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SHORTER COMMUNICATION

MASS-TRANSFER COEFFICIENTS OF ACETONE IN THE VOLUME CHANGING SYSTEM n-BUTYLACETATE/ACETONE/WATER

W. Halwachs* and H. R. Howind
Institut für Technische Chemie der TU Hannover, D-3000 Hannover, Callinstraße 3, West Germany

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NOMENCLATURE

c, concentration of acetone [mol/l];
V, volume of acetone, water, n-butylacetate [ml];
d, propeller diameter [cm];
F, interface [cm^2];
H, Henry-coefficient;
M, molecular weight [g/mol];
n, stirrer speed [min^{-1}].

Greek symbols

β, mass transfer coefficient [cm/s];
ν, kinematic viscosity [cm^2/s];
ρ, density [g/l].

Subscripts

Ac, acetone;
BuAc, n-butylacetate;
w, water.

Superscript

0, start condition.

INTRODUCTION

The knowledge of the mass-transfer coefficient β is of great importance in planning and design of liquid–liquid extraction contactors. Only a vanishing amount of quantitative mass-transfer data exists concerning the mass-transfer coefficient β, though there is a real industrial demand, especially for those systems which undergo a volume change during the transfer of the solute. Such an extraction system of great industrial interest for planning and laying out standard extraction processes is the mass transfer of acetone in the liquid–liquid system n-butylacetate–water.

The determination of β in stirred cells is most convenient, because it is possible to obtain time-constant hydrodynamic flow patterns and a definite interface area. In addition to that it is the only way to determine the time dependent mass-transfer coefficient in volume changing systems quantitatively (except single drop mass-transfer experiments including the liquid-scintillation technique [1], which is a more complicated method).

To show the experimental way of determination and the calculation of β via new simple algebraic equations in the case of a real industrial extraction process is the aim of this paper.

β-values measured in stirred cells could be helpful as a basis for the calculation of other extraction contactors, if it is possible to determine the specific interface, by a photometric method for example, to get the product $\beta \cdot F$.

A further aim of this paper was the presentation of some simple algebraic formulae for the calculation of mass-transfer coefficients in volume changing systems.

The concentration/time course during the transfer of the solute acetone was measured in both directions, water → n-butylacetate (stirrer speed: 264, 142, 106 min^{-1}) and n-butylacetate → water (stirrer speed: 264, 222, 140, 106 min^{-1}).

EXPERIMENTAL SECTION

Figure 1(a) shows an outline of the stirred cell in which the researches were done, Fig. 1(b) demonstrates the idealized flow pattern in the cell, Fig. 1(c) gives the profile of the cell to point out the geometry of the flow deflecting plate.

All mass exchange experiments were performed under isothermal conditions at 25°C, start concentration of acetone was always 0.86 mol/l. Two mechanical stirrers were used to avoid a disturbance of the liquid–liquid interface. Different flow patterns in the stirred cell are characterized by their Reynolds numbers:

$$Re = \frac{n \cdot \pi \cdot d^2}{60 \cdot \nu}.$$

To get a calm interface, Reynolds numbers of both phases should be equal, so in all runs $n_{BuAc} = n_w \cdot 0.86$.

Mass-transfer measurements are extremely sensitive referring to pollution. Therefore it was necessary to dismantle and decontaminate the stirred cell after each run for at least 15 h in a Deconex®-bath (supplied by Borer, Switzerland). Water was

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*To whom all correspondence should be sent.

