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SEPARATION OF ZINC AND CADMIUM IONS FROM SULFATE SOLUTIONS BY ION FLOTATION AND TRANSPORT THROUGH LIQUID MEMBRANES

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An experimental investigation is presented on zinc(II) and cadmium(II) ions separation from aqueous sulfate solutions, containing equimolar mixture on both metal ions, by ion flotation (IF) and transport through polymer inclusion membrane (PIM) processes. The IF experiments from dilute aqueous solutions ($c_{Me} = 1\cdot10^{-5}$ M) with an anionic surfactant (sodium dodecylbenzene sulfonate) and a cationic surfactant (hexadecylpyridinium chloride) are shown. With a cationic surfactant, the flotation separation of Cd/Zn grows with SO_4^{2-} concentration increase. In addition, the selective transport of Zn(II) and Cd(II) from aqueous sulfates source phase ($c_{Me} = 1\cdot10^{-2}$ M) through PIM containing cellulose triacetate (support), o-nitrophenyl pentyl ether (plasticizer) and di(2-ethylheksyl)phosphoric acid D2EHPA (ion carrier) is shown. The transport selectivity of Zn/Cd decreases with Na_2SO_4 concentration increase in source phase. Results obtained are discussed in terms of the stability constants of sulfate complex species for zinc(II) and cadmium(II).

Keywords: ion flotation, polymer inclusion membrane, zinc, cadmium, sulfates

INTRODUCTION

Selective separation of heavy metal ions from industrial and waste aqueous solutions is frequently required in hydrometallurgical processing (Davies 1987). The ion flotation and transport through liquid membrane are methods used for separation of metal ions from aqueous solutions. Ion flotation process has special position for the removal of ions from very dilute solutions, i.e. at the concentration below $1.0\cdot10^{-4}$M. Ion flotation involves the removal of surface inactive ions from aqueous solutions by the introduction of a surfactant and the subsequent passage of gas bubble through the aqueous solution (Lemlich 1972). The transport through liquid membrane is used for

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selective separation and concentration of metal ions from source aqueous phase, in which the concentration of metal ionic species is above $1\cdot10^{-4}$ M.

The selectivity of cationic surfactants foward anions has been established in several ion flotation experiments including chloride and cyanide metal complexes of Zn(II), Cd(II), Hg(II), and Au(III) (Walkowiak and Grieves 1976, Walkowiak et al. 1976 and 1992). Jurkiewicz (1984-85) investigated foam separation of Cd(II) ions by dodecyl sulfate from aqueous solutions in the presence of electrolytes and found a negative influence of these electrolytes on Cd^{2+} foam separation. Also Jurkiewicz (1985) investigated separation of thiocyanate and iodide complexes of cadmium(II) from acidic aqueous solutions with hexadecyltrimethylamonium bromide. The influence of chloride, bromide, iodide and thiocyanate ions on the separation of Zn(II) and Cd(II) ions was also studied by Jurkiewicz (1990). On the other hand, Skrylev el al. (1997) investigated the removal of Zn(II) and Cd(II) in the presence of inorganic ligands from sulfate solutions using fatty acid containing 10 ÷ 14 carbon atoms. The influence of zinc and cadmium concentrations on the effectiveness of flotation removal with potassium oleate was studied by Sinkova (1998); Zn(II) and Cd(II) ions were removed effectively from aqueous solution. Also Scorcelli et al. (Scorcelli et al 1999) studied the removal of Cd(II) using sodium dodecylsulfate as an anionic collector. The best removal (99 %) was obtained for metal cations to collector ratio equal to 1:3. Study of Cd(II) over Zn(II) ions separation in the presence of inorganic ligands by anionic and cationic collectors was conducted by Ulewicz et al. (2001) and Walkowiak (2002).

