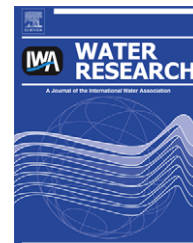


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## Development of polymer-based nanosized hydrated ferric oxides (HFOs) for enhanced phosphate removal from waste effluents

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### ABSTRACT

Phosphate originated from industrial effluents is one of the key factors responsible for eutrophication of the receiving waterways especially in the developing countries such as China. In the current study we proposed a novel process to immobilize nanoparticulate hydrated ferric oxide (HFO) within a macroporous anion exchange resin D-201, and obtained a hybrid adsorbent (HFO-201) for enhanced phosphate removal from aqueous system. The resulting HFO-201 possesses two types of adsorption sites for phosphate removal, the ammonium groups bound to the D-201 matrix and the loaded HFO nanoparticles. The coexisting sulfate anion strongly competes for ammonium groups, which bind phosphate through electrostatic interaction. However, it does not pose any noticeable effect on phosphate adsorption by the loaded HFO nanoparticles, which is driven by the formation of the inner-sphere complexes. Batch adsorption experiments also indicated that HFO-201 exhibits a little higher capacity for phosphate than the commercially available phosphate-specific adsorbent ArsenX<sup>TP</sup>, which possesses similar structure of HFO-201 and is produced by another patented technique. Fixed-bed column tests indicate that phosphate retention by HFO-201 from the synthetic waters results in the significant decrease of P from 2 mg/L to less than 0.01 mg/L, with the treatment capacity of ~700 bed volume (BV) per run, while that for D-201 was less than 200 BV under otherwise identical conditions. Such satisfactory performance of the hybrid adsorbent is mainly attributed to the specific affinity of HFO toward phosphate as well as the Donnan membrane effect exerted by the anion exchanger support D-201. Moreover, the exhausted HFO-201 was amenable to efficient in situ regeneration with a binary NaOH–NaCl solution for repeated use without any significant capacity loss. Similar satisfactory results were also observed by using a phosphate-containing industrial effluent as the feeding solution.

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## 1. Introduction

Phosphorus has been regarded as a limiting nutrient responsible for eutrophication of the water bodies, and enhanced removal of phosphate from waste effluents is thus of considerable significance before their discharge into the receiving waterways (Hecky and Kilham, 1988; Conley et al., 2009). Nowadays there are various techniques proposed for phosphate removal, which can be simply classified as chemical precipitation by using ferric or aluminium salts (Sherwood and Qualls, 2001; Clark et al., 1997; Narasiah et al., 1994), biological removal (Van Loosdrecht et al., 1997; Oehmen et al., 2007), and adsorption (de Vicente et al., 2008; Onyango et al., 2007). Chemical precipitation and biological processes have been widely used for phosphate removal from industrial effluents (Rahman and Mukhtar, 2008; Wang et al., 2005), however, it is still technically difficult or economically undesirable for both techniques to enhance phosphate removal as to meet increasingly stringent regulations on phosphate discharge. For instance, in China the maximum allowable phosphate was set as 0.5 mg·P/L for industrial effluent discharge (China EPA, 1996), and the current technologies usually cannot achieve the goal in a cost effective manner. Adsorption seems attractive for phosphate removal due to its operation simplicity and low operation cost (Onyango et al., 2007). To further improve the efficiency of an adsorption process for enhanced removal, it necessitates the development of phosphate-specific adsorbents (Saha et al., 2009; Chitrakar et al., 2006).

In the past decades ferric oxides have proved to exhibit specific removal of anionic pollutants, namely, arsenite/arsenate (Jang and Dempsey, 2008), selenite (Parida et al., 1997), or phosphate (Ryden et al., 1977; Khare et al., 2005), from contaminated waters. Moreover, they are environmentally benign, chemically stable over a wide pH range, and cost effective. However, ferric oxides are usually present as fine particles and difficult for use in a continuous flow system. To overcome the technical bottleneck, many researchers have focused on doping particulate ferric oxides within the traditional adsorbents of larger particle size (Jang et al., 2008; Zhang et al., 2008b; Xiong and Peng, 2008). All these hybrid adsorbents combine the excellent handling, flow characteristics of adsorbent supports with the specific affinity of ferric oxides toward the targeted anion pollutants. Nevertheless, surface chemistry of the support materials on performance of the resulting hybrid adsorbents is not well explored.

