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Design and development of a functionalized biochar sorbent with 2-(hydroxymethyl)-12-crown-4 ether for selective Li separation

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Key words: lithium, functionalized biochar, separation, crown ether, selective sorption

Abstract

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Sorption is a promising separation technique for lithium recovery from aqueous streams and industrial liquid waste; however, its efficiency is often limited by the presence of competing ions. In this study, a selective sorbent was developed using cherry pit-derived biochar as a low-cost precursor. Biochar produced via slow pyrolysis at 500 °C (BC) was chemically functionalized with 2-(hydroxymethyl)-12-crown-4 ether to enhance Li⁺ affinity, yielding the modified sorbent (F-BC). The physicochemical properties of the materials were characterized by FT-IR spectroscopy to confirm successful surface modification. Batch sorption experiments revealed a ~20-fold increase in lithium sorption capacity of F-BC relative to pristine biochar. Selectivity tests performed in multicomponent systems demonstrated preferential Li⁺ uptake over competing monovalent (Na⁺, K⁺) and divalent (Ca²⁺, Mg²⁺) ions, indicating favorable ion-recognition behavior of the functionalized surface. The enhanced performance is attributed to the specific complexation of Li⁺ with crown ether moieties. The results suggest that F-BC is a viable candidate for selective lithium recovery in complex aqueous matrices, with potential applications in resource recovery and wastewater treatment.

Introduction

The continuous growth of industrial production and technological development generates millions of tons of heavy metals annually. Improper management of industrial waste streams can lead to their release into surface and groundwater systems. At the same time, many of these contaminants represent valuable secondary resources, and their recovery is essential for the development of sustainable and circular production systems [1]. In particular, lithium has gained strategic importance due to its extensive use in energy storage technologies, while its global supply remains geographically uneven and sensitive to geopolitical constraints. Consequently, there is growing interest in recovering lithium from alternative sources, including dilute aqueous systems such as seawater and industrial effluents. However, the low concentration of Li^+ and the high excess of competing ions in such matrices present significant separation challenges. To address these limitations, advanced separation techniques for Li^+ recovery have been extensively investigated, including dialysis, ion exchange, membrane processes, and adsorption. Among these, adsorption-based approaches offer advantages in terms of operational simplicity, lower energy demand, and reduced formation of secondary pollutants compared to conventional methods [2]. The efficiency of adsorption processes is strongly dependent on the physicochemical properties and selectivity of the adsorbent material.

Selective Li^+ adsorption can be enhanced through the incorporation of functional ligands with specific ion-recognition capabilities. Crown ethers (CEs) are macrocyclic compounds composed of 3–20 oxygen atoms linked by alkyl chains, forming a structure with a hydrophobic exterior and a hydrophilic cavity capable of coordinating metal ions via ion–dipole interactions [4,5]. Their cavity size determines cation selectivity; for example, 12-crown-4 exhibits high affinity toward Li^+ , while 18-crown-6 preferentially binds K^+ . Due to these properties, CEs have been widely applied in separation processes, catalysis, and biochemical systems. Previous studies, such as Zheng et al. (2021), demonstrated that CE-functionalized nanofiber membranes can achieve high Li^+ sorption capacities (up to $168 \text{ mg}\cdot\text{g}^{-1}$ at pH 7) with excellent selectivity and regeneration performance [6]. Carbon-based materials are commonly employed as supports for CE immobilization due to their high surface area and chemical stability. In this context, biochar produced via thermochemical conversion of biomass and characterized by a carbon content typically exceeding 60 wt.% represents a low-cost and sustainable alternative to conventional carbon materials [7].

The aim of this study was to evaluate the suitability of biochar derived from food waste (cherry pits) as a support for surface functionalization with crown ether moieties, with the goal of developing a selective and efficient sorbent for Li^+ recovery from aqueous solutions.

Materials and methods

Preparation and 2H12C4 functionalization of biochar

Biochar (BC) was prepared from cherry pits at a temperature of 500°C via a slow pyrolysis process in an N₂ atmosphere (2 L/min) using a PYREKA 2.1 pyrolysis reactor (Pyreg, Dörth, Germany). The prepared pyrolysis product (Fig. 1) was homogenized and sieved.

