



High-rate removal of As(III) from aqueous system with sulfhydryl magnetic biological bamboo charcoal nanocomposites prepared by chemical co-precipitation method

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Abstract: Sulfhydryl magnetic biological bamboo charcoal nanocomposite (BBC@nFe-SH) was prepared by chemical co-precipitation method for the robust capture of As(III) from aqueous solutions. The novel BBC@nFe-SH shows favorable magnetic field strength (83376 A/m), which enables BBC@nFe-SH to be quickly recovered from aqueous solution. The maximum As(III) adsorption capacity is as high as 98.63 mg/g at pH 5.0 and 40 °C, reaching reaction equilibrium within 120 min. Various characterizations (e.g., SEM, FTIR, VSM and XPS) suggest that As(III) prefers to coordinate with surface oxygen groups bonded to the surface. BBC@nFe-SH displayed high stability and recyclability throughout the removal process, which could be easily activated by 1 mol/L NaOH after usage. Thus, the novel BBC@nFe-SH has promising applications for As(III) treatment.

Key words: arsenic-containing wastewater; bamboo biochar; chemical co-precipitation; adsorption; recyclability; wastewater treatment

1 Introduction

Arsenic, a metalloid element widely distributed in nature, is a ubiquitous environmental pollutant that can pose a great threat to the ecological environment and human health [1]. Arsenic can affect enzyme activity and gene expression, poison the gastrointestinal, cardiovascular, and nervous systems, and increase the incidence of cancer after entering the human body, thus seriously affecting the normal function of human organs. In addition, arsenic has accumulative

toxicity in organisms, and the damage of arsenic contamination to the ecological environment has been recognized to be long in duration and irreversible and to affect a wide range [2]. In natural aquatic environments, arsenic is predominantly present as As(III) and As(V), which are interconvertible under given conditions. Research has indicated that As(III) is more toxic to inorganic arsenic, which is 35–60 times more toxic than As(V). According to the World Health Organization, at least 50 million people are affected by arsenic pollution, especially in Southeast Asia and Latin America. China is also one of the most severely

polluted countries [3,4].

Existing techniques for arsenic removal from aqueous systems include precipitation, oxidation, ion exchange, membrane separation, biological treatment and adsorption [5–7]. Adsorption is recognized as one of the most promising methods due to its low cost, high efficiency, simple operation and wide range of applications [8,9]. Biochar is widely used in heavy metal treatment as an environmentally friendly adsorbent due to its distinct properties (abundant functional groups, strong cation-exchange ability and stable chemical properties) [10,11]. However, the surface charge of most biochars is negative, exhibiting lower adsorption efficiency to anionic compounds, such as AsO_4^{3-} , AsO_3^{3-} , SbO_4^{3-} and $\text{Cr}_2\text{O}_4^{2-}$, in water [12,13]. In this case, biochar is usually modified by activation, surface functional group modification and metal oxide modification to improve its adsorption performance to anions in water [14,15]. FAN et al [16] used sulfhydryl-modified rice straw biochar for the removal of Cd. The results showed that the adsorption performance was enhanced by 285.4% compared with that of common rice straw biochar. ZHU et al [17] modified wheat biochar with goethite to improve the adsorption property to As(III). The experimental results indicated that the adsorption capacity of goethite-modified biochar material to As(III) was almost 60 times that of common biochar. In practice, as an adsorbent for wastewater treatment, biochar is difficult to separate, thus causing secondary pollution to the environment [18,19]. Magnetic nanomaterials can be separated from aqueous solution through an external magnetic field [20,21]. Magnetization treatment of biochar not only improves the stability and regeneration capacity but also affects the

specific surface area, surface groups and porous structure of biochar [22].

Therefore, in this work, bamboo biochar (BBC) was prepared by the pyrolysis of *Phyllostachys pubescens* as a precursor. The magnetic biological bamboo charcoal nanocomposites (BBC@nFe) were obtained by reduction deposition of nano iron treatment of BBC through chemical coprecipitation process under a mild liquid solution. Subsequently, functional groups of BBC@nFe-SH were modified by sodium dimethyl dithiocarbamate to prepare sulfhydryl magnetic biological bamboo charcoal nanocomposites (BBC@nFe-SH). The effects of the initial solution concentration, pH, solution temperature and adsorbent dosage on As(III) removal by BBC@nFe-SH were investigated. Furthermore, adsorption kinetic and thermodynamic models were employed to preliminarily explore the adsorption mechanism of As(III) by BBC@nFe-SH combined with the characterization of BBC@nFe-SH.

2 Experimental

2.1 Preparation of BBC@nFe-SH

Phyllostachys pubescens, an easily available and environmentally friendly plant material, was used to prepare BBC@nFe-SHs. The bamboo charcoals before and after modification are named as bamboo biochar (BBC) and sulfhydryl magnetic biological bamboo charcoal nanocomposites (BBC@nFe-SH), respectively. The schematic diagram for the synthesis of BBC@nFe-SH is shown in Fig. 1.

In a typical procedure, *Phyllostachys pubescens* was treated by high-temperature pyrolysis (600 °C). After separation and drying, BBC was obtained and stored for later use. BBC (10 g) and water (250 mL)

