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RECOVERY OF ZINC(II) FROM MODEL HYDROCHLORIC ACID SOLUTIONS IN HOLLOW FIBER MODULES

The recovery of zinc(II) from model hydrochloric acid solutions in hollow fiber modules was studied. It was found that zinc(II) could be removed from these model solutions by 80% tributyl phosphate in kerosene. The efficiency of extraction was limited by low values of zinc(II) distribution coefficients. Two types of experiments were carried out: the extraction in one module and the extraction-stripping process in a set of two modules. Simplified models of mass transfer based on the solute concentration changes in the aqueous phases were formulated and verified.

Key words: hollow fiber module, extraction, stripping, hot-dip galvanizing, modelling

INTRODUCTION

Hot-dip zinc coating method has been introduced on an industrial scale by Sorel in 1836. It is nowadays one of the most commonly used method of protecting steel from corrosion. The method generates problems of spent pickling solution, which has recently become increasingly important due to tightening of environmental regulations. The pickling of steel goods is usually carried out with 20% HCl at room temperature, and the process is stopped when the concentration of hydrochloric acid achieves 10%. Such a solution can be successfully used for depleting the bad quality zinc protective layers, and the concentration of free hydrochloric acid decreases then near 1%. During the pickling the rust is dissolved and accumulation of iron, mainly iron(II), in the hydrochloric solution occurs. When pickled goods are housed on recycled hooks, i.e. covered in previous process with zinc, an accumulation of zinc(II) in the pickling solution is also observed. If waste pickling solution is additionally used

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to deplete the wrongly deposited zinc layer, then zinc(II) concentration in the spent pickling solution may increase to a few tens g/dm^3. Thus, the spent pickling solution may contain: Zn(II) (10 – 130 g/dm^3), Fe(II) (25 - 75 g/dm^3), Fe(III) (10 - 20 g/dm^3) ions and Cl$^-$ (approx. 5 mol/dm^3) anions. The most economically justified method of recovery of the metal ions is solvent extraction. Many popular extractants have been tested in last years (Cierpiszewski et al., 2002, Regel et al. 2001, Kirschling et al., 2001). Tributyl phosphate (TBP) has been chosen as the only extractant allowing efficient and selective extraction and stripping of zinc after reduction of Fe(III) to Fe(II).

Tributyl phosphate is a week extractant of zinc(II). It has to be used as a concentrated solution (above 80%) in kerosene. The use of undiluted extractant is inconvenient because of relatively high mutual solubility of TBP and water. Additionally, a third phase consisted of TBP, water and HCl may be formed in the system. Another constraint results from very small difference of water and TBP densities. This often causes problems with phases separation due to emulsification.

All the aforementioned problems could be avoided when the extraction-stripping process was carried out in the supported liquid membrane systems. Due to the immobilizing of organic phase in the pores of membrane the difference of densities as well as interfacial tension can be very small (de Gyves and de San Miguel, 1999).

The hollow fiber liquid membrane modules have been increasingly explored since 1980's as they provide large area of phases' contact. In the presented work single extraction as well as extraction-stripping processes were carried out in one and two parallel hollow fiber modules, respectively. The research was limited to zinc(II) extraction because it was shown previously (Regel et al., 2001) that Fe(II) ions do not deteriorate zinc(II) extraction as they are not transported by TBP. The aim of work was checking of the possibility of using TBP in membrane process of removing zinc(II) ions from model solutions as well as simplified mathematical modeling of such a process.

EXPERIMENTAL

The composition of model aqueous solution was as follows: [Zn(II)]=20 g/dm^3, [H$^+$]=0.54 mol/dm^3, [Cl$^-$]=5 mol/dm^3 (adjusted with NaCl). All materials (ZnCl$_2$, NaCl and HCl from P.O.Ch. Gliwice) were of pure grade. Deionised water from reverse osmosis was used. The original purity of tributyl phosphate (Fluka) was 97%. TBP was mixed with kerosene (Deutsche Exxon Chemical GmbH) in ratio 8:2 in order to avoid forming of a third phase (TBP + water + HCl) during experiments. Dispersion-free extraction-stripping experiments were carried out on a set-up depicted in Fig. 1. Commercial cross-flow Liqui-Cel® Extra-Flow 2.5×8" X30 membranes (Table 1) were used. In all experiments the aqueous feed phase flowed through the tube side and the organic phase through the shell side. Flows were always countercurrent in closed circulations. The total volume of organic phase was equal to about 500 ml. The total

initial volumes of aqueous feed and stripping phases were equal to 850 and 780 ml, respectively. In case of extraction experiments a set-up of one module and two tanks was used.

