A STUDY ON THE FLOW CHARACTERISTICS IN A PULSED DOUGHNUT-DISC TYPE PLATE EXTRACTION COLUMN

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Abstract—The axial dispersion coefficients in the continuous phase and holdup of dispersed phase have been studied in a 4.2 cm inside diameter and 200 cm height pulsed doughnut-disc type plates extraction column.

The axial concentraion gradient in a continuous extraction column was expressed mathematically in terms of Peclet number by axial dispersion model. Peclet numbers have been calculated from response curves using KCl solution as an impulse input tracer.

Experimental data have been taken for both continuous and dispersed phase with plate spacing, pulsing amplitudes, frequencies, and superficial velocities as system variables. Modified axial dispersion coefficients have been correlated by regression analysis of experimental data, and following equations were obtained.

1. Axial dispersion coefficient (single phase)
   $E_C = 3.5H^{-1.3}\ A^{1.54}\ f + 30.95\ U_C$
2. Axial dispsion coefficient (two phase)
   $E_C = 2.36\ H^{-0.8}\ A^{1.34}\ f + 20.89\ U_C$
3. Fractional holdup of the dispersed phase
   $\Phi_d = 4.2\times10^{-5}\ H^{-0.44}\ A\ f^{1.28}\ U_d^{0.93}$

INTRODUCTION

Continuous countercurrent liquid extraction is often carried out in a column in which countercurrent motion is effected only by the buoyancy force due to the difference in density of the two phases.

However, this force is relatively low and consequently the energy dissipation rate due to flow is not very high and the dispersed phase tends to consist of large droplets with a low specific interfacial area. This led to the adoption of externally agitating components which supply additional energy for creating a large specific interfacial area and generating turbulence.

The equipments used for externally agitated column have been investigated for the past 20 years; agitated impeller column [1,2], rotary disc column [3,4] reciprocating plate column [5,6,7], pulsed packed column [8], pulsed plate column [9,10], and Oldshue-Rushton column [11, 13]. All these means of agitation greatly enhanced the interfacial contact area by breaking up the dispersed droplets.

However, the axial mixing, as agitation is increased, tends to reduce the effectiveness of any countercurrent mass transfer process, by flattening the axial concentration gradients in each phase and thus reducing the overall driving force available.

This effect has led to a growing volume of countercurrent columns, allowing for backmixing, and many experimental measurements of backmixing in column have been carried out.

An important factor in understanding the fundamental mass transfer mechanisms in extraction column, hence is the effect of axial dispersion or residence time distribution of the continuous phase. The best reasonable values of Peclet number and holding time are obtained experimentally by injecting an impulse of tracer and measuring the change in concentration profile as it passes through the column. The axial dispersion coefficients in the continuous phase are determined from these values.

Much work is concerned with studying axial mixing effect in reciprocating plate columns or in pulsed extraction columns, special consideration being given to the effect of the plate geometry on the phenomena.

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However, the overall mass transfer rate holdup and flow characteristics of doughnut-disc type plate packed, pulsed column have not been studied.

The primary objective of this study is to determine experimentally the axial dispersion coefficients of the continuous phase in the type of column used by an impulse tracer technique and to correlate experimental data by regression analysis in terms of the parameters.

THEORY

Dispersion models have been widely used to represent the flow and mixing characteristics of complex chemical systems. These models have been used mainly to describe flow in empty and packed beds, systems that are closer to plug than ideally mixed flow.

If the density is constant and the system is in turbulent flow, the most general form of a dispersion model can be described by the equation of conservation of mass:

$$\frac{\partial c_i}{\partial t} + U\nabla c_i = \nabla (D\nabla c_i) + S_i + r_i$$

where S_i is the rate of input of species i from sources and r_i represents the rate of generation of species i by chemical reaction.

In tracer experiments without chemical reactions, $r_i = O$, and the source term is given by

$$S = \frac{I}{\pi}\delta(x-x_0)\ f(R)$$

where

I = injection rate of tracer

$\delta(x-x_0)$ = Dirac-delta function

$$f(R) = 1/R_i^2,\ R \leq R_i$$
$$= 0, \quad R_i \leq R \leq R_0$$

R_i = injector tube radius.

