

Synthesis and Characterization of β -Cyclodextrin Modified Biochar Environmental Remediation Materials

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Abstract

In this paper, biochar (BC) was used as raw material, activated by deionizing aqueous solution, NaCl solution, CA solution and HCl solution respectively. Epichlorohydrin (EPI) was used as crosslinking agent, and β -cyclodextrin (β -CD) was used to modify biochar (BC). The prepared modified biochar materials were labeled with β -CDBC, β -CDBC-Na, β -CDBC-CA and β -CDBC-H, respectively. The infrared spectrum, X-ray diffractometer, scanning electron microscope and specific surface area of the four modified materials were tested. The results showed that the C-O stretching vibration peak at 1020 cm^{-1} of the modified materials was slightly offset compared with that of biochar. The characteristic absorption peaks of XRD pattern decrease obviously at $2\theta = 26.7^\circ$ and 29.5° . It can be obviously observed on the electron microscope image that the surface is loaded or formed clathrates, and BET data and graphs also show that the specific surface area of the modified biochar is larger. Therefore, β -cyclodextrin successfully modified biochar and formed clathrates on the surface of biochar or was loaded in the pore structure of biochar, especially β -CDBC-CA achieved better modification effect. Because biochar and β -cyclodextrin raw materials are cheap, easy to prepare and green, and less prone to secondary pollution, it has a good advantage in environmental governance.

Keywords

Biochar, β -Cyclodextrin, Modification, Clathrate, Green Environmental Protection

1. Introduction

With the rapid development of science and technology, economy and agricultural production activities, a large number of organic pollutants, such as polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), DDT, PCDDs and carbamates, have been produced, especially in the process of industrial production, animal husbandry, transportation and residential life. It poses a serious threat to soil security [1].

Polycyclic aromatic hydrocarbons (PAHs) are a class of hydrocarbon organic compounds with two or more benzene rings. Due to man activities such as traffic discharges, sludge applications, wastewater irrigation, and incomplete combustion of fossil fuels [2] [3] make polycyclic aromatic hydrocarbons (PAHs) ubiquitous persistent organic pollutants (POPs) in soils and sediments. In addition, PAHs have carcinogenic, mutagenic and teratogenic properties. By destroying the normal function of liver and kidney, PAHs are genotoxic and carcinogenic to humans and animals [4], which has aroused human attention to environmental safety and health [5] [6]. Therefore, it is urgent to control the pollution of polycyclic aromatic hydrocarbons.

The treatment and remediation of persistent organic pollutants in soil can adopt a variety of remediation methods [7] [8], such as physical remediation technology, chemical remediation technology and biological remediation technology. Physical repair technology has good repair effect, but it needs specific equipment, high cost and large workload. Chemical remediation technology is easy to operate and is conducive to soil remediation in small areas, but the original properties of the soil may be destroyed and secondary pollution may be caused. Bioremediation technology is simple to operate, low cost, and not easy to cause secondary pollution, but its repair cycle is long, and the repair effect is easily affected by external environmental factors. Due to the characteristics of biochar, such as high specific surface area, strong electron exchange, multi-porosity and rich carbon components, it has a stronger adsorption capacity for organic pollutants compared with naturally generated carbon [9] [10]. At the same time, biochar materials are cheap, easy to prepare and environmentally friendly. Therefore, in order to solve problems such as secondary pollution, the modification method of biochar is adopted. Improve the adsorption capacity of organic pollutants and facilitate the removal of organic pollutants.

Biochar is a promising remediation agent due to its high porosity, large specific surface area, abundant surface functional groups and good cation exchange ability [11] [12] [13] [14]. It can be used as a bio-stimulant [13] and an adsorbent [15] in soils contaminated with PAHs. As a bio-stimulant, biochar introduces external nutrients to enhance the diversity and growth of soil microorganisms, thereby degrading soil pollutants [16] [17], while the pore structure and specific surface area of biochar can support and adsorb PAHs, thus achieving the purpose of repairing soil pollutants [18] [19]. However, since the original biochar pores or surfaces contain ash and impurities, which will affect its application effect, more research is to apply modified biochar to environmental remed-