Table 1. Geometrical parameters for the Liqui-Cel® Extra-Flow
2.5''×8'' X30 modules studied.

Fiber internal diameter	240 µm
Fiber wall thickness	30 µm
Effective pores size	0.03 µm
Porosity	40 %
Effective fiber length	15 cm
Effective surface area	1.4 m^2
Tubeside volume	145 ml
Shellside volume	195 ml
Fiber potting material	Polyethylene

During each experiment ten 15-mililitre samples of water phases were taken and the losses were not replenished with fresh solutions. This fact influenced the process and was taken into account in calculations.
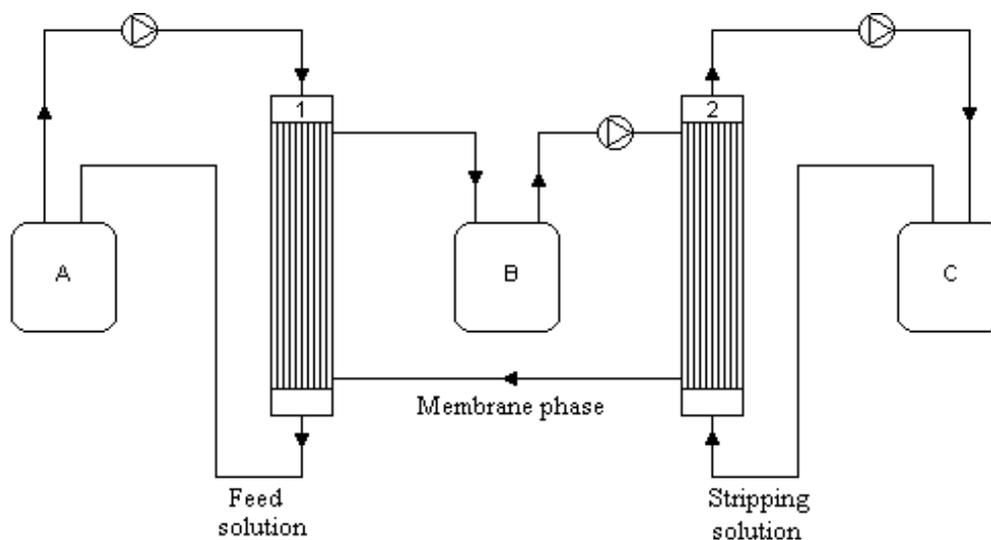

Fig. 1. Experimental two-module extraction-stripping system (A, B, C - tanks; 1 – extraction module, 2 – stripping module). In case of extraction experiments the system consisted of the module 1 and the tanks A and B

Since the fibers were made of polypropylene the pores in membrane's wall were more easily filled with less polar organic phase (Prasad and Sirkar, 1988). In order to avoid forming of dispersion 0.5-bar overpressure was attached on the aqueous phase sides. The concentration of zinc(II) in the aqueous phases was determined by titrating with EDTA.

RESULTS AND DISCUSSION

The distribution coefficients (m_{Zn}) calculated from consecutive experiments carried out in one hollow fiber module decrease in the first four experiments (Table 2). The organic phase was not exchanged during about 2-month research; the only method of its regeneration was washing it with distilled water for two hours after each experiment. Thus, some properties of the membrane phase were changing within the time of research. This assumption was supported by study carried out by Kertes and Halpern (1961) who reported that TBP, being an ester, was slowly hydrolysed in the presence of hydrochloric acid solutions giving di- and monoesters of ortophosphoric acid. In addition, it could not be excluded that some impurities always present in aqueous solutions gathered in the organic phase gradually deteriorating its extractive abilities. Another reason for the problem might be caused by impurities present originally in TBP. Commercial TBP of 97% purity could contain pirophosphoranes which exhibited superior ability to extract metal ions than TBP. However, they were much more susceptible to hydrolysis than TBP. Thus, aging of the membrane phase could directly affect the distribution coefficient of solute.

