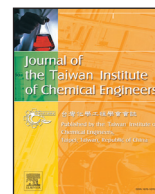




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Synthesis of magnetic biochar from iron sludge for the enhancement of Cr (VI) removal from solution

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ABSTRACT

Iron sludge was directly reutilized as ferric resource to synthesize magnetic Fe-coated cotton stalk biochar (Fe-CS) for Cr (VI) removal from aqueous solution. Physical analyses revealed that Fe-CS exhibited higher surface area of 129.2 m²/g and pore volume of 0.1711 cm³/g than those of the original cotton stalk biochar (OCS). Fe-CS showed an excellent enhanced adsorption for Cr (VI) (67.44 mg/g) removal compared with OCS (40.91 mg/g). The adsorption process followed the Langmuir isotherm model and the adsorption kinetic followed pseudo-second order model. Fe-CS can be easily separated from the solution using magnet and regenerated for reuse. The overall findings indicate that iron sludge derived from water treatment plant can be reutilized directly for iron resource, which can bring new direction in the treatment of iron sludge waste.

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1. Introduction

Chromium is observed in different kinds of industrial wastewaters, including metal processing, leather tanning and electroplating [1–3]. Chromium exists primarily as Cr (III) and Cr (VI) in natural waters. Cr (III) species are much less soluble and relatively stable than Cr (VI). In comparison, Cr (VI) species, such as HCrO₄[−], CrO₄^{2−} and Cr₂O₇^{2−} are highly soluble and mobile in solution [4,5]. Cr (VI) compounds are known to be highly toxic, carcinogenic and mutagenic, which exhibit a higher toxicity than Cr (III) [6–8]. In China, Cr (VI) pollution is a common contaminant in some economically backwardness countryside areas. For example, in Huan county, Gansu province, the concentration of Cr (VI) is higher than the permissible limit 0.05 mg/L [9,10].

The methods for Cr (VI) removal are mainly including chemical precipitation, ion exchange, membrane separation and adsorption [11–13]. Among these, adsorption is the most frequently applied for Cr (VI) remediation owing to its advantages such as fast, inexpensive and variety of adsorbent materials [14–17]. Recently, agricultural by-products derived adsorbents have been used for the removal of Cr (VI) [18,19]. Cotton stalk is an abundant agricultural byproduct in China countryside. The amount of cotton stalk waste annually generated is more than 40 million tons [20]. It has attracted more attention that cotton stalk can be carbonized at high temperature in the absence of oxygen to produce biochar [21–23].

It can be used as low cost adsorbent for the removal of dyes and harmful ions from solution [24]. However, the small particle size of biochar made it difficult to separate from the solution system after adsorption; several research groups have added iron oxides to synthesize hybrid magnetic chars to overcome this difficulty [25–27].

Iron salts had been widely used for the treatment of drinking water [28,29], while large amount of iron sludge would produce. It is usually disposed as solid waste. On the other hand, the iron sludge is a kind of iron-containing resource. The reuse of iron sludge has received large concern from scientific researchers. Yoo tried to recycle of iron sludge as a coagulant for the treatment of refractory organics in leachate [30]. It was found to be practically feasible and the coagulant dose and sludge production could be reduced up to 50%. However, the iron sludge could be reused only once in this system. Bolobajev tried to reuse the ferric sludge as an effective catalyst for the Fenton-based process [31]. As a result, the iron-containing sludge was reused without any regeneration and the process was as a feasible solution to minimize the production of hazardous ferric waste. In this process, iron sludge must interact with H₂O₂ to form the ferric-hydroperoxo intermediate. However, it has a disadvantage of increasing extra cost of H₂O₂ and H₂SO₄. To the best of the author's knowledge, there are few study on the directly reapplication of iron sludge without any chemical reagent.

Therefore, the objective of this study is to explore iron sludge as an iron source instead of ferric chemical reagent to fabricate magnetic biochar for Cr (VI) removal from aqueous solution. The adsorption parameters such as the initial pH, the contact time and the maximum adsorption capacity were investigated. The effects of iron element on Cr (VI) removal and the adsorption mechanisms

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were fully discussed. More importantly, magnetic property of the adsorbent allows the rapid separation from aqueous solution by magnet and good recyclability.

2. Experiment

2.1. Materials and equipment

The iron sludge waste used for this study was supplied by water treatment plant (Fe containment: 210–270 g/kg). The cotton stalk was obtained from Xinjiang province, China. Before usage, the original cotton stalk (CS) was crushed into power and sieved through 100 mesh size sieves. All chemicals used in this study were used as received without further purification. The microcomputer microwave chemical reactor (WBFY-205) used in this study was purchased from Shanghai Yue-Zhong instrument equipment Co., Ltd. The microcomputer microwave chemical reactor is equipped with a magnetic stirring system and the reactor is an open reaction system.

2.2. Microwave-assisted synthesis of magnetic biochar

The magnetic Fe-coated cotton stalk biochar (designed as Fe-CS) was synthesized as follows: CS (10 g), iron sludge waste (20 g) and deionized water (20 mL) were dispersed in a one-necked flask. Then the mixture was heated in the microwave chemical reactor at 300 W for 10 min. After cooling, the composite was washed with deionized water and dried at 80 °C for 12 h. Subsequently, the dried power was carbonized in a nitrogen atmosphere at 500 °C. As a comparison, original CS without iron sludge waste (designed as OCS) and CS dipping with chemical reagent ferric sulfate (designed as CFe-CS) were prepared under the same procedure.

