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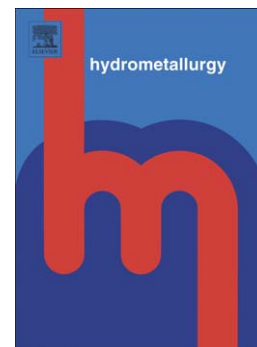
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Ion-exchange equilibria of Ni²⁺, Co²⁺, Mn²⁺ and Mg²⁺ with iminodiacetic acid chelating resin Amberlite IRC 748

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ABSTRACT

A chelating ion exchange resin, Amberlite IRC 748 which carries an iminodiacetate acid functional group has been investigated for its adsorption properties towards Ni, Co, Mn and Mg from aqueous solution. Experiments were conducted through batch adsorption by varying the resin volume at three different pH values. It was observed that the amount of metals adsorbed by the resin increased with increasing pH. The applicability of both the Langmuir and Freundlich isotherms were analyzed on the equilibrium data obtained. In all cases, the Langmuir isotherm shows an excellent fit to the experimental data while the Freundlich isotherm failed to predict the data at higher loadings. The resin exhibited high selectivity towards nickel ($K = 58000$) and cobalt ($K = 16800$), which can be easily separated from magnesium and manganese at pH 4 and 5. The obtained values of the equilibrium constant K (l/mol) matched the expected order of selectivity of this resin which is $Ni > Co > Mn > Mg$. The results also showed that the adsorption mechanisms of these metals onto the resin follow a combination of monolayer and multilayer adsorption.

Keywords Ion exchange; Equilibrium isotherms; Iminodiacetic acid; Chelating resin IRC 748; Nickel; Cobalt; Manganese; Magnesium.

1. Introduction

Nickel is one of the most important metals consumed by the stainless steel industry, which has been growing at the rate of 5-6% per annum in the last 20 years. It is extracted from both sulfide and lateritic oxide ores in about 20 countries around the world. In 1968, laterites supplied 28% of global nickel demand, rising to 42% in 2003 which is expected to increase to 51% by 2012 (Sudol, 2005).

The four major commercial routes that have been used for processing nickel laterite ores are ferronickel smelting, matte smelting, the reduction roast/ammoniacal leaching process known as the Caron process and pressure acid leaching (PAL). Among those processes, PAL is the only existing laterite process which exhibits good cobalt recovery (Kyle, 1996; Reid, 1996). This technology leaches the ore in sulfuric acid at elevated temperature and pressure to extract nickel and cobalt. Counter-current decantation (CCD) is then used to separate the solids and liquids. The first stage overflow pregnant leach liquor is passed on for further processing whilst the last stage underflow slurry is neutralized and pumped to a tailings dam. It was reported that up to 10% of the nickel and cobalt can report to the tailing as a result of settling inefficiency in the CCD stages (Nicol and Zainol, 2003).

In the previous work, the authors have established the technical feasibility of applying ion exchange to recover the nickel and cobalt from the CCD underflow. Chelating resin IRC 748 was chosen for the work which covered small scale batch tests, to the continuous six-stage mini plant and pilot plant.

Chelating ion exchange resins which are also known as complexing or specific ion resins are designed to have high specificity for an ion or groups of ions. These types of ion exchange resins adsorb metal ions through a combination of ionic and coordinating interactions instead of the simple electrostatic interactions in conventional cation or anion ion exchange (Harland, 1994). As the consequence, chelating resins offer greater selectivity than conventional resins. IRC 748 studied in this work, has imino-diacetic acid (IDA) as a functional group anchored to the styrene divinyl-benzene chains. Thus, the chelating group is tri-dentate containing one amino and two carboxylic oxygens as donor atom (Harju and Krook, 1995).

The aim of the present study is to investigate the fundamental aspects of loading metal ions on the resin. The loading behavior of the four metal ions involved in the PAL system namely Ni, Co, Mg and Mn were examined at various pH values. The equilibrium data obtained were then analyzed using the two of the most frequently used adsorption models. In addition, the coordination behavior of the metal ions on the resin was also considered in this study. The information gained from this study increases the understanding of the resin behaviour and selectivity.

2. Materials and methods

2.1. Reagents and ion exchange resin

Synthetic solutions of nickel sulfate, cobalt sulfate, magnesium sulfate and manganese sulfate were prepared with deionized water and employed for the study. All chemicals were of analytical grade. The solutions were adjusted to the required pH 3, 4 and 5 using dilute sodium hydroxide or sulfuric acid.

