Refereed Proceedings

Separations Technology VI: New Perspectives on Very Large-Scale Operations

Adsorption of Metals from Metal-Organic Complexes Derived from Bioleaching of Nickel Laterite Ores

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ADSORPTION OF METALS FROM METAL-ORGANIC COMPLEXES DERIVED FROM BIOLEACHING OF NICKEL LATERITE ORES

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ABSTRACT

This study aims to establish the potential of an iminodiacetic-based chelating resin (Purolite S930) in recovering Ni and Co from the biological leachate. The adsorption tests were carried out to compare the hydrogen (H) and sodium (Na) forms of resin. The equilibrium adsorption isotherms were determined using metal citrate complexes with concentrations from 15 to 2000 mg/L. Adsorption tests were carried out for a period of 24 hours to attain adsorption equilibrium. The pH of the solution was varied using citric acid with concentrations of 0.01-1.0 M. The adsorption equilibrium data were interpreted using the Langmuir and Freundlich models. Metal elution was carried out using 2M HNO_3 solution. The adsorption behaviors of nickel and cobalt citrate complexes were found to follow both Langmuir and Freundlich models. The results suggested that metal uptake were influenced by the hydrolysis of the resin and competition of metal complexes with the citrate anion and hydronium ion (H^+). Metal elution was influenced by the interaction or reaction of metal complexes with the resin which included surface complexation.

INTRODUCTION

Approximately 80% of known nickel and about 95% of cobalt global reserves are found in nickel laterite ore deposits ([1], [2], [3]) and the rest are in sulfide ores ([4]). Commercial extraction of nickel and cobalt from laterite ores constitutes one of the most challenging and high cost processing techniques. This is largely associated with the fact that there are no free nickel and cobalt minerals which limits the beneficiation of these ores. The processes are also subjected to strict environmental regulations, which continuously constrain the processing of laterite ores. Microbial leaching is an alternative technology which offers a much needed step-change in laterite processing ([3], [5], [6]). Biological

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leaching of low-grade nickel laterite ores involves the use of heterotrophic micro-organisms such as *Aspergillus* and *Penicillium* species. Excreted metabolites (organic acids) dissolves the heavy metals from mineral oxide ore to form metal-organic complexes. Previous studies have shown that citric is the most effective in dissolving Ni and Co minerals (7).

Metal recovery from bioleach solution is of practical importance. However, it has received only minor interest in literature (8). Most studies in this area have been limited to internal industrial investigations. There are several methods which can be used in recovering metals from solution. This includes electrowinning, chemical precipitation and adsorption. Adsorption using ion exchange chelating resins has been shown to be an effective method and has wide application in wastewater treatment (9) and hydrometallurgical processes (10). Chelating resins like iminodiacetatic-based resin have been shown to demonstrate high selectivity for transition metals, particularly copper and nickel. Their performances in recovering metals from solution were found to be superior compared to ordinary ion exchange resins because of their ability to complex with metals (11, 12). This study aims to establish the adsorption and desorption characteristics of nickel and cobalt citrate complexes on an iminodiacetate-based chelating resin (Purolite S930). Metal concentrations, citric acid concentrations (or solution pH) and sodium (Na) and hydrogen (H) forms of the resins were considered in this investigation.

EXPERIMENTAL DETAILS

Resins and Chemicals

The chelating resin, Purolite S930, based on iminodiacetic acid functional group (see Figure 1) was used in this investigation (13). The physical and chemical properties of the resin are shown in Table 1. Prior to use, the resins was rinsed with deionised water to remove all impurities followed by a regeneration process to clean and convert the resin to H and Na-form. The regeneration process involved washing the resin with 2 M HCl for 1 hour. This was followed by deionised-water rinse. The H-form of the resin was dried at $50^{\circ}C$ for 3 hours. To convert the resin to sodium form, the H-resin was contacted with 2 M NaOH for 1 hour and followed by deionised-water rinse. The resin was dried and stored at room temperature.
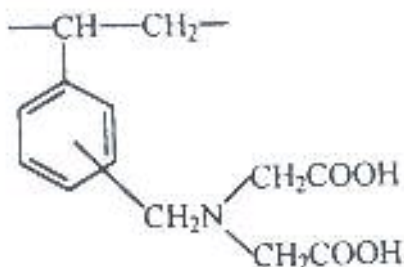

Figure 1. Chemical Structure of Purolite S930.

