

Mass Transfer Coefficients in a Trickle-Bed Reactor with High and Low Viscosity Organic Solutions

BADIE I. MORSI

Chemical and Petroleum Engineering Department, University of Pittsburgh, Pittsburgh, PA 15261 (U S A)

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ABSTRACT

Some experimental data are presented for the volumetric liquid-side mass transfer coefficients, coupled with the total liquid hold-up and two-phase pressure drop, for the weak

interaction regime in a catalytic-packed-bed reactor. The measured k_{La} , $\Delta P/Z$ and β values for viscous solutions were about four times those observed for low-viscosity organic solutions. An empirical correlation for predicting k_{La} values in trickling flow regime is proposed

TABLE 1

Some available literature data and correlations for the k_{La}

| Author | Study | Packing | | Correlation |
|----------------------------------|--|-------------------------------|----------------------|---|
| | | $d_p \times 10^3$ (M) | Nature | |
| Reiss [12] | Absorption of O_2 into water from air at 25 °C | 7.62 to 40.64 | R.R. | $k_{La} = 0.0173 E_L^{0.5}$ $E_L = \left(\frac{\Delta P}{Z} \right)_{LG} \frac{L}{\rho_L}$ |
| Hirose <i>et al.</i> [15] | Desorption of O_2 into N_2 from water at 15 °C | 2.59 to 12.2 | G.B. | $\frac{k_{La}}{\epsilon} = 0.163 \left(\frac{G}{\epsilon \rho_G} \right)^{0.6} E'_L{}^{0.31}$ $E'_L = \left(\frac{\Delta P}{Z} \right)_{LG} \frac{L}{\epsilon \beta \rho_L}$ |
| Gianetto <i>et al.</i> [13] | Desorption of O_2 in air from (2 N) NaOH solution | 6 | B.S. R.R. G.B. | $\frac{k_{La} \epsilon L}{\rho_L} = 0.0305 \left[\left(\frac{\Delta P}{Z} \right)_{LG} \frac{\rho_L \epsilon}{a_c L^2} \right]^{0.068} - 1$ |
| Ufford and Perona [14] | Absorption of CO_2 in water | 19 to 12.7 | B.S. R.R. R.R. | $k_{La} = i G^m L^n$ $i, m \text{ and } n \text{ are constants}$ |
| Sylvester and Pitayagulsarn [16] | Absorption of pure CO_2 in water | 3.18 to 3.18 | C | |
| Goto and Smith [17] | Desorption of O_2 in pure N_2 from distilled water saturated with pure O_2 | 0.541 to 2.91 CuZnO | C.P. | $\frac{k_{La}}{D_A} = \alpha \left(\frac{L}{\mu_L} \right)^n (Sc)^{0.5}$ $Sc = \mu_L / \rho_L D_A$ $\alpha \text{ and } n \text{ are constants}$ |
| Turek and Lange [22] | Hydrogenation of α -methylstyrene to cumen (organic solution) | 0.54 to 3.00 Pa/ Al_2O_3 | C.P. | N.A. |

R.R. = Raschig rings, G.B. = glass beads, B.S. = Berl saddles, C = cylinders, C.P. = catalytic particles
N.A. = Not available

1 INTRODUCTION

The design and scale-up of trickle-bed reactors require the knowledge of both the hydrodynamics, *e.g.* flow regimes, phases hold-ups and two-phase pressure drop, and the transport characteristics, such as gas-liquid and liquid-solid mass transfer coefficients and corresponding interfacial areas [1-11]. Most of the published experimental data on the volumetric liquid-side mass-transfer coefficient $k_L a$ were obtained with aqueous and highly ionic solutions [12-17]. The correlations based on these studies are briefly summarized in Table 1. This paper presents some experimental data on $k_L a$ for organic viscous and non-viscous solutions. The experimental data were obtained using a chemical technique where carbon dioxide was absorbed in low-viscosity diethanolamine (DEA)-ethanol and in highly viscous diethanolamine-ethyleneglycol organic solutions.