Assuming an one-dimensional flow with a constant axial dispersion coefficient E_c this equation, in dimensionless form, can be put into;

$$\frac{\partial C}{\partial \theta} + \frac{\partial C}{\partial z} = \frac{1}{p}\frac{\partial^2 C}{\partial z^2} + \delta(z-z_0)\,\delta(\theta)$$

where

$\theta = Ut/L = Ft/V$

$z = x/L$

$P = UL/E_c$ (Peclet number)

$C = c/c_0$

For an open vessel and a perfect delta function input, the concentration evaluated at $z=1$ is given by

$$C = \frac{1}{2}\left(\frac{P}{\pi\theta}\right)^{1/2} \exp\left(-\frac{P(1-\theta)^2}{4\theta}\right)$$

and the first and second moments are;

$$\mu_1 = 1 + \frac{2}{p}$$

$$\sigma^2 = \frac{2}{P} + \frac{8}{P^2}$$

From the response curves, the following deterministic moments can be obtained;

$$\mu_1 = \frac{1}{\alpha}\int_0^\infty tc\ dt$$

$$\sigma^2 = \frac{1}{\alpha}\int_0^\infty (t-\mu_1)^2 C\ dt$$

where

$$\alpha = \int_0^\infty C\, dt$$

Because of long tail of the response curves, accurate measurements were difficult. Hence the response curve has been divided into two sections. The tail section could be described by an exponential decay function [14]

$$C = C_A \exp(-K(t-t_a))$$

where (t_a, C_A) is a point on the curve, and K is a decay factor and the initial section could be analysed numerically. The parameter K was evaluated from the tail section of the response curves and found approximately in the range of (1/30) C_{max} and (1/6)C_{max}, leading to the deterministic moments to be estimated;

$$\alpha = \sum_{i=0}^{n} C_i \Delta t + \frac{C_T}{K}$$

$$\mu_1 = \frac{1}{\alpha}\left(\sum_{i=0}^{n} C_i\ t_i \Delta t + \frac{C_T}{K}\left(t_c + \frac{1}{K}\right)\right)$$

$$\sigma^2 = \frac{1}{\alpha}\left(\sum_{i=0}^{n} Ci\ t_i^2 \Delta t + \frac{C_T}{K}\left(t_c^2 + \frac{2t_c}{K} + \frac{2}{K^2}\right)\right) - \mu_1^2$$

In multiple linear regression for a system of K independent varibles, the regression equation can be written as

$$y_i = \beta_0 + \beta_1 X_{1j} + \beta_2 X_{2j} + \cdots + \beta_K X_{Kj} + \varepsilon_j$$
$$= \beta_0 + \sum_{i=1}^{n} \beta_i X_{ij} + \varepsilon_i$$
$$J = 1, 2, \ldots, n$$

or $Y = X\beta + E$

The estimation procedure requires that the random error component have $E(\varepsilon_j) = 0$ and $Var(E) = \sigma^2$.

The least square estimator and the regression coefficient vector can be found by minimizing the sum of squares of the errors. Thus the least square estimator β is

$$\beta = (X^T X)^{-1} X^T Y$$

The independent variables in this study are Peclet number and the holding time. These variables obtained by the parameter estimation using deterministic moments were taken as the initial values and in the two-level factorial design, the two levels of interest were set at ± 5%.

Using the independent variables coded to an (-1, 1) interval and the two-level fatorial design, the steepest descent path was obtained and by employing four experimental data the components of the least square estimator were found;

$$\beta_0 = (y_1 + y_2 + y_3 = y_4) / 4$$

$$\beta_1 = (y_1 - y_2 + y_3 - y_4) / 4$$

$$\beta_2 = (y_1 + y_2 - y_3 - y_4) / 4$$

EXPERIMENTAL

1. Experimental set-up

The main extraction column is made of 4.2cm inside diameter pyrex glass column with an overall height of 2.12m. The top and bottom sections of the main column are made of 10cm inside diameter lucite column is provided with injection ports (or sampling ports) at 16cm intervals on one side and two injection ports, 200cm apart on the other side. Concentrations of the tracer were measured at sampling port by a probe cell and a conductivity bridge network. The conductivity probe is made of 0.5mm diameter and 7mm length platinum wire.

