



Enhanced hexavalent chromium removal by activated carbon modified with micro-sized goethite using a facile impregnation method

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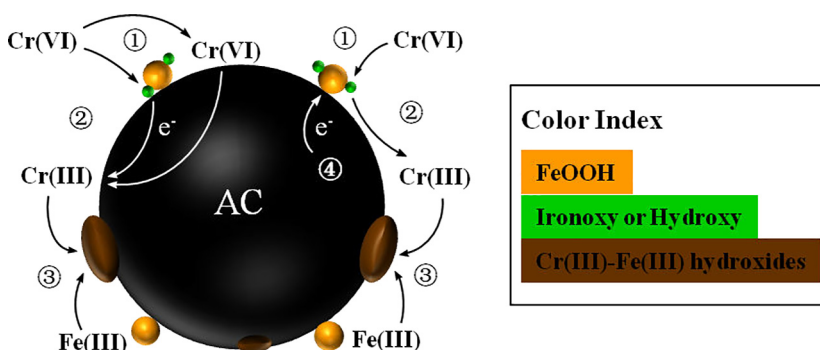
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HIGHLIGHTS

- Micro-sized FeOOH was homogeneously distributed on activated carbon.
- Cr(VI) was removed via adsorption, reduction and co-precipitation mechanism.
- The mFeOOH@AC composite has good stability and reusability.

GRAPHICAL ABSTRACT



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ABSTRACT

In this study, activated carbon (AC) was modified with micro-sized goethite (mFeOOH) using a facile and cost-effective impregnation method for enhanced Cr(VI) removal from aqueous solutions. X-ray diffraction (XRD) and scanning electron microscope (SEM) analysis showed that FeOOH particles with a diameter of 0.1–1 μm were dispersed homogeneously on the surfaces and pores of the AC. Fourier transform infrared spectrum (FTIR) and X-ray photoelectron spectra (XPS) analysis indicated that Cr(VI) was easily adsorbed onto the mFeOOH and reduced to Cr(III) by the AC, eventually deposited as Cr(III)-Fe(III) hydroxides (e.g., $(\text{Cr}_x\text{Fe}_{1-x})(\text{OH})_3$). Hence, the mFeOOH@AC achieved a significantly higher Cr(VI) removal efficiency of 90.4%, 4.5 times of that of the AC. The adsorption of Cr(VI) onto the mFeOOH@AC agreed well with the Langmuir adsorption model, demonstrating that the adsorption process was controlled by monolayer adsorption. This adsorption process also followed the pseudo second-order kinetics and the adsorption rate constant K_2 was determined to be 0.013 $\text{g}/(\text{mg} \cdot \text{min})$. The Cr(VI) removal efficiency decreased with pH values as the adsorption process was highly pH-dependent. After the desorption-adsorption process by 0.1 M HCl solution for 4 cycles, the removal efficiency of Cr(VI) was still kept up to 75.1%, indicating that the mFeOOH@AC has a good stability and can be easily regenerated. In addition, the mFeOOH@AC also exhibited a promising potential for reutilization since a Cr(VI) removal

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efficiency of 85.4% was achieved after stripping all the mFeOOH and Cr(III)–Fe(III) hydroxides by 1 M HCl solution and regeneration with mFeOOH. We demonstrate that the modified AC with micro-sized goethite can remarkably enhance its ability for Cr(VI) removal in water treatment.

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1. Introduction

Chromium is widely used in various industries such as steel manufacturing, leather tanning, metal finishing, wood preserving, and textile productions (Jayakumar et al., 2014; Wu et al., 2012; Yoon et al., 2011). During the process of production, due to the leakage of industrial effluents and improper practices of industrial sludge disposal, a large number of chromium components have been released into the surface and groundwater. In aqueous solutions, chromium usually exists in the oxidative states of hexavalent chromium (Cr(VI)) and trivalent chromium (Cr(III)) (Shi et al., 2011b). Compared to relatively innocuous Cr(III), Cr(VI) is more soluble, mobile, toxic, and carcinogenic (Chai et al., 2017; Yirsaw et al., 2016; Zhuang et al., 2014). It is hence easy to spread throughout the water body, contaminating the ground water and endangering the human health (Min et al., 2017). When the concentration of Cr(VI) reaches 0.1 mg/g body weight, Cr(VI) becomes lethal to human (Richard and Bourg, 1991). Hence, Cr(VI) has been considered as a priority pollutant by the United States Environmental Protection Agency (He et al., 2013) and it shall be removed or decontaminated from the environment.

A variety of technologies have been developed to remove Cr(VI) from aqueous solutions, including chemical reduction (Vlyssides and Israilides, 1997), membrane filtration (Fabiani et al., 1997), solvent extraction (Macchi et al., 1991), biological processes (Kapoor et al., 1999) and ion exchange (Tiravanti et al., 1997). However, most of these methods have technical or economic limits, such as high operational cost, incomplete removal, and toxic metal sludge generation. Compared to these methods, adsorption is a promising and efficient technology to remove Cr(VI) from aqueous solutions (Barakat, 2011; Sahu et al., 2009). By now, activated carbon (AC) is one of the most cost-effective adsorbents for various pollutants because of its abundant micropores, huge specific surface areas and functional groups such as hydroxyl and carboxyl (Park and Jang, 2002). Moreover, AC may reduce Cr(VI) to Cr(III) since Cr(VI) is a strong oxidant (Espinoza-Quiñones et al., 2010; Módenes et al., 2010), and the reduction of Cr(VI) benefits the immobilization of Cr via Cr(III) precipitation (Gheju and Balcu, 2011). However, the functional groups of AC are negatively charged and relatively nonpolar (Adhoum and Monser, 2002; Yin et al., 2007), resulting in a weak adsorption capacity for Cr(VI), generally below 10 mg/g (Monser and Greenway, 1996). This limits the application of AC for the removal of Cr(VI).

