



Effectiveness and mechanisms of phosphate adsorption on iron-modified biochars derived from waste activated sludge



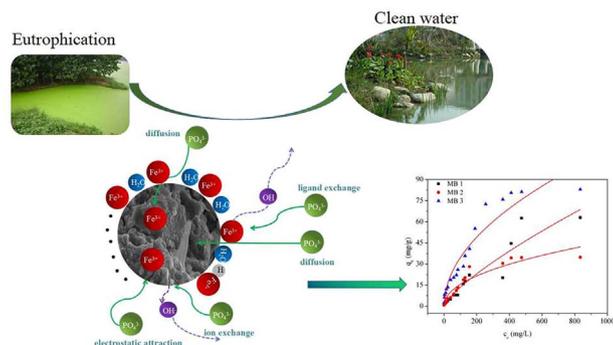
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GRAPHICAL ABSTRACT



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ABSTRACT

Excessive discharge of phosphate (P) into the surface water is the key factor to cause the eutrophication, so its removal has aroused much attention in recent years. In this study, different iron modification (chemical coprecipitation of Fe³⁺/Fe²⁺ or FeCl₃ impregnation) was used to improve the phosphate adsorption capacity of waste activated sludge (WAS)-based biochar. Comparative tests demonstrated that the FeCl₃-impregnated WAS-based biochar exhibited much superior phosphate adsorption capacity (111.0 mg/g) in all as-prepared samples and performed well even under the interferences with pH and coexisting ions. X-ray diffraction (XRD) analyzes indicated that the iron in FeCl₃-impregnated WAS-based biochar existed mainly in amorphous phase, as hematite and amorphous hydroxides forms, which was of great benefit to the phosphate adsorption. Besides, ligand exchange plays important role in the adsorption of phosphate. The WAS-based biochar kept over 60% phosphate removal efficiency after five recycles.

1. Introduction

Eutrophication, caused by the excessive emission of nutrient particularly phosphorous (P), has become a tough environmental problem

around the world (Zhao et al., 2015). High level phosphate can stimulate the growth of organisms, especially algae, in water bodies and thus deteriorate the quality of the aquatic ecosystems (Karaca et al., 2004). The natural recovery of phosphate is inefficient because

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phosphate are temporally converted into sedimentation and then released into waters again (Loganathan et al., 2014). Therefore, it is imperative to explore an efficient technology to remove phosphate from water.

To date, numerous technologies have been investigated to remove phosphorus from wastewaters, which are mainly divided into three categories: chemical, biological and physical methods (Karaca et al., 2004; Wang et al., 2012). Chemical phosphorus removal will generate plenty of sludge due to the precipitation of phosphorus and might potentially result in new pollution (Yao et al., 2011; Yeoman et al., 1988). Biological phosphorus removal is sensitive to the operation parameters, so its efficiency is unstable (Sun et al., 2017; Xie et al., 2017). Moreover, biological treatment involves the waste activated sludge disposal or other pretreatment, which would increase the cost of wastewater treatment (Neufeld and Thodos, 1969). Contrary to other expensive physical methods such as electrodialysis and reverse osmosis (Loganathan et al., 2014), adsorption is low cost and high efficiency. So it has been widely applied in phosphate removal from water (Bhatnagar and Sillanpää, 2011).

Various adsorbents have been used in phosphate removal, such as activated carbon (Loganathan et al., 2014), metal hydroxide (Chitrakar et al., 2005), blast furnace slag (Kumar et al., 2010) and so on. Recently, biochars have been concerned for their broad source, especially that they can be prepared from abundant waste cellulosic materials (Zhong et al., 2012; Zhao et al., 2016). However, the phosphate adsorption capacity of virgin biochar is unfavorable. Michalekova-Richveisova et al. (2017) demonstrated that the maximum phosphate adsorption capacity of three non-modified biochars were very low, which were 0.036 mg/g for corn cobs biochar, 0.132 mg/g for garden wood waste biochar and 0.296 mg/g for wood chips biochar, respectively. In order to improve the phosphate adsorption performance of biochar, iron modification is often employed. Compared with other modified methods, for instance the activation by sulfuric acid (Kumar et al., 2010) and lanthanum (Chouyyok et al., 2010), iron modification had simple and efficient features. It is well known that different iron modification may cause different valence states of iron in biochar. However, the relationship between the phosphate adsorption efficiency and valence states of iron existed in the biochar is vague.

