



Research article

Phosphate reclaim from simulated and real eutrophic water by magnetic biochar derived from water hyacinth

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ABSTRACT

In this study, the efficiency and mechanism of aqueous phosphate removal by magnetic biochar derived from water hyacinth (MW) were investigated. The MW pyrolyzed at 450 °C (MW450) exhibited the most prominent phosphate sorption capacity, which was estimated to be 5.07 mg g⁻¹ based on Langmuir–Freundlich model. At an initial phosphorus (P) concentration of 1 mg l⁻¹, >90% P removal was achieved over pH 3–9, but the efficiency decreased sharply at pH > 10. The presence of arsenate and carbonate could remarkably decrease P sorption, while the inhibition effects of antimonate, nitrate and sulfate were less significant. In further application of MW450 to reclaim P from eutrophic lake waters (0.71–0.94 mg l⁻¹ total P), ~96% P removals were attained in the batch studies and the effluent P concentrations in the column tests were reduced to <0.05 mg l⁻¹ within 509–1019 empty bed volumes. As indicated by XRD, MW450 surface was dominated by Fe₃O₄ and Fe₂O₃, resulting in a good ferromagnetic property of this composite (saturation magnetization 45.8 emu g⁻¹). Based on XPS, P sorption onto MW450 occurred mainly by surface complexation with the hydroxyl *via* ligand exchange. These results highlighted that MW derived from highly damaging water hyacinth could provide a promising alternative for P removal from most eutrophic waters.

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1. Introduction

Phosphorus (P) is a major but the least mobile nutrient in soil and, thus, shows the lowest phytoavailability for plant growth, especially for paddy rice (Rengel and Marschner, 2005; Yadav et al., 2012). In the main rice-producing areas in southern China, successive application of a high amount of P to advance rice yield has greatly contributed to eutrophication in freshwater lakes (Guo, 2007; Duan et al., 2009; Liang et al., 2016). Moreover, the lack of efficient sewage treatment in a range of less-developed regions has exacerbated water eutrophication by allowing continuous P input into various water bodies, especially those with low flow rates. In surface water, P concentration exceeding 0.05 mg l⁻¹ may cause eutrophic conditions (Hinesly and Jones, 1990). According to this criterion, a number of lakes in China are already in eutrophic state with total P up to 0.12–0.49 mg l⁻¹ (Fig. S1). This is not only

threatening water quality but also aquatic biodiversity and human wellbeing (Li et al., 2008; Gong et al., 2009; Zan et al., 2011; Chen et al., 2014; Hillman et al., 2014; Wu et al., 2014). Therefore, water eutrophication has now become a major environmental and public health challenge confronting China. Developing cost-effective technologies are critical in order to trap and reclaim P from eutrophic waters.

Since the late 1960s, a variety of physicochemical and biological technologies have been established mainly for phosphate removal from municipal and industrial effluents (Onar et al., 1996; Morse et al., 1998; Yao et al., 2011a). With a minimal requirement for system operation and little addition of chemicals, P sorption using widely available and high-affinity sorbents has exhibited important potential in cost-effectiveness and environmental friendliness (Li et al., 2006; Karageorgiou et al., 2007; Hamdi and Srasra, 2012; Vasudevan and Lakshmi, 2012). Recently, a biochar derived from the residue of anaerobically digested sugar beet tailings has been shown to remove P efficiently from aqueous solutions (Yao et al., 2011a, b). In their studies, the prominent P sorption capacity of biochar was mainly attributed to the enriched MgO particles with a high point of zero charge (PZC_{MgO} = 12), which facilitated P

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complexation at the hydroxylated MgO surface. In scaled-up applications, however, it could be difficult to recover the powdered biochar from the effluent. Moreover, most biochar with relatively high alkalinity (pH 7–12) and low pH_{PZC} (<4) generally has a low anion exchange capacity (<5 cmol kg⁻¹) and thus, very limited sorption capacity towards the negatively charged anions, such as P (Mohan et al., 2007; Beesley et al., 2011; Mukherjee et al., 2011; Hale et al., 2013).

To further explore the potential of biochar for efficient removal of problematic anion species, a magnetic biochar (MW) composed of iron oxides was prepared by chemical co-precipitation of Fe²⁺/Fe³⁺ on water hyacinth biomass followed by pyrolysis. The MW exhibited a notable capability to remove aqueous arsenate [As(V)] with the used sorbents being easily separated with a magnet (Zhang et al., 2016). Phosphate and As(V) are chemical analogs with many similarities (Elias et al., 2012) and, thus, it is conceivable that this MW could be effective in P reclaim from eutrophic waters. Furthermore, water hyacinth native to South America has been identified as a highly invasive water weed with rapid growth rates and extensive dispersal capabilities, which emerged in most tropical and subtropical areas in more than 50 countries including China (Plummer, 2005; Jafari, 2010; Shanab et al., 2010; Zhang et al., 2010). Seasonal outbreak of water hyacinth in a substantial number of surface waterbodies, partially due to water eutrophication, has caused extensive environmental, economical and societal problems (Villamagna and Murphy, 2010; Patel, 2012; Zhang et al., 2015). Therefore, the conversion of water hyacinth biomass into magnetic biochar could provide a useful concept for better management of this highly problematic and invasive species.

