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## THE DETERMINATION OF INDIVIDUAL MASS TRANSFER COEFFICIENTS IN LIQUID-LIQUID EXTRACTION

*An improved Lewis cell has been used as an efficient method to determine the mass transfer coefficient for any ternary multi-component system. In this paper the individual mass transfer coefficients were determined for three ternary systems: water–acetone–carbon tetrachloride, water–acetone–chloroform and water–acetone–toluene, using the improved Lewis cell. Criterial equations were developed to calculate the mass transfer coefficients when one or both (organic and aqueous) phases were agitated and when solute transfer occurs in both directions.*

Liquid–liquid extraction, as a separation operation, is based on mass transfer and is widely applied in the chemical industry. Different mechanisms and models have been proposed to explain the very complex phenomenology of this operation [1].

The intensity of mass transfer can be quantified using the individual mass transfer coefficients for each phase. For liquid–liquid extraction, the determination of these coefficients is a difficult task which has been approached by many researchers, using two different methods [2–6].

In one of these methods, two partially miscible liquids were used. One of them is saturated with the other, and the degree of saturation is monitored when they are contacted. This method is not very precise and can be applied only in a limited number of situations.

In another approach, ternary systems were used, having different values of the partition coefficients which are also not one. In this case, it may be considered that the mass transfer resistance is located in the phase with the lower equilibrium concentration. This hypothesis involves a great degree of uncertainty [7] and the method cannot be applied to any system.

In both the methods described briefly above, one of the contacted phases is dispersed as drops in the other, so that the interfacial area is difficult to determine, because the drops are deformable and the mixing intensity between the two phases is variable [8–10].

In this study, these disadvantages were overcome by using an improved Lewis cell, which operates continuously. The experimental set-up and the operating procedure allow the interfacial areas (contact surface between phases) to be determined precisely, while the mixing can be well controlled for every phase.

The Lewis cell [11] with several improvements remains the most efficient method of determining mass

transfer coefficients for any ternary system or for systems with more components, as stated in the G.L. Standard. Numerous researchers developed such modified cells, especially for reactive extraction study [3].

The Lewis cell presented in this work was used to determine the individual mass transfer coefficients for three ternary systems:

- water–acetone–carbon tetrachloride [4];
- water–acetone–chloroform;
- water–acetone–toluene.

Statistic analysis of the experimental data enabled a criterial equation for the calculation of the individual mass transfer coefficient, for each phase to be developed. This was possible when one or both phases were stirred and the transfer condition for the solute in both directions was performed. The equations have the

following general form:  $Sh = b Re_{ag}^p Sc^n \left(\frac{D_x}{D_y}\right)^q \beta^r$ .

### EXPERIMENTAL

The cell used for the study of mass transfer in both the continuous and batch operation mode was constructed of Cr–Ni stainless steel and glass (Figure 1). The compartments (1) and (2), where the aqueous and organic phases are introduced, have side walls made out of glass (3). The two edges are equipped with metallic seals (4), which support the bearings of the blade mixer (5), driven by separate motors, with changeable speed, between 80–1200 rpm. The compartments are separated by a metallic plate (6), which has a central hole with the diameter of 30 mm, where the interface between the two liquids is located (7). The hole diameter can be modified using several rings.

Several orifices were also made in the central plate to ensure feed of the compartments with liquids.