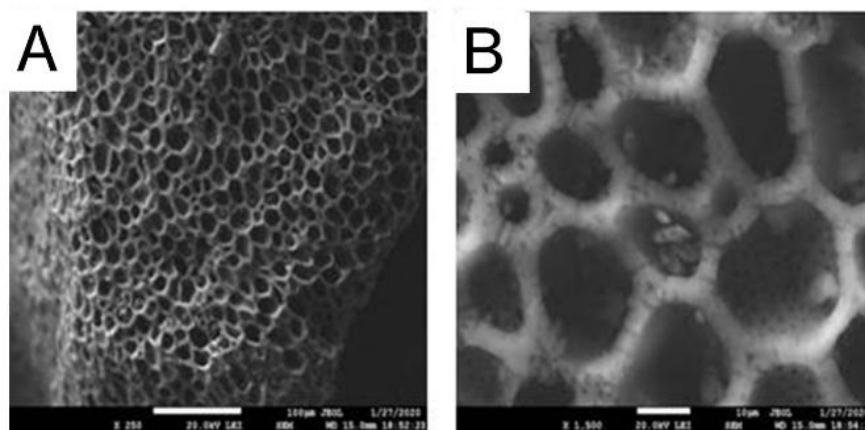


Fig. 1 SEM images of BC at 250× (A) and 1500× (B) magnification.

In the first step of activation, the biochar was oxidized with HNO₃ to introduce -COOH groups onto the sorbent surface. To 10 g of biochar, 300 mL of 20% HNO₃ was added, and the suspension was stirred on a magnetic stirrer at 300 rpm and 90 °C for 4 hours. Subsequently, the mixture was filtered and washed with deionized water until the filtrate reached a pH≥7. After filtration, the biochar was dried at 60 °C to constant weight. In the second step (Fig. 2), we added 20 mL of anhydrous DMF to 1 g of activated biochar and stirred for 10 min. We added 6.1 mg of DMAP to the suspension while stirring continuously. DMAP accelerates the formation of esters with the surface functional groups of the modified biochar while minimizing side reactions. We cooled the mixture to 0–5°C in an ice bath. In a small dry test tube, we dissolved 0.288 g of EDC · HCl and 0.173 g of NHS in 4 mL of DMF at 4°C. We added this activation solution dropwise to the biochar suspension. We stirred the resulting suspension for 1 h at 22°C. Subsequently, we dissolved 0.155 g of 2H12C4 in 3 mL of DMF and added the solution dropwise to the biochar suspension, stirring continuously at 22°C for 18 h. We separated the functionalized sorbent (F-BC) from the reaction solution by vacuum filtration, followed by a series of washes with DMF (to remove byproducts and unreacted reactants), methanol (to remove residual organic matter), and deionized water (to remove salts and water-soluble byproducts). The prepared BC and F-BC sorbents were re-sieved to obtain a fraction < 0.07 mm, which was subsequently used for FT-IR analysis and sorption characterization.

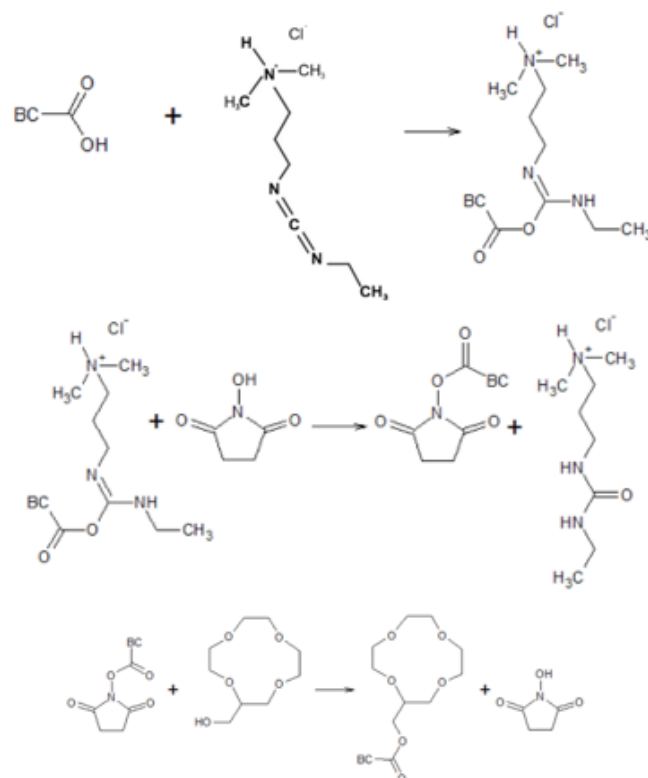


Fig. 2 Modification of BC and preparation of F-BC with 2H12C4; (A) activation of -COOH using EDC, (B) formation of an NHS ester – stabilization of the activated -COOH, (C) reaction of the NHS ester with 2H12C4

FTIR analysis

We performed Fourier-transform infrared (FTIR) spectroscopy using a Cary 630 instrument (Agilent, USA) with the attenuated total reflection (ATR) method. We subjected samples of the feedstock biomass, BC-H₂O, and BC-2H12C4 to this analysis. All analyzed samples were homogenized and, without further treatment, applied to a Ge crystal in sufficient quantity to cover its entire surface. FTIR spectra were recorded in the mid-infrared range of 650–4000 cm⁻¹ with a resolution of 4 cm⁻¹ and 64 scans. Before each sample measurement, a background measurement (of the clean crystal) was performed to eliminate interference. The measurements were conducted in transmission mode.

