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Modelling of the Extraction and Back-Extraction Equilibria of Zinc from Spent Pickling Solutions

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Abstract: This work reports the study of the chemical equilibria of the extraction and back-extraction of zinc from spent pickling solutions. Tributyl phosphate was used as a selective extractant and service water as a back extraction agent. Feed solutions had an average composition of 6.4 M Cl^-, 1.2 M Zn^{+2}, 1.7 M Fe^{+2} and traces of other heavy metals i.e., Pb, Ni, Cu, Mn. Under the experimental conditions the ion $ZnCl_4^{2-}$ was the predominant zinc species in feed. A careful experimental design was performed obtaining the equilibria mathematical models and parameters, $K_{EX} = 0.137\ (mol/L)^{-4}$, and $K_{BEX} = 1.46\ [-]$.

Keywords: Extraction equilibrium, back-extraction, zinc chloride, tributyl phosphate, water

INTRODUCTION

Steel goods are most often coated with zinc to protect their surfaces against atmospheric corrosion. Zinc films are deposited by immersing steel details in molten zinc around 450°C. In order to obtain a high quality zinc film, pre-treatment of the steel surface is required, including several processes, i.e.,

1. degreasing with a hot alkaline solution;
2. rinsing with water;

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3. pickling with HCl 20%;
4. rinsing, and
5. fluxing with zinc and ammonium chlorides.

In the pickling step, hydrochloric acid is consumed during the process, but the concentration of chloride ions does not change. As a result, the spent pickling solution contains zinc and iron, present mainly in the form of iron(II), which form appropriate chlorocomplexes. Typical metallic concentrations lie in the range 20–120 g/L for zinc and 100–130 g/L for iron, whereas the hydrochloric acid concentration lies in the range 1–6 mol/L (1). Besides, the solution may contain other heavy metals such as Pb, Ni, Cu, Mn, etc. at low concentration. Thus, this effluent has a strong hazardous character and needs to be treated before disposal (2).

The spent pickling solution is conventionally processed by the Ruthner process in which the hydrochloric acid is evaporated and granules of iron oxide are formed in a fluidized bed at temperatures above 600°C; however, the presence of zinc in amounts higher than 0.5 g/L disturbs the process (2–6).

The development of a clean process allowing the recovery of zinc under conditions that permit the recovery of electrolytic grade metal would reduce the effluent toxicity, recovering at the same time the component of higher added value.

Solvent extraction and membrane based solvent extraction have received considerable attention and have been proved to be very efficient in the removal of solutes from aqueous solutions (7–12).

With regard to the separation of zinc (II) from solutions containing hydrochloric acid, Cierpiszewski et al. (2002) (5) studied the behavior of different extractants in the separation between zinc and iron concluding that solvating extractants led to better extraction results than other analysed compounds. In fact, tributyl phosphate (TBP) is the most suitable reagent and enables both the extraction (EX) of zinc (II) from HCl solutions and subsequent back extraction (BEX) with water (3, 5, 13). In a previous work it was reported the viability of applying the non dispersive solvent extraction (NDSX) technique to the recovery of zinc(II) from spent pickling solutions using TBP and service water as extraction and back extraction agents respectively (2). In the latter work it was reported that the transport of trace metals such as Mn, Pb, Al, Cr, etc., to the stripping solution was negligible. The homogeneous formation of zinc chloride complexes in hydrochloric acid solutions and the extraction of the formed complexes into organic phases has also been reported (14–16). However, a suitable equilibrium model of the extraction and back-extraction reactions together with their representative equilibrium parameters, has not been reported due to the complex behavior of the system.

The aim of this work is to study the chemical equilibrium of the extraction of zinc(II) from high acidic medium using TBP as extractant, as well as the back-extraction reaction using service water, in order to obtain the mathematical model and parameters needed to design and optimisation of separation processes.

EXPERIMENTAL

Reagents and Analysis

Physicochemical characteristics of the industrial spent pickling solution used in the experimental study are shown in Table 1. Extraction and back-extraction agents were pure grade TBP (tributyl phosphate, MERCK) and service water, respectively.

