

Substituting the corrected shear stress into the expression for h_D^* yields the expression for the corrected mass transfer coefficient when $N_{Sc} = 1$.

$$\left(\frac{h_D^*}{h_{D0}^*} \right)_{N_{Sc}=1} = \left(\frac{h_D^*}{h_{D0}^*} \right)_{N_{Sc} \rightarrow \infty}^{4/3} \cdot \left(\frac{\alpha}{e^\alpha - 1} \right)^{1/3} (B_1)$$

Thus the term $[\alpha/(e^\alpha - 1)]^{1/3}$ is the predicted variable density correction when $N_{Sc} = 1$. Since the analysis is approximate

and the integral clumsy for $N_{Sc} \neq 1$, this correction is suggested for all Schmidt numbers near unity. Use of this correction is made in the Discussion Section of the paper. As in the case of constant properties, it is expected that this correction might apply reasonably well to geometries other than the flat plate.

Manuscript received October 14, 1963; revision received September 14, 1964; paper accepted April 5, 1965. Paper presented at A.I.Ch.E. San Juan meeting.

Interstage Mixing in an Oldshue-Rushton Liquid-Liquid Extraction Column

EDGAR B. GUTOFF

Polaroid Corporation, Waltham, Massachusetts

Interstage mixing was measured in a 4-in. diam. Oldshue-Rushton column fed with a salt solution at the top and distilled water at the bottom, with the diluted salt solution leaving at the top.

Mixing between stages is relatively slight at low agitator speeds, but increases very rapidly with speed once the turbulent region is reached. Interstage mixing is reported in terms of bulk backflows and also in terms of eddy diffusivities. The amount of interstage mixing is similar to that reported in the literature for rotating disc contactors.

Variations in the horizontal baffle plate design were also studied.

The use of vertical columns rather than mixers-settlers for the continuous extraction of a solute from one liquid into another is quite commonplace. Similar columns are also used as chemical reactors. The efficiency of the column depends mainly on the rate of mass transfer between the phases. However, a number of workers (2, 5, 8, 10, 11) have recognized that backmixing or internal circulation may be a major cause of a lowered efficiency in these columns. In fact, just recently Treybal (9) proposed a new type of column whose complex internal construction is designed to eliminate interstage mixing.

The degree of interstage mixing that takes place during liquid-liquid extractions is not normally easy to measure. The lowered efficiency that results from backmixing often shows up as a maximum in the curves of extraction efficiency vs. agitator speed. Higher speeds decrease the drop size and therefore increase the surface, and thus, along with the greater turbulence, tend to increase the rate of extraction. On the other hand, higher speeds also increase the interstage mixing; therefore there is often an optimum agitator speed for any given system.

Until now, the actual amount of backflow has not been determined, although Westerterp and Mayberg (10) found that an impeller in the top stage caused severe interstage mixing in the top three stages of a rotating disk contactor. Miyauchi et al. (3, 4), Eguchi and Nagata (1), and Sleicher (7) have taken interstage mixing into account by using an eddy diffusivity term in the mass transfer equations. From the actual performance of the column, it is then possible to determine the value of the eddy diffusivity. Strand, Olney, and Ackerman (8) and Westerterp and Mayberg (11) have directly measured this diffusivity term in a rotating disk contactor.

The approach taken here is to directly measure the amount of interstage mixing in a model, single phase system where no extraction is taking place. In this agitated column the contents of any one stage are assumed to be homogeneous, and concentration changes should then occur in a step-by-step fashion at the horizontal plates separating the stages. The results are explicit, and should directly relate to actual conditions.

EXPERIMENTAL

The Oldshue-Rushton type column (5) used for these tests is illustrated in Figure 1. The column consists of a 4-in. I.D. Plexiglas tube. The stainless steel horizontal plates have 2-in. diam. openings and are spaced 2 in. apart. The four 1/2-in. wide vertical baffles serve to support the horizontal plates. The 2-in. diam., six bladed turbine agitators are manufactured by the Mixing Equipment Company. Teflon steady bearings are used at the top and bottom of the column.