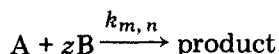
2 EXPERIMENTAL DETAILS

The experiments were carried out in a 0.05 m diameter co-current, down-flow column packed with 0.0024 m mean diameter spherical Co/Mo/Al₂O₃ catalyst particles to a 0.49 m height. The bed void fraction was

0.385. In all the experiments, the operating temperature was controlled at 20 ± 0.5 °C. The gas (inert + solute) flow rates were varied between 0.03 and 0.15 kg m⁻² s⁻¹, and the liquid (solvent + reactant) flow rates were varied between 1 and 8 kg m⁻² s⁻¹. The experimental set-up is described elsewhere [8, 11]. The physicochemical properties and kinetics data for the gas-liquid systems used are given in Table 2.

3 THEORY

Alvarez-Fuster and co-workers [18, 19, 23] reported that the chemical reactions between CO₂ and DEA, in both ethanol and ethyleneglycol solutions, are of a third order, being first order with respect to the solute CO₂ and second order with respect to the reactant DEA. These reactions can be represented as



The above irreversible reactions will be in the slow reaction regime if on one hand the following inequality is verified

$$Ha = \frac{1}{k_L} (D_A k_3 C_{B0}^2)^{1/2} < 0.3 \quad (1)$$

and on the other hand, the depletion test is achieved, *i.e.*

TABLE 2

Physicochemical properties and kinetic data at 20 °C for the organic systems used

| | | DEA-ethanol | | | DEA-ethyleneglycol | | |
|--|---|---|--------|--------|---|--------|--------|
| C_{B0} | (kmol m ⁻³) | 0.0435 | 0.0440 | 0.0450 | 0.0450 | 0.0640 | 0.0850 |
| ρ_L | (kg m ⁻³) | 805 | 805 | 806 | 1081 | 1109 | 1110 |
| $10^3 \mu_L$ | Pa s | 1.52 | 1.52 | 1.52 | 20.85 | 21.42 | 21.52 |
| $10^3 \sigma_L$ | N m ⁻¹ | 24.2 | 23.9 | 23.2 | 48.0 | 47.8 | 47.0 |
| Order of the chemical reaction with respect to | | | | | | | |
| solute | | 1 | | | 1 | | |
| reactant | | 2 | | | 2 | | |
| $D_A \times 10^9$ | m ² s ⁻¹ | 3.250 | | | 0.251 | | |
| He | Pa m ³ kmol ⁻¹ | 8.57×10^5 | | | 28.60×10^5 | | |
| $k_{m,n}$ | $\frac{1}{s} \left(\frac{\text{m}^3}{\text{kmol}} \right)^{m+n-1}$ | 133 | | | 304 | | |
| ϕ | kmol m ⁻² s ⁻¹ | $1.34 \times 10^{-5} PC_{B0} D_A^{0.5}$ | | | $0.78 \times 10^{-5} PC_{B0} D_A^{0.5}$ | | |

$$C_{A0} \ll C_A^* \quad (2)$$

If one postulates that the chemical reaction *within the liquid film is negligible*, it is then possible to write the following mass conservation equation:

$$k_L a (C_A^* - C_{A0}) = k_3 \epsilon \beta C_{A0} C_{B0}^2 \quad (3)$$

which can be rearranged as

$$\frac{C_{A0}}{C_A^*} = \frac{1}{1 + (k_3 \epsilon \beta C_{B0}^2 / k_L a)} \quad (4)$$

If the value of $(k_3 \epsilon \beta C_{B0}^2 / k_L a) \gg 1$, eqn. (4) gives

$$\frac{C_{A0}}{C_A^*} \ll 1.0 \quad (5)$$

Both inequalities (1) and (5) provide the conditions for the slow reaction regime as

$$\frac{k_L a}{k_3 \epsilon \beta} \ll C_{B0}^2 < \frac{k_L^2}{10 D_A k_3} \quad (6)$$

These two inequalities allow one to choose an appropriate value of C_{B0} , which satisfies the slow reaction conditions if approximate values for k_L , D_A and k_3 are known, and to verify the obtained values of $k_L a$ when k_3 , ϵ , and β are measured. In a trickle-bed reactor, if one assumes