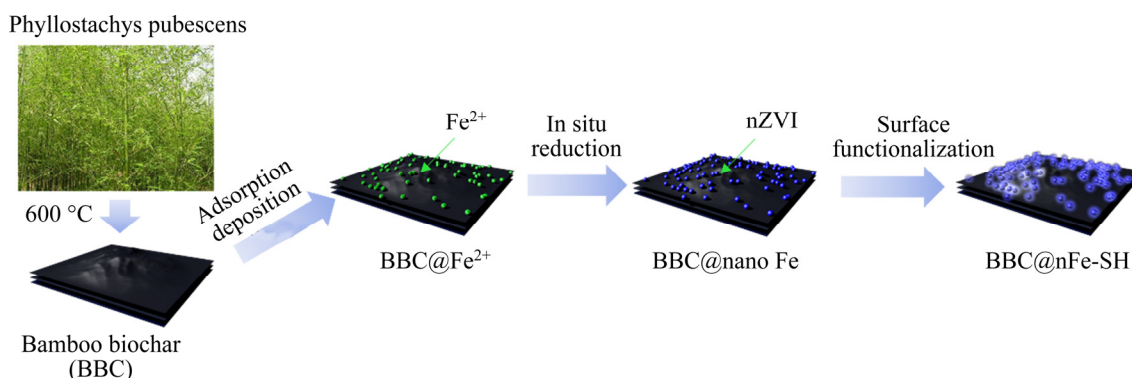


Fig. 1 Schematic illustration of preparing process of BBC@nFe-SH

were added to a three-neck flask in a water bath at 40 °C under stirring at 300 r/min to disperse BBC uniformly. Then, 100 mL of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ (0.2 mol/L) was added to the solution, and 150 mL of sodium borohydride (40 g/L) was slowly added at 2 drop/s. After mixing constantly for 2 h, the sample was washed with a mixture of deionized water and absolute alcohol until the pH was neutral. Finally, the BBC@nFe was obtained through slow oxidation in a constant-temperature drying oven at 80 °C for 24 h and sealed for later use.

BBC@nFe (5.0 g) and 300 mL of mixed liquid with a volume ratio of ethanol to water of 2:1 and 5.0 g of sodium dimethyl dithiocarbamate were added into a three-neck flask, and then the mixture was mixed in a water bath (60 °C) under stirring at 300 r/min for 2 h to obtain a suspension. The suspension was washed with deionized water and alcohol until the pH was neutral. Finally, the BBC@nFe-SH was obtained through filtration and dried in a constant-temperature drying oven at 60 °C for 12 h and sealed for later use.

2.2 Adsorption experiments

2.2.1 Batch experiments

The initial As(III) concentration was kept at 100 mg/L for all experiments. The initial solution pH, contact time and environmental temperature for As(III) removal by BBC@nFe-SH were investigated at an adsorbent dose of 2.0 g/L. The pH impact was firstly investigated at 25 °C and pH values ranging from 2 to 11 with a contact time of 12 h. The effect of contact time was studied at contact time ranging from 5 to 1440 min, 25 °C and a pH of 5.0. The effect of environmental temperature (25, 40 and 55 °C) was investigated at a pH of 5.0 and a contact time of 120 min. Moreover, the effect of adsorbent dosage on As(III) removal by BBC@nFe-SH was investigated at 25 °C, a pH of 5.0 and a contact time of 120 min.

2.2.2 Adsorption kinetics

Eight Erlenmeyer flasks were prepared, and 50 mL of solution containing 100 mg/L As(III) at pH 5.0 was added to each flask. Then, 0.1 g of BBC@nFe-SH was added to each flask, and the mixtures were shaken in a thermostatic shaker at 25 °C. Samples were collected after 5, 10, 20, 40, 60, 80, 120, and 180 min for analysis. The pseudo-first-order and pseudo-second-order kinetic models were used to calculate the adsorption results, which

can be described by linear forms in Eqs. (1) and (2), respectively:

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (1)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (2)$$

where q_e (mg/g) and q_t (mg/g) are the adsorbed As(III) amounts at equilibrium and time t (min), respectively; k_1 (min^{-1}) and k_2 ($\text{g}/(\text{mg} \cdot \text{min})$) are the rate constants of pseudo-first-order and pseudo-second-order adsorption, respectively.

2.2.3 Adsorption isotherm

All adsorption experiments were conducted in flasks, which contained 50 mL of aqueous solution of As(III) at the required concentrations (10, 20, 30, 40, 50, 60, 70, 80, 90, and 100 mg/L). The pH of each flask was adjusted to 5.0. After that, 0.1 g BBC@nFe-SH adsorbent was added, and the flasks were shaken in a thermostatic shaker at 25 °C for 120 min. The concentration of As(III) was determined after the reaction, and then the Langmuir and Freundlich isotherm equations were used to calculate the experimental results, expressed as Eqs. (3) and (4), respectively:

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{K_L q_m} \quad (3)$$

$$\lg q_e = \left(\frac{1}{n} \right) \lg C_e + \lg K_f \quad (4)$$

where C_e is the equilibrium concentration; q_m is the maximum adsorption capacity; K_L is the Langmuir constant; n is a parameter to evaluate the favorableness of the adsorption process; K_f is the Freundlich constant.

2.2.4 Adsorption-desorption experiments

The desorption of As(III) from the used BBC@nFe-SH was conducted by NaOH solution (1 mol/L). The adsorbent was repeatedly used after filtration and dried to evaluate the reusable performance of BBC@nFe-SH.

2.3 Analytical methods

The As(III)-containing wastewater used in this study was prepared with NaAsO_2 and deionized water. Specifically, a stock solution of As(III) with a content of 1 g/L was prepared by dissolving a certain amount of NaAsO_2 in a beaker, and working solutions of the required concentrations were obtained by diluting the As(III) stock solution with

deionized water.

Dissolved As(III) was analyzed by a hydride generating-atomic fluorescent spectrometer. All tests were finished in 24 h, and the blank test was given with deionized water. The detection limit in this method was 1 $\mu\text{g/L}$. The recovery of arsenic was up to 90%, and the relative standard deviation was less than 1%.

The morphology of the samples was observed by scanning electron microscopy (SEM, Zeiss Sigma HD) operated at 10000–80000 times magnification. The functional groups in the bamboo charcoal (before and after modification) and the used BBC@nFe-SH were analyzed by Fourier transform infrared spectroscopy (FTIR, Bruker Vertex 70). The elements and valence variation of BBC@nFe-SH before and after adsorption were characterized by X-ray photoelectron spectrometry (XPS, EscaLab Xi⁺). The bamboo charcoal before and after adsorption was pretreated at 1000 °C in an air atmosphere, and then the residual mass of the samples was analyzed by a thermogravimetric analyzer (TG, STA 2500).

