testing of larger units for the correlation will be limited in practice to pulse testing existing units whose annular area ratios approximate the ratios used in this study.

INTRODUCTION

A large number of reaction stages in series can approximate plug flow, the continuous analog of batch operation. This type flow is desirable whenever there is a need to minimize bypassing by reagents having a small residence time. Figure 1 shows how the total volume of a reaction train of continuous stirred tank reactors (CSTR) can be decreased by increasing the number of stages. To achieve 98% conversion with a reaction exhibiting second-order kinetics, a single CSTR would require 52 times the volume of a batch or plug flow reactor. With six CSTR stages, the required volume is only 1.8 times larger to achieve the same conversion. The importance of stage efficiency is illustrated with these curves. A 10-stage unit whose volume was designed for 98% conversion would result in only 80% conversion if the stages were operating at 50% efficiency. Results are less dramatic for first-order kinetics.

Use of multistage agitated contactors as reactors is advantageous, especially at the pilot level. Each stage can be operated at a different temperature, thereby simulating a nonisothermal batch mode. A multistage contactor with n stages may require a single seal and seal flush with the seal located in a gas cap. A system of n separate reactors in series would require n seals and flushes and has a higher probability of failure. Probability of failure increases further if solids are present and the reactors are run liquid-full. Additionally, the seal flush rate may be nearly as high for pilot size seals as for commercial units, resulting in a dilute reaction system.

Haug (1971) has reviewed and correlated the backmixing data available in the open literature. The bulk of the data was obtained on liquid-liquid contactors and the resulting correlation reflects the design regime where the annular area between the rotating shaft and the hole in the horizontal interstage baffles is a significant fraction of the column cross-sectional area. The correlation has been found to be inapplicable to the low backmix regime required for reacting systems. Some aims of this paper are to present correlation forms that have been found to be useful in the low backmix regime, to note that backmix ratios may have different dependencies on agitation level, to illustrate that the length of the interstage annular opening is an important parameter in this regime, and to quantify the relationship between backmixing and stage efficiency.

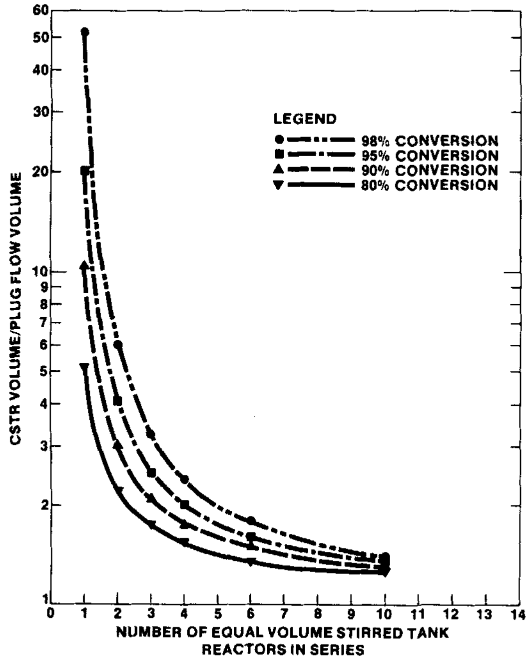


Figure 1. CSTR volume reduction second-order kinetics.

BACKMIXING MODEL

A cascade model of discrete, perfectly matched stages with equal and constant backmix flows is depicted in Figure 2. The finite difference differential equation is for the ith component of a reacting system on the jth stage. A recursive system of $(m-1)\times n$ differential equations is required for a system composed of m components and n stages. The forms of the first and last stages vary slightly from that of the general jth stage.

For a single nonreacting tracer component we have:

$$V_j\frac{dC_j}{d\theta} = (F+f)C_{j-1} - (F+2f)C_j + fC_{j+1}. \qquad (1)$$

The relative variance of the residence time distribution for n stages described by Eq. 1 was developed by Van der Laan and credited as a private communication reference in an article by Overcashier et al. (1959).

$$\frac{\sigma^2}{\mu^2} = \frac{1-x^2-\frac{2x}{n}(1-x^n)}{n(1-x)^2}\,. \qquad (2)$$

The variable x is the modified mix ratio given by:

$$x = f/(F+f)$$

and is related to the backmix ratio α by:

$$\alpha = x/(1-x)$$

The variable α is defined by:

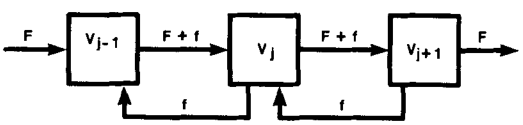
$$\alpha = f/F$$

where f is the backflow rate and F is the feed rate. From Eq. 2 it is seen than the relative variance at zero backmix is $1/n$.