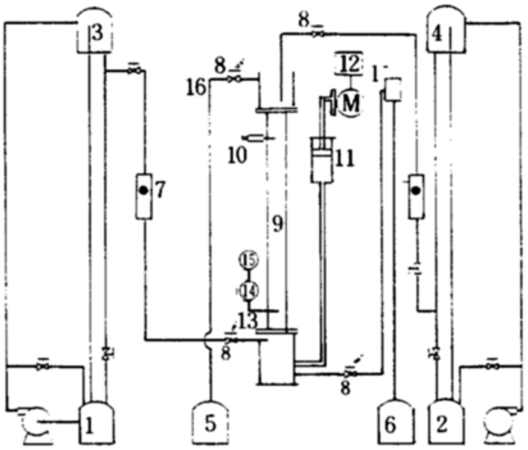
The doughnut plates and disc plates are made of 1mm thick stainless steel plates. their diameter and free area are listed in table 1. The doughnut type plates are supported in the main column by means of two vertical 4mm width and 1.5mm thickness stainless steel baffles. The disc plates are supported by means of a vertical 4mm diameter stainless steel rod. Stainless steel sleeves around the rod serve as spacers for the plates. The entire rod plate assembly can be installed and removed by lifting it from the open top of the column. By use of sleeves and baffle holes between plates, the center-to-center spacing H can be set.

The cylinder is made of 5.5cm inside diameter stainless steel tube and the piston ring is made of 5.5cm diameter with 2cm length teflon rod. Pulser is driven by a cam attached to amplitude adjuster driven by a 1/4 hp. 110 volt D.C. motor. The amplitudes were adjusted by an amplitude adjuster and the sinusoidal pulsation frequencies were adjusted by speed ranger transmission.

The overall schematic diagram is shown in Fig. 1. The heavy and light phases are fed to the column close to the stator ring (doughnut type plate) from the head tanks equipped with the overflow outlet to maintain the liquid level constant. The flow rate of both phases are measured by rotameters. The continuous phase outlet is equipped with an adjustable limp to maintain constant interface level.

Table 1. Description of System.

Column		System
effective height	(cm)	200
inside diameter	(cm)	4.2
opening diameter of stator	(cm)	2.13
disk diameter	(cm)	2.13
free area	(%)	25
continuous phase		distilled water
dispersed phase		refined kerosene
tracer		KCl (0.5 N)



1 . Organic Storage
2. Aqueous Storage
3 . Organic Head Tank
4. Aqueous Head Tank
5 . Organic Reservoir
6. Aqueous Reservoir
7 . Rotameter
Solenoid Valve
9 . Main Column
10. Sampling Needle
11. Pulsing Unit
12. R. P. M Control System
13. Condudtivity Cell
14. Wheatstone Bridge
15. Recorder
16. Dispersed Phased Outlet
17. Continuous Phase Outlet

Fig. 1. Schematic Flow Diagram.

Likewise, the dispersed phase outlet is equipped with an overflow. The pulsing unit is connected to bottom of the main column. The solenoid valves are installed at the inlet and outlet of the column in order to stop the flows simultaneously.

2. Operation

The experimental column is initially filled with the heavy phase (distilled water) set at desired flow rate. At the same time pulsing unit is started and maintained at a proper frequency and amplitude. The light phase (refined kerosene) set at the desired flow rate is then gradually introduced.

After steady state has been reached an half second impulse of 0.5N potassium chloride was carefully injected into the column using syringe at an injection port, and down stream concentration was detected by a probe cell and a conductivity bridge network as a response curve.