A commercial weak acid chelating resin IRC 748 (formerly known as IRC 718) supplied by Rohm and Haas was used in this study. This resin is characterized by imino-diacetate acid functional group as shown in Fig 1. The properties of the resin as reported by the supplier are given in Table 1. The resin obtained in the sodium form was acid-conditioned with 1 M sulfuric acid to remove the impurity and to convert the exchange sites to the desired H^+ form. The resin was washed to pH 5 with distilled water. The ion exchange resin beads in the size range of 355-500 μm were used in this study.

The resin was subjected to a titration procedure in order to establish various degrees of protonation and to calculate the total exchange capacity of the resin. The result showed that there were three equivalent points at pH values of 6.3, 8.0 and 10.6 at which the three protons are being neutralized. This observation is in agreement with the result obtained by other researchers related to the imino-diacetic type chelating resin (Heitner-Wirguin and Ben-Zwi, 1970; Szabadka and Inczedy, 1980). A value of 1.13 eq/l was found at the second equivalent point which is the total exchange capacity in terms of the acetate chelating groups contained in this resin. A capacity of 1.13 eq/l corresponds to a maximum capacity of 33.2 g/l or 89.7 g/kg for nickel. This value is about 94% of the value quoted by manufacturer which is 1.25 eq/l.

2.2 Equilibrium studies

This study was conducted using the automated titration set-up. This equipment maintained the desired pH by the addition of sodium hydroxide to neutralize the protons released by exchange of metal ions on the resin. It was used in a combination with a data acquisition computer utilizing Labview software.

Typically, 250 ml of each metal ion solution was repeatedly equilibrated with different calculated volumes (8-60 ml) of wet resin based on the resin capacity. The resin was then filtered, washed and stripped of the loaded metal ion. In this study, the resin was observed to reach its equilibrium after 4 hours. Elution of the loaded resin was carried out using 1 M sulfuric acid. Both the equilibrium solution and the eluate solution were sampled and analyzed using AAS or ICP which determined the amount of metal loaded onto the resin at the equilibrium. Each pair of equilibrium solution and loaded resin values was used as an equilibrium point on the isotherm for each metal at each pH.

3. Results and discussion

Langmuir and Freundlich models are the two frequently used isotherms to describe the adsorption equilibrium ion exchange relation between the solid phase and liquid phases (Grimshaw and Harland, 1981; Slater, 1991). These isotherms relate metal loading per unit volume of resin to the concentration of metal ion in the solution at the equilibrium state given the temperature is constant. The Langmuir isotherm assumed that a surface is composed of a finite of equivalent adsorption sites and the maximum coverage is obtained when all sites are filled (Langmuir, 1918). The Langmuir equation is commonly expressed as

$$RM = \frac{KR_{\max}C_e}{1 + KC_e} \quad (1)$$

where C_e is the equilibrium concentration of the metal ion in solution (mol/l), RM is the amount of metal ion adsorbed onto the resin (mol/l), R_{\max} is a constant related to the area occupied by a monolayer of metal ion and reflects the adsorption capacity (mol/l) of the

resin and K is an adsorption equilibrium constant which is a direct measure of adsorption intensity (l/mol). This isotherm can be rearranged to a linear form. The corresponding parameters of K and R_{max} can be determined from the slope and intercept of a plot of C_e/RM versus C_e .

The Freundlich isotherm is an empirical equation that describes adsorption based on heterogeneous surface energy (Freundlich, 1906) and is shown to be satisfactory for low metal ion concentrations. The Freundlich equation is presented as

$$RM = a(C_e)^b \quad (2)$$

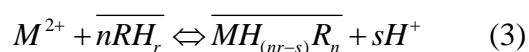
The constant b , normally varies between 0 and 1 is a measure of adsorption intensity or surface heterogeneity. When the b value is close to zero, it means that the adsorption becomes more heterogeneous. A value above one indicates unfavorable adsorption. Similarly, the Freundlich isotherm can be converted to a linear form by taking the natural logarithm of both sides. The empirical constant of a and b can be derived from the intercept and slope of $\log RM$ versus $\log C_e$.

