LIQUID-LIQUID MASS TRANSFER COEFFICIENTS IN A KARR EXTRACTION COLUMN

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ABSTRACT. A Karr extraction type of column was constructed using a cylinder (15.3 cm internal diameter and 100 cm long) with a stack of 17 plates. Its operation was successfully carried out in a counter current liquid-liquid flow employing kerosene oil dispersed phase and a continuous water phase with benzoic acid and n-butyric acid used as solutes. The effect of phase velocities, agitation rate of the stack, plate perforation diameter and free flow area were investigated. Based on the experimental results, correlations for volumetric mass transfer coefficient have been proposed in the operability of the column.

KEYWORDS: Karr column, phase interface, combined mass transfer coefficient, amplitude

INTRODUCTION

Multiphase contacting is commonly encountered in chemical engineering practice to affect mass transfer between the phases. Karr extraction column is an ideal multiphase contactor which offers vivid advantages over other in respect of power consumption, more interfacial areas, large through puts, simple design and scale up method etc (Prabhakar, 1987). The reciprocating plate column, as introduced by Karr has become one of the most popular and versatile extractive columns used in pharmaceutical, petrochemical and biochemical industries (Lo and Prochazka, 1983). Its performance as biodigester is very well recognized in air and water pollution control (Brauer, 1985). Enough work on hydrodynamics has been published in this column (Prabhakar, 1988; Baird and Rama rao, 1988). Mass transfer, heat transfer and reactions are being explored for its performance as reactor, separator, and heat exchanger (Aravamudan and Baird, 1999; Rama Rao and Baird, 2000; Bensalem, 1985).

This paper mainly concerns mass transfer aspects between the phases in the column and presents the correlations for estimating volumetric mass transfer coefficients in terms of design variables of the system.
Experimental

The experimental set-up is shown schematically in Figure 1 consisting of a glass column of 15.3 cm inside diameter and 100 cm long, of which 90 cm formed the test section (1). The perforated plates are mounted on a central rod and connected to a slider crank mechanism, which allowed imparting motion at the desired frequency and amplitude for the plate stack. The agitation rate \( Z \) is defined as the product of amplitude and and frequency of the plate stack. Water is fed as a continuous phase and kerosene as a dispersed phase at the bottom of the column respectively to achieve a counter current flow. The physical properties of the solvents and the range of variables investigated are presented in Table 1. Benzoic acid and n-butyric acid were used as solutes one at a time and the transfer was from dispersed phase to continuous phase and vice-versa. Kerosene and water at known initial solute concentrations were fed at the desired flow rates in to the column, keeping the plate stack under pulsing motion until a steady state was reached. The initial feed concentrations of benzoic acid in kerosene was maintained at about 0.1 g.mol l\(^{-1}\) and of n-butyric acid at about 0.125 g.mol.l\(^{-1}\) during experiments. The concentrations in the streams were estimated by volumetric method. All the experiments were conducted at a room temperature of 28 ± 1 °C.

<table>
<thead>
<tr>
<th></th>
<th>Density g cm(^{-3})</th>
<th>Viscosity, cp</th>
<th>Surface tension, dyn . cm(^{-1})</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>0.996</td>
<td>1.01</td>
<td>77.5</td>
</tr>
<tr>
<td>Kerosene</td>
<td>0.8</td>
<td>1.07</td>
<td>28.9</td>
</tr>
</tbody>
</table>

Continuous phase velocity (cm s\(^{-1}\)) = 0.02 - 0.91
Dispersed phase velocity (cm s\(^{-1}\)) = 0.03 - 0.85
Amplitude of pulsing (cm) = 1.4 - 5.0
Frequency of pulsing (Hz) = 0.66 - 7.5
Agitation rate of plate stack (cm s\(^{-1}\)) = 0.0 - 7.5
Plate perforation diameter (cm) = 0.5 - 1.2
Plate free flow area = 0.125 - 0.517
Number of plates = 18
Plate spacing (cm) = 5.0

RESULTS AND DISCUSSION

The study delineates the operation of the karr column into two distinct types of phase dispersion corresponding to mixer-settler and emulsion regions (Figure 2). The mixersettler region is characterized by heavy and light phases forming discrete clear layers in the stage.
Figure 1: Schematic diagram of experimental set-up (1, column; 2, plate stack; 3, central rod; 4, slider-crank assembly; 5, control valves; 6, water inlet; 7, water outlet; 8, kerosene inlet; 9, kerosene outlet; 10, liquid storage tank; 11, rotometers)

Figure 2: Variation in the dispersed phase holdup ($e$) with $z$

Figure 3: Variation in combined mass transfer coefficient with agitation rate
The heavy phase descends through the light phase during the upward movement of the plate stack, where as the light phase rises through the heavy phase as globules during its downward movement. With increase in the agitation rate the mixer-settler region leads to the emulsion region. The latter is characterized by uniform distribution of the dispersed phase droplets in the continuous phase with no discernible change in the phase distribution with upward or downward stroke of the plate stack.

