# Mixing and Mass Transfer at High Liquid Rates in the Steady State Counterflow Operation of Packed Columns

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Carbon dioxide absorption profiles have been measured for high liquid-gas ratios in an absorption column operated at atmospheric pressure under steady state counterflow conditions. Mixing and mass transfer parameters were extracted from these profiles using an extension of the technique described by Brittan (1967). The flow ranges covered are $15 < Re_s < 100$ and $300 < Re_L < 1\,600$.

The dispersion data reflect the same trends as shown by dynamic testing methods (Woodburn, 1974). However, with decreasing liquid-gas ratios the effect of dispersion on steady state mass transfer becomes less significant than would be inferred from dynamic testing results.

Under reverse gas flow conditions, the mass transfer coefficients corrected for the effects of axial dispersion show the same functional dependency on liquid rate as given by the Sherwood and Holloway (1940) correlation and provide a quantitative explanation for the anomalous behavior exhibited by their results in this liquid range.

## SCOPE

The majority of reported gas phase axial dispersion data for packed columns has been obtained at the low liquid rates normally used in absorption towers (de Maria and White, 1960; Dunn et al., 1962; Sater and Levenspiel, 1966). Under these conditions, the effect of dispersion on packed tower performance may be neglected (Brittan and Woodburn, 1966). There is evidence, however, that at the extremely high liquid-gas ratios necessary for the physical absorption of sparingly soluble gases, dispersion can have a pronounced effect on mass transfer performance (Woodburn, 1974).

The axial dispersive effect on mass transfer in packed columns is normally accounted for by a superimposition of the diffusion type of process on plug flow. The reported axial mixing coefficients used in the steady state model have invariably been determined using dynamic tracer methods. Buchanan (1971) maintains that the only dispersive process that should affect mass transfer performance is one of backmixing and that this is not the only dispersive mechanism that is detected in transient analyses. This implies that the measured dispersion coefficients are too large for application to steady state mass transfer situations.

Brittan (1967) has shown how the axial mixing coefficients may be estimated from experimentally determined steady state gas solute concentration profiles. The limited data reported by Woodburn (1974) using this technique, although consistent with the dynamic testing results, are too few to provide conclusive evidence as to the applicability of dynamic results to steady state situations. His results on mass transfer coefficients at the high liquid rates, where Sherwood and Holloway (1940) report anomalous behavior, indicate that correction for axial mixing caused by backmixing will yield coefficients consistent with the extrapolated Sherwood and Holloway correlation (Woodburn, 1974). Again, the reported data are too few to enable definite trends to be established.

Steady state carbon dioxide absorption profiles have been determined inside the same tower as used for Woodburn's (1974) dynamic testing. Mixing and mass transfer parameters have been extracted from these profiles using the technique of Brittan (1967). These coefficients can be compared with the unsteady state mixing results of Woodburn and the plug flow mass transfer coefficients of Sherwood and Holloway. The experiments were so designed that the range of liquid rates used embraced that liquid rate at which the onset of an induced reverse gas flow, or backmixing, has been observed (Uchida and Fujita, 1936, 1937; Woodburn, 1974). In this way, the observation of Buchanan (1971) relating to the effect of different dispersive mechanisms on mass transfer performance may be evaluated.

## CONCLUSIONS AND SIGNIFICANCE

Carbon dioxide was absorbed in water under the counterflow operation of a 0.292 m diameter column packed to a depth of 10.97 m with 0.025 m ceramic Raschig rings. Mixing and mass transfer parameters were extracted from experimentally determined absorption profiles by the fitting of the usual axial mixing model to these profiles. The flow ranges covered were $15 < Re_s$

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**$< 100$ and $300 < Re_L < 1\,600$.**

**It has been established that:**

**1. The axial mixing model is a good representation of the steady state absorption process in a packed column only for those liquid flow rates which are associated with an induced reverse flow of gas. This backmixing is responsible for the observed concentration inlet discontinuity which is a necessary condition for the applicability of the model.**

