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Use of the Ion Exchange Technique for Purification of Lithium Carbonate for Nuclear Industry

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1. Introduction

Lithium is an alkali metal, the lightest of existing metals, with a specific mass of 0.534 g cm^{-3}. Its atomic number is 3 and molecular weight is 6.94 g mol^{-1}. It has two stable isotopes, Li^{+6} (Lithium-6) with an atomic weight of 6.015 g mol^{-1} and Li^{+7} (Lithium-7) with an atomic weight of 7.017 g mol^{-1}. Isotopic abundances are 7.52% for Li^{+6} and 92.47% for Li^{+7}.

The obtainment of lithium compounds, especially lithium carbonate, Li_2CO_3, is the result of extraction processes from several minerals and from brines with lithium content. The most abundant minerals in lithium are lepidolite and spodumene, which is the main source of lithium in Brazil, Australia and Canada.

Lithium carbonate, Li_2CO_3, is the most used raw material for the manufacture of Li batteries, which are currently the largest consumers of Li. and for application in the nuclear area >99.9%.

Lithium-7 is a product used in the refrigeration system of the primary circuit of nuclear power reactors (PWR) to maintain a constant pH. To obtain lithium-7 it is necessary that the starting compound have a high degree of purity in order to reach the enrichment of the isotope of interest.

The present work aims to obtain the purification of lithium carbonate from ion exchange processes at a purity >99%.

2. Methodology

For the purification of lithium carbonate, the materials, reagents and equipment are listed below.

2.1 Materials, reagents and equipment

In the experiments, the materials, reagents and equipment used are glass column 50 cm in height and 2 cm in internal diameter; 50 mL centrifuge tubes in polypropylene, with a lid, self-supporting; 50 mL glass beakers; long-stem funnel of approximately 50 mL capacity; filter paper; Li_2CO_3, with a purity degree of 98.5%; resin AG 50W-X8; nitric acid, HNO_3, 65%, PA; methanol, CH_3OH, PA; lithium standard, 1000 μg L^{-1}; sodium standard, 10000 μg L^{-1}; calcium standard, 10000 μg L^{-1}; ultrapure water, Optical Emission Spectrometer with Argon Plasma Source (ICP- OES), Spectro Flame M120, Modula, Spectro Analytical Instruments; analytical balance with four decimal places, Ohaus Pioneer IDT 7; heating plate, Eletrolab; peristaltic dosing pump, Milan Equipamentos Científicos LTDA; pHmeter, Model 420A, Orion Analyzer and magnetic stirrer, Quimis.

2.2 Methodology

For the development of the methodology, tests were carried out with different conditions of resin quantity, lithium concentration, lithium solution volume, proportion of reagents in the elution solution, pH, eluent volume and flow rate (mL min^{-1}). The Table 1 shows the ion exchange process experimental conditions.

Table 1: Ion exchange operations experimental conditions.

Parameters	A	B	C	D	E
Column Height (cm)	20 x 2				
Resin Volume (mL)	30	40	40	40	40
Conditioning Solution (mL)	HNO_3 0.1 mol L^{-1} in 50% MeOH (v/v)				
Lithium Concentration	3.5 mg of Li^+				33 mg of Li^+
Volume Lithium Solution (mL)	2				34
pH	5				
Eluent Volume (mL)	300	600	800	1100	1100
Flow rate (mL min^{-1})	0.3	1.0	1.5	3.0	3.0

For all experiments, the cationic resin AG 50W-X8 was used. Experiments B, C, D and E were carried out with the aid of a peristaltic pump. In the first step, to carry out the ion exchange experiments, the lithium solution was prepared according to the procedure (A, B, C, D or E) and the resin was added to the column. Then it was added the lithium solution and then added to the eluting solution. The aliquots of the process were collected in 50 mL centrifuge tubes, filtrated, evaporated, filtrated again, and bulked up with ultrapure water. The analyzes were performed via ICP-OES for the determination of the lithium, sodium and calcium content in the aliquots obtained in the experiments.

3. Results and Discussion

The results of tests A, B, C, D and E were evaluated by separating the compounds Li, Na and Ca. Presenting 98.4%, 98.6%, 99.8%, 98.4% and 99.9%, respectively.