Table 2. Distribution coefficients of zinc(II) in one-module extraction system

No	Volumetric flow of the feed phase [dm³/min]	Volumetric flow of the membrane phase [dm³/min]	Final zinc(II) concentration in the feed phase[*] [mol/dm³]	Final zinc(II) concentration in the membrane phase[*] [mol/dm³]	Distribution coefficient m_{Zn}
1	1.6	0.46	0.17	0.20	1.18
2	2.4	0.46	0.18	0.21	1.17
3	3.2	0.46	0.19	0.18	0.95
4	4.0	0.46	0.19	0.17	0.90
5	2.4	0.57	0.18	0.18	1.00
6	2.4	0.97	0.18	0.16	0.89
7	2.4	1.60	0.18	0.18	1.00

[*]determined from the plateau of the relationship zinc(II) concentration versus time.

For the case of extraction-stripping process carried out in two parallel hollow fiber modules two distribution coefficients of zinc(II) were calculated, $m_{Zn,1}$ and $m_{Zn,2}$ for the extraction and stripping processes, respectively:

$$m_{Zn,1} = \frac{C^*_{Zn(o)}}{C^*_{Zn}} \qquad m_{Zn,2} = \frac{C^*_{Zn(o)}}{C^*_{Zn(R)}}$$

where C^*_{Zn}, $C^*_{Zn(o)}$ and $C^*_{Zn(R)}$ denote the equilibrium zinc(II) concentration in the feed, organic and stripping phases, respectively. They amounted: $m_{Zn,1} = 0.98$ and $m_{Zn,2} = 0.16$.

Typical profiles of zinc(II) concentration in the aqueous phases versus time are shown in Figs. 2 and 5a. The concentration of solute in the feed phase in extraction system decreased abruptly in the first period of experiments and after about 45 minutes reached approximately constant value. The situation was analogous in two-module system. However, the concentration of zinc(II) in the organic phase achieved a maximum value and then slowly decreased, whilst it reached maximum constant value in the extraction system. The extraction-stripping system achieved equilibrium after a longer period of time compared to one-module system.

Similar concentration profiles were obtained for HCl transfer, although the species was stronger accumulated in the membrane phase. In all experiments the molar ratio of HCl to zinc(II) removed from the feed phase was equal to about 1.2:1.

MODELS OF MASS TRANSFER

In the presented work simplified mathematical models of mass transfer were applied. The models described the solute concentration changes regarding all phases independently.

The models of mass transfer in hollow fiber modules were rarely presented in literature (Escalante et al.,1996). They were based on macroscopic mass balance of solute and took into consideration the solute concentration changes in feed and stripping phase volumes with time.

In case of one-module extraction experiments the model equation was:

$$-\frac{dC_{Zn}}{dt} = K_E \cdot \left(C_{Zn} - C^f_{Zn} \right) \qquad (1)$$

where C^f_{Zn} was the final concentration of zinc in the feed phase, obtained from the plateau of concentration profile versus time.

After integration including the initial condition $C_{Zn} = C_{Zn,i}$ for $t = 0$ the following equation was obtained:

$$C_{Zn} = C^f_{Zn} + \left(C_{Zn,i} - C^f_{Zn} \right) \cdot \exp\left(-K_E \cdot t \right) \qquad (2)$$