There are different types of liquid membranes, i.e. bulk (BLM), emulsion (ELM), supported (SLM), as well as polymer inclusion membranes (PIM). PIM provides metal ion transport with high selectivity, as well as easy setup and operation (Sugiura et al. 1987). The casting solution contains cellulose triacetate (support), a membrane plasticizer and an ion-exchange carrier. There are few papers, which deal with Cd(II) removal and Cd(II) over Zn(II) separation by supported liquid membranes. Breembroek et. al. (1998) has reported on cadmium extraction through a flat sheet and hollow fiber supported liquid membranes using tertiary amines as ion carriers. Also Urtiga and Ortiz (1999) studied cadmium removal from aqueous phosphoric acid by SLM. The separation of Cd(II) over Zn(II) from chloride media by a supported liquid membrane using quaternary ammonium salts as ion carriers was reported by Danesi et al. (1983) and Hoh et al. (1990). Li et al. (1997, 1998) studied transport of Cd(II) ions through liquid membrane with tri-n-octylamine, triisooctylamine and sorbitan monooleate. They investigated the effect of KI, KBr and KSCN concentration in the feed solutions on Cd(II) removal. The removal studies of Cd(II) from chloride aqueous solutions by PIMs were published by Hayashita (1996) and Hayashita et al. (1994). Preliminary research of Cd(II) over Znc(II) ions separation by PIM from chloride aqueous solutions was conducted by Kozłowski et al. (2000 and 2002).

The present work deals with the separation of zinc(II) and cadmium(II) metal ionic species from sulfate aqueous solutions containing equimolar mixture of both metals by ion flotation ($c_{Me} = 1\cdot10^{-5}$ M) and transport across polymer inclusion membranes ($c_{Me} = 1\cdot10^{-2}$ M).

EXPERIMENTAL

ION FLOTATION

The ion flotation experiments were carried out in a glass column 45.7 cm in high and 2.4 cm in diameter. The flow rate was maintained at 12 cm^3/min through a sintered sparger of 20–30 µm nominal porosity. The volume of each initial aqueous solution was 100 cm^3, and the temperature was maintained at 22 ± 2 ^{0}C.

The initial aqueous solutions were prepared with double distilled water and the compounds of $ZnSO_4$, $CdSO_4$, Na_2SO_4, H_2SO_4 all from POCh, Gliwice (reagents of analytical grade). The surfactants concentration, i.e. sodium dodecylbenzene sulfonate (DBSNa) and hexadecylpyridinium chloride (CPCl), in the initial solutions were kept constant at $2.0\cdot10^{-4}$ M throughout this investigation. The gamma radioactive isotopes, i.e. Zn-65 and Cd-115m were of sufficiently low specific activity to neglect the effect of carrier concentration (Zn-65: 9.2 GBq/g, Cd-115m: 2.2 GBq/g). These isotopes were from the Atomic Energy Institute (Świerk).

The time dependence of zinc(II) and cadmium(II) concentrations in the bulk solution (*c*) was recorded continuously during each ion flotation run by means of radioactive analytical tracer, and gamma radiation spectrometry, following a procedure described previously (Walkowiak and Ulewicz 1999). A single channel, gamma radiation spectrometer was applied as the detector of radioactive intensity of specific energy. The c versus time curves enabled the calculation of the percent removal (*M*):

$$M = [1 - (c_r/c_i)] \cdot 100\% \qquad (1)$$

where c_i is the initial metal concentration, c_r is the metal concentration in residual solution after foam ceased. Also selectivity coefficients (*S*) of ion metal *Me1* over *Me2* were calculated:

$$S_{Me1/Me2} = M_{Me1}/M_{Me2}\,. \qquad (2)$$

POLYME R INCLUSION MEBRANE

A solution of cellulose triacetate (as the support), di(2-ethylheksyl)phosphoric acid D2EHPA (as the ion carrier), and *o*-nitrophenyl pentyl ether (as the plasticizer) in dichloromethane was prepared. A portion of this organic solution was poured into a membrane mold comprised of a 9.0 cm glass ring attached to a plate glass with cellulose triacetate-dichloromethane glue. Dichloromethane, as the organic solvent, was allowed to evaporate overnight and the resultant polymer inclusion membrane was separated from the glass plate by immersion in water. The membrane was stored in water. The concentration of D2EHPA was 1.50 M based on plasticizer.

Transport experiments were conducted in a permeation cell in which the membrane film (at surface area of 4.9 cm^3) was tightly clamped between two cell compartments. Both, i.e. source and receiving aqueous phases (45 cm^3 each), were mechanically stirred at 600 rpm. The receiving phase was 1.0 M aqueous solution of sulfuric acid. The PIM transport experiments were carried out at the same temperature as IF runs. Small samples (0.1 cm^3 each) of the aqueous receiving phase were removed periodically via a sampling port with a syringe and analyzed to determine zinc and cadmium concentrations by atomic absorption spectroscopy method (AAS Spectrometer, Solaar 939, Unicam).

