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Removal of aluminum from aqueous solutions using sodium di-(*n*-octyl) phosphinate

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Abstract

Sodium di-(*n*-octyl) phosphinate (NaL), was used as a ligand agent to remove aluminum from aqueous solutions. Aluminum was removed from the aqueous phase as an insoluble complex in the form $AlL_{3(S)}$. The effects of the initial pH, between 1 and 7, and of the mole ratio of NaL to aluminum, between 0 and 6, were investigated. Adding acid to the initial solution decreases the removal of aluminum and precipitates phosphinate in the acid form as $HL_{(S)}$. Total removal of aluminum, as $AlL_{3(S)}$, was obtained with an initial pH of 2.75 and a mole ratio between the ligand and aluminum of 3. The removal of aluminum was not affected by the presence of calcium or chloride in the range of concentrations and pH studied. Over 95% of the aluminum, free of precipitating agent, was recovered by addition of a 3 M NaOH aqueous solution to the precipitate, followed by extraction of the sodium phosphinate with diethyl ether. The aluminum was completely recovered from the precipitated $AlL_{3(S)}$ in a solution at a concentration four times its concentration in the initial aqueous phase.

Keywords: Wastewater treatment; Aluminum removal; Metal recovery; Sodium di-(*n*-octyl) phosphinate

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1. Introduction

Water streams and groundwater containing metals in solution, present considerable health and environmental hazards. Numerous industrial effluents discharge aluminum into the environment. The advantages and disadvantages of precipitation, extraction, biosorption, ion exchange, and other techniques for the removal or recovery of metals from wastewater have been discussed by Peters et al. [1] and also by Patterson [2]. In the case of aluminum, the objective of these processes was to recover aluminum from the vast supply contained in industrial and mine wastewater. Typical examples are waste liquors discarded in the production of catalyst-supporting materials from clays; liquors from uranium mills using acid leach process; acid mine waters from iron and coal mines; waste solutions resulting from the extraction of copper from waste dumps at porphyry copper mining operations or from Bayer process [3–5].

Extraction processes used for the removal of aluminum from aqueous solutions use different organic phases like kerosene, benzene, 1-octanol, and also different extracting agents like di-(2-ethylhexyl-) phosphoric acid [6], 2-ethylhexyl 2-ethylhexyl-phosphonic acid [7], phenylacetic acid [8], bis(1,1,3,3-tetramethylbutyl) phosphinic acid (MBP) and its sulfur analogues [9], alkyl phosphoric acids [3]. A limitation of these methods is that residual organic solvent or expensive extractant remains as a contaminant in the treated wastewater. The removal of the extractant from the treated water adds to the costs of the process.

Precipitation processes widely used to remove metal ions from aqueous solutions include the formation of hydroxides, carbonates [10], or sulfides [11]. The disadvantages of most precipitation processes are the large consumption of precipitating agent and the difficulties found in the filtration of the sludge. The use of organic reactants to precipitate metals in solution [12] has been limited by poor regeneration and the high cost of the organic precipitating agent.

Biosorption processes used to remove aluminum from aqueous phase using *Sargasum fluitans* [13] or *Cyanidium caldarium* [14] are economic and environmentally friendly but they seem to be limited to the extraction of the metals from dilute solutions. The recovery of aluminum from the biomass has not been reported. Lee and Volesky [15] and

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Nomenclature

C_i	overall equilibrium concentration of i, i = Al, Ca, L (mol/l)
C_i^0	overall concentration of i in the initial solution (mol/l)
L	di-(*n*-octyl) phosphinate, ligand.
L_L	percentage loss of the ligand
n_{Al}	number of moles of aluminum in treated aqueous solution at equilibrium
n_{Al}^0	number of moles of aluminum in an initial aqueous solution
n_L	number of moles of phosphorus (ligand) in the treated aqueous solution at equilibrium
n_L^0	number of moles of phosphorous (ligand) in the initial aqueous solution
pH	equilibrium pH
pH^0	pH of the initial solution
r_{Ca}^0	mole ratio of total calcium to total metal in the initial solution
r_{Cl}^0	mole ratio of total chloride to total metal in the initial solution
$r_{NO_3}^0$	mole ratio of total nitrate to total metal in the initial solution
r_L^0	mole ratio of sodium di-(*n*-octyl) phosphinate, added to the system, to the total metal in the initial solution
R_i	percentage removal of metal i, i = calcium or aluminum
S	solid precipitate

Texier et al. [16] reported that the aluminum interferes with the biosorption process of the desired metals and should be removed first.

