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Lithium Recovery from Aqueous Solution by Adsorption using MCM-41 Functionalized by TBP

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ABSTRACT

A mesoporous material of type MCM 41 was obtained by the in-situ formation of a hybrid organic-inorganic silicic lattice. The resulting material was characterized by physico-chemical methods: N₂ sorption, XRD and FTIR. In order to improve the extraction capacity of this material with respect to lithium, the material was functionalized with tributyl phosphate (TBP). The structural and textural properties of the material were maintained after functionalization. Lithium extraction tests in the presence or absence of a co-extractor (FeCl₃), by the different solids (MCM-Not Calcined, MCM- Calcined and MCM-CI-TBP) have been carried out by varying the contact time, and the initial concentration. The obtained results are encouraging.

Keywords: Lithium, adsorption, mesoporous silicas.

1. Introduction

Protecting the environment has become a major economic and political issue, and better production and less pollution are the challenges facing manufacturers in all sectors. The lithium mining has major ecological and social consequences in the places where it is mined, particularly in terms of pollution and depletion of water resources. Lithium is rare, precious and dangerous to human health in high concentrations[1]. This element and its compounds are widely used in many fields, particularly in the rapid expansion of rechargeable batteries, which is increasing demand for lithium resources. Indeed, because of its scarcity and in order to conserve resources, it is important to recover lithium from these batteries. With this in mind, we have turned our attention to lithium recovery using mesostructured silica functionalized by TBP.

2. Experimental

2.1. Synthesis & functionalization

Mesoporous silica MCM-41 was prepared by a hydrothermal method using cetyltrimethylammonium bromide (CTAB) as the structure orientation template, and tetraethyl orthosilicate (TEOS) as the silica source. The schematic figure 1 shows the silica formation steps; the protocol adopted in this work is that proposed by Firouzi A. et al [2], [3].

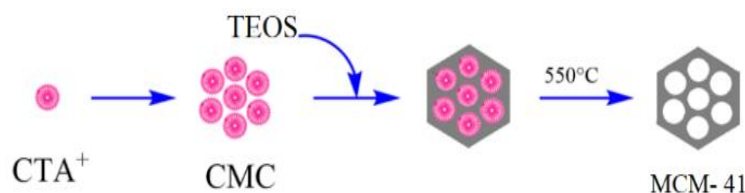


Figure 1: Silica formation steps

After calcination, the silica was impregnated by the dry method. TBP was solubilized in a suitable organic solvent, with stirring until the mixture was homogenized; a second step was to add the silica, and the mixture was then kept under stirring at room temperature, until the solvent had evaporated. It is very important to dry the impregnated silica to evaporate any traces of organic solvent[3].



2.2. Lithium adsorption

In the present work, the lithium solutions were prepared from lithium chloride (LiCl). Uncalcined silica (MCM-NC), calcined unimpregnated silica (MCM-CNI) and calcined silica impregnated with TBP (TBP-MCM41) were used for lithium adsorption tests. Iron trichloride (FeCl_3) was used as a co-extraction agent to improve the adsorption of lithium yield according to the literature [4]. The initial and final concentrations of lithium were determined by analysis on a flame spectrophotometer. The amount of lithium adsorbed on silica $Q_e(\text{mg/g})$ was calculated using the following equation:

$$Q_e = \frac{(C_0 - C_e)V}{m}$$

3. Results and Discussion

The amounts of lithium adsorbed in one hour by the materials MCM-NC, MCM-CNI and MCM-TBP respectively are shown in figure 2 a, the increase of the initial concentration favourably influences the adsorption of lithium, at 500 ppm the MCM-CNI adsorbs 152.5 mg / g, the amount adsorbed by the MCM-TBP increases from 8.57 mg/g to 100.5 mg/g with increasing initial concentration. The study of the influence of the contact time on the adsorption of lithium proves that the adsorption equilibrium can be reached after 10 minutes figure 2 b.

The presence of FeCl_3 at a molar ratio of 1:1 favours the adsorption of lithium, after 60 minutes the MCM-TBP adsorbs 8.57 mg/g without FeCl_3 , 24.3 mg/g in the presence of FeCl_3 1: 1 and 0.9 mg/g in the ratio 1: 4. This proves that the presence of FeCl_3 has increased the adsorption performance, but with the appropriate molar ratio (1:1), the Li- FeCl_3 -TBP interaction is represented by the following reactions[5]:

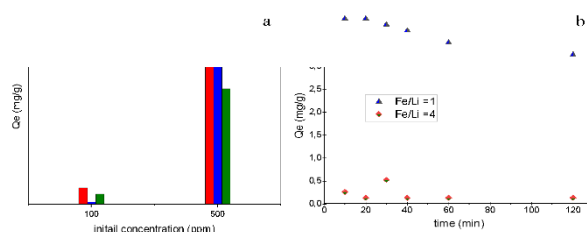
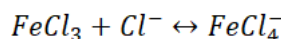
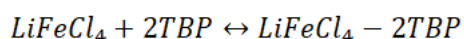
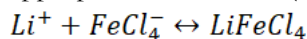


Figure 2: lithium adsorption

4. Conclusions

We have studied lithium extraction from TBP-impregnated calcined, uncalcined and calcined mesostructured silicas. Lithium extraction is positively influenced by increasing the initial lithium concentration. The time required to reach the equilibrium of the extraction is about 10 minutes. The presence of FeCl_3 as a co-extraction agent favours lithium extraction at low molar ratios.

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