



Arsenate adsorption from water using a novel fabricated copper ferrite

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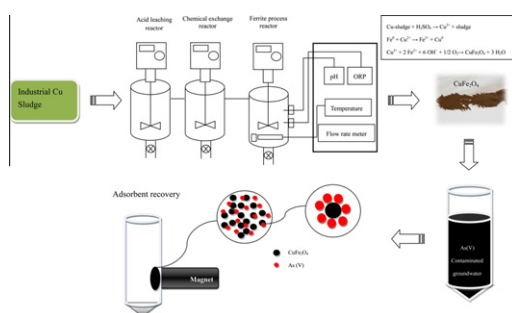
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HIGHLIGHTS

- ▶ A novel technology for fabricating CuFe_2O_4 was developed from industrial Cu sludge.
- ▶ The fabricated CuFe_2O_4 was effective in removing As(V) from contaminated water.
- ▶ The adsorbent can be rapidly recovered by a magnet because of its paramagnetism.
- ▶ As K-edge XANES showed the adsorbed As(V) was not reduced to more toxic As(III).
- ▶ Desorption rate is in an order of $\text{H}_3\text{PO}_4 > \text{Na}_3\text{PO}_4 > \text{H}_2\text{SO}_4 > \text{Na}_2\text{SO}_4 > \text{HCl} > \text{HNO}_3$.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:

Received 15 March 2012

Received in revised form 31 May 2012

Accepted 3 June 2012

Available online 12 June 2012

Keywords:

Adsorption
Arsenate
Industrial sludge
Copper ferrite
As K-edge XANES

ABSTRACT

A novel fabricated copper ferrite was investigated for its As(V) removal in this study. The adsorption of As(V) by this recycled copper ferrite exhibited an L-shaped nonlinear isotherm, suggesting limited binding sites on the adsorbent surface. The As K-edge XANES indicated that the adsorbed As(V) on copper ferrite was not reduced to more toxic As(III) by Fe^{2+} in the structure. The maximum As adsorption capacity of the copper ferrite was 45.66 mg g^{-1} at pH 3.7 and decreased with pH due to enhanced electrostatic repulsion between As(V) and the adsorbent surface. Desorption of As(V) using six different acid and salt solutions revealed that the desorption rate decreased in the order of $\text{H}_3\text{PO}_4 > \text{Na}_3\text{PO}_4 > \text{H}_2\text{SO}_4 > \text{Na}_2\text{SO}_4 > \text{HCl} > \text{HNO}_3$. These results suggest that the recycled copper ferrite without surface modification is an effective adsorbent for removing As(V) from water, confirmed by effective removal of As(V) from contaminated groundwater. Furthermore, the novel fabricated copper ferrite can be used to recover rapidly in 20 s using a magnet. The information obtained in this work shows great potential for developing a cost-effective adsorbent for immobilization of arsenate using the recycled copper ferrite.

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1. Introduction

Arsenic (As) contamination is threatening water supplies in many developing countries [1–3]. The anthropogenic sources of As include the waste and wastewater of various industries utilizing As and the materials used in agricultural production, such as

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herbicide and wood preservative [1,4]. In addition to man-made pollutants, elevated levels of As in surface waters and groundwater also occur naturally in certain areas of the world as a result of leaching from As-bearing minerals [2,3]. Because As is highly carcinogenic after long-term or high-dose exposure, the occurrence of As in water supplies can pose a deleterious impact on public health. Therefore, developing techniques to remove As from contaminated water is an important task for healthy living in a society.

The common treatment techniques for removing As from contaminated waters include precipitation [5–7], oxidation [8–10], coagulation [11–14], adsorption [15–18], ion-exchange [19–21], and membrane filtration [22–26]. The adsorption method is considered more advantageous over others because of its removal effectiveness, treatment cost, and ease in equipment handling. Commercial adsorbents such as activated carbons are effective for arsenic removal from water [27–29]. However, the high cost makes it less competition in market. Mohan and Pittman [1] has summarized some interesting literatures of low-cost adsorbents for arsenic removal. In other words, adsorbents were regarded as low-cost if they only need less pre-treatment procedures, or were either waste materials or by-products from the other industrial factories [30–33].

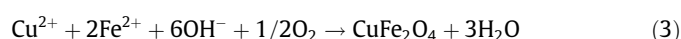
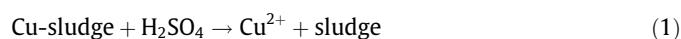
This study demonstrates the arsenate removal by using copper ferrite, which is recycled from the sludge in printed circuit board (PCB) industry. The copper ferrite, a magnetic Cu–Fe oxide, contains Cu^{2+} and Fe^{3+} in the spinel structure. It was reported that synthesized copper ferrite was effective for arsenic removal from solution due to Cu^{2+} and Fe^{3+} have strong affinity toward inorganic arsenic [34]. If the copper ferrite can be obtained or recycled from industrial sludge, then the adsorbent price could be decreased dramatically. Our earlier investigation has successfully recycled copper powder from PCB sludge by combination of acid leaching and chemical exchange [35]. After these two combinations of technologies, ferrite process has been conducted not only to make sure the supernatant but also the sludge can meet the environmental rules. The sludge generated from ferrite process hence is regarded as a general industrial waste due to its stability in environment. If there are no other resourced uses for the sludge, it could be disposed to the sanitary landfills.