To avoid interface deformation by turbulence, three baffles were placed inside each compartment. Each

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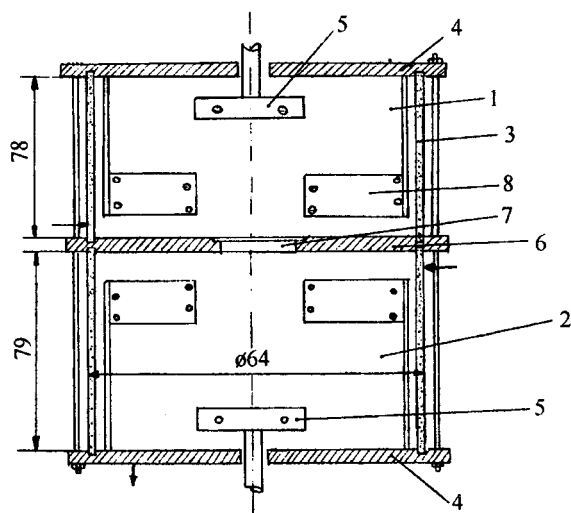


Figure 1. Extraction cell

baffle has four holes of 4 mm diameter. Similar holes were made on the surface of the agitator blade.

The experiments were performed using the following ternary systems: water–acetone–carbon tetrachloride, water–acetone–chloroform and water–acetone–toluene. These systems were chosen so that the partition and diffusion coefficients were quite different, and the reciprocating solubility of the solvents (water– carbon tetrachloride, water– chloroform and water– toluene) was considered negligible.

The partition coefficients for the system consisting of aqueous–organic phases have the following values at 25°C:

- water–acetone–carbon tetrachloride  $\beta_{XY} = 0.175$  – 0.52 (0.25 under the operating conditions) [12];
- water–acetone–chloroform  $\beta_{XY} = 2.35$  [13];
- water–acetone–toluene  $\beta_{XY} = 0.83$ .

The diffusion coefficient of the solute in the aqueous phase is  $D_x = 0.923 \times 10^{-9} \text{ m}^2/\text{s}$ , while in the organic phase it has the following values: carbon tetrachloride  $D_y = 1.763 \times 10^{-9} \text{ m}^2/\text{s}$ , chloroform  $D_y = 3.35 \times 10^{-9} \text{ m}^2/\text{s}$  and toluene  $D_y = 2.88 \times 10^{-9} \text{ m}^2/\text{s}$  [14].

The liquids are purified by distillation before use. In the first set of experiments, the cell was fed with an aqueous solution containing 7% wt. acetone and with a pure organic phase. In the second set of experiments, the cell was fed with an organic phase containing 7% acetone and with distilled water, as well. In this way, the mass transfer of the solute in two directions was studied. When the distillation of the carbon tetrachloride and chloroform was carried out, the azeotrope with acetone was considered. Acetone was previously removed from the organic liquids using water.

The acetone concentrations at the entrance and exit for both phases were determined using the refractometric method. The acetone balance was used to verify the accuracy of the measurements. To establish the dominant resistance, the experiments occurred under the following conditions:

- mixing of the aqueous phase only;
- mixing of the organic phase only;
- mixing of both phases.

The rotational speed of the mixers ranged from 0 to 1200 rpm. When the speed was larger than 1000 rpm for the aqueous phase and 900 rpm for the organic phase, the interface began to deform and the flux of the transferred solute increased.

The feeding flow rate of the organic phase was 4.14 l/h, while that for the aqueous phase was 3.78 l/h.

## RESULTS AND DISCUSSIONS

The cell used for experimentation has a very small specific surface (namely  $1.4 \text{ m}^2/\text{m}^3$ ) calculated as the ratio of the interface area between phases to the volume of both compartments of the cell. This type of specific surface calculation applies for industrial extraction columns, as well. Under these conditions, it is to be expected that the specific flux of the transferred solute would be small and dependent on the cell compartments.

The specific fluxes of the transferred solute were calculated based on the input and output compositions of the extract and raffinate, knowing the surface of the contact interface between the phases ( $s=7.068 \times 10^{-4} \text{ m}^2$ ).

The specific fluxes increase continuously with the agitator speed up to a limit value. Over this value, the flux becomes independent of the rotation speed (Figure 2).

The flux remains constant in the rotation range between 650–1100 rpm, only when the aqueous phase is stirred (curve 1); its value is  $105 \text{ kgAc}/\text{m}^2$ .