In the case of ion exchange, the process is generally used for effluent with low aluminum concentration or as a secondary treatment in order to polish wastewater [2]. Problems associated with this treatment are the fouling of the system and the costly regeneration of the resins.

Recently, Esalah et al. [17,18] studied the use of an organophosphorus compound as precipitating reagent to extract heavy metals from aqueous solutions. The successful removal of lead, cadmium and zinc from aqueous solutions was obtained by formation of a solid complex with sodium di-(*n*-octyl) phosphinate. No phosphinate was detected in the aqueous phase after removal. A 99% recovery of the ligand from the solid complex was obtained and a concentration of 100 times that of the metal in the initial solution was attained. In addition, any ligand remaining in the wastewater can be removed by addition of calcium chloride or calcium carbonate. Treatment of the insoluble calcium salt of the ligand with sodium hydroxide produces its sodium form [17].

This study is part of a series in which different metals are selectively removed from dilute aqueous solutions by formation of an insoluble solid complex with sodium di-(*n*-octyl) phosphinate $(C_8H_{17})_2POONa$. Aluminum is normally found in wastewaters and its removal may be of interest by itself or due to the interference that aluminum may cause for the removal of more valuable metals. In this work, we report the removal of aluminum from low pH aqueous solutions. The speciation of aluminum is well documented in literature [19]. Complexing agents, called ligands, capable of satisfying the coordination number of a metal, form complexes with metals in solution. When these ligands are not present or their concentration is low, metal ions are normally hydrated with water molecules, and combined with hydroxyl ions in the form hydroxyl complexes. At high metal concentration and high pH, polynuclear hydroxyl complexes are expected to be present [19–22]. The possible presence of species Al^{3+}, and the hydroxyl forms $Al(OH)^{2+}$, $Al(OH)_2{}^+$, $Al(OH)_3$, and $Al(OH)_4{}^-$ were considered to explain the complexation of the metal with the ligand used in this work.

2. Experimental

2.1. Materials and methods

Di-(*n*-octyl) phosphinic acid $(C_8H_{17})_2POOH$, was synthesized by the peroxide catalyzed reaction, between 1-octene and hypophosphorus acid following the Peppard et al.'s modification [23] to the procedure described by Williams and Hamilton [24]. The acid was first purified and then converted to its sodium salt. The stock solution of aluminum was a standard aqueous solution (1000 ppm) with 2% HNO_3 purchased from Fisher Scientific (Montreal, Quebec). All the other chemicals were of analytical grade also purchased from Fisher Scientific (Montreal, Quebec).The experiments were carried out using deionized water with conductivity less than 0.8 μS/cm, prepared with and Easy pure, RF, Compact Ultrapure Water system. The pH measurements were performed with an accuTupH reference/pH electrode (Fisher Scientific, Montreal, Quebec) filled with saturated potassium chloride solution (Fisher Scientific, Montreal, Quebec). An OAKTON pH/mV Benchtop Meter model WD-35616-00 with a resolution of 0.01 pH and accuracy of ±0.01 pH was used to monitor pH measurements. The solutions were filtered with 0.2 μm filters (Fisher Scientific, Montreal, Quebec). The concentrations of aluminum and phosphorus, and thus of ligand, were measured by inductively coupled plasma (ICP) atomic emission spectroscopy using a Thermo Jarrel Ash Trace Scan (Thermo Jarrel Ash, Waltham, MA). The wavelength used to detect phosphorus was 213.618 nm and that for aluminum was 396.152 nm. The solid complex was analyzed by X-ray diffraction (Philips Diffractometer PW 1710).

2.2. Experimental procedure for the precipitation of aluminum

The experiments were carried out at room temperature (22 ± 1 °C). A feed solution containing 0.02 mmol of aluminum at a fixed pH was treated with an aqueous solution containing 7000 ppm of the ligand agent so to obtain the desired ligand/aluminum molar ratio. The total volume was fixed to 20 ml, by addition of distilled water, giving a concentration of 25 ppm of aluminum. The pH adjustment of the initial solution was made by addition of aqueous HNO_3 or NaOH before adding the ligand. The samples were shaken and then left to equilibrate for 24 h. The equilibrium was tested by analyzing the concentration of aluminum and ligand in the aqueous solution at different times (15 and 30 min, 1 and 2 h, 1 and 2 days). For low concentrations of aluminum less than 24 h were necessary to reach equilibrium. For higher concentrations of aluminum, shorter times were required. Thus, a contact time of 24 h was fixed for all the experiments. At the end of each experiment, a white insoluble solid complex and a clear aqueous phase were obtained. The solution was filtered using 0.2 μm filters and the equilibrium pH of the filtrate was measured. The concentration of phosphorus and metal remaining in the aqueous solution were determined by ICP spectrophotometer. In parallel, the solid complex was analyzed by a X-ray diffraction and the spectrum obtained was compared to the database JCPDS (International Center of Diffraction Data).

