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Evaluation of Liquid Phase Mass Transfer Coefficient from the Residence Time Observation in a Glass-beads Packed Bed

ガラス球充填層の滞留時間分布からの液側物質移動係数の算定

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Abstract

The method, which enabled to evaluate the liquid phase mass transfer coefficient from the observation of residence time curve in packed bed of insoluble glass beads, was proposed. The mass transfer coefficient was derived from the moment integration of the residence time curve (or step response). The coefficient was correlated with flow condition in terms of the J_D factor against Reynolds number. The correlation between the J_D factor and Reynolds number shows good agreement with mass transfer data of previous investigators with soluble-beads-packed beds. This method is simple and useful tool for evaluation of liquid phase mass transfer coefficient in packed beds.

和 文 要 旨

不溶性のガラス球を充填した層の滞留時間分布曲線を実測することにより、実測曲線から液側物質移動係数を算定し得る方法を提案した。液側物質移動係数は滞留時間分布曲線（または、ステップ応答）をモーメント積分することにより算定した。算定した物質移動係数は流動条件と J_D ファクター対レイノルズ数の形で相関した。本方法で求めた相関は、従来の溶解性の球を充填した層による物質移動データと良好な一致を示した。本方法は簡単で有効な物質移動係数の測定法であることがわかった。

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Introduction

The liquid phase mass transfer between flow-through-fluid and solids in a packed bed is essential in many kinds of unit operations, such as ion exchange, catalytic reaction, adsorption, solid-liquid extraction, etc., and has been studied by many investigators. Carberry³ derived a theoretical equation for liquid phase mass transfer in a packed bed, applying the boundary layer theory to the flow around each bead packings in a bed. Pfeffer¹³ led a theoretical equation based on the free surface model in low Reynolds number region. Kataoka, Yoshida and Ueyama⁸ proposed a theoretical equation on the ground of the hydraulic radius model⁴. Their theoretical equations agreed well with the previous experimental data^{12 14 15}. The all previous experiments were carried out with soluble solid particles, such as benzoic acid bead.

In this work, the method which can evaluate the liquid phase mass transfer coefficient from the residence time curve was proposed. The residence time curves in a bed packed with insoluble glass spheres were measured, when step concentration change was imposed on the inlet stream. The pressure drop in the bed was also measured to ensure the validity of the glass-beads-packed bed employed for the experiment. The variance from the mean residence time was calculated by the moment integration⁷. From the variance, the liquid phase mass transfer coefficient was calculated. The coefficient was plotted as J_D factor against Reynolds number, and was discussed on the agreement with the previous data of soluble-beads-packed beds.

Experimental

The schematic diagram of the experimental apparatus is shown in Fig. 1. The cylindrical column (see Fig. 2), made of transparent methacrylic resin, was 55mm in inside diameter and 307mm in length of packed section. A flow distributor⁹ which had the tournamented paths of flow and gave uniform radial flow distribution was used. For more uniform distribution of flow, the test section ($\epsilon = 0.386$) of 3mm glass beads packing was fitted with packing section