The largest values of the flux are obtained when both phases are agitated (curve 3). Starting from point

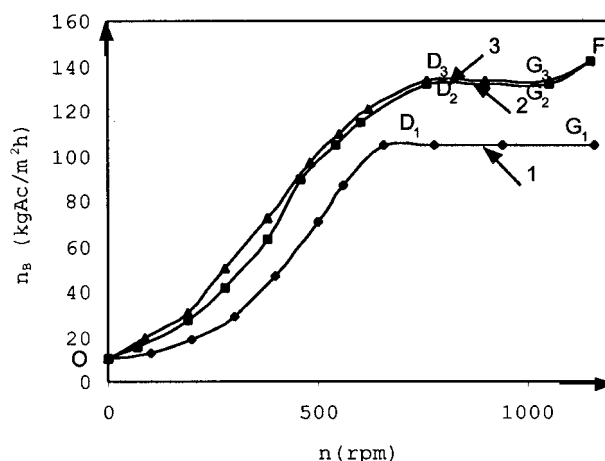


Figure 2. Dependence of the specific flux of the transferred solute on the stirring intensity, for transfer from the aqueous to the organic phase (water–acetone–chloroform ternary system), 1 – the aqueous phase stirred, the organic phase non-stirred; 2 – the organic phase stirred, the aqueous phase non-stirred, 3 – both phases stirred;

DG – domains were the specific flux becomes independent of the agitator speed;

GF – domains were the interface begins to deform.

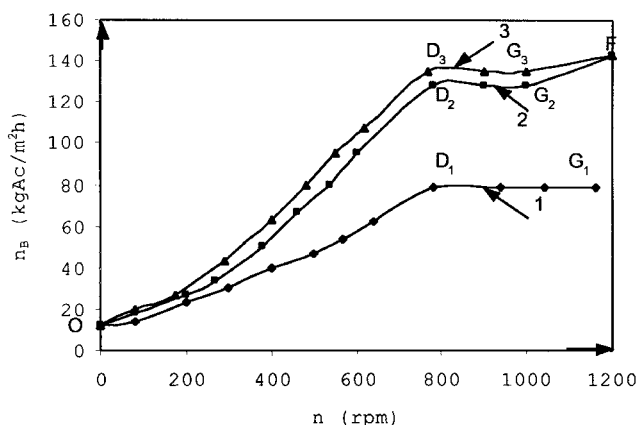


Figure 3. Variation of the specific flux of the transferred solute with agitation intensity for acetone transfer from the organic to the aqueous phases (water–acetone–chloroform ternary system) 1 – the aqueous phase stirred, the organic phase non–stirred; 2 – the organic phase stirred, the aqueous phase non–stirred; 3 – both phases stirred.

$D_3$ , increasing the rotation rate does not affect the mass transfer of acetone from the aqueous to the organic phase. The specific flux remains constant, at 134 kgAc/m<sup>2</sup>.h.

The dependence of the specific flux of the transferred solute on the mixing intensity is presented for the acetone transfer from the organic to the aqueous phase in Figure 3.

The smallest values of the flux of the transferred solute are also obtained when only the aqueous phase is agitated (curve 1), and when the acetone equilibrium concentration is small.

For speeds higher than 1000 rpm, the flux shows a significant increase because of the deformation of the interface. Visually, undulations on the surface can be observed, leading to an increase of the surface.

For the ternary system: water–acetone–chloroform, the difference between curves 1 (which represent the specific flux of the transferred solute when only the aqueous phase is stirred) and curves 3 (which represent the specific flux of the transferred solute when both phases are stirred) is less than that of the water–acetone–carbon tetrachloride system. A possible explanation of this behavior could be the difference between the diffusion coefficients of the solute in the aqueous phase and in the organic phase.

