



Effect of chemical treatments on lithium recovery process of activated carbons



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ABSTRACT

In this work, surface characteristics of chemically treated activated carbons (ACs) were studied for lithium ion in aqueous solution. From the results, it was found that the chemical treatments introduced functional groups onto the ACs surfaces. The amount of lithium ion recovery was enhanced by basic treatments. Also, we characterized lithium ion adsorption-desorption efficiency. The lithium adsorption-desorption efficiency exhibited good stability after five adsorption-desorption cycles. Consequently, it could be concluded that lithium ion recovery behaviors are greatly influenced by the basic characteristics of AC surfaces, resulting in enhanced electron acceptor-donor interaction at interfaces.

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Introduction

Lithium and its compounds are widely used in many fields, especially in rapid expansion of rechargeable batteries, wireless communications technologies, and hybrid vehicles, which increase the demand of lithium resources [1]. Because of lithium's attractive properties, no other element can be substituted for it, and its consumption increases annually [2]. Due to their high energy density, high cell voltage, long storage life, low self-discharge rate and large temperature range, lithium batteries are often preferred to conventional systems with aqueous electrolytes [3]. Lithium is harvested from salt lakes, which include about 33 million tons of lithium [4]. Geopolitically, there is an inherent risk regarding the accessibility of lithium since this metal is available mainly in specific geographic regions (South America, such as Argentina, Bolivia and Chile) and an instability in these regions can greatly affect the lithium supply [5]. In order to meet the measures of the demand of lithium, the recycling of lithium secondary battery wastes and the extraction of lithium from seawater have promoted, but now it is difficult to try recycling waste that it is due to lack of high purification technology [6]. Therefore, many researchers have interested in fields of the recycling useful metals

from seawater. Although the concentration of lithium is very low about 17 ppm, but seawater contain enormous quantity of the element lithium about 260 billion tons [7,8]. From now, many researchers have studied various techniques for lithium recovery from seawater and the salt lakes, such as solvent extraction [9], membrane processes, ion exchange resins [10], co-precipitation [11], and adsorption [12,13]. Specially, the study of adsorbent has been performed with various materials, such as LiMn_2O_4 (Lithium Manganese Oxide), which have attracted attention in lithium adsorbents from seawater because of their high selectivity [14].

Activated carbons as an efficient adsorbents are widely used to recover Ni(II) from wastewater due to its high surface area, excellent adsorption capacity and various surface with functional groups. However, its practical use is extremely restricted by its expensive production costs, hence the production of activated carbon from lower cost precursor has important values [15]. In recent years, many researchers have studied activated carbons from many cheaper and renewable precursors, such as several agricultural by-products including rice husk, grapeseed, chestnut shell, and peanut hull. Also, modified activated carbons have been suggested as an useful adsorbents improving the adsorption capacity for metal [16,17].

The chemical modification of carbon materials is a frequently used method in the preparation of carbon-based adsorbents for heavy metal recovery [18]. Various reagents have been used as oxidizers: concentrated nitric or phosphoric acid, sodium hypochlorite, permanganate, potassium hydroxide, hydrogen peroxide, and ozone-based gas mixtures [19]. Among surface modification methods, acid treatment can be used to increase the acid value in a

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Table 1
Distributions of activated carbons prepared by chemical treatments.

Specimens	Chemical treatments	Concentration (mol/L)
NAC	H ₂ O	–
AAC-0.01	H ₃ PO ₄	0.01
AAC-0.1		0.1
AAC-1		1.0
BAC-0.01	KOH	0.01
BAC-0.1		0.1
BAC-1		1

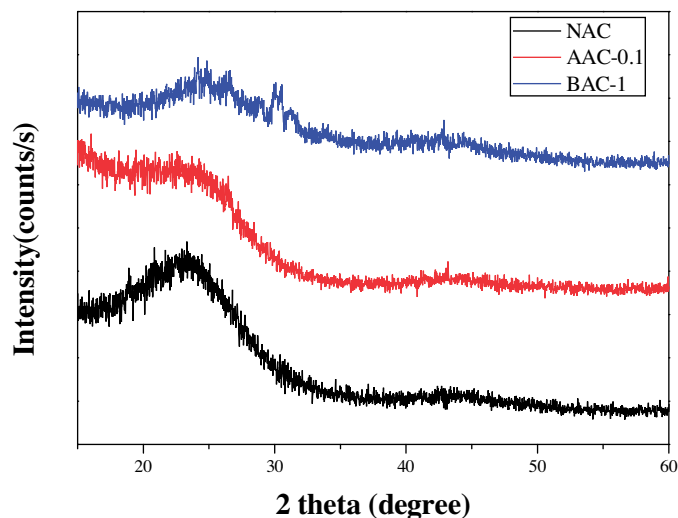


Fig. 1. XRD patterns of ACs prepared by chemical treatments.