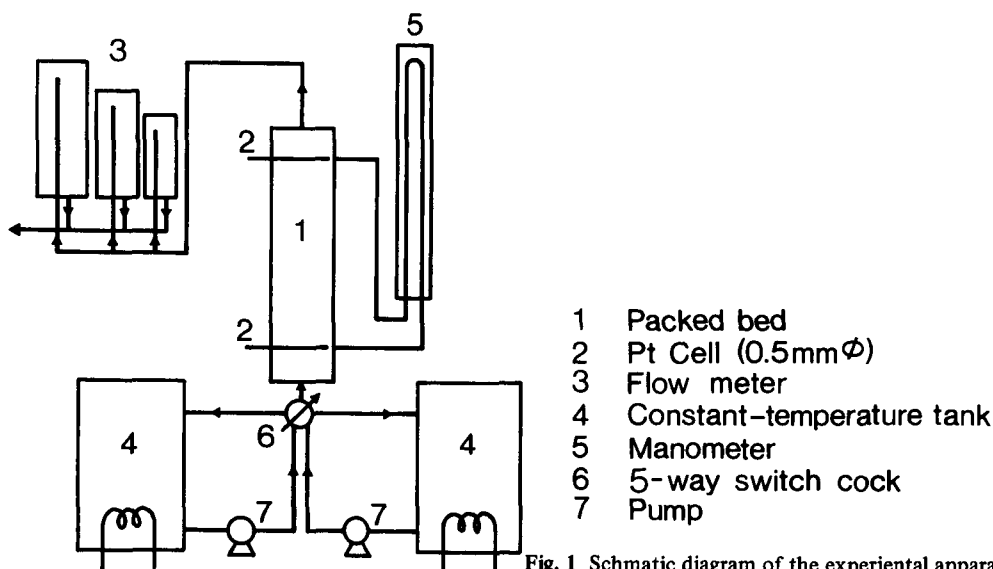


Fig. 1 Schematic diagram of the experiental apparatus

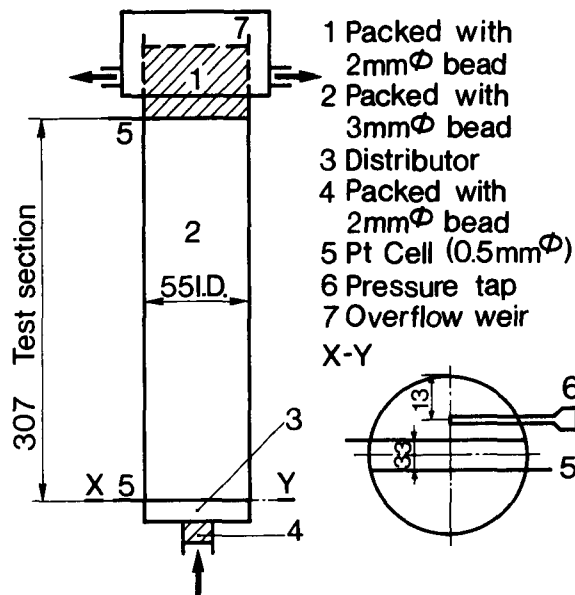


Fig. 2 Beads packed bed

with bead of 2mm diameter before and behind. The step response of test section was observed as follows. Stepwise (not perfectly step-shaped) concentration changes both at inlet and outlet of the test section were detected by electric conductivity measurement, when step change in concentration was imposed on the fluid at the upstream flow distributor. The concentration change was given by a five-way switch cock⁹ made of Pylex glass. Two fluids, distilled water at $25 \pm 0.2^\circ\text{C}$ containing, respectively, 0.002N and 0.005N hydrochloric acid, were switched by the cock to select either, and to give step-wise concentration change. Concentration was detected by electric conductivity cells of 0.5mm ϕ Platinum wires which were set at two boundary cross sections for averaging concentration change. The two recorded step-wise responses were processed by a digital computer with step-testing algorithm, which had been developed elsewhere¹⁸, and converted to a residence time (or step response) curve for the test section, via frequency domain.

Analysis by dynamic model

The liquid phase mass transfer coefficient in packed beds has ever been evaluated from the steady state outlet concentration when soluble-solid-beads are used as packings in beds, through which liquid is flowing. In our analysis, soluble tracer substance was introduced from the inlet of bed, and diffused from the body flow (or main flow part) to the stagnant boundary layer on insoluble packings, and diffused back from the boundary layer to the body flow. The diffusion to the stagnant layer gave rise to apparent axial dispersion of tracer, as had been shown in a previous work^{11 16}. The mass transfer coefficient was able to be interpreted as the reciprocal of the mass transfer resistance between stagnant layer on the packed bead surface and the body flow. The detail explanation by dynamic model to obtain the mass transfer coefficient was given as follows. This dynamic model assumes two phases for the fluid in