Experiments conducted at different initial zinc concentrations were carried out by diluting the initial effluent. After dilution of the samples, the concentration of Cl^- ions was adjusted to 2.5 M by addition of HCl, in order to avoid the influence of this variable in the studied system.

The metallic content of the feed solution was analysed using a Perkin Elmer Plasma Emission Spectrometer ICP 400; the iron(II) concentration was determined in a Spectrophotometer Spectroquant Nova 400; the concentration of chloride ions was analysed using an Ion Chromatograph Dionex-120 and finally the zinc content of the samples was determined using Atomic Absorption Spectrophotometry, Perkin Elmer 3110.

Experimental Methodology

Equilibrium experiments were carried out in separatory funnels, using a rotatory SBS stirrer (5–150 rpm), at room temperature (18 ± 2°C). Extraction

Table 1. Physico-chemical analysis of the feed solutions

Property color (pH)	Value (green $\approx$ 0)	
Composition	mg/L	mol/L
Zn	81,760	1.250
Fe	96,000	1.720
Fe + 2	96,000	1.720
Mn	174	3.17E-3
Pb	104.64	5E-4
Al	38.21	1.4E-3
Cr	18.99	0.365
Ni	18.73	3.2E-4
Cd	26.63	5.9E-5
Cu	5.18	8.1E-5
Co	0.14	2.4E-6
Cl-	225,920	6.400

experiments were carried out starting with four different initial concentrations of zinc(II) (1.2, 0.6, 0.3, and 0.15 mol/L) and using different volume ratios of the feed and organic phases: $V_a/V_o = 1/1, 1/2, 1/4$. Table 2 shows the experimental planning. In the extraction analysis 6 batches were prepared with similar conditions (E1-E6) in order to provide enough organic phase to replicate back-extraction experiments. The extractant (undiluted TBP) and the feed solution were mechanically shaken for 1 hour, time enough to reach equilibrium (experimentally determined). After equilibration, the solution was allowed to settle down for phase separation (few minutes). The content of zinc(II) in the aqueous solutions was analysed after complete separation of the phases. The study of zinc(II) stripping was performed in a similar way. Volumes of the loaded extractant (5–20 mL), were equilibrated for 1 hour at the selected organic/stripping ratios $V_o/V_s = 1/1, 1/2, 1/4$, using service water as stripping agent. Table 3 shows the experimental design. After separation of the liquid phases, the concentration of zinc(II) was measured. The contents of zinc(II) in the organic phase were calculated from the mass balance of the respective aqueous solutions.

RESULTS AND DISCUSSION

In the system under study that has a very high chloride concentration, metal ions formed chlorocomplex species according to the following reaction (3, 17).

$$M^{n+} + iCl^{-} \Longleftrightarrow MCl_i^{n-i} \tag{1}$$

where the stoichiometric coefficient "i" can take different values, $i = 1, 2, 3$, and 4 and

$$\beta_i = \frac{\left[MCl_i^{n-i}\right]}{[M^{n+}] \cdot [Cl^{-}]^i} \tag{2}$$

is the equilibrium parameter of the homogeneous reaction responsible of the formation of the complex species.

Table 2. Experimental planning in the study of the extraction of zinc(II) with pure TBP

	V_a/V_o		
$[Zn]_o$	1/1	1/2	1/4
1.2 M experiments	E1-E6	E25-E30	E49-E54
0.6 M experiments	E7-E12	E31-E36	E55-E60
0.3 M experiments	E13-E18	E37-E42	E61-E66
0.15 M experiments	E19-E24	E43-E48	E67-E73

Table 3. Experimental planning of the study of the back-extraction of zinc(II) from loaded TBP (from experiments E1-E24) with service water

