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Removal of Heavy Metals from Aqueous Solutions by Precipitation-Filtration Using Novel Organo-Phosphorus Ligands

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Abstract: An organophosphorus mixture of sodium mono- and di-(n-hexa-decyl) phosphinate was synthesized and purified, and then used as a ligand to remove heavy metals by precipitation from aqueous nitrate, chloride, and sulfate solutions. The new ligand offers more advantages over the previously studied sodium dioctyl and dodecyl phosphinates. The sodium form of the mono- and di-(n-hexa-decyl) phosphinate has a much lower solubility in water, which contributed to much lower back contamination and much lower loss of the reagent, even when excess amount of ligand was employed. Moreover, an excess amount of the ligand did not alter the filtration characteristics of the resultant precipitate. The heavy metals: lead, cadmium, mercury, cobalt, and nickel were precipitated with the sodium mono- and di-(n-hexa-decyl) phosphinate, NaL, in the form of $PbL_{2(s)}$, $CdL_{2(s)}$, $HgL_{2(s)}$, $CoL_{2(s)}$, and $NiL_{2(s)}$. In the absence of free acid in the feed, a maximum removal of each metal corresponded to the stoichiometric ratio. The residual concentrations of each of the metals at the optimum conditions were measured for the different media and found to be lower than 10 ppb, lower than the acceptable levels for most regions. Lead, as a model heavy metal, was studied in more detail. Adding an acid to the feed solution reduced the removal of lead as some of the phosphinate ligand was converted to the acid form. The presence of chloride and sulfate in the feed solution; up to mole ratios to lead of 5000, and of calcium in the feed solution; up to mole ratio to lead of 200, had no effect on the removal of lead. The ligand was more selective to lead than the other four metals, and the selectivity was in the order Pb > Cd > Co & Ni > Hg. The ligand was regenerated up to 99.99% and the metals were recovered in 100 times more concentrated aqueous solutions.

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INTRODUCTION

Wastewaters encompass many types of effluents; including industrial waste, sewage effluent, mining waste, etc. Industrial wastewater can be contaminated with a wide variety of toxic chemicals, with heavy metals and organics being the most significant and the potentially troublesome contaminants. The entry of heavy metals into the food chain or into the water supply poses high risk. Battery industry, electroplating industry, and smelting operations are the main sources of heavy metal contamination. There are many industrial processes that produce acidic wastewater streams containing low, yet significant concentrations of heavy metals. These contaminants could lead to adverse effects on human and animal health. The increasing need for removal of heavy metals from industrial wastewaters and the stringent guidelines on maximum allowable heavy metal concentrations motivate continuous search for new effective separation processes. Industries are generally required to adhere to local discharge limits on wastewater concentrations. The limits vary from region to region, but generally are in the range of 5 ppb to 5 ppm (1). The heavy metals of particular concern when considering treatment of industrial wastewater are mercury, cadmium, lead, zinc, copper, chromium, cobalt, and nickel. They typically exist in industrial wastewater streams among other soluble metals, acids and anions; including calcium, chlorides, sulfates, etc.

Many separation and concentration processes are potentially applicable to metal removal from industrial wastewater. Most of these processes are well-developed and many have been commercially applied on a large scale. In general, to be removed from wastewater, metals must be precipitated or otherwise attached to an insoluble material through adsorption or ion exchange. The available methods used for removal of heavy metals include: precipitation, ion exchange, membrane separations, solvent extraction, foam flotation, adsorption, and biosorption processes, etc. Peters et al. (1) and Patterson (2), described various methods employed for heavy metal removal in industry and discussed the advantages and the disadvantages of their application. Precipitation processes are widely used to remove metal ions from solution. The precipitation of metal hydroxides is, by far, the most commonly used technique. Carbonate precipitation, on the other hand, did not reduce the residual concentration to the same extent as the hydroxide precipitation (3). Sulfide precipitation has been demonstrated to be an effective alternative to hydroxide precipitation for removing different heavy metals (4). In general, the disadvantages of the precipitation process are the large chemical consumption and the poor settling and filtering properties of the sludge. In addition, the residual metal concentrations do not always meet minimum acceptable standards. Moreover, exceeding the optimum dosage of the reagent may in fact result in re-dissolving the precipitate (9).