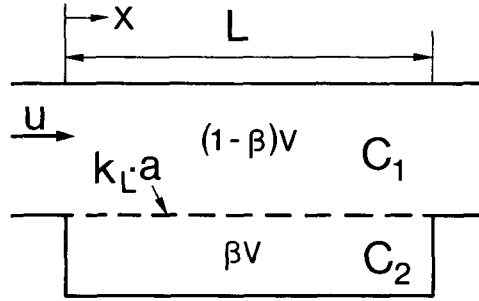


Fig. 3 Illustrated dynamic model with a motion phase and a rest phase

packed beds, that is, a motion phase and a rest phase which are corresponding respectively to the bulk flow through the beds and the stagnant fluid in the vortices before and behind the packings and in boundary layer. These fluid behaviours were confirmed by the photographic observations of streamlines in a two dimensional packed bed¹ and a three dimensional bead-packed bed¹⁰. In the motion phase, fluid is assumed to flow without dispersion in plug flow at velocity of u and the volume is $V(1-\beta)$, as illustrated in Fig. 3. The rest phase is stagnant phase having the volume of $V\beta$. Resistance for mass transfer between these two phases is also postulated as concentrated at the boundary of two phases. This two-phase-concept is just identical with the double-film-theory, because steady states of Eqs. (1) and (2) (stated below) give rise to material balance of double-film-theory. According to these assumptions, material balance of tracer substance gives Eqs. (1) and (2).

$$(1-\beta)\frac{\partial C_1}{\partial t} = -u\left(\frac{\partial C_1}{\partial x}\right) + k_L \cdot a(C_2 - C_1) \quad (1)$$

$$\beta\frac{\partial C_2}{\partial t} = k_L \cdot a(C_1 - C_2) \quad (2)$$

where a is the surface area per bed volume. When soluble spheres are utilized, C_2 is equal to C_s . Boundary conditions are written as Eqs. (3) and (4).

$$X=0 \quad C_1 = C^{in} \quad (3)$$

$$X=L \quad C_1 = C^{out} \quad (4)$$

Transfer function, derived from Eqs. (1) ~ (4), can be written as Eq. (5).

$$G(S) = \frac{C^{out}(S)}{C^{in}(S)} = \exp\left[-(1-\beta)S - \frac{N_L \cdot \beta S}{\beta S + N_L}\right] \quad (5)$$

where $N_L = k_L \cdot a \times (L/u)$. Mean residence time, $\bar{\tau}$, and variance from mean residence time, $\bar{\tau}^2$, are also derived from the transfer function and represented as Eq. (6).

$$\bar{\tau} = 1.0 \quad \text{and} \quad \bar{\tau}^2 = 2\beta^2/N_L \quad (6)$$

For the observed residence time curve (or step response), $C_s(\tau)$, experimental mean residence time, $\bar{\tau}_{exp}$, and experimental variance from mean residence time, $\bar{\tau}_{exp}^2$, are represented as Eqs. (7) and (8) by utilizing the difference, $\bar{C}_s(\tau) = [C(\infty) - C_s(\tau)]$ from steady state value, $C(\infty)$ ¹⁷.

$$\bar{\tau}_{exp} = \int_0^{\infty} \bar{C}_s(\tau) d\tau \quad (7)$$

$$\bar{\tau}_{exp}^2 = 2 \times \int_0^{\infty} \bar{C}_s(\tau) \cdot \tau d\tau - (\bar{\tau}_{exp})^2 \quad (8)$$

By these Eqs. (7) and (8), the variance of the step response can be computed by the τ -weighted numerical integration with experimental step response curve, $C_s(\tau)$. By equating Eq. (6) with Eq. (8), i.e., $\bar{\tau}^2 (=2\beta^2/N_L) = \bar{\tau}_{\text{exp}}^2$, $N_L = 2\beta^2/\bar{\tau}_{\text{exp}}^2$ can be derived. This relationship between $\bar{\tau}_{\text{exp}}^2$ and N_L enables to evaluate N_L or k_L from experimentally observed variance, $\bar{\tau}_{\text{exp}}^2$. On the estimation, β needs to be also evaluated experimentally. $(1-\beta)$ is dead time of residence time curve as is found in Eq. (5) (the term, $\exp[-(1-\beta)S]$). From the dead time of experimental response curve, which shows sharp increment after dead time, β can be read and used to calculate N_L . The number of transfer unit, N_L is rewritten as $N_L = k_L \cdot a \times (L/u)$. a is the effective interfacial area, and is equal to $6 \times (1-\epsilon)/d_p$ in packing of spheres. Thus, the mass transfer coefficient k_L becomes to $N_L \times u/(L \times a)$.