It is obvious that stirring overcomes the effect of molecular diffusion, but when only a single phase is agitated, the difference between the molecular diffusion coefficients should affect the process. For example, in the water–acetone–carbon tetrachloride system the diffusion coefficient in the unstirred phase is  $D_x=0.923 \times 10^{-9}$  m<sup>2</sup>/s, while for the water–acetone–chloroform system, this value is  $D_y=3.35 \times 10^{-9}$  m<sup>2</sup>/s.

The literature does not offer information regarding the influence of the direction of solute transfer on liquid–liquid extraction. This study tries to highlight this effect. For each ternary system, experimental data were

acquired when acetone was transferred from the aqueous phase to the organic one and vice-versa. The specific flux is greater in the water–acetone–carbon tetrachloride system, if the solute is transferred from the phase in which the equilibrium concentration is smaller to the phase in which the equilibrium concentration is higher. This situation is the same for the other two systems studied, as well. These results have been confirmed by the experimental data of other researchers [13].

The rules stated to evidence the effect of the transfer direction and the individual mass transfer coefficients on the transferred flux are valid for those domains where the concentration and the specific flux become independent of the agitator speed (the ranges DG, for the curves from Figures 2 and 3).

The experimentally determined specific fluxes, as well as the individual driving force from the equilibrium curves, allow for the calculation of the individual mass transfer coefficients, using the following equations:

$$n_B = k_{X,XY} (X_{A,i} - X_{A,e}) = k_{Y,XY} (Y_{S,e} - Y_{S,i}) \quad (1)$$

$$n_B = k_{X,YX} (X_{S,e} - X_{S,i}) = k_{Y,YX} (Y_{A,i} - Y_{A,e}) \quad (2)$$

where B refers to acetone, A– component of the initial solution where B is dissolved; S–extraction solvent;  $n_B$  [kgAc/(m<sup>2</sup> · s)]; X [kgAc/m<sup>3</sup> water]; Y [kgAc/m<sup>3</sup> organic phase] and k [m/s].

The variation of the individual mass transfer coefficients when both phases are stirred is presented in Figure 4, for the system water–acetone–carbon tetrachloride.

These coefficients show higher values when the transfer occurs from the aqueous to organic phase (curves 1 and 2), that is from the phase with a higher solute equilibrium concentration to that with a lower one.

For the same direction of solute transfer, the individual mass transfer coefficients of the phases have similar values. When the transfer occurs in both directions, the individual mass transfer coefficient of the receiving phase is higher than that of the other one.

The dependence of the individual mass transfer coefficients for the system water–acetone–chloroform on the agitator speed, when both phases are stirred, is shown in Fig. 5.

These coefficients have higher values in both phases, if the acetone is transferred from the organic to the aqueous phase (curves 1 and 2, Figure 5), i.e. from the phase with the higher solute equilibrium concentration to that with the lower one.

For the same direction of transfer, the differences between the individual mass transfer coefficients of the phases are higher for water–acetone–chloroform (curve 1, 2 and respectively 3, 4; Figure 5) than for the ternary system water–acetone–carbon tetrachloride (curve 3, 4 and respectively 1, 2; Figure 4), because the partition coefficients differ greatly from 1. Also, the individual mass transfer coefficient for the receiving phase in the