2.3. Recovery of the ligand and of the aluminum

After separating the complex from the aqueous phase, the ligand was regenerated and the aluminum concentrated in aqueous solution. The regeneration of the ligand was done by adding 3 M NaOH, followed by extraction with diethyl ether. After agitation and subsequent phase disengagement, two phases were obtained. The lower aqueous phase was analyzed with an ICP to determine the concentration of aluminum and phosphorus. The upper ether phase containing the ligand agent in its sodium form was evaporated, and sodium di-(*n*-octyl) phosphinate was regenerated.

3. Results and discussion

In the present work, the separation of aluminum from an aqueous solution using sodium di-(*n*-octyl) phosphinate was studied. The effect of pH, mole ratio between ligand and metal and the presence of different ions were investigated. At least two replicates were run for each experiment. The concentrations of the species are reported in molarity (M) and/or parts per million by mass (ppm). The percentage removal of aluminum, the percentage loss of the ligand agent in the treated solution and the equilibrium pH are reported. The percentage removal of aluminum, R_{Al}, is defined as

$$R_{\mathrm{Al}} = \frac{n_{\mathrm{Al}}^0 - n_{\mathrm{Al}}}{n_{\mathrm{Al}}^0} \times 100 \qquad (1)$$

where n_{Al}^0 is the initial number of moles of aluminum in the aqueous solution and n_{Al} is the number of moles left in the treated aqueous solution at equilibrium.

The percentage loss of the ligand, L_{L}, is defined as

$$L_{\mathrm{L}} = \frac{n_{\mathrm{L}}}{n_{\mathrm{L}}^0} \times 100 \qquad (2)$$

where n_{L} is the number of moles of phosphorus ligand in the aqueous solution at equilibrium, and n_{L}^0 is the number of moles of phosphorous ligand in the initial aqueous solution. In the figures, the data are shown as symbols and lines; the superscript zero (0) is used for the properties of the initial aqueous solution, before treatment with the ligand, so to distinguish them from the values at equilibrium.

In order to explain the behavior of aluminum in the presence of ligand, we first present the results of the effect of pH in a solution containing aluminum nitrate and no ligand and in a solution containing the ligand and no aluminum.

Fig. 1 shows the effect of pH on the precipitation of aluminum, from a solution of aluminum nitrate at a concentration C_{Al}^0 of 0.925 mM (25 ppm in 20 ml). The initial pH (pH^0) was adjusted with a solution of 1 M HNO_3 or 0.2 M NaOH and no ligand was added to the solution. As seen in Fig. 1, the aluminum is precipitated for pH between 4 and 10 with a maximum precipitation at a pH around 7. In this pH range, the aluminum precipitates as $Al(OH)_3$. Depending on the pH of the solution, different hydroxyl complexes can be formed by the aluminum ions, Al^{3+}, due to the following reaction:

$$Al^{3+} + nOH^- \leftrightarrow Al(OH)_n^{3-n} \qquad (R1)$$

where $n = 1$, 2, 3 and 4. Thus, at pH below 5, aluminum is in the form of soluble Al^{3+}, $Al(OH)^{2+}$ and $Al(OH)_2{}^+$ as the value of the pH increases. At pH around 9 the soluble complex $Al(OH)_4{}^-$ forms. The consumption of OH^- in reaction (R1) displaces to the right the ionization reaction of water (R2):

$$nH_2O \leftrightarrow nH^+ + nOH^- \qquad (R2)$$

The net result of reaction R2 is a production of free protons, which is reflected in a decrease of pH. Thus, aqueous solutions of aluminum nitrate are acidic and conversely, the precipitation of aluminum from a aluminum nitrate solution tends to decrease the pH of the solution.

