



Synthesis and lead absorption properties of sintered activated carbon supported zero-valent iron nanoparticle



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ABSTRACT

In this study, the zero-valent iron nanoparticles (Fe⁰NPs) were immobilized on the sintered activated carbon (SAC). The prepared Fe⁰NPs-SAC was characterized with using scanning electron microscope (SEM), X-ray diffraction (XRD), Brunauer-Emmett-Teller (BET) analyzer and atomic absorption spectrophotometry (AAS). The SEM morphology illustrated that Fe⁰NPs were well embedded in the SAC. The XRD peaks revealed that the addition of Fe⁰NPs did not change the crystal degree. The BET data showed that its surface area was 436.96 m²/g, average pore diameter was 2.377 nm and total pore volume was 0.060 cm³/g, while the corresponding values of SAC were 513.29 m²/g, 2.225 nm and 0.065 cm³/g. The AAS results indicated the iron loading amount was related with Fe⁰NPs/SAC ratio and reaction time in the preparing process. Subsequently, Fe⁰NPs-SAC was used as adsorbent to treat simulated lead bearing wastewater. The effects of pH, adsorbent dosage and iron loading amount were investigated. Moreover, water erosion resistance of the Fe⁰NPs-SAC was determined through released iron content in the static and shaking water. The results showed the prepared Fe⁰NPs-SAC was not only effective but also safe adsorbent for purification of drinking water.

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1. Introduction

With the rapid development of industry, waste streams containing heavy metal ions are more and more often encountered. It brings a greatly growing threat to the safety of drinking water [1,2]. So far, adsorption is one of the most common approaches to remove those heavy metal ions from drinking water, due to its technological and cost advantages [3].

Zero-valent iron nanoparticles (Fe⁰NPs) have been reported as an effective adsorbent [4]. It is an ideal candidate for the remediation of contaminated groundwater in situ [5]. Due to its strong reducing power and high removal capacity, Fe⁰NPs have been used in permeable reactive barriers for intercepting heavy metal ions in contaminated groundwater [6] or added to household filters for heavy metal ions removal in developing nations [7]. However, because of the aggregation arisen from its high specific surface areas and strong reactivity, its wide application may be limited [8].

Therefore, there is an increasingly stringent necessity to reduce the aggregation of Fe⁰NPs in the process of water treatment.

Activated carbon (AC) is a common supported material due to its exceptionally high surface area (ranges from 500 to 1500 m²/g), well-developed internal micro-porosity and remarkable chemical stability and durability [9–11]. However, AC usually appeared as powders [12]. Their direct application in water treatment system may cause fast loss and further pollution of drinking water due to its tiny particle size [13]. Therefore, our study used sintered technology to merge powdery AC into a large-scale SAC with almost no rearrangements in its internal structure. Such SAC used as a supported material can make the iron particles evenly distribute. Furthermore, SAC itself is an efficient adsorbent for a wide array of chemical pollutant (but not effective for heavy metal ions) [14]. The combination of those two materials would improve the purify efficiency and extend the application range [15]. Therefore, in our study, a novel material of Fe⁰NPs-SAC was prepared and applied for the simulated lead bearing wastewater. It would be an outstanding adsorption material against the pollutants in the drinking water.

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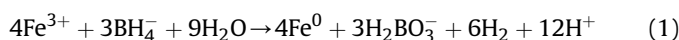
2. Materials and methods

2.1. Chemical reagents

Sintered activated carbon (SAC) was prepared in our laboratory by the same route introduced in our previous article [16]. Simulated lead bearing wastewater was prepared using dissolving required amount of $\text{Pb}(\text{NO}_3)_2$ (NCB Testing Technology, CO., LTD) in the distilled water. The pH of the solution was adjusted with using HCl or NaOH solution. Other reagents, such as ferric chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$), sodium borohydride (NaBH_4) and PEG-20000 were all of analytical grade. All solutions were prepared with the distilled water. In addition, 0.45 μm filter membranes were purchased from German Membrane CO., LTD.

2.2. Preparation of $\text{Fe}^0\text{NPs-SAC}$

The preparation of Fe^0NPs was based on the following reaction equation:



NaBH_4 (0.25 M) was introduced into a blend of FeCl_3 (0.045 M) and 3% (m/v) PEG-20000 solution through titration (titration rate 20–30 d/min) with volume ratio of 1:1 [17].

The formed Fe^0NPs were obtained using vacuum filtration through 0.2 μm filter papers. Then, they were washed several times respectively using distilled water and ethanol, and dried in a vacuum oven at 70 °C for 2 h. The prepared Fe^0NPs were stored in anhydrous ethanol solution in order to avoid oxidation.

The ultrasonic-assisted impregnation method [18] was used to add the Fe^0NPs into SAC. The SAC material was cut in small cubes with the side of about 1 cm, washed thoroughly with distilled water and dried at 60 °C for 4 h. Then, the pretreated SAC were added in the Fe^0NPs solution with ultrasonic treatment. Finally, the $\text{Fe}^0\text{NPs-SAC}$ were taken out of the solution, washed thoroughly with distilled water and dried at 60 °C for 4 h.