EXPERIMENTAL PROCEDURE

Tracer testing in flow systems has been discussed by Levenspiel (1962) and Bischoff (1966). An inert tracer is fed to the first of n cascaded stages, and the resulting residence time distribution curve is monitored in the nth stage effluent. The analytical procedure that is chosen should have the capability of accurately measuring a 100-fold change in concentration. For ease of analysis, it is useful for the measuring instrument to be linear with concentration. Red ink was chosen for our inert tracer. Several bottles were mixed to yield a uniform lot, and then a concentration calibration curve was obtained on a Beckman Model B Spectrophotometer used in conjunction with a constant voltage transformer.

Figure 3 is a schematic diagram of the glass prototype system used in the initial investigations. The diagram depicts downward flow of fluid, but nearly identical results were obtained in a limited number of replicate tests using upflow fluid. Special measures were taken to maintain the feed rate as constant as possible. A Cuno filter was used in a water supply line connected to city water. A pressure regulator maintained the supply pressure at 2.7×10^5 N/m^2. A Moore Model 635U differential pressure regulator was used to maintain a constant differential pressure across a manually-set control valve. Flow rates were visually monitored with a Brooks Shorate rotameter with tube #R-6-25-B using 0.635 cm stainless steel or Pyrex floats. Flow rates were continuously verified by noting the times required to catch 2 kg of effluent in tared containers on gravimetric balances.



$$V_j\frac{dc_{i,j}}{d\theta} = (F+f)c_{i,j-1} - (F+f)c_{i,j} - fc_{i,j} + fc_{i,j+1} - RATE_j\, V_j$$

$$RATE_j = R(\vec{c}_j, TEMP_j)$$

Figure 2. Material balance with chemical reaction.

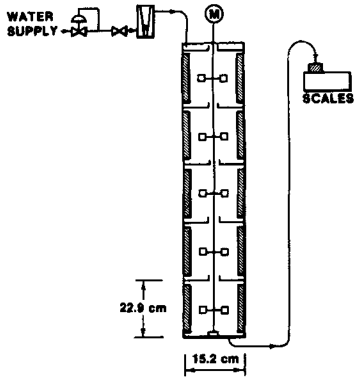


Figure 3. Glass prototype.

Percent relative error $(\sigma/\mu) \times 100$ for the flow rates averaged 2.1% for the five-stage prototype. When the differential pressure controller was added to the pilot unit, the percent relative error was reduced to 1.1%. When converted to relative variance, these error percentages correspond approximately to an α backmix ratio of 0.001.

Trial runs verified the quantity of ink required to pulse the contactors so that the effluent concentration was in the desired range. From 20 to 55 mL of red ink were injected in the feed to the first stage in less than 1.5 min. Tracer addition times were small compared to the 1.25 to 2.5 hours average residence time of the fluid in the prototypes. Average residence time in the pilot reactor ranged from 1 to 8 hours. Sampling frequency of the effluent from the last stage was set at 2 kg intervals and increased to 1 kg intervals near the maximum concentration of the tracer to obtain well-defined curves.

After the inflection point on the backside of the distribution curve was passed, the sampling frequency was decreased to 2 kg between samples. Run times required from three to four displacements to measurably extinct the effluent tracer concentration. It was noted that sometime after the backside inflection, the decay ratio of succeeding readings made at the same time intervals leveled out on a constant value for a given run.

The volume of ink used was calculated from

$$\text{Area} = \int_0^\infty \text{Conc}(\theta)_n d\theta \tag{3}$$

and compared to the actual pulse volume for each run. Integrations were made using the Continuous System Modeling Program III. The first moment about the origin was obtained from

$$M_1' = \int_0^\infty \theta \cdot \text{Conc}(\theta)_n d\theta \tag{4}$$

from which the mean μ is obtained:

$$\mu = M_1'/\text{Area}. \tag{5}$$

Variance σ^2 is defined as the second moment about the mean

$$\sigma^2 = M_2 = \frac{\int_0^\infty (y-\mu)^2 f(y)}{\int f(y)} \text{(normalized)}. \tag{6}$$

By expanding Eq. 6, we can redefine the variance as a function containing a second moment about the origin, where

$$M_2' = \int_0^\infty \theta^2 \cdot \text{Conc}(\theta)_n d\theta \tag{7}$$

and

$$\sigma^2 = M_2'/\text{Area} - \mu^2. \tag{8}$$

Equation 8 is a useful transformation since it allows calculation of the variance in a single pass through a numerical integrator routine without initially knowing the mean.

