



Sorption of dissolved inorganic and organic phosphorus compounds onto iron-doped ceramic sand



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ABSTRACT

Sorption-based processes are widely used in the wastewater treatment. We present results of a study contrasting the sorption behavior of several dissolved organic phosphorus (DOP) compounds and phosphate onto three ceramic sands with different physicochemical properties. The synthesized sorbent materials were characterized by nitrogen adsorption analyses, X-ray diffraction (XRD) and infrared spectroscopy (FTIR). The DOP compounds were chosen to represent different molecular weights and structures. The effects of solution pH, P concentration and contact time on sorption capacity was investigated by kinetic and equilibrium sorption isotherm studies. The order of maximum sorption capacity observed for the three P compounds onto iron-doped ceramic sand samples, on a per gram dry weight basis, is phosphate (DIP) > β -glycerophosphate disodium salt hydrate (β -GP) > adenosine 5'-triphosphate disodium salt hydrate (ATP-P). Langmuir isotherm sorption equation well describes the experimental sorption isotherms. Kinetic studies revealed that the sorption process followed both pseudo-first order and pseudo-second order kinetic models. The iron-doped ceramic sand samples had a higher phosphorus removal capacity than the raw, which could be attributed to its better intra-particle diffusion and higher binding energy. P compound size and structure, and the nature of the sorbents all appear to play a role dictating relative sorption capacity. The sorption process was complex; both surface sorption and intra-particle diffusion were simultaneously occurring and contribute to the sorption mechanism.

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1. Introduction

Phosphorus is an essential, often limiting, nutrient for growth of organisms in most ecosystems, and has been recognized as one of the main species responsible for eutrophication of fresh water bodies (Baldy et al., 2007). Excess phosphorus entering into rivers and lakes triggers the disordered growth of undesirable algae and other aquatic plants (De-Bashan and Bashan, 2004). As regulations continue to become more stringent, reducing or removing phosphorus from polluted water body is one of the most important tasks for wastewater treatment. In concert with the provision of efficient nutrient removal at large wastewater treatment facilities, treatments of sewage discharged from small rural communities in China are adsorbing more attention for improving the environmental quality in streams and rivers. Therefore, it is urgent to find efficient as well as economically attractive solutions for these small wastewater producers.

Different on-site solutions have been launched, such as bio-film systems, sand filters and constructed wetland systems (Erickson et al., 2012; Helness and Odegaard, 2001; Mateus et al., 2012). The degree of treatment required in the system is determined by regulations, which may contain standards for suspended solids (SS), biochemical oxygen demand (BOD), total nitrogen (TN) and total phosphorus (TP). Most systems are able to fulfill the requirements for SS, BOD and TN removal. However, it is often a problem to remove phosphorus in these small-scale, on-site systems (Lantzke et al., 1998). The main removal mechanism for phosphorus in these systems is plant uptake, microbial immobilization, retention by media and precipitation in the water column. Several studies have investigated that the amount of phosphorus which could be removed by harvesting plants usually constitutes only a small proportion of the amount of phosphorus loaded into the system with the sewage (Brix, 1997). Consequently, attention should be paid to explore the availability of materials to remove P from the sewage. Conventional sorbents may not be feasible in practical sewage treatment and eutrophication control because either some of them such as sands, limestone, fly ash, etc. do not have sufficient sorption capacities or some like steel slag, zeolite and red mud have potential toxic effects.

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Numerous studies have been carried out on the sorption of phosphate onto iron-doped materials (Liu et al., 2013; Sousa et al., 2012), and revealed that iron has a high affinity toward phosphate and it is selective in the sorption process (Shanableh and Elsergany, 2013). Removal has been attributed to ion exchange, specific sorption to surface hydroxyl groups or coprecipitation. The bound phosphorus in the sewage exists in inorganic and organic states. Both inorganic and organic phosphorus provide significant risk for eutrophication (Jarvie et al., 2006). While directly bioavailable as phosphate, Dissolved Organic P (DOP) is rendered bioavailable after enzyme hydrolysis cleaves phosphate from DOP (Ruttenberg and Sulak, 2011). However, fewer studies have explored DOP sorption on sorbents in practical sewage treatment, and these have focused exclusively on lake sediment (Wang et al., 2007) or seawater systems (Ruttenberg and Sulak, 2011). While studies of DOP sorption onto sediments can be instructive when considering similar processes in sewage treatment systems, they are not directly relevant because of the profoundly different ionic strength and pH of sediments versus sewage treatment systems.

We are unaware of any studies that examine both inorganic and organic phosphorus sorption onto iron-doped ceramic sand which could be used in sewage treatment systems. Consequently, understanding the reactions between phosphorus and sorbents is important for understanding and modeling its movement and fate in the sewage treatment systems. Both sorption under equilibrium conditions and kinetics are important in this regard. The objective of this work was to study the feasibility of using ceramic sands as sorbent for phosphorus removal from sewage and to perform a comparative study of the behavior of several dissolved organic phosphorus (DOP) compounds and phosphate on three ceramic sands samples with different physicochemical properties. The sorbents were characterized by scanning electron microscope (SEM), nitrogen adsorption analyses, X-ray diffraction (XRD) and infrared spectroscopy (FTIR). The sorption isotherms, kinetics and initial concentrations of the phosphorus effect were evaluated in batch experiments. These work could give some insights into the inorganic and organic phosphorus removal process using ceramic sands and represent an initial evaluation of iron-doped ceramic sands for future engineering applications.

