



Development of multi-stage column for lithium recovery from an aqueous solution



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ABSTRACT

Spinel lithium manganese oxides (LMOs) are the most effective lithium adsorbent for the recovery of lithium from an aqueous solution with high selectivity. Developing a systematic method to apply LMOs powder will have a profound impact on the effective and practical process of lithium recovery. We previously fabricated a reservoir system in which the LMO powder was packed in the inside of fabric membrane and then tested by dipping it in an aqueous solution. However, this method exhibited lower adsorption capacity compared with that of LMO powder by stirring in an aqueous solution. Here, we demonstrate a new method to selectively recover lithium from an aqueous solution through an integration of several columns filled with LMO powder. Briefly, our system comprises a series of columns equipped with fabric filters that accumulate LMO powder onto the fabric membrane in which the aqueous solution containing Li^+ ion is introduced into the system by applying pressure. We report that the multi-stage column (MSC) uniformly packed with LMOs exhibited higher adsorption capacity ($30.7 \text{ mg} \cdot \text{g}^{-1}$) compared with that ($12.3 \text{ mg} \cdot \text{g}^{-1}$) of a reservoir system at a lithium concentration of $30 \text{ mg} \cdot \text{L}^{-1}$. To confirm the feasibility of the multi-stage column (MSC), we also tested it to the recovery of lithium from Salt Lake, which was artificially manufactured.

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1. Introduction

Lithium resources are the raw materials for various industry-related products, such as cathode materials for rechargeable batteries, light aircraft alloys, catalyst, and nuclear fusion fuel, which play a significant role in the growth of the economy. Recently, the demand for lithium has been accelerated due to the rapid growth of the worldwide lithium battery market (Wang et al., 2008; Yang et al., 2000; Zhang et al., 2003; Zhao et al., 2004). It is expected that the depletion of lithium resources, which has recently become an issue, will be an obstacle to the development of industrial technologies in the near future.

There are several methods, such as adsorption, solvent extraction, and coprecipitation, to extract lithium from seawater, brine, and geothermal water (Abe and Chitrakar, 1987; Dang and Steinberg, 1978; Miyai et al., 1988; Yanagase et al., 1983). Among these methods, the adsorption method to recover lithium from seawater is the most attractive, considering that seawater is a vast lithium resource containing approximately 2.5×10^{14} kg of lithium compared with other reservoir

sites, although the lithium concentration is low ($0.17 \text{ mg} \cdot \text{dm}^{-3}$) (Chitrakar et al., 2000, 2001; Wang et al., 2009).

A number of researchers have recently reported the fabrication of spinel-type lithium manganese oxides (LMOs), such as LiMn_2O_4 , $\text{Li}_{1.33}\text{Mn}_{1.67}\text{O}_4$, and $\text{Li}_{1.6}\text{Mn}_{1.6}\text{O}_4$, and their application for the recovery of lithium from aqueous solutions (Ammundsen et al., 1995; Feng et al., 1992; Ooi et al., 1990; Sagara et al., 1989; Wang et al., 2006a). LMOs possess noticeable adsorptive properties and especially high selectivity to lithium, including chemical stability with low toxicity, which is suitable for the application of LMOs in an aqueous solution. The feasibility test of LMO to recover lithium from salt lake brine and convert it to the final product (Li_2CO_3) was also reported (Shi et al., 2011). However, the difficulty of recovering the adsorbent powder after the end of operation has limited the practical application of adsorbent powder. To resolve the poor recyclability of powder-type adsorbents, several groups have developed immobilization techniques, such as membranization (Umeno et al., 2002), foaming methods (Han et al., 2012; Ma et al., 2011; Ryu et al., 2013), granulation (Hong et al., 2013), and the use of a reservoir system (Chung et al., 2008), to increase the utilization of the adsorbents used in lithium recovery. The above-mentioned methods (Han et al., 2012; Hong et al., 2013; Ma et al., 2011; Ryu et al., 2013; Umeno et al., 2002) still require improvement for the practical application of such adsorbents because the active

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reaction sites of the adsorbent powder can be blocked by the use of binders, and the fabrication step is more complicated (because at least two-step processes, such as the preparation of adsorbent powder and their formation with the use of binders, are required). Our previous work (Chung et al., 2008) described the use of a reservoir system containing the adsorbent powder inside of the membrane reservoir. One disadvantage of this approach, however, is that the adsorption time to recover lithium from the aqueous solution with low lithium concentration is relatively long although adsorbent powder can be applied without the formation using binders.

In this study, we present a new process using a multi-stage column (MSC) for the highly efficient recovery of lithium from an aqueous solution. Our approach was based on the integration of multiple columns that contain LMO powder onto the filter membrane and manipulation of the flow of aqueous solution as a function of the applied pressure. In addition, this paper describes details of the column design and the effect of controllable pressure for the supplied aqueous solution into the system, as well as the application of LMO powder using the MSC to the recovery of lithium from artificially manufactured Salt Lake. The application of LMO powder using the MSC resulted in dramatic enhancement of the adsorption capacity of lithium from the aqueous solution without recovery difficulty of LMO powder after the end of process.

