



Technical note

Separating lithium and magnesium in brine by aluminum-based materials

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ABSTRACT

Separating lithium and magnesium from high Mg/Li mass ratio brine is an internationally recognized concern. The following study used the reaction between the aluminum-based material with the brine in order to investigate the reaction process of the lithium precipitation as well as the influences on the lithium precipitation rate and Mg/Li mass ratio in precipitation. The results showed that the direct reaction process of the aluminum-based material with the brine helped to eliminate the product coating on the surface of the raw material while enhancing the lithium precipitation rate. Aluminum-based materials exhibit the excellent performance of precipitating lithium and separating lithium and magnesium. The lithium precipitation rate reached 78.3%. The Mg/Li mass ratio in the precipitate was only 0.02, under the optimal conditions. The results were informative for the separation of lithium and magnesium in a salt lake brine.

1. Introduction

The rapid development of new energy industry has increased, the demand for lithium resources that can promote the development and the utilization of lithium resources (Scrosati et al., 2011; Gunther et al., 2017). The salt lakes are rich in lithium resources, and account for over 80% of lithium resources reserves in the world (Kesler et al., 2012; Mauro et al., 2012). Lithium extraction from brines with a lower mass ratio of Mg/Li is the main method used for lithium compounds generation, because of the low production cost (Ramesh et al., 2014; Kenta et al., 2016; Grosjean et al., 2014). Many of the world's salt lake brines have a high Mg/Li mass ratio, with many ratios being 35:1 and reaching up to 1837:1 (Song et al., 2017; Xing et al., 2016; Liu et al., 2015). It is difficult to separate lithium from magnesium, since Li^+ and Mg^{2+} have similar chemical properties. Lithium extraction from a high Mg/Li mass ratio brine remains restricted.

There have many methods used for extracting lithium from salt lake brines, including precipitation (Peiró et al., 2013; An et al., 2012), calcination leaching (Liu et al., 2009; Meshram et al., 2014), solvent extraction (Xiang et al., 2017; Xiang et al., 2016; Ji et al., 2016), and adsorption method (Xiao et al., 2015; Shi et al., 2011; Zandevakili et al., 2014). The precipitation method is a large-scale industrial production method used to extract the lithium from brine. This method is appropriate for the recovery of lithium from a low Mg/Li mass ratio brine (Tran et al., 2013). If there is too much magnesium in the brine, a large amount of NaOH will be used, which is too expensive for industrial production. Calcination leaching uses the insolubility of MgO as a

calcination product. The mixtures that contained $\text{MgCl}_2 \cdot \text{H}_2\text{O}$, LiCl, KCl, and NaCl are obtained by evaporating the brine, followed by roasting at 700 °C (Zhu et al., 2008). During this process, the $\text{MgCl}_2 \cdot \text{H}_2\text{O}$ decomposes into MgO and HCl. The LiCl solution is obtained by leaching the roasting product with the water. This process requires large amounts of energy, and the HCl is produced, which causes corrosion to the equipment and environmental contamination (Feng, 2013). Solvent extraction is an efficient method for extracting lithium from brine, as it has a good selectivity when extracting lithium. The pH of the solution typically requires adjustment via acid to meet the demand of the solvent extraction (Tan and Qin, 2011). The organic solvents have specific solubility in water, which could damage the ecosystem of the salt lake (Shi et al., 2016). The adsorption method is based on the selectivity of the adsorbent to Li^+ . The key to the adsorption process is to acquire an effective adsorbent with good recyclability. The current adsorbent has a small capacity, a low adsorption speed, a large dissolution loss, and is costly (Park et al., 2014; Moazeni et al., 2015), making industrialization difficult.

The current study developed a novel, green method that separates lithium and magnesium from high Mg/Li mass ratio brine. The aluminum-based materials were used to react with the lithium in brine. This method is in accordance with the concept of green extraction, as it does not require organic solvents, acid, or alkali.

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2. Experimental

2.1. Materials preparation

The aluminum-based material was obtained by mixing aluminum powder with NaCl (mass ratio of Al/NaCl is 3:7) and then grinding with a ball crusher for 30 min.

2.2. Experimental procedure

The experiments were carried out in the glass beaker. The solution was added into the beaker (500 mL), agitated with a mixer impeller, and then a thermostatic bath was used to maintain the temperature. The aluminum-based material was added into the solution once the temperature was reached. After the predetermined reaction time, a vacuum filtration was used to separate the solution from the precipitate. The precipitate was washed several times with deionized water. The Li and Mg content in precipitation, was analyzed by atomic absorption spectroscopy. The precipitation rate of the lithium and the magnesium, as well as the mass ratio of Mg/Li in the precipitate were calculated.

2.3. Analysis

During the separation of magnesium and lithium, most of the magnesium remained in filtrate, with a small amount of magnesium in precipitate. The Mg content must be obtained in precipitate in order to obtain an accurate precipitation rate. The precipitate was completely dissolved in a 100 mL hydrochloric acid solution (2 mol/L) under high temperature, and then analyzed. The Li^+ and Mg^{2+} concentration in the dissolved solution were determined by atomic absorption spectroscopy (AAS, Persee of Beijing, China). The precipitation rate of Mg^{2+} and Li^+ were calculated.

