

CORRELATION OF MASS TRANSFER VOLUMETRIC COEFFICIENT WITH POWER INPUT IN STIRRED LIQUID-LIQUID DISPERSIONS

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A continuous-phase mass transfer volumetric coefficient $k_L a$ in stirred liquid-liquid dispersions was measured under many stirring conditions by using the hydrolysis reaction of ester with sodium hydroxide. Droplet diameter was also observed with a microscope during the period of reaction progress, allowing the continuous-phase mass transfer coefficient k_L to be determined. The resultant k_L was correlated with the power input per unit volume and the Sauter mean diameter d_{32} . With the help of the k_L correlation equation, d_{32} was well correlated with the power input per unit volume swept out by the impeller. The $k_L a$ values estimated from both the k_L correlation equation and the d_{32} correlation equation agreed well with the observed values.

Introduction

Liquid-liquid mixing in an agitated vessel is the most common process in chemical industries. The effects of the liquid properties and the stirring conditions on mass transfer coefficient and effective interfacial area must be known in order to clarify the mass transfer in a liquid-liquid dispersion system. Although studies on mass transfer through a droplet surface have been conducted by many investigators,^{8,11,12,13} there are no available correlation equations of continuous-phase mass transfer coefficient k_L and effective interfacial area at the surface of a tiny droplet.

In the previous paper,⁷⁾ k_L for tiny droplet was measured by using the hydrolysis of pure ester with alkali, and it was shown that the mass transfer behavior of the tiny droplet is very similar to that of a suspended solid sphere.

The aim of this paper is to clarify the effects of the liquid properties and the stirring conditions on the continuous-phase mass transfer volumetric coefficient $k_L a$ for the tiny droplet by evaluating k_L and the Sauter mean diameter d_{32} . k_L and d_{32} are measured under various experimental conditions with the reaction method developed in the previous paper⁷⁾, and correlation equations for k_L and d_{32} are proposed. A continuous-phase mass transfer volumetric coefficient $k_L a$ for tiny droplet is evaluated by using the correlation equations for k_L and d_{32} and is compared with the observed $k_L a$.

1. Reaction Model

We consider the hydrolysis of ester with alkali as a slow reaction.¹⁾



For a droplet of ester-toluene mixture in aqueous phase, the reaction is considered to proceed under the following assumptions vi and vii in addition to the assumptions i to v in the previous paper.⁷⁾

- vi) Toluene does not dissolve at all into the aqueous phase.
- vii) The volume of the mixture equals the sum of those of the pure ester and toluene.

The reaction rates of the ester with the alkali can be expressed as

$$dC_A/dt = k_L a(C_{Ai} - C_A) - k_2 C_A C_B \quad (2)$$

$$dC_B/dt = -k_2 C_A C_B \quad (3)$$

The concentration profiles of C_A and C_B are shown in Fig. 1 in reference 1. At low volume fraction of droplet ϕ , the apparent concentrations of the ester and the alkali, \bar{C}_A and \bar{C}_B , can be expressed as

$$\bar{C}_A = C_A(1 - \phi) + \phi C_{AP} \approx C_A + \phi C_{AP} \quad (4)$$

$$\bar{C}_B = (1 - \phi)C_B \approx C_B \quad (5)$$

Initial conditions at $t=0$ are expressed as

$$C_A = C_{A0} = C_{Ai}, \quad C_B = C_{B0} = \bar{C}_{B0}, \\ \bar{C}_A = \bar{C}_{A0}, \quad \bar{C}_B = \bar{C}_{B0} \quad (6)$$

From assumption vii the droplet volume V_p and the ester concentration in the droplet C_{AP} satisfy the