(i) the gas-side resistance to mass transfer is negligible,

(ii) the two fluid phases are flowing in a plug flow, and

(iii) the total pressure varies linearly with the packing height, it is then possible to write the following two mass conservation equations:

$$\Phi = - \int_{Y_1}^{Y_0} \dot{N}_G dY = \int_0^Z k_L a (C_A^* - C_{A0}) \Omega dh \quad (7)$$

and

$$\int_{C_{B0i}}^{C_{B0}} Q_L dC_{B0} = \int_{Y_1}^Y z \dot{N}_G dY \quad (8)$$

The solubility of the solute gas in the liquid phase at thermodynamic equilibrium can be expressed by Henry's law as

$$C_A^* = \frac{P}{H_e} \quad (9)$$

In order to calculate $k_L a$ under various operating conditions in a trickle-bed reactor, the following procedure was used:

(i) From eqn. (3), C_{A0} is expressed as

$$C_{A0} = C_A^* \left(1 + \frac{C_{B0}^2}{\gamma k_L a} \right)^{-1} \quad (10)$$

where $\gamma = (k_3 \epsilon \beta)^{-1}$.

(ii) Integration of eqn. (8) gives

$$C_{B0} = \frac{\alpha + Y}{\theta} \quad (11)$$

where $\theta = Q_L / z \dot{N}_G$, and $\alpha = \theta C_{B0i} - Y_1$.

(iii) Substitution of C_{B0} from eqn. (11) into eqn. (10) gives

$$C_{A0} = C_A^* \left\{ 1 + \frac{(\alpha + Y)^2}{\gamma \theta^2 k_L a} \right\}^{-1} \quad (12)$$

(iv) Under the operating conditions used, the gas was assumed ideal, Dalton's law is applied, and eqn. (9) is rewritten as

$$C_A^* = y \frac{P_{tm}}{H_e} = \frac{Y}{(1 + Y)} \frac{P_{tm}}{H_e} \quad (13)$$

where P_{tm} is the total mean pressure and y is the mole fraction of the solute gas in the gaseous mixture.

(v) By substituting C_{A0} from eqn. (12) into eqn. (7) and using eqn. (13) to express C_A^* , the following expression is obtained:

$$k_L a = \frac{\int_{Y_0}^{Y_1} \{(1 + Y)/Y\} dY}{R_0 - \gamma \theta^2 \int_{Y_0}^{Y_1} \{(1 + Y)/Y(\alpha + Y)^2\} dY} \quad (14)$$

where

$$R_0 = \frac{P_{tm} \Omega Z}{\dot{N}_G H_e}$$

and

$$P_{tm} = \frac{P_{ti} + P_{to}}{2}$$

The analytical integration of eqn. (14) allowed the calculation of $k_L a$ as follows. If $\alpha = 0$, eqn. (14) becomes

$$k_L a = \frac{(Y_1 - Y_0) + \ln(Y_1/Y_0)}{R_0 - (\gamma \theta^2 / 2) \{(2/Y_0) - (2/Y_1) + (1/Y_0^2) - (1/Y_1^2)\}} \quad (15)$$

and if $\alpha \neq 0$, eqn. (14) yields

$$k_L a = \left\{ (Y_1 - Y_0) + \ln \left(\frac{Y_1}{Y_0} \right) \right\} \times \left[R_0 - \gamma \theta^2 \left(\frac{\alpha - 1}{\alpha} \right) \left(\frac{1}{\alpha + Y_0} - \frac{1}{\alpha + Y_1} \right) - \frac{\gamma \theta^2}{\alpha^2} \ln \left\{ \frac{Y_1(\alpha + Y_0)}{Y_0(\alpha + Y_1)} \right\} \right]^{-1} \quad (16)$$

4 EXPERIMENTAL RESULTS AND DISCUSSION

In the present work $k_L a$, the total liquid hold-up β , and the pressure drop ΔP were independently measured for each reading. At stabilized liquid and gas flow rates, the liquid conversion was measured by an acidimetric titration of the reactant in a liquid phase, whereas the absorbed gas was determined by a chromatographic analysis of the solute in the gaseous phase at the inlet and exit of the reactor. The total liquid hold-up was measured by a gravimetric method [10, 20, 21] and the two-phase pressure drop using differential manometers located at the top and bottom of the packing.