In this study, holdup is defined as the average percent of the total volume between all doughnut type plates occupied by the dispersed organic phase at steady state operation. Holdup measurements were made when two phases were completely separated after the solenoid valves were shut off in order to stop the flows.

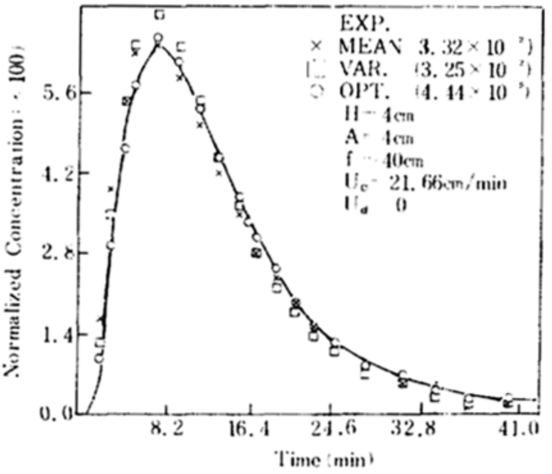


Fig. 2. Comparison between Experimental Data and Theoretical Predictions(Single Phase).

3. Data Processing

The obtained conductivity gain from the response curve on the recorder chart was corrected into the exact concentration value using the relationship between concentration versus conductivity gain which was determined in the preliminary experiment. Typical moment analyses and regression analyses do not necessarily require real concentration value but arbitrary units [15]. Thus, all the data processing was carried out with the residence time distribution curve form direct conductivity gain.

The typical results of the analyses are compared with experimental data for single-phase and two-phase operations in Figures 2 and 3, respectively. The values of the sample variance for regression analyses were lower than those for moment analyses, hence, good agreement with response curves.

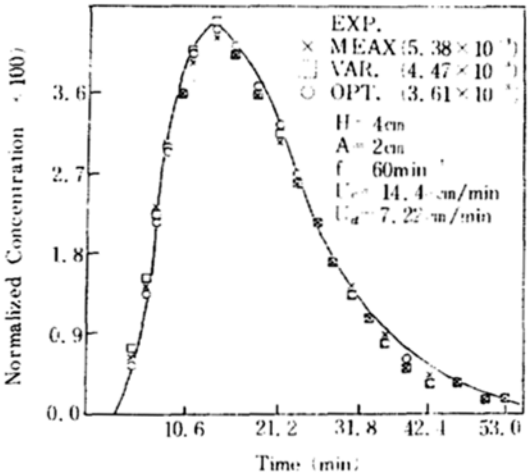


Fig. 3. Comparison between Experimental Data and Theoretical Predictions(Two Phase).

RESULTS AND DISCUSSIONS

An axial dispersion model has been adopted and found successful in depicting the flow characteristics in a doughnut-disc plates packed, pulsed column.

While the dispersion model has been commonly employed for pipe flow, packed beds and so forth where discrete stages were not readily identified, the attempt to apply this model to present system has been made due to a considerable interchange of liquid between the free area of interplate regions.

1. Single Phase Operation

1-1. The effect of continuous phase superficial velocity

The effects of U_C on the axial dispersion coefficient are shown in Fig. 4. The axial dispersion coefficients increased linearly with continuous phase superficial velocity.

In the previous investigations [11, 17, 18] a linear effect of the superficial velocity on the axial dispersion coefficient has also been reported. These relations were obtained from measurements in pulsed column of different types by employing impulse response technique.