**2. The invariance of the Peclet number with liquid rate for irrigation rates greater than those corresponding to the onset of induced reverse gas flow, reported previously (Woodburn, 1974) for the dynamic testing method, has been confirmed by the present steady state results.**

**3. With decreasing liquid-gas ratio, for any given liquid rate, the steady state results indicate that the effect of dispersion on steady state mass transfer is less than would be inferred from dynamic testing data. This verifies Buchanan's (1971) observations that dynamic testing techniques are sensitive to dispersive mechanisms that may be of no consequence to steady state situations.**

**4. Correction of the mass transfer coefficient for the axial mixing caused by the recirculating gas fraction yields results that show the same functional dependency on liquid rate as given by the Sherwood and Holloway (1940) correlation. The anomalous behavior shown by the data of these authors at the high liquid rates may therefore be attributed to the onset of induced reverse gas flow.**

In the evaluation or prediction of packed tower performance, it is normal to assume plug flow in both the gas and liquid phases. Owing to the complexity of the bed structure, however, there is a spatial variation in local velocities at any given axial position, and hence a spread in the residence times of fluid elements ensues. Other factors such as stagnant zones and reverse gas flow also contribute to the distribution of residence times. This dispersion affects mass transfer within the column in that the concentration profiles are modified, with a consequent change in the concentration driving force for mass transfer. Over the past 20 yr this dispersion phenomenon has received increased attention in attempts to explain the poor performance of industrial installations, and to date it is still not clear what are the precise effects of dispersion on the operation of packed columns.

Brittan and Woodburn (1966) used the axial mixing data of de Maria and White (1960) to show that axial dispersion did not seriously affect the performance of their column over the range of liquid rates investigated. Subsequently, Woodburn (1974) has shown that at high liquid rates, significant deviations from the plug flow solutions may occur.

Furzer and Ho (1967), using the distillation results of Yoshida et al. (1954) and published axial dispersion coefficients, showed quantitatively that axial dispersion in the phases is an important factor in the relation between the height of a transfer unit and packed height. Mellish (1968) remarked, however, that the dispersion data were obtained in columns in which there would almost certainly be pronounced wall flow and suggested that consequently the mixing coefficients used were too large.

A more basic objection to the use of dispersion coefficients obtained by means of dynamic testing techniques was lodged by Buchanan (1971). He suggested that for the axial mixing model to be appropriate, the measured dispersion should be caused by backmixing and that forward mixing (as caused by velocity gradients within each pore, the variability in flow path dimensions, and the existence of stagnant pockets exchanging material with the flowing streams) should have no influence on the steady state concentration profiles. Unsteady state tracer experiments detect all these dispersive mechanisms, and, he continued, unless the backmixing component contribution to the dispersion predominates over the forward mixing effects, the measured coefficients cannot be used with justification in the prediction of steady state mass transfer.

Sherwood and Holloway (1940) reported mass transfer coefficients over a wide range of liquid flow rates. Their results showed a simple power law dependence of the coefficient on the liquid rate up to a rate of approximately 25 kg/m²s ($Re_L \doteqdot 700$). Above this liquid rate, the mass transfer coefficient decreased on increased irrigation. The results of Cooper et al. (1941) indicated a gas rate dependency at high liquid-gas ratios. They suggested that the fast counterflowing liquid caused a recirculation of gas within the column with a consequent reduction in the concentration driving force.

Uchida and Fujita (1936, 1937) detected a reverse flow of gas at high liquid rates. They reported the onset of the induced reverse gas flow to occur at a liquid rate corresponding to a liquid Reynolds number of 694. The effect of liquid rate on macroscale gas recirculation rates was reported recently by Woodburn (1974). The results were correlated by the expression

$$Re_R = 131.5 \log_{10} Re_L - 370.4$$

The onset of the induced reverse gas flow occurred at a liquid Reynolds number of approximately 685. Further evidence of the onset of a new dispersive mechanism was also furnished by the Peclet number data, wherein the data exhibited an invariance in the Peclet number with increasing liquid rate above $Re_L \doteqdot 650$.