Sorption experiments

To characterize the sorption properties of BC and F-BC for the separation of Li⁺ from aqueous solutions, the effects of contact time between the sorbent and the sorbate, the initial concentration of the sorbate, and the competition from selected monovalent (Na⁺, K⁺) and divalent (Ca²⁺, Mg²⁺) ions in equimolar concentrations (1:1). To calculate the amount of Li sorbed by BC- and F-BC-based sorbents, we used the following basic equation:

$$Q_{eq} = ((C_0 - C_{eq}) \cdot V) / m$$

where Q_{eq} represents the amount of sorbed Li (mg/g), C_0 represents the initial concentration of Li in the solution (mg/L), C_{eq} represents the equilibrium concentration of Li in the solution after sorption (mg/L), V is the volume of the solution (L), and m is the mass of the sorbent (g)

The concentrations of unsorbed Li in the solution after sorption were determined using F-AAS (Agilent AA240) in both absorption and emission modes. A reference material with c Li = 1000 mg/L (Sigma-Aldrich, Germany) was used for the optimization and calibration of the Li determination.

In the case of equilibrium sorption, the experimental data obtained were evaluated using the Langmuir and the Freundlich adsorption isotherm models [8].

Results and discussion

FTIR Analysis

Based on the obtained IR spectra, we were able to identify various functional groups on the surface of the feedstock (Fig. 3A) cherry pits and on the surface of biochar (BC) as well as 2H12C4-functionalized F-BC (Fig. 3B). In the spectrum of the feedstock, we identified the presence of a broad band in the range of ν 3200–4000 cm^{-1} , which corresponds to O–H vibrations and may indicate surface hydration or the presence of oxygen-containing functional groups. These bands are often broad and intense, reflecting the presence of hydrogen bonds [9, 10].

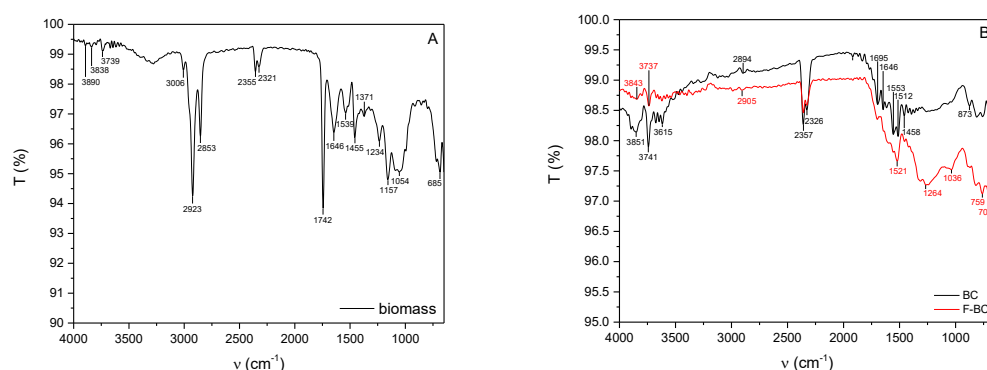


Fig. 3 FTIR spectra of the input biomass (A) and the BC and F-BC sorbent samples (B).

In the range of $\nu \sim 3000\text{--}2800 \text{ cm}^{-1}$, we observe intense absorption bands which, according to the literature, can be attributed to C–H valence vibrations of methyl and methylene groups [11]. In the range of $2325\text{--}2360 \text{ cm}^{-1}$, we identified a band that could correspond to the presence of atmospheric carbon dioxide (CO_2), since all samples are porous in nature [12]. In the spectrum of the input biomass, we identified an absorption band in the range of ν 1750–1700 cm^{-1} , which corresponds to the vibrations of the C=O double bonds of groups, specifically ketones and