1-2. Effects of amplitude and frequency

Effects of amplitude and frequency on the axial

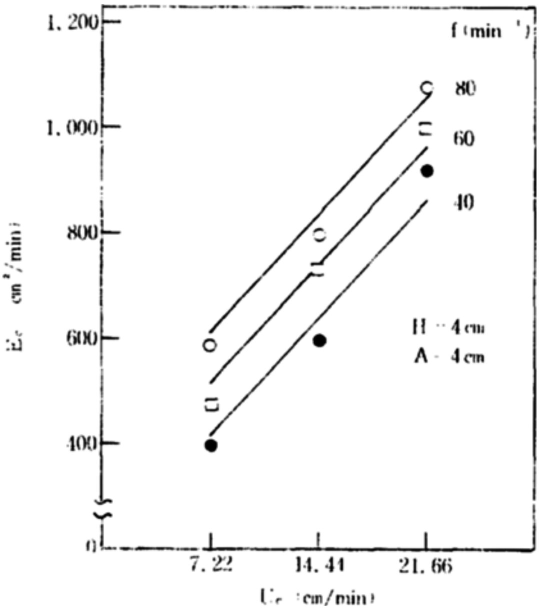


Fig. 4. Effect of U_c on Axial Dispersion Coefficient (Single Phase).

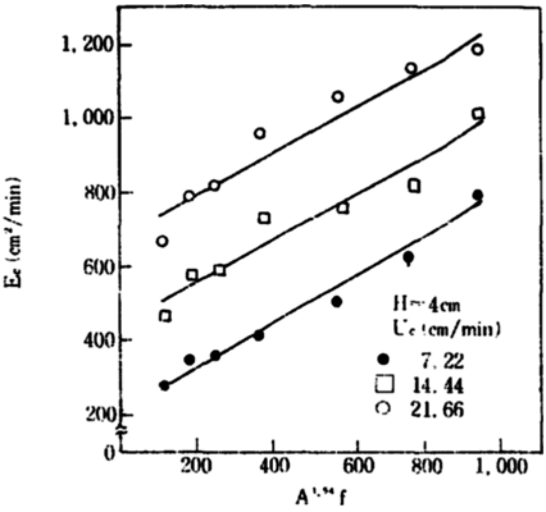


Fig. 5. Effect of Pulsing Factor on Axial Dispersion Coefficient(Single Phase).

dispersion coefficient are shown in Fig. 5. The dispersion coefficient increased linearly with the frequency, and this trend is in accordance with most previous data for pulsed columns of different types [9, 16]. However, the effect of amplitude is significantly nonlinear. The dispersion coefficient increased approximately to the 1.54 power of amplitude, whereas the majority of pulsed column data have shown the dependence. This trend agrees however with the data of Kim[5] using a reciprocating plate with the relatively large free area between the plates. Some of the pulsed column dada [17] have shown a slightly greater effect of amplitude than of frequency but the effect has not been very pronounced.

Most of previous data on pulsed columns using sieve-plates have been collected with plates having free area 0.2 an relatively small holes.

1-3. The effect of plate spacing

The effect of the plate spacing on the axial dispersion coeficient are shown in Fig. 6. The axial dispersion coefficient decreased with the plate spacing and this trend is in accordance with previous data for pulsed column of different types [9] and reciprocating plate column [5].

The increase in the axial dispersion coefficient with the increase of plate spacing has been generally observed in the mixed model.

However, in this column the turbulence level tends to decline with an increase in plate spacing leading to the results.

1-4. Sigle phase correlations

About 50 data points were obtained for single phase conditions for the dispersion coefficient as a function of amplitude, frequency, continuous phase superficial velocity and plate spacing. The data were correlated by regression analysis as follows:

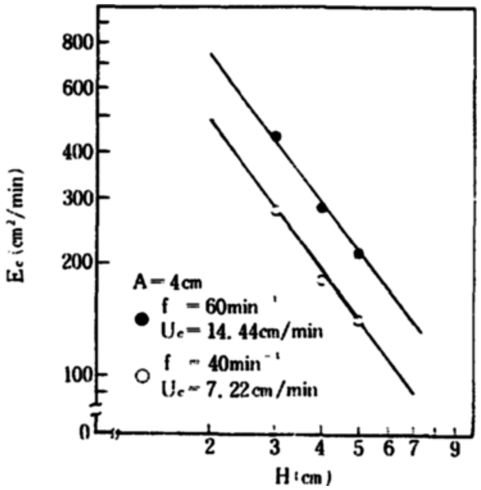


Fig. 6. Effect of Plate Spacing on Axial Dispersion Coefficient(Single Phase).

$$E_c = 3.5\ H^{-1.3}\ A^{1.54}\ f + 30.95\ U_c$$

The results are shown in Fig. 7.