Fig. 1 also depicts the effect of the pH on the precipitation of sodium di-(*n*-octyl) phosphinate ligand, in the absence of aluminum in the aqueous phase. A solution with an initial concentration of ligand of $C_{\mathrm{L}}^0 = 2.1$ mM at an initial pH around 9 (that is the natural pH of the ligand at this concentration in aqueous solution), was acidified with an aqueous solution of 1 M of HNO_3. As shown in Fig. 1,

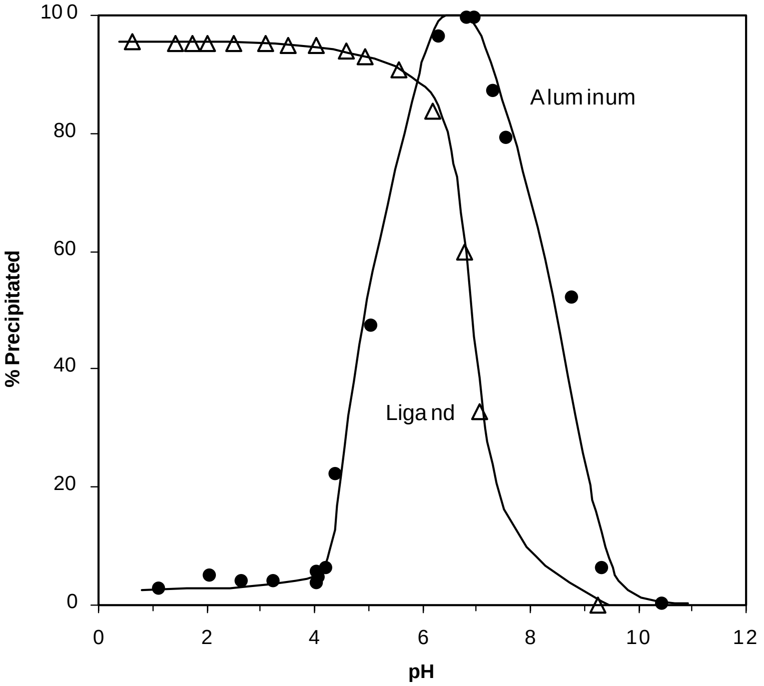

Fig. 1. Effect of the pH on the % precipitation of aluminum (●) and % precipitation of sodium di-(*n*-octyl) phosphinate (△) from an aqueous solution.

the precipitation of the ligand increases as the pH decreases, reaching over 95% removal at pH below 5. This is due the formation of water insoluble di-(*n*-octyl) phosphinic acid $(C_8H_{17})_2POOH$, that we write as $HL_{(S)}$, for short:

$$H^+ + L^- \leftrightarrow HL_{(S)} \tag{R3}$$

3.1. Effect of the mole ratio ligand to aluminum

Fig. 2 shows the percentage removal of aluminum and the percentage loss of the ligand for a feed solution of 0.925 mM (25 ppm) aluminum, at an initial $pH^0 = 2.75$, treated with sodium-(*n*-octyl) phosphinate at different mole ratios of ligand to aluminum. The removal of aluminum increased linearly up to a value of the mole ratio of 3 with small increase in the equilibrium pH, owing to the precipitation of aluminum. At this ratio, the equilibrium concentration of aluminum and ligand remaining in the aqueous solution, within the confidence interval 95%, were 0.7 ± 0.1 ppm and 5 ± 0.5 ppm, respectively. Based on these results, the following reaction is used to describe the formation of the insoluble complex $[((C_8H_{17})_2POO)_3Al]$, for a pH below 4:

$$Al^{3+} + 3L^- \leftrightarrow AlL_{3(S)} \tag{R4}$$

For pH higher than 4, the formation of hydroxyl complex occurs and the reactions R1 and R2 have to be considered as discussed above.

The sudden increase in pH at mole ratio above 3 was due to the formation of di-(*n*-octyl) phosphinic acid (R3). The highest percentage removal with the lowest loss of ligand agent was observed at mole ratio of 3. Higher molar ratios increased the loss of precipitating agent and had not noticeable effect on the percentage removal of aluminum.