Since the starting compound (Li_2CO_3) had 98.5% in Li purity. In Procedure E, the lithium peak was identified separately from the other interfering compounds (sodium and calcium) and the recovery of lithium obtained was 99.9%. Figure 5 presents the separation between Li and Ca in procedure E.

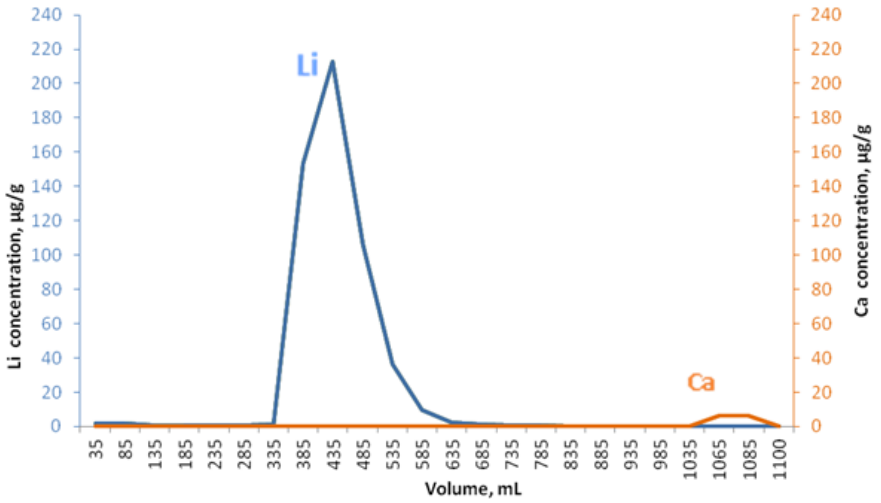

Figure 1: Lithium and calcium separation.

The Figure 2 shows the separation of Li and Na obtained in procedure E.

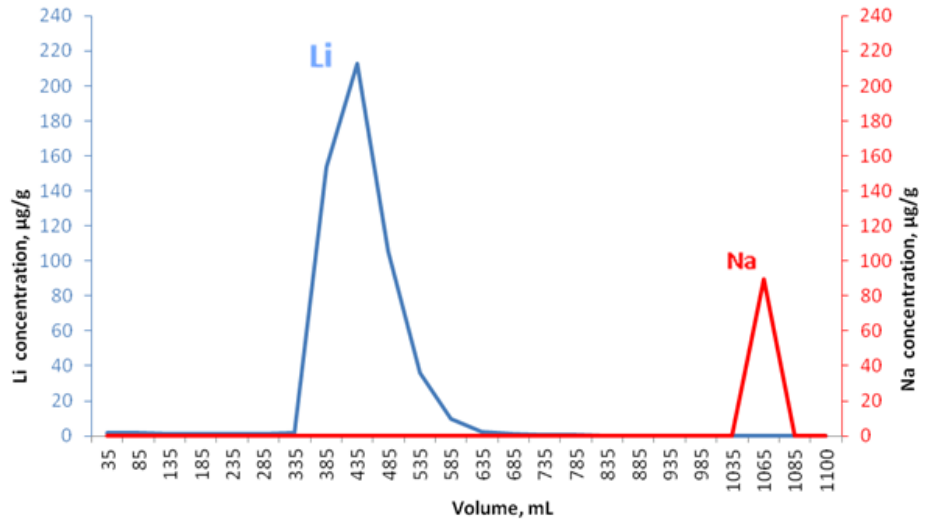

Figure 2: Lithium and sodium separation.

According to the results obtained in the analyses and evaluation of the results and experimental conditions, it is considered that the resolution/separation of the peaks of these cations is positive.

4. Conclusions

Based on the data presented, the purification of lithium with a high degree of purity required for nuclear application was reached at a value of 99.9% using the ion exchange technique.
A great advantage of this process is that it does not require the use of large amounts of water and the possibility of reusing the resin. Even if still on bench scale, the entire cycle of this process can be reproduced for higher scales as a pilot assembly or even on an industrial scale.

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