The objectives of the present study are (1) to demonstrate the kinetics and equilibrium isotherms of P sorption by the MW; (2) to identify the effects of pH and accompanying anions on P sorption; (3) to elucidate the underlying mechanisms governing P removal; and (4) to determine P removal efficiency by the MW from actual eutrophic lake waters, which is an important but missing link to advance P reclaim by the sorption technique.

2. Materials and methods

The magnetic biochar derived from water hyacinth (MW) used in this study is an Fe oxide-biochar nanocomposite prepared according to Zhang et al. (2016) and Chen et al. (2011).

2.1. P sorption by MWs

In light of Chinese discharge standards of pollutants for municipal wastewater treatment plants (GB18918-2002), the maximum allowed threshold of P in treated municipal wastewater ranges from 1 to 5 mg l⁻¹. Moreover, the upper limit of P concentration allowed in direct discharge from the phosphate fertilizer industry is 15 mg l⁻¹ according to the discharge standard of water pollutants for phosphate fertilizer industry proposed by Ministry of Environmental Protection of China (GB15580-2011). Based on the above standards, three representative P levels, i.e. 1, 5, 15 mg l⁻¹, were chosen for the following P sorption tests. P solutions were prepared by dissolving KH₂PO₄ (guaranteed reagent grade) in distilled water.

2.1.1. Experiment 1 comparison of P removal efficiency by MW250-550

To compare P removal efficiency by different MWs, 0.2 g of each MW sample was added to 40 ml of P solution containing 1 or 15 mg l⁻¹ P. The no-biochar control was also included. Each treatment had at least three replicates and the initial pH of each P solution was adjusted to 7 with 1 M NaOH or 1 M HCl. The mixtures

were then subjected to mechanical agitation for 24 h at 25 ± 0.5 °C. Upon filtration through 0.22 μm filters, P concentrations in the filtrates were determined by the ammonium molybdate spectrophotometric method (GB11893-89, Ministry of Environmental Protection of China) with an ultraviolet spectrophotometer (Shimadzu, UV-2550, Japan) at wavelength 700 nm. Based on the results of this experiment, MW450 was chosen for further tests due to its higher P removal capacity.

2.1.2. Experiment 2 selection of the optimal solid/solution ratio

To identify the effect of adsorbent dose on P removal, MW450 was mixed with the P solution (1 mg l⁻¹ P, pH = 7) at solid/solution (w/v) ratio from 1:100 to 1:1000. After 24 h of agitation, levels of P were determined as described above.

2.1.3. Experiment 3 sorption isotherm

The sorption isotherms of P by MW were determined by mixing 0.2 g MW450 with 40 ml P solution of different P concentrations from 0.186 to 150 mg l⁻¹. No-P and no-MW450 controls were also included. Each treatment had at least three replicates. The mixtures were adjusted to pH 7, shaken for 24 h at room temperature, and then filtered. Preliminary experiments had demonstrated that equilibrium was reached in 24 h. After this period, the suspensions were filtrated and the filtrates were used for P measurements.

2.1.4. Experiment 4 sorption kinetics

The kinetics were studied by adding 0.2 g of MW450 into 40 ml of P solutions containing 1, 5 or 15 mg P l⁻¹. The mixtures were agitated for a specified time period from 5 min to a maximum of 48 h. At each time interval, three replicates from each treatment were taken and the filtrates were analyzed for P.

2.1.5. Experiment 5 effect of pH and coexisting anions on P removal

In order to study the effects of pH on P sorption, P solutions (1, 5 and 15 mg l⁻¹ P) were adjusted to pHs ranging from 3 to 12 using 1 M NaOH or 1 M HCl. Aliquots of MW450 were then added to the P solutions at varying pH and P concentrations were detected after 24 h of agitation.

The impacts of the co-existence of As(V), antimonate [Sb(V)], nitrate (NO₃⁻), sulfate (SO₄²⁻), and carbonate (HCO₃⁻) on P sorption were investigated by preparing the stock solutions of these oxy-anions from NaH₂AsO₄, KSb(OH)₆, NaNO₃, Na₂SO₄ and NaHCO₃, respectively. Aliquots (0.2 g) of MW450 were added to P solutions (initial P of 5 mg l⁻¹, i.e. 0.16 mmol l⁻¹) with single competitive anion of different concentrations from 0.15 to 0.5 mmol l⁻¹. The mixtures were shaken for 24 h. After filtration, As and Sb concentrations in the equilibrium solutions were analyzed with an atomic fluorescence spectrometer (AFS6500, Haiguang, Beijing). Aqueous NO₃⁻, SO₄²⁻, HCO₃⁻ concentrations were determined by the ultraviolet spectrophotometric method (Chen et al., 2011; Frenzel and Rauterberg, 1992; Mecozzi and Monakhova, 2013).

2.2. Lab-scale application of MW450 for P removal from eutrophic lake waters

Lake Dianchi located in Yunnan province is one of the most eutrophic freshwater lakes in China. Long-lasting algal blooming in this lake has drawn great attention from both public and scientific communities (Qu and Fan, 2010) concerning its degradation. In the present study, water samples were collected from three sub-areas of this lake, which differed remarkably in the eutrophication degree (Fig. 1 and Table 1). Basic chemical properties of the water samples, including pH, P, total nitrogen (TN), permanganate index (CODMn), chlorophyll *a* (Chl *a*) were assessed according to Wei (2002).

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