

## CO-CURRENT MASS TRANSFER STUDIES IN A PACKED COLUMN

K. O. Ipinmoroti

*Federal University of Technology, Akure, Ondo State, Nigeria*

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The extraction of succinic acid with *n*-butanol was studied in a packed column run counter-currently. A rapid approach to equilibrium was noticed. The stage efficiencies ranged from 76 – 97% for contact times of between 18 and 32 sec. Overall mass transfer coefficients based on both phases were similar and positively influenced by increase in total throughput.

Values of overall heights of transfer units were also similar for both phases (0.21 – 0.61m), but lower than the corresponding literature values for counter-current works.

*Keywords* : Mass transfer, Succinic acid, Packed column.

## INTRODUCTION

In a solvent extraction process, the feed needs to be contacted intimately with the extractant and the resultant mixture separated into two layers. The rate of mass transfer between the two phases depends, among other things, on the interfacial area. In order to increase this, it is usual to disperse one phase in the other. Many commercial solvent extractors are currently available in the market including a variety of differential column contactors which, apart from other advantages, require less space than, say, the traditional mixer-settlers.

For a typical spray column extractor, it has been found that sometimes as much as 90% of the column height may be needed to compensate for axial mixing alone [1]. This can be drastically reduced by introducing some suitable packing materials into the column. The packing reduces back-mixing of the continuous phase, promotes mass transfer by breaking up large drops, and enhances internal mixing by setting up a coalescence-redispersion cycle of the drops.

The convectional method of operating a column extractor is to make the two liquid phases run counter-currently. Despite the acceptable performance of this mode of operation, it is desirable to find ways of overcoming the flooding problem which critically limits the throughput. One idea is to run the column co-currently. While it is obvious that this technique might be limited by pressure drop, thus necessitating the use of more powerful pumps; from the hydrodynamic point of view, it could be advantageous as flooding would be eliminated.

The rate of transfer of succinic acid from an aqueous medium into a dispersed *n*-butanol phase has thus been investigated in a packed column operated co-currently in order to ascertain whether this mode of operation offers any significant advantages.

*Theoretical aspects of mass transfer in a packed column.* Taylor [2] had earlier derived an equation for calculating the mass transfer coefficient of the dispersed phase for co-current flow in a packed column, i.e.

$$\frac{K_d a t (1+r)}{h_d} = \log_e \left( \frac{Y_1 - mX_1}{Y_2 - mX_2} \right) \quad (1)$$

(Fig.1).

If the fractional approach to equilibrium  $E_f$  is given by

$$E_f = AB/AC$$

then it can be shown that (2)

$$1 - E_f = \frac{Y_2 - mX_2}{Y_1 - mX_1} \quad (3)$$

Thus,

$$K_d a = \frac{h_d \log_e \left( \frac{1}{1 - E_f} \right)}{t (1+r)} \quad (4)$$

Where

$t$  = Contact time.,  $r$  = Extraction factor.,  $h_d$  = Hold up of the dispersed phase.,  $m$  = Slope of the equilibrium line.,  $K_d$  = Mass transfer coefficients based on the dispersed phase.,  $a$  = Interfacial area.

An identical equation may be written for the mass transfer coefficient based on the continuous phase,  $K_c$ . The sizes of the dispersed droplets were not measured and  $K_d a$  and  $K_c a$  were evaluated instead of  $K_d$  and  $K_c$  respectively. For overall heights of transfer units, equations.

$$(HTU)_{od} = Fd/K_d a A \quad (5)$$

$$\text{and } (HTU)_{oc} = Fc/K_c a A \quad (6)$$

were used where  $F_d$  and  $F_c$  stand for flow rates and  $A$  is the cross-sectional area of the column.

### EXPERIMENTAL

All chemicals were obtained from the British Drug House Ltd. (BDH) in sufficiently pure grades so that further purification was considered unnecessary. The concentrations of succinic acid in the aqueous and organic phases were determined by titration with standard sodium hydroxide using phenolphthalein indicator, and a potentiometric end point respectively. The equilibrium line for the system had been reported somewhere else [3] and was checked and found satisfactory.

Mass transfer experiments were carried out in a glass column (0.076M i d and 1.70M high) with four sampling points at distances of 0.30M, 0.50M, 0.65M and 0.80M from the bottom respectively. The column was randomly packed with ceramic Rasching rings (0.025M long) to a height of 1.0M. Since *n*-butanol and water are mutually and partially soluble in each other, each phase was initially saturated with the other. Runs were carried out at the laboratory temperature of  $25 \pm 2^\circ$  with *n*-butanol dispersed in an upward flow of a continuous aqueous succinic acid. Samples were withdrawn from the column 2 min. after

start-off as trial runs had indicated that this time was sufficient to attain a steady state.