iation. For low molecular weight polycyclic aromatic hydrocarbons (LMW PAHs) (2 - or 3-ring), pine needles, wheat straw and bluegrass biochar all have high absorption of naphthalene. Fu *et al.* [20] found that dissolved black carbon in biochar could also adsorb polycyclic aromatic hydrocarbons and hydrophobic organic compounds of chlorinated benzene through hydrophobic action. Similarly, phenanthrene adsorption effects of biochar were also found on poplar and sawdust biochar [21] and corn stalk, sawdust and pig manure biochar. Another study showed that for 4-cyclic PAHs, biochar extracted from wood, corn straw and soybean straw can adsorb pyrene with removal efficiency of 60% - 99.5% [22]. In addition, the removal rate of more complex PAHs, such as benzo (a) anthracene, benzo (b) fluoranthene, benzo (k) anthracene, benzo (a) pyrene, dibenzo (a, h) anthracene, using coconut waste and orange waste bio-charcoal can reach 23.8% - 84.0%. Zhou *et al.* [23] studied the adsorption properties of wood waste biochar for PAHs. These studies show that biochar prepared from different biomass has great potential and high efficiency for the remediation of organic pollutants, especially for polycyclic aromatic hydrocarbons.

β -cyclodextrin has a hydrophobic inner cavity and a hydrophilic outer cavity structure, and this type of structure has the inclusion of many compounds such as polysubstituted benzene ring and naphthalene ring, which can not only improve the solubility of organic matter, but also improve the adsorption capacity [24] [25]. Due to the poor solubility of β -CD in water, the application range of β -CD is limited. Therefore, fixing β -CD on a specific carrier can improve the dispersion and reuse of β -CD, and can effectively make up for the limitations of β -CD use. At present, the applied research direction of β -CD is mainly concentrated in the field of medicine [26] [27] [28] and environmental governance [29] [30] [31]. Extraction of PAHs with hydroxypropyl - cyclodextrin (HPCD) aqueous solutions has been demonstrated and provides a good estimate of the mineralized or biologically accessible fraction of the phenanthrene. Inorganic materials are easy to separate and have greater convenience in recycling as the loading material of β -CD. Mesoporous silica [32], iron oxide nanoparticles [33], gold surface [34], etc., have been studied for loading materials. Therefore, materials with high specific surface area and large pore volume are an ideal carrier for β -CD.

In recent years, cyclodextrins (CDs) can be used as surfactants due to their unique cavity structure, especially in the field of soil pollution control. More recently, cyclodextrin only has solubilization effect on some hydrophobic organic pollutants, and polycyclic aromatic hydrocarbons are also hydrophobic organic substances. At the same time, biochar has the characteristics of high specific surface area, strong electron exchange, many pores, rich carbonaceous components and surface functional groups, and can also be used for adsorption and removal of pollutants. Therefore, this paper mainly studies the crosslinking of biochar and β -CD to prepare modified biochar materials, improve the performance of biochar, and prepare biochar materials with better adsorption capacity. It is convenient for subsequent use in the treatment of organic pollutants in soil

polycyclic aromatic hydrocarbons. What is more valuable is that β -cyclodextrin and biochar materials themselves are cheap, non-toxic and biodegradable, and belong to environmentally friendly materials, which will not produce secondary pollution.

2. Experiment

2.1. Experimental Drugs and Experimental Instruments

Biochar is the straw biochar (BC) obtained by pyrolysis at a limited oxygen temperature under the condition of 300°C. The experimental raw materials are phenanthrene (PHE), analytically pure, potassium bromide (ArK), spectroscopically pure, sodium hydroxide (NaOH), hydrochloric acid (HCl), citric acid (CA), Epichlorohydrin (EPI), (Analytical pure), purchased from China Sino-pharm Chemical Reagent Co., LTD., the ultra-pure water used in the experiment is deionized water made by the pure water equipment system.

Experimental instruments for testing and characterization include Fourier transform infrared spectrometer (Nicolet-6700, USA), D8ADVANCE X-ray diffraction analysis (XRD) (Bruker, Germany), JEM-6510 scanning electron microscopy, and nitrogen adsorption desorption instrument (BET) (ASAP-2460, USA MAC Instruments).

2.2. Experimental Synthesis Method

Four groups of 20 g dried biochar were added to 500 mL 1 mol/L NaCl solution, CA solution, HCl solution and deionized water respectively, soaked for a period of time, washed repeatedly with deionized water until neutral, and then dried in an oven at 110°C until constant weight. Add 500 mL deionized water into the treated bio-charcoal, adjust the pH value to 4, add 20 mL EPI, shake at 25°C for 12 h, remove and pour out the liquid, add 7.78 g β -CD and 500 mL 6% NaOH solution, continue to shake for 12 h, remove and wash until neutral. Dry in the oven at 65°C to constant weight, that is, β -CDBC-Na, β -CDBC-CA, β -CDBC-H and β -CDBC, respectively.