After calculating the experimental relative variance σ^2/μ^2 for a given run, the modified mix ratio was obtained from Eq. 2 by an iterative procedure.

RESULTS

Mechanical design requirements for the pilot reactor were established by pulse testing a five-stage glass simulator having a 15.2 cm internal diameter and 22.9 cm stage height. Physical descriptions of the prototype variations are presented in Table 1 along with operating variables and experimental backmix ratios. It is noted

TABLE 1. FIVE-STAGE GLASS PROTOTYPE

Run No.	Interstage Baffle Hole Size, cm	Annular Path Length, cm	Annular Area/ Column Area	Annular Velocity cm/s	Agitator Speed, rps	Backmix Ratio, f/F
1	1.59	0.476	0.00391	3.15	6.67	0.77
2	1.59	0.476	0.00391	3.19	6.77	0.75
3	1.59	0.476	0.00391	3.14	8.37	0.94
4	1.59	0.476	0.00391	3.22	10.1	1.14
5	1.59	0.476	0.00391	3.23	13.3	1.48
6	1.59	0.476	0.00391	6.01	6.78	0.28
7	1.59	0.476	0.00391	5.85	13.4	0.69
8	1.91	0.476	0.00868	2.60	13.3	1.85
9	1.91	0.476	0.00868	2.83	6.83	0.49
10	1.91	0.476	0.00868	2.82	13.3	1.46
11	1.91	0.476	0.00868	1.43	6.7	0.97
12	1.91	0.476	0.00868	1.42	13.4	2.88
13	1.91	3.65	0.00868	1.41	13.3	0.13
14	1.91	3.65	0.00868	2.83	6.76	0.055
15	2.22	3.65	0.0143	1.69	6.67	0.055
16	2.22	3.65	0.0143	0.853	13.3	0.50
17	2.22	3.65	0.0143	0.868	10.0	0.24
18	2.22	4.60	0.0143	0.848	13.4	0.36
19	2.22	1.83	0.0143	1.70	6.60	0.20
20	2.22	1.83	0.0143	0.868	13.3	1.46
21	2.22	2.48	0.0143	0.853	13.3	0.91

Stage diameter, T = 15.2 cm
Stage height, H = 22.9 cm
Impeller diameter, D = 7.62 cm
Shaft diameter, d_s = 1.27 cm
Total volume = 20.5 L
Four baffles at wall, 22.5 cm long × 2.22 cm deep × 0.16 cm thick.
Six-blade turbine impellers; blades 1.59 cm wide × 1.43 cm high × 0.16 cm thick.
Support shaft bearing on top stage.

that a tight seal was required between the interstage baffles and the glass wall to eliminate additional interstage circulation paths. The first seven data points in Table 1 are for thin (0.476 cm) interstage baffles having a smaller annular opening. The annular area divided by column cross-sectional area (a/A) is 0.00391. For these data, a plot of the backmix ratio α as the ordinate versus the reciprocal annular velocity as the abscissa yields a family of straight lines having a common nonzero intercept on the reciprocal velocity axis. For these data

$$\alpha = 0.481(1/U_a - 0.08)(\text{rps}). \tag{9}$$

The model fits the experimental data to an average of 3.2% relative error.

Previous correlations have not shown the fine distinction of a nonzero intercept on the reciprocal velocity axis. This partially explains why the available correlations give poor estimates in this region. The correlation of Haug (1971) is

$$\alpha = 0.0098\left[\frac{\epsilon ND}{U_s}\left(\frac{D^2a}{THA}\right)^{1/2}\right]^{1.24} \tag{10}$$

Using this equation for the first seven data points of Table 1, it is found that the correlation yields α's which average 4.5 times higher than the experimental data.

Data points 8–12 of Table 1 were obtained with thin interstage baffles having an intermediate annular opening (a/A 0.00868). Parameter tuning yields

$$\alpha = 0.0711\,(1/U_a - 0.02)(\text{rps})^{1.58} \tag{11}$$

for these data. The model represents the data with an average of 4.2% relative error. Note that the dependence on agitation (rps) has changed from the first power to a power of 1.58 between Eqs. 9 and 11.