3 Results and discussion

3.1 Characterization

3.1.1 SEM images and BET specific surface area

The surface morphologies of BBC and BBC@nFe-SH are presented in the SEM images in Figs. 2(a) and (b), respectively. The surface morphology and microstructure of BBC were greatly changed after modification. There were various structural lumps on the surface of BBC,

with different sizes and irregular forms. After modification, the content of impurities on the surface of BBC@nFe-SH was obviously reduced, and a comparatively regular shape appeared. Moreover, numerous spherical particles with sizes of 150–300 nm were attached on the BBC@nFe-SH surface, which were most likely magnetic iron nanospheres.

The original morphology of BBC@nFe-SH and the distribution of the four elements C, Fe, O, and S in this morphology area are presented in Fig. 2(c). The color depth and density of element points represented the content of elements to some degree. Figure 2(c) shows that the contents of C and O were higher, while those of Fe and S were relatively low. This is because the main component of BBC@nFe-SH was bamboo charcoal, which had the highest content of C and O, while Fe and S were mainly found by magnetization and functional group modification. From Fig. 2(c), S exhibited an even element distribution, and Fe and S were distributed intensively in the area of less C distribution. Furthermore, granular spheres can be seen clearly in this area, which confirms the above SEM analysis that the granular spheres are magnetic nanospheres. The surface area and microstructures of BBC@nFe-SH and biochar control samples were observed by BET instrument. According to BET analysis, the specific surface areas of raw BBC and BBC@nFe-SH were 263.142 and 371.291 m^2/g , respectively. Larger specific surface area will directly affect the removal efficiency of pollutants. In order to explore the recyclability mechanism of materials in more detail,

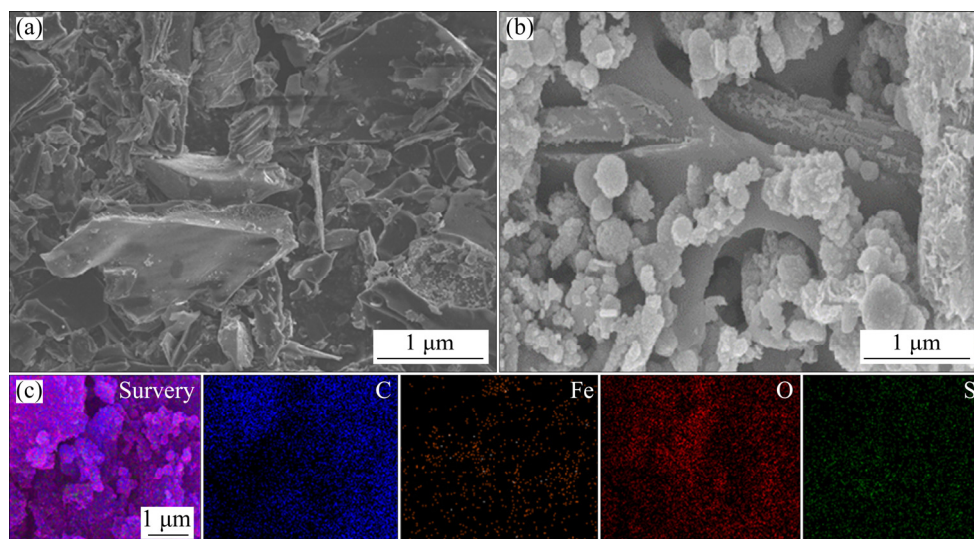


Fig. 2 SEM images of BBC (a), BBC@nFe-SH (b), and EDAX-mapping spectra of BBC@nFe-SH (c)

magnetic hysteresis loop patterns of BBC and BBC@nFe-SH were measured by vibrating sample magnetometer. The results show a ferromagnetic property of 83376 A/m, much stronger than that of the raw BBC (18 A/m).

3.1.2 FTIR spectra

The surface groups of BBC@nFe-SH were analyzed by FTIR and compared with those of BBC and BBC@nFe (Fig. 3). The broad peak at approximately 3135 cm^{-1} for BBC was attributed to the stretching vibration of hydroxyl groups ($-\text{OH}$) or the surface bond water of BBC. The absorption at 1619 cm^{-1} was attributed to the stretching vibration of the surface carboxyl groups of BBC or the carbonyl groups ($\text{C}=\text{O}$) of esters, and the peak at 1400 cm^{-1} corresponded to the symmetric bending vibration of methyl groups ($-\text{CH}_3$). From the FTIR spectra, the $-\text{OH}$ peak transmission of BBC@nFe-SH decreased, implying the formation of a large number of $-\text{OH}$ groups after modification. Additionally, the appearance of slight vibrations at $1619\text{--}11621\text{ cm}^{-1}$ indicated that the quantity and quality of $\text{C}=\text{O}$ changed [23]. In the

spectra of BBC@nFe-SH, the peak at about 536 cm^{-1} was ascribed to the stretching vibration of $\text{Fe}-\text{O}$, which demonstrated that iron ions were successfully supported on the surface of the adsorbent [24]. Compared to BBC@nFe, a new characteristic peak appeared at $900\text{--}1600\text{ cm}^{-1}$ in BBC@nFe-SH, and the peaks observed at 1514 , 1145 and 988 cm^{-1} were attributed to the stretching vibrations of $\text{N}-\text{C}=\text{S}$, $\text{C}=\text{S}$ and $\text{C}-\text{S}$, respectively [25]. Compared to BBC@Fe, the spectrum of BBC@nFe-SH showed obvious changes in terms of peak shifts due to the modification of the sulfate radicals. New peaks appeared at 1145 and 988 cm^{-1} , which were attributed to the characteristic stretching vibrations of $\text{C}=\text{S}$ and $\text{C}-\text{S}$, respectively. Moreover, it is obvious that the amount of oxygen-containing functional groups of BBC@nFe-SH increased. New organic functions were introduced, which could form As(III) complexes by ionic reactions and complexing reactions, thus enhancing the adsorption property of As(III) to BBC@nFe-SH [26].