The values of apparent mass transfer coefficient K_E were collected in table 3. In most experiments the simple model equation did not reflect properly the solute concentration changes at the initial period of extraction process (see Fig. 2) when the solute concentration decreased abruptly. The discrepancies disappeared when the curves reached plateau.
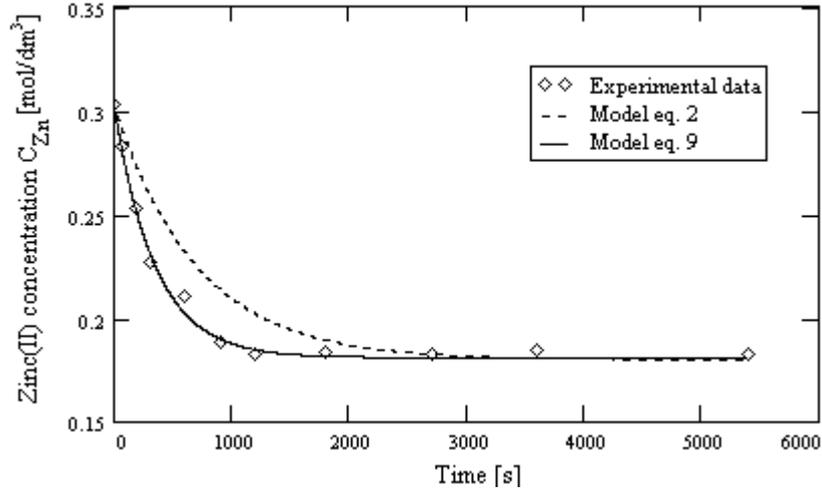

Fig. 2. Exemplary data of zinc(II) concentration changes with time in extraction system
(experiment No. 7 - see Table 2) and the results of fitting model equations

The probable reason for observed discrepancies was the decrease of feed phase volume caused by sampling. The volume of each of 10 samples taken for analysis was equal to 13-15 ml. This resulted in the total loss of feed phase of 15-18% during whole experiments. At the same time the volume of organic phase remained unchanged.

To improve the model equation the volume changes were included into the C_{Zn}^f parameter:

$$C_{Zn,N}^f = \frac{C_{Zn,i} \cdot V_{w,i} - \sum_{j=1}^{N} C_{Zn,j} \cdot V_{w,j}}{V_{w,i} + V_o \cdot m_{Zn} - \sum_{j=1}^{N} V_{w,j}} \qquad (3)$$

where $C_{Zn,i}$ is the initial concentration of zinc(II) in the feed phase, $C_{Zn,j}$ – the concentration of zinc(II) in the sample j, $V_{w,i}$ – the initial volume of the feed phase, $V_{w,j}$ – the volume of sample j taken for analysis and V_o – the volume of organic phase.

The above equation was correct unless the distribution coefficient varied within the range of experimental concentration changes. When the driving force expressed as the

difference of actual and final concentrations of zinc(II) in the feed phase reached zero the sampling did not change the value of C_{Zn}^f any more.

To include the variation of the final solute concentration into model equation it was necessary to express it as a function of time (Fig. 3). The points represented the consecutive values of C_{Zn}^f after sampling. The correlation could be approximated by the following equation:

$$C_{Zn}^f(t) = C_{Zn,N}^f + \left(C_{Zn,0}^f - C_{Zn,N}^f\right) \cdot \exp(-\alpha \cdot t) \tag{4}$$

where $C_{Zn,0}^f$ - the final zinc(II) concentration reached in the system if no samples were taken, $C_{Zn,N}^f$ - the final zinc(II) concentration reached after taking all samples.

The constant α had to be found by means of regression. Then, the model equation became as follows :

$$-\frac{dC_{Zn}}{dt} = K_E^{'} \cdot \{C_{Zn} - \left[C_{Zn}^f + \left(C_{Zn,0}^f - C_{Zn,N}^f\right) \cdot \exp(-\alpha \cdot t)\right]\} \tag{5}$$

After integrating by means of the method of variation of parameters the resultant equation was found to be:

$$C_{Zn} = C_{Zn,N}^f + \frac{K_E^{\copyright}}{K_E^{\copyright} - \alpha} \cdot \left(C_{Zn,0}^f - C_{Zn,N}^f\right) \cdot \exp(-\alpha \cdot t) +$$

$$+ \left[C_{Zn,i} - C_{Zn,N}^f - \frac{K_E^{\copyright}}{K_E^{\copyright} - \alpha} \cdot \left(C_{Zn,0}^f - C_{Zn,N}^f\right)\right] \cdot \exp(-K_E^{\copyright} \cdot t) \tag{6}$$

The apparent mass transfer coefficients calculated by the least squares method were collected in Table 3. The model equation (6) fitted experimental data very well (see Fig. 2).