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Table 1. Properties of commercial chelating resin ([13]).

	Purolite S930
Matrix	Macroporous Styrene-divinylbenzene
Functional group	Iminodiacetic acid
Particle size (mm)	0.3-1.0
Bed density (g/L)	710-745
Particle density (g/cm^3)	1.17
Moisture content (%)	55-65
Operating temperature, max (^{o}C)	70
PH range (operating)	H$^+$ form 2-6, Na$^+$ form 6-11
Total capacity (meq/g wet)	H$^+$ form 1.52, Na$^+$ form 1.29

The synthetic metal solutions (15 to 2000 mg/L) used in this study were prepared using analytical-reagent-grade $Ni(NO_3)_2.6H_2O$ and $Co(NO_3)_2.6H_2O$ salts dissolved in 0.01, 0.1, 0.5 and 1.0 M citric acid. These acid concentrations (0.01 and 0.1 M) are typical of those generated by heterotrophic fungi used in bioleaching of nickel laterite ores ([3], [14]). The acid concentration was extended to higher (0.5 and 1.0 M) values as such conditions allow better leaching efficiencies to be achieved and could therefore be of future interest in this area.

Batch adsorption

The batch adsorption tests were carried out in a 250-mL reactor at room temperature. A 100-mL of metal solution at various concentrations was agitated with 2.0 g dry resins in incubator-shaker at 400 rpm for 24 hours. This period was sufficient to attain equilibrium adsorption. The metal concentration at the completion of adsorption was determined using atomic absorption spectroscopy. The adsorption capacity (Q_e) of the resins per unit mass was calculated from the following equation:

$$Q_e = \frac{C_i - C_e}{m} V \qquad (1)$$

where C_i and C_e are the initial and equilibrium concentrations of metal (mg/L), V is the volume of metal solutions (L), and m is the weight of dry resins (g).

Metal stripping

After the adsorption test, the resins were separated from the solution by filtration. The elution process was conducted in a 100-mL of 2 M HNO_3 under similar conditions to batch adsorption. The (%) efficiency was determined from the ratio of the mass of metal desorbed from the resins ($M_{desorbed}$) to the mass of metal adsorbed onto the resins ($M_{adsorbed}$).

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$$\%Efficiency = \frac{M_{desorbed}}{M_{adsorbed}} x100 \tag{2}$$

RESULTS AND DISCUSSION

Adsorption equilibrium isotherms of nickel and cobalt citrate onto S930

In order to optimise the design of an adsorption system in recovering metals from the leachate solution, it is important to establish the most appropriate correlations for the equilibrium data for each system. Two isotherm equations have been tested in the present study, the Langmuir (15) and Freundlich (16) models. The applicability of the isotherm equations was compared by judging the correlation coefficient, R^2. The Langmuir isotherm model is associated with the monolayer adsorption of solute on specific homogenous sites of the adsorbent. The Langmuir equation is the most widely used two parameter equation commonly expressed as:

$$\frac{1}{Q_e} = \frac{1}{Q_m b C_e} + \frac{1}{Q_m} \tag{3}$$

where C_e is the concentration of metal remaining in the solution after adsorption (mg/L) and Q_e is the amount of adsorbed metals per unit mass of resins at equilibrium (mg/g). The corresponding Langmuir parameters Q_m and b are the constants related to the area occupied by a monolayer of adsorbate, reflecting the adsorption capacity (mg/g) and a direct measure for the intensity of the adsorption process (L/g), respectively. The values of Q_m and b are evaluated from the slope and intercept of the plot of $1/Q_e$ vs $1/C_e$.

The Freundlich isotherm is an empirical equation which has been shown to satisfactory describe adsorption at low concentrations of solutions. The equation is commonly given by:

$$Q_e = A C_e^{1/n} \tag{4}$$

where A is a constant for the system, related to the bonding energy. The constant A can be defined as the adsorption or distribution coefficient and represents the quantity of heavy metal adsorbed onto the resin adsorbents for a unit equilibrium concentration (ie. C_e = 1 mg/L). The slope 1/n, varies between 0 and 1, is a measure of adsorption intensity or surface heterogeneity, becoming more heterogeneous as its value gets closer to zero. A value for 1/n below one indicates a normal Langmuir isotherm, while 1/n above one indicates a cooperative adsorption. A plot of log Q_e as a function of log C_e enables the empirical constants A and 1/n to be determined from the intercept and slope of the linear regression.