A salt solution is introduced into the top of the column, and distilled water into the bottom. Diluted salt solution is removed from the top. The amount of interstage mixing is determined from the salt concentration at the bottom c_b , the salt concentration of the effluent diluted salt solution c_d , and the distilled water flow F_w , as follows.

The nomenclature illustrated in Figure 1 is used. A salt balance on the top portion of the column above stage n gives

$$c_s F_s + c_n (F_b + F_w) = c_d F_d + c_{n-1} F_b \quad (1)$$

while the overall balance is

$$c_s F_s = c_d F_d \quad (2)$$

Therefore

$$c_n (F_b + F_w) = c_{n-1} F_b \quad (3)$$

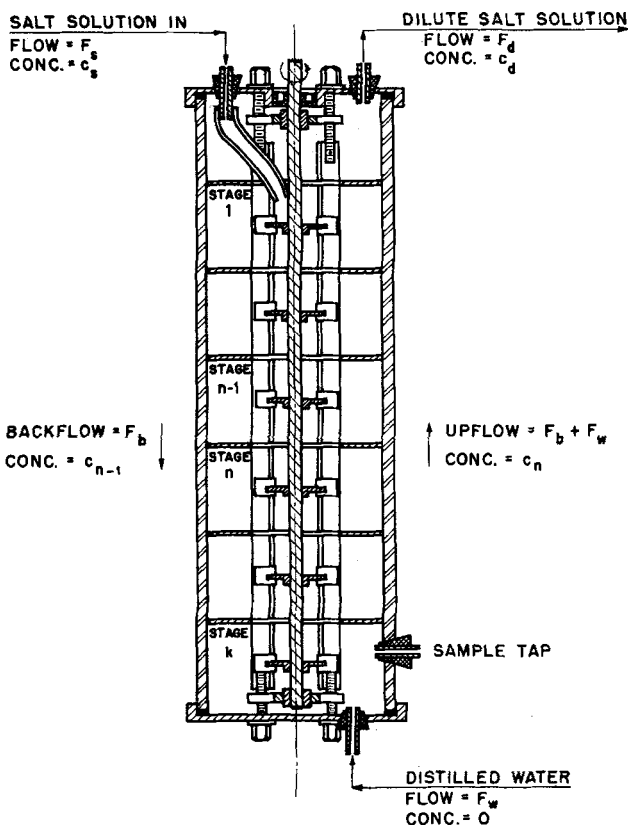


Fig. 1. Experimental Oldshue-Rushton type column.

TABLE 2. COMPARISON OF INTERSTAGE MIXING UNDER SEVERAL TEST CONDITIONS

This table lists the interstage mixing or backflow for various test conditions at a turbine speed of 140 rev./min. The data are taken from the curves of Figure 2.

Test condition	Distilled water flow, ml./min.	Backflow, ml./min.
Standard 2-in. holes in plates	150	600
1 1/4-in. holes in plates	150	230
1 1/4-in. holes in plates	75	440
1/8-in. screens in openings	150	440
1/16-in. screens in openings	150	175
Alternate turbines out, with or without plain or baffled tubes in calming section	150	230
Baffles cut out above plates	150	650
2-in. rotating center disks, 0.4-in. annulus	150	910
3.5-in. rotating center disks, same open area	150	1260

6. In variation 5 each tube had four 1/2-in. vertical baffles.

7. The horizontal plates were replaced by ones having annular openings. The solid central portion was 2 in. in diameter and rotated with the shaft on which it was fastened, and the outer portion had a 2.8-in. opening.

8. The horizontal plates were replaced by central disks, 3.47 in. in diameter, mounted on the shaft. The vertical baffles were cut out to accommodate these discs. The open area was the same as in variation 7.

Some experiments were also run with half the flow rate of distilled water entering the bottom of the column.