The two inequalities presented in relationship (6) were verified, and an example is shown in Table 3. This confirmed that a slow reaction regime in the bulk liquid was established for measuring the $k_L a$ values presented in this investigation. Also, the assumption of negligible gas-side resistance to mass transfer in trickle bed reactor was satisfied as shown in Appendix A and Table 3.

TABLE 3

Verification of a slow reaction regime and of a negligible gas phase resistance

| System | L | G | β | $k_L a$ | $\frac{\epsilon \beta k_3 C_{B0}^2}{k_L a}$ | $\frac{k_L^2}{10 D_A k_3 C_{B0}^2}$ | $R_G^a \times 10^4$ |
|---------------------------------|-----|-------|---------|---------|---|-------------------------------------|---------------------|
| | | | | | | | |
| DEA-ETH ($C_{B0} = 0.045$) | 4 | 0.101 | 0.540 | 0.023 | 2.46 | 1.95 | -2.66 |
| | 4 | 0.141 | 0.506 | 0.025 | 2.14 | 3.23 | +2.33 |
| | 6 | 0.050 | 0.680 | 0.015 | 4.78 | 2.04 | -1.13 |
| | 6 | 0.102 | 0.607 | 0.041 | 1.54 | 3.50 | +4.70 |
| | 8 | 0.050 | 0.741 | 0.027 | 2.85 | 3.86 | -5.50 |
| DEA-ETG ($C_{B0} = 0.045$) | 4 | 0.139 | 0.814 | 0.073 | 2.66 | 9.30 | +1.50 |
| | 4 | 0.181 | 0.774 | 0.066 | 2.78 | 11.36 | +3.90 |
| | 6 | 0.088 | 0.924 | 0.085 | 2.57 | 13.21 | +1.80 |
| | 6 | 0.141 | 0.859 | 0.139 | 1.47 | 16.44 | -5.20 |
| | 8 | 0.089 | 0.949 | 0.106 | 2.13 | 19.79 | -1.20 |

^a R_G values are very sensitive to the round-off errors in β , $k_L a$, and C_{A0}/C_A^*

Figures 1, 2 and 3 present the effect of solution viscosity on $k_L a$, $(\Delta P/Z)_{LG}$, and β respectively. From these figures, it can be seen that the two-phase pressure drop and mass transfer coefficients increase with increasing G at constant L and also increase with increasing L at constant G . The total liquid hold-up values, on the other hand, increase with L at constant G and decrease