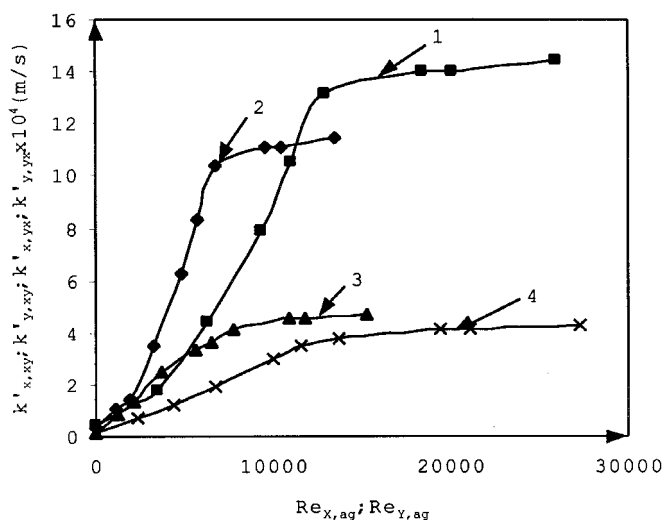


Figure 4. Variation of the individual mass transfer coefficients with agitation intensity in the water-acetone-carbon tetrachloride ternary system, for solute transfer in both direction, when both phases are agitated 1 -  $k'_{y,xy}$ ; 2 -  $k'_{x,xy}$ ; 3 -  $k'_{x,yx}$ ; 4 -  $k'_{y,yx}$

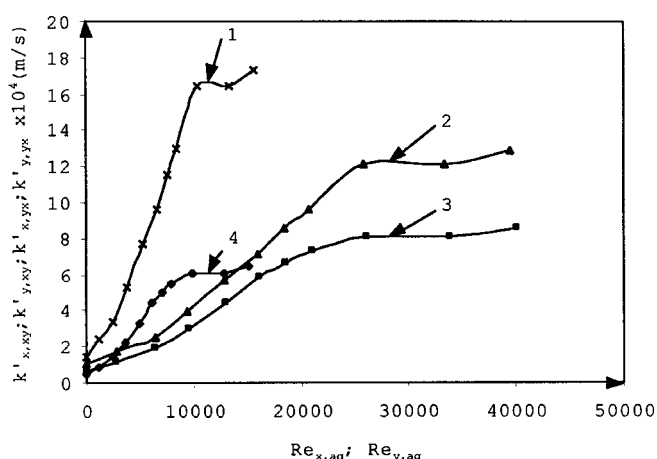


Figure 5. Variation of the individual mass transfer coefficients with agitation intensity in the water-acetone-chloroform ternary system, for solute transfer in both direction, when both phases are agitated 1 -  $k'_{x,yx}$ ; 2 -  $k'_{y,yx}$ ; 3 -  $k'_{y,xy}$ ; 4 -  $k'_{x,xy}$

water-acetone-chloroform system in both directions, is higher than that in the other phase.

Using Eqs. 1 and 2, the individual mass transfer coefficients were calculated for the water-acetone-toluene system when both phases were agitated. The results were presented in Figure 6.

The small difference between the densities of the aqueous and organic phases, as well as the values of the partition coefficients being close to 1 for this ternary system, determine that the curves presented in Figure 6 are closer together.

The results for the water-acetone-toluene system are similar to those obtained for the water-acetone-carbon tetrachloride system. This can be explained by the fact that for both these ternary systems, the solute concentration is higher in the aqueous phase. In the

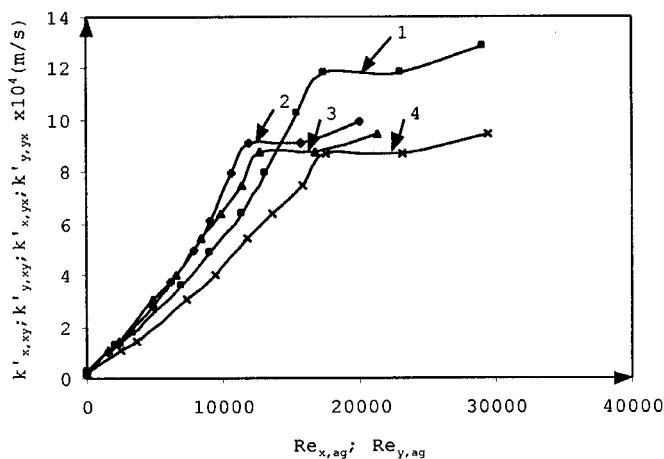


Figure 6. Dependence of the individual mass transfer coefficients of the stirring intensity in the water-acetone-toluene system, for solute transfer in both directions, when both phases are agitated 1 -  $k'_{y,xy}$ ; 2 -  $k'_{x,xy}$ ; 3 -  $k'_{x,yx}$ ; 4 -  $k'_{y,yx}$

third ternary system investigated and for the same transfer direction as in the ternary water-acetone-toluene system the values of the individual mass transfer coefficients were found to be higher in the receiving phase.

