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SELECTIVE SEPARATION OF INDIUM BY IMINODIACETIC ACID CHELATING RESIN

M. C. B. Fortes[1], A. H. Martins[2*] and J. S. Benedetto[1]

[1]Centro de Desenvolvimento da Tecnologia Nuclear, (CDTN/CNEN),
Fax: +(55) (31) 3499-3399, Rua Prof. Mario Werneck, s/n, C. P. 941,
CEP 30.161-970, Cidade Universitaria, Pampulha,
Belo Horizonte - MG, Brazil
[2]Universidade Federal de Minas Gerais (UFMG),
Departamento de Engenharia Metalúrgica e de Materiais,
Fax: +(55) (31) 3238-1815, Rua Espirito Santo 35, sl 206AG,
CEP 30.160-030, Centro, Belo Horizonte/MG, Brazil
E-mail: ahmartin@demet.ufmg.br

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Abstract - Indium can be recovered by treating residues, flue dusts, slags, and metallic intermediates in zinc smelting. This paper investigates the adsorption characteristics of indium and iron on an iminodiacetic acid chelating resin, Amberlite®IRC748 (Rohm and Haas Co.-USA). High concentrations of iron are always present in the aqueous feed solution of indium recovery. In addition, the chemical behaviour of iron in adsorptive systems is similar to that of indium. The metal concentrations in the aqueous solution were based on typical indium sulfate leach liquor obtained from zinc hydrometallurgical processing in a Brazilian plant. The ionic adsorption experiments were carried out by the continuous column method. Amberlite®IRC748 resin had a high affinity for indium under acidic conditions. Indium ions adsorbed onto the polymeric resin were eluted with a 0.5mol/dm^3 sulphuric acid solution passed through the resin bed in the column. 99.5% pure indium sulfate aqueous solution was obtained using the iminodiacetic acid chelating resin Amberlite®IRC748.
Keywords: Indium; Purification; Ion exchange.

INTRODUCTION

Trace amounts of indium are found in many minerals, particularly in sulphide ores of zinc and to a lesser extent in association with lead, tin, and iron. Commercial recovery of indium is achieved by treating residues, flue dusts, slags and metallic intermediates in zinc smelting. Indium is a crystalline, silvery white metal and is very soft, ductile, and malleable. It generally increases the strength, corrosion resistance, and hardness of metals. The major uses of indium are in alloys and instruments, mainly in the electronics industry (Habashi, 1997). Several methods can be used to extract indium, depending on the source material and its indium content. In addition to solvent extraction from acidic media with different extractants (Hoffman, 1991; Rodriguez et al., 1995; Barakat, 1998; Alguacil, 1999; Fortes et al., 2003), several authors have reported the use of chelating resins for selective adsorption of indium (Sekine et al., 1985; Maeda and Egawa, 1991; Trochimczuk et al., 1994; Yuchi et al., 1997).

In the present study, the adsorption characteristics of indium and iron on the iminodiacetic acid chelating resin Amberlite®IRC748 (Rohm and Haas) were investigated. The metal concentrations in the aqueous solution were based on typical indium sulphate leach liquor obtained from zinc hydrometallurgical processing in a Brazilian plant and treated with solvent extraction for removal of impurities. Due to the high iron concentration in this

*To whom correspondence should be addressed solution, this paper emphasises the selective adsorption of indium.

EXPERIMENTAL PROCEDURE

The iminodiacetic acid chelating resin Amberlite®IRC748 was purchased from Rohm and Haas. Before use the resin was washed with distilled water for 24h, dried on a stove at 35±5°C, and pre-equilibrated with sulphuric acid solution (H_2SO_4 0.5M).

Stock solutions of In and Fe^{2+} were prepared by dissolving indium oxide (99.5% In_2O_3, Merck, Germany) and iron II sulfate (21-23% Fe II, Hoechst, Brazil), respectively, in sulphuric acid solution (95-98% H_2SO_4, Quimex, Brazil). The indium concentration in the feed solution was 2.0g/L and the iron concentration was 6.5, 5.0, and 1.5g/L at a pH of 1.5. The choice of these concentrations was based on typical indium sulphate leach liquor obtained from zinc hydrometallurgical processing in a Brazilian plant and treated by solvent extraction for removal of impurities (Benedetto, 1999). Analytical grade reagents and distilled water were used throughout the experiments.

The conditioned resin, equivalent to 10g dry weight, was placed in a glass column (10mm inner diameter, 400mm length) and had a bed volume of 27mL. Metal ion solution was passed through the resin bed at different flow rates (1.0, 2.0, 5.0, and 10.0mL/min). The metal content in the effluent was determined by atomic absorption spectrophotometry (932 AA Computerized Model, GBC Scientific Equipment, Australia). The metal adsorbed by the resin was eluted with sulphuric acid solution.