Organic-based ligands, and macroligands, are widely used for heavy metal removal. The heavy metal ions are too small to be retained by filters, so they are first associated with the ligand to form a water-soluble complex or a precipitate. Ultrafiltration is then employed for the separation of the water-soluble complex, while conventional filtration is used for the separation of the precipitate. Removal of heavy metals by complexation-ultrafiltration has been investigated by many researchers (5,6). In this method, the two processes; the complexation and the ultrafiltration, must be optimized simultaneously. Many companies have marketed different ligands, reagents, for the precipitation of heavy metals from wastewater. However, the available reagents lack sufficient ability to strongly bind the metals, and they fail to provide long-term stability as ligand-metal precipitates under a variety of moderate conditions (7,8). In addition, high ligand to metal dosage is typically required. Husein (9) used different compounds containing the sodium carboxylate group to precipitate lead from aqueous solutions. Among these, sodium caprate produced the best filtration properties with the highest percentage removal (9,10). About 99% of sodium caprate was regenerated. Esalah et al. (11,12) and Vera et al. (13), on the other hand, have used sodium di-(n-octyl)- and sodium di-(n-dodecyl) phosphinates to precipitate lead, cadmium, and zinc from different aqueous solutions at different conditions. Low residual concentrations of the metals, lower than accepted levels in most regions, were obtained and the ligand was regenerated to more than 99.9%. Nonetheless, the residual concentration of phosphinate ligands posed an environmental concern.

In the current work, and in order to reduce the solubility of the metal-organophosphorus complex, and hence the residual concentration of the metal as well as the ligand, novel organophosphorus, namely; sodium mono and di-(n-hexa-decyl) phosphinate compound were used. This ligand has a chain length twice of that used by Esalah et al. (9,11). The selectivity of the ligand for heavy metals over calcium, and its ability to remove heavy metals from different aqueous media, which mimic industrial wastewater was demonstrated.

EXPERIMENTAL WORK

The ligand, a mixture of sodium, mono,- and di-(n-hexadecyl) phosphinate was synthesized and purified following the procedure presented in the work of Esalah (14), for Sodium di-(n-octyl) phosphinate. The experiments were carried out at 22 ± 1°C. Samples of 25 ml volume at certain pH containing the heavy metal, calcium, and nitrate, sulfate, or chloride concentrations were treated with specified masses of the ligand, which were added in the solid form. The pH of the solution was adjusted prior to adding the ligand by addition of HNO_3. All samples were shaken in tightly closed jars and then left to reach equilibrium. Equilibrium condition was verified by analyzing the system at a time interval between 5 min. and one week. At equilibrium, two phases existed; a white precipitate and a clear aqueous phase. The two phases were separated using Fisher brand fast flow filter papers. After phase separation, the equilibrium pH was measured using AB15 plus pH meter (Accumet, Fisher Scientific). The concentrations of the different metals were measured using inductively coupled plasma (ICP) (IRIS Intrepid II XDL, Thermo-Instruments Canada Inc.). A metal stock standard was prepared by dissolving a specified mass of the corresponding metal nitrate in deionized water. A standard containing 1 ppm was prepared by diluting 40 ppm of the stock standard in deionized water. A typical standard error in the metal concentration measurement was ±0.001 ppm. The concentration of the ligand in the aqueous phase was measured by a TOC-V_E total carbon analyzer (Shimadzu). The removal of heavy metals from a feed containing a mixture of heavy metals was evaluated following the same procedure. A feed solution containing lead, cadmium, mercury, cobalt, and nickel at about equal concentrations, 40 ppm, was treated with different masses of the ligand in order to achieve different ligand to heavy metal mole ratios.