Dimensional analysis of the variables and dimensional constants that influence mass transfer during liquid-liquid extraction leads to a group of dimensional criteria that can be used to calculate the individual mass transfer coefficients, for each phase, when one or both phases are stirred and for solute transfer in both directions. These equations have the following general form:

$$Sh = b Re_{ag}^n Sc^n \left( \frac{D_1}{D_2} \right)^q \beta^r \quad (3)$$

were  $n=1/3$ ,  $q=1/2$  and are presented in Table 1.

The exponents of Re and  $\beta$  have different values depending on the stirred phase and solute transfer direction.

The distribution coefficient  $\beta$  appears in the criterial equation in two situations (Eqs. 2 and 3, Table 1):

- in number Sherwood,  $Sh'$  for the aqueous phase, when both phases are stirred and the transfer direction is  $Y \rightarrow X$ ;
- in the number Sherwood,  $Sh'$  for the organic phase, when both phases are stirred and the transfer direction is  $X \rightarrow Y$ .

These equations were verified for the three studied ternary system. The results are presented in Figures 7 and 8, respectively. The errors were less than 3%.

## CONCLUSIONS

The experimental installation has an improved Lewis cell as the central element. The cell can be exploited in continuous and discontinuous modes, also allowing for steady-state operation for a short time when

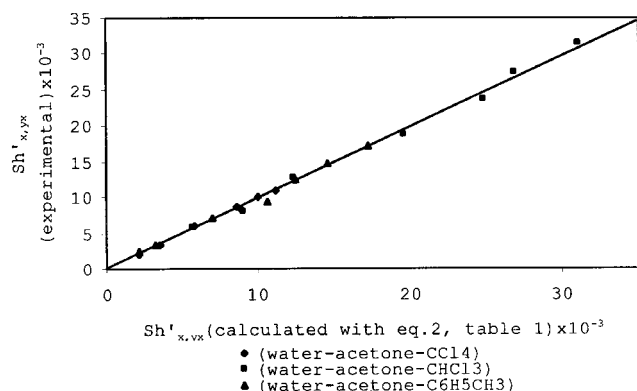


Figure 7. Verification of the relationship (2) from Table 1

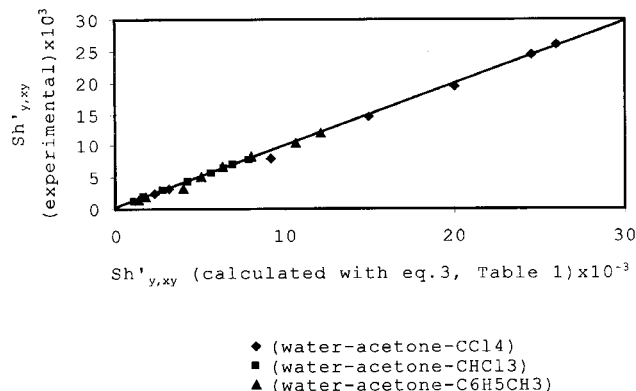


Figure 8. Verification of the relationship (3) from Table 1

continuous running is used. The Lewis cell is equipped with stirrers driven by motors with changeable speeds, which are placed in each compartment of the cell. Each compartment of the cell is equipped with baffles with holes, to avoid liquid rotation and stagnant zones. The mass transfer area can be rigorously determined and modified under certain limits. The improved Lewis cell is one of the most efficient methods for determining mass transfer coefficients for any ternary system.

