

Short communication

Separation and recovery of lead from a mixture of some heavy metals using Amberlite IRC 718 chelating resin

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Abstract

A weakly acidic ion exchanger resin containing the iminodiacetate group has been used to separate and recover metal ion from their mixtures. Batch and column methods were applied for the separation studies. Previous study on the kinetics and thermodynamics shows that the selectivity of this resin for transition metals was quite high as compared to that of alkaline earth metals. The efficiency of this resin in different experimental conditions was established and because of the large difference in the distribution coefficient values separation of the metal ions from their mixture was performed.

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1. Introduction

Chelating resins contain insoluble fictionalized polymer, which can provide more flexible working conditions together with good stability and high capacity for certain metal ions [1–6]. The interest in this type of chelating resins is due to the rapid adsorption of metal ions, greater selectivity and less swelling compared to the analogous organic polymers. Comprehensive reviews have been written on the synthesis, properties and application of chelating resins [7–9]. Without doubt, in analytical as well as in preparative inorganic chemistry, there exists a need for chelating polymers that combine the ease of operation of conventional ion exchangers and the selectivity of organic and analytical reagents [10]. The selectivity of most organic reagents for metals lies predominantly in their ability to form chelates with certain cations. This led to the synthesis of organic polymers containing chelating groups as an exchanging moiety [11–13]. Applications of polymeric resins containing iminodiacetate groups as active sites are well documented in the literature. Among the commercially available products, Chelex-100 (Bio-Rad) is one of the most well characterized regarding its applications [14–18]. Less information exists, however, on the behavior and practical ana-

lytical aspects of similar resin types which may potentially be employed for trace metal pre-concentration [19–22].

An iminodiacetate resin, namely Amberlite IRC 718 has certain active sites, which coordinates with transition metal ions and heavy metal ions. This resin has an ability to function over a wide pH range, hence it can be used effectively to recover heavy metal ions found in a mixture. Due to the weak acidic group, elution from this resin is extremely effective, yielding a concentrate and separate metal ion solution of the desired metal ion. The high selectivity of Amberlite IRC 718 for transition metal ions as well as its activity over a wide pH range makes it a good choice for a variety of specialized processing applications. This resin will also prove useful in the removal and recovery of metal ions from plating baths, drag out and rinse water in the plating industry. The macro reticular structure of this resin ensures its stability in the aggressive solution of plating baths. It also removes hardness to less than 0.1 ppm from brine fed to chloroalkali electrolysis cells. Detailed kinetic and thermodynamic studies on this resin for alkaline earth metals, and a few transition metal ions are studied systematically by the authors [23,24]. Although this resin showed its affinity for transition metal ions, but very little evaluation from the metal separation point of view has been performed on this exchanger. The purpose of the present work is to explore the possibility of a few analytical separations of metal ions from synthetic and actual binary mixtures, namely $Pb^{2+}-Mg^{2+}$, $Pb^{2+}-Fe^{3+}$, $Pb^{2+}-Ni^{2+}$, $Pb^{2+}-Ca^{2+}$.

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Distribution coefficient values in different solutions were studied. The separations are based on the affinity differences of these metal ions toward the chelating resin as a function of pH. Thus, a systematic investigation for the separation of Pb(II) from the nitrate mixtures was performed on Amberlite IRC 718 chelating resin in a batch and column separation mode.

2. Experimental

2.1. Apparatus and reagents

Temperature controlled shaker (SICO) was used for shaking the resin with metal containing aqueous solution. Atomic absorption spectrophotometer (TJA SH 8000) was used for the analysis of trace metals.

Sodium form of Amberlite IRC 718 was a Rohm & Haas Company product (USA). However, before using it for metal exchange it was shaken with dilute NaOH solution for 1 h, decanted and washed with distilled water to remove any sodium ion adhering to the surface. It was then dried at 40 °C. The dried product of a constant mesh size 100–200 mesh was used for further experimental work.

Nitrate salts of the metals studied were of reagent grade. Their solutions were standardised by the EDTA method [25].

2.2. Ion exchange capacity

Ion exchange capacities were determined by batch process. The pre-washed resin in sodium form (0.5 g) was equilibrated with 50 ml of a 0.1 M solution of metal nitrate in a stoppered flask for 24 h with constant shaking at room temperature. The content of the flask was filtered and the filtrate was titrated complexometrically for the metal ions left unexchanged with sodium ions. Ion exchange capacities are given in Table 1.