The regeneration of the ligand and the stripping of the metals were performed by treating the metal-ligand precipitate with 10 ml of 3 N HNO_3 in one approach, and with 10 ml of 3 N NaOH in another approach. The addition of the acid or the base was followed by agitation for 3 h. The sodium or the acid form of the ligand precipitated at the bottom, while the aqueous phase contained the stripped metal. The aqueous phase was analyzed for its content of the heavy metal and the ligand using ICP and TOC; respectively.

RESULTS AND DISCUSSION

Equilibrium experiments showed that 5 min. is sufficient to achieve equilibrium in all the samples, except the ones involving mercury. The data are presented on the figures in terms of the percentage removal of the metal, and equilibrium pH versus each of the independent variables. The percentage removals of the metal; R_M, is defined as

$$R_M = \frac{C_M^o - C_M}{C_M^o} \times 100 \tag{1}$$

where, C_M^o is the initial concentration of metal, and C_M is the equilibrium concentration of metal in the treated solution.

Removal of Lead

Effect of Mole Ratio of Ligand to Lead

The effect of the mole ratio of ligand to lead was investigated by treating a feed solution containing 0.19 mM (40 ppm) lead at initial pH, pH°, of 5.3, with different mole ratios of the ligand to lead. The percentage removal of lead and the equilibrium pH are plotted in Fig. 1.

The removal of lead increased linearly up to a value of the mole ratio of two, for which the highest percentage removal corresponded to the lowest amount of ligand required. The equilibrium concentration of lead at this point was less than 10 ppb, which is well below the accepted level of lead in most of the regions. At higher mole ratios, an increase in the mole ratio had no noticeable effect on the percentage removal of lead or loss of the reagent, since the sodium form of the ligand has very low solubility in water. Based on these results, the stoichiometry of the overall precipitation reaction is believed to be:

$$Pb^{2+} + 2L^- \leftrightarrow PbL_{2_{(s)}} \tag{R1}$$

Aqueous solutions of lead salts are typically acidic. The decrease in the pH results from the hydrolysis of Pb^{2+} following reactions R2 and R3 below.

$$H_2O \leftrightarrow H^+ + OH^- \tag{R2}$$

$$Pb^{2+} + OH^- \leftrightarrow PbOH^+ \tag{R3}$$

Figure 1 shows that the equilibrium pH of the solution has increased when the mole ratio of the ligand to lead increased up to a mole ratio of 2, the small increase observed in the pH is believed to be due to reversing reaction R3 as a result of lead precipitation. The sudden increase in pH at mole ratios above 2 is believed to be due to the formation of the acid form of the ligand:

$$H^+ + L^- \leftrightarrow HL_{(s)} \tag{R4}$$

The trends in Fig. 1 confirm that, even though, phosphinates with long alkyl length are surfactants by nature (15), micellization, if any, did not