Recently, several studies have been proposed to increase Cr(VI) removal efficiency by modifying AC with acids, oxidants and metal oxides (Adhoum and Monser, 2004; Zhao et al., 2005). In particular, AC modified with iron or iron oxides showed improved removal capacities for Cr(VI). Iron oxides are widely distributed in the environment and have been applied to remove heavy metal ions from aqueous solutions due to their low toxicity, fast kinetics and strong affinity toward heavy metals (Zhou et al., 2015). However, iron and iron oxides are prone to aggregation and subsequent rapid sedimentation in aqueous solutions because of their high surface energy (Xu et al., 2012), leading to the decrease of their reactivity in the adsorption of heavy metal ions. Besides, powdery iron oxides are difficult to be separated from aqueous solutions after their usage. It is hence needed to enhance the dispersity of iron oxide particles by supporting them onto physical materials such as bentonite, multiwalled carbon nanotube, zeolite, smectite and pillared clay (Lv et al., 2013; Ruan et al., 2015; Shi et al., 2011b). For instance, Ruan et al. (2015) prepared a bentonite-biochar-Fe₂O₃ composite by facile pyrolysis that significantly enhanced the adsorption capacity of Fe₂O₃

for Cr(VI). Wu et al. (2012) used montmorillonite to support Fe⁰, which greatly reduced the aggregation of Fe⁰ and improved its adsorption and reduction of Cr(VI).

In the present study, we chose AC as a supporting material for another promising iron oxide, goethite (FeOOH). It is one of the most common iron oxides which widely spread in water, soil, sediment and rock in the environment (Guo et al., 2016). Compared to iron or other iron oxides (e.g., Fe⁰, Fe₃O₄, and Fe₂O₃), FeOOH is more stable under aerobic conditions and can be easily synthesized by chemical processes. Besides, FeOOH generally exhibits strong affinity to oxyanions such as Cr(VI) (Qin et al., 2014) via ion exchange and complexation owing to the existing of hydroxyl and ironoxy groups on its surface. However, it has been found that FeOOH with a size less than 5 μm will be adverse to its practical application such as separation, recovery and reuse owing to its easily aggregation (Zuo et al., 2016). It is hence necessary to support FeOOH onto physical materials in order to prevent it from aggregating. Among various physical materials, carbonaceous materials such as AC, biochar, graphene and graphene oxide sheets have been widely selected as supporters for metal oxides (Cong et al., 2012; Guo et al., 2016; Koduru et al., 2016; Xu et al., 2013). Here we chose AC as an effective supporting matrix for FeOOH because ferric ion can be easily adsorbed onto AC by electrostatic adsorption. We propose this modified FeOOH@AC approach to investigate the dispersity of FeOOH and the enhancement of Cr(VI) removal. In this study, we will focus primarily on the performance of the FeOOH@AC for the Cr(VI) removal capacity. Solid phase characterization was performed to illustrate the mechanism of the Cr(VI) removal by the FeOOH@AC. Fourier transform infrared spectrum (FTIR) and X-ray photoelectron spectroscopy (XPS) were employed to measure the reduction of Cr(VI) and the oxidation of AC during the adsorption process. Desorption-adsorption experiments were also carried out to evaluate the stability and reutilization of the FeOOH@AC.

2. Materials and methods

2.1. Materials

The activated carbon (AC) was received (Chaoyang Senyuan Activated Carbon Limited Company) with an average diameter of 0.1 mm and a surface area of 770 m²/g, respectively. Chromium stock solution (1000 mg/L) was prepared by dissolving 2.829 g of potassium dichromate (K₂Cr₂O₇) into 1000 mL of deionized water. The required concentrations of the chromium standard solution were made by diluting the stock solution. Nine hydrated ferric nitrate (Fe(NO₃)₃·9H₂O) and other chemicals were analytical grade (Guangzhou Chemical Reagent Factory).

2.2. Adsorbents preparation

Before use, the AC was pretreated with 0.1 M HCl for 24 h and ultrasonic treatment for 15 min and rinsed with deionized water until it became neutral. It was then dried at 103 °C for 2 h and subsequently packed into zip lock bags and stored in a desiccator. The unsupported mFeOOH was used as a control and was synthesized using a standard method (Schwertmann and Cornell, 2000). In brief, 100 mL of 1 M ferric nitrate solution was prepared in a polyethylene bottle and 180 mL of 5 M KOH was quickly poured into the polyethylene bottle. The solution was mixed at a stirring rate of 130 rpm. Immediately, the suspension was diluted to 2 L with deionized water and then kept in a sealed

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