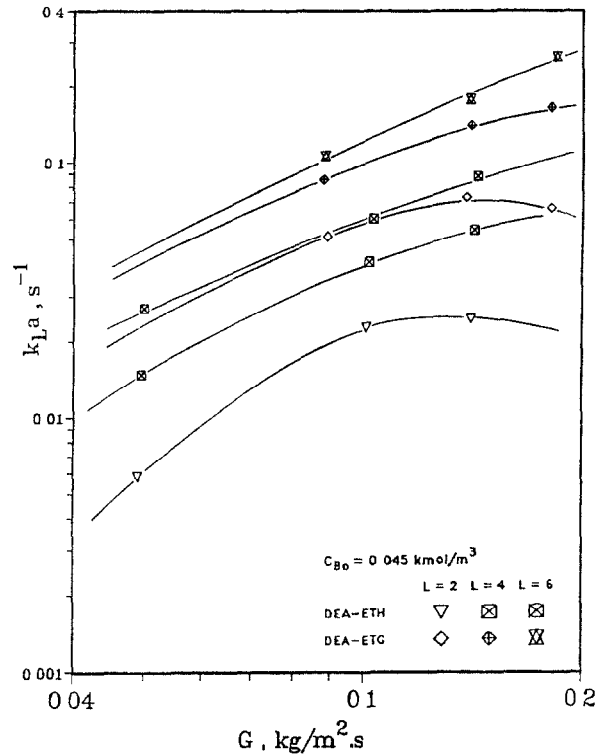


Fig 1 Influence of the solution viscosity on $k_L a$

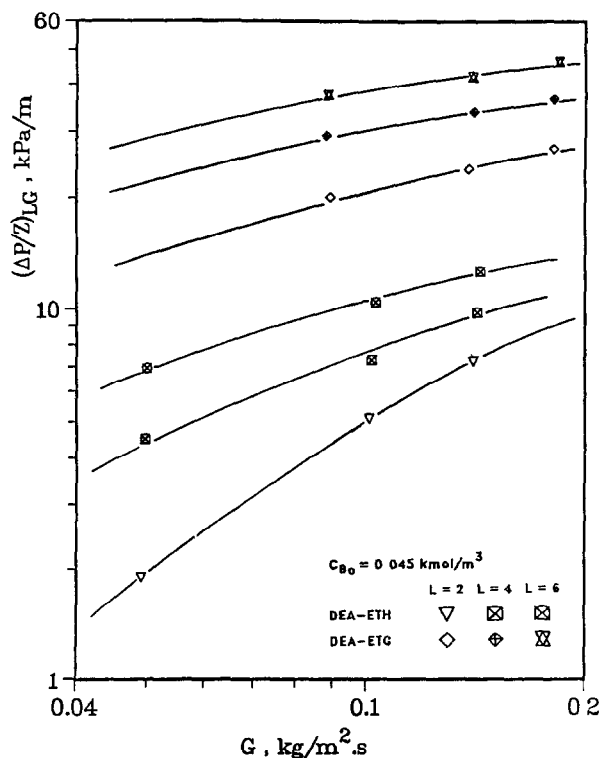


Fig 2 Influence of the solution viscosity on $(\Delta P/Z)_{LG}$

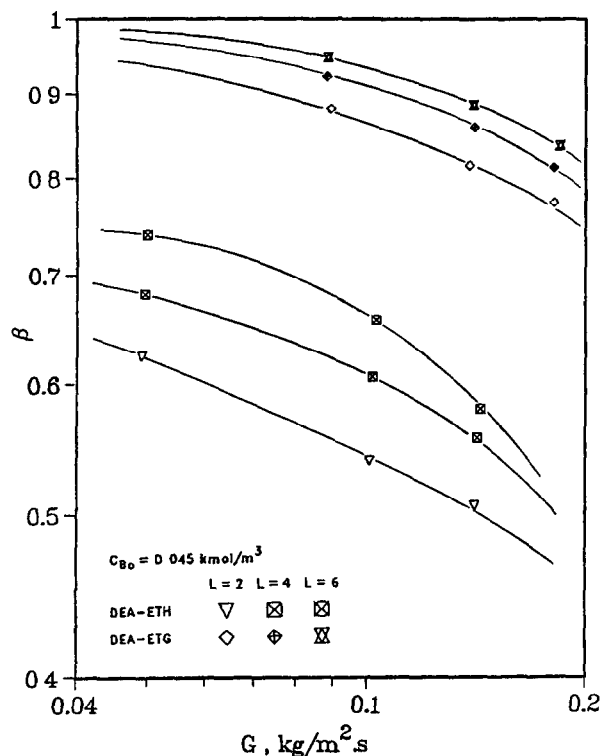


Fig 3 Influence of the solution viscosity on β

with G at constant L . This behavior of low-viscosity (DEA-ethanol) and high-viscosity (DEA-ethyleneglycol) solutions is similar to that observed by Morsi *et al* [9, 11] for non-viscous and viscous organic liquids. From Figs. 1, 2 and 3 it is also clear that at the same L and G , the k_{La} , $(\Delta P/Z)_{LG}$, and β values increase with increasing the solution viscosity. Quantitatively, the values obtained for viscous DEA-ethyleneglycol solution are about four times those measured for non-viscous DEA-ethanol solutions. This behavior can be related to higher static liquid hold-up, a thicker liquid film, higher friction losses, and elevated gas-liquid interfacial areas expected for viscous solutions when compared with those expected for non-viscous solutions. Morsi *et al* [11] proposed correlations for predicting the two-phase pressure drop and total liquid hold-up for organic viscous liquids. The correlations were tried and they were able to predict the experimental values with high accuracy