wet oxidation treatment. Especially, the oxidation treatment of carbon-based materials in a wet phosphoric acid results in a significant increase in the acidic surface groups such as carboxyl groups [20]. In the case of ACs modified with basic reagents, the acid value is almost the same in as untreated ACs caused by an increase of lactone groups as well as carboxyl groups. However, most chemical treatments of carbon materials have been carried out to increase the adhesion interfacial force in polymeric matrix composite systems [21]. There are few reports on the modification of activated carbons to enhance the loading capacity.

Finally, we studied that the effect of chemical treatments in the structural and textural properties for lithium recovery process in acid-base treated ACs from aqueous solution.

Experimental

Materials and sample preparation

Activated carbon (AC) prepared from charcoal by oxidation using molecular oxygen was purchased from Tokyo Chemical

Industry Co. Ltd. (Tokyo, Japan). The specific surface area of the activated carbons, pore volume, and average pore diameter are 773 m²/g, 0.877 cm³/g, and 2.45 nm, respectively. Prior to the acid and base treatments, ACs were washed several times with distilled water and dried in an oven at 80 °C for 24 h. One gram of the ACs was immersed with 100 mL of different concentration phosphoric acid (H₃PO₄, Duksan Co.) and potassium hydroxide (KOH, Duksan Co.) solutions for 2 h at room temperature. The H₃PO₄-, KOH-treated ACs samples were filtered and washed with distilled water and dried in an oven for 24 h at 80 °C. The H₃PO₄-treated ACs had as-received H₃PO₄ concentrations denoted as AAC-0.01, AAC-0.1, and AAC-1. The KOH-treated ACs had as-received KOH concentrations denoted as BAC-0.01, BAC-0.1, and BAC-1. The prepared samples were shown in Table 1.

Characterization

X-ray diffraction (XRD) analysis was carried out to investigate the phase of the samples using the CuK α radiation ($\lambda = 1.543 \text{ \AA}$) at 30 kV and 30 mA (D2 Phaser; Bruker Co.). The morphologies were observed by SEM measurement (S-4300; Hitachi Co.). Textural properties were measured at 77 K using a nitrogen adsorption analyzer (BEL-SORP; BEL Co.). Zeta-potential measurements were performed by putting 70 mg of ion samples, which is dispersed into 70 mL of distilled water by sonication, using a dynamic light scatter analyzer (Nanotracer wave Q, Microtrac Co.). The degrees of acid and base value of the ACs surfaces were determined according to the Boehm titration method. One gram of sample is placed in 100 mL of 0.1 N NaOH for acid value measurement. The vials were sealed and shaken for 24 h. Then, 20 mL of filtrate were pipetted and titrated with 0.1 N HCl. And the base value measurement was performed by same procedure but sequence of putting NaOH and HCl was changed.

Measurement of lithium ion adsorption-desorption

The lithium ion adsorption and desorption behaviors were studied in a batch system. 125 mg of ACs samples were immersed in 250 mL of LiCl (Aldrich Co., Korea) solution at room temperature for 24 h. The initial lithium ion concentration was fixed at about 50 ppm at neutral pH. The mixture was centrifuged, and the lithium ion concentration of the remaining solution was determined using the supernatant. Lithium ion adsorption capacity was calculated based in the change of the initial and final lithium ion concentrations using the following equation:

$$Q = (C_0 - C_\alpha) / w \times V \times 100 \quad (1)$$

where, Q is the adsorption capacity (wt%), C_0 and C_α are the initial and final concentrations (mg/L), respectively. V is the volume of solution (L), and w is the mass of adsorbent used (g). After absorbed lithium, the sample was desorbed in 0.5 M HCl solution at room



Fig. 2. SEM images of (a) NAC (b) AAC-0.1 and (g) BAC-1 prepared by chemical treatments.

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