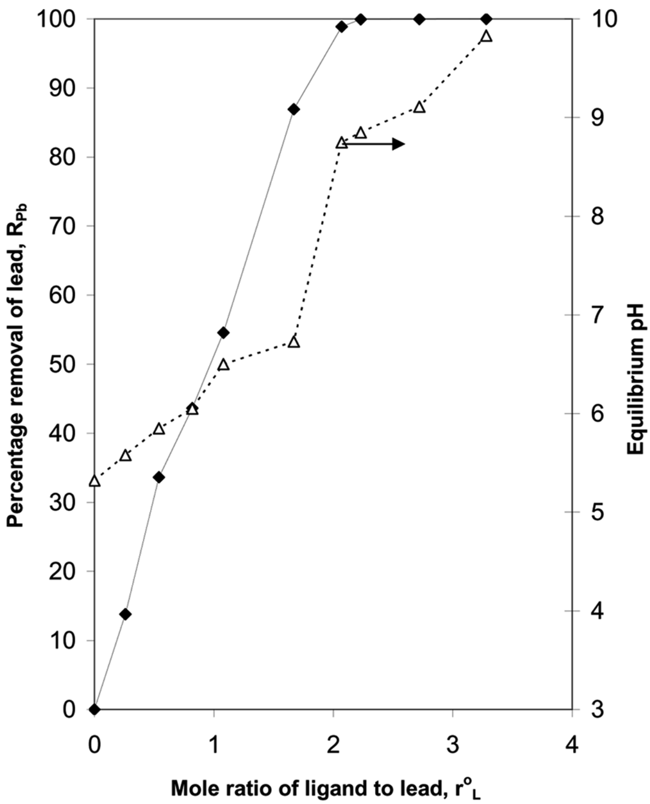

Figure 1. Effect of mole ratio of ligand to lead on the removal of lead and equilibrium pH. $C^o_{Pb} = 0.19$ mM (40 ppm), $pH^o = 5.3$.

interfere with the precipitation reaction. It is worth noting that any colloidal form of the metal could be easily detected by the ICP.

Effect of pH

The effect of decreasing the pH of the feed, pH^o, from 5.3 to 0.51 on the removal of lead and on the equilibrium pH was investigated. The pH of the feed was adjusted by adding HNO_3 solution. The concentration of lead in the feed was fixed at 0.23 mM, and the mole ratio of the ligand, L^-, to lead was fixed at 2.0.

Figure 2 shows that decreasing the pH of the feed, pH^o, decreased the percentage removal of lead and the equilibrium pH. The decrease in the percentage removal of lead is attributed to a competition between lead

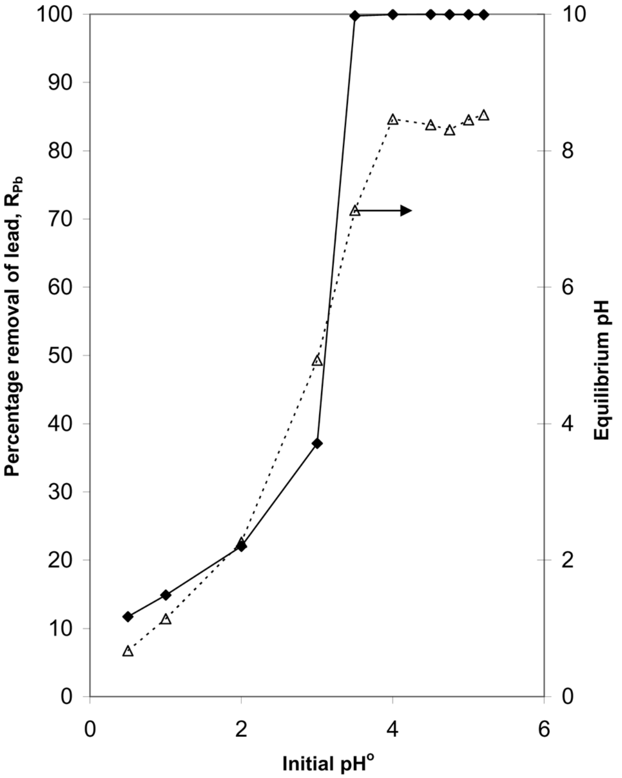

Figure 2. Effect of pH on the removal of lead, $C^o_{Pb} = 0.23\,mM$, $r^o_L = 2.0$.

and hydrogen ions following reactions R1 and R4 above. The decrease in the equilibrium pH is caused by the free acid left in the solution. The equilibrium pH was higher than the pH of the feed due to the precipitation of the acid form of the ligand as in reaction R4, and the formation of the hydroxo-complexation as in reaction R3. The fact that the acid form of the ligand mostly precipitates is supported by the very low residual concentration, less than 3 ppm, of the ligand in the solution at equilibrium.