3.1.3 TG-DTA curves

TG-DTA analysis was carried out in an air atmosphere to determine the thermal stability of the BBC@nFe-SH. As shown in Fig. 3(b), two significant changes are observed in the TG and DTA curves from 0 to $1000\text{ }^{\circ}\text{C}$. Firstly, the mass loss at $30\text{--}200\text{ }^{\circ}\text{C}$ is attributed to the evaporation of physisorbed water. Secondly, the mass loss between 200 and $600\text{ }^{\circ}\text{C}$ is attributed to the removal of organic groups. In general, when the temperature is increased to approximately $200\text{ }^{\circ}\text{C}$, the mass loss is only less than 10% , and the mass loss of the material over the entire heating process is not large, which proves that BBC@nFe-SH has good thermal stability.

3.2 Single-factor analysis results of As(III) removal by BBC@nFe-SH

3.2.1 Effect of initial pH

The initial pH of aqueous solution is an important factor affecting the adsorption process of As(III). In the present work, the effect of the initial solution pH on As(III) adsorption by BBC@nFe-SH was investigated at different initial pH values ranging from 2.0 to 12.0 . The result in Fig. 4(a) showed that pH had a great influence on the adsorption of the adsorbent. Under acidic

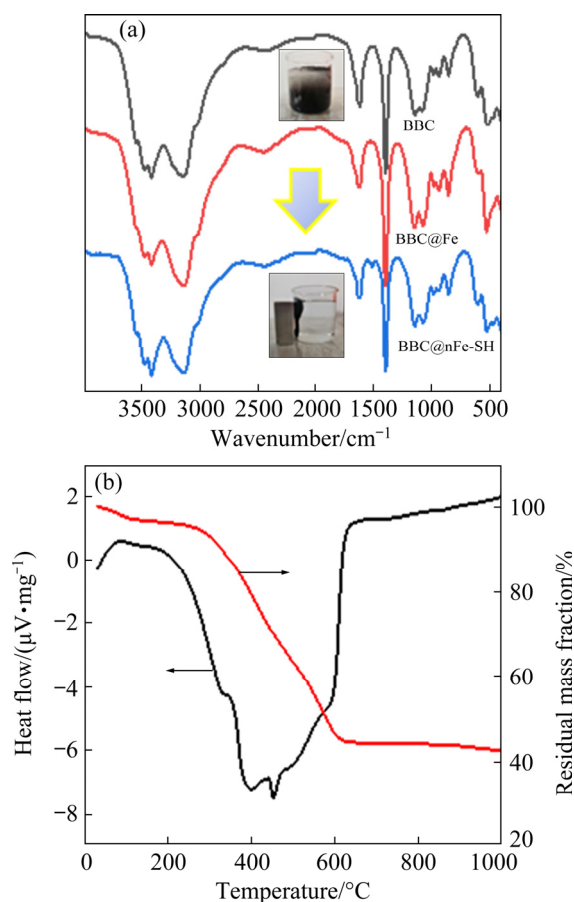


Fig. 3 FTIR spectra of BBC, BBC@Fe and BBC@nFe-SH (a), and TG-DTA curves of BBC@nFe-SH (b)

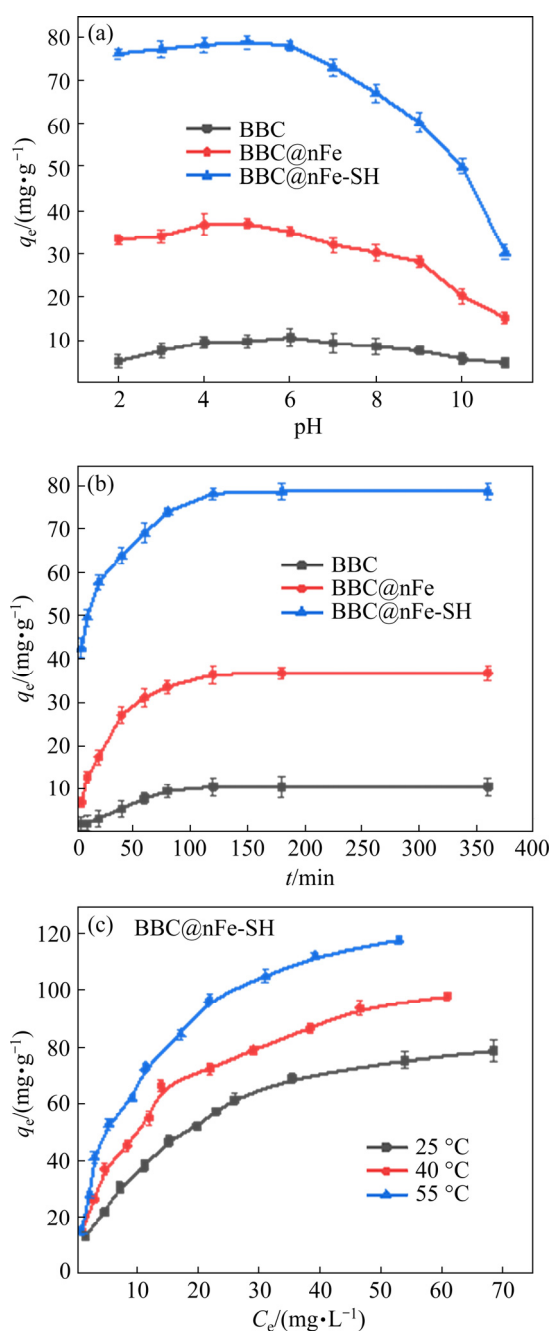


Fig. 4 Effects of pH (a), contact time (b) and temperature (c) on As(III) adsorption

conditions, the adsorption of As(III) on BBC@nFe-SH remained at a high level. With increasing pH from 6.0 to 11.0, however, the adsorption capacity decreased dramatically. BBC@nFe-SH under acidic conditions showed higher adsorption performance, which may be ascribed to the increasing electrostatic repulsion between the adsorbent and As(III). Due to the increase in pH, the adsorbent surface charge gradually changed from positive to negative, while the As(III) in aqueous solutions was negatively charged, resulting in electrostatic

repulsions between As(III) species and the absorbent surface. Moreover, more hydroxyl ions in the solution led to significant competition for As(III) adsorption sites, thereby inhibiting the adsorption of As(III) on the adsorbent. Iron oxide in BBC@nFe-SH can oxidize AsO_3^{3-} to AsO_4^{3-} at a low pH due to its oxidation ability to As(III). When $\text{pH} > 8.0$, As(III) cannot be oxidized because iron oxide in BBC@nFe-SH has a certain reductive ability, which might be the reason why the adsorption ability of BBC@nFe-SH decreased dramatically under alkaline environments.