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following relations:

$$\begin{aligned} V_P &= V_A + V_T \\ C_{AP}V_P &= C_A^\circ V_A \end{aligned} \quad (7)$$

where V_A and V_T are the volumes of the pure ester and toluene composing the droplet, respectively, and C_A° is the molarity of the pure ester. Equations (4) and (7) hold also at $t=0$, and they give rise to the following relation:

$$\frac{C_{AP}\phi}{C_{AP0}\phi_0} = \frac{\bar{C}_A - C_A}{\bar{C}_{A0} - C_{A0}} = \frac{V_P - V_{T0}}{V_{P0} - V_{T0}} \quad (8)$$

where V_T is constant during the reaction period and equals V_{T0} , because of assumption vi. When the droplet diameter d_p is constant during the reaction period (assumption ii: the constancy of the droplet diameter during the reaction period), the interfacial area of the droplet per unit volume of the aqueous phase, a , is proportional to the volume of the droplet, V_p . Then Eq. (8) gives rise to the following expression:

$$\frac{a}{a_0} = \frac{V_P}{V_{P0}} = X_{A0} \left(\frac{\bar{C}_A - C_A}{\bar{C}_{A0} - C_{A0}} \right) + (1 - X_{A0}) \quad (9)$$

where

$$X_{A0} = V_{A0}/V_{P0} = 1 - (V_{T0}/V_{P0}) \quad (10)$$

The interfacial concentration of n -amyl acetate in aqueous phase for the mixture of the ester and toluene, C_{Ai} , was measured by Alwan *et al.*,¹⁾ where it was not directly proportional to the concentration of the ester in the mixture. Then we assume that C_{Ai} is expressed by Eq. (11) for any ester.

$$C_{Ai} = m(C_{AP})^n \quad (11)$$

where the exponent n is estimated experimentally. Equation (11) must hold for the pure ester as

$$C_{Ai}^\circ = m(C_A^\circ)^n \quad (12)$$

where C_{Ai}° is the solubility of the ester in an aqueous phase. With the help of Eqs. (8) and (10), combination of Eq. (11) with Eq. (12) gives rise to the following expression:

$$C_{Ai} = C_{Ai}^\circ \left\{ \frac{X_{A0}\alpha}{X_{A0}\alpha + (1 - X_{A0})} \right\}^n \quad (13)$$

where

$$\alpha = (\bar{C}_A - C_A)/(\bar{C}_{A0} - C_{A0}) \quad (14)$$

Equations (2), (3) and (13) come to be the following dimensionless expressions with the help of Eqs. (5) and (10) and mass balance ($\bar{C}_{A0} - \bar{C}_A = \bar{C}_{B0} - \bar{C}_B$).

$$\frac{dC_A^*}{dt^*} = K \{ X_{A0}\alpha + (1 - X_{A0}) \} (C_{Ai}^* - C_A^*) - C_A^* \bar{C}_B^* \quad (15)$$

$$d\bar{C}_B^*/dt^* = -C_A^* \bar{C}_B^*/\theta_B \quad (16)$$

$$C_{Ai}^* = C_{Ai}^{\circ*} \left\{ \frac{X_{A0}\alpha}{X_{A0}\alpha + (1 - X_{A0})} \right\}^n \quad (17)$$

where

$$\alpha = \{ 1 - \theta_B(1 - \bar{C}_B^*) - C_A^* \} / (1 - C_{A0}^*) \quad (18)$$

and

$$\begin{aligned} C_A^* &= C_A/\bar{C}_{A0}, \quad \bar{C}_B^* = \bar{C}_B/\bar{C}_{B0}, \\ C_{Ai}^* &= C_{Ai}/\bar{C}_{A0}, \quad \theta_B = \bar{C}_{B0}/\bar{C}_{A0}, \\ K &= k_L a_0/k_2 \bar{C}_{B0}, \quad t^* = k_2 \bar{C}_{B0} t \end{aligned} \quad (19)$$

The dimensionless initial conditions are

$$C_A^* = C_{Ai}^*, \quad \bar{C}_B^* = 1 \quad \text{at} \quad t^* = 0 \quad (20)$$