	V_a/V_o		
$[Zn]_o$	1/1	1/2	1/4
$ORG^{E1\text{-}E6}$ experiments	B1-B2	B3-B4	B5-B6
$ORG^{E7\text{-}E12}$ experiments	B7-B8	B9-B10	B11-B12
$ORG^{E13\text{-}E18}$ experiments	B13-B14	B15-B16	B17-B18
$ORG^{E19\text{-}E24}$ experiments	B19-B20	B21-B22	B23-B24

Different values of β_i have been reported for the same system by various authors mainly due to its sensitivity to the presence of other ions that can be quantified by the value of the ionic strength and/or the water activity. (3, 5, 6, 13, 15–17) Regel et al. (2001) (3) reported that in a system containing 1 M Fe^{+2}, 0.1 M Zn^{+2}, 5 M Cl^- and 0.6 or 3.6 M HCl, with an ionic strength around 5, over 90% of the zinc was in the form of $ZnCl_4^{2-}$ and only a few percent existed as $ZnCl_3^-$. Similar results were reported by Cierpiszewski et al. (2002) (5), Mishonov et al. (2004) (6), Niemzcewski et al. (2004) (13) and Miesiac et al. (2004), (18) for 5 M Cl^-, (0.5–2.5)M HCl, and very different zinc concentrations (0.0076-1)M. Table 4 summarizes the distribution of zinc(II) chlorocomplexes in aqueous solutions reported in the literature.

An increase in the ionic strength leads to an increase in the formation of complex species with high stoichiometric coefficients of Cl^- (13). In this work, aqueous feed solutions (undiluted and diluted solutions from real spent pickling solutions) contained high concentrations of Cl^- 1.6–6.4 M, of Zn^{+2} 0.15–1.2 M, and of Fe^{+2} 0.2–1.7 M, as well as traces of other heavy metals i.e., Pb, Ni, Cu, Mn, etc. Therefore, according to the literature, the ion $ZnCl_4^{2-}$ can be expected to be the main anionic species of zinc(II) in the aqueous phase.

The extraction of zinc(II) complex species from hydrochloric acid using tributyl phosphate was studied by Morris et al. (1962) (16) and Forrest et al. (1968) (15). For aqueous feed solutions of high acidity, the zinc(II) complex species are extracted according to the following equation

$$H_2ZnCl_4 + 2TBP \Longleftrightarrow \overline{H_2ZnCl_4 2TBP} \tag{3}$$

This reaction is shifted to the right by an increase of the aqueous phase acidity and of the chloride concentration. Under milder conditions, lower HCl concentrations, other reactions become important,

$$HZnCl_3 + 3TBP \Longleftrightarrow \overline{HZnCl_3 3TBP} \tag{4}$$

$$ZnCl_2 + 2TBP \Longleftrightarrow \overline{ZnCl_2 \cdot 2TBP} \tag{5}$$

Table 4. Distribution of zinc(II) chlorocomplexes in acidic solutions

	Feed (M)						
Author	Zn^{+2}	Fe	Cl^-/HCl	$ZnCl_4^{2-}$	$ZnCl_3^-$	$ZnCl_2$	$ZnCl^+$
Regel et al. 2001	0.1	1 M Fe^{+2}	5 M Cl^- 0.6–3.6 M HCl	90%	Few percent	—	—
Mishonov et al. 2004	0.9	—	5.2 M Cl^- 0.5–2.3 M HCl	78%	11%	6%	5%
Cierpiszczewski et al. 2002	7.6E-3	0.627 Fe^{+3}	5 M Cl^-	92%	7%	—	—
Niemczewska et al. 2004	0.3	—	5 M Cl^-	92%	8%	0%	—
Miesiac et al. 2004	0.076	—	5 M Cl^-/2.5 M HCl	94%	6%	—	—
	0.076	—	3.40 M Cl^-/2.5 M HCl	54%	34%	10%	2%
	0.305	—	5 M Cl^-/2.5 M HCl	92%	8%	—	—
	0.305	—	3.8 M Cl^-/2.5’M HCl	49%	36%	0.12%	3%

Reaction 4 becomes important for concentrations of hydrochloric acid in the range 0.8–2.7 M, and is shifted to the right with increasing concentration of protons. Reaction 5 is dominant for concentrations of HCl and Cl^- below 0.8 and 1 M respectively (16).