The nickel citrate adsorption isotherms on both H and Na-forms of Purolite S930 are shown in Figure 2. These tests were conducted at various pH by preparing the nickel complexes with different citric acid concentrations comprising of 0.01, 0.1, 0.5 and 1.0 M. The corresponding Langmuir and Freundlich model parameters are shown in Tables 2 and 3. It appears, from the determination of coefficients reported in Tables 2 and 3, that there is no distinction between the fit of the Langmuir and Freundlich models for

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nickel citrate adsorption on H and Na-forms of S930. However, adsorption of nickel citrate in 1.0 M provides the highest sorption capacity but the R^2 is worse than those in other concentrations. The adsorption data in 1.0 M could be described by the linear sorption model ($Q_e = K_P C_e$) which provides a good fit ($R^2 = 0.98$) and $K_P = 0.003$. The low partition coefficient K_P indicates poor adsorption of nickel citrate onto H-form resin which follows the reduction trend of metal uptake as acid concentration increases. In general, this suggests that adsorption of citrate complexes onto S930 occurs by a combination of monolayer and multilayer adsorption except in 1.0 M on H-form resin.
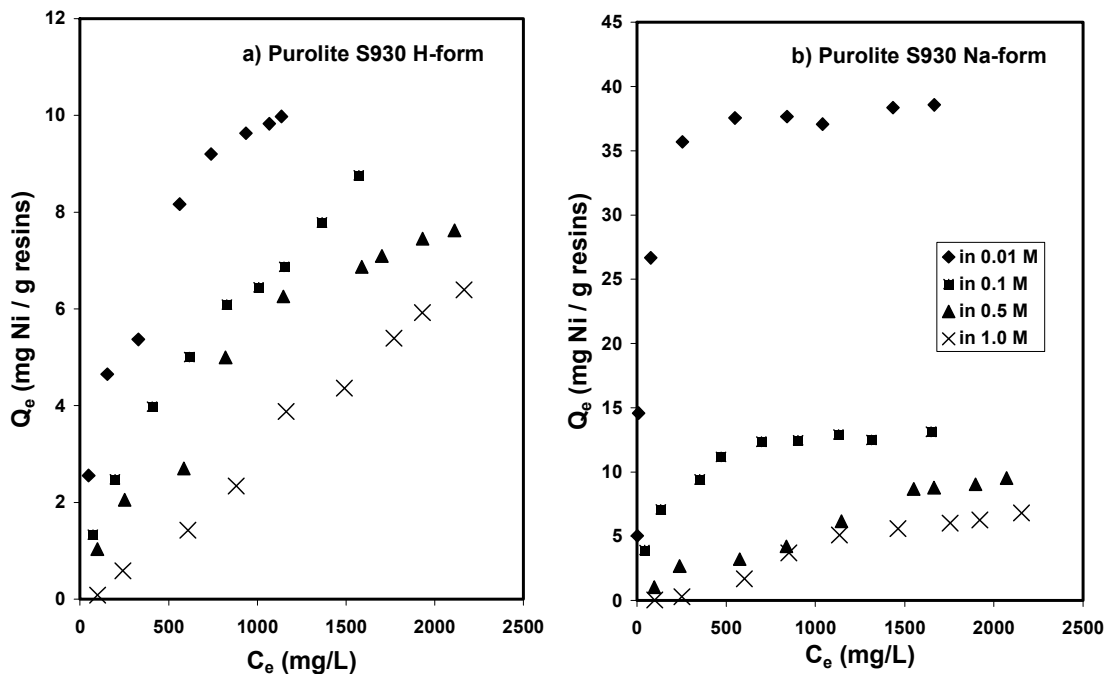

Figure 2. Adsorption isotherms of nickel citrate onto a) Purolite S930 H-form and b) Purolite S930 Na-form.