2. Two Phase Operation

The bulk of these data have been obtained in the emulsion flow regime in which the dispersion phase droplets move freely within the continuous phase. There were only some indication of clustering and coalescence near the doughnut plates and the disc plates during pulsation.

Quite similar relations to effects in single phase operation variables has been obtained. The effect of dispersed phase superficial velocity on the axial disper-

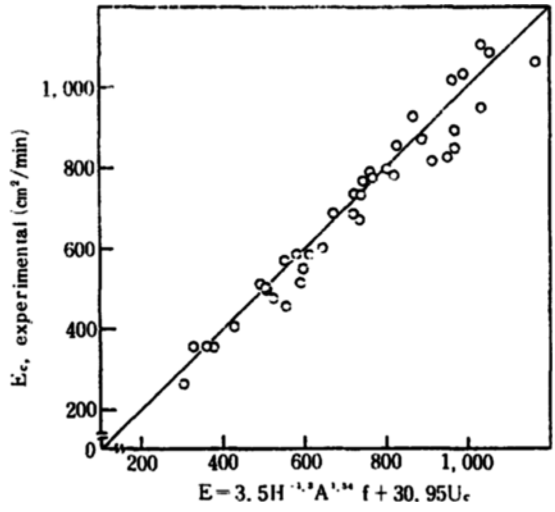


Fig. 7. Correlation for Axial Dispersion Coefficient(Continuous Phase only).

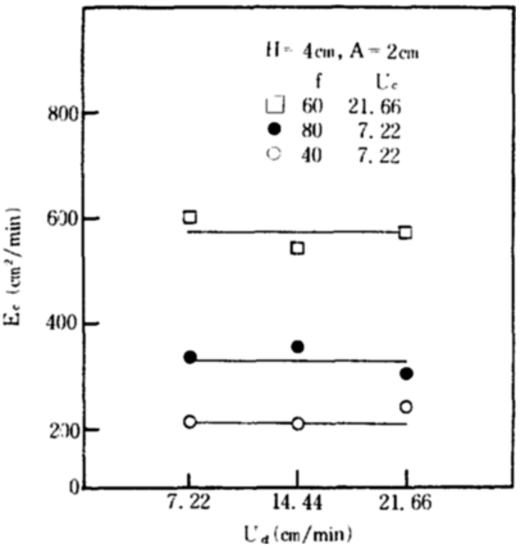


Fig. 8. Effect of U_d on Axial Dispersion Coefficient (Two Phase).

sion coefficient is relatively small as shown in Fig. 4 through 8.

All experimental data for two-phase have been correlated by regression as follows:

$$E_c = 2.36\ H^{-0.8}\ A^{1.34} f + 20.89\ U_c$$

The results are shown in Fig. 9.

3. Holdup

The size of kerosene droplets decreased with pulsing factors (frequency and amplitude) in two phase operation. Thus the difference of density of two phase decreased and volume fractional holdup of the dispersed phase in stage increased.

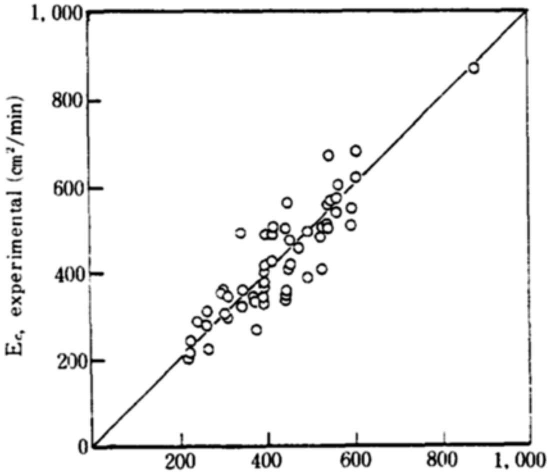


Fig. 9. Correlation of Axial Dispersion Coefficient for Continuous Phase(Two Phase).