It is important to notice that those works (15, 16) determined the compositions of the extracted zinc(II) complex species using model dilute solutions. Thus, Eqs. (3)–(5) provide only general trends and boundaries of concentrations because it is very difficult to transfer the data, including the regions of complex existence, from dilute model solutions to the actual spent pickling solutions (5).

Equilibrium Study of the Extraction of Zinc(II)

According to the literature, extraction of zinc(II) with TBP can be described by a general reaction of the form (15, 16),

$$ZnCl_2xHCl + \alpha TBP \Longleftrightarrow \overline{ZnCl_2xHCl\alpha TBP} \tag{6}$$

Considering $ZnCl_4^{2-}$ as the predominant zinc species in the studied conditions, the equilibrium reaction transforms into,

$$ZnCl_22HCl + \alpha TBP \Longleftrightarrow \overline{ZnCl_22HCl\alpha TBP} \tag{7}$$

and the equilibrium parameter can be expressed as,

$$K_{EX} = \frac{\overline{ZnCl_22HCl\alpha TBP}}{ZnCl_22HCl \cdot TBP^{\alpha}} = \frac{\overline{ZnCl_22HClTBP}}{ZnCl_22HCl \cdot (TBP_O - \alpha \cdot ZnCl_22HClTBP)^{\alpha}} = \frac{n}{ZnCl_22HCl} \tag{8}$$

where

$$n = \frac{\overline{ZnCl_2xHClTBP}}{(TBP_O - \alpha \cdot ZnCl_2xHClTBP)^{\alpha}} \tag{9}$$

Figure 1 shows the experimental data of the equilibrium isotherm obtained under the conditions specified in Table 1. Experimental points are the average value of 6 replicate experiments.

The equilibrium isotherm shows that the distribution coefficient varies in the range of the studied conditions. However in order to have an equilibrium expression that can explain the experimental behaviour in the whole range of concentrations, data of Fig. 1 were fitted to the linearized expression of Eq. (8). Plots of "n" in ordinates versus $ZnCl_22HCl$ in abscissas were done for different values of α, obtaining the best fitting for $\alpha = 4$ (Fig. 2). The value of the regression coefficient, $r^2 = 0.98$, is high enough to consider

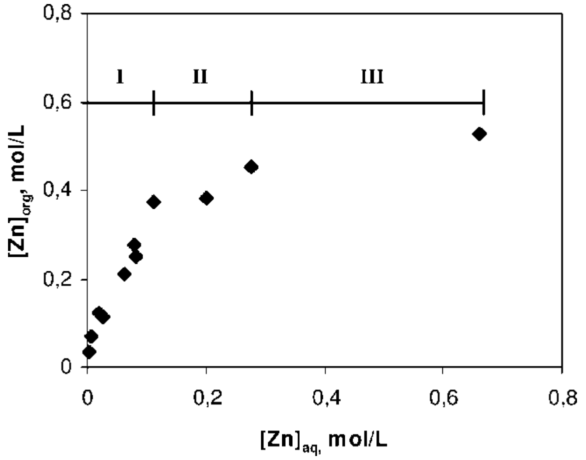

Figure 1. Equilibrium isotherm of zinc(II) extraction. Experiments E1-E76.

that the equilibrium reaction can be described by the stoichiometric equation,

$$ZnCl_2 2HCl + 4TBP \Longleftrightarrow \overline{ZnCl_2 2HCl 4TBP}^{-4} \tag{10}$$

being the value of the equilibrium parameter

$$K_{EX} = 0.137(\text{mol/L})^{-4} \tag{11}$$

No previous references have reported a value of the equilibrium parameter of the extraction of zinc(II) with TBP. However, several authors have reported values of the distribution coefficient (D). According to Fig. 1 the equilibrium