2.3. Material Characterization Methods

The surface morphology and structure (SEM) of modified biochar materials were observed by the scanning electron microscope. The surface functional groups of the repaired materials were determined by Fourier transform infrared spectrometer (FTIR) and the infrared spectrometer of model Nicolet-6700 was used. The specific surface area and pore size distribution of the samples were determined by BET and ASAP-2460 instrument. The adsorption-desorption isotherm was determined at a temperature of 77K.

3. Results and Discussion

3.1. Infrared Characterization

The FTIR spectra of biochar, β -cyclodextrin and β -cyclodextrin modified bio-

char are shown in **Figure 1**. The C-O stretching vibration peak of β -cyclodextrin modified biochar material is slightly shifted at 1020 cm^{-1} . The peaks near 1652 cm^{-1} are mainly aromatic C=O stretching vibrations (similar to carboxyl group) or aromatic C=C stretching vibrations, and the peak strength of the products after crosslinking of raw biochar and β -cyclodextrin is significantly increased here. Aliphatic methylene ($-\text{CH}_2$) asymmetric stretching peaks and methylene in-plane rocking vibration peaks appeared at 2935 cm^{-1} and 743 cm^{-1} , and ester group (C-O) stretching vibration peaks and O-H out-of-plane vibration peaks appeared at 1157 cm^{-1} and 937 cm^{-1} . The intensity of these peaks was decreased, which may be due to the difference in the crosslinking mode between cyclodextrin and biochar caused by the activation mode. The structure of the product may be that β -cyclodextrin is loaded inside the pore diameter of biochar or forms clathrates on the surface of biochar. In general, the modification does not cause significant changes in the surface functional groups of biochar.

3.2. XRD Characterization

Figure 2 shows the X-ray diffraction pattern of biochar and its modified biochar material. It can be seen from the figure that obvious diffraction peaks appear at $2\theta = 26.7^\circ$ and 29.5° , corresponding to quartz SiO_2 and calcite CaCO_3 , and the characteristic peaks correspond to the crystal faces of biochar. The results showed that all biochar materials had a certain degree of graphitization. The spectrogram of the modified biochar material is superimposed on the characteristic peaks of cyclodextrin and biochar, and no new crystals are formed. It can be observed from the XRD patterns of the four modified biochar materials that the characteristic absorption peaks at $2\theta = 20.9^\circ$, 26.7° , 28.1° , 29.5° and 42.7° are weakened in different ways, which can be obviously observed in the XRD patterns of β -CDBC-CA materials. It can be concluded that the citric acid-activated

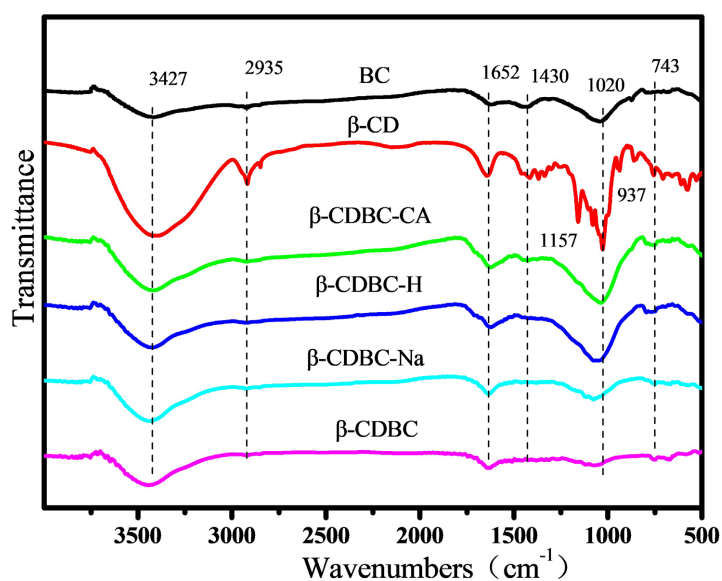


Figure 1. Infrared spectrum of β -cyclodextrin modified biochar material.

