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Journal of Water Process Engineering

journal homepage: www.elsevier.com/locate/jwpe

Separation of magnesium and lithium from brine using a Desal nanofiltration membrane



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ARTICLE INFO

Article history: Received 31 March 2015 Received in revised form 9 June 2015 Accepted 17 June 2015 Available online 20 July 2015

Keywords: Nanofiltration Brine Mg²⁺/Li⁺ separation

ABSTRACT

Owing to the high ratio of Mg^{2+} to Li^+ in most of the salt lake brines in China, it is difficult to extract lithium. Therefore, the separation efficiency of a nanofiltration membrane was investigated in this study. Operating conditions such as operating pressure, inflow water temperature, pH, and Mg^{2+}/Li^+ ratio were investigated. Relationship between the rejection rates of magnesium and lithium was established. Moreover, the extractions of lithium from salt lake brines were also evaluated. The results indicate that the separation of magnesium and lithium was highly dependent on the Mg^{2+}/Li^+ ratio, operating pressure, and pH. When the Mg^{2+}/Li^+ ratio was <20, the competitive coefficient was sensitive to the Mg^{2+}/Li^+ ratio. The permeate flux of membrane for the East Taijiner brine was higher than that for the West Taijiner brine.

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1. Introduction

Lithium, as the lightest metal in nature, is used in batteries, lubricants, refrigerants, ceramics, medicines, and fusion, which has attracted much attention [1–7]. In recent years, although global economy has suffered, the performance of the lithium industry is still outstanding. The demand for lithium in global market is increasing rapidly with the promotion of new energy, automotive, and electronics industries [8,9]. However, lithium ore resources cannot meet this demand. Therefore, salt lakes and seas with a much larger lithium resource are being considered [10]. Currently, the ion-sieve adsorption method is mainly used to recover lithium from aqueous solutions. However, the Mg²⁺ in brine with a high Mg^{2+}/Li^{+} ratio is easily hydrolyzed, thus increasing the viscosity and decreasing the pH of the brine. These factors lower the brine diffusion rate of the adsorbent and ion-sieve adsorption capacity. Therefore, a method is urgently needed to decrease the Mg²⁺/Li⁺ ratio, separate magnesium and lithium, and increase the lithium concentration in brine.

As a pressure-driven separation process, nanofiltration (NF) technology is based on ultrafiltration and reverse osmosis [11]. This technology has the advantages of a low operating pressure and a high flux and has been widely used in environment and water

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http://dx.doi.org/10.1016/j.jwpe.2015.06.012 2214-7144/© 2015 Elsevier Ltd. All rights reserved.

treatment fields [12]. On account of the special pore-size distribution and preparation technology, NF membranes have a high specific separation performance. These membranes can retain substances with molecular weights between 200 and 1000; moreover, they selectively capture bivalent and multivalent ions. Monovalent ions and small molecules are rejected relatively slowly by these membranes. Because of these characteristics of NF membranes. Somrani et al. [13] studied the separation of lithium from salt lake brines by the NF technology. All the studies were performed using a low flow-rate ratio. This method efficiently separated magnesium and lithium, affording a lithium-rich brine that was converted to lithium carbonate or lithium chloride. Wen et al. [14] analyzed the results of lithium extraction by a DK NF membrane and found that Donnan exclusion, dielectric exclusion, and steric hindrance are the main influencing factors of NF performance. Furthermore, when ionic radius and membrane pore size were close, steric hindrance played a significant role in the separation.

The lithium recovery from brine with a high Mg²⁺/Li⁺ ratio by a Desal (DL) NF membrane have not been studied. In this study, a DL NF membrane was used to study the separation and enrichment of lithium from brine with a high Mg²⁺/Li⁺ ratio systematically. The influencing factors such as the solution concentration, operating pressure, water inlet temperature, and pH of solution were systematically investigated. Moreover, the effects of Na⁺ and Ca²⁺ on the separation of magnesium and lithium were evaluated. Finally, the separation of magnesium and lithium was conducted by using simulate East/West Taijiner brine containing rich lithium resources.



Fig. 1. Schematic diagram of experimental apparatus.

Table 1	
Working parameters of DL nanofiltration.	