Equations (15) and (16) are solved simultaneously by the Runge-Kutta method.⁷⁾

The optimum value of K is determined to obtain the continuous-phase mass transfer volumetric coefficient $k_L a_0$ for each run.⁷⁾

The interfacial area of the droplet per unit volume of liquid a_0 is obtained from the observed Sauter mean droplet diameter d_{32} as

$$a_0 = 6\phi_0/d_{32} \quad (21)$$

The initial volume fraction of droplets ϕ_0 , which satisfies the following mass balance equation of ester, is obtained by an iteration method.

$$\phi_0 = (V_{Aad} + V_{T0})/V_w - (C_{Ai}^\circ/C_A^\circ) \{ 1 - (V_{T0}/V_w)/\phi_0 \}^n \quad (22)$$

Here the continuous-phase mass transfer coefficient k_L is obtained from the value of $k_L a_0$.

2. Experimental

Two kinds of experimental systems are used, as shown in Table 1. The experimental apparatus and procedures are almost the same as those described in the previous paper.⁷⁾ Baffled reaction vessels are made of glass, and paddle impellers used are made of stainless steel. The impeller is set in the center of the vessel.

The mixture of the ester (n -amyl acetate or n -hexyl acetate) and toluene was added to the stirred water to obtain a dispersed liquid with an apparent concentration of ester of 0.025 mol/l. The water was saturated with the ester and the droplet size was in dynamic equilibrium, usually in 10~15 minutes. The hydrolysis reaction was started by instantaneous addition of sodium hydroxide aqueous solution whose concentration was adjusted to become 0.050 mol/l in the vessel. At this moment no change in droplet size was observed. The progress of the reaction was monitored by measuring the variation of the alkali concentration with both electric conductivity meter and titration. The reaction temperature was 30°C and

the stirred speed of the impeller was 150 to 550 rpm. The initial volume fraction of droplet ϕ_0 was 0.0018 to 0.0081 and the volume fraction ratio ϕ/ϕ_0 unity to

0.0015.

The droplet diameter was measured with a microscope during the reaction period by the same procedure as described in the previous paper.⁷⁾

The physical properties and reaction rate constant of the esters used are listed in Table 2.⁷⁾ For the interfacial concentration of ester C_{Ai} in Eq. (11), the exponent n was estimated experimentally. For the mixture of *n*-amyl acetate and toluene the value of n was estimated as 1.18 from the experimental data by Alwan *et al.*,¹⁾ whereas for the mixture of *n*-hexyl acetate and toluene there were no available data to estimate this value. In this paper, the value of n for *n*-hexyl acetate was estimated to be 1.27 as the value which made the difference between the observed and calculated curves of alkali concentration vs. time minimum for any value of K .

3. Results and Discussion

3.1 Correlation of mass transfer coefficient

By using the Sauter mean droplet diameter d_{32} as a characteristic length and the power input per unit volume P_V as a factor related to a characteristic velocity, the observed mass transfer coefficients, k_L , for both experimental systems are correlated in dimensionless form in Fig. 1. The k_L values are close to the correlation lines for suspended solid sphere by Brian *et al.*,³⁾ Asai *et al.*,²⁾ and Harriott,⁶⁾ though the

Table 1. Experimental systems

	Experiment I			Experiment II		
Dispersed phase	Mixture of ester and toluene (0, 10, 30, 50 vol% of toluene)			Pure ester		
Ester	<i>n</i> -amyl acetate <i>n</i> -hexyl acetate			<i>n</i> -hexyl acetate		
Continuous phase	NaOH aq. solution			NaOH aq. solution		
Vessel	$D = 10, 14, 17$ cm			$D = 14, 17$ cm		
Impeller	d	b	n_p	d	b	n_p
	5.0 cm	1.0 cm	6	7.0 cm	1.4 cm	2
	7.0	1.4	"	"	"	4
	8.5	1.7	"	"	"	6
				"		8
				7.0	0.91	6
				"	1.75	"
				"	2.33	"
				"	3.5	"
Temperature	30°C			30°C		