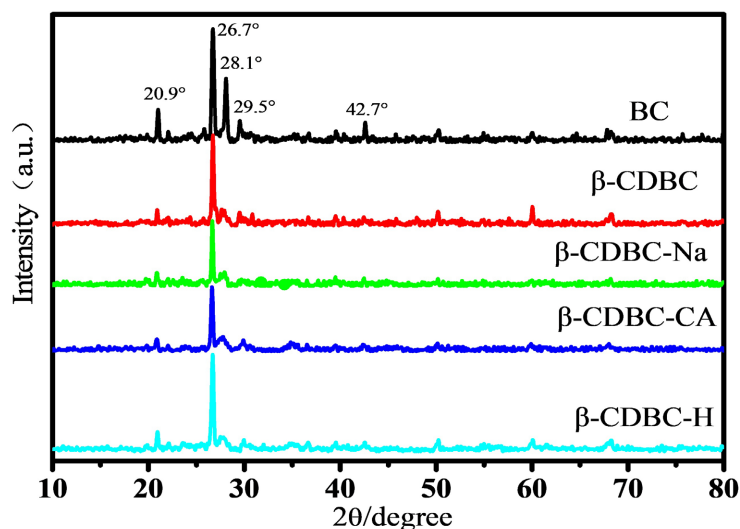


Figure 2. XRD pattern of β -cyclodextrin modified biochar material.

biochar material enables the successful cross-linking of β -cyclodextrin with biochar and the biochar material may be encapsulated in the β -cyclodextrin cavity, forming a clathrate, or successfully loaded on the biochar surface.

3.3. SEM Characterization

It can be seen from **Figure 3(a)** that the surface of the biochar raw material is relatively flat and smooth when magnified 1000 times, and the layered porous structure can also be obviously observed. It can be seen from **Figure 3(b)** that the surface of β -CDBC-CA material can be observed that the surface of biochar becomes not smooth, and a thin layer is formed at the same time, increasing the porosity of raw materials. From the side, it can be seen that biochar still retains a porous structure, so it can be considered that the β -cyclodextrin modified biochar is successful. β -cyclodextrin forms clathrates on the surface of biochar or is supported in the pore structure of biochar, which is consistent with the results of XRD characterization.

3.4. BET Characterization

The BET analysis results of biochar and its β -cyclodextrin modified biochar are shown in **Table 1**. In general, the adsorption capacity of the material is proportional to its total surface area, and the larger the specific surface area, the more contact points between the solid surface and the gas or liquid, thereby increasing the chance of adsorption. The surface and porosity analysis of the biochar treated in four different ways showed that the non-activated modified biochar (β -CDBC), NaCl-activated modified biochar (β -CDBC-Na) and HCl-activated modified biochar (β -CDBC-H) were better than that of the biochar raw material (BC). The specific surface area and pore volume increased to varying degrees, especially the specific surface area of HCl activated modified biochar (β -CDBC-H) increased more obviously, which may be because HCl played a certain role in

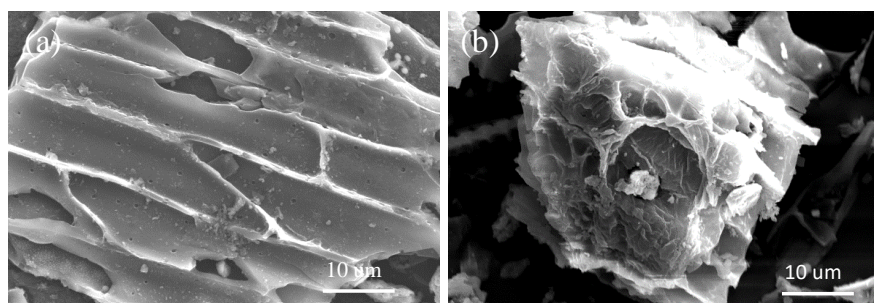


Figure 3. SEM image of β -cyclodextrin modified biochar material. (a) Electron microscope image of biochar ($\times 1000$); (b) Electron microscopic image of β -CDBC-CA material ($\times 1000$).

Table 1. Specific surface area of β -cyclodextrin modified biochar materials

Sample	Specific surface area (m^2/g)	Pore volume (cm^3/g)	Pore size (nm)
BC	5.1761	0.008141	16.4115
β -CDBC	6.9937	0.012770	73.0367
β -CDBC-Na	8.2936	0.015077	72.0367
β -CDBC-CA	18.2654	0.029689	65.0180
β -CDBC-H	17.8629	0.030612	68.5480

cleaning the pores of biochar when activating biochar. However, the specific surface area and pore volume values of NaCl-activated modified biochar (β -CDBC-Na) were smaller than those of the other two types of modified biochar, and the pore size was relatively largest, which may be due to the fact that NaCl-activated biochar mainly focuses on ion exchange and does not clean the pore channels of biochar. Compared with the other two kinds of biochar, citric acid activated biochar had a maximum specific surface area of $18.2654 \text{ m}^2/\text{g}$ and a pore size of 65.0180 nm , which may be due to the fact that citric acid not only cleaned the pore size of biochar to a certain extent, At the same time, this may also be due to the esterification of citric acid with hydroxyl (-OH) groups in the outer cavity of β -cyclodextrin, which is easy to form macromolecules loaded on the surface of biochar to form inclusion compounds, so that the citric acid-activated modified biochar material (β -CDBC-CA) has a larger specific surface area, which is consistent with the results of electron microscopy characterization. Modified biochar has a larger specific surface area and is more conducive to the adsorption of organic pollutants.