The exchange capacity (amount of ions adsorbed on mmol/g of the resin) as well as distribution and separation ratio was calculated from the analytical data. The distribution ratio was calculated from the mmol of metal adsorbed per gram of the resin divided by mmol of metal per millilitre of solution. The separation factor was calculated from the distribution ratio of each metal.

RESULTS AND DISCUSSION

Figures 1 to 4 show the adsorption curves of indium and iron for different flow rates of feed solution.

It was observed in all results that the iminodiacetic acid chelating resin Amberlite®IRC748, prefered indium ions. This occurred independent of the flow rate of feed solution. In Figures 1 to 4, the indium adsorption reached the maximum, expressed by a ratio of metal concentration in the effluent ($C_{effluent}$) to that in the feed solution ($C_{feed\ solution}$) of zero. First, the iron was adsorbed and then it was substituted by indium ions until equilibrium was reached. After equilibrium was reached, iron was no longer adsorbed. As can be seen, the iron $C_{effluent}/C_{feed\ solution}$ was 1.

It also can be observed in Figures 1 to 4 that initial iron adsorption decreased with the increase in flow rate of feed solution up to 5.0mL/min for a higher flow rate of 10.0mL/min, the initial adsorption of iron became larger. Therefore, to achieve a selective adsorption of indium, the appropriate flow rate of feed solution was less than or equal to 5.0mL/min.

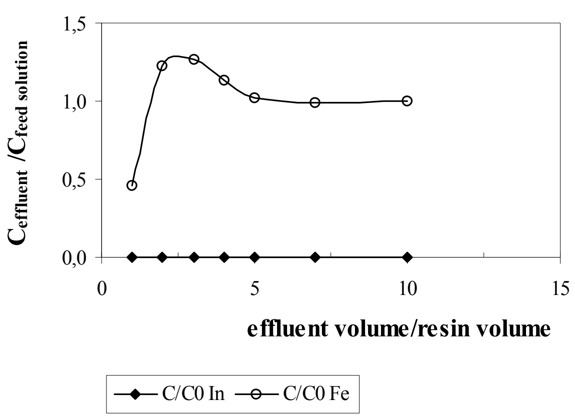

Figure 1: Loading curves of indium and iron in 10.0g of Amberlite®IRC748 resin. Solution with 2.0g/L In^{+3} and 5.0g/L Fe^{+2} , pH 1.5, and a flow rate of 1.0mL/min

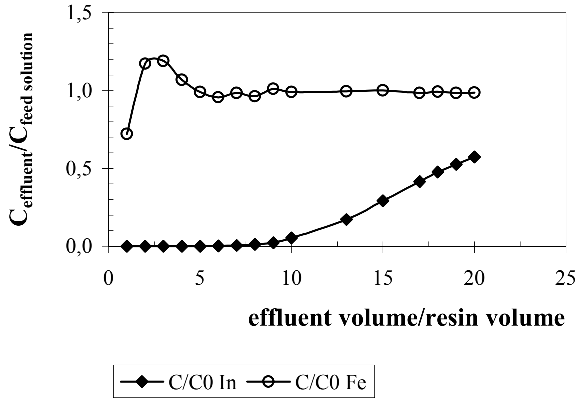

Figure 2: Loading curves of indium and iron in 10.0g of Amberlite®IRC748 resin. Solution with 2.0g/L In^{+3} and 5.0g/L Fe^{+2} , pH 1.5, and a flow rate of 2.0mL/min

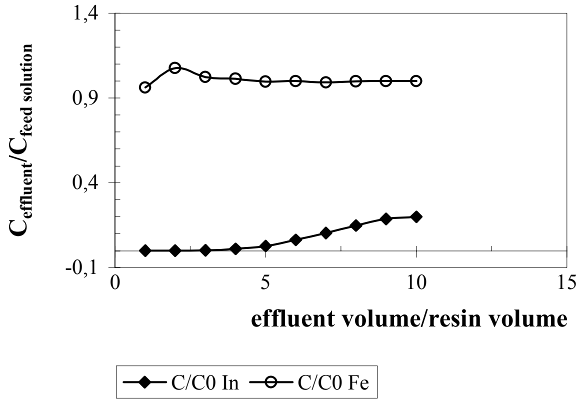

Figure 3: Loading curves of indium and iron in 10.0g of Amberlite®IRC748 resin. Solution with 2.0g/L In^{+3} and 5.0g/L Fe^{+2} , pH 1.5, and a flow rate of 5.0mL/min