The inorganic chemicals, their purity, and producer were the same as in ion flotation experiments. The organic chemicals, i.e. cellulose triacetate (Fluka), di(2-ethylheksyl)phosphoric acid (Fluka), *o*-nitrophenyl pentyl ether (Fluka), and dichloromethane (POCh) were of analytical reagent grade. The percent removal and selectivity coefficients were calculated according to equations (1) and (2) in which c_i and c_r are the initial and residual metal concentrations in the aqueous source phase.

RESULTS AND DISCUSSION

First, the competitive ion flotations were studied to determine the sulfates influence on flotation kinetics of Zn(II) and Cd(II) ions with an anionic and a cationic surfactant (Fig. 1).

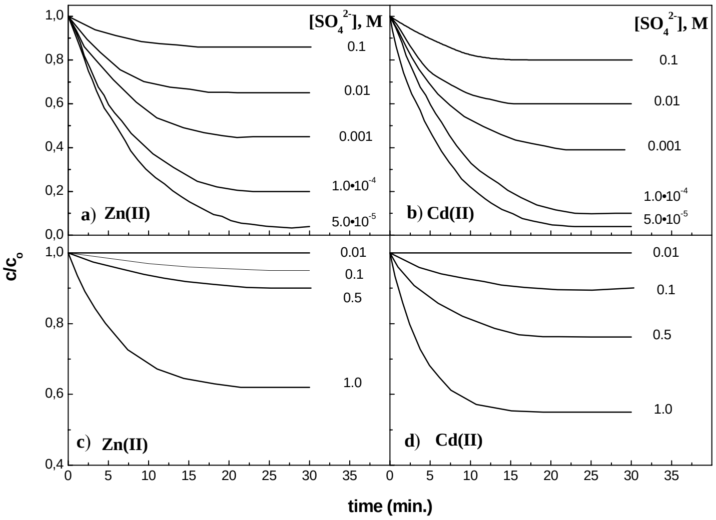

Fig. 1. Rate curves of zinc(II) and cadmium(II) concentration vs. time from aqueous solution containing equimolar mixture of both metals ($c_{Me} = 1.0 \cdot 10^{-5}$ M) in presence of sulfates with DBSNa (a, b) and with CPCl (c, b), $c_{surf.} = 2.0 \cdot 10^{-4}$ M

According to Figs 1a and 1b, with the increase of sulfates concentration the rate and removal of both floated ions, i.e. zinc(II) and cadmium(II), with DBSNa decreases. At concentration of sulfates equal to $5\,10^{-5}$ M, percent removal of zinc and cadmium reaches 97 %, and 99 %, respectively, whereas at concentration of sulfates equal to 0.10 M, percent removal of zinc and cadmium decreasses to 12 %, and 19 %, respectively. Separation of Cd/Zn does not occurred because percent removal of both metal ions is comparable. Contrary influence of sulfates concentration on competitive ion flotation of zinc and cadmium is observed using a cationic surfactant, i.e. CPCl (Figs 1c and 1d). In this case both flotation rate and percent removal of Zn(II) and Cd(II) increase with SO_4^{2-} concentration increase but the separation of cadmium over zinc is low (Fig. 2). With sulfates concentration increase the percent removal of cadmium(II) grows faster than zinc(II).

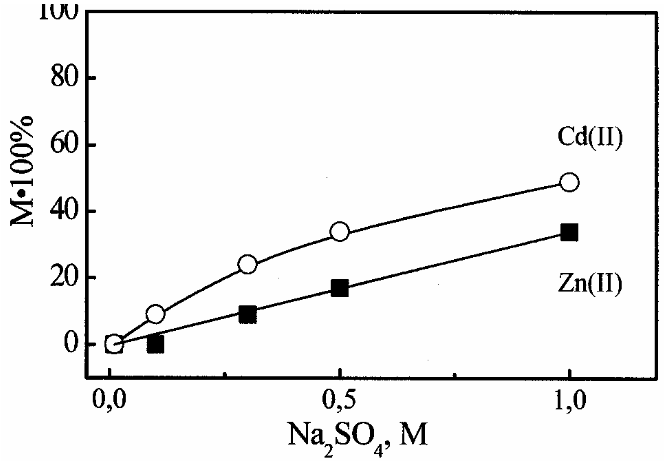

Fig. 2. Percent removal of zinc(II) and cadmium(II) in competitive ion flotation with CPCl from aqueous solutions containing equimolar mixture of both metals vs. sulfates concentration. Experimental conditions as in Fig.1