2. Materials and methods

2.1. Sorption materials

Unprocessed ceramic sand supplied by the Xinbei Building Material Factory (Nanjing, China) was used in this study. The ceramic sand (≤ 100 mesh, signed as T) was first rinsed with boiling distilled water to remove impurities, and then was washed with the 0.1 mol L^{-1} HCl solution to remove salts precipitated in its pores. Afterwards, the ceramic sand was repeatedly washed with the distilled water to remove all acid residues. Finally, the cleaned sample was dried at 105°C for 24 h to volatilize impurities, and then modified with 5 mol L^{-1} HNO_3 for 6 h at the room temperature. Oxidized sample was thoroughly washed by distilled water to remove residual acid adsorbed before iron loading. The ceramic sand oxidized by HNO_3 was named T/N hereinafter. The iron-doped ceramic sand samples were prepared by introducing 1 g of unoxidized and oxidized ceramic sand into 100 mL of 0.2 mol L^{-1} iron solution made from $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, respectively. The pH during iron loading was kept at 2.0. The mixtures were stirred continuously for a few minutes, and then heated at 110°C until they become dry. Afterwards, they were cooled at room temperature, rinsed to remove Fe residues, dried, and sieved. The two prepared iron-doped ceramic sand are denoted as T-Fe and T/N-Fe, respectively.

2.2. Characterization of materials

Mineralogy of the ceramic sand samples were obtained using an X-ray diffraction (XRD) analysis instrument (D/MAX-IIA, Japan) with $\text{CuK}\alpha$ radiation (40 kV, 30 mA) over the 2θ range $10\text{--}80^\circ$. Surface morphology and structure of the ceramic sand samples were examined with scanning electron microscope (Hitachi, S-520, Japan). Surface area measurements were obtained by N_2 adsorption at 77 K using the Micromeritics ASAP-2020 Surface Analyzer in the relative pressure range of 0.01–1. The surface functional groups were identified by transmission infrared spectra obtained from a Fourier transform spectrophotometer (Nicolet Nexus 470, USA) using pellets of KBr containing about 0.5% finely ground ceramic sand samples. These pellets were dried overnight at 120°C before the spectra were recorded and the spectra were the result of averaging 60 scans with the range of $400\text{--}4000 \text{ cm}^{-1}$. The elemental composition of the ceramic sand samples was carried out in the Testing and Analysis Center of Fudan University using the atomic adsorption spectrophotometer (Hitachi, Z-5000, Japan).

2.3. Phosphorus compounds

In addition to phosphate, two organic phosphorus compounds representing different molecular sizes and P bond types, were selected for experimentation (Table 1). The choose of β -glycerophosphate disodium salt hydrate was used to examine the relative sorption affinity of mid-lever molecular weight phosphorus. Adenosine 5'-triphosphate disodium salt hydrate was chosen to evaluate the sorption of a high molecular weight phosphorus with a tri-polyphosphate tail. Phosphate was included to provide a referent against which the different DOP compounds could be compared.

2.4. Sorption isotherm experiments

Sorption isotherm experiments were conducted for phosphate, β -GP, and ATP-P sorption onto T, T-Fe, and T/N-Fe. Since all chemicals are of analytical reagent grade, they were used without further purification. The stock solution containing 1000 mg L^{-1} of phosphorus (in P) was prepared by dissolving Potassium phosphate dibasic trihydrate, β -Glycerophosphate disodium salt hydrate, and Adenosine 5'-triphosphate disodium salt hydrate in distilled water, respectively, and desired solutions were prepared by dilution of the stock solution. Sorbents (0.5 g) (T, T-Fe, T/N-Fe) and 20 ml solutions of certain phosphorus concentration ($0.45\text{--}94.43 \text{ mg L}^{-1}$) in a 50 ml Erlenmeyer flask, and the mixture was agitated in a thermostated shaker (Model THZ-C, China) at a speed of 160 r/min at room temperature (25°C) for 24 h to reach equilibrium. In pH studies, the solution pH was adjusted to 4.15–10.54 with 0.01 mol L^{-1} HCl or 0.01 mol L^{-1} NaOH solutions after the mixing of 20 ml phosphorus solution with 0.5 g ceramic sand samples, and the initial phosphorus concentration was 20 mg L^{-1} . pH values were measured by a pH meter (Model pHs-2C, China).

After sorption, the supernatant liquids were filtered with $0.45 \mu\text{m}$ filter paper and the residual phosphorus concentrations were determined by UV/vis spectrophotometer (Model UV-759S, China) at 700 nm wave length. Phosphate from sorption experiments was determined in duplicate using the molybdenum blue method described by Murphy and Riley (1962). DOP was analyzed according to Jeffries et al. (1979), which employed an acid (sulfuric acid) potassium persulphate ($\text{K}_2\text{S}_2\text{O}_8$) oxidation procedure. Sorbed P was assumed to be the difference between the initial P concentration C_0 (mg L^{-1}) and the solution P concentration after completion of the experiment, which was at or near equilibrium C_e (mg L^{-1}).

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