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journal homepage: www.elsevier.com/locate/envpolEfficient arsenate removal by magnetite-modified water hyacinth biochar[☆]Feng Zhang^{a, b}, Xin Wang^{a, *}, Ji Xionghui^{b, **}, Lijuan Ma^a^a College of Resources and Environmental Science, Hunan Normal University, Changsha, Hunan, 410081, China^b Soil and Fertilizer Institute of Hunan Province/Ministry of Agriculture Key Lab of Agri-Environment in the Midstream of Yangtze River Plain, Changsha, 410125, China

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ABSTRACT

Magnetic biochars (MW) prepared by chemical co-precipitation of Fe²⁺/Fe³⁺ on water hyacinth biomass followed by pyrolysis exhibited important potential in aqueous As(V) elimination. In comparison, MW250₁ outperformed other MWs and exhibited the highest As(V) sorption capacity which was estimated to be 7.4 mg g⁻¹ based on Langmuir-Freundlich model. With solution pH ranging from 3 to 10, As(V) removal efficiency by MW250₁ kept stable and consistently higher than 90%. Besides, ~100% removal of 0.5 mM As(V) can be obtained in the presence of P ≤ 0.1 mM or Cr/Sb ≤ 0.5 mM, indicating a wide applicability of MW250₁ for treatment of As-containing water. The predominance of Fe₃O₄ on MW250₁ surface was evidenced by XRD. Ligand exchange between As(V) anion and the hydroxylated surface of Fe₃O₄ as well as H bond was largely responsible for As(V) sorption as suggested by FTIR. XPS analysis further revealed the dominance of As(V) in the sorbed As on MW250₁ surface with co-occurrence of a minor proportion of As(III) (11.45%). In parallel, oxidative transformation of Fe₃O₄ to Fe₂O₃ was also suggested by XPS. By a lab-scale column test, the potential and suitability of MW250₁ in As-containing water treatment was further confirmed, which could also provide an alternative way to manage and utilize this highly problematic invasive species.

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1. Introduction

Arsenic (As) is highly toxic to most living organisms and regarded as class I human carcinogen (Dummer et al., 2015; Nieto-Delgado and Rangel-Mendez, 2012). Long-term exposure to As can cause cancers of the lung, bladder and skin (Tuutijarvi et al., 2009). As-contaminated water used for drinking, food preparation and irrigation of food crops has been posing great threats to public health (Chen et al., 2007; Fendorf et al., 2010; Hettick et al., 2015; Meharg and Zhao, 2012; Rodríguez-Lado et al., 2013). In South and Southeast Asia, the geochemical occurrence of As in groundwater has caused the worst environmental disaster in human history and has been exposing millions of people to chronic As toxicity

through water drinking and rice consumption (Brammer and Ravenscroft, 2009; Fendorf et al., 2010; Meharg, 2004). In Hunan province which is the heartland of Chinese nonferrous mining and smelting, wide-spread As contamination in surface water and paddy soils has been determined in a number of mine-affected regions as a result of long-term discharge of As-containing drainage, improper storage of tailings, and deposition of metallurgical fume (Hu et al., 2015; Okkenhaug et al., 2012; Wang et al., 2015b; Williams et al., 2009). For example, the district of Shimen realgar mine (Changde, Hunan), which had the largest realgar deposit in Asia, has been subjected to the most severe As contamination in China with the local river water containing up to 765 μg l⁻¹ As (Hu et al., 2015).

In most terrestrial water bodies, inorganic arsenate [As(V)] is the dominate As species (Dovick et al., 2015; Ma et al., 2012; Salaiun et al., 2012) and mainly existed as H₂AsO₄⁻/HASO₄²⁻ at pH 2–11 (Ma et al., 2012; Mohan and Pittman, 2007). Previous studies have proved that As(V) has strong affinity to iron (Fe) oxides especially those with nanosize, forming inner-sphere monodentate or bidentate-binuclear complexes (Morin et al., 2008). Although As(V)

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sorption capacity of Fe (hydr)oxides tends to increase dramatically as the particle size decreases (Yavuz et al., 2006), nano-sized Fe (hydr)oxides have a strong tendency to form aggregates due to high surface energy, which would reduce its surface area substantially and hence As(V) removal efficiency. To solve this issue, loading of nanosized Fe oxides onto a low-cost and widely available supporting material could be an effective pathway (Chen et al., 2007; Deliyanni et al., 2013; Gu et al., 2005; Wang et al., 2015a; Zhang et al., 2013).

Biochar derived from pyrolysis of organic feedstocks in an oxygen-limited atmosphere at relatively low temperatures (<700 °C) has exhibited potential in carbon sequestration (Clough et al., 2013; Jha et al., 2010; Manyà, 2012; Woolf et al., 2010). With relatively abundant mineral ash and carboxylic functional groups, most biochars exhibit high alkalinity with relatively low anion exchange capacity (AEC, <5 cmol kg⁻¹). As a result, biochar generally has an ignorable to relatively low sorption capacity towards As anions (Beesley et al., 2014; Mohan et al., 2007). This was confirmed in our preliminary experiment with only 18.1% As(V) being removed by biochar converted from water hyacinth at initial As(V) of 5 mg l⁻¹ and 1/200 solid/water ratio after 24 h equilibration. However, limitless supply of waste agriculture biomass and less energy consumption during pyrolysis at relatively low temperature may offer apparent advantages for biochar as low-cost supporting materials. Therefore, production of biochar/FeO_x composite should be a useful manner to acquire novel As(V) sorbents with superiorities that cannot be attained by any of the components acting alone.