Table 3. Apparent mass transfer coefficients for one-module extraction system

No	Apparent mass transfer coefficient $K_E \cdot 10^4$ [s^{-1}]	Parameter $\alpha \cdot 10^3$ [s^{-1}]	Apparent mass transfer coefficient $K_E^{\copyright} \cdot 10^4$ [s^{-1}]
1	10.09	2.03	11.54
2	11.91	2.11	12.18
3	8.17	1.94	7.41
4	13.78	2.23	18.94
5	10.44	1.49	17.92
6	17.57	2.41	27.72
7	14.23	2.14	28.94

A disadvantage of equation (6) was the necessity of calculating two constant parameters, i.e. K_E' and α while the equation (1) contained only one parameter, K_E.
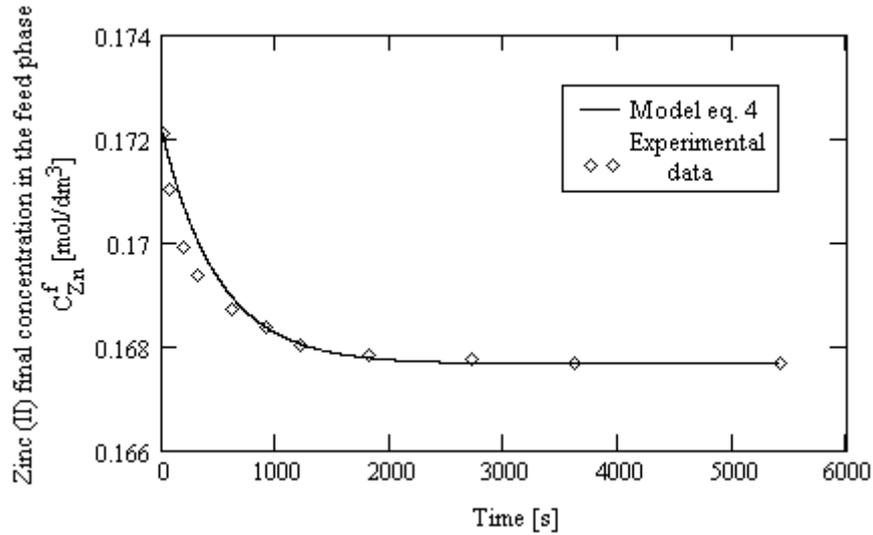

Fig. 3. The variation of zinc(II) final concentration in the feed phase with time
(Experiment No. 1 – see Table 2)

Table 4. Apparent mass transfer coefficients for two-module extraction-stripping system
(volumetric flow of the membrane phase $Q_o = 0.25$ dm^3/min).

No	Volumetric flow of the feed phase [dm^3/min]	Volumetric flow of the stripping phase [dm^3/min]	Apparent mass transfer coefficient $K_E \cdot 10^4$ [s^{-1}]	Apparent mass transfer coefficient $K_R \cdot 10^4$ [s^{-1}]
8	1.6	1.6	6.28	3.65
9	1.7	1.7	6.05	3.70

No	Time t_f [s]	Apparent mass transfer coefficient $K_{R1} \cdot 10^4$ [s^{-1}]	Apparent mass transfer coefficient $K_{R2} \cdot 10^4$ [s^{-1}]
8	256	0.93	15.83
9	332	2.40	17.02

For extraction-stripping process a set of two equations was proposed for describing solute concentration changes (Escalante et al., 1996). For the presented system they were:

$$-\frac{dC_{Zn}}{dt} = K_E \cdot \left(C_{Zn} - C_{Zn}^f\right) \tag{7}$$

$$\frac{dC_{Zn(R)}}{dt} = K_R \cdot \left(C_{Zn(R)}^f - C_{Zn(R)}\right) \tag{8}$$

After integrating, taking K_E and K_R as constants and assuming $C_{Zn} = C_{Zn,i}$ and $C_{Zn(R)} = 0$ when t = 0 one could obtain:

$$C_{Zn} = C_{Zn}^f + \left(C_{Zn,i} - C_{Zn}^f\right) \cdot \exp\left(-K_E \cdot t\right) \tag{9}$$

$$C_{Zn(R)} = C_{Zn(R)}^f \cdot \left[1 - \exp\left(-K_R \cdot t\right)\right] \tag{10}$$

The apparent mass transfer coefficients could be calculated by simple regression. Due to similar conditions in both carried experiments respective values were approximately equal (Table 4).