Table 2. Adsorption model parameters of nickel citrate onto S930 H-form.

[CA], M	Langmuir parameters			Freundlich parameters		
	Q_m (mg/g)	b (L/g)	R^2	A (mg/g)	1/n	R^2
0.01	8.04	9.5×10^{-3}	0.97	0.46	0.44	0.98
0.1	7.94	2.7×10^{-3}	0.99	0.10	0.60	0.99
0.5	7.04	1.7×10^{-3}	0.96	0.05	0.67	0.97
1.0	16.50	3.0×10^{-4}	0.92	2.5×10^{-4}	1.34	0.97

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Table 3. Adsorption model parameters of nickel citrate onto S930 Na-form**.**

[CA], M	Langmuir parameters			Freundlich parameters		
	Q_m (mg/g)	b (L/g)	R^2	A (mg/g)	1/n	R^2
0.01	30.40	1.2×10^{-1}	0.99	4.89	0.38	0.95
0.1	12.36	1.1×10^{-2}	0.99	0.84	0.42	0.98
0.5	12.00	1.0×10^{-3}	0.97	0.04	0.71	0.96
1.0	10.09	9.0×10^{-4}	0.97	1.0×10^{-5}	1.90	0.97

The adsorption isotherms of cobalt citrate onto S930 H- and Na- forms are shown in Figure 3 and the corresponding model parameters are reported in Tables 4 and 5. As shown, the cobalt citrate adsorption equally fits both the Langmuir and Freundlich models. This suggests that cobalt citrate adsorption also occurs by monolayer and multilayer adsorption.
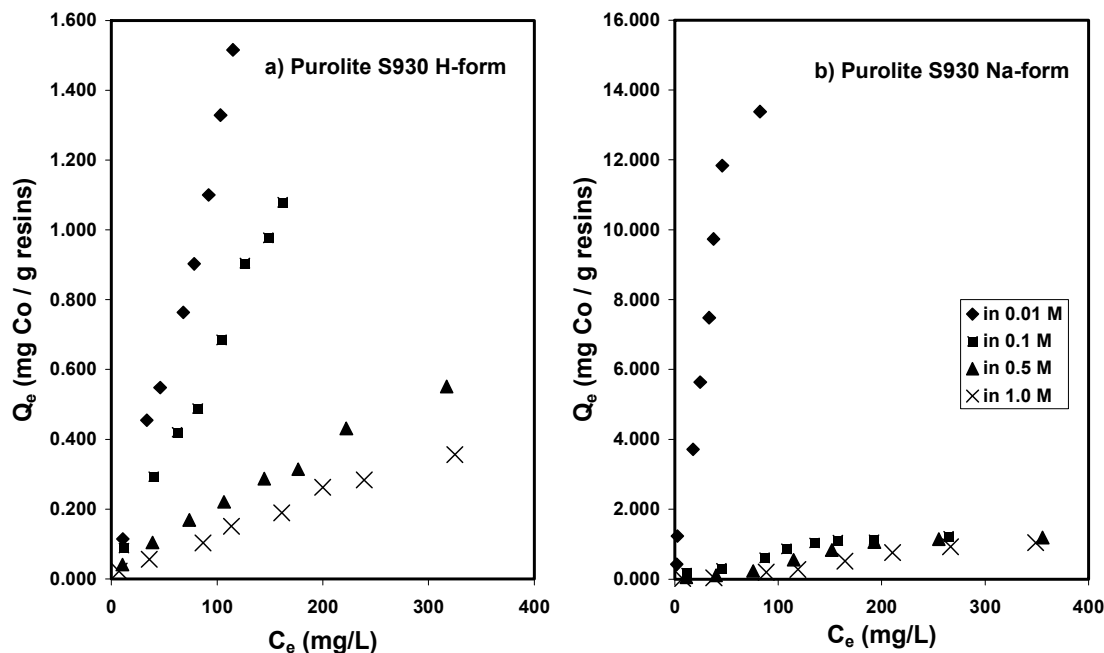

Figure 3. Adsorption isotherms of cobalt-citrate onto a) Purolite S930 H-form and b) Purolite S930 Na-form**.**

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Table 4. Adsorption model parameters of cobalt-citrate onto S930 H-form.