3.2.2 Effect of contact time

Figure 4(b) describes the effect of contact time on the removal of As(III) by BBC@nFe-SH. The adsorption amounts of As(III) increased with increasing time. In the initial adsorption stage, the adsorption rate of As(III) increased quickly and then slowed down with increasing contact time. The rapid sorption stage is likely due to the abundant functional groups on the surface of the material, which could provide a large number of adsorption sites for As(III). As the reaction time increased, more available sites for As(III) became occupied. The adsorption rate of As(III) slowed down until reaching equilibrium, and the concentration of As(III) in aqueous solution finally reached a stable value. In the adsorption process, the adsorption capacity reached saturation in 120 min and was seldom influenced with time thereafter.

3.2.3 Effect of temperature

As temperature can change the interaction between the adsorbent and ions by affecting the stability of the adsorbent, the effect of temperature on As(III) adsorption was investigated under three different temperature conditions (25, 40 and 55 °C). As shown in Fig. 4(c), the adsorption ability of As(III) on BBC@nFe-SH increased with increasing temperature. This result indicated that temperature exerted a great influence on the adsorption process, which may be due to the improvement in the As(III) diffusion velocity in solution with increasing temperature. The chaos of the system increased, the reaction activation energy was enhanced, and subsequently, the binding ability of active sites increased. Additionally, the inner porous expansion of BBC@nFe-SH resulted in an increase in the effective collision probability with As(III), followed by an increase in the adsorption capacity. Therefore, increasing the temperature was beneficial to

improving the adsorption of As(III) onto BBC@nFe-SH, and the temperature of 55 °C theoretically gave a better adsorption result. Based on actual considerations, 25 °C was selected as the optimal adsorption temperature.

3.3 Adsorption kinetics

Adsorption kinetics was used to explore the relationship between the adsorption rate and activation energy dynamics, which provides a theoretical basis for analyzing the variation in heavy metal concentration with time in the adsorption process [27,28]. In the present work, the pseudo-first-order and pseudo-second-order equations were applied to fitting the adsorption process, as shown in Figs. 5(a) and (b), respectively, and the kinetic fitting results are listed in Table 1.

As shown in Table 1, the correlation coefficient of the pseudo-second-order kinetic model ($R^2=0.997$) was higher than that of the pseudo-first-order kinetic model ($R^2=0.875$), and the calculated value of q_e (81.00 mg/g) for As(III) was in better agreement with the experimental q_e . This indicated that the adsorption process agreed well with the pseudo-second-order kinetic model. Therefore, it is concluded that the rate control in the adsorption process of As(III) by BBC@nFe-SH was achieved through chemical adsorption (electron

sharing and electron gain or loss) or on the basis of chemical reaction. The adsorption process was a chemical adsorption process mainly involving ion exchange and complexation reactions between As(III) and functional groups in the adsorbent.

3.4 Adsorption isotherms

Langmuir and Freundlich isotherms were employed to describe the adsorption process of As(III) in this study, as shown in Figs. 6(a) and (b). The Langmuir isotherm model is widely applied for monolayer adsorption that assumes identical adsorption sites and no interaction between adsorbates [29,30]. The Freundlich adsorption isotherm is an empirical model that assumes ideal and multilayer adsorption on heterogeneous surfaces. According to Freundlich isotherms, there are different adsorption sites with varying adsorption energies on the surface of adsorbents, and the adsorption capacity increases with increasing adsorbent dosage. The parameters obtained from the two isotherm models are summarized in Table 2.

As shown in Table 2, the isotherm data fit well to the Langmuir isotherm model, as the correlation coefficient obtained from the Langmuir isotherm model ($R^2=0.993$) was larger than that from the Freundlich isotherm model ($R^2=0.957$), indicating

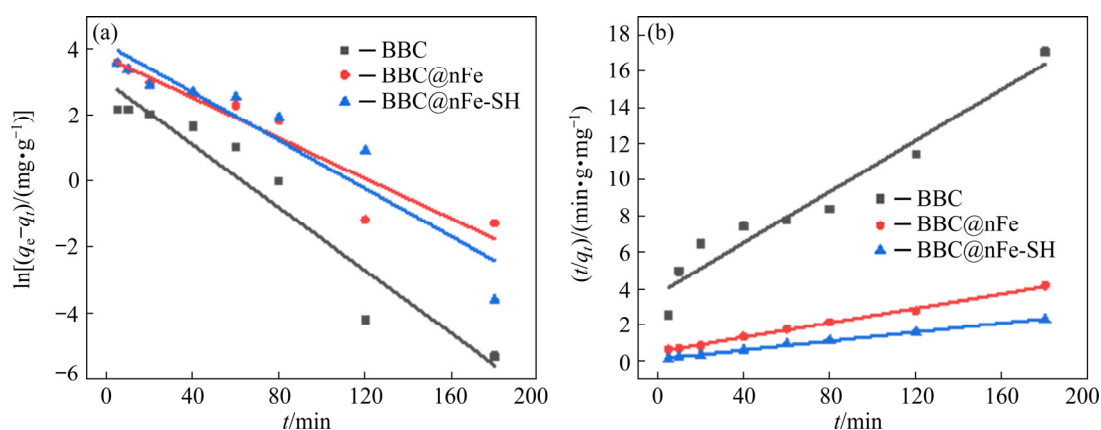


Fig. 5 Kinetics curves for As(III) adsorption on BBC@nFe-SH and their control samples from aqueous solutions: (a) Pseudo-first-order model; (b) Pseudo-second-order model