The adsorption isotherm coefficients obtained from the slopes and intercepts of the linear plots are listed in Table 2. The extent of fit of the calculated loading to the experimental data was measured using the coefficient of determination R^2 . The resulting values of R^2 which are high for both Langmuir and Freundlich isotherms suggests that the adsorption of the four metal ions are applicable with the two isotherm models. Its also indicates that the adsorption of these metal ions on the resins by mean of combination of monolayer and multilayer adsorption mechanisms.

The value of Langmuir equilibrium constant K , which reflects quantitatively the affinity between the resin and metal ions matched the expected order of selectivity of this resin which is $Ni > Co > Mn > Mg$ (Rohm and Hass, 1998). Even though there was no available data for manganese at pH 3 and 4, the higher K value than magnesium at pH 3 should give enough hints that the K value of this metal at higher pH should be higher. The resin reached its maximum capacity for both nickel and cobalt at pH 4 and 5 and whilst for manganese the resin only reached 2/3 of its capacity even at high pH value.

The resin exhibited high selectivity for nickel ($K = 58000$) and cobalt ($K = 16800$) which suggest that these metal ions can be easily separated from magnesium and manganese at pH 4 to 5. The value of b , which decreased with increasing pH, shows that the adsorption of these metal ions is favored at higher pH. The plot of the observed equilibrium adsorption isotherms of Ni, Co, Mg and Mn at pH 3, 4 and 5 as compared to the Langmuir and Freundlich isotherm are shown in Figures 2 to 5. In all cases, the Langmuir is shown to give a better fit than the Freundlich isotherm especially at higher loading values.

An alternative approach to the development of an adsorption isotherm is by expressing the equilibrium involved in the adsorption reaction on the resin. By assuming at this stage that the number of protons exchanged by one metal ion adsorbed is not specified, one can write a general reaction for the uptake of divalent metals by an imino-diacetic resin as



where $n = 1$, $r = 2$ and $s = 1$ or 2 , M is a divalent metal ion (mol/l), $\overline{nRH_r}$ is the concentration of unused or free resin sites (mol/l) and $\overline{MH_{(nr-s)}R_n}$ is the metal concentration on the resin (mol/l).

Equilibrium constant can be written for this general adsorption equation as

$$K_0 = \frac{[\overline{MH_{(nr-s)}R_n}][H^+]^s}{[M^{2+}][\overline{RH_r}]^n} \quad (4)$$

As the pH is maintained constant, the above equation can be simplified to produce a second constant K

$$K = \frac{[\overline{MH_{(nr-s)}R_n}]}{[M^{2+}][\overline{RH_r}]^n} \quad (5)$$

Introducing the maximum concentration of functional group on the resin as $[\overline{R_{\max}}]$ and rearranging gives

$$[\overline{MH_{(nr-s)}R_n}] = \frac{K[\overline{R_{\max}}][M^{2+}]}{1 + K[M^{2+}]} \quad (6)$$

This equation has the same form as the Langmuir isotherm in Equation 1. Thus the Langmuir isotherm corresponds to the equilibrium involved in the adsorption of metal ions on this type of resin.

Two limits can then be considered depending on the concentration of metal ions.

$$\text{For } K[M^{2+}] \ll 1, [\overline{MH_{(nr-s)}R_n}] = K[\overline{R_{\max}}][M^{2+}] \quad (7)$$

Thus, the extent of loading of metal on the resin is linearly related to the equilibrium concentration in solution. This condition is satisfied if the resin has a low affinity for the metal (low K) and/or the metal concentration in solution is low.

The second limit arises if $K[M^{2+}] \gg 1$,

$$[\overline{MH}_{(nr-s)}R_n] = [\overline{R}_{\max}] \quad (8)$$

This corresponds to a fully loaded resin which results from a large K (high affinity for the metal) or high metal ion concentrations in solution. In this case, increasing the solution concentration will not produce further loading of metal onto the resin.

An alternative method of analyzing selectivity data was suggested by Hall et al. (1966) in terms of which the essential features of the Langmuir isotherm can be expressed in terms of a dimensionless separation factor or equilibrium parameter K_R , which is defined by

$$K_R = \frac{1}{1 + KC_0} \quad (9)$$

where C_0 is the initial solution concentration (mol/l) and K is the Langmuir constant (l/mol). The value of K_R can then be used to describe the isotherm by the following

Value of K_R	Type of isotherm
$K_R > 1$	Unfavourable
$K_R = 1$	Linear
$0 < K_R < 1$	Favourable
$K_R \rightarrow 0$	Irreversible

The result of such an analysis is presented in Table 3. As expected from the shape of the isotherms, the values of K_R reflect the favorable equilibrium in all cases, especially at the higher pH values where the values of K_R close to zero. The isotherms for the adsorption of magnesium and manganese ions are almost linear at pH 3 and favorable at pH 4 and 5. This classification also clearly shows that the order of selectivity is Ni > Co > Mn > Mg as suggested previously.