The effect of initial pH was also investigated by comparing two sets of feed samples having the same lead concentration, 0.23 mM, and pH values of 5.3 and 3.1 at different mole ratios of precipitating agent to lead. No acid was added to the first set, and the initial pH of the other one was adjusted by adding nitric acid. The mole ratio of the precipitating agent to lead, r^o_L, was varied from 0 to 6.4. The percentage removal of lead, and the equilibrium pH plotted versus r^o_L are presented in Fig. 3.

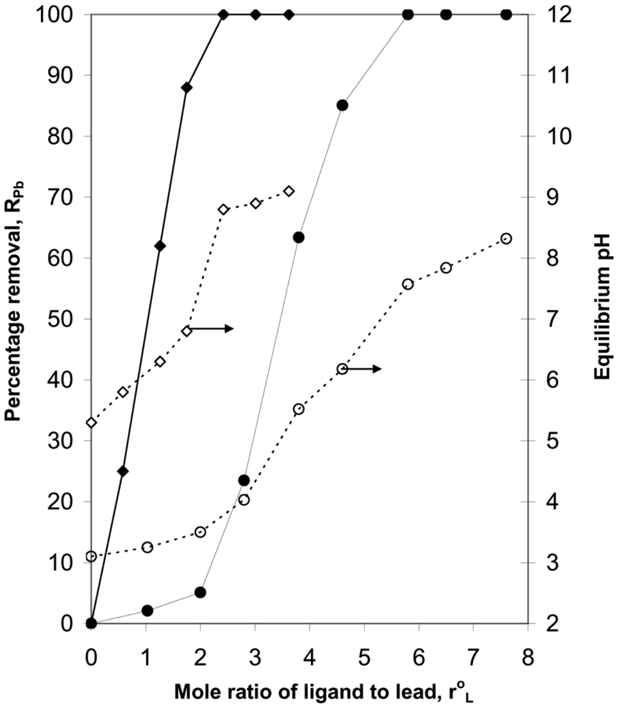

Figure 3. Effect of mole ratio of ligand to lead on removal of lead and equilibrium pH at (◆,◇) pH° = 5.3 and (●, ○) pH° = 3.1; C^o_{Pb} = 0.23 mM.

For the first set of samples (pH° = 5.3), because of low hydrogen concentration, only lead precipitated as $PbL_{2_{(s)}}$ following reaction R1. The highest percentage removal of lead was obtained at $r^o_L = 2.0$. Addition of acid to the other set, pH° = 3.1, reduced the removal of lead due to the precipitation of $HL_{(s)}$ following reaction R4. The highest percentage removal of lead for each set was obtained at different r^o_L. The lower the pH, the higher the value of r^o_L required to achieve the same removal. When the initial pH in not very low, the following formula proposed by Esalah et al.(9) can be used to estimate the required amount of ligand for maximum removal of lead

$$r^o_L = 2 + \frac{10^{-pH^o}}{C^o_{Pb}} \tag{2}$$

where, pH^o is the initial pH and C^o_{Pb} is the initial concentration of lead. From this equation, for $C^o_{Pb} = 0.23\,\text{mM}$ and $pH^o = 3.1$, the mole ratio of ligand to lead required for maximum removal is 5.45 which is in agreement with the experimentally estimated value. For very low initial pH, $pH^o < 0.5$, a large amount of ligand is required and in this case adjusting the pH of the system is recommended.

Effect of Chlorides and Sulfates

Chloride and sulfate typically exist in most water streams. They tend to form soluble chloride-metal and sulfate-metal complexes, which increase the solubility of heavy metals. The effect of the presence of chloride and sulfate on the removal of lead was investigated by adding sodium chloride and sodium sulfate, respectively, to the feed solution. A feed solution of 0.19 mM, 40 ppm, lead, containing chloride, sulfate or nitrate, was treated with the ligand at a mole ratio to lead of two; $r_L^o = 2.0$. The concentration of chloride, sulfate, or nitrate was increased from 0 to 1.0 M. Nitrate was employed to account for the effect of ionic strength, since nitrate does not participate in any complexes. The percentage removal of lead, and the equilibrium pH are plotted versus the concentration of chloride and sulfate in Fig. 4.