3.2. Effect of the pH of the initial solution

The effect of adjusting the pH of the initial solution, pH^0, from 1 to 8 by addition of HNO_3 or NaOH is shown in

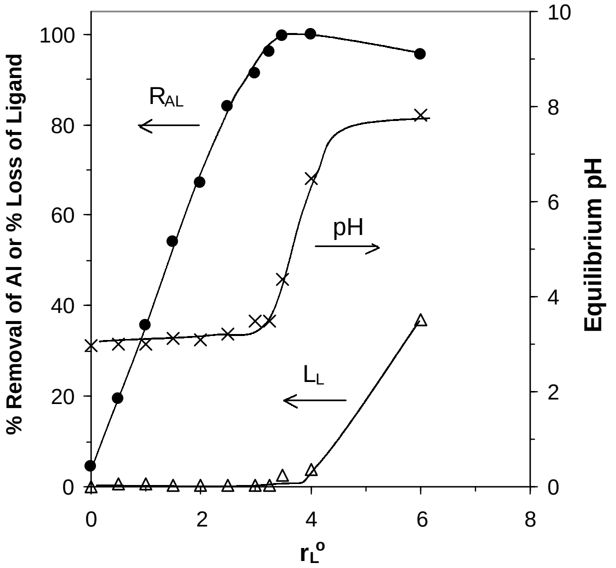

Fig. 2. Effect of mole ratio of sodium di-(*n*-octyl) phosphinate to aluminum on the removal of aluminum (●), % loss of ligand (△) and the equilibrium pH (×). Initial solution contains aluminum nitrate at $C^0_{Al} = 0.925$ mM and initial $pH^0 = 2.75$.

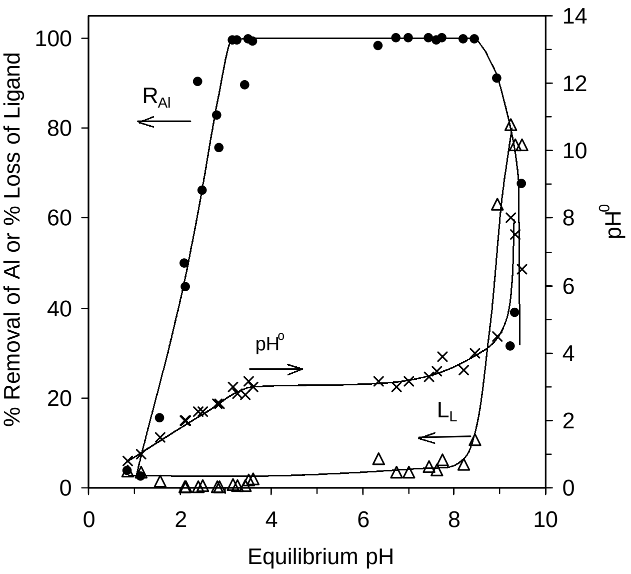

Fig. 3. Effect of the initial pH on the removal of aluminum, the loss of the ligand agent and equilibrium pH using sodium di-(*n*-octyl) phosphinate. Initial solution contains aluminum nitrate at $C^0_{\mathrm{Al}} = 0.925\,\mathrm{mM}$. The mole ratio of ligand to aluminum was $r^0_{\mathrm{L}} = 3.1$: % removal of aluminum (●), % loss of ligand (△), initial pH^0 (×).

Fig. 3. The initial solution had a concentration of aluminum of 0.925 mM (25 ppm) for a total volume of 20 ml at a fixed pH^0. The mole ratio of the ligand agent to aluminum (r^0_{L}) was fixed at 3.1 for all the experiments. As shown in Fig. 3, the 100% removal efficiency of the aluminum was obtained for a pH^0 of 2.75. Decreasing pH^0 below 2.75 lowered the percentage removal of aluminum and also the equilibrium pH, while the percentage loss of the ligand remained constant. This result can be explained by reactions R2, R3 and R4, where the H^+ ions compete with aluminum for the formation of a solid complex with the ligand. The equilibrium pH is always slightly higher than the initial pH since part of the H^+ ions are consumed in the formation of the acidic form of the ligand. As pH^0 increased from around 3 to 4, the percentage removal of aluminum, which was 100%, and the percentage loss of the ligand remained constant, while the equilibrium pH increased from around 3 to 8. In this pH range, aluminum hydroxyl complexes, with an eventual precipitation of the aluminum trihydroxide, are formed following reaction R1. The ligand competes with the OH^- ions for the aluminum. The formation of complexes frees more ligand that will precipitate and thus consume H^+ ions. This results in a shifting of reaction R2 towards the formation of OH^- and the equilibrium pH increases. As pH^0 increase, the degree of complexation of aluminum with OH^- is higher and fewer H^+ ions are available in the aqueous solution. At an equilibrium pH around 7, the excess of ligand remains in the aqueous solution. While less ligand precipitates either with H^+ ions or aluminum, the removal efficiency of aluminum decreases at pH around 9 due to the formation of the soluble $\mathrm{Al(OH)_4}^-$ complex.