Using Eq. 10, α's calculated for data points 8–12 average 4.2 times higher than the experimental values. For a longer annular path length (points 13 and 14) the average prediction is 67 times higher than the experimental values.

Figure 4 summarizes the backmix data obtained on the glass prototype with different annular lengths but at a constant annular area to column area a/A ratio of 0.0143. The data points are listed as 15–21 in Table 1. Annular lengths were varied by adding a sleeve on top of each stage divider. The data are fit by

$$\alpha = 0.0167\,(1/t - 0.125)\,\frac{(\text{rps})^2}{U_a}. \tag{12}$$

Relative deviation of the model from the data averaged 8.0%.

In Eq. 12 the nonzero intercept on the reciprocal path length axis is similar to Eqs. 9 and 11 with their nonzero intercepts on the reciprocal velocity axis. In Eqs. 9 and 11, no backmixing occurs at some large annular velocity value (12.5 and 50 cm/s). Additional decreases in backmixing are not obtained by operating at higher velocities. Similarly, in Eq. 12 some annular path length for a fixed annular area to column cross-sectional area ratio should correspond to having individual reactors in series (t = 8 cm for our data). Again, backmixing would not be reduced further by having a longer annular path. Equation 12 is valid at this point for the geometry of the experimental apparatus.

A ten-stage pilot unit was built using information developed with the prototype. A description of the pilot unit is given in Table 2 in addition to experimental backmix data. Backmix data for the pilot unit are plotted in Figure 5. The lines on the graph represent the equation

$$\alpha = 0.02\,(1/U_a - 0.15)(\text{rps})^2. \tag{13}$$

The model fits the data well, predicting the points with a 4.8% average relative error. For this unit, a/A is 0.00862, d_s/d_h is 0.86, and annular path length is 3.81 cm. Equation 10 on the average predicts backmix ratios that are 35 times higher than the experimental values.

Two runs were made with solutions thickened with carboxymethylcellulose to get a feel for the effect of reduction in interstage mixing by viscous dampening. Increasing the average viscosity from 0.52 to 16 cp (mPa/s) reduced α from 0.54 to 0.084 (Table 2, points 6 and 14). The backmix ratio was reduced to zero at or some time before the solution viscosity reached 46 cp (point 13). A zeroth order approximation of viscosity dependence using points 6 and 14 suggests that α is dependent on (viscosity)$^{-1/2}$.

The mixing Reynolds number, N_{Re_1}, ranged from 7.4×10^4 to 1.5×10^5 for the prototype runs and from 6.7×10^3 to 3.1×10^5 for the pilot reactor. This would place the interstage mixing in the turbulent regime for all data points.

The annular flow Reynolds number, N_{Re_2}, ranged from 156 to 370 in the prototype and from 2 to 924 for the pilot reactor. If no shaft rotation were present, stable laminar flow would exist in the annulus below a Reynolds number of 2,000. With shaft rotation, the theoretical results of Goldstein (1937) would indicate that turbulent flow would result for all annular Reynolds numbers greater than about 55. Only data points 13 and 14 of Table 2 (higher viscosity experiments) are in the laminar flow region in the annulus.

BACKMIXING AND STAGE EFFICIENCY

The backmix ratio α has been defined as f/F, the backmix rate in each stage divided by the feed rate. The modified backmix ratio

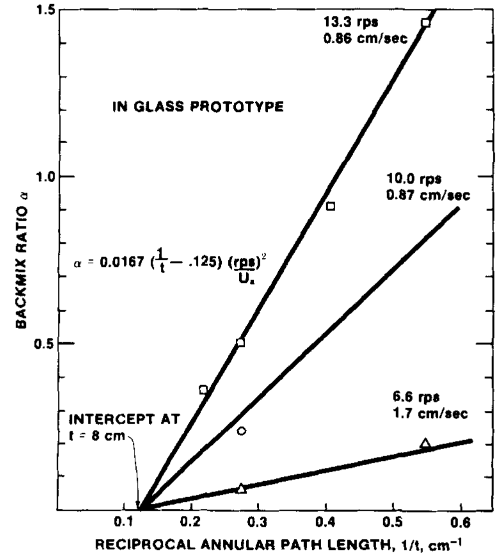


Figure 4. Backmix reduction with increasing annular path length.