carboxylic acids [13]. Bands are found at $\nu \sim 1640\text{--}1530\text{ cm}^{-1}$, the presence of which may correspond to the occurrence and vibration of C=C aromatic bonds present in plant materials [14]. A band located at $\nu \sim 1450\text{ cm}^{-1}$ indicates that O-CH₃ (methoxyl) functional groups contribute to the basic structure of the phenylpropane units of lignin [15]. At $\nu \sim 1370\text{ cm}^{-1}$, there is a clear band corresponding to C-H vibrations originating from cellulose, hemicellulose, and lignin. The band at $\nu 1234\text{ cm}^{-1}$ corresponds to C-C and C-O vibrations originating from lignin, and C-O-C at 1157 cm^{-1} from cellulose and hemicellulose [16]. Infrared vibrations at $\nu \sim 1030\text{ cm}^{-1}$, corresponding to C-O or -OH stretching, indicate the presence of ethers and esters, which act as a linking element between various monomers of lignocellulosic components, as well as components of phenylpropane units linked to lignin. A more intense absorption band at $\nu \sim 685\text{ cm}^{-1}$ indicates the presence of mono-, polycyclic, and substituted aromatic compounds contributing to the basic structure of the lignocellulosic components of biomass [15].

In the biochar samples (Fig. 3B), we characterized several bands. In the BC sample, we observed the same bands as those found in the biomass sample; specifically, at $\nu 3200\text{--}4000\text{ cm}^{-1}$, we observed O-H vibrations [9], at $\nu 2910\text{--}2890\text{ cm}^{-1}$, C-H valence vibrations [11], and at $2325\text{--}2360\text{ cm}^{-1}$, those characteristic of CO₂ [12]. At $\nu 1695\text{--}1640\text{ cm}^{-1}$, we observed a band characteristic of C=C aromatic bond vibrations. For delocalized conjugated C=C bonds in aromatic rings, a vibration band at $\nu 1560\text{--}1510\text{ cm}^{-1}$ is characteristic [14]. At a wavenumber of 1458 cm^{-1} , we observed a band typical of the deformation vibrations of methylene (CH₂) and methyl (CH₃) groups [9]. The band at $\nu 900\text{--}700\text{ cm}^{-1}$ is characteristic of -CH deformation vibrations, which indicate recombination and the formation of an aromatic structure [12].

In the FT-IR spectrum for the F-BC sample, in addition to the bands described for the BC sample, we observed absorption bands at 1264 cm^{-1} and 1036 cm^{-1} . The band at 1264 cm^{-1} corresponds to C-O vibrations (acetyl esters), and the band at 1036 cm^{-1} corresponds to C-O vibrations and deformations of OH groups present in ethers and esters [17].

Sorption Experiments

The sorption characteristics of the sorbents studied indicate that the prepared functionalized biochar (F-BC) is suitable for the effective sorption separation of Li from aqueous solutions. The chemical modification of the biochar increased the sorption capacity (Q_{eq}) of BC by nearly 20-fold. Based on the sorption kinetics (Fig. 4), we can describe the sorption process as a relatively fast process, with sorption equilibrium established within 120 minutes of contact time.

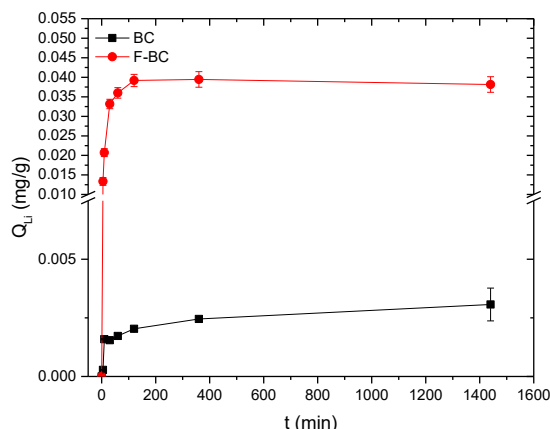


Fig. 4 Sorption kinetics of Li by BC- and F-BC-based sorbents (contact time 5–1440 min; $c_0 = 1$ mg/L; pH = 6.7; sorbent/sorbate ratio: 2.5 g/L)

The effect of the initial Li concentration on the sorption capacity of BC and F-BC indicated potential saturation of the innovative sorbent even within the selected concentration range of 0.5–6 mg/L (Fig. 5). The experimental data were described using the empirical models of the Langmuir and Freundlich adsorption isotherms. We achieved higher fitting efficiency with the Langmuir isotherm, with a coefficient of determination $R^2 = 0.999$ for both BC and F-BC. The obtained isotherm parameters further indicate predicted Q_{\max} values of 0.32 mg/g for F-BC and 0.02 mg/g for BC.