2.3. Characterization of the materials

Fourier transform infrared spectroscopy (FTIR) spectra were obtained using Bruker TENSOR 27 FTIR and the spectra were recorded in 400–4000 cm⁻¹ range. The morphologies and chemical constituents of the two materials were obtained by scanning electron microscopy (SEM, JEOL JSM-5600LV). The nanocrystal size of Fe-CS was observed by transmission electron microscopy (TEM, FEI, USA). Their structure and phase composition were studied by X-ray diffractometer (XRD, Shimadzu XRD-6000). The surface areas of OCS and Fe-CS were measured by N₂ adsorption-desorption technique (JW-BK122W). The surface electronic states were analyzed by X-ray photoelectron spectroscopy (XPS, ESCALAB 250Xi)

2.4. Cr (VI) adsorption experiments

The adsorption of Cr (VI) was studied by batch experiments in an isothermal shaker (160 r/min). The adsorption experiments were carried out at 25 °C and the adsorbent for Cr (VI) adsorption was 0.4 g/L. The effects of initial solution pH on the adsorption capacity of OCS and Fe-CS were investigated within pH 1.0–11.0. The initial pH of the solutions was adjusted by H₂SO₄ and NaOH solution by using pH meter. For the regeneration studies, Fe-CS was separated by using magnet after adsorption Cr (VI). The Cr (VI) adsorbed Fe-CS was dipped in 0.1 M NaOH solution and then washed with distilled water.

The amount of Cr (VI) adsorbed onto the adsorbent was calculated by the following equation:

$$q = \frac{(C_0 - C)V}{m}, \quad (1)$$

where q (mg/g) is the amount of adsorbed Cr (VI), C_0 (mg/L) and C (mg/L) are the initial and final Cr (VI) concentration, respectively. V (L) is the volume of the Cr (VI) solution, and m is the weight of the adsorbent.

2.5. Analytical methods

The concentrations of Cr (VI) were determined by the diphenyl-carbazide colorimetric method on a UV-Vis spectrophotometer (UV-1200, China). The total Cr was measured by the atomic absorption spectrophotometer (solan 969, USA). The pH of the solution was measured by an acidimeter (PHS-2 Model, China). Three replicates were measured on each sample.

2.6. Non-linear regression analysis

In order to measure the accuracy of the applied models, the determination coefficient (R^2) and chi-square test (χ^2) were assessed. The determination coefficient (R^2) can be expressed as

$$R^2 = \frac{1 - \sum_{i=1}^i (Q_{e,i} - Q_{m,i})^2}{\sum_{i=1}^i (Q_{e,i} - \bar{Q}_{m,i})^2}, \quad (2)$$

where, Q_e (mg/g) is the adsorptive capacity at equilibrium; Q_m (mg/g) is the maximum adsorption capacity; i is the number of observation. The chi-square test (χ^2) can be expressed as

$$\chi^2 = \sum_{i=1}^i \frac{(q_{e,i} - q_{m,i})^2}{q_{e,i}}, \quad (3)$$

where, q_e (mg/g) is the amount of Cr (VI) adsorbed; q_m (mg/g) denotes the maximum adsorption capacity of Cr (VI); i is the number of observation. If the data from model are similar to the experimental data, χ^2 will be smaller.

3. Results and discussion

3.1. Characterization of OCS and Fe-CS

To determine the functionalities of OCS and Fe-CS, FTIR analysis was performed. As shown in Fig. 1, the band at about 3438 cm⁻¹ was assigned to hydroxyl group which contains hydrogen bonding. The peaks found at 1650 cm⁻¹ and 1566 cm⁻¹ originated from the C=O asymmetric stretching vibrations of carboxylates and the skeletal C=C vibrations in aromatic ring vibration, respectively. The peak at about 1430 cm⁻¹ was related with the symmetric stretching of carboxylates. The spectra of Fe-CS showed a strong band at 1160 cm⁻¹, which was assigned to the characteristic peak of S=O groups. Compared with Fig. 3(a), the peaks in Fe-CS at around 677 cm⁻¹ and 588 cm⁻¹ were related to the characteristic vibration of Fe-O group [32].

XRD patterns of OCS and Fe-CS are given in Fig. 2. The diffraction spectrum of OCS did not show any obvious crystalline peak indicating the amorphous phase of the cotton stalk biochar. However, with the introduction of iron sludge, several characteristic peaks for Fe₃O₄ were observed at $2\theta = 30.29^\circ$, 35.39° , 43.12° , 53.43° , 56.40° and 62.53° . These peaks were indexed to the (220), (311), (400), (422), (511) and (440) crystalline planes, respectively [33]. These results indicated that a magnetic iron-coated cotton stalk biochar was successfully synthesized. According to the powder diffraction file (PDF) from the jade 6 software, CaSO₄ and CaCO₃ were identified on Fe-CS, which may from the iron sludge.

Fig. 3(a) and (b) showed the morphologies of OCS and Fe-CS. The SEM image of OCS revealed a relatively smooth and plain surface (Fig. 1(a)). However, the surface of Fe-CS was much more coarse and porous than OCS, which is expected to exhibit enhanced adsorption capacity of Cr (VI) (Fig. 1(b)). Fig. 4 clearly showed the presence of Fe₃O₄ nanoparticles dispersed on the surface of OCS and the diameters of Fe₃O₄ nanoparticles was

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