2.3. Distribution coefficient (K_d) determination

A batch equilibrium method was employed for the determination of distribution coefficient of Pb^{2+} , Mg^{2+} , Ni^{2+} , Fe^{3+} and Ca^{2+} on this resin. The effect of different pH on the uptake of metal ions by Amberlite IRC 718 was studied using the following procedure.

A 0.25 g of the resin was suspended in 50 ml of 300 ppm metal solution at different pH and in distilled water and was equilibrated for 24 h. The resin after equilibration was separated by

decantation and was washed with distilled water. The filtrate and washings were analysed for the metal content left unexchanged in the solution by Atomic absorption spectrophotometer. The distribution coefficient K_d was then calculated using the equation given below.

$$K_d = \frac{I - F}{F} \frac{V}{W} \quad (1)$$

where I and F are the amounts of metal ions added and left unexchanged after equilibrium, V the total volume of the solution taken in milliliters and W is the weight of the resin used in grams. Amberlite IRC 718 was used in sodium form. It was washed with distilled water to remove the surface adhered sodium ions and dried at 50 °C.

3. Results and discussion

The over all process can be shown as:

Exchange: This resin in sodium form was then equilibrated with metal ions thus displacing the sodium ions inside the resin with the di and tri valent metal ions as shown in Eq. (2).

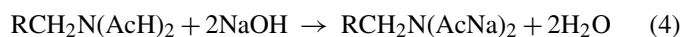


The metal ions adsorbed by the resin was then eluted with different eluents depending on the K_d values.

Regeneration: After the metal elutions were complete the resin was regenerated to hydrogen form using mineral acids such as HCl.



Conversion to Na form: The resin in H^+ form is then converted to the Na^+ form by passing a dilute sodium hydroxide solution on it.



Thus, the hydrogen ion in the resin gets exchanged with the sodium ions. The resin in sodium form is then washed with distilled water and is recycled back to the system.

Ion exchange capacities of Amberlite IRC 718 for Mg^{2+} , Ca^{2+} , Fe^{3+} , Ni^{2+} and Pb^{2+} are given in Table 1. Ion exchange capacities of Amberlite IRC 718 for Mg^{2+} , Ca^{2+} , Fe^{3+} , Ni^{2+} and Pb^{2+} were found to be 2.13, 2.3, 2.55, 1.73 and 3.96 meq g^{-1} , respectively. Since this resin is a weak cation exchanger with a chelating group responsible for variable selectivity for different metal ions. The results of K_d values presented in Table 2 indicate that Amberlite IRC 718 shows a selective sorption of Pb, Ni and Fe. Selectivity of this resin for these metal ions was also confirmed by the thermodynamic studies performed by the author on this resin [25]. The reason for selective sorption and desorption of certain metal ions can be attributed to the larger difference in K_d values. Thus, based on the selectivities of these metal ions on K_d value determination, separation of lead from a few binary mixtures were studied. Further these results were applied to the actual lead alloys.

Table 1
Ion exchange capacities and distribution coefficient values on Amberlite IRC 718 for Cu, Pb and alkaline earth

Metal ion	Salt used	Metal concentration (M)	Ion exchange capacity (meq g^{-1})
Mg^{2+}	$Mg(NO_3)_2$	0.1	2.13
Ca^{2+}	$Ca(NO_3)_2$	0.1	2.3
Fe^{3+}	$Fe(NO_3)_3$	0.1	2.55
Ni^{2+}	$Ni(SO_4)_2$	0.1	1.73
Pb^{2+}	$Pb(NO_3)_2$	0.1	3.96

Table 2
Distribution coefficient of different metals at different pH at equilibrium contact time 1 h

Metal ion	Input (mg)	D-value obtained at						DMW
		pH 1	pH 2	pH 3	pH 4	pH 5	pH 6	
Pb	11.11	48.0	144.0	180.0	201.0	212.5	212.0	214.02
Mg	9.24	0.0	8.0	19.0	10.0	95.8	100.5	124.29
Ni	10.86	4.0	11.2	14.2	25.8	175.8	250.6	275.5
Fe	22.04	0.0	7.0	18.0	24.0	80.5	110.5	122.23
Ca	12.8	0.0	18.0	27.0	32.0	35.0	34.2	35.5

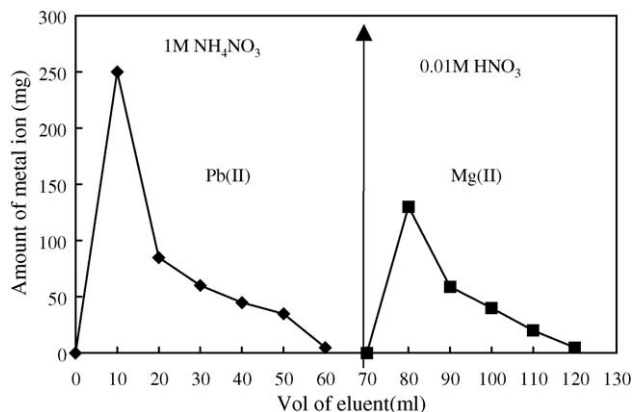


Fig. 1. Separation of Pb(II)-Mg(II).

