



Preparation and characterization of a novel magnetic biochar for arsenic removal



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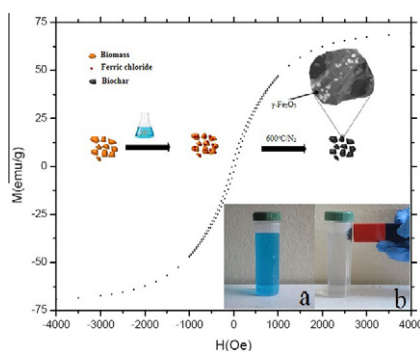
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HIGHLIGHTS

- ▶ A magnetic biochar can be fabricated via pyrolysis of FeCl₃ treated biomass.
- ▶ Colloidal or nanosized γ -Fe₂O₃ particles grow within biochar matrix.
- ▶ Biochar/ γ -Fe₂O₃ composite has excellent ferromagnetic properties.
- ▶ Biochar/ γ -Fe₂O₃ composite has strong sorption ability to As(V).

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:

Received 19 July 2012

Received in revised form 26 November 2012

Accepted 28 November 2012

Available online 8 December 2012

Keywords:

Black carbon

Biochar

γ -Fe₂O₃

Nanocomposite

Adsorption

ABSTRACT

A magnetic biochar based adsorbent with colloidal or nanosized γ -Fe₂O₃ particles embedded in porous biochar matrix was fabricated via thermal pyrolysis of FeCl₃ treated biomass. The synthesized samples were studied systematically by X-ray diffraction, X-ray photoelectron spectroscopy, transmission electron microscopy, selected-area electron diffraction pattern, scanning electron microscopy, energy-dispersive X-ray analysis, superconducting quantum interference device, and batch sorption measurements. The characterization analyses showed that large quantity of γ -Fe₂O₃ particles with size between hundreds of nanometers and several micrometers tightly grow within the porous biochar matrix. Biochar/ γ -Fe₂O₃ composite exhibited excellent ferromagnetic property with a saturation magnetization of 69.2 emu/g. Batch sorption experimental results showed that the composite has strong sorption ability to aqueous arsenic. Because of its excellent ferromagnetic properties, the arsenic-laden biochar/ γ -Fe₂O₃ composite could be easily separated from the solution by a magnet at the end of the sorption experiment.

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1. Introduction

Arsenic is a toxic heavy metal widely distributed in the Earth's crust, which has also been used in pesticides and antibiotics for agricultural and medical uses. Thus, it can get into water systems,

especially groundwater aquifers, from various ways. Drinking of arsenic contaminated water has become a serious threat to the public health and has affected millions of people across the world (Mudhoo et al., 2011). As a result, the US Environmental Protection Agency has set the arsenic standard for drinking water to a level of less than 10 μ g/L. This stringent arsenic standard inevitably requires many water utilities to upgrade their present treatment systems or to consider new purification technologies. During the past decades, much effort has been spent developing high-efficiency and cost-effective adsorbents for arsenic removal, such as metal

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oxides, modified activated carbons, resins, and gels (Aredes et al., 2012; Gu et al., 2005; Mohan and Pittman, 2007).

Biochar is a black carbon created through thermal or hydrothermal conversion of biomass for environmental applications. It is a stable solid, rich in carbon, and can be used as a carbon sink in soils for thousands of years (Wardle et al., 2008). When applied to soils, biochar can introduce multiple benefits, including improving soil fertility, raising crop productivity, increasing soil nutrient and water holding capacity, and reducing emissions of greenhouse gases from soils to mitigate global warming (Lehmann et al., 2008). Several research efforts have been made to evaluate biochar as an adsorbent in water treatment applications for removing various contaminations, including heavy metals, nutrients, and organic compounds (Beesley et al., 2010; Inyang et al., 2012; Yao et al., 2012). It has been reported that biochar converted from hardwoods may have potential ability, but may not be high, to immobilize arsenic from aqueous solutions (Beesley and Marmiroli, 2011). There are a wide range of raw materials can be used to produce biochar-based adsorbents at relatively low cost, which offers promising opportunities for the development of a new arsenic removal technology. Further improvements of the technology through enhancing the chemical and physical properties of biochar-based adsorbents can be envisioned because most of the biochars are directly produced from precursors without any modifications.

Recently, an alternative technology was developed to use engineered/modified biochars to remove various pollutants, including phosphate, heavy metals, and organic compounds, from aqueous solutions (Inyang et al., 2010, 2011; Yao et al., 2011a, b; Zhang et al., 2012). For example, anaerobically digested biomass residues were used as feedstock materials to produce engineered biochars that have high adsorption abilities for heavy metals and phosphate (Inyang et al., 2012, 2011; Yao et al., 2011a, b). H_2O_2 modification of biochar hydrothermally produced from peanut hull can lead to an activation and greatly enhance its ability to remove heavy metals, particularly lead, from water (Xue et al., 2012). Because exhausted biochar adsorbents may contain a great number of pollutants, when biochar-based adsorbents are applied to treat natural water bodies, there is a need to develop a technique to collect the pollutant-laden adsorbents from aqueous solutions to avoid secondary contaminations. Such technique can realistically accomplish the commercialization of biochar-based adsorbents.