RESULTS AND DISCUSSIONS

The results of these experiments are presented in Table 1* and Table 2, and plotted in Figure 2. The accuracy of the results may be easily estimated. The conductivity determination should be accurate to 1%. In solving Equation (5), the accuracy of c_k/c_d is then 2%. The accuracy of the ratio $(F_b + F_w)/F_b$ is therefore $[1/(k - 1)]$ (2%), or, for the $k = 4$ to seven stages used here, 0.3% to 0.7%. The error in the ratio of backflow to wash water flow, F_b/F_w , is higher, and, for the values used, the accuracy is about 0.7% to 8%. The greater errors are at the higher degrees of backmixing. The wash water flow, measured by a calibrated capillary flow meter, should be accurate to better than 3%. The value of the actual amount of backmixing flow would therefore be accurate to between 4 and 11%.

It can be seen from Figure 2 and Table 2 that:

1. At speeds above about 90 rev./min. the interstage mixing increases very rapidly with turbine speed. Thus at 90 rev./min. the backmixing is 163 ml./min., while at 192 rev./min. it rises to 1,100 ml./min. To a first approximation this backmixing varies linearly with turbine speed. A similar linear relationship was found by Westerterp and Mayberg (10) for rotating disk contactors. Below about 90 rev./min., the amount of interstage mixing does not change much with agitation. With no agitation at all the backmixing is higher than at low speeds, probably because of the directed downward flow of the entering salt solution.

2. Reducing the effectiveness of the baffling by cutting out the baffle for 3/16-in. just above the horizontal plates slightly increases the interstage mixing.

* Tabular material has been deposited as document 8371 with the American Documentation Institute, Photoduplication Service, Library of Congress, Washington 25, D. C., and may be obtained for \$1.25 for photoprints or 35-mm. microfilm.

or

$$\frac{c_n}{c_{n-1}} = \frac{F_b}{F_b + F_w} \quad (4)$$

As all stages are geometrically identical and as Equation (4) holds for two stages, for a total of k stages, Equation (4) becomes

$$\frac{c_k}{c_d} = \left[\frac{F_b}{F_b + F_w} \right]^{k-1} \quad (5)$$

The distilled water and effluent diluted salt flows were measured with capillary flow meters. The salt solution to the system was pumped in with a calibrated Sigmamotor pump. Concentrations of the ammonium nitrate salt solutions in and out, and at the bottom stage (c_k) were determined from their conductivities, as measured with an Industrial Instruments Model RC-16B2 conductivity bridge using a conductivity cell having a cell constant of 0.1. The conductivities were found to be directly proportional to the salt concentration in the range studied, and so the conductivities were used directly in the equations in place of the concentrations. The conductivities used in solving Equation (5) were taken above the conductivity of the distilled water, although in most cases this correction was negligible.

The experiments were run in the column as illustrated in Figure 1, and also with several modifications. These changes were made to study their effects on the severity of interstage mixing.

1. The vertical baffles were cut out about 3/16-in. above the plates to allow for some circulation on the plates.

2. The horizontal plates were replaced by plates having 1 1/4-in. openings, rather than the standard 2-in. openings.

3. Double thick screening of 1/2-in. and of 1/16-in. mesh were placed in the openings in all the horizontal plates. The column thus tends to approach the construction of the Scheibel column.

4. The agitators were removed from alternate stages, so that each stage consisted of an agitated section and a calming section.