The fitting to experimental data was satisfactory only in the case of extraction process (see Fig. 5). The model equation (10) exhibited significant discrepancies with experimental points. They were especially strong for the initial period of experiments; the solute concentration appeared to increase much slower than predicting from equation (10).

The model correlation (10) did not reflect the real nature of solute transfer. In most extraction-stripping processes the solute was temporary accumulated in the membrane phase (Fig. 4). In order to derive a model correlation that would properly describe the observed solute concentration changes in stripping phase a mass balance of membrane phase was introduced. The solute concentration changes in organic phase were expressed by the difference between actual and final solute concentrations. Two equations were obtained:

$$V_o \cdot \frac{dC_{Zn(o)}}{dt} = K_E \cdot V_w \cdot \left(C_{Zn} - C_{Zn}^f\right) - K_{R1} \cdot V_R \cdot \left(C_{Zn(o)}^f - C_{Zn(o)}\right)$$

$$\text{for } C_{Zn(o)} < C_{Zn(o)}^f \tag{11}$$

$$V_o \cdot \frac{dC_{Zn(o)}}{dt} = K_E \cdot V_w \cdot \left(C_{Zn} - C_{Zn}^f\right) - K_{R2} \cdot V_R \cdot \left(C_{Zn(o)} - C_{Zn(o)}^f\right)$$

$$\text{for } C_{Zn(o)} \geq C_{Zn(o)}^f \tag{12}$$

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where V_w, V_o and V_R denote the volume of the feed, organic and stripping phases, respectively.

Equation (11) described the variation of solute concentration changes from the initial time $t = 0$ until the moment $t = t_f$ when the actual solute concentration for the first time reached its final value (see Fig. 4). After that the driving force expressed as $(C_{Zn(o)}^f - C_{Zn(o)})$ became negative and the process was thereafter described by equation (12). Combining equations (11) and (12) with (9) two differential equations were obtained. They could be solved by the method of variation of parameters.
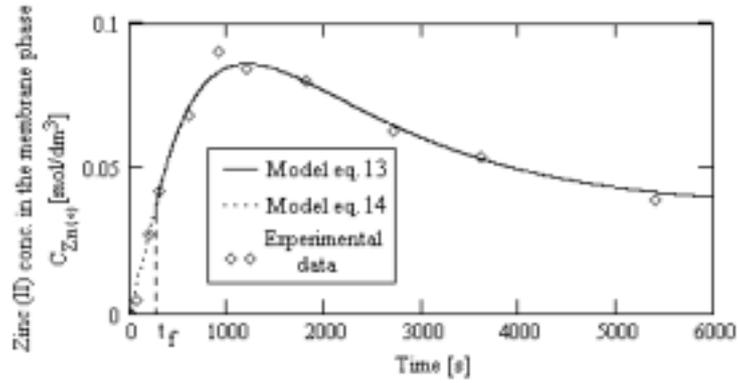

Fig. 4. Zinc(II) concentration profile in the membrane phase in two-module extraction-stripping process
(Experiment No. 8 – see Table 4)

For the initial conditions $t = 0$ $C_{Zn(o)} = 0$ and $t = t_f$ $C_{Zn(o)} = C_{Zn(o)}^f$ the solutions were as follows:

$$C_{Zn(o)} = \frac{V_w \cdot K_E \cdot \left(C_{Zn,i} - C_{Zn}^f\right)}{K_E \cdot V_o + V_R \cdot K_{R1}} \cdot \left[\exp\left(\frac{V_R \cdot K_{R1}}{V_o}\right) - \exp\left(-K_E \cdot t\right)\right] +$$

$$+ C_{Zn(o)}^f \cdot \left[1 - \exp\left(\frac{V_R \cdot K_{R1}}{V_o}\right)\right]$$