## PARAMETER ESTIMATION

The method proposed by Brittan (1967) for the generation of a steady state gas solute profile has been extended to incorporate a significant pressure gradient along the column to yield the following differential equation (Woodburn, 1972):

$$\frac{\partial^2 f}{\partial \eta^2} + \frac{y^o}{(1 - y^o f)} \left( \frac{\partial f}{\partial \eta} \right)^2 - \left\{ \frac{P}{(1 - y^o f)} + \frac{\Delta\pi}{(1 - y^o f)(1 + \Delta\pi(1 - \eta))} + N \right\} \frac{\partial f}{\partial \eta} - \frac{PNA}{F} \frac{[1 + \Delta\pi(1 - \eta)](1 - y^o f)}{(1 + \Delta\pi)(1 - y^o)} \Big( f -$$

$$\left.\frac{m(1+\Delta\pi)Fv^{o}{}_{\mathrm{CO_2}}x^{1}}{(1+\Delta\pi(1-\eta))v_{\mathrm{H_2O}}y^{o}}\right)+PN(1-y^{o}f)\left(\frac{f}{(1-y^{o}f)}-\frac{f^{1}}{(1-y^{o}f^{1})}\right)+\frac{N\Delta\pi f}{[1+\Delta\pi(1-\eta)]}-\frac{\Delta\pi^{2}f}{[1+\Delta\pi(1-\eta)]^{2}}=0 \quad (1)$$

There is a high degree of interdependence between the Peclet number *P* and the number of transfer units *N*. For small pressure gradients, the two groups appear almost symmetrically in Equation (1). This illustrates the difficulty in estimating the groups individually from the profile.

However, the boundary conditions provide a sensitive relation for *P*:

$$\eta = 1 \quad \frac{\partial f}{\partial \eta} = 0 \qquad (2)$$

$$\eta = 0 \quad \frac{1-f_o}{1-y^{o}} = -\frac{1}{P}\left(\frac{\partial f_o}{\partial \eta} - \frac{f_o \Delta\pi}{1+\Delta\pi}\right) \qquad (3)$$

The latter condition results from a mass balance for the solute in the gas phase at the inlet to the tower, assuming no axial dispersion in the gas supply pipe.

A least-squares procedure was used over all the experimental points on a steady state profile to find the best value of *N*. For each value of *N*, regression was undertaken on the Peclet number in order to match the value used to compute the profile with the inlet boundary condition. The iterative procedure for the determination of *P* used the Newton approximation at the $\eta = 0$ boundary with the partial differential coefficients required being carried through the Runge-Kutta-Gill procedure used to generate $f(\eta)$, in the manner described by King (1967), with initial values at $\eta = 1$.

It is clear that the dispersion coefficient is uniquely determined by the choice of the value for the mass transfer coefficient, which in turn is that value which gives the best fit of the model to the experimental profile. Note that for the estimation of the Peclet group, it is a necessary condition that there exists an inlet concentration discontinuity as implied by the boundary condition (3).

## EXPERIMENTAL DESIGN

Thirty two steady state absorption profiles have been determined over the range of high liquid rates used in the dynamic tests reported previously (Woodburn, 1974). The range of gas rates for any particular liquid rate was restricted by the requirement that there be a significant concentration gradient at the gas inlet to be able to invoke the boundary condition given by Equation (3). For small absorption factors, the profile is flat at the inlet, and the solution procedure is insensitive to *P*.