2.3. Characterization

The surface morphology of the $\text{Fe}^0\text{NPs-SAC}$ material was observed with using a SEM (Nova Nano SEM 430) operated at 20 KV and 80Ma. Additionally, an energy-dispersive X-ray spectrometry (EDS) analysis was carried out to determine the elements composition (C, O and Fe) of the prepared $\text{Fe}^0\text{NPs-SAC}$ material [19].

X-ray diffraction (XRD) patterns of the elements in $\text{Fe}^0\text{NPs-SAC}$ material were recorded using a Bruker D8 Advance X-ray diffraction instrument equipped with Cu K α X-ray radiation [20].

The BET specific surface area properties of $\text{Fe}^0\text{NPs-SAC}$ were determined using a JW-BK222 automatic physical adsorber. The nitrogen adsorption was measured at 20 °C and 101 kPa. The pore size distribution was calculated by the density functional theory (DFT) method. Total pore volume (V_t) was estimated with liquid volume of nitrogen at a relative pressure of about 0.995 [21].

The iron loading was measured using an AAS (Z-2000 from Japan). The measure conditions were set up as follows: air flow rate was 15.0 L/min; acetylene flow rate was 1.8 L/min; electric current of lamp was 10.0 mA; slit width was 1.3 nm; wave length was 248.3 nm. The samples prepared with different $\text{Fe}^0\text{NPs/SAC}$ ratio (1.2 mmol/g, 1.8 mmol/g, 2.4 mmol/g, 3.0 mmol/g and 3.6 mmol/g) or different reaction time (30 min, 60 min and 90 min) were measured in order to determine their relationship with the iron loading.

2.4. Adsorption properties

Adsorption properties of $\text{Fe}^0\text{NPs-SAC}$ on heavy metal ions was determined by Pb removal experiments using $\text{Pb}(\text{NO}_3)_2$ solution with Pb(II) concentration of 50 mg/L as simulated lead bearing wastewater. The $\text{Fe}^0\text{NPs-SAC}$ and $\text{Pb}(\text{NO}_3)_2$ solution were mixed in the 100 mL conical flask with 180 rpm mechanical agitation at 25 °C. After given intervals (5, 10, 20, 30, 45, 60, 90, 120, 180 min), 500 μL of aqueous sample was collected and filtered with 0.45 μm filter membrane. The concentration of Pb(II) was measured using the AAS. The measure conditions were set up as follows: air flow rate was 15.0 L/min; acetylene flow rate was 2 L/min; electric current of lamp was 7.5 mA; slit width was 1.3 nm; wave length was 283.3 nm [22]. The Pb (II) uptake was calculated by the following equation:

$$q_t = (c_0 - c_1)V/m \quad (2)$$

where c_0 (mg L^{-1}) and c_1 (mg L^{-1}) were the initial and final concentrations of Pb(II) in the aqueous solution, V (L) was the total volume of the aqueous solution, and m (g) was the mass of the adsorbent.

The adsorption percentage R was calculated by the difference between the initial and final concentration of adsorption solution given as:

$$R = (c_0 - c_1)/c_0 \times 100\% \quad (3)$$

During the process of Pb(II) removal, the effect of initial adsorbent dosage was determined with the adsorbent dosage ranging from 100 to 200 mg/L. Meanwhile, the effect of iron loading amount was determined using samples with 6.02%, 9.07% and 14.23% iron content, respectively.

2.5. Resistance to water erosion

The resistance of prepared $\text{Fe}^0\text{NPs-SAC}$ to water erosion was determined through an attrition test as the method of Wang et al. [23]. Sample 1–4 with iron content of 6.02%, 7.23%, 9.07% and 14.23% were respectively used in this test. In the static test, about 0.2 g of each sample was respectively placed in 50 mL distilled water for 48 h at room temperature. In the shaking test, similar works were carried out with shaking speed of 180r/min. After the test, the released iron content in the water was measured using AAS.

3. Results and discussion

3.1. SEM analysis

As shown in Fig. 1a, the size distribution of representative single Fe^0NP was around 45–80 nm. It can be due to the –OH groups in PEG-20000 which can slightly reduce aggregation of Fe^0NPs [24]. Additionally, the micro-structure of SAC was presented in Fig. 1b. It illustrated that the interaction between carbon particles and PE adhesive resulted in the SAC's porous structure and excellent stability during the sintered process [25]. Such porosity increased the surface area and provided the possibility to disperse Fe^0NPs . As shown in Fig. 1c and d, the Fe^0NPs are deposited as a thick cluster in an appropriate scale on the surface or internal space of SAC, which had important meanings for its application as an adsorbent. Generally, nanoparticles at high concentration would tend to agglomerate so as to reduce the surface energy, which lead to larger size and poorer disperse [26].

Additionally, through the EDS analysis, C, O and Fe elements

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