Table 2. Physical properties and reaction rate constant of esters at 30°C

Ester	$D_A \times 10^{10}$ [m ² /s]	C_{Ai}^0 [mol/l]	k_2 [l/(mol·s)]	$\sigma \times 10^3$ [N/m]			
				0 (pure)	10	30	50 [vol. % toluene]
<i>n</i> -amyl acetate	8.75	0.0135	0.113	15.31	15.98	18.49	21.69
<i>n</i> -hexyl acetate	8.13	0.0039	0.097	14.77	15.69	18.30	21.59

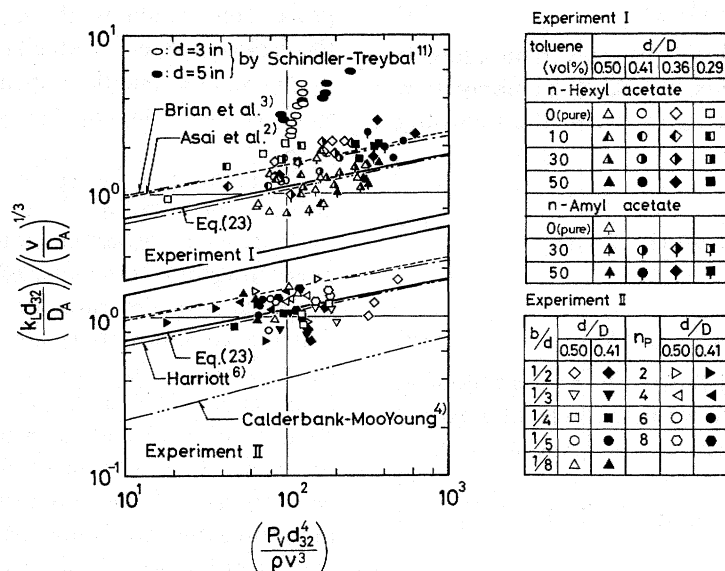


Fig. 1. Dimensionless correlation of mass transfer coefficient for tiny droplet

data by Schindler and Treybal¹¹⁾ are above these correlation lines. The dimensionless correlation equation for the present experimental data can be obtained with the least squares method as

$$\frac{k_L d_{32}}{D_A} = 0.45 \left(\frac{P_V d_{32}^4}{\rho v^3} \right)^{0.193} Sc^{1/3} \quad (23)$$

where the exponent of the variable $(P_V d_{32}^4 / \rho v^3)$ is assumed to be equal to the value of 0.193 by Asai *et al.*,²⁾ because the range of the variable is not large enough to determine the exponent in the present experiment. Equation (23) seems to satisfy well the result by Harriott.⁶⁾

3.2 Estimation of characteristic droplet diameter

In this experiment, a small volume of the dispersed liquid was drawn from the vessel and d_{32} is obtained from the diameters of 400 droplets every five minutes. There is a fear that the d_{32} value might be the that for a local droplet diameter distribution. In this section the characteristic droplet diameter \bar{d}_p over the vessel is evaluated with the observed k_{La} value and it is shown that the observed d_{32} is almost the same as the \bar{d}_p .

The correlation equation, Eq. (23), was originally derived for uniform droplet diameter. Considering the droplet size distribution, the continuous-phase mass transfer volumetric coefficient for the droplet k_{La_0} can be derived from Eqs. (21) and (23), as follows (see Appendix):

$$k_{La_0} = 0.45 \left(\frac{6\phi_0 D_A}{\bar{d}_p^2} \right) \left(\frac{P_V \bar{d}_p^4}{\rho v^3} \right)^{0.193} Sc^{1/3} \quad (24)$$

where \bar{d}_p is defined by Eq. (A-4) in **Appendix**. Equation (24) is rearranged to the following expression.

$$\bar{d}_p = 0.522 \left\{ \frac{6\phi_0 D_A}{(k_{La_0})} \right\}^{0.815} \left(\frac{P_V}{\rho v^3} \right)^{0.157} Sc^{0.272} \quad (25)$$

Then the characteristic droplet diameter \bar{d}_p can be obtained from the measurement of the observed mass transfer volumetric coefficient, k_{La_0} , via Eq. (25).