An operating diagram similar to that shown in Fig. 1 was then plotted for each run and  $E_f$  value evaluated from it.

### RESULTS AND DISCUSSION

Tables 1 and 2 summarise the data for stage efficiencies, mass transfer coefficients and heights of transfer units for a packed height of 0.65M, the height at which end effects became minimal. Others for different heights are not tabulated to save space.

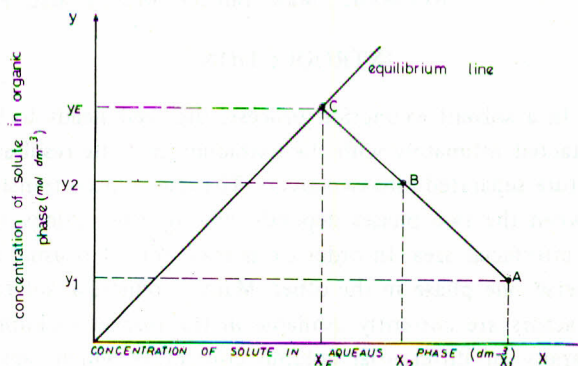


Fig. 1. Operating diagram for a column extractor

Table 1.  $E_f$  and mass transfer co-efficients for packed height of 0.65M

$F_c$										
(1/min)	1.0	1.0	1.0	2.0	2.0	2.0	3.0	3.0	4.0	4.0
$F_d$										
(1/min)	0.5	1.0	2.0	1.0	2.0	4.0	1.5	3.0	2.0	4.0
$E_f$ (-)	0.94	0.89	0.95	0.91	0.92	0.97	0.95	0.97	0.97	0.97
$K_c a \times 10^2$ ( $S^{-1}$ )	0.71	0.83	1.47	1.22	1.90	3.44	2.28	3.95	3.56	5.26
$K_d a \times 10^2$ ( $S^{-1}$ )	0.67	0.78	1.41	1.14	1.79	3.31	2.13	3.71	3.32	4.98

Table 2. Heights of transfer units

$F_c$										
(1/min)	1.0	1.0	1.0	2.0	2.0	2.0	3.0	3.0	4.0	4.0
$F_d$										
(1/min)	0.5	1.0	2.0	1.0	2.0	4.0	1.5	3.0	2.0	4.0
(HTU) <sub>oc</sub> (mm)	51.5	44.0	24.9	59.9	38.5	21.3	48.1	27.8	41.1	27.8
(HTU) <sub>od</sub> (mm)	27.3	46.9	51.8	32.1	40.8	44.2	25.7	29.4	22.0	29.4

$E_f$  values ranged between 76 and 97% for contact periods of between 18 and 32 sec. Godfrey and Slater [4] reported an approach to equilibrium of 89% for water-acetonetoluene system for a contact time of 40.8 sec. This suggests that mass transfer was faster in the water-succinic acid-*n*-butanol system. Since both of them involve physical mass transfer alone, physical properties such as molecular diffusivities of acetone and succinic acid in water, and interfacial tension could account for the difference.

In general,  $E_f$  values appear to increase with increase in flow ratio of the dispersed to the continuous phases in the column, that is, as hold-up and interfacial area increased. Roy *et al* [5] found the reverse in their work for kerosine-benzene-water system. This might be due to the fact that unlike this work, in their study solute transfer was from the dispersed phase into the continuous phase, thus encouraging coalescence.

Value of  $K_{ca}$  and  $K_{da}$  varied widely with flow rates and packing heights. At a packing height of 0.65M, they ranged within  $(0.71 - 5.30 \times 10^{-2} \text{ S}^{-1})$  and  $(0.67 - 5.00) \times 10^{-2} \text{ S}^{-1}$  respectively. These values are higher than the counter-current results reported for kerosine-benzoic acid-water system by Allerton *et al*. [3] and for toluene-benzoic acid-water by Roy *et al* [6]. They, however, compare favourably with the work of Briggs *et al* [7] for the system, benzene-acetic acid-water.  $K_{da}$  values decreased slowly with increase in packing heights up to a height of about 0.60M and seemed to level out (Fig. 2). Roy *et al* [4] had