[CA], M	Langmuir parameters			Freundlich parameters		
	Q_m (mg/g)	b (L/g)	R^2	A (mg/g)	1/n	R^2
0.01	3.86	3.8×10^{-3}	0.97	1.0×10^{-2}	1.05	0.99
0.1	3.47	2.2×10^{-3}	0.99	8.0×10^{-3}	0.95	0.99
0.5	0.71	4.4×10^{-3}	0.99	6.7×10^{-3}	0.76	0.99
1.0	0.15	2.3×10^{-2}	0.98	4.3×10^{-3}	0.75	0.98

Table 5. Adsorption model parameters of cobalt-citrate onto S930 Na-form.

[CA], M	Langmuir parameters			Freundlich parameters		
	Q_m (mg/g)	b (L/g)	R^2	A (mg/g)	1/n	R^2
0.01	11.64	4.6×10^{-2}	0.98	0.36	0.87	0.96
0.1	0.99	1.8×10^{-2}	0.92	2.6×10^{-2}	0.73	0.95
0.5	0.62	7.9×10^{-3}	0.96	2.7×10^{-3}	1.11	0.95
1.0	0.53	3.9×10^{-3}	0.94	1.0×10^{-3}	1.20	0.94

Resin hydrolysis

Figures 2 and 3 suggest the adsorption capacities of nickel and cobalt citrate complexes were significantly higher on the Na-form of resin. The iminodiacetic based resin used in this study is weakly acidic by nature and thus selective for the hydronium ion. This resin can only be partially hydrolysed in water. In the Na-form, S930 reacts alkalically in water (this is also referred to as hydrolysis of the resin) which promotes the rise of solution pH value as the counter ion exchanges with H^+ from water:

$$RNa_2 + 2\ H_2O \Leftrightarrow RH_2 + 2\ Na^+ + 2\ OH^- \tag{5}$$

The interactions of the H and Na forms of the resins with water are reflected by the change in solution pH after adsorption (see Table 7). As shown, the H-form of resin resulted in a decrease in pH, whereas Na-form of resin promoted a rise in pH. These results confirm the stability of the H-form of resin toward hydrolysis and the ability of Na-form to hydrolyze in solution at various pHs. Hydrolysis of resins occurs only at low metal concentrations. Although the pH may still rise at high metal concentrations, hydrolysis become less prevalent and only minimal change in pH is observed.

The equilibrium pH value has a very strong effect on the distribution of metals. The hydrolysis of the resin results in the formation of a three-component ion exchange

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system $H_3O^+/Na^+/M^{2+}$ (<u>11</u>). Metal sorption starts when the pH rises to the range where most acidic ion exchange sites start to exchange hydronium ion for metal and the capacity reaches the maximum value in the pH range where all the ion exchange sites take part in the reaction and the functional group is able to form chelate rings with the metal cations. This is confirmed by the increase in nickel and cobalt citrate adsorption with decreasing citric acid concentrations in the metal complex solutions (see Tables 2-5). It should be noted that an increase in solution pH can also promote the precipitation of metals. This additional reaction should be considered when interpreting the effect of resin hydrolysis and solution pH on metal adsorption. In general, iminodiacetic-based resins are used in the Na-form because they enhance metal sorption as they undergo hydrolysis to generate a solution pH of 2-5 (<u>17</u>, <u>18</u>, <u>19</u>).

At the lower pH, hydrogen ions will attach to the carboxyl group and nitrogen atom of the iminodiacetatic functional group thus making the resin surface net positive (<u>11</u>):

$$RCH_2N(COOH)_2 + H^+ \rightarrow [RCH_2\text{-}NH\text{-}(COOH)_2]^+ \tag{6}$$

The corresponding anion, cations and metal species in solution at equilibrium are generated through the following equations. Citric acid (H_3L) dissociates as:

$$H_3L \Leftrightarrow H_2L^- + H^+ \tag{7}$$

$$H_2L^- \Leftrightarrow HL^{2-} + H^+ \tag{8}$$

$$HL^{2-} \Leftrightarrow L^{3-} + H^+ \tag{9}$$

The stability of these species is shown in Figure 4. The corresponding nickel and cobalt citrate complexes formed and their stability constants are shown in Table 6.
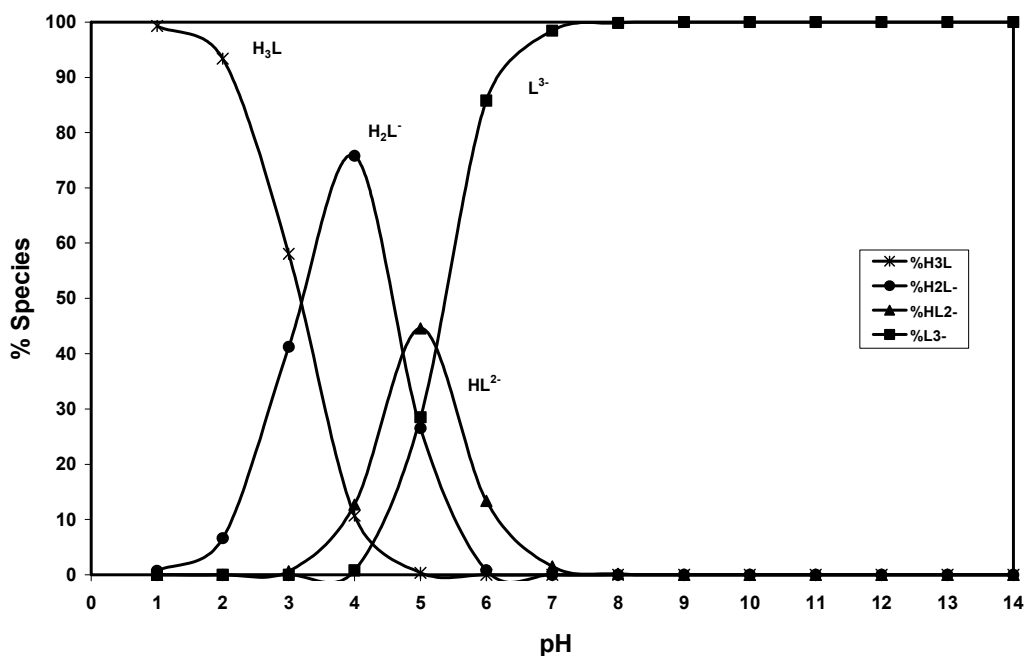
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Figure 4. Citric acid speciation as a function of solution pH.

Table 6. Complexation and stability constants of nickel and cobalt citrate complexes.

System	Complexation / Chelation	Stability constants ([20])
Ni-citrate	$Ni^{2+} + H_2L^- \Leftrightarrow NiH_2L^+$	$pK_1 = 1.75$
	$Ni^{2+} + HL^{2-} \Leftrightarrow NiHL^0$	$pK_2 = 3.19$
	$Ni^{2+} + L^{3-} \Leftrightarrow NiL^-$	$pK_3 = 5.11$
Co-citrate	$Co^{2+} + H_2L^- \Leftrightarrow CoH_2L^+$	$pK_1 = 1.25$
	$Co^{2+} + HL^{2-} \Leftrightarrow CoHL^0$	$pK_2 = 3.19$
	$Co^{2+} + L^{3-} \Leftrightarrow CoL^-$	$pK_3 = 4.83$

Table 6 suggests the positively charged Ni^{2+} and NiH_2L^+ species will be dominant at low pH. This suggests that low pH is unlikely to promote the adsorption of the metal complexes because of the similarities in the surface charge of the resin and nickel complexes. The uptake of negatively charged anions will be promoted ([21]). These results suggest the importance of solution pH in the uptake of metal complexes onto the resin. This study has shown that the solution pH can be varied either by directly changing the pH of solution by addition of an acid or alkali or by the choice of Na-form of the resin. Overall, weakly acidic conditions promote adsorption of Ni and Co

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complexes as the acidic sites on the resins are able to participate in the exchange of ions and complexes. Acidic solutions promote the adsorption of H^+ and generation of net positive surfaces that repel the adsorption of positively charged metal complexes but promote the adsorption of negatively charged anions.

Table 7. The pH of solutions before and after adsorption of nickel citrate onto S930.