The resultant characteristic droplet diameter, \bar{d}_p , is plotted against the power input per unit volume swept out by impeller, P_{VI} , in **Fig. 2**, where \bar{d}_p for pure ester is well correlated with P_{VI} instead of $P_V^{5.10}$ and is proportional to the -0.4 power of P_{VI} for both Experiments I and II. Considering the effect of interfacial tension σ , the observed diameter is related to the length parameter, $(\sigma^{0.6} / \rho^{0.2} P_{VI}^{0.4})$, in **Fig. 3**, where the solid line is a correlation equation derived for all data in Experiments I and II with the least squares method, i.e.,

$$\bar{d}_p = 0.261 (\sigma^{0.6} / \rho^{0.2} P_{VI}^{0.4}) \quad (26)$$

The Sauter mean diameter d_{32} observed with a videocamera is compared with $(\bar{d}_p)_{cal}$ calculated with

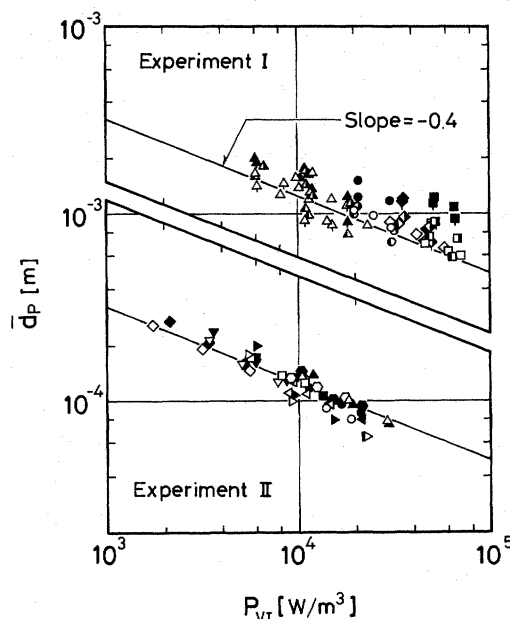


Fig. 2. Observed characteristic droplet diameter. Keys are referred to Fig. 1.

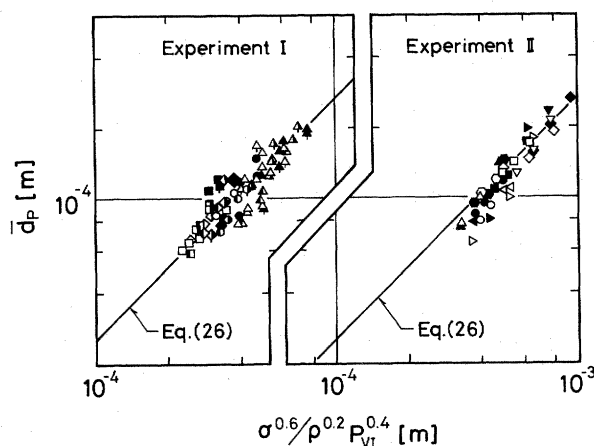


Fig. 3. Observed characteristic droplet diameter with respect to length parameter. Keys are referred to Fig. 1.

Eq. (26), as shown in **Fig. 4**. Equation (26) agrees well with the observed value of d_{32} except for the data for small d/D ratio in Experiment I and the weak stirring condition in Experiment II. Then the Sauter mean droplet diameter in liquid-liquid dispersions can be estimated well with Eq. (26); i.e.

$$d_{32} = 0.261 (\sigma^{0.6} / \rho^{0.2} P_{VI}^{0.4}) \quad (27)$$

McManamey⁸⁾ correlated the Sauter mean droplet diameter with the same equation as Eq. (27), though the coefficient 0.221 in his equation was smaller than the 0.261 in Eq. (27). Davies⁵⁾ correlated also the maximum droplet diameter with an equation similar to Eq. (27), where the estimated coefficient in Eq. (27) from his equation was 0.23 to 0.30.