5. In variation 4 a 2-in. diam., 2-in. high metal tube was inserted in each calming section.

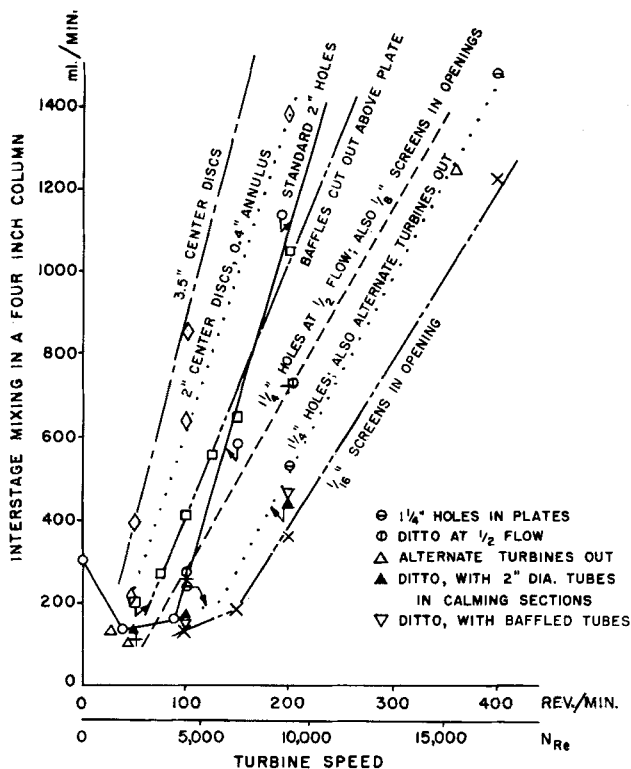


Fig. 2. Interstage mixing in the experimental 4-in. column as a function of the turbine speed. In all cases but one, the flow of distilled water is 150 ml./min.

3. Working with smaller holes in the horizontal plates, such that the open area is approximately one-half, and therefore the net upward velocity of the wash water is double, reduces the interstage mixing. Simultaneously reducing the wash water flow to one-half its normal value slightly increases the interstage mixing, but not back to its original value.

4. Inserting double thicknesses of screening in the holes of the horizontal plates reduced the amount of backmixing. With $\frac{1}{8}$ -in. screening the effect is the same as using $\frac{1}{4}$ -in. holes in the plate at one-half the flow rate, while using $\frac{1}{16}$ -in. screening is considerably more effective. The use of screening brings the column closer to the Scheibel type, where the horizontal plates are replaced by wire mesh packing.

5. Removing alternate turbines, so that each stage is now twice the height and consists of a mixing section and a calming section, reduces the interstage mixing to about the level using the $\frac{1}{4}$ -in. plates. Inserting 2-in. diam. tubes either baffled with a four $\frac{1}{2}$ -in. vertical baffles or unbaffled, had little further effect.

6. Replacing the 2-in. diam. axial opening in the horizontal plate by an annular opening greatly increased the amount of interstage mixing. The further out from the center, the greater the backmixing. This increased backmixing is partly due to the nature of the flow pattern of the fluid around the turbine agitator. The fluid leaves the turbine radially, and near the walls of the column flows vertically. At the horizontal plates the flow becomes radially inward, and finally vertically again back towards the turbine blades. Thus, the closer the openings in the horizontal plate are to the column walls, the greater will be the vertical component of the flow tending to drive the fluid into the adjacent stages. Also, as the horizontal plates are rotating, they exert a pumping action which can drive liquid into adjacent stages.

It should be emphasized that these results pertain to this particular system with these particular flows.

The data obtained here can also be analyzed in terms of an eddy diffusivity for backmixing. Under steady conditions there is no net transfer of salt across any cross section of the column and thus the backflow by diffusion is equal to the salt carried upward by the flowing water. Therefore (11)

$$E \frac{dc}{dx} + Vc = 0 \quad (6)$$

Integrating from $c = c_a$ at $x = 0$ to $c = c_k$ at $x = kH$, where H is the height of one stage, gives

$$\ln(c_k/c_a) = -VHk/E \quad (7)$$

For the standard arrangement with stages 2 in. high and with 2-in. and also $\frac{1}{4}$ -in. openings in the horizontal plates, eddy diffusivities of 0.25 to 1.90 sq.cm./sec. were obtained, as shown in Table 3. This compares with values of 0.66 to 1.08 sq.cm./sec. that Westertep and Mayberg (11) obtained in a rotating disk contactor operating at speeds from 900 to 1,800 rev./min.

Strand et al. (8) have shown that (E/VH) , a reciprocal Peclet number, should be related to $D_r N/V$, where D_r is the rotor diameter and N is the rotor speed, and also to several geometrical factors. As shown in Table 3, values of E/VH equal to 1.6 to 12.1 were obtained when $D_r N/V$ varied from zero to 1,100. For the same range of $D_r N/V$ values, Strand et al. (8) found for 6-in. and 42-in. rotating disk contactors similar values of E/VH , ranging from 0.6 to 10. Thus the degree of backmixing in both the Oldshue-Rushton column and in the rotating disk contactor are of the same order of magnitude.