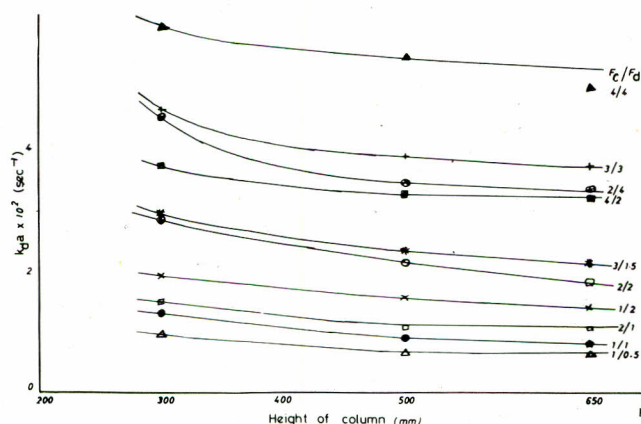


Fig. 2.  $K_{da}$  values against column height

made a similar observation, while Leacock and Churchill [8] even found that  $K_{da}$  values were almost insensitive to any variation in packing height for the binary system *iso*-butanol-water.  $K_{da}$  values were also found to increase with increase in total flow (Fig. 3). The graphical lines are close, thus suggesting that the influence of total flow was more critical than that of the flow ratio.

The highest values of mass transfer coefficients appeared to correspond to equal flow rates of the phases. Perhaps this is not surprising as a large aqueous flow rate could give a smaller hold-up of the solvent phase, while, on the other hand, a larger solvent flow rate could lead to drop coalescence with both effects giving a reduction in interfacial area for mass transfer. When the dispersed flow rate was varied at constant continuous phase flow rate, a rapid increase in  $K_{da}$  with increase in solvent flow rate was noticed (Fig. 4). This is consistent with the work of Leacock and Churchill [8] and that of Roy, *et al* [5].

Values of the overall height of transfer units based on the solvent phase,  $(HTU)_{od}$ , ranged between 0.022 and 0.052M, while those of the aqueous phase  $(HTU)_{oc}$ , were between 0.021 and 0.060M for a packed height of 0.65M, the higher value of a corresponding pair being in favour of the higher flow rate. The differences between  $(HTU)_{oc}$  and  $(HTU)_{od}$  are not significant, thus showing that resistance to mass transfer was similar in both phases.

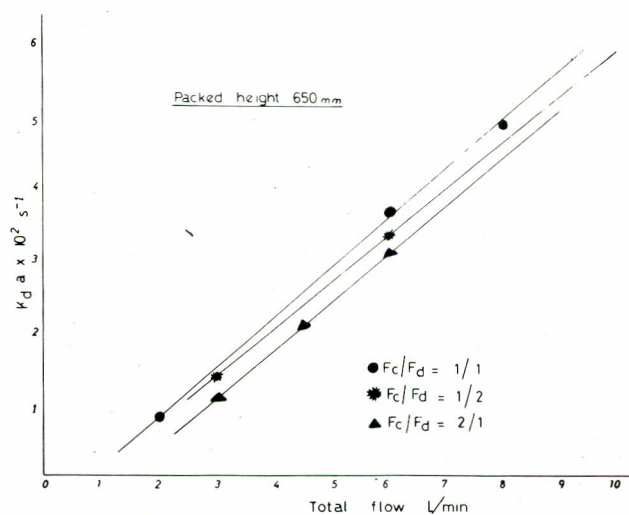


Fig. 3. Effect of total flow on  $K_{da}$

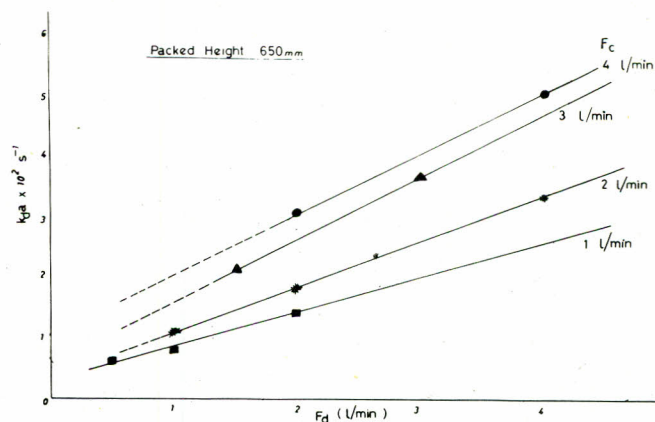


Fig. 4. Effect of flow rates on  $K_{da}$

## CONCLUSIONS

The rate of extraction of succinic acid from an aqueous phase into *n*-butanol in a packed column operated co-currently was very fast and the experimental data gave mass transfer coefficients that compare favourably with published values for counter-current works. The resistance to mass transfer in the two phases appeared similar.

Although the extraction rate was most favourable for equal flow rates of the phases, solvent inventory might make it uneconomical to run a column in this manner. For a more detailed study of the mass transfer characteristics of this system, the column height should preferably not be less than 0.60M. Below this the height end-effect was large and unpredictable.

The relatively low values of the overall heights of transfer units imply that only short columns are required for the extraction, and since only one equilibrium stage can

be achieved in a co-current run, for an industrial process, a cascade of short co-current columns connected counter-currently to mixer-settlers might prove valuable.

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