In Fig. 4, the percentage removal of aluminum and the equilibrium pH for three sets of samples having an aluminum concentration of 0.925 mM (25 ppm in 20 ml), and at pH^0 values of 2, 2.5 and 2.75, respectively, are compared at different values of the mole ratio of ligand to aluminum. No pH adjustment was made to the first set. The initial pH of the other two sets was adjusted by addition of NaOH. A decrease in the pH of the initial solution from 2.75 to 2 causes a decrease in the removal of aluminum and of the equilibrium pH, for a fixed value of the mole ratio of ligand to aluminum. The largest percentage removal of aluminum for each set was obtained at different value of the ratio r^0_{L}. The lower the pH, the higher the value of the ratio required to produce maximum removal. This can be explained by the increase in the concentration of hydrogen ions due to the ligand forming the acid solid complex $\mathrm{HL_{(S)}}$, following reaction (R3). The change of the equilibrium pH with the ratio of ligand to aluminum for each set was previously considered in the discussion of Fig. 3.

3.3. Effect of chloride in the initial solution

Chloride, which is present in most water streams, forms soluble chloro complexes with aluminum:

$$\mathrm{Al}^{3+} + n\mathrm{Cl}^- \leftrightarrow \mathrm{Al(Cl)}_n^{3-n} \qquad \text{(R5)}$$

where $n = 1$, 2, 3, and 4. The effect of the chloride concentration on the removal of aluminum was investigated by adding sodium chloride to an initial solution of aluminum chloride. An initial solution containing 0.925 mM (25 ppm) aluminum, as aluminum chloride at pH^0 = 2.75, was treated with ligand agent at a ratio $r^0_{\mathrm{L}} = 2.5$. The mole ratio of chloride to aluminum (r^0_{Cl}) was increased from 10 to 400 by addition of sodium chloride. The chloride system was compared with nitrate system where aluminum nitrate was the source of the aluminum and the nitrate concentration was increased by addition of $\mathrm{NaNO_3}$. The initial pH was adjusted using HCl or $\mathrm{HNO_3}$, depending on the case.

Fig. 5 shows the percentage removal and the equilibrium pH as a function of r^0_{Cl} or $r^0_{\mathrm{NO_3}}$. The effect with both sodium salts followed the same trend. By increasing the value of r^0_{Cl} or $r^0_{\mathrm{NO_3}}$, the percentage removal of aluminum decreased while the equilibrium pH remained constant. The increase in the sodium concentration due to the addition of sodium salts displaced reaction (R6) to the left, resulting in a decrease in the availability of ligand to react with aluminum:

$$\mathrm{NaL_{(S)}} \leftrightarrow \mathrm{Na}^+ + \mathrm{L}^- \qquad \text{(R6)}$$

For a weak electrolyte (such as $\mathrm{AlL_{3(S)}}$), an increase in the ionic strength of the system, by addition of another electrolyte without a common ion (Al^{3+} or L^-), decreases its mean ionic activity coefficient and thus it increases its degree of dissociation, i.e. its solubility (17).