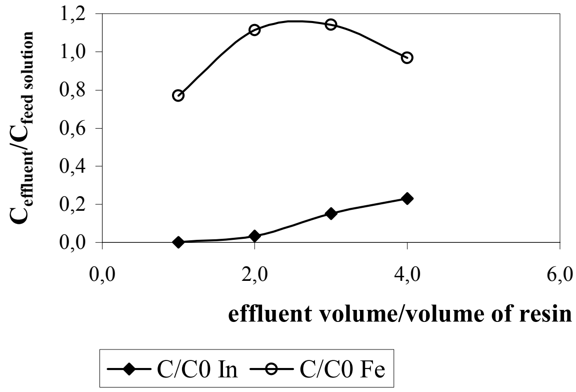

Figure 4: Loading curves of indium and iron in 10.0g of Amberlite®IRC748 resin. Solution with 2.0g/L In^{+3} and 5.0g/L Fe^{+2} , pH 1.5, and a flow rate of 10.0mL/min

The lower the flow rate of feed solution the greater was the adsorption of indium. This fact suggests that the longer the contact time, the higher the uptake of indium, as observed at the breakthrough point.

The results described above suggest that to achieve a high percentage of indium adsorption, besides using 5.0mL/min as a flow rate of feed solution, the experiment can be interrupted after obtaining the equivalent of four bed volumes, which represents the ratio between effluent volume and resin volume.

In Table 1 the results of the mass balance of indium and iron ions, the percentage of each metal that was adsorbed by the resin in relation to the total feed solution and the separation ratio are given. The experiments were done with 3.25, 2.5, and 0.75 Fe/In ratios. The ratio 2.5 Fe/In represents the intermediate concentration of solvent extraction in the indium concentration process developed by the Centre for the Development of Nuclear Technology–CDTN/CNEN, Brazil. At a ratio of 3.25 Fe/In, the study gives a 50% level of reliability process. Finally, 0.75 Fe/In is the ratio of indium to iron found at the outlet in the solvent extraction process developed (Benedetto, 1999).

Table 1: Mass balance of indium and iron ions for loading column experiments

Ratio Fe/In	Metal	Feed Solution (mmoles)	Quantity Adsorbed (%)	Exchange Capacity (mmol adsorbed/ g of resin)	Distribution (%)		Separation Factor
					Feed	Resin	
3.25	In	1.936	99.9	0.193	12.9	90.7	65.5
	Fe	13.061	1.5	0.020	87.1	9.3	0.02
2.5	In	1.846	99.7	0.184	16.8	95.1	95.8
	Fe	9.115	1.0	0.010	83.2	4.9	0,01
0.75	In	1.872	99.9	0.187	38.9	96.3	2.47
	Fe	2.939	2.5	0.007	61.1	3.7	0.03

In Table 1 it can be observed that indium adsorption was almost complete (above 99.7%), while iron adsorption was negligible. Observing the separation factor, it is clear that the iminodiacetic acid chelating resin Amberlite®IRC748 was selective for indium, even when the iron concentration in the feed solution was high.

The elution of the metal ions adsorbed on iminodiacetic acid chelating resin Amberlite®IRC748 was investigated using different eluant flow rates (5.0, 10.0, and 15.0mL/min) and concentrations of sulfuric acid solution (0.5 and 0.1M H_2SO_4). Figures 5 to 8 show the experimental results for indium and iron ion elution of the column. The column was filled with 10g of dry conditioned resin and loaded with an average of 1.82mmoles of indium and 0.09mmol of iron.

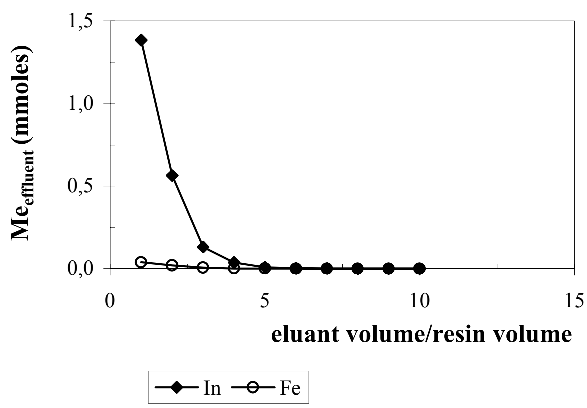

Figure 5: Recovery of indium from Amberlite®IRC748 resin with 0.5M H_2SO_4 solution at a flow rate of 5.0mL/min