5 CORRELATION OF k_{La}

Figure 4 shows a comparison between the present experimental data for organic solutions and the correlations proposed by Satterfield [1], and Charpentier [2]. From this figure it can be seen that the present data cannot be correlated by either of these correlations. This is not surprising because these reported correlations were proposed for highly ionic aqueous solutions. Figure 5 presents a plot of k_{La} as a function of E_L (the specific energy dissipated in the two-phase flow, when neglecting G and considering the intrinsic liquid velocity). This representation takes into account the influence of the total liquid hold-ups on k_{La} values. The experimental data points were scattered, but a correlation with $\pm 50\%$ could be obtained. The experimental values of k_{La} were correlated as a function of friction forces in two-phase downflow, and the diffusion coefficient of the solute gas in the liquid phase by the following equation:

$$\frac{k_{La}}{D_A^{1/2}} = 1.4 \times 10^{-3} \lambda_{LG}^{1.46} \quad (17)$$

for $2000 < \lambda_{LG} < 50\,000 \text{ Pa m}^{-1}$, where

$$\lambda_{LG} = \left(\frac{\Delta P}{Z} \right)_{LG} + \bar{\rho} g \quad (18)$$

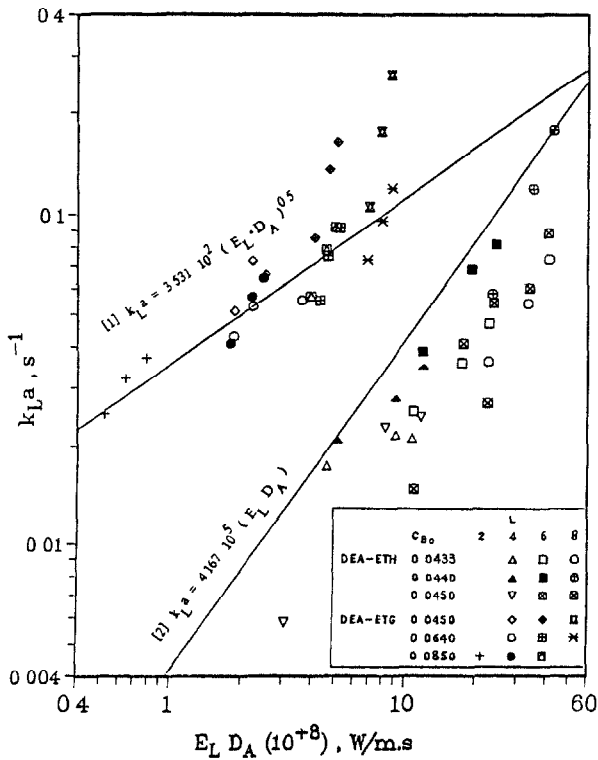


Fig 4 Comparison of k_La experimental values with the correlation of (a) Satterfield [1], (b) Charpentier [2]

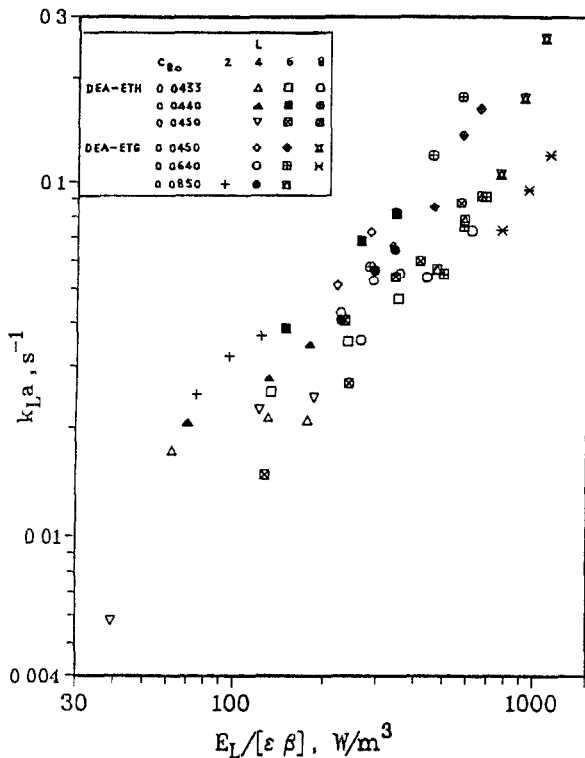


Fig 5 Correlation of k_La as a function of $E_L/\epsilon\beta$

and

$$\bar{\rho} = \frac{L + G}{L/\rho_L + G/\rho_G} \quad (19)$$

This empirical correlation is strictly valid for the weak gas-liquid interaction regime (trickling flow) and under the conditions mentioned above. Figure 6 shows this correlation, and as can be seen eqn. (17) predicts the experimental k_La values with $\pm 35\%$.