Ni (mg/L)	pH before adsorption (Both H- & Na- forms)				pH after adsorption							
					S930 H-form				S930 Na-form			
	Citric Acid [M]				Citric Acid [M]				Citric Acid [M]			
	0.01	0.1	0.5	1.0	0.01	0.1	0.5	1.0	0.01	0.1	0.5	1.0
0	2.55	2.07	1.63	1.56	2.33	2.02	1.62	1.54	5.42	2.66	1.76	1.62
100	2.52	2.08	1.86	1.50	2.25	2.02	1.79	1.47	4.93	2.54	2.00	1.74
500	2.26	1.98	1.70	1.42	2.11	1.94	1.65	1.40	3.69	2.35	1.89	1.68
1000	2.14	1.89	1.59	1.34	2.03	1.86	1.55	1.32	2.94	2.22	1.80	1.60
1500	2.07	1.83	1.51	1.27	1.97	1.82	1.49	1.27	2.77	2.13	1.74	1.56
2000	1.98	1.79	1.46	1.22	1.93	1.78	1.45	1.22	2.67	2.07	1.69	1.52

Metal stripping

Loaded resins were separated by filtration and stripped with 2 M HNO_3. The efficiency of metal elution was plotted as a function of metal concentrations used in adsoprtion (see Figures 5 and 6). The range of concentration of metal covers the leachable metal dissolution from bioleaching of low grade nickel laterite ores reported by Tang (7). It appears the concentration of the metal have no significant effect on the elution of the metals from the resin. However, metals prepared from lower citric acid concentrations were desorbed more effectively. This suggests that metal complexes were adsorbed weakly on the resin rather than through a more stable surface complexation. Surface complexation would require the metals to dissociate from its own citrate complex and to then react with iminodiacate functional group. The stability of the adsorbed Ni and Co complexes (reflected by poor elution efficiency) at the lower pH (or higher citric acid concentration) in Figures 5 and 6 confirms the possible surface complexation under these conditions.
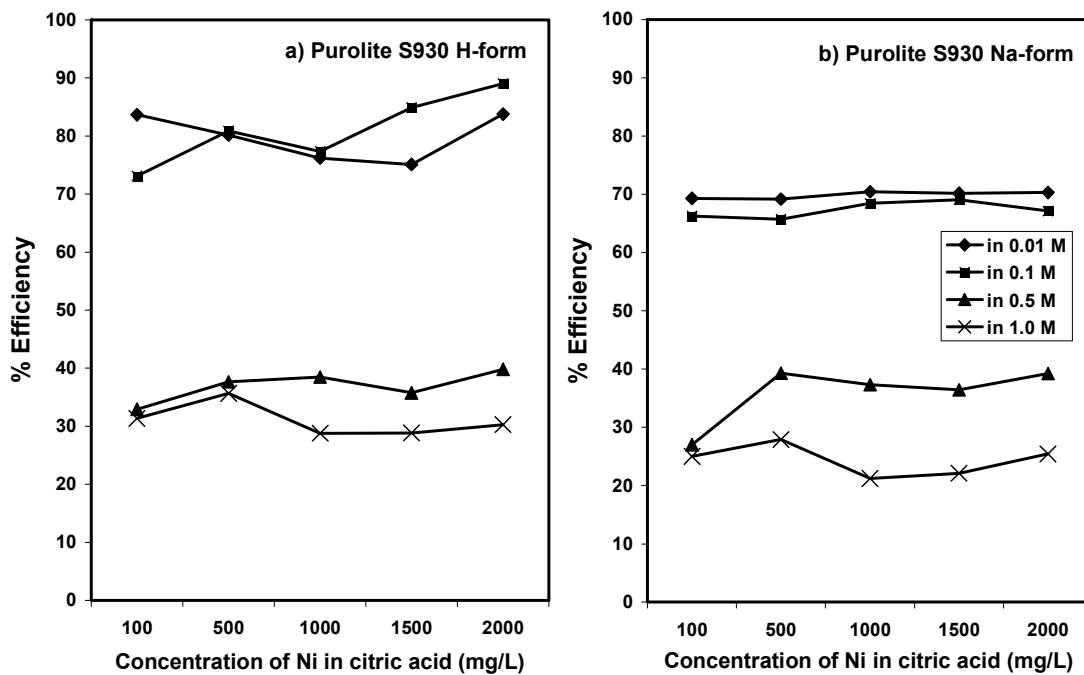
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Figure 5. The % efficiency of nickel elution as a function of nickel concentrations from a) H- form and b) Na form of resins.
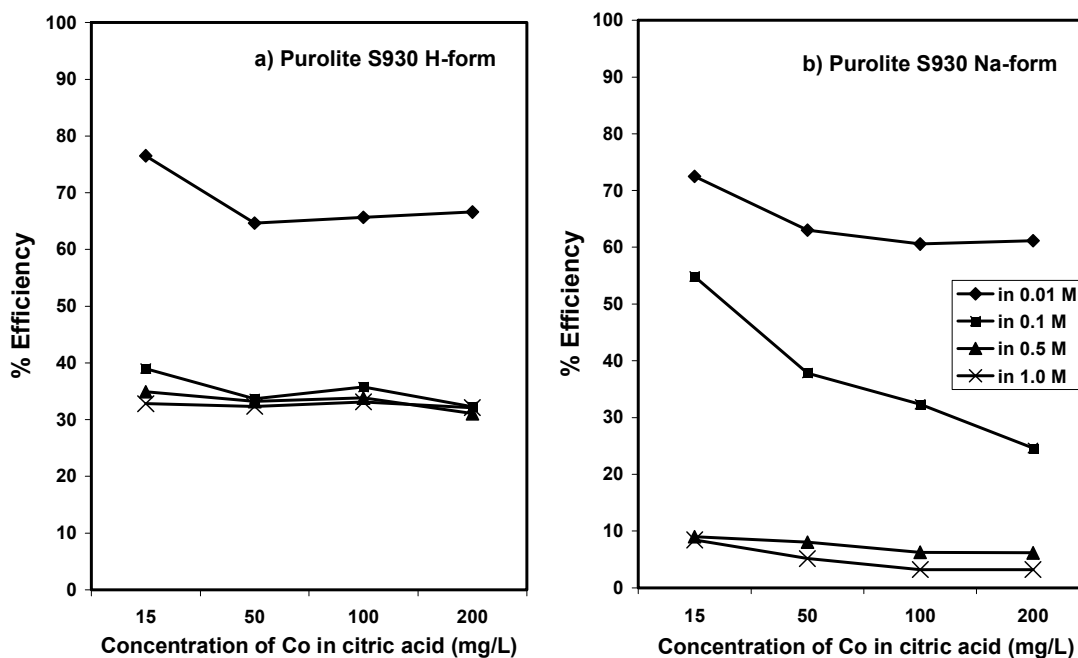
Figure 6 The % efficiency of cobalt elution as a function of metal concentrations from a) H- form and b) Na form of resins.