Table 1 Kinetic parameters for adsorption of As(III) by BBC before and after modification

Sample	Pseudo-first-order kinetic model			Pseudo-second-order kinetic model		
	$q_{\text{cal}}/(\text{mg} \cdot \text{g}^{-1})$	k_1/min^{-1}	R^2	$q_{\text{cal}}/(\text{mg} \cdot \text{g}^{-1})$	$k_2/(\text{g} \cdot \text{mg}^{-1} \cdot \text{min}^{-1})$	R^2
BBC	19.89	0.047	0.916	14.16	0.0180	0.937
BBC@nFe	41.679	0.031	0.900	50.00	0.0001	0.996
BBC@nFe-SH	62.178	0.036	0.875	81.00	0.0001	0.997

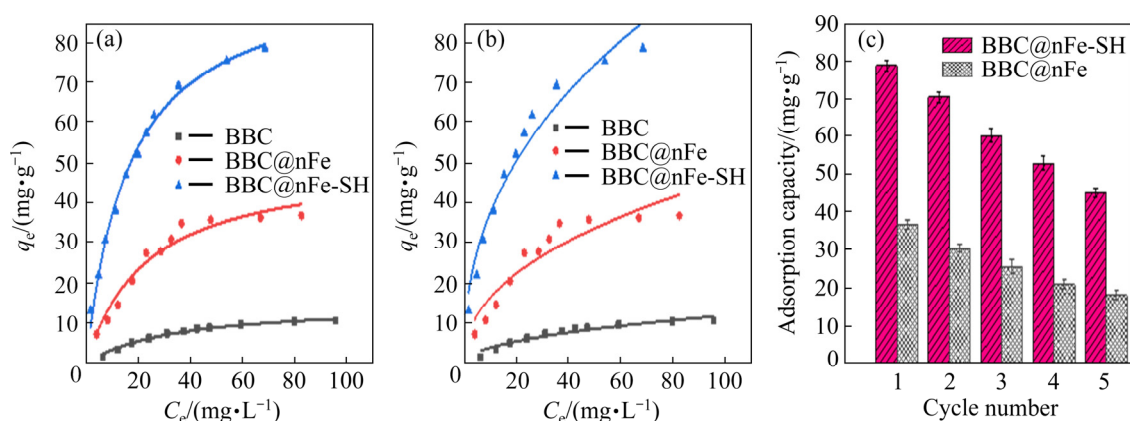


Fig. 6 Isothermal curves (a, b) and adsorption cycles (c) for As(III) adsorption on BBC@nFe-SH and their control samples from aqueous solutions: (a) Langmuir isotherm model; (b) Freundlich isotherm model

Table 2 Adsorption isothermal parameters for adsorption of As(III) by BBC@nFe-SH

Sample	Langmuir isotherm model			Freundlich isotherm model		
	$q_m/(\text{mg} \cdot \text{g}^{-1})$	$K_L/(\text{L} \cdot \text{mg}^{-1})$	R^2	n	$K_F/(\text{mg} \cdot \text{g}^{-1})$	R^2
BBC	15.030	0.029	0.982	2.045	1.257	0.917
BBC@nFe	50.820	0.041	0.995	2.096	5.795	0.950
BBC@nFe-SH	98.625	0.060	0.993	2.347	14.072	0.959

that the adsorption of As(III) on BBC@nFe-SH was more closely related to monolayer adsorption. From the fitting results of the Langmuir isotherms, BBC@nFe-SH possessed the strongest adsorption capacity of As(III), with a maximum adsorption capacity (q_m) of 98.625 mg/g and adsorption constant (K_L) of 0.060 L/mg, demonstrating that the adsorption of As(III) onto BBC@nFe-SH is easy to perform.

Biochar, known as a simple and low-cost adsorption material, is widely used in the treatment of As(III)-containing wastewater. Many studies and experiments have been performed to study the adsorption performance of As(III) on different biochars and modified biochars [31]. In this work, BBC@nFe-SH obtained from the modification of BBC was used to treat As(III)-containing wastewater. By comparing the adsorption capacity with that of other biochars, it can be seen that BBC shows better adsorption performance than most of the biochars except for biochar from rice husk and palm pomace, as listed in Table 3.

Due to the firm fixation of organic molecules, the composites have stronger stability, so BBC@nFe-SH still maintains high arsenic adsorption performance after 5 cycles of use (Fig. 6(c)). The results reveal that BBC@nFe-SH

Table 3 Comparison of removal properties of BBC@nFe-SH and other adsorbents for As(III)

Adsorbent	Adsorption capacity/($\text{mg} \cdot \text{g}^{-1}$)	Source
Perilla leaf biochar	11.01	[32]
Goethite modified biochar	65.20	[17]
Sludge biochar	0.07	[33]
Rice husk biochar	19.3	[34]
Biochar from palm pomace	18.9	[35]
Biochar modified by Fe, Mn and La	14.9	[36]
Chitosan derivatives	94.0	[37]
BBC@nFe-SH	98.63	This work

exhibits excellent adsorption ability equivalent to chitosan derivatives and iron-manganese composite oxides, far higher than other conventional biochar materials.