Based on the correlations of Strand et al. (8), our data in terms of E/VH , can be correlated with $(D_r N/V)$ (D_p/D_c) where D_p/D_c is the ratio of the plate opening to the column diameter. These relationships are shown in Figure 3. Of course, neither the turbine diameter nor the column diameter were varied, and the variations in plate opening and in wash water velocity are small. The data do show, however, that the same type of correlations for interstage mixing hold in the Oldshue-Rushton column as in the rotating disk contactor.

Two models, one assuming step-by-step concentration changes with a bulk backmixing flow, and the other assuming differential concentration changes caused by eddy diffusivity, were used to analyze the data. Both cannot be correct and it is probable that neither is completely correct. It is unlikely that there is a step discontinuity in concentration in the plate openings between stages, with no concentration gradients within a stage. On the other

TABLE 3. EDDY DIFFUSIVITIES AND RECIPROCAL PECLET NUMBERS

These values were obtained in the 4-in. column with seven 2-in. stages.

Plate opening, diam. in.	Water in, ml./min.	Turbine speed, rev./min.	$\frac{D_r N}{V}$	E , Eddy diffusivity, sq. cm./sec.	$\frac{E}{VH}$
2	150	0	0	0.45	2.89
2	147	40	112	0.24	1.58
2	150	90	247	0.28	1.79
2	150	150	411	0.80	5.11
2	150	192	526	1.48	9.44
1.25	150	100	274	0.38	2.41
1.25	150	200	549	0.74	4.70
1.25	150	400	1098	1.90	12.10
1.25	75	100	549	0.38	4.84
1.25	75	200	1098	0.93	11.93

$$N_{Re} = \frac{(\text{turbine diam.})^2 (\text{turbine speed in revolutions/unit time}) (\text{density})}{\text{viscosity}} \quad (11)$$

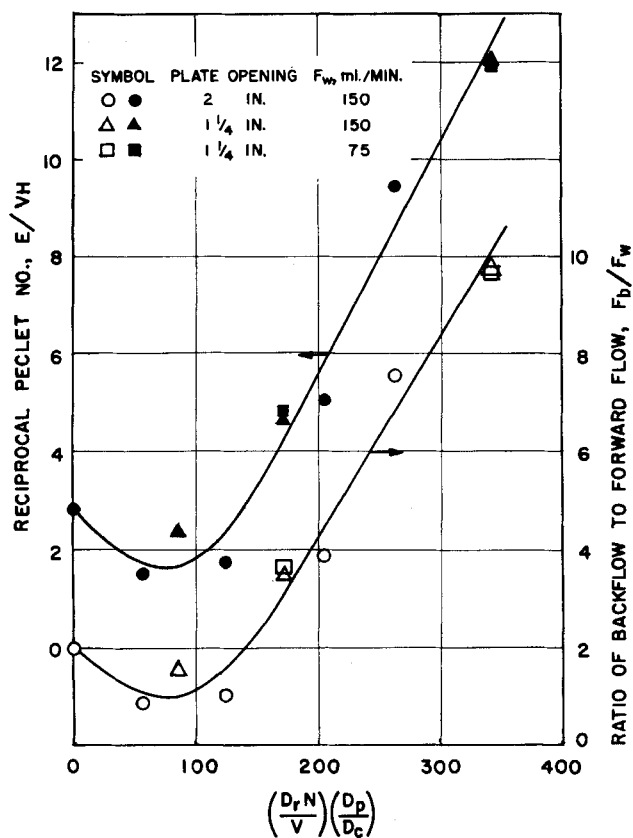


Fig. 3. Correlation between the ratio of backflow to forward flow, and also the reciprocal Peclet number, with term $(D_r N/V)(D_p/D_c)$.

hand, it is also unlikely that a uniform concentration gradient exists, for steepest gradients would occur in the openings between the stages, with lesser gradients within the stages. But although neither model is completely correct, both can contribute useful information, as has been shown above.