$$\text{for} \qquad t < t_f \tag{13}$$

$$C_{Zn(o)} = \frac{V_w \cdot K_E \cdot \left(C_{Zn,i} - C_{Zn}^f\right)}{K_{R2} \cdot V_R + V_o \cdot K_E} \cdot \left\{\exp\left[-K_E \cdot t\right] + \right.$$

$$\left. - \exp\left[\left(\frac{V_R \cdot K_{R2}}{V_o} - K_E\right) \cdot t_f - \frac{V_R \cdot K_{R2}}{V_o} \cdot t\right]\right\} + C_{Zn(o)}^f$$

$$\text{for} \qquad t \geq t_f \tag{14}$$

Introducing a mass balance for the stripping phase:

$$V_R \cdot C_{Zn(R)} = V_w \cdot C_{Zn,i} - V_w \cdot C_{Zn} - V_o \cdot C_{Zn(o)} \qquad (15)$$

and combining equations (13) and (14) with (15) one could obtain:

$$
\begin{aligned}
C_{Zn(R)} = \frac{V_w \cdot C_{Zn,i} - V_w \cdot \left[C_{Zn}^f + \left(C_{Zn,i} - C_{Zn}^f \right) \cdot \exp(-K_E \cdot t) \right]}{V_R} + \\
+ \left\{ -\frac{V_o \cdot V_w \cdot K_E \cdot \left[C_{Zn,i} - C_{Zn}^f \right]}{V_R \cdot \left[V_o \cdot K_E + V_R \cdot K_{R1} \right]} \cdot \left[\exp\left(\frac{V_R \cdot K_{R1}}{V_o} \right) - \exp(-K_E \cdot t) \right] - \\
- \frac{V_o \cdot C_{Zn(o)}^f}{V_R} \cdot \left[1 - \exp\left(\frac{V_R \cdot K_{R1}}{V_o} \right) \right] \right\} \\
\text{for} \qquad t < t_f \qquad (16)
\end{aligned}
$$

$$
\begin{aligned}
C_{Zn(R)} = \frac{V_w \cdot C_{Zn,i} - V_w \cdot \left[C_{Zn}^f + \left(C_{Zn,i} - C_{Zn}^f \right) \cdot \exp(-K_E \cdot t) \right]}{V_R} + \\
+ \frac{V_o}{V_R} \cdot \left\{ -\frac{V_w \cdot K_E \cdot \left[C_{Zn,i} - C_{Zn}^f \right]}{V_R \cdot K_{R2} - V_o \cdot K_E} \cdot \left[\exp(-K_E \cdot t) + \right. \right. \\
\left. \left. - \exp\left(\frac{V_R \cdot K_{R2} \cdot (t_f - t)}{V_o} - K_E \cdot t_f \right) \right] - C_{Zn(o)}^f \right\} \\
\text{for} \qquad t \geq t_f \qquad (17)
\end{aligned}
$$

The apparent mass transfer coefficients could be calculated by means of the least squares method (Table 4). The results of fitting of the equations (16) and (17) were presented in Fig. 5.

When deriving of the model equations for extraction-stripping process the variation of phase volumes caused by sampling was neglected. This did not introduce a significant error because the same volumes of both aqueous phases were removed at the same time. Thus, the influence of sampling was much smaller than in the case of extraction process.