Recently, Sengupta and his group have implemented the Donnan membrane principle to fabricate more efficient hybrid adsorbents for trace arsenic removal (Cumbal and Sengupta, 2005). Similar related researches have also been performed in our laboratory for enhanced removal of trace heavy metals from waters. As for phosphate removal, Sengupta et al. fabricated a novel hybrid adsorbent HAIX by doping hydrated ferric oxides (HFO) within a polymeric anion exchanger by a propriety technique (Blaney et al., 2007). The basic preparation procedures involved ion exchange of anionic oxidants onto the anion exchanger, rinsing or soaping the exchanger by Fe(II) solution to result in an in situ precipitation of iron hydroxide, and thermally treatment of the resulting

composites (SenGupta and Cumbal, 2005). The positively charged exchanger support is expected to result in permeation enhancement and pre-enrichment of phosphate anions within the polymeric phase prior to preferable adsorption by HFO particles. Obviously, such phenomena would be greatly favorable for efficient removal of trace phosphate. As they reported, phosphate can be efficiently removed from 200 to 260 µg/L to less than 50 µg/L (Blaney et al., 2007).

In the current study we proposed a new method to fabricate a similar polymeric hybrid adsorbent HFO-201, and examined its potential use for enhanced removal of phosphate from industrial effluents. Considering phosphate in many industrial effluents after chemical precipitation or biological removal usually ranges from 1 to 5 mg/L (Clark et al., 1997; Barth et al., 1968), we set the initial P concentration as 2 mg/L in batch adsorption tests. The industrial effluent from a pesticide plant in China was also selected as the feeding solution to further demonstrate the applicability of HFO-201 in enhanced P removal from wastewater.

## 2. Methodology

### 2.1. Materials

All chemicals were reagent grade from Shanghai Reagent Station (Shanghai, China) and used without further purification. The stock solution containing 1000 mg/L of orthophosphate (in P) was prepared by dissolving  $\text{KH}_2\text{PO}_4$  into the de-ionized water. Another HFO-loaded hybrid adsorbent ArsenX<sup>np</sup>, which has been demonstrated as a superior adsorbent for phosphate and arsenic (Cumbal and Sengupta, 2005; Blaney et al., 2007), was obtained from Purolite (China) Co. Limited for comparison purpose. The commercially available polymeric anion exchanger D-201 (in  $\text{Cl}^-$  type), with spherical beads ranging from 0.6 to 1.0 mm in diameter, was provided by Zhengguang Electrical Resin Co. Ltd. (Hangzhou, China). Prior to use, D-201 was subjected to extraction with ethanol in a Soxhlet apparatus to remove the possible residue impurities.

### 2.2. Preparation of HFO-201

It is well demonstrated that the anion exchanger resin is an ideal support material to fabricate an HFO-loaded hybrid adsorbent for phosphate or arsenate (Cumbal and Sengupta, 2005; Blaney et al., 2007). This is mainly because the immobilized ammonium groups bound to the polymeric matrix would favor permeation enhancement and even preconcentration of the targeted pollutants prior to their effective sequestration by the loaded HFO particles (Cumbal and Sengupta, 2005). However, the traditional HFO precursor,  $\text{Fe}^{3+}$ , cannot be readily ion exchanged onto an anion exchanger like D-201 as a result of the electrostatic repulsion. Herein, we developed a novel and propriety process to fabricate the target hybrid adsorbent (Pan et al., 2005). First, a binary  $\text{FeCl}_3$ -HCl solution was prepared and flew through a column packed with D-201 beads, where the resulting  $\text{FeCl}_4^-$  anions present in solution were preferably ion exchanged onto D-201 due to its much lower hydration energy than chloride (Gu

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