Midoux *et al* [24] previously correlated the gas-liquid interfacial areas, and from their correlation the following equation can be deduced for the systems used in this study

$$a = 0.66 \lambda_{LG}^{0.65} \quad (20)$$

for $1000 \leq \lambda_{LG} \leq 60\,000 \text{ Pa m}^{-1}$.

The division of eqn. (17) by eqn. (20) allows the estimation of the true liquid-side mass transfer coefficient k_L . It is obvious that the estimated k_L values depend on $\lambda_{LG}^{0.81}$, i.e. on the hydrodynamics of the gas-liquid interaction. The values are also proportional to $D_A^{1/2}$ which is consistent with k_L values predicted by the penetration theory (the Higbie model).

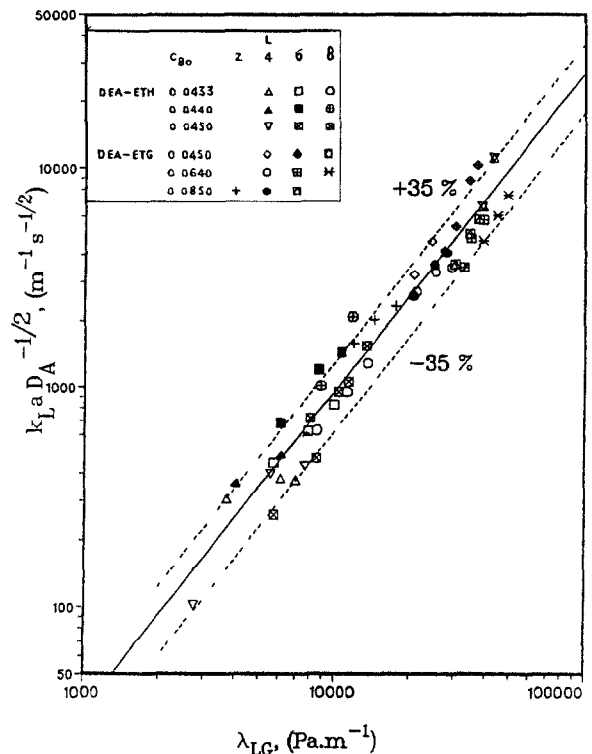


Fig 6 Correlation of k_La

6 CONCLUSIONS

From the preceding results and discussions, the following conclusions can be drawn:

(i) The $k_L a$, $(\Delta P/Z)_{LG}$, and β values for viscous solutions of DEA-ethyleneglycol were about four times those obtained for low-viscosity DEA-ethanol solutions, under the same G and L .

(ii) The solutions used enabled the formulation of an empirical correlation to predict the $k_L a$ values.

(iii) The estimated k_L values were directly dependent on the gas-liquid interaction and were proportional to the square root of the solute gas diffusion coefficient in the liquid phase.

7 NOTATION

| | |
|-------------------|--|
| a | gas-liquid interfacial area per unit volume of the reactor ($\text{m}^2 \text{m}^{-3}$) |
| a_c | specific surface area of the packing per unit volume of the reactor ($a_c = 6(1 - \epsilon)/d_p$) ($\text{m}^2 \text{m}^{-3}$) |
| C_A^* | solubility of dissolved solute in the liquid (kmol m^{-3}) |
| C_{A0} | concentration of free solute dissolved in the liquid bulk (kmol m^{-3}) |
| C_{B0}, C_{B0i} | concentration of reactant, at the reactor entrance (kmol m^{-3}) |
| D_A | diffusion coefficient of the solute gas ($\text{m}^2 \text{s}^{-1}$) |
| d_p | diameter of the particle (m) |
| E_L | specific energy dissipated in two-phase downflow ($E_L = (\Delta P/Z)_{LG} \cdot L/\rho_L$) (W m^{-3}) |
| g | acceleration due to gravity (m s^{-2}) |
| G | superficial gas mass velocity ($\text{kg m}^{-2} \text{s}^{-1}$) |
| Ha | Hatta number (dimensionless) |
| He | Henry's constant ($\text{Pa m}^3 \text{kmol}^{-1}$) |
| h | axial distance from the top of the column (m) |
| k_3 | reaction rate constant of third order ($\text{m}^3 \text{kmol}^{-1})^2 \text{s}^{-1}$) |
| k_G | gas-side mass transfer coefficient ($\text{kmol Pa}^{-1} \text{m}^{-2} \text{s}^{-1}$) |
| $k_G a$ | volumetric gas-side mass transfer coefficient ($\text{kmol Pa}^{-1} \text{m}^{-3} \text{s}^{-1}$) |
| k_L | liquid-side mass transfer coefficient (m s^{-1}) |