The dominant mass transfer resistance is concentrated in the organic phase for water-acetone-carbon tetrachloride and water-acetone-toluene ternary systems and in the aqueous phase for the water-acetone-chloroform system. The mass transfer intensity is higher when both phases, are stirred than

when only a single phase is agitated. The mass transfer intensity from a certain agitated phase increases significantly with increasing stirrer speed upto a certain limit, after which the transferred flux becomes independent of the agitator speed.

The smallest value of the transferred flux of the solute was obtained when only the phase with the lower equilibrium concentration was agitated (organic phase for water-acetone-carbon tetrachloride and water-acetone-toluene and aqueous phase for water-acetone-chloroform ternary systems). This conclusion is valid for all studied ternary systems.

The specific transferred flux of the solute is higher when the transfer direction is from the phase with the

Table 1. Criterial equations developed to calculate the individual mass transfer coefficients for each phase, when one or both phases are agitated and for solute transfer in both directions

Equation	Ternary system	Re	Sc	Comments
$Sh'_{x,xy} = 54.19 \cdot Re^{1.19} Sc^{1/3} \left( \frac{D_x}{D_y} \right)^{1/2}$ (1) in the aqueous phase	water-acetone- $CCl_4$ water-acetone- $CHCl_3$ water-acetone- $C_6H_5CH_3$	$Re_x < 9500$ $Re_y < 17500$	100÷1000	both phases agitated the transfer direction $X \rightarrow Y$
$Sh'_{x,yx} = 231.601 \cdot Re^{0.95} Sc^{1/3} \left( \frac{D_x}{D_y} \right)^{1/2} (-0.0074 \cdot \beta^2 - 0.011 \cdot \beta + 1.091)$ (2) in the aqueous phase	water-acetone- $CCl_4$ water-acetone- $CHCl_3$ water-acetone- $C_6H_5CH_3$	$Re_x < 10300$ $Re_y < 17500$	100÷1000	both phases agitated the transfer direction $Y \rightarrow X$ , $\beta = X/Y$
$Sh'_{y,xy} = 54.15 \cdot Re^{1.19} Sc^{1/3} \left( \frac{D_x}{D_y} \right)^{1/2} (0.341 \cdot \beta^2 - 1.249 \cdot \beta + 1.474)$ (3) in the organic phase	water-acetone- $CCl_4$ water-acetone- $CHCl_3$ water-acetone- $C_6H_5CH_3$	$Re_x < 9500$ $Re_y < 17500$	100÷1000	both phases agitated the transfer direction $X \rightarrow Y$ , $\beta = Y/X$
$Sh'_{y,yx} = 111.246 \cdot Re^{0.95} Sc^{1/3} \left( \frac{D_y}{D_x} \right)^{1/2}$ (4) in the organic phase	water-acetone- $CCl_4$ water-acetone- $CHCl_3$ water-acetone- $C_6H_5CH_3$	$Re_x < 10300$ $Re_y < 17500$	100÷1000	both phases agitated the transfer direction $Y \rightarrow X$
$Sh_{x,xy} = 4.721 \cdot Re^{1.45} Sc^{1/3} \left( \frac{D_x}{D_y} \right)^{1/2}$ (5) in the aqueous phase	water-acetone- $CCl_4$ water-acetone- $CHCl_3$ water-acetone- $C_6H_5CH_3$	$Re_x < 9500$	100÷1000	only the aqueous phase agitated $X \rightarrow Y$
$Sh_{x,yx} = 180.5 \cdot Re^{0.96} Sc^{1/3} \left( \frac{D_y}{D_x} \right)^{1/2}$ (6) in the aqueous phase	water-acetone- $CCl_4$ water-acetone- $CHCl_3$ water-acetone- $C_6H_5CH_3$	$Re_x < 10300$	100÷1000	only the aqueous phase agitated $Y \rightarrow X$
$Sh_{y,xy} = 694.21 \cdot Re^{0.795} Sc^{1/3} \left( \frac{D_x}{D_y} \right)^{1/2}$ (7) in the organic phase	water-acetone- $CCl_4$ water-acetone- $CHCl_3$ water-acetone- $C_6H_5CH_3$	$Re_y < 17500$	100÷1000	only the organic phase agitated $X \rightarrow Y$
$Sh_{y,yx} = 39.129 \cdot Re^{1.015} Sc^{1/3} \left( \frac{D_y}{D_x} \right)^{1/2}$ (8) in the organic phase	water-acetone- $CCl_4$ water-acetone- $CHCl_3$ water-acetone- $C_6H_5CH_3$	$Re_y < 17500$	100÷1000	only the organic phase agitated $Y \rightarrow X$