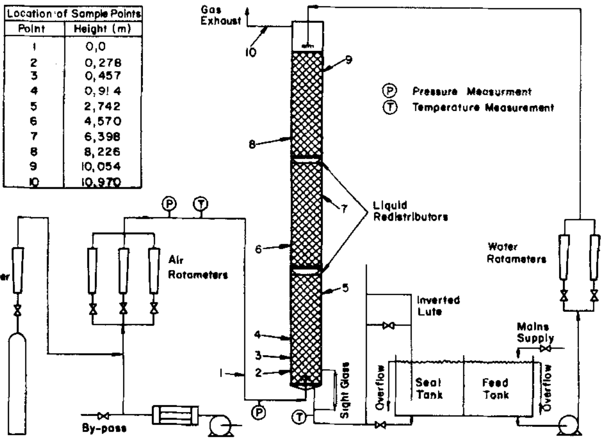
Mixing and mass transfer coefficients were extracted from the steady state profiles. In addition, plug flow profiles using the equation developed by Brittan (1964) were determined.

## EXPERIMENTAL APPARATUS AND PROCEDURE

### Absorption Column

The experimental work was undertaken in a 0.292 m internal diameter stainless steel column randomly packed to a depth of 10.97 m with 0.025 m ceramic Raschig rings. A flow diagram of the apparatus is shown in Figure 1.

In view of the critical importance of end effects in the parameter estimation technique used in this work, care was taken to reduce these effects as much as possible. Water was introduced to the top of the packing section via a four-point nozzle distributor, such that there were no gas-liquid interactions above the packing. The inlet gas entered the column through a centrally located 0.025 m pipe which passed through the packing support into the packing. The free space beneath the packing support had the minimum possible volume and was in the form of a very shallow cone. The water level, visible on a boiler type of sight glass, was held immediately below the bottom of the packing. In this manner, gas-liquid interactions below the packed section were minimized. Redistribution plates of the wall wiper type were located at distances of 3.66 and 7.32 m from the bottom of the column.



| Location of Sample Points | |
|---|---|
| Point | Height (m) |
| 1 | 0,0 |
| 2 | 0,278 |
| 3 | 0,457 |
| 4 | 0,914 |
| 5 | 2,742 |
| 6 | 4,570 |
| 7 | 6,398 |
| 8 | 8,226 |
| 9 | 10,054 |
| 10 | 10,970 |

Fig. 1. Schematic diagram of column.

### Carbon Dioxide Analysis

Gas sample probes were located within the column at the positions indicated in Figure 1. The feed and exit gases were also sampled.

A Gow-Mac model 100 gas chromatograph was used for the gas analysis. The stationary phase was water absorbed on Poropak T, and the detector current was maintained at 200 mA. Gas sampling was completely automated by means of a system of Kuhnke solenoid valves, control being exercised by a CDC 1700 SC computer.