| | |
|---------------|--|
| $k_L a$ | volumetric liquid-side mass transfer coefficient (s^{-1}) |
| $k_{m,n}$ | reaction rate constant with partial orders m and n ($(\text{m}^3 \text{kmol}^{-1})^{m+n-1} \text{s}^{-1}$) |
| L | superficial liquid mass velocity ($\text{kg m}^{-2} \text{s}^{-1}$) |
| m, n | reaction partial orders with respect to solute, reactant |
| \dot{N}_G | inert gas molar rate (kmol s^{-1}) |
| P, P_i | partial pressure of the solute gas, at gas-liquid interface (Pa) |
| Q_L | volumetric rate of the liquid phase ($\text{m}^3 \text{s}^{-1}$) |
| R_G | volumetric gas-side mass transfer resistance (s) |
| Y, Y_1, Y_0 | gaseous molar ratio of the solute to inert, at the inlet, at the outlet of the reactor |
| Z | packing height (m) |
| z | stoichiometric coefficient (dimensionless) |

Greek letters

| | |
|---------------------|---|
| β | total liquid hold-up expressed in fraction of interparticle void volume (dimensionless) |
| $(\Delta P/Z)_{LG}$ | two-phase pressure loss per unit length of the packed bed (Pa m^{-1}) |
| ϵ | void fraction of the packed bed (dimensionless) |
| μ_L | dynamic viscosity of the solution (Pa s) |
| ρ_L | solution density (kg m^{-3}) |
| $\bar{\rho}$ | average fluids density (kg m^{-3}) |
| σ_L | solution surface tension (N m^{-1}) |
| Φ | total rate of the solute gas absorption (kmol s^{-1}) |
| ϕ | specific rate of the solute gas absorption ($\text{kmol m}^{-2} \text{s}^{-1}$) |
| λ_{LG} | two-phase parameter (Pa m^{-1}) |
| Ω | cross-sectional area of the column (m^2) |

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APPENDIX A

From the conservation equation, the following expressions can be obtained:

$$\Phi = k_L a (C_A^* - C_{A0}) \Omega Z \quad (\text{A1})$$

$$\Phi = k_G a (P_A - P_A^*) \Omega Z \quad (\text{A2})$$

$$\Phi = k_3 \epsilon \beta C_{A0} C_{B0}^2 \Omega Z \quad (\text{A3})$$

Henry's law provides

$$C_A^* = P_A^* / H_e \quad (\text{A4})$$

By eliminating C_{A0} and P_A^* among the above equations, expression (A5) can be deduced

$$\frac{P_A}{k_L (C_A^* - C_{A0})} = \frac{1}{k_G} + \frac{H_e}{k_L} + \frac{a H_e}{k_3 \epsilon \beta C_{B0}^2} \quad (\text{A5})$$

If the gas-side resistance to mass transfer is negligible, i.e. $P_A = P_A^*$, expression (A5) can be rewritten as

$$\frac{1}{k_G a H_e} = R_G \quad (\text{A6})$$

where

$$R_G = \frac{1}{k_L a (1 - C_{A0}/C_A^*)} - \frac{1}{k_L a} - \frac{1}{k_3 \epsilon \beta C_{B0}^2} \quad (\text{A7})$$

R_G represents the volumetric gas-side mass transfer resistance expressed in seconds. The calculated values of R_G , listed in Table 3, clearly show that the gas-side resistance to mass transfer was negligible. Therefore, the assumption made to neglect the gas-side resistance to obtain $k_L a$ values in the trickle-bed reactor was satisfied.