Figure 4 shows no significant difference in the percentage removal or in the equilibrium pH in these three systems. This attests to the ability of the organophosphorus ligand to form more stable complexes with the heavy metal than any metal-anion complexes that typically exist in wastewater stream.

Effect of Calcium

Effective removal methods must be selective for the target heavy metal against alkaline earth metals. Calcium is one of the metals that exists in almost all water streams. When a given ligand has appreciable affinity to calcium, or other alkaline earth metals, calcium competes with the target heavy metal and consumes the ligand according to the following reaction:

$$Ca^{2+} + 2L^- \leftrightarrow CaL_{2_{(s)}} \qquad \text{(R5)}$$

The effect of calcium concentration on the removal of lead was investigated by addition of calcium nitrate to a feed solution containing 0.19 mM (40 ppm) lead. The mole ratio of the ligand to lead was maintained at 2.0. The mole ratio of calcium to lead, R_{Ca}^o, was increased from 0 to 200. The percentage removal of lead and the equilibrium pH are presented in Fig. 5 as a function of the mole ratio of calcium to lead.

The results show that the presence of calcium in the feed, in the range of mole ratio of calcium to lead considered, had no effect on the removal

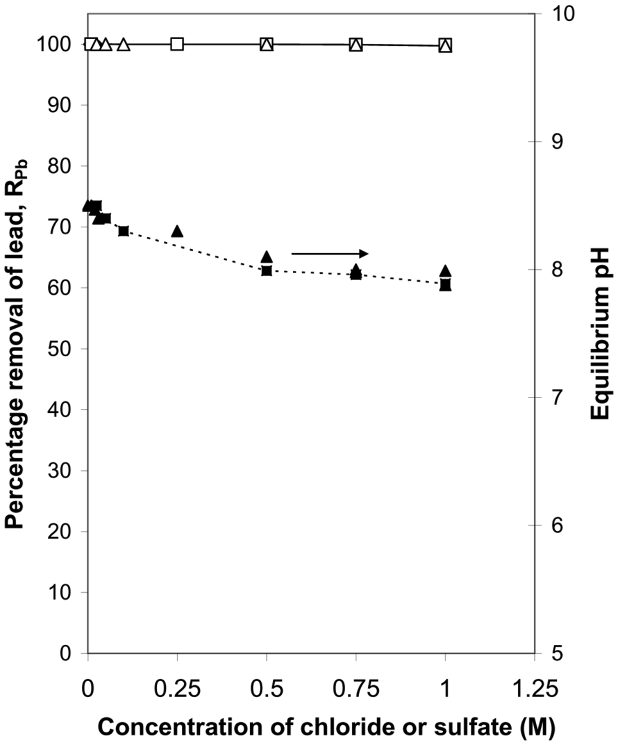

Figure 4. Effect of (□,■) chloride and (△,▲) sulfate concentration on the removal of lead, $r_L^o = 2.0$, $C_{Pb}^o = 0.19$ mM (40 ppm).

of lead. The ligand showed no affinity to calcium in presence of lead. This could be attributed to the acidity and electronegativity of the cations, where Pb^{2+} is more acidic and electronegative than Ca^{2+}. The electronegativity of lead and calcium are 1.8 and 1.0, respectively (14). This again, shows that the employed ligand is an excellent reagent for heavy metals removal from different types of wastewater streams.

Selectivity to Different Heavy Metals

Feed solutions containing 0.19 mM (40 ppm) lead,0.36 mM (40 ppm) cadmium, 0.2 mM (40 ppm) mercury,0.68 mM(40 ppm) cobalt, and 0.68 mM (40 ppm) Ni at $pH^o = 3.86$ with mole ratios of ligand to the total number of moles of metals, r_L^o, varying between 0 and 2.9, were prepared. The percentage removal of each metal and the equilibrium pH are shown in the Fig. 6 for the different mole ratios of the ligand.