The phase structures of the samples were characterized with X-ray diffraction analysis (XRD, Rint-2000, Rigaku) using $\text{Cu-K}\alpha$ radiation. The morphologies of the samples were characterized with a scanning electron microscope (SEM, JSM-5600, JEOL, Tokyo, Japan).

3. Results and discussion

3.1. Reaction process of lithium precipitation

A comparison of the lithium precipitation between the direct reaction process (aluminum-based materials reacted directly with the LiCl solution) and the indirect reaction process (aluminum-based materials reacted directly with the water firstly and then the reaction products reacted with the LiCl solution) was achieved. Fig. 1 shows the diffraction peaks of $\text{LiCl}\cdot 2\text{Al}(\text{OH})_3\cdot x\text{H}_2\text{O}$ in both the direct reaction process and the indirect reaction process. The direct reaction process was more efficient in producing the lithium precipitation. An AAS analysis reveals that the lithium precipitation rate of direct reaction process reached 70.53%. The indirect reaction process was 7.07%.

The morphologies of the products that were obtained during the different processes were compared to find the difference between the two. Fig. 2 shows that, the precipitates obtained by the different reaction processes were flaky. The particle size of the precipitate obtained by the direct reaction process was smaller than the indirect reaction process. This was caused by the precipitate of the indirect reaction becoming deposited on the surface of the particles, resulting in the growth and agglomeration of the particles. The sediment delays the reaction and decreases the lithium precipitation rate. The mechanism of the direct reaction process is depicted as Eq. (1). The hydrogen was produced during the direct reaction process, which could reduce the coating of the sediment on the surface of the aluminum-based materials, while promoting the reaction. The reaction products of the aluminum-based materials with deionized water were added into LiCl

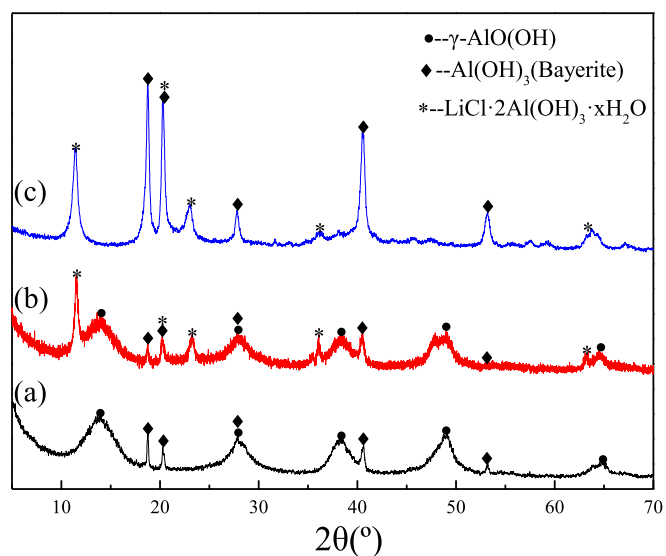


Fig. 1. X-ray diffraction patterns of various reaction precipitations (a): $\text{Al} + \text{H}_2\text{O}$, (b): (a) + LiCl, and (c): $\text{Al} + \text{H}_2\text{O} + \text{LiCl}$.

solution in order to eliminate the negative effects of the sediment film in the lithium precipitation process. The sediment was mechanical milled in a planetary ball mill (QM-3P4, Nanjing University Instrument Plant, China) with a ball to powder weight ratio of 20:1 for the predetermined time. Fig. 3 shows the XRD patterns of the reaction product that were milled for various milling times. The diffraction peaks of the $\text{LiCl}\cdot 2\text{Al}(\text{OH})_3\cdot x\text{H}_2\text{O}$ were observed when the milling time was greater than 210 min. The intensity of the peaks gradually increased as the milling times increased, which indicated that the milling process promoted the lithium precipitation reaction. This result further confirmed that the coating on the surface of the aluminum-based materials was harmful to the lithium precipitation process.



3.2. Separation of lithium to magnesium

The aluminum-based materials exhibited excellent performance when precipitating lithium in the solution. The majority of the salt lake brines in China are characterized with a high Mg/Li mass ratio. They have a small quantity of Li^+ and a quantity of Mg^{2+} coexisting within the salt lake brine. When the Mg^{2+} was precipitated, it was impossible to separate the Mg^{2+} and the Li^+ in solution. The behaviors of the aluminum-based materials in the mixed solution, containing 1 mol/L LiCl and 1 mol/L MgCl_2 were studied. Fig. 4 shows that there were peaks of $\text{LiCl}\cdot 2\text{Al}(\text{OH})_3\cdot x\text{H}_2\text{O}$ and Al, but no substances containing magnesium. The AAS analysis revealed that most of the magnesium remained in the solution. The precipitation rate of the magnesium was 0.8%. The precipitation rate of lithium is 64.8%, which indicated that the aluminum-based material could be used to separate the lithium and the magnesium in a salt brine.

3.2.1. Effect of reaction temperature on separation of lithium and magnesium

The effect of the reaction temperature was investigated between 20 °C to 90 °C. The Li^+ with an initial concentration of 1 g/L and the Mg^{2+} with an initial concentration of 20 g/L, and a reaction time of 3 h. The results are shown in Fig. 5. The reaction temperature had a significant effect on the lithium precipitation process. The Li^+ precipitation rate was 18.14% when reaction temperature was 20 °C. The Li^+ precipitation rate increased with the increase of temperature, where it reached 65.8% at 80 °C. The high temperature was beneficial

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