Next, the competitive transport of zinc(II) and cadmium(II) ions from aqueous source phase ($c_{Me} = 1.0\cdot10^{-2}$ M) containing sulfates through polymer inclusion membranes with D2EHPA into receiving aqueous phase was investigated. The kinetics of zinc(II) and cadmium(II) ions transport through PIM from aqueous source phase containing equimolar mixture of both metals is shown in Fig. 3.

Comparison of both processes kinetics, i.e. IF and transport across PIM (Figs 1 and 3) shows that transport through polymer inclusion membranes is much slower than ion flotation of adequate metals, and it takes 24 hours to remove more than 86 % of zinc from source aqueous phase. In contrast to IF, the PIM transport of zinc(II) is faster than cadmium(II). The dependence of percent removal of Zn(II) and Cd(II) ions from a source aqueous phase as a function of Na_2SO_4 concentration is presented in Fig. 4.

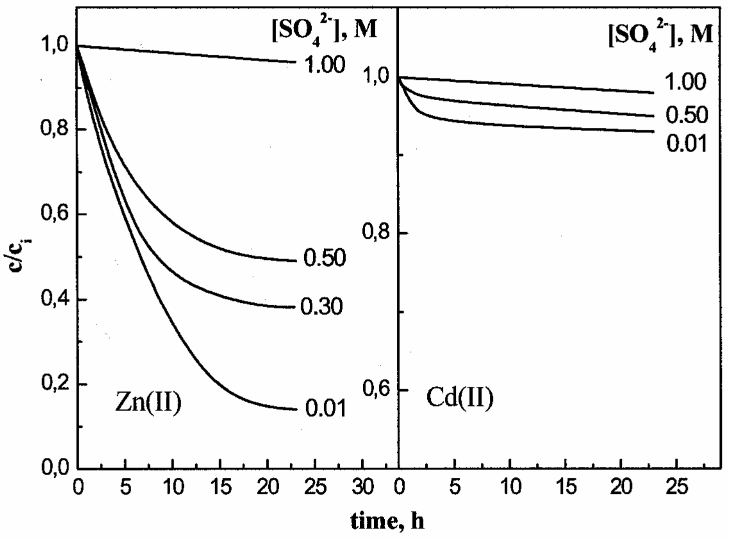

Fig. 3. Kinetics of zinc(II) and cadmium(II) transport through PIM with D2EHPA from source aqueous phase vs. Na_2SO_4 concentrations, ($c_{Me} = 1.0 \cdot 10^{-2}$ M), pH =4.0. Membrane: 2.6 cm^3 ONPOE / 1g CTA, 1.5 M D2EHPA

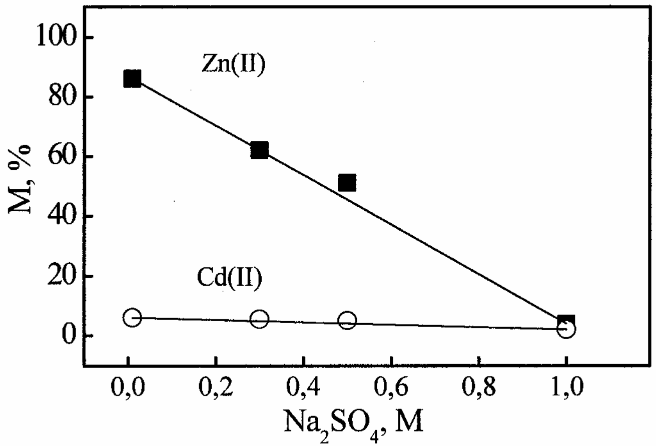

Fig. 4. Percent removal of zinc(II) and cadmium(II) ions through PIM from source aqueous phase containing equimolar mixture of both metals ($c_{Me} = 1{,}0 \cdot 10^{-2}$ M) vs. Na_2SO_4 concentration in source phase. Experimental conditions as in Fig.3