In the models presented above the driving force of the process was defined as the difference between the actual and the final concentrations in the appropriate phase. Future discussion must consider models with the driving force expressed as the difference between the actual solute concentration in one phase and the equilibrium concentration in the other phase.
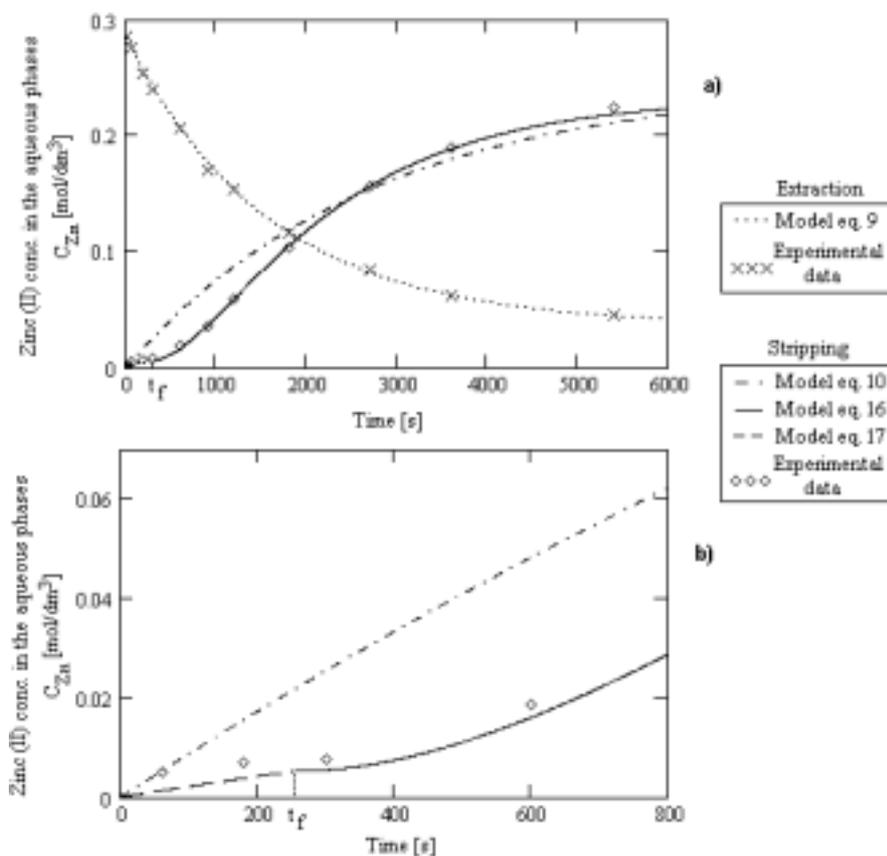
Fig. 5. The variation of the zinc(II) concentration in the feed and stripping phases with time
(Experiment No. 8 – see Table 4): a) full range of the concentration changes; b) first period of
experiment involving the model equation 17

CONCLUSIONS

The experimental results presented and discussed in the work suggested that zinc(II) could be effectively removed from model acidic chloride solutions. However, the final efficiency of the process was limited by relatively small values of the distribution coefficient of zinc(II) between TBP and aqueous solution. This fact implied that at least several modules should be used to achieve a sufficiently low level of zinc(II) in waste water.

In the presented work simplified mass transfer models in hollow fiber modules were explored. In these models the mass transfer coefficients were found by fitting to experimental data. The driving force was defined as the actual and final solute

concentration difference. The presented models properly described the extraction system. In case of two-module system, however, the equation describing the solute concentration variation in the stripping phase showed significant discrepancies with experimental data. The simple model did not include temporary accumulation of solute in the membrane phase. The suggested improved model described the observed phenomena much better. However, it was quite complicated.

The influence of sampling on the extraction process was found to be significant. This was caused by the fact that the volumes of samples were large and the loss of feed phase was not replenished with fresh solution. The loss of feed phase influenced the final solute concentration and thus the driving force was changed.

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Tórz M., Alejski K., Szymanowski J, *Odzysk jonów cynku(ii) z modelowych roztworów kwasu solnego w modułach z włóknami kapilarnymi,* Fizykochemiczne Problemy Mineralurgii, 36, (2002) 101-113 (w jęz. ang.)

W pracy przedstawiono wyniki badań ekstrakcji i reekstrakcji jonów cynku(II) w modułach z włóknami kapilarnymi z modelowych roztworów kwasu solnego o składzie zbliżonym do roztworów potrawiennych pochodzących z ocynkowni stosujących metodę cynkowania ogniowego. Stosowanym ekstrahentem był fosforan tributylu, fazą odbierającą natomiast woda. Wykazano, że cynk(II) może być usuwany z roztworów modelowych w modułach z włóknami wydrążonymi, przy czym o wydajności procesu decydował niski współczynnik podziału cynku(II) między fazę organiczną i wodną. Sformułowano oraz pozytywnie zweryfikowano uproszczony model matematyczny procesu, bazujący na bilansie masowym jonów cynku w fazach wodnych.