The authors attempt to resource this sludge as an adsorbent and test its adsorption feasibility of arsenate in solutions. The adsorbent characterization including particle size, crystalline phases, surface area, point of zero charge (pH_{PZC}), and saturation magnetization were studied systematically. Furthermore, As *K*-edge XANES spectra were used to understand the transformation of different As phases on copper ferrite. The information obtained in this work shows a great potential for developing a cost-effective adsorbent for immobilization of arsenate using recycled copper ferrite.

2. Material and methods

2.1. Recycled copper ferrite

A green low-cost adsorbent, copper ferrite, was manufactured from PCB sludge by combination of acid leaching, chemical exchange, and ferrite process. Literature [35] has reported detailed procedure for preparation of copper powder or copper ferrite. Briefly, acid leaching was conducted with 500 g of the industrial sludge as 10 L diluted sulfuric acid was added for extracting copper from solid to solution. Fe powder was used as sacrificed metal to substitute Cu^{2+} by chemical exchange in liquid. To ensure the supernatant qualify fulfill the effluent standards, ferrite process was performed after chemical exchange. A green low-cost adsorbent, copper ferrite, was manufactured after the ferrite process. The main corresponding reaction of acid leaching, chemical exchange, and ferrite process is described as Eqs. (1)–(3), respectively.



If one wants to obtain a large number of CuFe_2O_4 , Eq. (2) may not be able to perform in this combination system. In other words, the CuFe_2O_4 could be manufactured by using acid leaching and then followed by ferrite process.

The manufactured green low-cost adsorbent was collected using a magnetic separation method by taking advantage of its magnetism. The copper ferrite product was then washed with de-ionized water several times until pH of the solution reached near 7. The solid was then dried at 50 °C for 24 h in an oven and stored for further tests.

2.2. Characterization of copper ferrite

The XRD spectrum is used for determining the crystallographic identity of the produced material and phase purity. The recycled copper ferrite was characterized using XRD (D8 Advance, Bruker, Germany) with a graphite monochromatic copper radiation over the 2θ range of 10–80°. The BET surface area was determined using an ASAP 2010 analyzer (Micromeritics, USA) and N_2 adsorption at 77 K. The surface morphology and particle size were examined by scanning electron microscopy (JSM-6330, Japan). The saturation magnetization of the recycled copper ferrite was measured using a Superconducting Quantum Interference Device (MPMS-XL7, Quantum Design, USA) at 27 °C.

The isoelectric point, also referred as PZC (point of zero charge), is the pH at which the particles in suspension have net charge of zero and no mobility in the electric field. The determination of PZC in this study was carried out according to the procedures described by Smiciklas et al. [36]. Briefly, aliquots of 0.1 M KNO_3 were prepared in a series of flasks and the pHs of the aliquots were adjusted to values ranging from 2 to 12 using 0.1 M KOH or HCl solution. The ferrite samples were then added into each of the flasks to have a solid-to-solution ratio of 1:200 (w/w). The suspensions were allowed to equilibrate for 24 h in a shaker thermostated at 27 ± 1 °C. Then, the suspensions were magnetically separated from the aqueous phase by using a magnet with 4000 Gauss and the pH values (pH_f) of the residual solutions were measured using pH meter. For each sample, the values of the final pH (pH_f) were plotted against the values of the corresponding initial pH (pH_i). The experimental pH_f at the stable values was defined as PZC for the sample.

2.3. As(V) adsorption

Stock solutions of As(V) (1000 mg L^{-1}) were prepared by dissolving sodium arsenate ($\text{NaH}_2\text{AsO}_4 \cdot 7\text{H}_2\text{O}$) (Merck, Darmstadt, Germany) in de-ionized water. Working solutions for experiments were freshly prepared from the stock solution. All experiments were performed in triplicate in 15 mL centrifuge tubes under 30 rpm shaking at 27 ± 1 °C. An initial As(V) concentration of 10 mg L^{-1} and 10 mL As(V) solution was used for every experiment unless specially stated.

As(V) adsorption experiments were conducted using a batch method. To investigate the effect of pH on As(V) adsorption of copper ferrite, the pH of As(V) solutions were controlled at 2.3 ± 0.1 , 3.7 ± 0.1 , 5.4 ± 0.1 , 7.1 ± 0.1 , 8.9 ± 0.1 , 9.8 ± 0.1 , 11.2 ± 0.1 , 12.4 ± 0.1 by adding NaOH or HNO_3 solution under the conditions of As(V) concentration 80 mg L^{-1} and copper ferrite 0.02 g. Different adsorbent dosages (0.0025 g, 0.005 g, 0.01 g, 0.02 g) were used to determine the required dosage that fulfills the regulation concentration of $10 \mu\text{g L}^{-1}$ under the conditions of pH 3.7 and initial As(V) concentration of 100 mg L^{-1} . The As(V) adsorption isotherms of copper ferrite were also recorded in batch experiments to estimate arsenic uptake under the conditions of $\text{CuFe}_2\text{O}_4 = 0.001 \text{ g}$ and pH 3.7, 7.1, and 11.2.

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