Fig. 4 shows that the percent removal of zinc ions decreases with Na_2SO_4 concentration increase and at SO_4^{2-} concentration equal to 1.0 M transport through PIM does not occur. This causes that separation coefficient of Zn/Cd is the highest for low concentrations of sulfates, i.e. 0.010 M. The separation coefficients of zinc(II) over cadmium(II) in polymer inclusion membrane processes at 0.01, 0.3, 0.5 and

1.0 M concentrations of sulfates, reache 14.3; 11.7; 10.2 and 2.0, respectively. Wherease the separation coefficients of cadmium(II) over zinc(II) in ion flotation were between 1.2 ÷ 1.6. The dependence of separation coefficients of metal ions versus SO_4^{2-} concentration for both processes is different. For ion flotation, $S_{Cd/Zn}$ values are insignificantly growing with SO_4^{2-} concentration increase while coefficients values of $S_{Zn/Cd}$ for polymer inclusion membranes are decreasing with Na_2SO_4 concentration increase. To explain IF and PIM experimental results, the contributions of formed complex species of zinc(II) and cadmium(II) in aqueous sulfates solution were calculated. The percent molar contributions of sulfates complex species for those metals versus SO_4^{2-} concentration are presented in Fig. 5. α_0 is the percent molar contribution of uncomplexed cations (i.e. Zn^{2+} and Cd^{2+}); α_1, α_2, α_3 and α_4 are percent molar contributions of complexed ions with 1, 2, 3, and 4 ligands, respectively. Values of stability constants for Zn(II) + SO_4^{2-} and Cd(II) + SO_4^{2-} systems were taken from Stability Constants of Metal-Ion Complexes (1982).

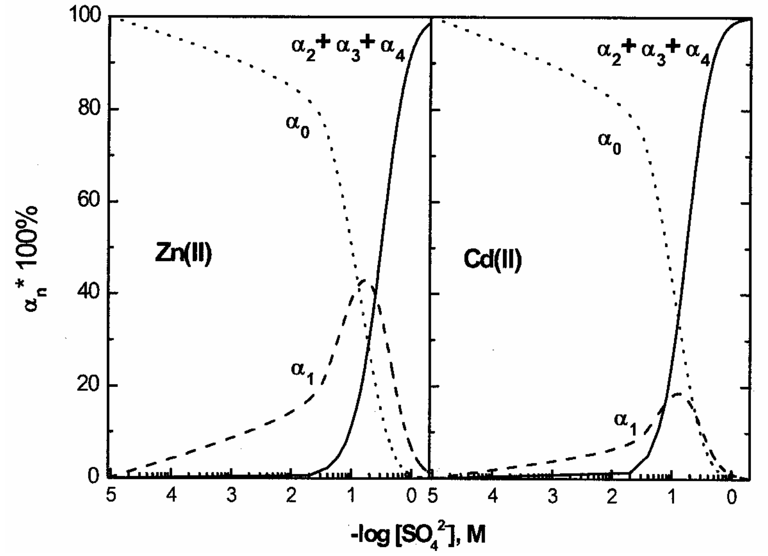

Fig. 5. Percent molar contributions ($\alpha_n \cdot 100\%$) of sulfate complex species for zinc(II) and cadmium(II) vs. sulfates concentration

In the presence of sulfates at concentration range from $5 \cdot 10^{-5}$ to 0.10 M zinc(II) and cadmium(II) exist as cations in 100 - 50 % and 100 - 43 %, respectively. But percent removal of both metals in ion flotation process with an anionic surfactant is much lower than values of α_0 molar contributions (Fig. 1). This is caused by interfering influence of sodium cations on ion flotation process. Percent removal of zinc(II) in IF process with a cationic surfactant increases in smaller part than cadmium(II) with sulfates concentration increase (Fig. 2). This correlates with low percent molar contribution of Zn(II) anionic forms (i.e. $\alpha_2 + \alpha_3 + \alpha_4$) which reaches 89.6 % and 96.7 % for Zn(II) and Cd(II), respectively (Fig. 6). On the other hand, Cd(II) and Zn(II) percent removal is much lower than values of $\alpha_2 + \alpha_3 + \alpha_4$ molar contributions and reaches 55 % and 38 % for 1.0 M Na_2SO_4, respectively (Fig. 2).