3.1. Column separation

A glass column (length 43 cm and ID 0.39 cm²) was packed with a known amount of resin (2 g) of 100-mesh size in the sodium form up to 10 cm. After washing the resin with distilled water the sample solutions containing the metal ions desired to be separated were passed through the column at a flow rate of 1–2 ml min⁻¹.

The quantitative separation of Mg²⁺-Pb²⁺, Pb²⁺-Fe³⁺, Pb²⁺-Ni²⁺, Pb²⁺-Ca²⁺ ions has been achieved. The effluent fractions were collected in 10 ml portion and analysed for the metal ions in it using atomic absorption spectrophotometer. Results of the possible separation achieved on the column from synthetic solutions are given in Table 3. The order and the eluents are presented in Figs. 1–4. Separations of these metals from some alloys and synthetic mixtures (containing metals corresponding to the composition of some lead alloys) are given in Tables 4 and 5. The order of metal ions eluted and the time of elution are shown in Figs. 1–4.

Table 3
Separation of some synthetic standards

S. no.	Metal ion	Amount loaded (ppm)	Amount recovered	Eluent	Recovery (%)
1	Mg ²⁺	250	248	45 ml 0.01 M HNO ₃	99.2
2	Pb ²⁺	550	540	50 ml 1 M NH ₄ NO ₃	98.18
3	Fe ³⁺	500	495	60 ml 0.05 M HClO ₄	99.0
4	Ni ²⁺	300	299	50 ml 0.5 M HCl + 0.5 M NH ₄ NO ₃	99.66
5	Ca ²⁺	500	490	50 ml 0.01 M HNO ₃	98.0

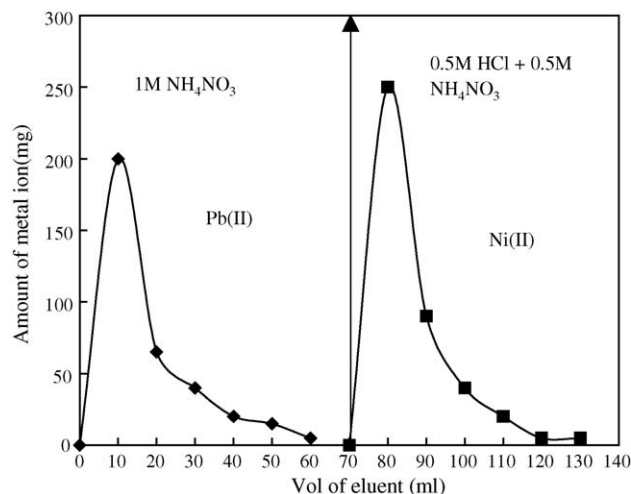


Fig. 2. Separation of Pb(II)-Ni(II).

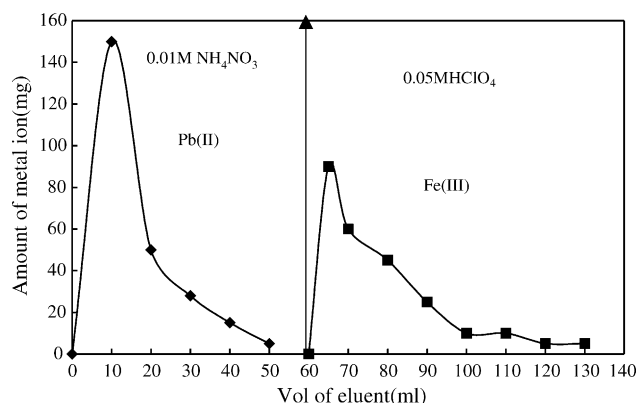


Fig. 3. Separation of Pb(II)-Fe(III).