$\gamma\text{-Fe}_2\text{O}_3$ particles have been widely used not only as information storage ferrofluids, but also as promising candidates for biomolecule imaging, sensing, and clean energy. Furthermore, $\gamma\text{-Fe}_2\text{O}_3$ is an effective sorption agent for various chemical compounds, such as heavy metals, organic dyes, and antibiotics (Baikousi et al., 2012; Xu et al., 2011; Zhu et al., 2011). Adsorption of arsenic on iron oxide particles was found to be a highly effective option for arsenic removal (Aredes et al., 2012; Velickovic et al., 2012). Additionally, exhausted/spent iron oxide particles may be magnetically collected after use, which favors the recycling of the pollutant-laden adsorbent. However, due to their high surface energy arising from strong van der Waals forces, the $\gamma\text{-Fe}_2\text{O}_3$ nanoparticles have a tendency to form aggregates in aqueous solutions, which dramatically decrease the surface area and adsorption abilities as well as increase the cost. So biochar/ $\gamma\text{-Fe}_2\text{O}_3$ composite may be created to obtain properties that cannot be achieved by any of the components acting alone.

In this work, a novel self-assembled magnetic biochar/ $\gamma\text{-Fe}_2\text{O}_3$ composite with robust interconnected 3D biochar networks was fabricated via thermal conversion (pyrolysis) of FeCl_3 pre-treated biomass (cotton wood). The biochar network can efficiently prevent the $\gamma\text{-Fe}_2\text{O}_3$ particles from aggregating. In addition, the biochar/ $\gamma\text{-Fe}_2\text{O}_3$ composite is not only an ideal candidate as a high-efficiency adsorbent to remove arsenic from water but is also

easily magnetically collected after adsorption. It is anticipated that the versatile method presented here can be extended to induce the assembly of functional metal oxides and biochar from various biomaterials via one step into different composites with multi-functionalities for different applications.

2. Methods

2.1. Materials

Ferric chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) and sodium arsenate dibasic heptahydrate ($\text{Na}_2\text{HAsO}_4 \cdot 7\text{H}_2\text{O}$), and methylene blue ($\text{C}_{16}\text{H}_{18}\text{N}_3\text{S}$) of analytical grade were purchased from Fisher Scientific and used as received. All chemical solutions were prepared using deionized (DI) water (18.2 M Ω) (Nanopure water, Barnstead), which was also used to rinse and clean the samples. Cottonwood, a common agricultural residue material, was collected in Gainesville, FL as the feedstock biomass for biochar-based composite production.

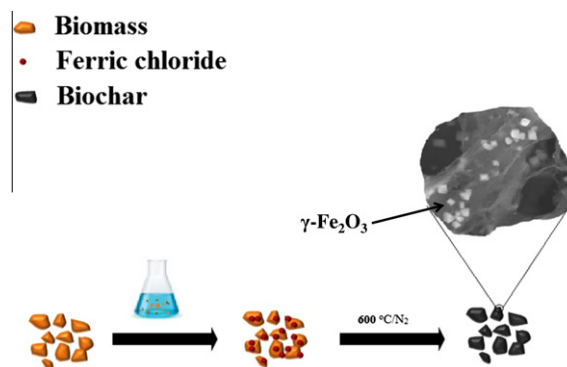
2.2. Preparation of biochar/ $\gamma\text{-Fe}_2\text{O}_3$ composite

Biochar/ $\gamma\text{-Fe}_2\text{O}_3$ composite was produced by using a method as shown in Scheme 1. The solution of ferric chloride was prepared by dissolving 40 g of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in 60 mL of DI water. The biomass was immersed into the prepared FeCl_3 solution for 2 h. The mixture was then dried at 80 °C for 2 h under air. The pre-treated biomass was pyrolyzed in a furnace at a temperature of 600 °C in N_2 environment for one hour. Biochar/ $\gamma\text{-Fe}_2\text{O}_3$ composite produced from the pyrolysis was gently crushed and sieved into two size fractions: <0.5 and 0.5–1 mm. Only the latter was used in the experiments to minimize the presence of residual ash particles. In addition, the samples were then washed with DI water for several times, oven dried (80 °C), and sealed in a container before use.

2.3. Characterizations

The amount of $\gamma\text{-Fe}_2\text{O}_3$ in the composite was determined by modifying the method of Oliveira et al. (2002). Briefly, about 0.1 g of the processed sample was placed in a crucible and was burned at 700 °C in air for 20 min. A magnet was then used to collect the $\gamma\text{-Fe}_2\text{O}_3$ from the ash to determine its content in the composite.

The microscopic features of the sample were characterized by field emission gun scanning electron microscopy (FEG-SEM, JEOL 6335F) equipped with an energy-dispersive X-ray analyzer and transmission electron microscopy (JEOL 200CX TEM) equipped with selected area electron diffraction (SAED) patterns. X-ray photoelectron spectra (XPS) of the sample were obtained with a Perkin



Scheme 1. Illustration of strategy for preparation of biochar/ $\gamma\text{-Fe}_2\text{O}_3$ composite.

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