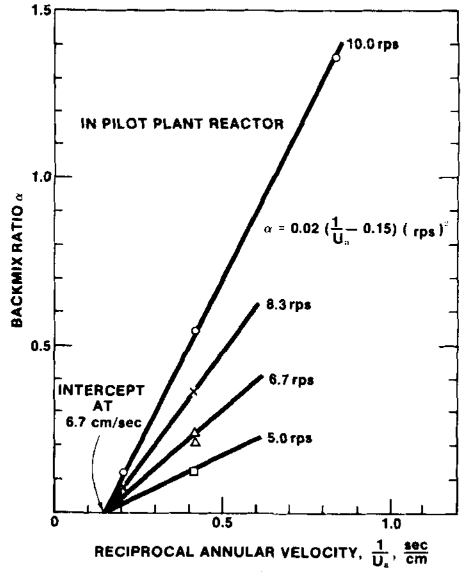


Figure 5. Backmix reduction with increasing annular velocity.

TABLE 2. TEN-STAGE PILOT PLANT UNIT

Run No.	Annular Velocity cm/s	Agitator Speed, rps	Backmix Ratio, f/F	Viscosity at Operating Temp., cp (mPa/s)
1	4.82	5.00	0.0036	—
2	4.82	8.33	0.074	—
3	4.80	10.0	0.116	—
4	4.79	6.67	0.031	—
5	2.39	6.67	0.24	—
6	2.42	10.0	0.54	0.52
7	2.42	8.33	0.36	—
8	2.41	5.00	0.117	—
9	2.42	3.33	0.020	—
10	2.40	6.67	0.204	—
11	9.31	10.0	0.00	—
12	1.20	10.0	1.36	—
13	2.40	10.0	0.00	46
14	2.40	10.0	0.084	16

Stage diameter, T = 20.3 cm
Stage height, H = 30.8 cm
Impeller diameter, D = 12.7 cm
Shaft diameter, d_s = 3.175 cm
Interstage baffle hole, d_h = 3.691 cm
Total volume = 96.5 L
Four baffles at wall, 28.3 cm long × 1.91 cm deep × 0.16 cm thick offset 0.64 cm from wall.
Six-blade turbine impellers; blades 3.18 cm wide × 2.54 cm height × 0.16 cm thick.
Support bearing between stages 4 and 5 starting at top, foot bearing.

x is $\alpha/(1+\alpha)$. For a given α, the relative variance for n stages is found from Van der Laan's relationship expressed as Eq. 2. A study of Eq. 2 reveals that the reciprocal of the relative variance for a given α and n yields the number of no-backmix ($\alpha = 0$) stages having an identical relative variance.

The relationship

$$\%\ \text{Efficiency} = \frac{(\mu^2/\sigma^2 - 1)}{(n-1)}(100) \tag{14}$$

is chosen to allow efficiency to range from zero to 100% for each n. If efficiency is defined as $(\mu^2/\sigma^2)/n$, the range is from $100/n$ to 100% and a separate efficiency curve is required for each n. This occurs because n stages can deteriorate to a single stage (not zero stages).

The solution of Eq. 14 is given in Figure 6, a plot of α vs. % efficiency. Given α, % efficiency can be determined from Figure 6. Knowing % efficiency, the equivalent number of no backmix ideal stages can be determined by solving for μ^2/σ^2 in Eq. 14.

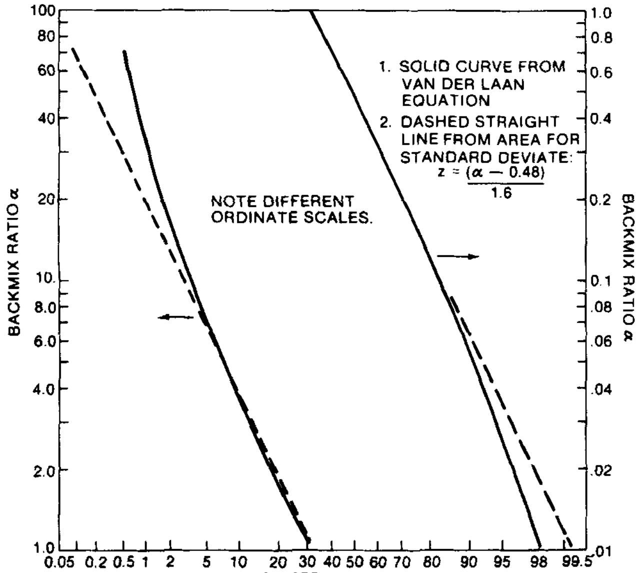


Figure 6. Relation between backmix ratio α and stage efficiency.