higher acetone equilibrium concentration, for all studied ternary systems.

When both phases are agitated and the transfer occurs in both directions, the individual mass transfer coefficient is greater in the receiving phase.

Statistical analysis of the experimental data allowed the development of some criterial equations, used to calculate the individual mass transfer coefficients for each phase when one or both phases are stirred and the solute transfer occurs in both directions. The general form of these equations is:

$$Sh = b Re_{ag}^p Sc^n \left( \frac{D_x}{D_y} \right)^q f(\beta).$$

## NOTATIONS

### Symbols used

A	[-]	– component of the initial solution were B is dissolved
Ac	[-]	– acetone
d	[m]	– agitator diameter
D	[m <sup>2</sup> · s <sup>-1</sup> ]	– diffusion coefficient
k	[m · s <sup>-1</sup> ]	– individual mass transfer coefficient when only one phase is stirred
k'	[m · s <sup>-1</sup> ]	– individual mass transfer coefficient when both phases are stirred
n	[rpm]	– stirrer speed
n <sub>B</sub>	[kgAc/m <sup>2</sup> s]	– the specific flux of the transferred solute
Re	[-]	– Reynolds number, = $\rho \cdot n \cdot d^2 / \eta$
s	[m <sup>2</sup> ]	– mass transfer surface
S	[-]	– notation for the extraction solvent
Sc	[-]	– Schmidt number, = $\eta / \rho \cdot D$
Sh	[-]	– Sherwood number, = $k \cdot d / D$
X	[kgAc/kgwater]	– acetone concentration in the aqueous phase
Y	[kgAc/kgorganic phase]	– acetone concentration in the organic phase
$\rho$	[kg · m <sup>-3</sup> ]	– density

$\eta$	[Pa · s]	– viscosity
$\beta$	[-]	– distribution coefficient

### Subscript

ag	– agitation
B	– solute
e	– equilibrium
S	– solution
X	– aqueous phase
XY	– the transfer direction is from the aqueous to the organic phase
Y	– organic phase
YX	– the transfer direction is from the organic to the aqueous phase

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## IZVOD

### ODREĐIVANJE INDIVIDUALNIH KOEFICIJENATA PRENOŠENJA MASE U EKSTRAKCIJI TEČNOST–TEČNOST

(Naučni rad)

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Koeficijenti prenošenja mase se mogu kod bilo kod ternernog višekomponentnog sistema odrediti primenom metode zasnovane na korišćenju modifikovane Lewis-ove ćelije. U ovom radu određivani su koeficijenti prenošenja mase kod tri različitih ternernih sistema: voda–aceton–ugljentetrahlorid; voda–aceton–hloroform i voda–aceton–toluen.

Izvedene su odgovarajuće kriterijumske jednačine koje omogućavaju izračunavanje koeficijenata prenošenja mase rastvorka u jednoj ili obe faze (vodenoj i organskoj) koje se mešaju.

Ključne reči: Tehnološke operacije  
 • Ekstrakcija • proces prenošenja mase • prenošenje mase • Prenošnje acetona • Kriterijalne jednačine •  
 Key words: Unit operations • Extraction • Transport processes • Mass transfer • Acetone Transfer • Criterial equation •