The derivation of the equilibrium constant for metal ions in Equation 5 can be simplified to the following equation

$$\frac{K_0}{[H^+]^s} = K \quad (10)$$

from which,

$$K_0 = K[H^+]^s \quad (11)$$

Thus, if $s = 2$, i.e. two protons are exchanged for divalent each metal ion adsorbed, K should increase by a factor of ten for each unit increase of the pH in order to produce a constant value for K_0 . The values of the K_0 calculated for $s = 2$ and $s = 1$ are given in Table 4.

The results show that K_0 varies with pH in both cases (either 1 or 2 protons involved) and that the general adsorption equation involving only the exchange between protons and the metal ions as written in Equation 3 cannot be the only reaction that takes place during the adsorption process. Taking logs of both sides of Equation 10 yields the

following equation which can be used to obtain a nominal value for the number of protons involved for each metal ion at a particular pH.

$$\text{Log } K = s \cdot \text{pH} + \text{Log } K_0 \quad (12)$$

The results in Table 5 shows that for each metal ion, the nominal number of protons, s , decreases with increasing pH with the slope approaching the theoretical maximum value of 2 at lower pH values for nickel and cobalt. A possible explanation for these observations can be obtained by an analysis of the information gained from the titration results given in the previous section and compared to the typical titration of the resin by the other researchers.

It is possible that the time allowed for the equilibration at pH values below about 6 was not sufficient. This resulted in higher apparent equilibrium pH values for these points. Typical behavior for a polyfunctional weak cationic ion exchange resin with respect to the imino-diacetic species present at different pH values is given in Figure 6 (Heitner-Wirguin and Ben-Zwi, 1970).

From the figure, it is apparent that the resin is fully protonated at pH value below 2.21. On the other hand, the resin in this study has been washed with water to pH about 3.3. As the pH increases the first proton is neutralized by pH 3.99. Thus, at pH values between 2.21 and 4 the resin is composed of functional groups RHH and RH. This explains the average number of protons of 1.5 in this pH range as shown in Table 5. The neutralization of the second proton then commences at pH above 3.99 and that is completely neutralized by pH 7.41. In this pH range the resin is expected to behave as a mixture of the RH and R forms. This is consistent with the number of proton exchanged

of about 1 between pH 4 and 5. However, for some unknown reason, this explanation cannot be applied to the data for loading of magnesium.

Another observation made was that the total of the amount of NaOH added to neutralize the protons released should correlate to the nickel loading at equilibrium. It is expected that the amount of NaOH added would be identical with the amount of nickel loading. This is, however, not the case. It was observed that the equilibrium NaOH added corresponds to a higher nickel loading than was obtained especially at higher pH values and at lower metal loading. It appears that at higher pH values and lower metal loading, sodium was also loading onto the resin and this resulted in higher additions of NaOH to neutralize the extra protons released into the solution.

4. Conclusions

The adsorption equilibrium of Ni, Co, Mn and Mg on the imino-diacetic acid chelating resin IRC 748 has been discussed in this report. Modeling of the equilibrium adsorption isotherms of metal ions on the resin showed that the Langmuir isotherm provided an excellent fit while the Freundlich isotherm failed to predict the loading at high loading. The resin proves to have high selectivity for nickel and cobalt which suggests that these metals can be easily separated from magnesium and manganese at pH 4 and 5. The magnitude of the Langmuir equilibrium constant of each metal ion was found to follow the order of selectivity of the resin which is $\text{Ni} > \text{Co} > \text{Mn} > \text{Mg}$. The number of protons exchanged with the metal ions in solution was found to vary with the pH of solution. This anomaly can be explained by the protonation states of the iminodiacetic species present at different pH values. Some sodium co-loading on the

resin also seems apparent at high pH which might contribute to the variation of the K_0 values with pH. Thus, further effort will be directed to incorporating the effect of added sodium into the metal ions equilibrium system.