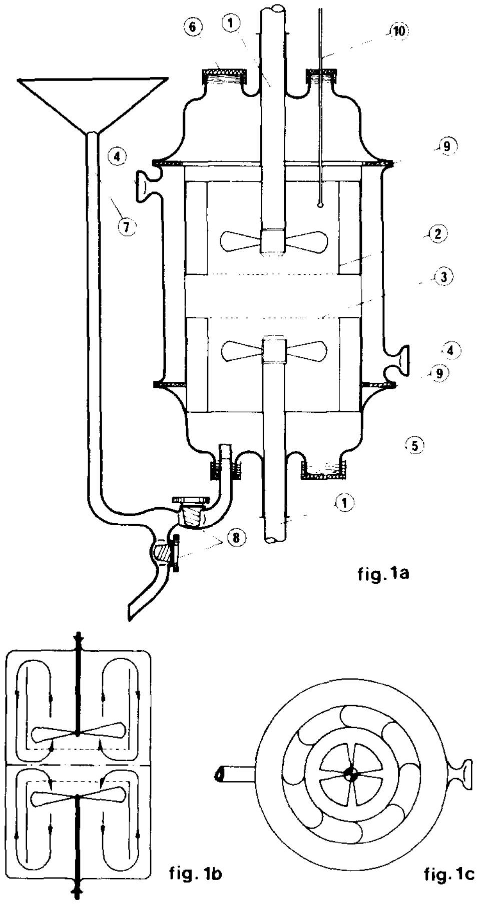

FIG. 1(a). Schematic diagram of the stirred cell. (1) Stirrer, (2) flow deflecting plate, (3) sieve, (4) thermostat connection, (5),(6) silicone membrane, (7) funnel, (8) Teflon stopcock, (9) caulking (Teflon, silicone), (10) thermometer. (b). Idealized flow pattern in the stirred cell. (c). Profile of the stirred cell.

bidistilled in a quartz still, acetone and *n*-butylacetate were of a p.a. quality. Before each measurement water was saturated with *n*-butylacetate and *n*-butylacetate with water. For the mass-transfer direction water → *n*-butylacetate the total start-volume of the water phase was 442 ml (414 ml H_2O + 28 ml acetone), *n*-butylacetate volume was 330 ml. In reverse transfer direction total start volume of *n*-butylacetate was 330 ml (309.1 ml *n*-butylacetate + 20.9 ml acetone), the volume of the water phase had been adjusted at 442 ml. The cross-section of the interface was 39.3 cm^2.

The Henry-coefficient $H = c_{BuAc}/c_w$ at equilibrium was determined to be 0.85.

ANALYTICS

The acetone concentration was analysed in the water phase by means of photometry at 262 nm wavelength (Beckman spectrophotometer DB). The concentration of acetone in *n*-butylacetate was calculated via the material balance. During each run acetone samples were taken with a 100 μl syringe and diluted 1:21 with water, so that *n*-butylacetate did not disturb the photometric analysis of acetone.

Table 1. Extinction of acetone in water as a function of the concentration (calibration). $\lambda_{max} = 262\,\mu m$; Dilution 1:21

Concentration (mol/l)	Extinction
0	0
0.1375	0.111
0.273	0.215
0.545	0.44
0.68	0.55
0.95	0.767

Straight line: $\tan\alpha = 0.809$.
Correlation coefficient $r^2 = 0.99994$.

RESULTS AND DISCUSSION

Because the mass transfer of acetone is accompanied by a volume change of both phases and acetone analysis could be performed only in the water phase β must be calculated from measured concentration/time data by the following equations:

$$\beta = \frac{-\dfrac{dc_w}{dt}\left[\dfrac{c_w \cdot V_w^0 \cdot V_{Ac}^0}{c_w^0(V_w^0 + V_{Ac}^0) - c_w V_{Ac}^0} + V_w^0\right]}{F\left(c_w - \dfrac{c_{BuAc}}{H}\right)\left(1 - c_w \cdot \dfrac{M_{Ac}}{\rho_{Ac}}\right)} \tag{1}$$

with

$$c_{BuAc} = c_w \cdot \frac{(V_{Ac}^0 - V_{Ac})(V_w^0 + V_{Ac})}{(V_{BuAc}^0 + V_{Ac}^0 - V_{Ac}) \cdot V_{Ac}} \tag{2}$$

and

$$V_{Ac} = \frac{c_w \cdot V_{Ac}^0 \cdot V_w^0}{c_w^0(V_w^0 + V_{Ac}^0) - c_w \cdot V_{Ac}^0} \tag{3}$$

V_{Ac}^0 is the total acetone volume, V_{Ac} the present acetone volume in the water phase, V_w^0 and V_{BuAc}^0 are the volumes of water and *n*-butylacetate, respectively, without acetone.

This chain of three simple algebraic equations can easily be adapted for other volume changing liquid–liquid extraction systems, with the upper phase corresponding to BuAc and the lower phase to *w*. There is no need for both phases to be of equal volume and H may change in a known (empirically determined) manner with the concentration of C_w. Therefore equations (1)–(3) are very convenient for the calculation of extraction processes in the industrial praxis, if the transport of a solute is accompanied by a change of both phase volumes.

Figure 2 shows β in the $w \rightarrow$ BuAc direction for three stirrer speeds (264, 142, 106 min^{-1}) as a function of the extraction time. With increasing stirrer speed higher β values prevail. Contamination of the interface and changing solvation effects with decreasing concentration lead to a decrease of β as a function of the time.