Discussion

By Kataoka et al.⁸, the relationship between modified J_D factor and Reynolds number is presented with hydraulic radius model by Eq. (9).

$$J_D = \left(\frac{1-\epsilon}{\epsilon}\right)^{1/3} \cdot k_L \cdot S_c^{2/3} / u = 1.85 (\text{Re}_P')^{-2/3} \quad (9)$$

where $S_c = 251$ and $\text{Re}_P' = \left(\frac{\epsilon}{1-\epsilon}\right) d_p u / \nu$. J_D obtained experimentally is plotted against modified Reynolds number as shown in Fig. 4. Equation (9) and Carberry's equation are also shown by a solid line and a broken line in Fig. 4. The experimental data are in good agreement with the

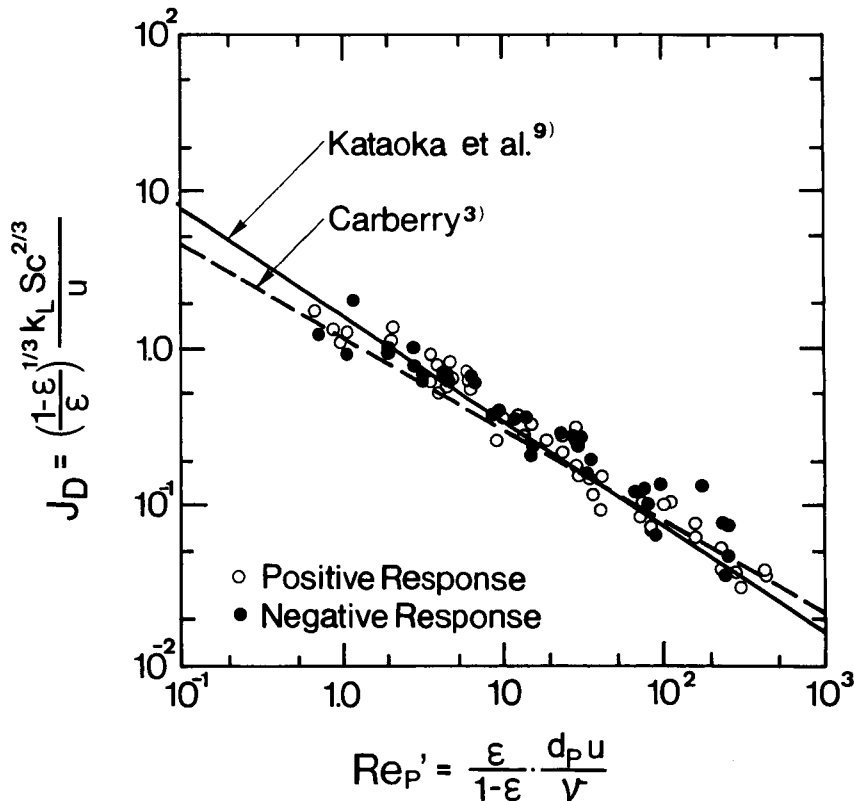


Fig. 4 Plots of J factor of mass transfer against Reynolds number

equation. This fact is indicated that mass transfer coefficient can be also calculated from the dynamic experiment with insoluble particles. Mass transfer coefficient in packed bed is influenced by shapes of packing, because it involves the effect of fluid mixing. When a packing is complex form previous methods is often difficult to evaluate mass transfer coefficient in the packed bed. However, this method can simply evaluate mass transfer coefficient in bed packed with complex packings.

Pressure drop, ΔP , was also measured for the packed bed used for the dynamic analysis. The modified friction factor, $f (= \frac{\epsilon}{1-\epsilon} \times \frac{\Delta P g d_p}{L u^2 \rho})$ is plotted against modified Reynolds number, Re_p' as shown in Fig. 5. The friction factor, f decreases with Reynolds number. Ergun's equation⁶, Kozeny-Carmans' equation⁴ and Burke and Plummerts' experimental data² are also shown. Experimental data are in best agreement with Ergun's equation and Burke and Plummerts' experiment. The packed bed utilized was shown to be valid as a normal Packing bed.