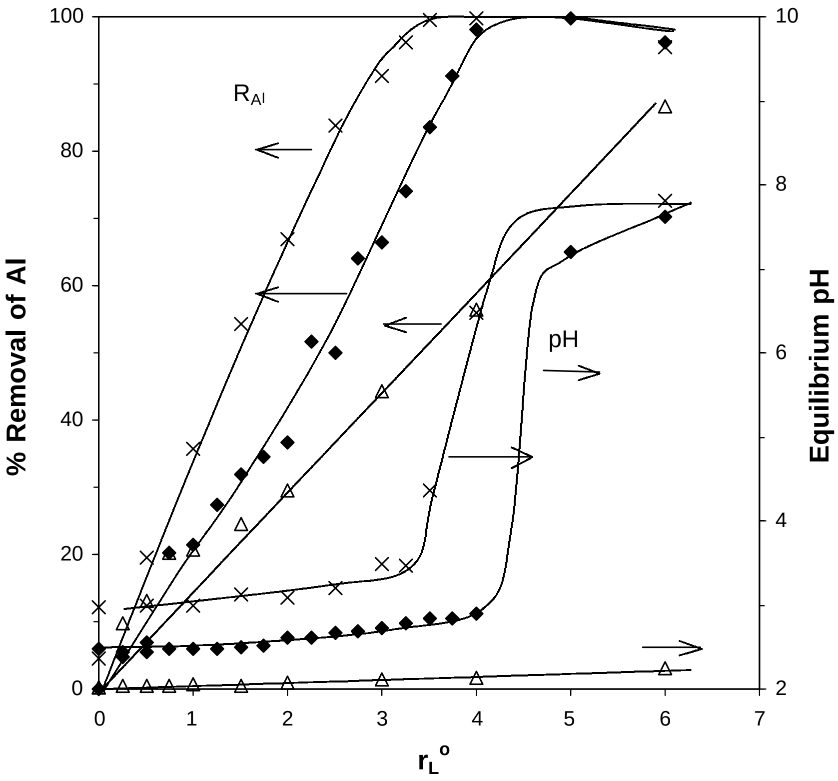

Fig. 4. Effect of mole ratio of sodium di-(*n*-octyl) phosphinate to aluminum on the removal of aluminum. Initial solution contains aluminum nitrate at $C^0_{\mathrm{Al}} = 0.925\,\mathrm{mM}$ and initial: $\mathrm{pH}^0 = 2.75$ (×), $\mathrm{pH}^0 = 2.50$ (◆), $\mathrm{pH}^0 = 2.00$ (△).

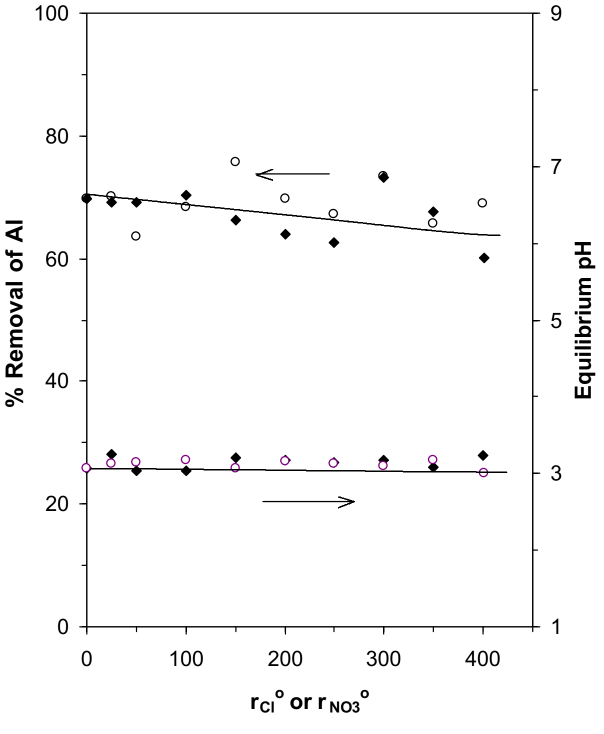

Fig. 5. Effect of chloride (using NaCl) and nitrate (using $\mathrm{NaNO_3}$) concentration on the removal of aluminum and equilibrium pH using sodium di-(*n*-octyl) phosphinate. $C^0_{\mathrm{Al}} = 0.925\,\mathrm{mM}$, $r^0_{\mathrm{L}} = 2.5$, $\mathrm{pH}^0 = 2.75$. Chloride (◆), nitrate (○).

3.4. Effect of calcium in the initial solution

Calcium, an alkaline earth metal, is generally present in most water streams. Calcium can be removed from an aqueous solution using sodium di-(*n*-octyl) phosphinate by forming the insoluble complex $\mathrm{CaL_{2(S)}}$ (17). Thus, $\mathrm{Ca^{2+}}$ can compete with aluminum for the ligand. The selectivity of the ligand for aluminum, in presence of calcium, was thus investigated. As shown in Fig. 6, the effect of the pH of the initial solution on the removal of aluminum, and on the equilibrium pH, was investigated by adding $\mathrm{Ca(NO_3)_2}$ at a mole ratio of ligand to aluminum of $r^0_{\mathrm{L}} = 4$ and a mole ratio of calcium to aluminum of $r^0_{\mathrm{Ca}} = 1.5$. The concentration of aluminum in the initial solution was 0.925 mM (25 ppm) and the initial pH was varied from $\mathrm{pH}^0 = 1$ to 8. As observed in the figure, the percentage removal of aluminum, the percentage removal of calcium and the equilibrium pH, all increase as the initial value of pH^0 increases. These trends can be explained by reactions, R1, R2, R3, R4, combined with the calcium complex formation with the ligand and with the formation of calcium hydroxyl complexes. Also, these results show that the ligand has more affinity toward the aluminum than calcium. However, as the pH increases, the formation of an insoluble aluminum hydroxide leaves ligand free to react with calcium and to remove it. Finally, the equilibrium pH is always higher than the initial pH^0 in the acidic region, $\mathrm{pH} < 7$. In this region, the $\mathrm{OH^-}$ ions are released from the

