

Purification of lithium ions from lithium hydroxide solution by ion exchange method using cationic resin AG 50W-X8 e HPR 1200

João C. Ferreira¹, Daniela da Costa G. Santos², Juliana I. Otomo², Vanderlei S. Bergamaschi¹, José O. V. Bustillos².

¹*jcferrei@ipen.br, Centro de Células à Combustível e Hidrogênio – CECCO – Instituto de Pesquisas Energéticas e Nucleares IPEN. Av. Prof. Lineu Prestes 2242 – Cidade Universitária. São Paulo. SP*

²*Centro de Química e meio Ambiente. CEQMA – Instituto de Pesquisas Energéticas e Nucleares IPEN. Av. Prof. Lineu Prestes 2242 – Cidade Universitária. São Paulo. SP*

1. Introduction

In recent times, lithium compounds with high chemical purity are gaining prominence in the energy matrix due to the applications of Li-ion vehicle batteries in the energy matrix. In this way, lithium has become an important alternative to the use of fossil fuels in order to reduce greenhouse gases caused by gas emissions. In Brazil, the lithium industries only produce lithium compounds (lithium carbonate and lithium hydroxide) of technical grade, that is, with lower purity than those required by battery manufacturers. Given this context, this work aimed to develop a technology for obtaining lithium compounds with a purity level equal to or greater than 99.95% lithium, using the ion exchange process [1]. Lithium spodumene ore extracted directly from rocks has several chemical impurities such as sodium, potassium and calcium; therefore, to be used as a battery grade, this ore needs to go through purification processes. In this work, lithium compounds were dissolved with nitric and hydrochloric acids, using Dowex 50W-X8 [2,3] and Amberlite HPR1200 resins as a cation exchanger based on the distribution coefficients of calcium, potassium and sodium ions to obtain an effective separation of lithium. Columns in series (120 cm long x 10 cm i.d.) were used to obtain between 98.8 and 99.98% purity in lithium hydroxide, these pure solutions being subsequently used in the isotopic separation of lithium-7.

2. Methodology

In this work, 2 L of 100 mg/L Lithium Hydroxide Load solution was used for each experiment, dissolving the lithium hydroxide supplied by Companhia Brasileira de Lítio (CBL) in the mixture solution NH_4OH 0.01M/ NH_4Cl 0.01M, pH = 4.

150 mL of 50W-X8 cationic resins and 150 mL of HPR 1200 cationic resins were added to the second column in the first column. The regeneration of the 50 W-X8 and HPR 1200 resin was carried out using 100 ml of 2M hydrochloric acid solution prepared with 16.70 of HCl and 83.30 ml of ultrapure water and washing the resin with ultrapure water until reaching pH=4. Both columns were conditioned with a mixture of 0.01 M NH_4Cl and 0.01 M NH_4OH .

The loading solution was percolated through the 50W-X8 column and the HPR 1200

column at a flow rate of 3.5 mL min⁻¹, by gravity, with the first 100 mL being discarded. Checking before discarding whether there has been a change in conductivity. During the percolation process, a conductivity meter was used to determine whether ion exchange was, in fact, occurring. Samples of 50 mL were collected for subsequent analysis using the ICP-OES and Flame Photometer

The elution of the resins was carried out in three stages:

1 – Elution was carried out in column 1, containing the 50W–X8 resin first with 300 mL of 2 M hydrochloric acid solution, washed with 600 mL of water and then elution with 250 mL of ammonium hydroxide solution 1 M.

2 – Columns 2 with HPR-1200 resins were eluted with a mixture of 400 mL ammonium acetate and ammonium nitrate, 300 mL of water and 500 mL of 0.5 M hydrochloric acid solution and then with 2 M hydrochloric acid solution .

All columns were washed with water until the pH decreased to pH=2, and were waited to be conditioned for the next experiment.

3. Results and Discussion

Table 1 – Results of experiments using HPR 1200 resin

Identification	pH	V. Sol.	% CaO	% K ₂ O	% NaOH	% LiOH
Cargo Solution	1,1	2000 mL	0,22	33,61	8,55	58,13
Eluted 17	4,88	300 mL	0,006	0,03	0,004	99,96
Eluted 18	0,73	300 mL	0,13	1,07	0,4	98,4
Eluted 20	0,53	300 mL	0,11	1,65	0,51	97,73
Eluted 1 A	3,0	300 mL	-	-		

Table 2 – Table 1 – Results of experiments using 50W-X8 resin

Identification	pH	V. Sol.	% CaO	% K ₂ O	% NaOH	% LiOH
Cargo Solution	1,1	2000 mL	0,22	33,61	8,55	58,13
Eluted 17	4,88	300 mL	0,016	1,24	3,35	95,39
Eluted 18	0,73	300 mL	0,06	2,50	6,4	91,04
Eluted 20	0,53	300 mL	0,15	1,25	2,85	95,75
Eluted 1 A	3,0	300 mL	-	-		

Conclusion

Initially starting from a charge solution with 58.13% purity in lithium hydroxide, a purity level of 99.96% purity was achieved in eluate 17 using the cationic resin HPR – 1200, while the use of resin 50 W X-8 the results in large columns and with a lithium concentration of 100 mg L⁻¹ were not very promising.

References

- [1] SWAIN, B. Recovery and Recycling of Lithium: A Review. *Sep. Purif. Technol.* 2017, 172, 388–403
- [2] BASPINEIRO, C.F.; FRANCO, J.; FLEXER, V. Performance of a Double-Slope Solar Still for the Concentration of Lithium Rich Brines with Concomitant Fresh Water Recovery. *Sci. Total Environ.* 2021, 791, 148192.
- [3] ABE, M.; ICHSAN, E.A.A.; HAYASHI, K. “Ion-exchange separation of lithium from large amounts of sodium, calcium, and other elements by a double column of dowex 50W-X8 and crystalline antimonite(V) acid”. *Anal Chem*, vol. 52, pp. 524-527 (1980).