Water hyacinth represents the most problematic invasive species in southern China and is listed as one of the first batch of 9 invasive plant species by Ministry of Environmental Protection of China. More than 10 provinces as exemplified by Guangdong, Zhejiang, Hainan, Fujian, Yunnan and Hunan have been suffering from water hyacinth invasion which has caused substantial damage to local water quality, fishery, shipping, and aquatic biodiversity, etc. (Chu et al., 2006; Gao and Li, 2003; Lu et al., 2007; Yan et al., 2001, 2016; You et al., 2014; Zhang et al., 2015). Presently, it is generally difficult to utilize the collected biomass of water hyacinth in a profitable way in China, which in turn fails to provide economic support to a sustainable control of this invasive species.

In this work, water hyacinth was selected as feedstock for magnetic biochar production. As(V) sorption behaviors and the influence of pH and chemical analogs on As(V) sorption were investigated in detail. Packed-bed column test was also carried out to evaluate the suitability of this magnetic biochar for continuous As(V) removal from contaminated waters. By using scanning electron microscope (SEM)-energy dispersive X-ray spectroscopy (EDS), Fourier transform infrared spectroscopy (FTIR), X-ray diffraction analysis (XRD) and X-ray photoelectron spectroscopy (XPS), the underlying mechanisms of As(V) sorption by this magnetic biochar were further explored and discussed.

2. Materials and methods

2.1. Magnetic biochar preparation

To prepare magnetic biochar, sun-dried water hyacinth (collected from Lake Meixi in Changsha, China, 28°11'33"N Lat, 112°53'54"E Long) was subjected to co-precipitation with a mixture of ferrous and ferric chloride and subsequent pyrolysis under N₂ atmosphere (Chen et al., 2011). Briefly, an aliquot of 67 g sieved (0.2 mm) water hyacinth biomass was added into a 1000 ml solution (1:15 w/v) containing ferrous chloride and ferric chloride at 1:1 M ratio (0.1: 0.1, 0.5:0.5 and 1:1 mol l⁻¹). The suspension pH was then raised to 10 with 5 mol l⁻¹ NaOH. After stirring for 1 h, pH

of the suspension was checked and re-adjusted to 10. The deposit was then separated by filtration through a 0.22 μm filter and then subjected to slow pyrolysis (6.5 °C/min) under 250 or 450 °C for 1 h. The resulting products were rinsed with deionized distilled water and grounded to pass through a 200 meshes sieve. After drying in an oven at 65 °C for 5 h, the samples were collected and referred to as MW250_x, or MW450_x, with X representing the concentration of ferrous and ferric chloride in the initial mixture.

2.2. Characterization of MWs

The specific surface area, total pore volume (Single point adsorption), average pore radius and particle diameter of the MW samples were measured using Brunauer- Emmett-Teller (BET) analysis method (ASAP2020, Micromeritics, USA). The pH of MW was determined by adding 1 g sample to 20 ml deionized water. The suspension was then shaken for 1 h and allowed to stand for 5 min before measuring the pH. To test the amount of Fe loaded, MW samples were digested using CEM Mars 6 microwave digestion system (CEM, Matthews, NC, USA) according to EPA 3051a. Fe concentrations in the filtrated solution were determined by an atomic adsorption spectrum (AAS, PinAAcle 900T, PerkinElmer). The magnetic properties of MW250₁ before and after reaction with As(V) solution were measured using a vibrating sample magnetometer (VSM) at 300 K (VSM250, Changchun Yingpu Magneto-electricity Technology). To identify the Fe minerals loaded, X-ray diffraction (XRD) analysis of MW samples was conducted on a computer-controlled X-ray diffractometer (D/max 2500, Rigaku, Japan) equipped with a stepping motor and graphite crystal monochromator. Furthermore, water hyacinth biochar (BC250), magnetic biochar (MW250₁ and MW450₁) were examined using scanning electron microscopy (SEM, FEI, Quanta 200) at 25 keV coupled with energy dispersive X-ray spectroscopy (EDS, EDAX, Genisis) to unravel their surface morphology and surficial element composition.

2.3. As(V) sorption with MWs

2.3.1. Experiment 1 optimal parameters selection for MW production

To identify the optimum pyrolysis temperature and initial Fe²⁺/Fe³⁺ concentration for more efficient As(V) sorption by MWs, a 0.2 g aliquot of MW material prepared as described above was added into a 40 ml solution with 118 μg l⁻¹ or 4946 μg l⁻¹ As(V) followed by agitation at 150 rpm at 25 ± 1 °C. After being shaken for 24 h, the suspensions were then filtered through 0.22 μm filters and total As concentrations in the filtrate were determined by an atomic fluorescence spectrometer (AFS-9700, BJHG, Beijing), which has proved advantageous in terms of selectivity, linearity and detection levels over optical emission spectrometry (OES) and AAS (Rahman et al., 2000).

2.3.2. Experiment 2 kinetics study

In kinetics study, 5 mg l⁻¹ As(V) was prepared from the stock solutions of 100 mg l⁻¹ As(V) (as Na₂HAsO₄·7H₂O). A 0.2 g aliquot of MW250₁ sample was added to 40 ml As(V) solution followed by agitation in the dark at 150 rpm for 36 h. Preliminary experiment has demonstrated that apparent equilibrium was established in 24 h. At each time interval (0.5, 1, 2, 4, 6, 8, 24, 48, 72 h), three replicates of the suspension were sacrificed and centrifuged followed by filtration through 0.22 μm filters. As concentrations in the filtrates were determined by AFS.

2.3.3. Experiment 3 isotherm study

In isotherm study, MW250₁ sample was added to As solution

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