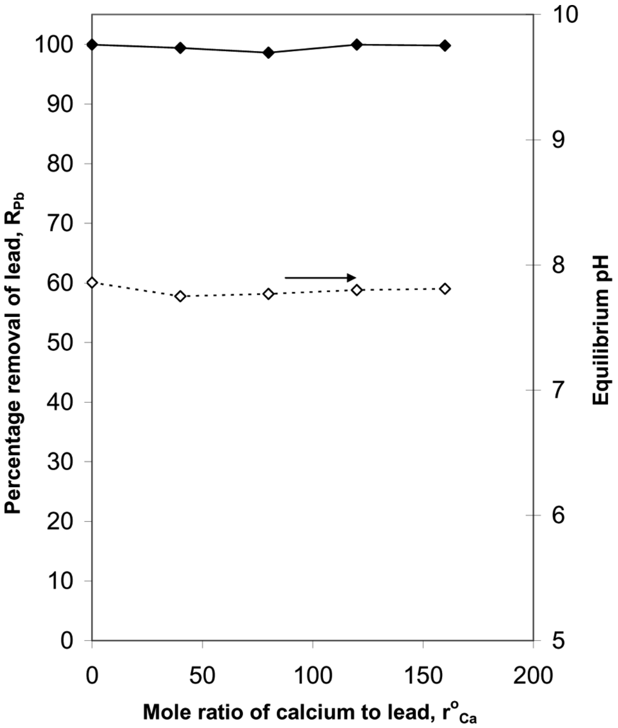

Figure 5. Effect of mole ratio of calcium to lead on the removal of lead, $r^{o}_{L} = 2.0$, $C^{o}_{Pb} = 0.19\,mM$.

The percentage removal of each metal and the equilibrium pH increased with increasing the mole ratio up to $r^{o}_{L} = 1.88$. In this range of mole ratios, although the other metals are present in higher molar concentrations than lead, the percentage removal of lead was the highest, followed by cadmium, cobalt/nickel, and then mercury. At $r^{o}_{L} = 1.88$, a percentage removal over 99% of each metal was obtained, except for mercury. The residual concentrations of lead, cadmium, cobalt, and nickel were below 10 ppb. The residual concentration of mercury was about 19 ppm, corresponding to a percentage removal of 45%. Increasing the mole ratio above 1.88 had no effect on the removal of mercury. This poor removal of mercury may be attributed to slow kinetics of mercury-ligand precipitate formation. Kinetics of mercury removal by this ligand is currently under investigation in our laboratory.

The affinity of the ligand for a particular ion among others present in a solution depends both on the ligand and the ion. Metal ions exist in aqueous solution with a certain number of water molecules bound to