In case of polymer inclusion membrane transport, which is significantly slower process (in comparison with IF), the main factor influencing on the Zn/Cd separation is different affinity of D2EHPA to Zn(II) and Cd(II) in experimental conditions (Binghua et al. 1996). The separation of Zn/Cd ratio decreases with Na_2SO_4 concentration increase.

CONCLUSION

Zinc(II) and cadmium(II) can be effectively removed from aqueous sulfate solutions in hydrometallurgical processes of ion flotation and transport across polymer inclusion membrane. The removal of zinc(II) and cadmium(II) ions by ion flotation with an anionic surfactant (DBSNa) decreases with SO_4^{2-} concentration increase. Separation of Cd/Zn does not occurr because percent removal of both metal ions is comparable The ion flotation with a cationic surfactant (CPCl) allow to partial separation of both metal ions, i.e. cadmium(II) over zinc(II), from dilute aqueous solutions ($c_{Me} = 1\cdot10^{-5}$ M). The removal of zinc(II) and cadmium(II) ions by ion flotation with CPCl increases with SO_4^{2-} concentration increase. Competitive transport of zinc(II) and cadmium(II) from an aqueous sulfate source phase ($c_{Me} = 1\cdot10^{-2}$ M) through polymer inclusion membranes containing di(2-ethylheksyl) phosphoric acid (D2EHPA) as ion carrier into aqueous sulfuric acid solution enables separation of zinc(II) over cadmium(II). The selectivity coefficients of Zn/Cd for PIM decreases with Na_2SO_4 concentration increase in source phase. In both studied separation methods, i.e. IF with DBSNa and transport across PIM, zinc(II) and cadmium(II) are removed from an aqueous sulfuric solutions in the form of Zn^{2+} and Cd^{2+}, respectively. On the other hand, in IF with CPCl zinc(II) and cadmium(II) are removed from solutions in the of forms anionic complexes. Results are discussed in terms of the sulfate complex species stability for zinc(II) and cadmium(II).

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Zbadano selektywność procesów wydzielania jonów cynku(II) i kadmu(II) z wodnych roztworów siarczanowych zawierających równomolową mieszaninę obu metali za pomocą flotacji jonowej (IF) i transportu przez polimerowe membrany inkluzyjne (PIM). Pokazano wyniki IF z rozcieńczonych roztworów wodnych ($c_{Me}=1,0 \cdot 10^{-5}$ M) za pomocą kolektora anionowego (dodecylobenzenosulfonian sodu) i kationowego (chlorek hexadecylopirydyniowy). Wykazano, że dla kolektora anionowego ze wzrostem stężenia siarczanów wydzielanie jonów cynku i kadmu maleje, a separacja obu metali nie jest możliwa ponieważ jony obu metali wydzielane są w porównywalnym stopniu. Natomiast w przypadku kolektora kationowego ze wzrostem stężenia siarczanów w roztworze wydzielanie jonów cynku i kadmu wzrasta ale wzrost selektywności flotacji Cd/Zn nie jest duży. W pracy prezentowane są również wyniki transportu Zn(II) i Cd(II) z wodnej fazy zasilającej ($c_{Me}=1,0 \cdot 10^{-2}$ M) przez PIM zbudowaną z trójoctanu celulozy (nośnik), eteru o-nitrofenylopentylowego (pastyfikator) i kwasu di(2-ethyloheksylo) fosforowego (przenośnik jonów). Selektywność transportu Zn/Cd przez PIM malała wraz ze wzrostem stężenia Na_2SO_4 w fazie zasilającej. Wyniki przedyskutowano w świetle zakresów trwałości poszczególnych form jonów kompleksowych Zn(II) i Cd(II) z siarczanami. W procesie IF z CPCl wydzielano jony cynku i kadmu w formie anionów, natomiast w procesie IF z DBSNa i PIM wydzielano kationowe formy obu metali.