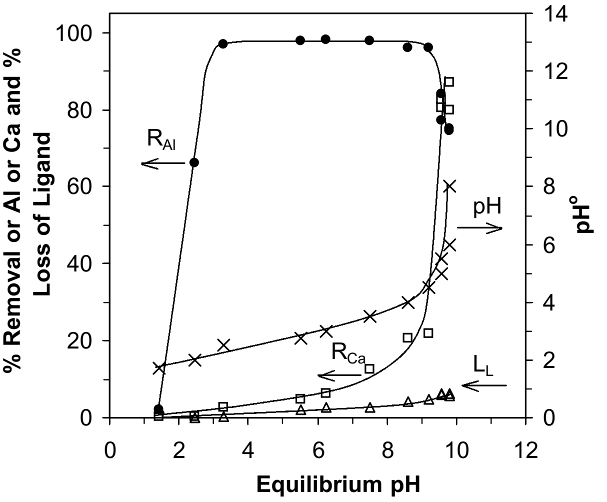

Fig. 6. Effect of pH of the initial solution on the % removal of aluminum, the equilibrium pH and % loss of ligand in the presence of calcium. Initial solution contains $C^0_{Al} = 0.925$ mM, $C^0_{Ca} = 1.4$ mM, $r^0_{Ca} = 1.5$, $r^0_L = 4$; % removal of calcium (☐), % removal of aluminum (●), % loss of ligand (△), pH feed (×).

metal hydroxyl complexes as metal–ligand complexes are formed. Any extra ligand in this case is removed from the solution by forming the insoluble complex $LH_{(S)}$.

3.5. Regeneration of the ligand and recovery of the aluminum

To regenerate the ligand agent in its sodium form and recover the aluminum at a high concentration, sodium hydroxide and diethyl ether were used. While sodium hydroxide dissolved the precipitate, the ligand was extracted in the organic phase. In this work, only diethyl ether was studied due to its common use in the industry and its ability to extract this specific ligand (24). Other organic solvents, such as chloroform, could be of potential interest for future studies.

As solid complex ($AlL_{3(S)}$) was formed by adding 5 ml of a 7000 ppm sodium di-(*n*-octyl) phosphinate solution to 15 ml of a 50 ppm aluminum nitrate solution. The aluminum di-(*n*-octyl) phosphinate solid complex was separated from the aqueous solution and then dissolved using 10 ml of 3 M NaOH. A volume of 3 ml of diethyl ether was then added to the solution and the mixture was agitated vigorously. After phase separation, two distinct phases were formed: a lower (aqueous) phase, containing the aluminum and an upper (ether) phase, containing the ligand agent. The ligand agent was recovered from the organic phase by evaporating the ether. No aluminum was detected in the regenerated ligand, confirming the insolubility of aluminum di-(*n*-octyl) phosphinate in diethyl ether. Over 95% of the ligand added to the initial aluminum solution was regenerated and recovered as sodium di-(*n*-octyl) phosphinate. The volume of the aqueous basic solution, containing the aluminum, was about 5 ml. Due to the low aqueous basic solution used in this study, the concentration of aluminum was thus increased four times with respect to that of the initial solution.

4. Conclusions

Aluminum was successfully removed from aqueous aluminum nitrate solutions using sodium di-(*n*-octyl) phosphinate to form a solid complex AlL_3. The percentage removal is strongly dependent of the pH of the initial solution and on the mole ratio ligand to metal. At $pH^0 = 2.75$, $pH = 4$, and $r = 3$, a 100% of removal was achieved. The presence of chloride, nitrate, or calcium had not effect on the removal of aluminum within the ranges of concentrations and pH of the initial solution considered in this study. Essentially complete recovery of aluminum and regeneration of the phosphinate was obtained.

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