In this study, the influence of inert atmosphere on the valence states of iron existed in biochars will be explored. Firstly, biochar derived from waste activated sludge (WAS) was modified by three different methods: co-precipitation of $\text{Fe}^{2+}/\text{Fe}^{3+}$ under nitrogen atmosphere, co-precipitation of $\text{Fe}^{2+}/\text{Fe}^{3+}$ under air and impregnation with ferric chloride. Then the properties of three iron-modified biochars were characterized and their phosphate removal efficiency was compared. Lastly, the underlying mechanism was discussed.

2. Materials and methods

2.1. Materials

The WAS taken from a secondary sedimentation tank of a municipal wastewater treatment plant in Changsha, China was concentrated by settling at 4 °C for several days. After pouring the liquid supernatant, the concentrated sludge was dried in oven at 105 °C and sieved into fine powders for further use.

The phosphate stock solution (1000 mg/L) was prepared by dissolving potassium dihydrogen orthophosphate (KH_2PO_4) powders in deionized water and desired solutions were prepared by diluting the phosphate stock solution. All the chemical reagents used in the study are of analytical grade.

2.2. Biochar preparation and modification

The virgin biochar, produced by pyrolyzing dewatered sludge at 550 °C without any modification, did not achieve obvious phosphate

removal but release 15.88 mg/g phosphate. When immersed by 0.7 M ZnCl_2 solution, the phosphate removal rate of biochar raised to 28.5% for 20 mg/L phosphate solution (data not shown). Thus further modification of biochar is necessary.

The WAS-based biochar was generated through an impregnation and pyrolysis process. Briefly, the dewatered sludge powders was soaked with 0.7 M ZnCl_2 solution with magnetic stirring for 24 h. The impregnated samples were pyrolyzed in 550 °C and retained for 2 h under nitrogen flow to prepare the biochar. Lastly, the biochar was crushed and sieved to 0.5–1 mm size fraction, washed with deionized water to remove impurities, oven dried at 65 °C and sealed in glass container.

As-prepared biochars were modified by three different iron modification as follows:

- (1) Biochar decorated by co-precipitation of $\text{Fe}^{2+}/\text{Fe}^{3+}$ under nitrogen atmosphere (labeled MB1): Under room temperature and nitrogen atmosphere, 1 g biochar was added into a 30 mL solution, which contained 1.17 g $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ and 1.09 g $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, stirred vigorously at 150 rpm for several minutes followed by the dropwise adding of NaOH to adjust pH to 11. Aqueous suspension kept stirring for 45 min and aged overnight without stirring. The separated solid was washed several times with deionized water and dried in vacuum drying chamber.
- (2) Biochar decorated by co-precipitation of $\text{Fe}^{2+}/\text{Fe}^{3+}$ under air atmosphere (labeled MB2): The process was same with (1) except that there was no nitrogen protection and the solid was dried in oven.
- (3) Biochar decorated by impregnation with ferric chloride under air atmosphere (labeled MB3): The process was same with (2) except that the solution only contained 2.18 g $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$.

2.3. Batch adsorption experiment

The batch adsorption experiments were conducted to determine the phosphate adsorption capacity of three novel biochars. 0.1 g biochars were loaded into a 250 mL Erlenmeyer flask, containing 50 mL of phosphate solutions with the concentration of 20 mg/L. The initial pH was adjusted to 7.0 ± 0.2 by adding 2 M HCl or NaOH. Then Erlenmeyer flasks were shaken at 120 rpm and 22 ± 0.5 °C in a temperature controlled water bath shaker until the adsorption equilibrium. The phosphate adsorption performance was compared by the decrease of the phosphate amount in the solution.

2.4. Adsorption isotherm

To examine the adsorption isotherm of the biochar, 0.1 g adsorbent was added into 50 mL phosphate solutions with different initial concentration (5–1000 mg/L). The adsorption capacity at equilibrium was calculated according to Eq. (1):

$$q_e = V \times \frac{(c_e - c_0)}{m} \quad (1)$$

where q_e is the equilibrium adsorption capacity (mg/g), V is the volume of solution (mL), c_e and c_0 represent the concentrations at the equilibrium and initial time (mg/L), respectively, and m is the weight of adsorbent (g).

2.5. Adsorption kinetics

In this experiment, 0.2 g biochar was loaded into 100 mL phosphate solutions with the concentration of 20 mg/L at room temperatures. The residual phosphate content at different time intervals (0, 5, 10, 20, 40, 60, 90, 120, 240, 720 and 1440 min) was analyzed to determine the adsorption kinetics.

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