5. Acknowledgements

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Figure 1. Chemical structure of Amberlite IRC 748 chelating resin

Figure 2. Adsorption isotherms of nickel onto IRC 748. (a) pH 3 (b) pH 4 (c) pH 5 (conditions: resin volume = 8-85 ml; initial $M^{2+} = 2.5$ g/l; vol. solution = 250 ml; $T = 25 \pm 1^\circ\text{C}$; time = 4 hours).

Observed (◆), Langmuir (.....), Freundlich (-----)

Figure 3. Adsorption isotherms of cobalt onto IRC 748. (a) pH 3 (b) pH 4 (c) pH 5 (conditions as Fig. 2 except resin volume = 5-100 ml)

Observed (◆), Langmuir (.....), Freundlich (-----)

Figure 4. Adsorption isotherms of magnesium onto IRC 748. (a) pH 3 (b) pH 4 (c) pH 5 (conditions as Fig. 2 except resin volume = 10-72 ml)

Observed (◆), Langmuir (.....), Freundlich (-----)

Figure 5. Adsorption isotherms of manganese onto IRC 748 at pH 3 (adsorption conditions as Fig. 2 except resin volume = 12-100 ml)

. Observed (◆), Langmuir (.....), Freundlich (-----)

Figure 6. Stability regions of the iminodiacetate species

Figure 1

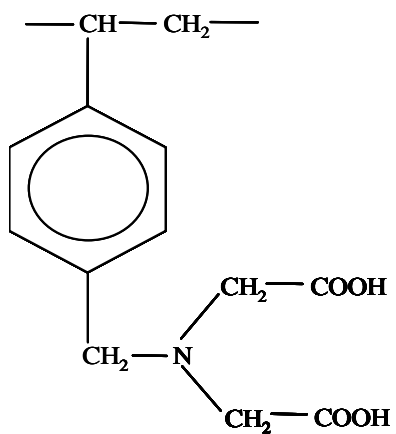


Figure 2

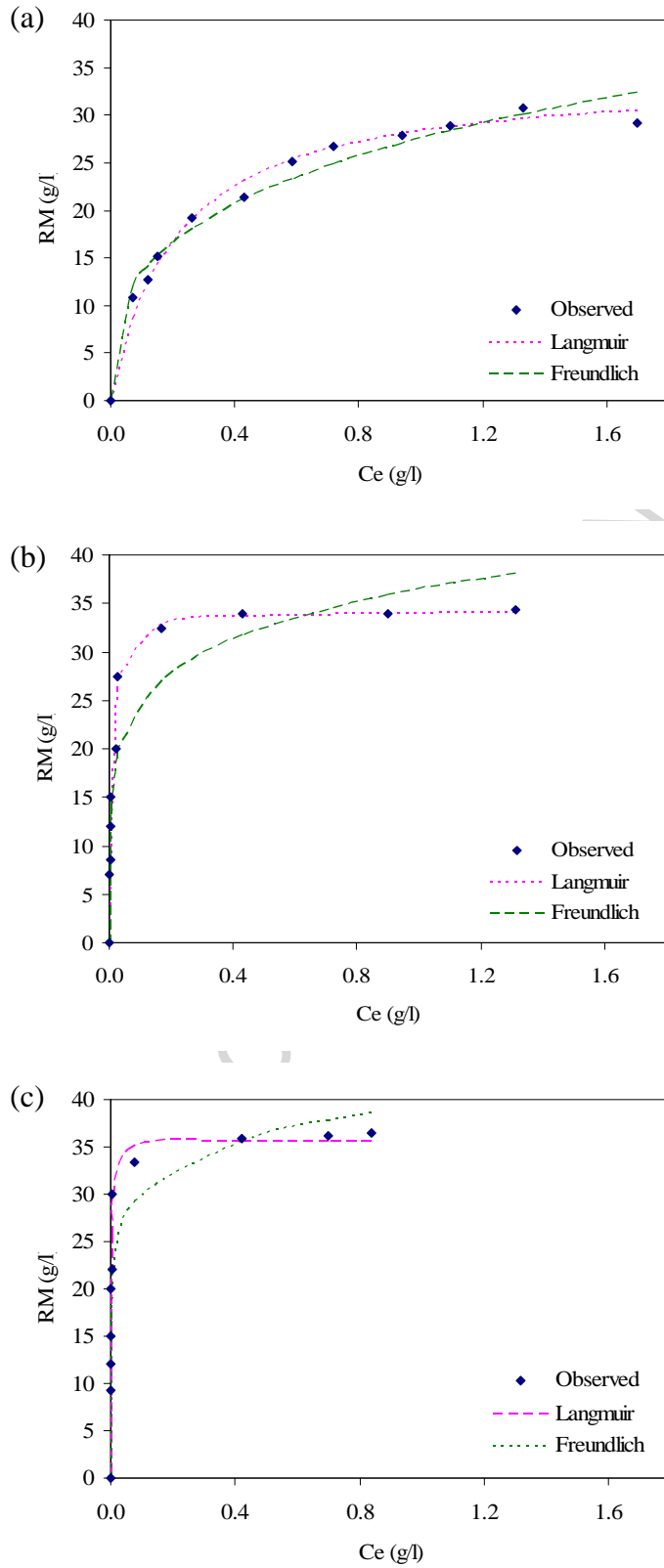


Figure 3

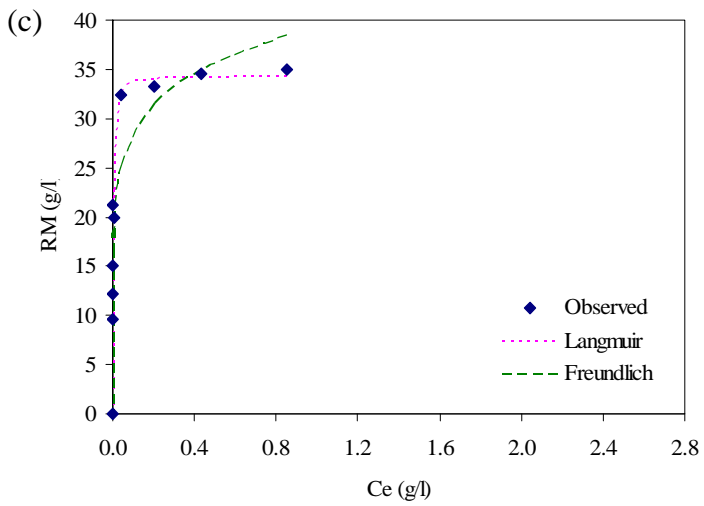
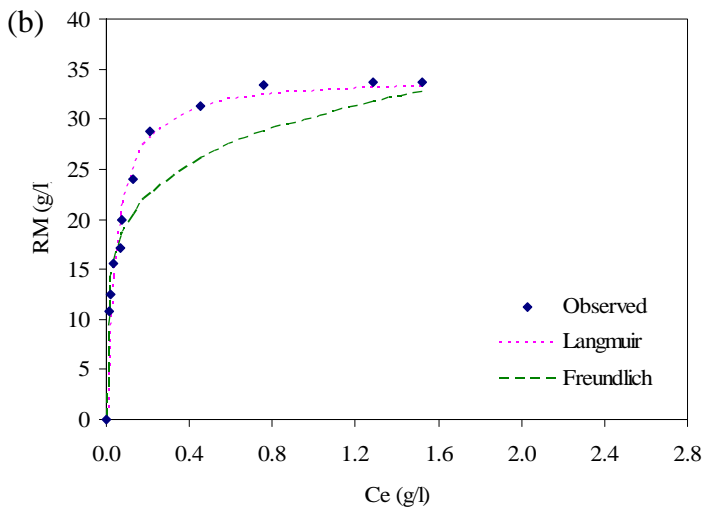
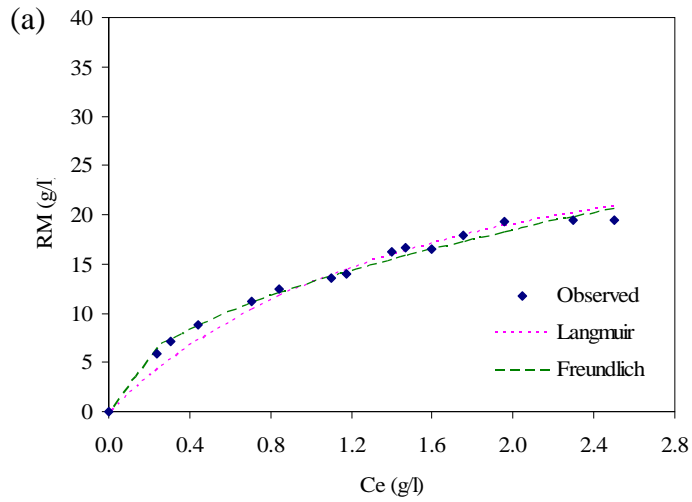