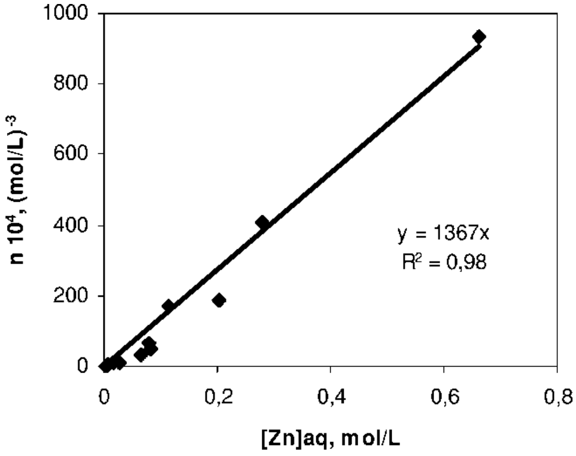

Figure 2. Linear fitting of n·10^4 values to zinc(II) concentration in the aqueous phase.

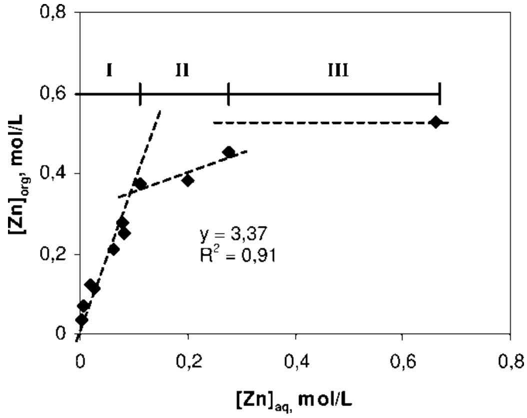

Figure 3. Linear regression of equilibrium data of region I.

isotherm can be divided in three regions with apparent constant distribution coefficients. Therefore, in order to compare the values of the extraction distribution coefficient (D_{EX}) reported in the literature with the results of this work, the value of D_{EX} was calculated in region I (Fig. 3) obtaining,

$$D_{EX} = \frac{[Zn]_{org}}{[Zn]_{aq}} = 3.37;\ r^2 = 0.91 \tag{12}$$

that is very close to the value of 3.33 reported by Niemczewska, et al. (2004), (13), who worked with a feed solution containing 0.31 M Zn^{+2}, 0.55 M H^+, and 5 M Cl^-.

Thus, it is concluded that working with solutions of high acidity and low concentration of zinc ($[Zn]aq \leq 0.15$ mol/L), the extraction equilibrium with TBP can be described by a distribution coefficient, whereas at higher concentrations, it is better described by a chemical equilibrium expression.

Equilibrium Study of the Back-Extraction of Zinc(II)

Figure 4 gives a representation of the experimental data of the back-extraction isotherm; replicate runs were performed for each point. As shown, zinc(II) can be effectively stripped from the organic phase using water as back-extraction agent (3, 5). Equilibrium data were fitted to a linear isotherm of the form,

$$[Zn]_{aq} = 1.46 \cdot [Zn]_{org};\ r^2 = 0.98 \tag{13}$$

being $K_{BEX} = 1.46\ [-]$.

Cierpiszewski et al. (2002), for (5) studied the stripping isotherm of zinc(II) from loaded TBP with water. The organic phase was loaded with a solution that contained 0.9M Zn^{+2} and 1M Fe^{+2} dissolved as chloride salts

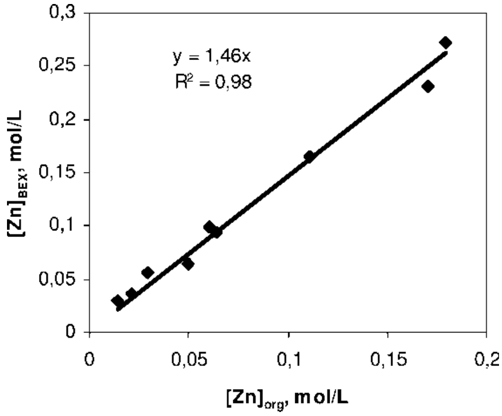

Figure 4. Zinc(II) stripping from loaded undiluted tributyl phosphate with water.