Further it is observed that the emulsion region is of practical importance and stable for the operation of the column to affect good mass transfer between the phases of contact due to uniform mixing and drop size distribution of dispersed phase through out the column.

The volumetric mass transfer coefficient $K_{oa}$ (Mass transfer coefficient is the reciprocal of Resistance in the direction of mass transfer) was estimated from the inlet and outlet concentrations of solute in the dispersed and continuous phases and mass balance and equilibrium relationships for each experiment. From the knowledge of the solute concentrations of the feed streams and the effluents, the combined mass transfer coefficient $K_{oa}$ is obtained as $K_{oa} = (N/\theta)_{av}/V \log \text{mean } \Delta C$. Figure 3, Figure 4 and Figure 5 show the variation in $K_{oa}$ with system variables. $K_{oa}$ increases with increase in agitation rate, the rise is slow in mixer-settler region ($Z < 2 \text{ cm s}^{-1}$) and rapid in the emulsion region ($Z > 2 \text{ cm s}^{-1}$). At very high agitation rates $K_{oa}$ is found to decrease before on set of flooding (Figure 3). The combined mass transfer coefficient increases with an increase in agitation rate and disperse phase velocity mainly due to increase in interfacial area. This trend is observed until just before the onset of flooding (Figure 3 and Figure 4a).

The $K_{oa}$ is little influenced by the flow velocity of continuous phase (Figure 4b) compared to dispersed phase velocity (Figure 4a). The volumetric coefficient $K_{oa}$ varies little with perforation diameter (Figure 5a) but falls rapidly with increase in plate free flow area (Figure 5b). The increase in mass transfer coefficient with $U_d$ is due to less resistance to mass transfer in dispersed phase than in the continuous phase.

The experimental data is correlated in emulsion region for estimating $K_{oa}$ in terms of design variables as follows:

**Benzoic acid as solute,**

\[
K_{oa} \text{ (sec}^{-1}) = 0.8 \times 10^{-3} \ U_e^{0.25} \ U_d^{0.85} \ Z^{1.5} \ S^{-1.0}
\]

**n-butyric acid as solute,**

\[
K_{oa} \text{ (sec}^{-1}) = 0.23 \times 10^{-3} \ U_d^{0.6} \ Z^{1.4} \ S^{-0.92} \ d^{-0.135}
\]
Figure 4: Variation in the combined mass transfer coefficient with the phase velocities ($U_c$ and $U_d$)

Figure 5: Variation in the combined mass transfer coefficient with the plate geometry: perforation diameter and fractional free area
Figure 6 depicts the experimental versus predicted volumetric mass transfer coefficients in emulsion region of operation of the column for different solutes. The values are in close agreement with each other within tolerable limits of entire data. Figure 6a represents the entire data for n-butyric acid as solute and Figure 6b for benzoic acid.

Figure 6: Correlation for the combined mass transfer coefficient, $K_{oa}$
CONCLUSION

The mass transfer coefficient in Karr column is influenced by agitation rate of the plate stack, the free flow area, the phase velocities and the perforation diameter of the plate. The volumetric coefficients are found to be 15 to 20 times higher with agitation than without agitation of the plate stack. The high mass transfer rates are found close to flooding thus ensuring maximum through put through the column before onset of flooding. The volumetric efficiency and the phase interface are found very high reflecting its potential as mass transfer equipment. Its simple design, cost of fabrication, scale-up and economical operation prompt for commercialization of the unit.

NOMENCLATURE

a  interfacial area (cm$^{-1}$)
d  plate perforation diameter (cm)
g  acceleration due to gravity (cm s$^{-2}$)
H  height of a test section (cm)
K$a  volumetric mass transfer coefficient (s$^{-1}$)
N  number of stages
P  plate spacing (cm)
S  fractional free area of plate
U$c  continuous phase superficial velocity (cm s$^{-1}$)
U$d  dispersed phase superficial velocity (cm s$^{-1}$)
V  volume of the test section (cm$^3$)
(DC)$_{in}$  log mean concentration gradient
Z  agitation rate (cm s$^{-1}$)
$\varepsilon$  dispersed phase holdup
m  volumetric efficiency (s$^{-1}$)
$\theta$  clock time

REFERENCES