Figure 4

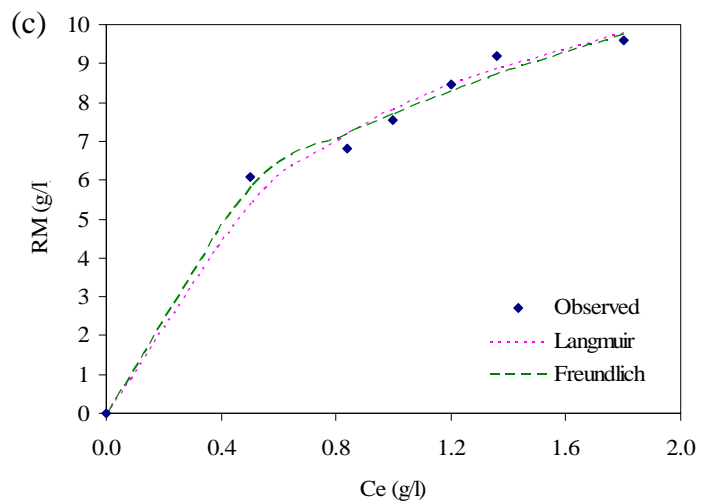
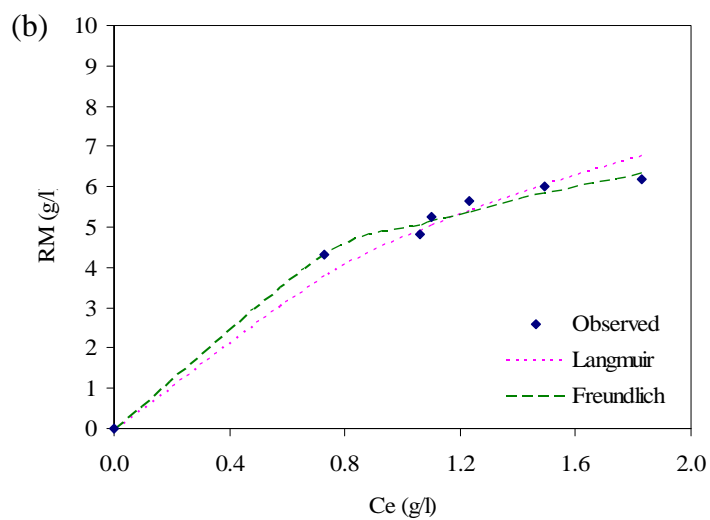
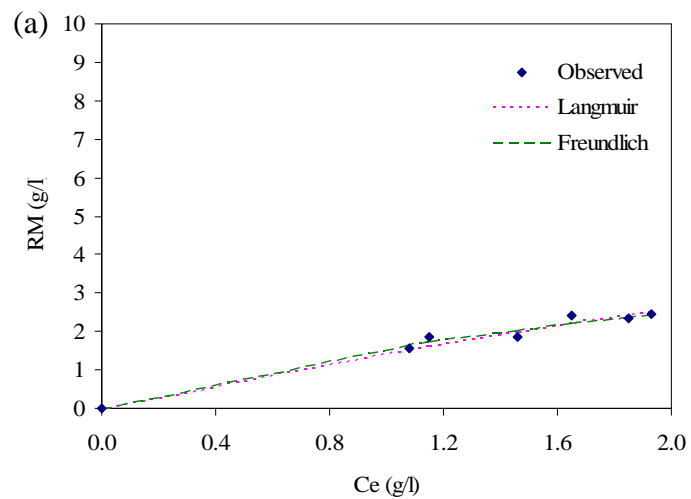