Comparing the two models in Equations (5) and (7), and noting that Equation (5) yields

$$\ln \frac{C_d}{C_b} = (k-1) \ln \left(\frac{F_b}{F_b + F_w} \right) \quad (8)$$

we find that

$$(k-1) \ln \left(\frac{F_b}{F_b + F_w} \right) = -\frac{VH}{E} k \quad (9)$$

or

$$\frac{VH}{E} = \left(\frac{k-1}{k} \right) \ln \left(1 + \frac{F_w}{F_b} \right) \quad (10)$$

Thus, the Peclet number, VH/E , can be directly related to a function of F_b/F_w , the ratio of the backflow to the net forward flow. If both models were true, this relationship should be independent of the number of stages. From Equation (10) this would occur when the number of stages is high and $(k-1)/k$ approaches unity. With many stages, the assumption of uniform concentration gradients would be more closely approximated and both models should tend to be equivalent.

It is also instructive to examine the Reynolds number at which the rapid increase of interstage mixing begins. On the abscissa of Figure 2 the Reynolds numbers

are also marked. For 2-in. diam. turbines in water at room temperature, Equation (11) becomes

$$N_{Re} = 43 (\text{turbine rev./min.}) \quad (12)$$

It is seen that in this particular system the transition region occurs at a Reynolds number of about 4,000. In the literature (6) it is reported that the transition region between laminar and turbulent regions in an agitated system extends from $N_{Re} = 10$ to 10,000. It therefore appears that under the conditions employed here the onset of the rapid increase in interstage mixing is associated with the onset of the turbulent flow region. It may be assumed that many eddies are propelled from one stage to the next.

ACKNOWLEDGMENT

Thanks are due to Richard Varney for carrying out the experimental work.

NOTATION

- c = salt concentration, units of salt per unit volume
- D = diameter, units of length
- E = eddy diffusivity for backmixing, units of length² per unit time
- F = solution flow rate, units of volume per unit time
- H = height of a stage, units of length
- k = number of stages in the column
- N = rotor speed, revolutions per unit time
- N_{Re} = turbine Reynolds number, dimensionless
- V = superficial forward or washwater velocity, units of length per unit time
- x = distance along the column, units of length

Subscripts

- b = back or interstage flow in the column
- c = column
- d = diluted salt stream leaving the column
- k = the bottom stage
- $n, n-1$ = the n^{th} and $(n-1)^{\text{th}}$ stages
- p = horizontal plate opening
- r = rotor
- s = incoming salt stream
- w = incoming washwater stream

LITERATURE CITED

1. Eguchi, W., and S. Nagata, *Mem. Fac. Eng., Kyoto Univ.*, (in English), **21**, 70 (1959).
2. Nagata, S., and W. Eguchi, *ibid.*, (in English), **19**, 102 (1957).
3. McMullen, A. K., T. Miyauchi, and T. Vermeulen, *Rept. No. UCRL-3911*, Supplement, U. S. Atomic Energy Comm. (January 22, 1958).
4. Miyauchi, T., *Rept. No. UCRL-3911*, U. S. Atomic Energy Comm. (August 15, 1957).
5. Oldshue, J. Y., and J. H. Rushton, *Chem. Eng. Progr.*, **48**, 297 (1952).
6. Rushton, J. H., E. W. Costich, and H. J. Everett, *ibid.*, **46**, 395, 467 (1950).
7. Sleicher, C. A., Jr., *A.I.Ch.E. J.*, **5**, 145 (1959).
8. Strand, C. P., R. B. Olney, and G. H. Ackerman, *ibid.*, **8**, 252 (1962).
9. Treybal, R. E., *Chem. Eng. Progr.*, **60**, No. 5, 77 (1964).
10. Vermijs, H. J. A., and H. Kramers, *Chem. Eng. Sci.*, **3**, 55 (1954).
11. Westertorp, K. R., and W. H. Meyberg, *ibid.*, **17**, 373 (1962).

Manuscript received September 30, 1964; revision received March 30, 1965; paper accepted March 31, 1965.