in 3.16M HCl. An approximately linear isotherm with a slope close to 1 was obtained. Regel et al. (2001) (3) determined the stripping isotherm using the loaded organic phase obtained in extraction experiments where the aqueous phase contained 3.6M HCl and the concentration of zinc(II) in the organic phase was below 0.24M. The obtained isotherm was approximately linear and the following equation was reported:

$$[Zn]_{aq} = 1.13 \cdot [Zn]_{org} - 0.32;\ r^2 = 0.99 \tag{14}$$

Thus, the results obtained in this study are in good agreement with those reported by other authors.

Additionally, the equilibrium data of the back extraction study have been compared to the extraction data. The system under study is the reversible reaction of zinc chloride chlorocomplexes with TBP, thus, according to the extraction reaction Eq. (10), the back-extraction equation can be described by

$$\overline{ZnCl_2 2HCl4TBP} \Longleftrightarrow ZnCl_2 2HCl + 4TBP \tag{15}$$

characterized by the equilibrium parameter

$$K'_{BEX} = \frac{ZnCl_2 2HCl \cdot TBP^4}{\overline{ZnCl_2 2HCl4TBP}}$$

$$= \frac{ZnCl_2 2HCl \cdot (TBP_O - 4 \cdot \overline{ZnCl_2 2HCl4TBP})^4}{\overline{ZnCl_2 2HCl4TBP}} = \frac{ZnCl_2 2HCl}{n} \tag{16}$$

Figure 5 gives a representation of the equilibrium concentration of zinc in the back-extraction phase in abscissas versus the parameter "n" in ordinates.

Although the data reported in Fig. 5 could be fitted to a straight line, the value of the slope $m = 0.0108$ leads to a value of the back-extraction

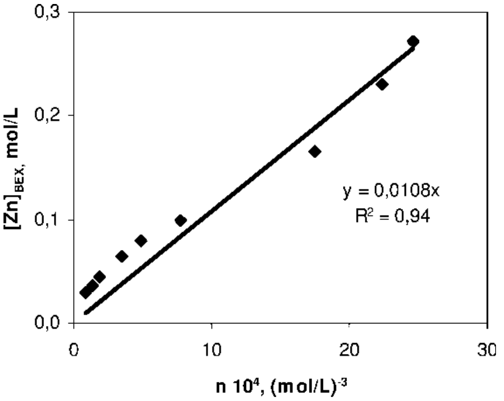

Figure 5. Linear fitting of zinc(II) concentration in the back extraction phase to $n \cdot 10^4$ values.

equilibrium parameter $K'_{BEX} = 108\ (\text{mol/L})^4$ that is considerable higher than the corresponding value obtained from the reverse of the extraction parameter, i.e.,

$$K'_{BEX} = \frac{1}{K_{EX}} = 7.3(\text{mol/L})^4 \qquad (17)$$

The difference could be attributed to the influence of the acidity of the aqueous solutions used in the studied equilibria. Therfore it is concluded that under the experimental conditions the back-extraction equilibrium is best described by a linear isotherm $[Zn]_{aq} = 1.46 \cdot [Zn]_{org}$.

CONCLUSIONS

Taking into account the formation of different chlorocomplex species in the feed solution (high concentration of HCl), $ZnCl_4^{2-}$ was considered as the predominant species in the experimental range of concentrations. Experimental data were satisfactorily fitted to a mathematical model with a stoichiometric coefficient of the extractant $\alpha = 4$, being the equilibrium parameter $K_{EX} = 0.137\ (\text{mol/L})^{-4}$. The back extraction equilibrium has been described by a linear expression of the type $[Zn]_{aq} = 1.46 \cdot [Zn]_{org}$, that is in agreement with related studies reported in literature.

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