Figure 4(a) shows the adsorption and desorption curves of biochar and its modified biochar. According to IUPAC classification, the adsorption curves of the four materials belong to class IV, mainly mesoporous adsorbent materials. It can also be observed from **Figure 4(b)** that the pore size is concentrated in the range of $30 - 50 \text{ nm}$, and there are also some macro-porous structures in the range of $50 - 100 \text{ nm}$. Therefore, single-multilayer adsorption can occur on the mesoporous wall of β -CDBC-CA in the mesoporous, which is conducive to the adsorption of polycyclic aromatic hydrocarbons of pollutants.

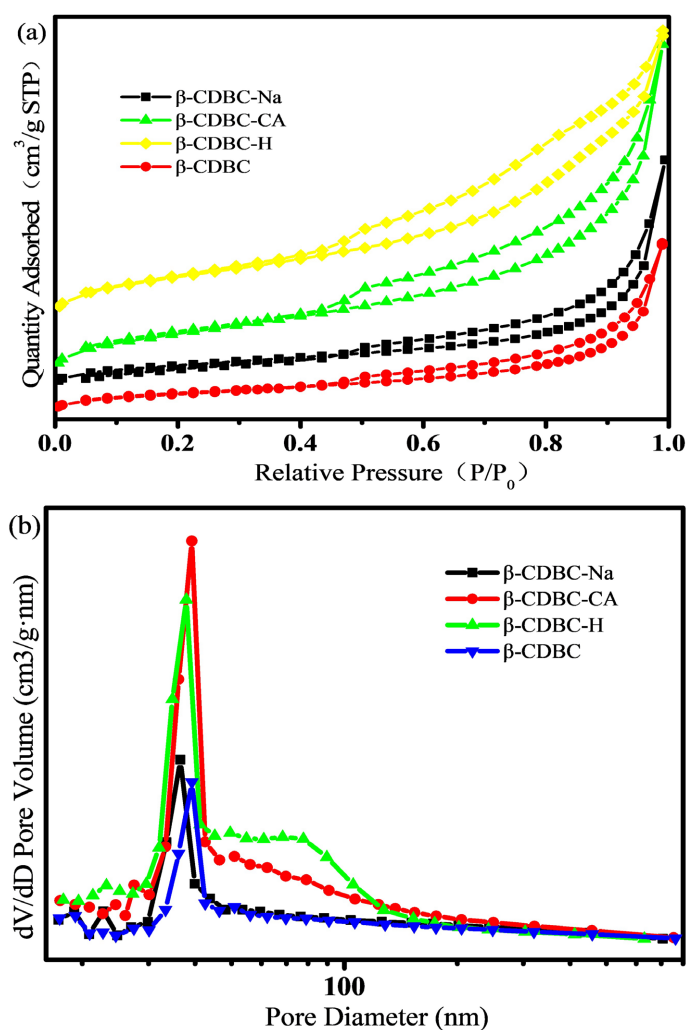


Figure 4. BET characterization of β -cyclodextrin modified biochar materials. (a) Adsorption-desorption curves of modified biochar materials; (b) Pore size curves of modified biochar materials.

4. Conclusion

In summary, biochar (BC) material was activated by deionizing aqueous solution, NaCl solution, CA solution and HCl solution in this paper. Epichlorohydrin (EPI) was used as a crosslinking agent, and β -cyclodextrin (β -CD) was used to modify biochar (BC). The prepared modified biochar materials were labeled with β -CDBC, β -CDBC-Na, β -CDBC-CA and β -CDBC-H, respectively. The results showed that β -cyclodextrin successfully modified biochar and formed clathrates on the surface of biochar or was supported in the pore structure of biochar, which increased the specific surface area of biochar, especially β -CDBC-CA achieved better modification effect. The modified biochar material can be well applied to the adsorption and removal of organic pollutants.

Data Availability

The data used and/or analyzed during the current study are available in the sup-

plemental materials or from the corresponding author on reasonable request.

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Conflicts of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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