All experimental data can be correlated by regression as follows:

$$\Phi_d = 4.2\text{x}10^{-5}\ H^{-0.44}\ A\ f^{1.28}\ U_d^{0.93}$$

From this equation it is apparent that the dispersed phase holdup is strongly influenced by the amplitude and the frequency. The results are shown in Fig. 10.

V. CONCLUSIONS

1. An axial dispersion model has been found satisfactorily applicable in depicting the flow characteristics of the continuous phase in a pulsed, doughnut-disc type plate extraction column.
2. Peclet number and the holding time have been successfully estimated from this model by employing the response surface method and the axial dispersion coefficient has been subsequently calculated therefrom.
3. The axial dispersion coefficient has been successfully correlated with the pulsing factors (frequency and amplitude), the plate spacing and the superficial velocity of the continuous phase. The dispersed phase holdup has also been correlated with the pulsing factors and the superficial velocity of the dispersed phase.

The regression analyses have been applied and the following results were obtained.

- Axial dispersion coefficient (single phase)

  $E_c = 3.5\ H^{-1.3}\ A^{1.54} f + 30.95\ U_c$

- Axial dispersion coefficient (two phases)

  $E_c = 2.36\ H^{-0.8}\ A^{1.34} f + 20.89\ U_c$

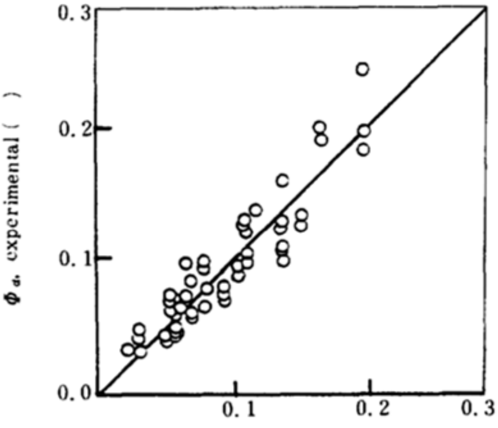


Fig. 10. Correlation of Dispersed Phase Holdup Ratio (org./total).

- Fractional holdup of the dispersed phase

$$\Phi_d = 4.2\text{x}10^{-5}\ H^{-0.44}\ A\ f^{1.28}\ U_d^{0.93}$$

NOMENCLATURE

A : pulsing amplitude (cm)
c : tracer concentraction (g-mole/cm^3)
C : dimensionless tracer concentration $\left(\frac{C}{Co}\right)$ (--)
Co : initial tracer concentration (g-mole/cm^3)
C_A : tracer concentration when $C = \frac{1}{6}\ C_{max,}$ at $t = t_A$ (--)
C_T : tracer concentration when $C = \frac{1}{30}\ C_{max,}$ at $t = t_c$ (--)
D : eddy diffusivity tensor
E_c : axial dispersion coefficient for continuous phase (cm^2/min)
f : pulsing frequency (min^{-1})
F : volumetric flow rate (cm^3/min)
H : Plate spacing (cm)
I : injection rate of tracer (g-mole/cm^3 sec)
L : effective length of the column (cm)
K : exponential factor defined in equation
P : peclet number for continuous phase (--)
R : radial position (cm)
R_i : radial position (cm)
R_0 : tube radius (cm)
t : time (min)
U : superficial velocity (cm/min)
V : volumetric flow rate of continuous phase (cm^3/min)
Var : variance
X : matrix of independent variable (x_{ij})
y_i : response
Y : response (y_i) vector
z : dimensionless axial length (x/L)

Greek Letters

α : area under response curve
β : regression coefficient
$'\beta$: regression coefficient vector
ε : error
E : error vector
Φ_d : fractional hold-up
μ_1 : mean (1st moment)
δ : delta function
σ^2 : variance (2nd central moment)
Δ : gradient

Subscripts

c : continuous phase
d : dispersed phase

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