3.3 Droplet size distribution

The observed cumulative volume frequency, F_V ,

with respect to the dimensionless diameter, d_p/d_{32} , is shown in Fig. 5. All data collapse approximately on a single line on normal distribution paper. The solid line in this figure represents the following equation, decided with all data in both Experiments by the least squares method.

$$F_V = 0.5 \left[1 + \operatorname{erf} \left(\frac{d_p/d_{32} - 1.03}{0.26\sqrt{2}} \right) \right] \quad (28)$$

The dotted line also represents the correlation equation by Wang and Calabrese.¹⁴⁾ The two lines are almost the same, though their slopes are slightly

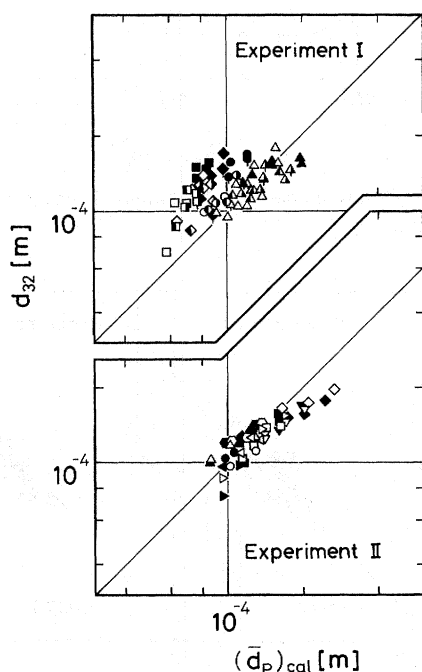


Fig. 4. Comparison of observed Sauter mean droplet diameter with calculated characteristic droplet diameter. Keys are referred to Fig. 1.

different from each other.

3.4 Estimation of volumetric mass transfer coefficient

The mass transfer coefficient k_L can be estimated from Eq. (23). Then the mass transfer volumetric coefficient in liquid-liquid dispersions can be calculated from Eqs. (21), (23) and (27) for given operating conditions. The calculated mass transfer volumetric coefficient from these equations, $(k_L a_0)_{\text{cal}}$, is compared with the observed one, $(k_L a_0)_{\text{obs}}$, in Fig. 6, where the latter agrees well with the former. The data for $d=3$ in by Schindler and Treybal¹¹⁾ agree also well with the calculated values.

Evaluating the effects of the stirred speed of impeller, the impeller diameter and the diameter ratio of impeller to vessel on $k_L a_0$, the mass transfer volumetric coefficient is expressed as

$$k_L a_0 \propto N^{2.052} d^{1.368} (d/D)^{0.578} \quad (29)$$

where the power number, $P/\rho N^3 d^5$, is assumed to be constant for the baffled agitated vessel. All the values of exponents on N , d and (d/D) in Eq. (29) are small compared with those obtained by Skelland and Lee¹²⁾ and Keey and Glen.⁸⁾

Conclusions

For stirred liquid-liquid dispersions of ester in aqueous alkali, the continuous-phase mass transfer coefficient at droplet surface was measured by using the hydrolysis reaction of the ester with the alkali. The observed mass transfer coefficient was close to that for a suspended solid sphere.