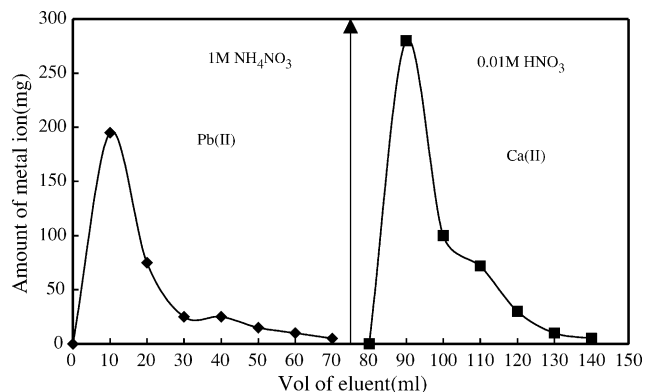


Fig. 4. Separation of Pb(II)-Ca(II).

Table 4
Separation of Pb and Mg from synthetic mixtures

Sample no.	Separation achieved	Amount of metal eluted in ppm	% Recovery
1	Pb ²⁺ 500 ppm	480 ppm Pb ²⁺ with 60 ml 1 M NH ₄ NO ₃	96
	Mg ²⁺ 250 ppm	254 ppm Mg ²⁺ with 50 ml 0.01 M HNO ₃	101.6
2	Pb ²⁺ 250 ppm	248 ppm Mg ²⁺ with 50 ml 0.01 M NH ₄ NO ₃	99.2
	Fe ³⁺ 300 ppm	250 ppm Fe ³⁺ with 75 ml 0.05 M HClO ₄	83.3
3	Pb ²⁺ 350 ppm	345 ppm Pb ²⁺ with 60 ml of 1 M NH ₄ NO ₃	98.57
	Ni ²⁺ 400 ppm	410 ppm Ni ²⁺ with 60 ml 50 ml 0.5 M HCl + 0.5 M NH ₄ NO ₃	102.5
4	Pb ²⁺ 350 ppm	348 ppm Pb ²⁺ with 70 ml of 1 M NH ₄ NO ₃	99.42
	Ca ²⁺ 500 ppm	497 ppm Ca ²⁺ 60 ml 0.01 M HNO ₃	99.4

All the values are the average of three repetitions.

Table 5
Quantitative separation of lead from synthetic lead alloys

Sample no.	Synthetic alloy and its composition ($\mu\text{g ml}^{-1}$)	Amount found in eluent	Error (%)
1	Solder (Pb 500, Sn 500)	495.5	-0.9
2	Solder (Pb 430, Sn 570)	431.6	+0.37
3	Wood's metal (Pb 250, Sn 125, Bi 500, Cd 125)	249.5	-0.2
4	Rose's metal (Pb 280, Sn 220, Bi 500)	281.3	+0.26

3.2. Separations of lead from some synthetic lead alloys

Earlier studies on the kinetic and thermodynamic of the exchange of these metal ions on this resin clearly indicated the possibility of separation of lead from different metal ions in a mixture [23,24]. Variation of different parameters like pH, temperature showed a marked effect on the exchange of different metal ions on this resin. Thus, a few individual cations were attempted for their exchange and elution on Amberlite IRC 718. A glass column (length 43 cm and ID 0.39 cm²) was packed with 2 g of 100 mesh size resin in the sodium form, occupying 10 cm of the column space. The resin was pre-conditioned with the appropriate solution for 2 h. The metal ion solution after pH adjustment was passed through this column at the flow rate of 1–2 ml min⁻¹. The effluent fraction were collected in 10 ml portions and analysed for the concentration of the metal ion by AAS. Results are presented in Table 1 along with the various eluents used. Based on these results, separation of lead ions from several binary synthetic mixtures containing Pb²⁺, Mg²⁺, Ni²⁺, Fe³⁺ and Ca²⁺ was done on Amberlite IRC 718. The column with a known amount of resin was pre-conditioned and the aliquot of the binary synthetic mixture of an appropriately buffered metal ion solution was introduced from the top of the resin bed. The eluent was collected in 5 ml fraction at a flow rate of 0.5 ml min⁻¹ and was analysed for the metal ion eluted. Results shown in Table 2 indicate that lead ion can be separated selectively by this resin.

Thus, on the basis of these results of the lead separation from a few binary mixtures, the separation of lead from some lead alloys was attempted. Since actual samples of these alloys were not available, a few synthetic mixtures were prepared by mixing metal ion solutions in the ratios corresponding to the standard composition of these alloys. Lead was then separated from these

mixtures following the procedure described above and the results are shown in Table 3.

4. Conclusions

Amberlite IRC 718 containing the iminodiacetate group has been used to separate and recover metal ion from their mixtures. Batch and column methods were done for the separation of Pb from the binary mixture with Fe(III), Ca(II) and Ni(II) from synthetic and a few real life samples. The efficiency of this resin in different experimental conditions was established and because of the large difference in the distribution coefficient values separation of the metal ions from their mixture was possible.

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