3.5 Adsorption mechanism

The FTIR spectrum revealing the variation in the functional groups of BBC@nFe-SH is shown in Fig. 7. Compared to the unadsorbed BBC@nFe-SH, the chief peak transmittance increased and shifted to different degrees after adsorption. The

absorbances at 3135, 3413, 3472 and 3545 cm^{-1} decreased significantly, implying that the hydroxyl groups on the surface of BBC@nFe-SH significantly decreased [38]. In addition, the small vibrations of the peak positions of —OH , C=O and Fe—O indicated that new functional groups were formed from the complex reaction between these oxygen-containing functional groups and As(III). After the adsorption, the emerging characteristic peak at 943 cm^{-1} was attributed to As—O stretching [39]. The adsorption peak of the C=S stretching vibration shifted from 1145 to 1149 cm^{-1} . The adsorption peak of the N—C=S stretching vibration shifted from 1145 to 1149 cm^{-1} , and the peak of the C—S stretching vibration was weakened. These results indicated that the coordination occurred between As(III) and both C=S and C—S [40].

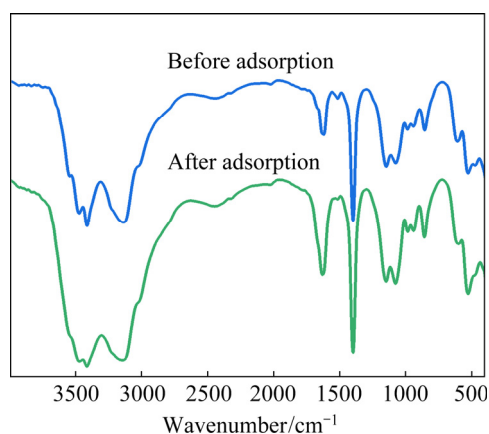


Fig. 7 FTIR spectra of BBC@nFe-SH before and after As(III) adsorption

X-ray photoelectron spectrometry was used to analyze BBC@nFe-SH before and after As(III) adsorption (Fig. 8). As shown, based on the appearance of the element peaks of C, O, Fe, S and N in BBC@nFe-SH, the existence of the N—C=S and Fe—O functional groups can be confirmed, which confirms the above mentioned FTIR analysis [41]. After adsorption, peaks of As 3p, As 3d, As 2p_{3/2}, and As LMM were detected on the surface of BBC@nFe-SH, demonstrating the effective adsorption of As(III) by BBC@nFe-SH. The iron peaks of Fe 2p₁, Fe 2p_{3/2}, Fe LMM, Fe LMM₁ and Fe LMM₂ occurred before and after adsorption, showing favorable magnetic properties and recycling potential after the elution of BBC@nFe-SH. In the spectrum, the molar fractions

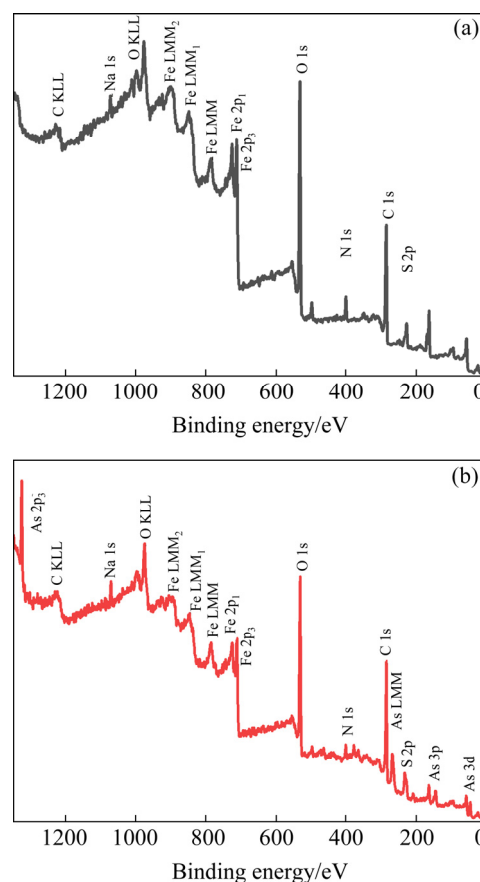


Fig. 8 Full scan spectra of BBC@nFe-SH before (a) and after (b) As(III) adsorption

of O, Fe and S all decreased while the molar fraction of C increased after adsorption. The molar ratio of O to C decreased from 0.797 to 0.637, demonstrating that oxygen-containing functional groups were reduced after adsorption due to participation in the reaction. In addition, Fe and S can promote the adsorption of As(III) to some degree, thus enhancing the adsorption ability of BBC@nFe-SH.

As shown in Fig. 9(a), the main forms of Fe on the surface of BBC@nFe-SH were Fe(III) and Fe(II). A previous study suggested that the peaks appearing at 711.39 and 724.69 eV in Fe 2p corresponded to Fe_3O_4 [42]. The shift of the Fe 2p_{3/2} photoelectron peak after adsorption suggested that Fe was involved in the adsorption process of As(III). The C 1s peaks positioned at 284.70, 286.20 and 288.62 eV were assigned to C—C , C—O and C=O , respectively [43]. From the photoelectron spectroscopy of C, the content and form of C in BBC@nFe-SH before and after adsorption did not appear to change apparently. The broad O 1s peak in Fig. 9(b) shows the existence of