The same is valid for the reverse transfer direction (BuAc → w, see Fig. 3).

Figure 4 illustrates the dependency of β as a function of the stirred speed n_w in the initial phase of extraction, 5 min after the beginning of mass transfer.

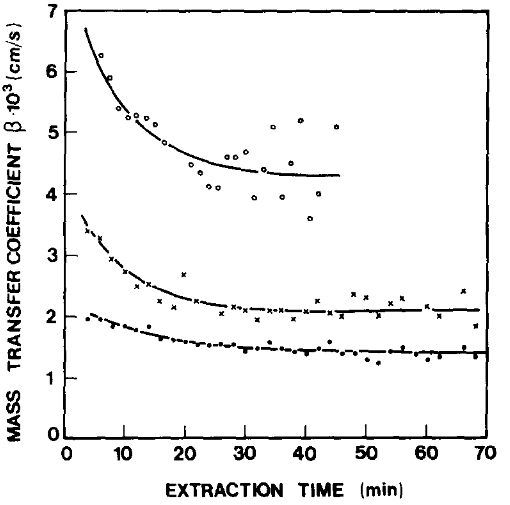

FIG. 2. Mass-transfer coefficient of acetone for three stirrer speeds. Direction $w \rightarrow$ BuAc. (○) 264 m^{-1}, (×) 142 min^{-1}, (●) 106 min^{-1}.

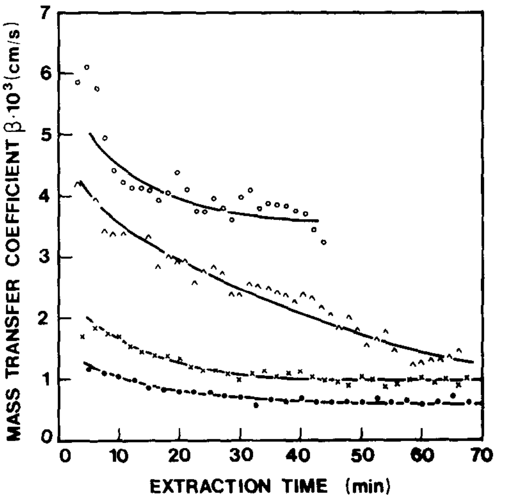

FIG. 3. Mass-transfer coefficient of acetone for four stirrer speeds. Direction: BuAc $\rightarrow w$. (○) 264 min^{-1}, (Λ) 222 min^{-1}, (×) 140 min^{-1}, (●) 106 min^{-1}.

Table 2. β as a function of n_w after 5 min

n_w (min^{-1})	$\beta \cdot 10^3$ (cm/s) $w \rightarrow$ BuAc	BuAc $\rightarrow w$
264	6.2	5.0
222	—	3.8
142, 140	3.3	1.9
106	2.0	1.2

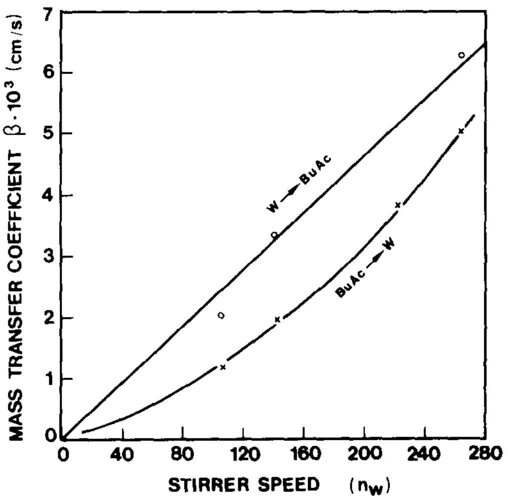

FIG. 4. Mass-transfer coefficient β as a function of the stirrer speed n_w and the direction of transfer 5 min after start of the extraction.

Corresponding values are represented in Table 2. Concerning the $w \rightarrow$ BuAc mass transfer direction larger β values prevail in the total n_w range. This phenomenon was observed before by other authors [2, 3, 6]. The explanation of this effect refers to the formation of convectional cells (roll cells) on the interface, which occur if a solute is transferred from a solvent of definite kinematic viscosity to a solvent of lower kinematic viscosity [2, 4, 5]. These convectional cells lead to an enhancement of mass transfer.

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