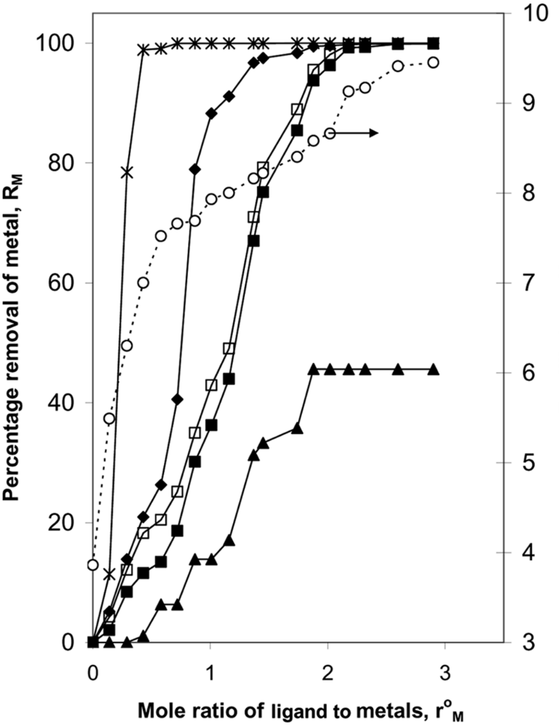

Figure 6. Selectivity of the ligand to heavy metals: pH° = 3.86, (∗) $C^{o}_{Pb} = 0.19$ mM (40 ppm); (◆) $C^{o}_{Cd} = 0.36$ mM (40 ppm); (□) $C^{o}_{Ni} = 0.68$ mM (40 ppm); (■) $C^{o}_{Co} = 0.68$ mM (40 ppm); (▲) $C^{o}_{Hg} = 0.2$ mM (40 ppm).

them. The larger the ionic radius, the smaller the hydrated radius and the lower the hydration energy. Smaller hydrated ions bind more strongly to ligands. This may explain the selectivity of the ligand to lead over cadmium, and cadmium over cobalt and nickel, since the ionic radii of Pb^{2+}, Cd^{2+}, Co^{2+}, and Ni^{2+} are 133, 109, 83 and, 83 pm; respectively (14). Except for mercury, the ligand left very low residual concentrations of the heavy metals, well below the accepted levels in most regions with very low reagent loss and back contamination, below 3 ppm.

Regeneration of Sodium Mono- and Di- (n-Hexadecyl) Phosphinate (Ligand)

Two sets of samples of metal-ligand precipitates were produced by treating one-liter solutions containing 40 ppm of lead with the ligand at a mole ratio, r_L^o, of 2. The precipitates were then treated with 10 ml solution of 3 N nitric acid, in one set, and with 10 ml solution of 3 N NaOH, in the other set. The acid and the sodium forms of the ligand precipitated. In both cases, after filtration, the ligand was 99.99% regenerated in its acid or sodium form, and the metal was concentrated to about 100 times.

CONCLUSIONS

Heavy metal pollution is a growing environmental concern, since these metals exhibit toxic effect at very low concentrations. The more and more stringent rules pertaining to their maximum accepted levels in wastewater streams rendered many current removal technologies obsolete and motivated the development of new ones. The current work employs a mixture of sodium mono, and di-(n-hexadecyl) phosphinate ligands for the precipitation of different heavy metals; including lead, cadmium, mercury, cobalt, and nickel from aqueous solutions. Upon a single treatment, the residual metal concentration decreased to below 10 ppb, except for Hg, and the residual ligand concentration was limited to below 3 ppm, even when excess amount of ligand was added. This low residual ligand concentration is attributed to low solubility of the sodium form of the ligand. Nevertheless, the limited solubility of the ligand did not pose mass transfer limitations, and, except for mercury, very quick precipitation reactions were encountered. The residual metal concentration of 10 ppb satisfies the maximum accepted metal concentration in most regions.

The system with lead, as a model heavy metal, was studied in details. The presence of chloride, sulfate, or calcium had no effect on lead removal. Decreasing the pH of the feed decreased the percentage removal of lead, because some of the ligand precipitated as acid form. This fact was exploited in the regenerate step of the ligand by adding a proper amount of an acid at certain concentration. The regeneration of the ligand in its sodium form and the recovery of the metal could also be achieved by adding a 3 N NaOH solution to the ligand/heavy metal precipitate. The ligand was found to be selective to lead over the other heavy metals and the selectivity was in the order: lead $>$ cadmium $>$ Cobalt and nickel $>$ mercury, at the conditions considered in this study.

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NOMENCLATURE

C_i^o	Initial concentration of species i (in the feed)
C_i^o	equilibrium concentration of species i ligand
pH^o	initial pH (pH of the feed)
$pH^{\square}$	final (equilibrium) pH
r_i^o	mole ratio of total species i to total metal(s) in the feed
R_M	percentage removal of metal

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