CONCLUSIONS

Adsorption of nickel and cobalt citrate complexes onto an iminodiacetate chelating resin (Purolite S930) was found to behave as follows:

1. Nickel and cobalt citrate adsorption were found to follow both the Langmuir and Freundlich models suggesting both metals adsorbed on S930 by monolayer and multilayer adsorption mechanisms.
2. Metal adsorption was found to be significantly influenced by the solution pH and the ability of the resin to hydrolyse in solution.
    a. At low pH, H-form of the resin does not hydrolyse effectively in solution, whereas Na- form hydrolyses resulting in an increase in solution pH. An increase in solution pH allows acidic sites on the resin to exchange resulting in uptake of metal complexes.
    b. At high pH, the selective uptake of H^+ on the resin resulted in the development of a net positive charge on the resin surface. This resulted in the repulsion of positively charged metal complexes but promoted the adsorption of citrate anions.
3. Metal elution was found to be independent of the metal concentrations used in the adsorption. However, citric acid concentration appears to have a significant effect on the efficiency of metal elution. It is proposed that metal complexes are stable at high pH and are adsorbed intact. The result is a weak adsorption that is easily eluted (70-90%). At high pH, dissociation of metal complexes from their citrate anions allows the metals to adsorb on the resin by surface complexation. This results in a stable adsorption that could not be desorbed efficiently (4-40%).

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ACKNOWLEDGEMENT

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KEY WORDS

Adsorption, Metal-organic complexes, Bioleaching

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