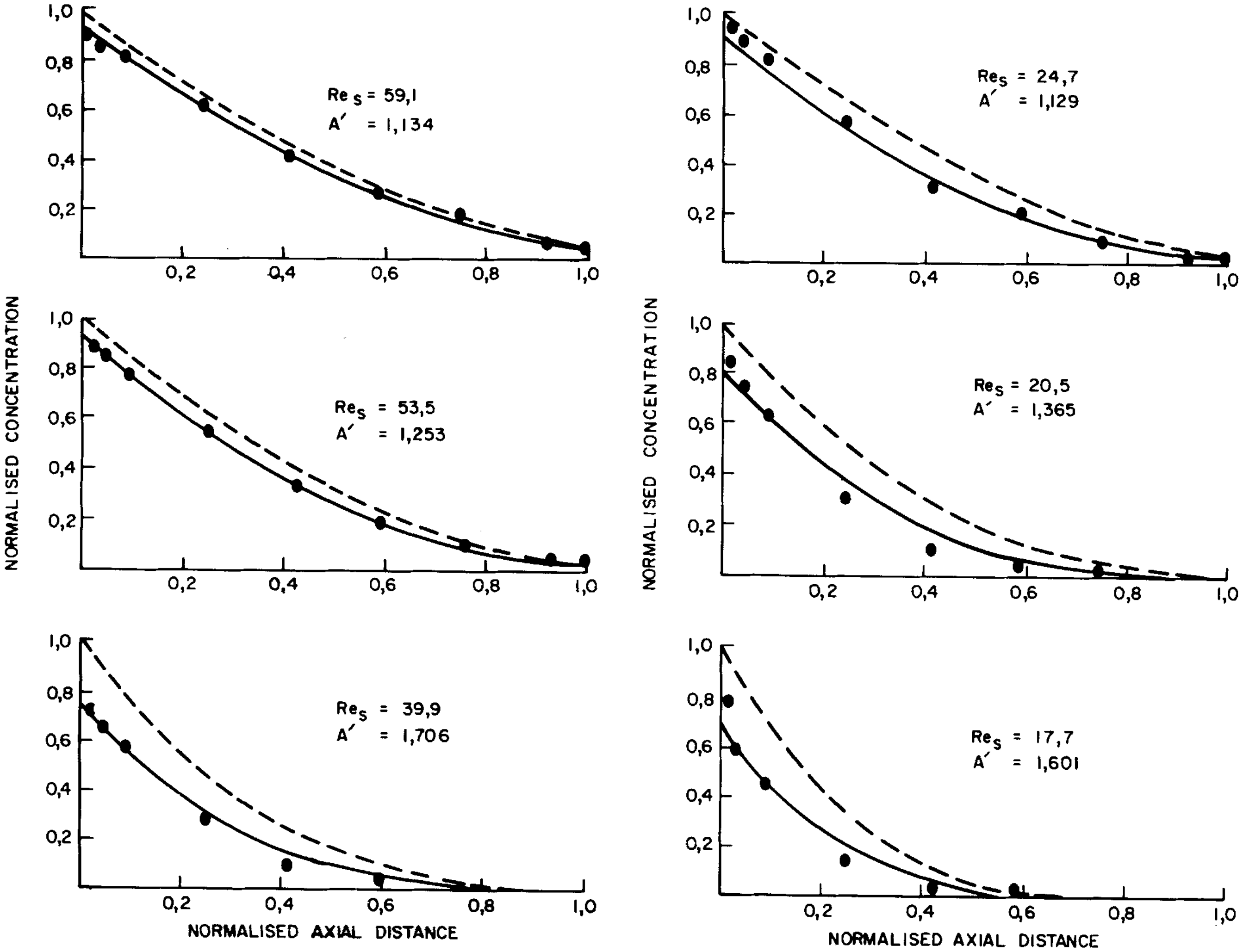
The GC imbalance signal was monitored by the computer, and peak areas were calculated immediately and stored on a disk for further processing. A scan of the sample points took approximately 15 min. Because usually five or six repetitive scans were done, the column was required to operate steadily for 1½ hr during any one run.

The principal experimental difficulty was associated with the withdrawal of gas samples in the presence of large amounts of counterflowing liquid. If water is entrained in the gas sample, then the possibility of absorption within the probe exists. However, provided the gas withdrawal rate is very much higher than that of the liquid, the liquid in the probe should become saturated and should not affect the gas solute concentration significantly.

Further experimental details are given elsewhere (Hatton, 1976).

## STEADY STATE ABSORPTION PROFILES

Experimental steady state absorption profiles for two different liquid rates and a number of absorption factors are shown in Figure 2. Also shown are the regressed curves for the axial mixing model and the profiles computed in terms of the plug flow model. The mass transfer



(a). $Re_L = 1\,312$.

(b). $Re_L = 500$.

Fig. 2. Absorption profiles: axial mixing (——) and plug flow (– – –) models.

coefficients estimated for these six runs using the axial mixing and plug flow models are given in Table 1. Peclet numbers from steady state and dynamic testing are also reported. The higher liquid rate ($Re_L = 1\,312$) is associated with a measurable induced reverse flow of gas. At the lower liquid rate, no gas recirculation has been detected.

It is clear that for the higher liquid rate, excellent fits to the experimental profiles are obtained using the axial mixing model, and there is no doubt as to the presence of the inlet concentration discontinuity. Although satisfactory agreement is found between the experimental points and the regressed curve for the lower liquid rate (corresponding to $Re_L = 500$), the presence of the concentration discontinuity is by no means confirmed. In fact, a curve could more realistically be traced through the experimental points to intersect the ordinate axis at a normalized concentration of unity. These observations were apparent for all the liquid rates used in this work; namely, an inlet concentration discontinuity exists only under those liquid flow rates which are associated with macroscale recirculation effects, or backmixing.