The coordinates in Figure 6 are scales of logarithms and probability. A straight line on this type of plot has properties that can be manipulated as if they had statistical significance. In this instance, a straight line can be used to approximate the curve over a large region of interest. As a close approximation to the solution, the standard deviate z is calculated from

$$z = (\alpha - 0.48)/1.6 \tag{15}$$

Here 0.48 is the mean (50% point) and 1.6 is the standard deviation of the α vs. efficiency curve. After calculating the standard deviate for a given α, the area under the normal curve $F(z)$ is obtained from a statistical table. For $\alpha < 0.48$, efficiency is $100 \cdot F(z)$. For $\alpha > 0.48$ efficiency is $[1 - F(z)] \cdot 100$. Approximated efficiencies using this methodology are shown as a dashed straight line in Figure 6. The approximations are good in the 5 to 90% efficiency range (α values from 7 to 0.05).

DISCUSSION AND SIGNIFICANCE

Realizing that Eq. 10 available in the literature for estimation of backmix ratios has been overextended in the low backmix regime, we recognize three reasons for lack of fit. First, the bulk of the correlated data was from the region of large a/A. Second, there are no provisions in Eq. 10 for adjustments due to annular path length. Third, converting Eq. 10 from superficial velocity U_s to annular velocity U_a by substituting $U_a = U_s \cdot (A/a)$ results in

$$\alpha = 0.0098 \left(\frac{\epsilon ND}{U_a TH}\right)^{1.24} \left(\frac{A}{a}\right)^{0.62} . \tag{16}$$

There is some discomfort in accepting a functional form that at constant annular velocity predicts a decrease in the backmix ratio as the annular area is increased.

For a thin horizontal plate and a small annular opening, we have found the backmix ratio to be proportional to rps: to $(\text{rps})^{1.58}$ for a thin plate with an intermediate annulus, and to $(\text{rps})^2$ for intermediate and larger annuli having extended annular lengths. The concepts of minimum annular velocity and minimum annular path lengths for zero backmix are readily acceptable from a physical visualization of the individual stages approaching separate reactors as a limit.

Equations 12 and 13 are special cases of

$$\alpha = k_5 \left(\frac{1}{U_a} - k_6 \right) \left(\frac{1}{t} - k_7 \right) (\text{rps})^2 \qquad (17)$$

with either k_6 or k_7 being too small to be regressed from the limited data. With sufficient data, both k_6 and k_7 may be obtained for some apparatus. The parameters k_6 and k_7 may depend on the size of the apparatus as well as on certain geometric ratios (a/A, d_s/d_h, $d_s - d_h$, etc).

Stage efficiency deteriorates rapidly in multistage contactors if care is not taken to minimize interstage backmixing. Contactors with a backmix ratio of 1 have only a 38% actual efficiency ($\mu^2/\sigma^2/n$); α's less than 0.06 are required to achieve 90% stage efficiency.

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NOTATION

A = column cross section area, cm^2
a = annular area in horizontal baffles dividing compartments, cm^2
C = concentration, units/cm^2
D = impeller diameter, cm
d_h = diameter of hole through which shaft passes
d_s = diameter of shaft, cm
F = feed rate, mL/s
f = backflow rate, mL/s
H = height of a stage, cm
k_1, k_3, k_5 = correlation parameters
k_2 = reciprocal of minimum annular velocity yielding zero backmix for all agitation levels at a fixed annular path length
k_4 = reciprocal minimum annular path length that yields zero backmix for all agitation levels
k_6 = same as k_2 but varies with path length
k_7 = same as k_4 but varies with a/A
M_1' = first moment about origin (unnormalized)
M_2 = second moment about mean (unnormalized)
M_2' = second moment about origin (unnormalized)
m = number of components
N = agitator speed, s^{-1}
N_{Re_1} = mixing Reynolds number = D^2N/ν
N_{Re_2} = annular flow Reynolds number = $d_h(1 - k)(U_a/\nu)$
n = number of stages or nth stage
T = column diameter, cm
t = annular path length, cm
U_s = superficial velocity, cm/s
U_a = annular velocity, cm/s
V = volume of a compartment, mL
x = modified backmix ratio, $f/(F + f)$

Greek Letters

α = backmix ratio, f/F
ϵ = impeller correction factor $\cong$ (number of impeller blades/6)$^{1/2}$
θ = residence time, hours
κ = d_s/d_h
μ = mean of distribution
ν = kinematic viscosity
σ^2 = variance of distribution

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