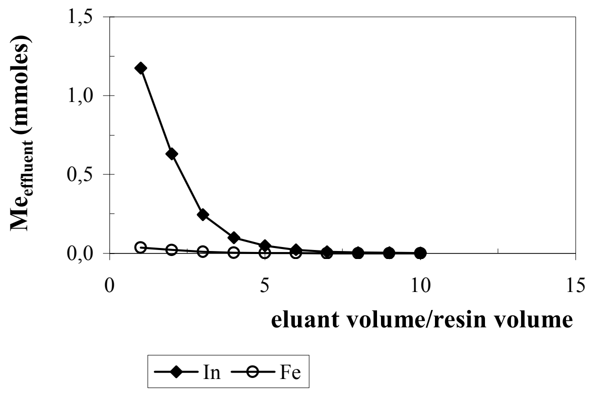

Figure 6: Recovery of indium from Amberlite®IRC748 resin with 0.5M H_2SO_4 solution at a flow rate of 10.0mL/min

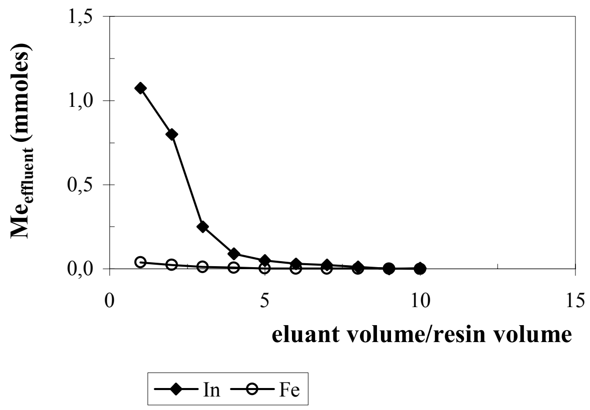

Figure 7: Recovery of indium from Amberlite®IRC748 resin with 0.5M H_2SO_4 solution at a flow rate of 15.0mL/min

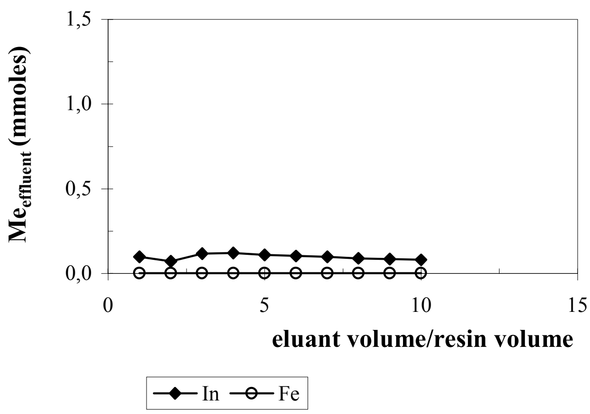

Figure 8: Recovery of indium from Amberlite®IRC748 with 0.1M H_2SO_4 solution at a flow rate of 5.0mL/min

From the experimental results, showing in Figure 5, it can be observed that indium could be effectively eluted by treating with 0.5M H_2SO_4 solution and an eluant flow rate of 5.0mL/min. The first volume of eluant contained the largest amount of indium with almost complete recovery from the resin in five volumes. Increasing the flow rate of the eluant solution to 10.0 and 15.0mL/min, the elution of indium was complete after the equivalent of six 6 and seven bed volumes, respectively, while almost no iron was no eluted.

In Figure 8 it can be observed that using 0.1M H_2SO_4 solution as an eluant, complete elution occurred only after more than ten bed volumes passed through the column. This is not interest of, since the final elution solution would be much diluted.

In order to selectively separate indium from a zinc effluent solution with the iminodiacetic acid chelating resin Amberlite®IRC748 (Rohm and Haas), it is suggested that the loading stage should be carried out with a flow rate of 5.0mL/min and by stopping the feed after four bed volumes. The adsorbed ions could then be eluted with 0.5M H_2SO_4 and an eluant solution flow rate of 5.0mL/min. With these experimental results, using a feed solution of 0.75 Fe/In, the final solution had 2.0g/L of indium with 99.5% purity.

CONCLUSIONS

The remarkable difference between the behaviour of indium and that of iron adsorption and elution under the experimental conditions indicates that the selective separation of these ions with the iminodiacetic acid chelating resin Amberlite®IRC748 is feasible.

It was possible to obtain a final solution with 99.5% pure indium by loading the column with the iminodiacetic acid chelating resin Amberlite®IRC748, using a 5.0mL/min flow rate and stopping the feed solution after four bed volumes, followed by elution of the resin bed with 0.5M H_2SO_4 solution at a 5.0mL/min flow rate.

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