The Peclet number determination using the axial mixing model depends implicitly on the presence of an inlet discontinuity, and thus the model is physically unrealistic when no such discontinuity exists. Hence the model should

TABLE 1. MIXING AND MASS TRANSFER PARAMETERS FOR THE EXPERIMENTAL CONDITIONS OF FIGURE 2

| | | | $K_La$ s$^{-1}$ | | $Pe_G$ | |
|---|---|---|---|---|---|---|
| $Re_L$ | $Re_S$ | $A$ | Axial mixing model | Plug flow model | Steady state testing | Dynamic testing* |
| 1 312 | 59.1 | 1.134 | 0.0375 | 0.0256 | 0.062 | 0.025 |
| | 53.5 | 1.253 | 0.0342 | 0.0222 | 0.047 | 0.023 |
| | 39.9 | 1.706 | 0.0394 | 0.0178 | 0.023 | 0.019 |
| 500 | 24.7 | 1.129 | 0.0436 | 0.0169 | 0.033 | 0.033 |
| | 20.5 | 1.365 | 0.0431 | 0.0117 | 0.020 | 0.029 |
| | 17.7 | 1.601 | 0.0514 | 0.0111 | 0.019 | 0.025 |

* Woodburn (1974).

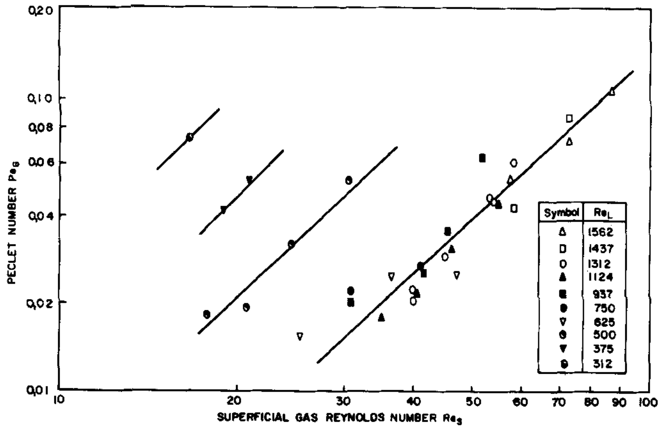


Fig. 3. Peclet numbers from steady state measurements.

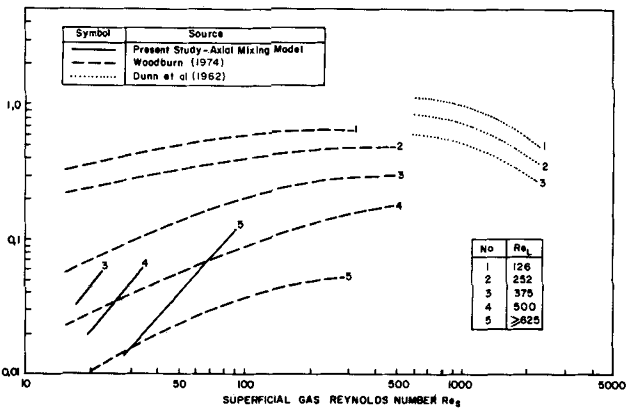


Fig. 4. Comparison of the Peclet numbers with published data.

only be applied to steady state mass transfer situations for those liquid rates for which macroscale recirculation occurs.

With increasing absorption factor (or increasing liquid-gas ratio), the deviation between the plug flow and experimental profiles becomes more pronounced. This is a direct consequence of the effect of the liquid-gas ratio on the concentration gradient prevailing along the column. For low liquid-gas ratios, this gradient is small, and any forward or backmixing of the solute will not change its concentration appreciably. Under these conditions it is to be expected that the column will appear to exhibit plug flow behavior. On increasing the liquid-gas ratio, the concentration gradient increases, and the effect of the dispersion becomes more significant.