Figure 5

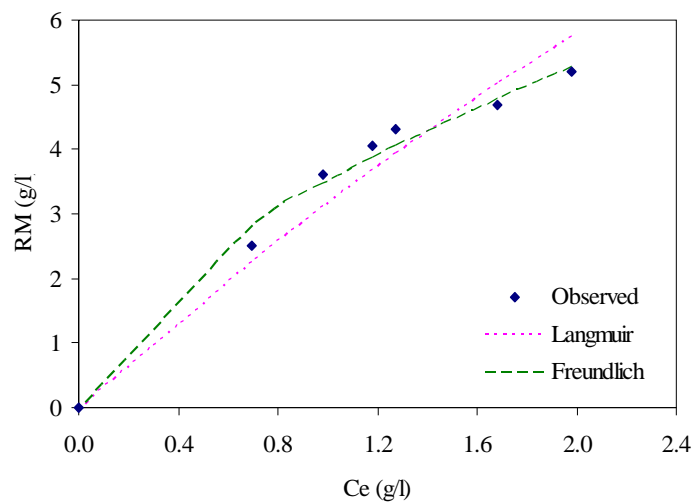
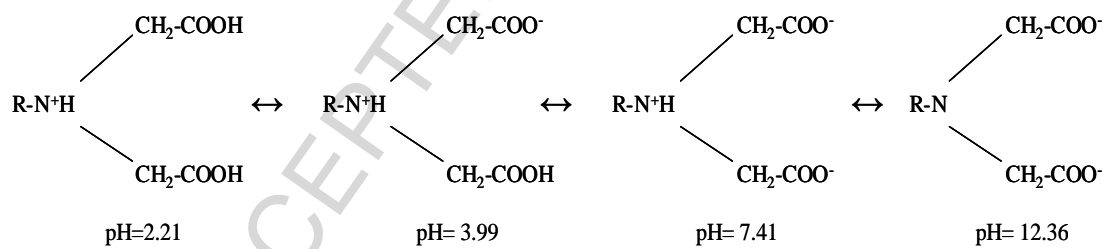


Figure 6



Tables

Table 1. Physical and chemical properties of the Amberlite IRC 748

Source	Rohm and Haas, USA
Ionic form as shipped	Sodium
Functional group	Iminodiacetic acid
Matrix	Styrene DVB
Structure	Macroporous
Bead size	0.3-1.1mm
Bulk density (g/l)	685-760
Total capacity (eq/l)	1.25
Operating pH range	1.5-14

Table 2. Comparison of the adsorption constants obtained for the Langmuir and Freundlich adsorption isotherms at different pH values^a

Metal	pH	Langmuir Constants				Freundlich Constants		
		Observed R_e (mol/l)	R_{max} (mol/l)	K (l/mol)	R^2	a (mol/l)	b	R^2
Nickel	3	0.50	0.57	320	1.00	2.07	0.36	0.99
	4	0.59	0.59	8500	1.00	1.30	0.18	0.84
	5	0.62	0.61	58000	1.00	1.04	0.11	0.77
Cobalt	3	0.33	0.46	61	0.98	1.70	0.50	0.97
	4	0.52	0.53	1600	1.00	1.40	0.23	0.92
	5	0.59	0.60	16800	1.00	1.35	0.15	0.87
Magnesium	3	0.10	0.28	7	0.85	0.58	0.70	0.80
	4	0.26	0.38	29	0.97	0.98	0.49	0.93
	5	0.39	0.55	37	0.97	1.20	0.41	0.93
Manganese	3	0.09	0.26	17	0.91	1.11	0.72	0.94

^a Temperature 25° C, initial solution concentration 2.5 g/l, stirring rate 200 rpm

Table 3. Calculated values of the constant K_R for metal ions

Metal	pH	K_R
Nickel	3	0.0684
	4	0.0028
	5	0.0004
Cobalt	3	0.2821
	4	0.0145
	5	0.0014
Magnesium	3	0.6122
	4	0.2759
	5	0.2300
Manganese	3	0.5638

Table 4. Calculated values of the thermodynamic constant K_0 assuming either 2 or 1 protons exchanged/metal ion

Species	pH	$[H^+]$ (mol/l)	K (l/mol)	$K_0 = K.[H^+]^2$ (mol/l) x 10^{-6}	$K_0 = K.[H^+]$
Nickel	3	10^{-3}	320	320.00	0.32
	4	10^{-4}	8500	85.00	0.85
	5	10^{-5}	58000	5.80	0.58
Cobalt	3	10^{-3}	61	61.00	0.06
	4	10^{-4}	1600	16.00	0.16
	5	10^{-5}	16800	1.68	0.17
Magnesium	3	10^{-3}	7	7.00	7×10^{-3}
	4	10^{-4}	29	0.29	29×10^{-4}
	5	10^{-5}	37	0.00	37×10^{-5}

Table 5. Number of protons exchanged s for each metal ion adsorbed on the resin within the pH range tested

Metal ions	pH range	Protons exchanged, s
Nickel	3-4	1.5
	4-5	0.8
Cobalt	3-4	1.5
	4-5	1.3
Magnesium	3-4	0.6
	4-5	0.4