Model	Operating pressure (MPa)	Available membrane area (m²)	Available pH range	Water yield (m ³ d ⁻¹)	Average rejection of MgSO ₄ (%)
DL-2540	0.48-2.76	2.51	2-11	3.18	96

2. Experimental

2.1. Apparatus and membrane

The schematic diagram of the experimental apparatus is shown in Fig. 1. The feed was first filtered through the filter to remove the particle impurities. Then, the solutions were pumped to the membrane module. The feed flow rate was controlled by returning a part of the solutions into the feed storage barrels. The flow rates of penetrant and concentrated solution were measured using the rotor flow meter. The penetrant and concentrated solutions were fed into the raw liquid tank to stabilize its feed concentrations. A DL-2540 membrane was used as the NF membrane module, and the working parameters are shown in Table 1. The experimental solutions were prepared from LiCl, MgCl₂, CaCl₂, NaCl (AR), and deionized water (conductivity <0.5 μ s cm⁻¹).

2.2. Experimental procedure

The pristine NF membranes were soaked in deionized water for 24 h at room temperature to ensure that the pores of NF membranes were completely wet and stretched. Then, the membranes were washed with deionized water for 2–3 h under low-flow and low-pressure conditions until the preservatives and other chemicals attached to the surface of the membranes were removed. Finally, the entire cycle was repeated for 3 h to preload the NF membranes effectively and to ensure a stable operation in the subsequent experiments.

The permeate flux of pure water was determined to calculate the permeate coefficient, then the pore sizes of membrane were determined. The operating conditions such as operating pressure, inflow water temperature, pH, and Mg^{2+}/Li^+ ratio were investigated. Relationship between the rejection rates of magnesium and lithium was established. Moreover, the extractions of lithium from salt lake brines were also evaluated

The total influent flow $(C_{\text{Li}}^+ = 0.152 \text{ g L}^{-1}, C_{\text{Mg}}^{2+} = 9.12 \text{ g L}^{-1}$, and $n_{\text{Mg}}^{2+}/n_{\text{Li}}^+ = 60)$ was conducted at a cross-flow rate of 600 L h^{-1} , which was controlled using the bypass and concentrated-solution exit valves, to weaken the concentration polarization phenomenon. The operating pressure ranged from 1.0 to 2.0 MPa with a step length of 0.2 MPa, and the experiments were carried out at room temperature (18–20 °C).

During the process, the operating pressure (1.0-2.4 MPa) and flow velocity were controlled by the rotation rate of high-pressure pump, reflux regulator and into-the-membrane regulator. The pH (3.0–6.0) was adjusted by HCl and NaOH. The temperature (20–35 °C, error: 0.5–1 °C) was controlled by the heat exchanger of the raw liquid tank. The Mg²⁺/Li⁺ ratio (20–90) was adjusted only by the concentration of Mg²⁺.

In the experiments with brines, the flow into the membrane should be under $600 Lh^{-1}$ in order to reduce the concentration polarization phenomenon due to the complicated components and high ion concentrations of brines. The operating pressure should be high (2.0–3.0 MPa), and the temperature was kept at 20–22 °C.

As shown in Table 2, the mass ratio of Mg^{2+}/Li^+ in the prepared East Taijiner brine was 44, and the other ion concentrations were close to those in the real brine, which was quite reliable.

The mass ratio of Mg^{2^+}/Li^+ in the prepared West Taijiner brine was 64, the difference was that the concentration of Na⁺ in the West Taijiner brine was low, and the concentration of Li⁺ was high (Table 3).

The separation efficiency of a NF membrane can be described by the apparent rejection coefficient R_{obs} , separation factor SF, and membrane flux as follows:

$$R_{\rm obs} = \frac{C_{\rm F} - C_{\rm p}}{C_{\rm F}} \times 100\% \tag{1}$$

$$SF = \frac{\left(C_{(Mg^{2+})}/C_{(Li^{+})}\right)_{P}}{\left(C_{(Mg^{2+})}/C_{(Li^{+})}\right)_{F}}$$
(2)

$$J_{\rm v} = \frac{F}{T \cdot S \cdot 3600} \times 10^{-6}$$
(3)

where C_P , C_F , $C_{(Mg}^{2+})$, and $C_{(Li^+)}$ are the concentrations of the penetrant, raw solution, magnesium, and lithium, respectively. *T* is the time consumed by the volume of 1-L penetrant, and S is the effective membrane area. When SF = 1, magnesium and lithium do not separate; when SF < 1, Li⁺ penetrates the membrane preferentially and becomes more concentrated when SF is lower [15].

Moreover, the competition coefficient B is defined as follows:

$$B (Mg^{2+}) = \frac{R_{Mg^{2+}}}{R_{Li^+}}$$
(4)

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