## AXIAL MIXING

The mixing data extracted from the experimental profiles using the axial dispersion model are shown in Figure 3. The curves are compared with those of Dunn et al. (1962) and Woodburn (1974) in Figure 4. Over the gas range investigated, the Peclet number data obtained in the present work indicate the same general trends as the earlier unsteady state results. There is an increase in the Peclet numbers with gas rate and, for the low liquid rates, a decrease with increasing liquid flow. For liquid Reynolds numbers greater than approximately 625, the Peclet number becomes independent of liquid rate.

At the low gas rates, the Peclet numbers obtained on the basis of the steady state measurements are of the same order of magnitude as the dynamic testing results. At the higher gas rates, however, considerably less dispersion is indicated by the steady state results than by those obtained using the dynamic technique. As much as a fourfold difference is apparent. These results tend to confirm that the unsteady state testing methods do detect dispersive mechanisms that are of no consequence in steady state mass transfer situations. This conclusion is in accord with the observations made by Buchanan that some dispersive mechanisms detected using tracer experiments are irrelevant in the steady state operation of packed columns.

## MASS TRANSFER

Because the axial mixing model is not sensitive to changes in the dispersion coefficient when the gas-phase concentration gradient is very small, it was necessary to adjust the operating conditions to obtain a reasonable gradient along the column. This greatly restricted the range of gas rates that could be employed with any given liquid rate and also resulted in relatively high liquid-gas ratios. Under these conditions, the gas-film resistance may be significant. Calculations based on the correlation of Shulman et al. (Treybal, 1968) for the gas-film mass transfer coefficients and that of Sherwood and Holloway (1940) for the liquid-film coefficients indicated that the gas-film resistance accounts for only 4 to 6% of the overall resistance to mass transfer under all the operating conditions used in this work.

It was earlier suggested that the deviation from the simple power law variation of the liquid film coefficient with liquid rate shown by the results of Sherwood and Holloway (1940) is caused by the onset of an additional dispersive mechanism, namely, induced reverse gas flow. It was postulated that correction for this axial mixing would yield coefficients consistent with the simple power law correlation.

Since only a small gas-film resistance is indicated for all the flow conditions considered, it follows that the experimental $K_La$ values (corrected for the dispersion effects) may be considered to be close to the true liquid-film coefficients $k_La$ and have been plotted as such in Figure 5. Under the flow conditions where backmixing occurs, the corrected mass transfer coefficients show the same functional dependency on liquid rate as given by the Sherwood and Holloway correlation, although the values are approximately one half those predicted by the extrapolated correlation (this discrepancy in the magnitudes of the coefficients is probably due to liquid maldistribution effects in the large column used in this

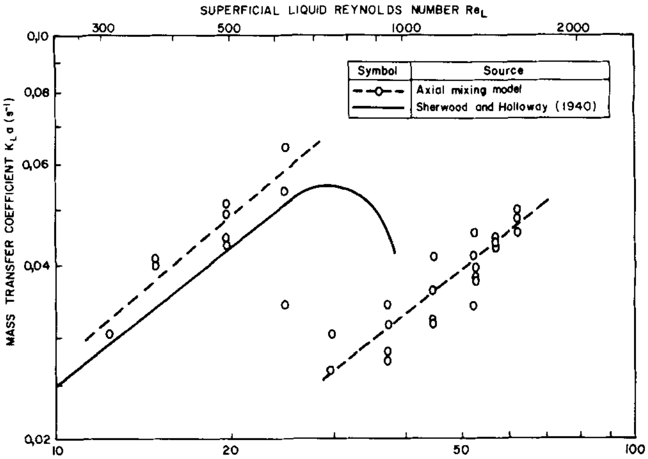


Fig. 5. Liquid-film mass transfer coefficients.

work). These results give credence to the inference that the anomalous behavior of the Sherwood and Holloway curve at these liquid rates is a direct consequence of axial dispersion caused by backmixing.