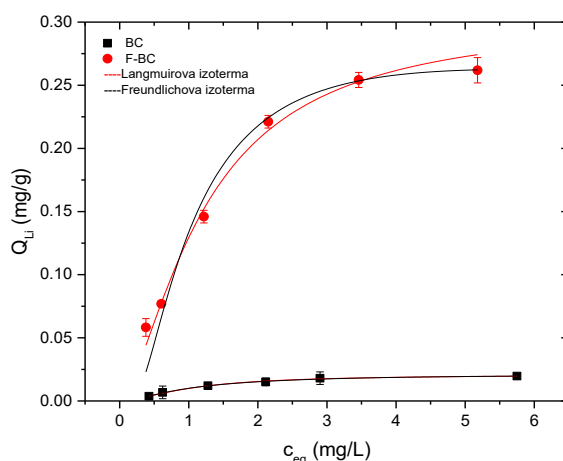


Fig. 5 Dependence of the sorption capacity of BC and F-BC for Li under the following conditions: contact time 120 min; $c_0 = 0.5$ –6 mg/L; pH = 6.2–6.5; sorbent/sorbate ratio: 3 g/L. Experimental data fitted with Langmuir and Freundlich adsorption isotherms

To assess the selectivity of the prepared F-BC-based sorbent, we conducted a series of experiments aimed at investigating the effect of equivalent concentrations of monovalent (Na^+ , K^+) and divalent (Ca^{2+} , Mg^{2+}) ions on Li^+ sorption at a concentration of 1 mg/L. As evident from Fig. 6, the F-BC sorbent is a highly selective sorbent compared to BC. Competition from monovalent ions did not result in a statistically significant decrease in the concentration of sorbable Li^+ compared to BC, where the decrease for the group of monovalent ions was more than 50%. For divalent ions and F-BC, no competitive effect on sorption capacity was observed compared to BC, where the decrease in Q_{eq} was 14%.

Zheng et al. (2021) used crown ether modification of nanomaterials, demonstrating high selectivity of the modified material for Li^+ in the presence of competitive monovalent and divalent ions (Na^+ , K^+ , Ca^{2+} , and Mg^{2+}). They attribute this selectivity to the unique structure of 2-(hydroxymethyl)-12-crown-4 ether, whose cavity diameter is very similar to the radius of Li^+ ions [6].

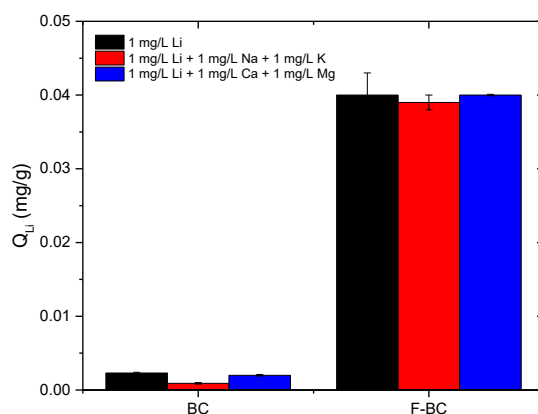


Fig. 6 Effect of monovalent ions (Na^+ and K^+) as well as divalent ions (Ca^{2+} and Mg^{2+}) on the sorption capacity of BC and F-BC for Li (under conditions of 120 min contact time; $c_0 = 1$ mg/L; c_0 (Na^+ , K^+ , Mg^{2+} , Ca^{2+}) = 1 mg/L; pH = 6.5; sorbent/sorbate: 3 g/L).

Conclusion

The application of Li-selective sorbents represents an effective method for extracting Li from aqueous matrices, such as seawater. The sorption capacity of biochar functionalized with 2-(hydroxymethyl)-12-crown-4-ether reached values nearly 20 times higher than that of unfunctionalized biochar (BC). The Li sorption process by the F-BC sorbent is a relatively fast

process, and Li saturation of the biochar occurs within 120 minutes of contact time. The sorption process can be described by the Langmuir adsorption isotherm model. The value of the Q_{\max} parameter obtained by nonlinear regression analysis for F-BC was 0.32 mg/g compared to 0.02 mg/g for BC. The F-BC sorbent exhibited high selectivity for Li^+ in the presence of competing monovalent and divalent ions (Na^+ , K^+ , Ca^{2+} , and Mg^{2+}). These results indicate that the functionalized biochar (F-BC) we prepared is suitable for the selective sorption of Li ions from aqueous solutions and liquid wastes.

Acknowledgments

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