A characteristic droplet diameter for the mass transfer in liquid-liquid dispersions was estimated from the observed mass transfer volumetric coefficient, by using the correlation equation of the mass transfer coefficient. This diameter agreed well with the Sauter mean droplet diameter and was correlated with the

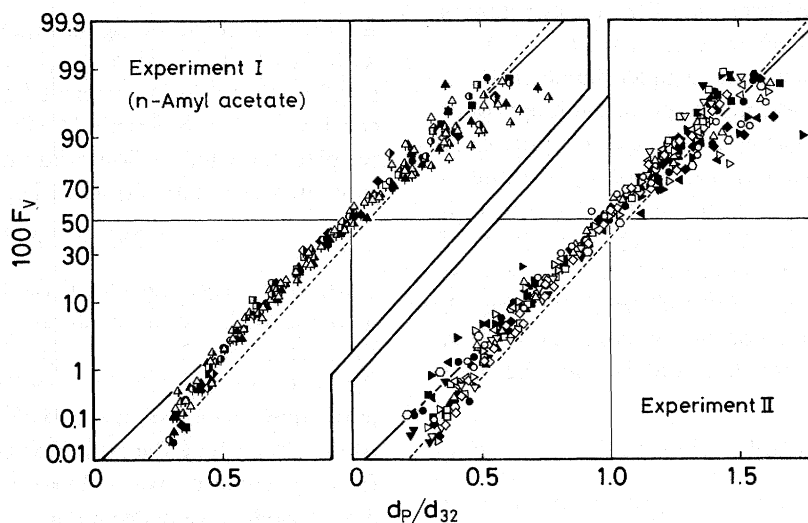


Fig. 5. Droplet size distribution. Keys are referred to Fig. 1.

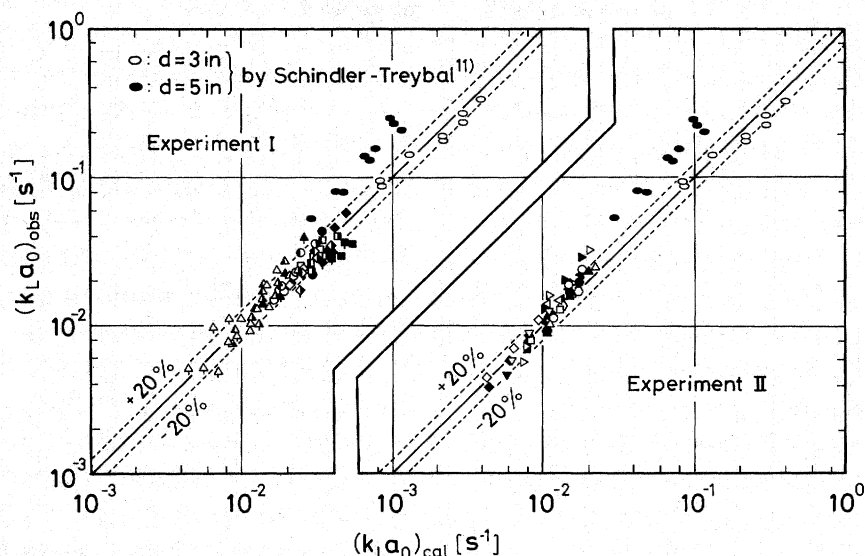


Fig. 6. Comparison of observed mass transfer volumetric coefficient with calculated one. Keys are referred to Fig. 1.

power input per unit volume swept out by impeller. The mass transfer volumetric coefficient estimated from the obtained correlation equations of both the mass transfer coefficient and droplet diameter agreed well with those observed.

Appendix: Derivation of characteristic droplet diameter \bar{d}_p

When the continuous-phase mass transfer coefficient at the droplet surface satisfies Eq. (23) in the text, the mass transfer coefficient for i -th droplet k_{Li} is related to the diameter of its droplet d_{pi} as follows

$$k_{Li} = C d_{pi}^{-0.23} \quad (\text{A-1})$$

The mass transfer volumetric coefficient $k_L a$ is defined as

$$k_L a = \frac{k_L A}{V} = \frac{\sum_i k_{Li} (\pi d_{pi}^2)}{\left(\sum_i \frac{\pi}{6} d_{pi}^3 \right) / \phi} \quad (\text{A-2})$$