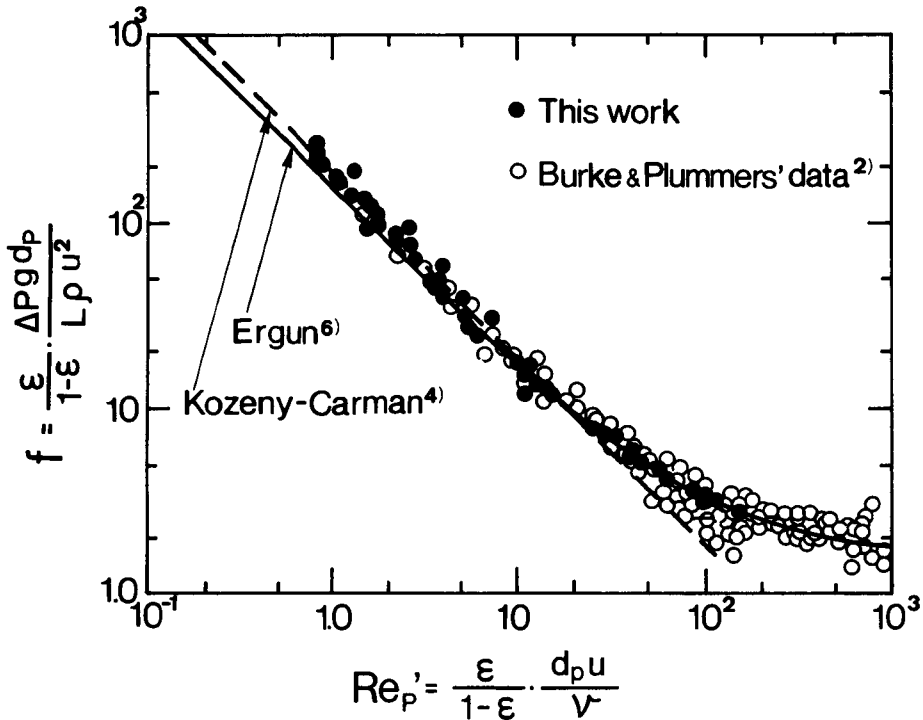


Fig. 5 Plots of friction factor against Reynolds number

Conclusion

By considering the dynamic mass transfer model, liquid phase mass transfer coefficient can be evaluated from the experimental residence time curves utilizing insoluble spheres. The mass transfer coefficient which is determined by this method agrees with that by the steady state method utilizing soluble particle.

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Nomenclature

a	= the surface area of packings per bed volume	[cm ² /cm ³]
C	= dimensionless concentration change of tracer substance normalized by gain ($C = 0 \sim 1$ for positive response, $C = 1 \sim 0$ for negative response)	[-]
$C(\infty)$	= steady state value of C ($= 1.0$ for positive response and $= 0$ for negative response)	[-]
$C_s(\tau)$	= residence time curve (or step response)	[-]
$\bar{C}_s(\tau)$	= difference from steady state value ($= C(\infty) - C_s(\tau)$)	[-]
d_p	= diameter of packing spheres	[cm]
f	= modified friction factor ($= \frac{\epsilon}{1-\epsilon} \times \frac{\Delta p g d_p}{L \rho u^2}$)	[-]
g	= acceleration of gravity	[m/sec ²]
J_D	= modified J factor ($= (\frac{1-\epsilon}{\epsilon})^{1/3} \times k_L \cdot Sc^{2/3} / u$)	[-]
k_L	= liquid phase mass transfer coefficient	[cm/sec]
L	= axial length of packed bed (or test section)	[cm]
N_L	= number of transfer unit	[-]
Re_p'	= modified Reynolds number ($= (\frac{\epsilon}{1-\epsilon}) d_p u / \nu$)	[-]
S	= Laplace's operation	[-]
Sc	= Schmidt number (ν/D)	[-]
t	= real time	[sec]
u	= interstitial linear velocity	[cm/sec]
V	= volume of fluid in voids of test section	[cm ³]
X	= axial distance, flow direction	[cm]
β	= fraction of stagnant phase	[-]
ΔP	= pressure drop	[Kg/cm ²]
ϵ	= void fraction	[-]
ν	= kinematic viscosity	[cm ² /sec]
ρ	= density of fluid	[g/cm ³]
τ	= dimensionless time ($= ut/L$)	[-]
$\bar{\tau}$	= mean residence time with regard to τ	[-]
$\bar{\tau}^2$	= variance from $\bar{\tau}$, 2nd moment	[-]

<Subscript>

1	= body flow (or motion phase)
2	= boundary layer (or stagnant phase)
exp.	= experimental

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