The good agreement between the experimental $K_La$ values and the Sherwood and Holloway correlation at the lower liquid rates is believed to be fortuitous. The axial mixing model is physically unrealistic for these flow rates owing to the assumed inlet discontinuity which does not exist. Consequently, since backmixing is implied in the model, the corresponding Peclet numbers overestimate the mixing effect, although they are consistent with reported unsteady state data. Because of the almost symmetrical appearance of the groups $P$ and $N$ in the defining differential equation, it follows that the estimated $K_La$ values are too large. This is consistent with Buchanan's (1971) observations that where no backmixing occurs, the mixing coefficients estimated from dynamic techniques are too large for application to steady state situations.

## CONCLUSIONS

1. The axial mixing model adequately describes the steady state absorption process in packed columns only for those liquid flow rates which are associated with an induced reverse flow of gas. This backmixing is responsible for the observed concentration inlet discontinuity which is implied by the model.
2. The invariance of the Peclet number with liquid rate for $Re_L > 625$ reported previously for the dynamic testing method (Woodburn, 1974) has been confirmed by the present steady state results.
3. With decreasing liquid-gas ratio, the effect of dispersion on steady state mass transfer performance is less than would be predicted on the basis of mixing coefficients obtained from unsteady state tracer methods.
4. Correction of the mass transfer coefficient for the axial mixing caused by the recirculating gas fraction yields results that show the same functional dependency on liquid rate as given by the Sherwood and Holloway (1940) correlation. It may be inferred that the anomalous behavior shown by their results at the high liquid rates is caused by the onset of induced reverse gas flow, or backmixing.

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## NOTATION

$A$ = absorption factor = $U_L/mU_s^o$
$d_p$ = packing characteristic length, m
$E_G$ = gas-phase axial dispersion coefficient, $m^2\ s^{-1}$
$F$ = temperature correction factor = $(273 + T)/(273 + T^o)$
$f$ = dimensionless gas solute volume fraction = $y/y^o$
$k_La$ = liquid-film mass transfer coefficient, $s^{-1}$
$K_La$ = overall mass transfer coefficient, $s^{-1}$
$L_M$ = superficial mass liquid rate, $kg\ m^{-2}\ s^{-1}$
$m$ = equilibrium constant = $(y/x)\ (v_{H_2O}/v_{CO_2})$
$N$ = number of transfer units = $k_LaZ/U_L$
$P$ = gas-phase column Peclet number = $U_GZ/E_G$
$Pe_G$ = gas-phase Peclet number = $U_Gd_p/E_G$
$Re_L$ = liquid Reynolds number = $U_L\rho_Ld_p/\mu_L$
$Re_R$ = gas Reynolds number for induced reverse gas flow = $U_R\rho_Gd_p/\mu_G$
$Re_s$ = gas Reynolds number = $U_s\rho_Gd_p/\mu_G$
$U_G$ = interstitial gas velocity, $m\ s^{-1}$
$U_L$ = superficial liquid velocity, $m^3\ m^{-2}\ s^{-1}$
$U_R$ = recycle gas velocity, $m^3\ m^{-2}\ s^{-1}$
$U_s$ = superficial gas velocity, $m^3\ m^{-2}\ s^{-1}$
$v$ = specific volume, $m^3$ kg mole$^{-1}$
$x$ = volume fraction carbon dioxide in liquid
$y$ = volume fraction carbon dioxide in gas
$Z$ = packing depth, m
$z$ = axial coordinate, m
$\eta$ = dimensionless axial coordinate = $z/Z$
$\mu$ = fluid viscosity, $Nsm^{-2}$
$\Delta\pi$ = normalized column pressure drop
$\rho$ = fluid density, $kg\ m^{-3}$

### Subscripts

0, 1 = conditions within the tower at the gas inlet and exit conditions, respectively
$L$, $G$ = liquid and gas phases, respectively

### Superscripts

0, 1 = conditions external of the column at the gas inlet and exit positions, respectively

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