Introduction of Eq. (A-1) into Eq. (A-2) gives rise to the following relation:

$$k_L a = C \cdot \frac{6\phi \sum_i d_{pi}^{1.77}}{\sum_i d_{pi}^3} = C \cdot 6\phi (\bar{d}_p)^{-1.23} \quad (\text{A-3})$$

Then the characteristic droplet diameter \bar{d}_p can be derived as

$$\bar{d}_p = \left(\frac{\sum_i d_{pi}^3}{\sum_i d_{pi}^{1.77}} \right)^{1/1.23} \quad (\text{A-4})$$

On the other hand, the Sauter mean droplet diameter d_{32} is defined as

$$d_{32} = \left(\frac{\sum_i d_{pi}^3}{\sum_i d_{pi}^2} \right) \quad (\text{A-5})$$

From the experimental data of droplet diameter distribution, the diameters of \bar{d}_p and d_{32} by Eqs. (A-4) and (A-5) are calculated and compared with each other. The characteristic droplet diameter agrees with the Sauter mean diameter within an error of 1%.

Nomenclature

a	= interface area per unit volume	[m ⁻¹]
b	= impeller width	[m]
C_A	= concentration of ester	[mol · l ⁻¹]
C_{Ai}	= concentration at interface	[mol · l ⁻¹]
\bar{C}_A	= apparent concentration of ester	[mol · l ⁻¹]
C_A°	= concentration of pure ester	[mol · l ⁻¹]
C_{Ai}°	= solubility of ester in water	[mol · l ⁻¹]
C_B	= concentration of alkali	[mol · l ⁻¹]
\bar{C}_B	= apparent concentration of alkali	[mol · l ⁻¹]
C_{AP}	= concentration of ester in droplet	[mol · l ⁻¹]
d	= impeller diameter	[m]
d_p	= droplet diameter	[m]
\bar{d}_p	= characteristic droplet diameter defined by Eq. (A-4)	[m]
d_{32}	= Sauter mean diameter	[m]
D	= vessel diameter	[m]
D_A	= diffusivity	[m ² · s ⁻¹]
F_V	= cumulative volume frequency of droplet	[—]
H	= liquid depth	[m]
K	= dimensionless parameter ($= k_L a_0 / k_2 \bar{C}_{B0}$)	[—]
k_L	= mass transfer coefficient	[m · s ⁻¹]
$k_L a$	= mass transfer volumetric coefficient	[s ⁻¹]
k_2	= reaction rate constant	[l · mol ⁻¹ · s ⁻¹]
m	= proportional constant in Eq. (11)	[(mol · l ⁻¹) ¹⁻ⁿ]
N	= stirred speed of impeller	[s ⁻¹]
n	= exponent in Eq. (11)	[—]
P	= power input	[W]
P_V	= power input per unit volume ($= P / (\pi D^2 H / 4)$)	[W · m ⁻³]
P_{VI}	= power input per unit volume swept out by impeller ($= P / (\pi d^2 b / 4)$)	[W · m ⁻³]
Sc	= Schmidt number	[—]
t	= time	[s]
V_{Add}	= volume of pure ester added in a vessel	[m ³]
V_p	= volume of droplet	[m ³]
V_A, V_T	= volume of pure ester and toluene composing droplet	[m ³]
V_w	= volume of water added in a vessel	[m ³]
X_A	= volume fraction of pure ester in droplet	[—]
α	= dimensionless variable defined by Eq. (14)	[—]

θ_B	= molar ratio ($= \bar{C}_{B0}/\bar{C}_{A0}$)	[—]
ν	= kinematic viscosity	[m ² ·s ⁻¹]
ρ	= density	[kg·m ⁻³]
σ	= interfacial tension	[N·m ⁻¹]
ϕ	= volume fraction of droplet	[—]

<Superscript>

* = dimensionless

<Subscript>

A = ester

B = alkali

cal = calculated value

0 = initial value

obs = observed value

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