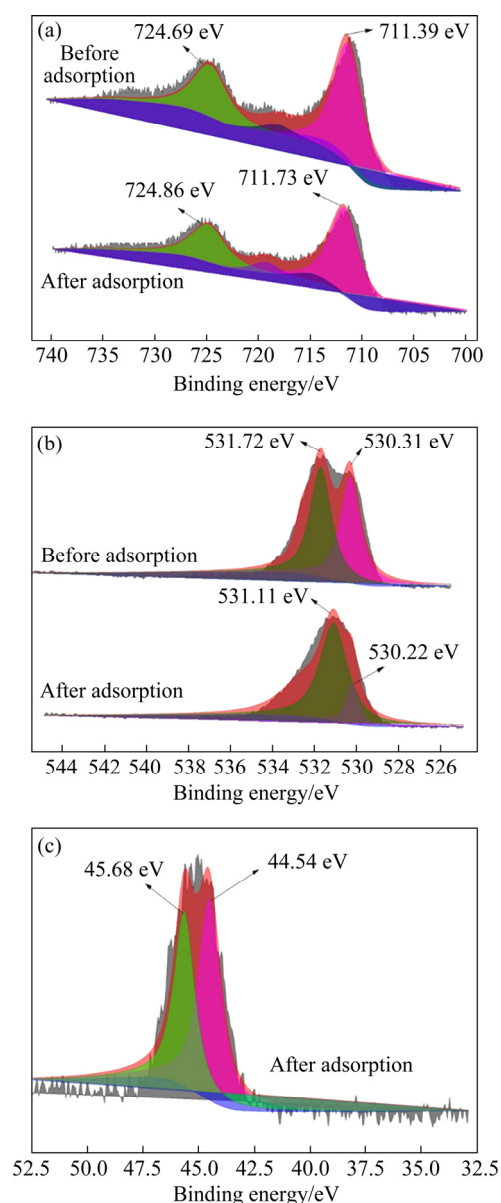


Fig. 9 High resolution XPS spectra of Fe 2p (a), O 1s (b) and As 3d (c)

oxygen in different states, such as organic oxygen (carboxyl, carbonyl and hydroxyl groups) and inorganic oxygen (iron oxides). The O 1s binding energy of 530.31–532.60 eV was ascribed to the binding between inorganic oxygen and iron. The peaks with binding energies at 531.72 and 530.31 eV were assigned to Fe—O—C and Fe—OH, respectively [44]. After the adsorption of As(III) on BBC@nFe-SH, a blue shift of the O element peak was observed, and the binding energy was weakened to some degree, most likely as a result of As(III) adsorption, demonstrating that considerable oxygen-containing functional groups were involved in the adsorption process.

After the adsorption of As(III) on BBC@nFe-SH, two peaks at 44.54 and 45.68 eV in As 3d were found (Fig. 9(c)). Research indicated that As 3d binding energies of 45.50, 44.20 and 41.50 eV were assigned to NaHAsO₄ of As(V), NaAsO₂ of As(III) and As(0), respectively [45]. In addition, different speciations and valences can result in variations in the As 3d binding energy. For instance, binding energies of 44.90, 45.50 and 46.70 eV were assigned to AsO₄³⁻, HAsO₄²⁻ and H₂AsO₄, respectively [46,47]. Therefore, the binding energies at 44.54 and 45.68 eV represented As(III) and As(V) in the present work and accounted for 56% and 44%, respectively, which was consistent with other research. In addition, some studies have shown that iron oxides have strong oxidation of As(III) at low pH values and can oxidize As(III) to As(V). The N—C=S functional group showed a relatively strong trapping ability for As(III) but a weak adsorption ability for As(V). In addition, the zeta potential results showed that the surface zeta potential was the lowest after alkali activation (Fig. 10). It is likely that the organic groups on the surface have been deprotonated. According to the adsorption experimental results, the surface deprotonation process is conducive to the coordination of composites with heavy metals.

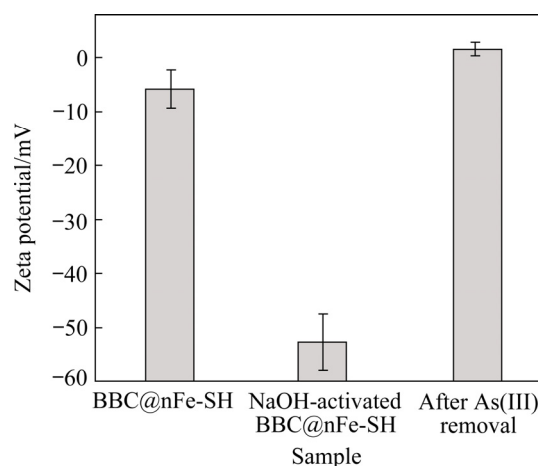


Fig. 10 Zeta potentials of BBC@nFe-SH before and after As(III) adsorption

4 Conclusions

(1) Through magnetization and functional group modification of bamboo biochar, novel BBC@nFe-SH was successfully prepared as adsorbents for efficient removal of As(III) from aqueous solutions. The results of SEM, TG-DTA

and FTIR analysis reveal that BBC@nFe-SH exhibits a better magnetic property and possesses more oxygen-containing functional groups.

(2) The maximum adsorption capacity of BBC@nFe-SH reaches 98.63 mg/g at pH 5.0 and 40 °C. The removal reaction reaches equilibrium within 120 min. The coordination organic groups of C=S and C—S dramatically prohibit the removal rate of As(III). In addition, Fe, S and ozone molecules directly interact with As(III), which strengthens the performance of BBC@nFe-SH.

(3) In the whole reaction process, BBC@nFe-SH displays higher stability and recyclability in the adsorption process. Thus, BBC@nFe-SH with high adsorption capacity and strong magnetism will be a promising adsorbent for the removal and recovery of As(III) from aqueous solutions.

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多巯基磁性生物竹炭纳米复合材料的化学共沉积制备及其高效去除水溶液中的 As(III)

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摘要: 通过简单的化学共沉淀方法制备多基团磁性生物竹炭纳米复合材料(BBC@nFe-SH)并用于废水中 As(III) 的去除。研究表明, BBC@nFe-SH 的磁场强度达到 83376 A/m, 具有极好的磁回收性。在 pH 5.0、40 °C 的条件下 BBC@nFe-SH 对 As(III) 的最大吸附容量为 98.63 mg/g, 且在 120 min 内达到反应平衡。SEM、FTIR、VSM 和 XPS 等分析表明, As(III) 的去除机制为 BBC@nFe-SH 表面的含氧基团与 As(III) 的络合作用。在水处理过程中 BBC@nFe-SH 表现出较好的稳定性和可回收性, 可以通过 1 mol/L NaOH 淋洗实现材料的再生活化。因此, BBC@nFe-SH 在含砷废水处理方面具有极大的应用潜力。

关键词: 含砷废水; 生物竹炭; 化学共沉淀; 吸附; 循环利用; 废水处理

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