2. Experimental section

2.1. Preparation of spinel type lithium manganese oxide (LMO)

Spinel type lithium manganese oxide was synthesized via a solid state reaction using Li_2CO_3 (99%, Aldrich, USA) and MnCO_3 (99.9%, Aldrich, USA) as the starting materials (Ryu et al., 2013). The Li/Mn molar ratio was 1.33/1.67. Prior to the heat treatment, the weighed reactants were well mixed using a ball mill. The mixture was placed in a ceramic boat and then heated in a box furnace at 500 °C for 4 h under air atmosphere. The crystalline forms of the obtained products were determined using an X-ray diffractometer (XRD, D/MAX 2200, Rigaku), the morphology was examined using analytical scanning electron microscopy (SEM, S-4700, Hitachi), and the particle size of the prepared LMO powder was investigated by the use of a particle size analyzer (Mastersizer 2000, Malvern).

2.2. Fabrication of the multi-stage column system

Fig. 1 shows a schematic diagram of the multi-stage column (MSC) and the experimental device. The experimental device consisted of a diaphragm pump, a multi-stage column, a reservoir, a pressure gauge, a pressure control valve, switch valves, and a flow meter. The column was fabricated using a fabric filter attached between polyethylene (PE) plates. The size of the PE plates was 85 mm (L) × 85 mm (W) × 10 mm (T). The column was assembled in the order of PE

plate/fabric filter/spacing PE plate/fabric filter/PE plate and was subsequently pressed to prevent the loss of LMO powder during the experiment. The distance between the two PE plates was fixed at 10 mm using a spacing PE plate in a manner that the LMO powder was packed inside the columns. The diaphragm pump (All-FLO PUMP Co., USA) was operated by flowing compressed air into the pump. The pressure of the supplied solution containing lithium ion was adjusted by controlling the pressure control valve connected to the end of the column. The porosity and average pore diameter of used fabric filter in the column was investigated using a mercury porosimeter (Micrometrics autopore IV 9500).

2.3. Li^+ adsorption and desorption experiments

The experiment was conducted using the following four steps. In the first step, LMO powder was packed inside the column by circulating the solution of LMO powder and distilled water using the diaphragm pump. In the second step, $0.5 \text{ mol} \cdot \text{L}^{-1}$ of HCl solution was circulated to extract lithium from the LMO structure, which resulted in the formation of the lithium adsorbent. In the third step, a solution containing lithium ions was circulated into the system until the adsorption reaction reached an equilibrium state. In the final step, $0.3 \text{ mol} \cdot \text{L}^{-1}$ of HCl solution was circulated into the system to recover lithium ions from the lithium adsorbent. After each step, distilled water was supplied and/or air was flushed into the system to remove the remaining solution.

The influent solution flowed into the channel of each spacing PE plate to uniformly pack LMO powder onto the fabric filter of the multi-stage column (MSC) and then was discharged through the multiple channels of the MSC so that the pressure that could be caused from the fabric filters of the column was minimized. The adsorption reaction of lithium adsorbent was investigated using 30 L of LiOH (Li^+ concentration of $30 \text{ mg} \cdot \text{L}^{-1}$) at 25 °C. The amount of LMO powder packed in the column was 5 g. The pressure of a compressed solution in the system was controlled from 3 to $6 \text{ kg} \cdot \text{f} \cdot \text{cm}^{-2}$ by adjusting the pressure control valve connected to the end of column while the 30 L of the solution was circulated into the system using the diaphragm pump (maximum operating pressure: $6 \text{ kg} \cdot \text{f} \cdot \text{cm}^{-2}$). For the comparison test, the reservoir system was prepared using the same fabric filter as that of the MSC. 5 g of delithiated LMO powder was packed inside of the fabric filter, which was subsequently was placed in the rectangular shaped column of 5 cm W × 10 cm L × 10 cm H. 30 L of the solution was circulated at a flow rate of $10 \text{ L} \cdot \text{min}^{-1}$ through the reservoir system. The concentration of ion of the collected samples was analyzed by the use of an inductively coupled plasma atomic emission spectrophotometer (ICP-AES, Optima 7300D, Perkin Elmer).

The lithium adsorption capacity (q_e) was examined by:

$$q_e (\text{mg}_{\text{Li}}/\text{g}_{\text{adsorbent}}) = \frac{(C_o - C_e)V}{m} \quad (1)$$

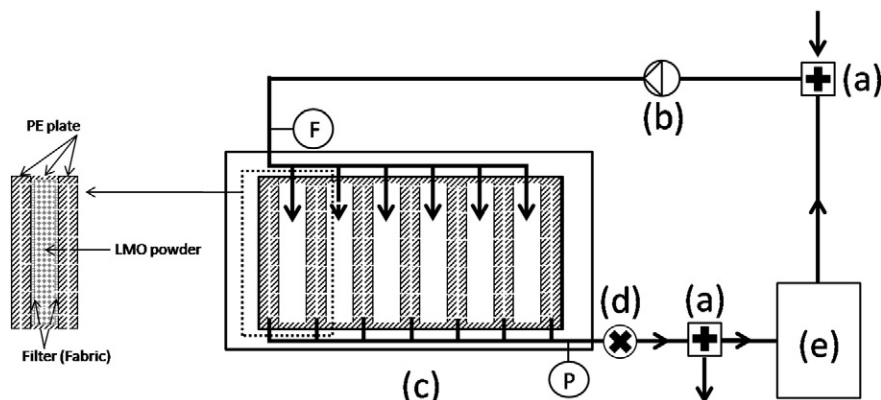


Fig. 1. Schematic diagram of the MSC and the experimental device: (a) switch valve, (b) diaphragm pump, (c) column, (d) pressure control valve, and (e) reservoir.

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