

## Regular Article

# Adsorption of phosphate from aqueous solution using iron-zirconium modified activated carbon nanofiber: Performance and mechanism



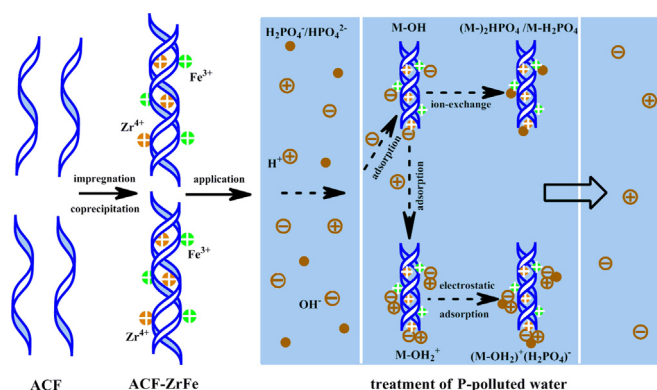
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## GRAPHICAL ABSTRACT



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## ABSTRACT

Phosphate (P) removal is significant for the prevention of eutrophication in natural waters. In this paper, a novel adsorbent for the removal of P from aqueous solution was synthesized by loading zirconium oxide and iron oxide onto activated carbon nanofiber (ACF-ZrFe) simultaneously. The adsorbent was characterized by scanning electron microscopy (SEM), Fourier transform infrared (FT-IR) spectroscopy and X-ray photoelectron spectroscopy (XPS). The results showed that P adsorption was highly pH dependent and the optimum pH was found to be 4.0. The isotherm of adsorption could be well described by the Langmuir model and the maximum P adsorption capacity was estimated to be 26.3 mg P/g at 25 °C. The kinetic data were well fitted to the pseudo-second-order equation, indicating that chemical sorption was the rate-limiting step. Moreover, co-existing ions including sulfate ( $\text{SO}_4^{2-}$ ), chloride ( $\text{Cl}^-$ ), nitrate ( $\text{NO}_3^-$ ) and fluoride ( $\text{F}^-$ ) exhibited a distinct effect on P adsorption with the order of  $\text{F}^- > \text{NO}_3^- > \text{Cl}^- > \text{SO}_4^{2-}$ . Further investigations by FT-IR spectroscopy and pH variations associated with the adsorption process revealed that ligands exchange and electrostatic interactions were the dominant mechanisms for P adsorption. The findings reported in this work highlight the potential of using ACF-ZrFe as an effective adsorbent for the removal of P in natural waters.

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## 1. Introduction

Phosphate (P), an indispensable macronutrient widely found in aquatic environments, is highly required for the growth of living organisms and the normal functioning of ecosystems. However, about 1.3 Mt of P in every year from all over the world discharged into aquatic systems will lead to over-fertilization and cause eutrophication and ecosystem degradation [1–4]. Therefore, it is necessary to take highly effective, reliable, and economical methods to remove P from wastewater prior to its discharge.

Stringent regulations have been implemented by many countries with respect to the presence of P in water. These strict regulations have triggered a variety of P removal technologies, including chemical precipitation which relies on ferric or aluminum salts [1,5], adsorption methods [6–10], biological treatments [11–13], and ion exchange [14]. Among these available treatment methods, adsorption is considered to be an effective approach due to its easily-handle operation, low cost and eco-friendly characters [15–18]. Various adsorbents such as fly ash [19], aluminum hydroxide [20], iron-based compounds [21–24] and Ca-based sorbents [25] have been tested for P adsorption. However, the direct addition of adsorbents in water treatment system may cause fast loss of adsorbents and reduce their reusability as a result of their tiny powder size, which usually restrict the application of adsorbents [26]. To further improve the recyclability and adsorption capacity, it is of particular significance to develop more novel phosphate-specific adsorbents. In recent investigations, zirconium-based materials have been paid more attention in controlling P pollution due to their high binding affinity towards P, non-toxicity and acceptable cost. Awual et al. [27] reported fibrous loaded with zirconium for P removal from water. Su et al. [28] synthesized amorphous zirconium oxide nanoparticles via a simple and low-cost hydrothermal process and the new adsorbent exhibited remarkable P adsorption capacity under various conditions. In addition, iron-based materials have been reported as showing good P removal efficiency and possessing advantageous characteristics in terms of cost, chemical stability and environmental consequence [29]. In order to inherit the advantages of these two kinds of adsorbents, a composite sorbent containing zirconium and iron oxides was developed and exhibited promising performance for P adsorption [15].

Activated carbon nanofiber (ACF), which is a highly microporous carbon material and shows favorable adsorption performances due to its nano-structure, abundant micrometer porosity, high specific surface area and uniform micropore size distributions [30,31], is believed to have potential applications in adsorption. The particular characteristics of ACF mentioned above are due to the versatility of being loaded with metal oxide. For instances, metal oxides such as titanic oxide [31], manganese oxide [32], iron oxides [33] and Lanthanum iron oxides [34] have been successfully introduced to the surface of ACF. Manganese-modified ACF was found to have a high efficiency of arsenic removal by providing abundant amounts of binding sites on the obtained Mn-ACF adsorbent surface [32]. The adsorption capacity for P of Lanthanum oxide doped ACF [34] was enhanced, which was principally attributed to the formation of new coordination sites from the hydroxyl groups on lanthanum oxide. Nevertheless, no report is available on the study of the phosphate adsorption by Fe–Zr modified ACF which may have promising adsorption capacity, as the functional groups may be formed in ACF after the modification.

In this study, a new adsorbent for the removal of P from aqueous solution was prepared by loading zirconium and iron particles on the surface of ACF (ACF-ZrFe) simultaneously. The main objectives of this study are to: (i) characterize the synthesized adsorbent by scanning electron microscopy (SEM), X-ray photoelectron

spectroscopy (XPS) and Fourier transform infrared spectroscopy (FT-IR); (ii) systematically evaluate the influences of various experimental parameters, such as initial P concentration, pH values, reaction time and co-existing substances on adsorption performance; (iii) reveal the mechanisms of P adsorption onto ACF-ZrFe.

## 2. Experimental procedures

### 2.1. Materials

All reagents such as zirconium oxychloride octahydrate, ferric nitrate, potassium phosphate monobasic and ammonium hydroxide were of analytical grade and purchased from Sinopharm Chemical Reagent Co., Ltd, China. ACF was procured from Nantong Senyou Carbon Fiber Co., Ltd., Nantong, China. This ACF has specific surface area of 1300–1400 m<sup>2</sup>/g, with thickness of 1–5 mm. Stock solution of P was prepared using anhydrous KH<sub>2</sub>PO<sub>4</sub> and pH was pre-adjusted by HCl or NaOH solutions. All water samples containing P were filtrated through 0.45 μm membrane filters and then quantitatively determined by the molybdenum blue method [35] on a SHIMAD UV-2401PC UV/Vis spectrometer with the detection wavelength of 700 nm. All solutions were prepared using deionized water.

### 2.2. Synthesis of adsorbent

ACF was first cut into desired size pieces (0.5 cm × 0.5 cm), which was then fully washed four times with deionized water and kept in boiling water for 30 min to remove impurities and soluble salts. The treated ACF was dried at 100 ± 5 °C for 4 h. The zirconium-iron-modified ACF (ACF-ZrFe) was prepared by a one-step synthesis method as follows. 0.5 g of ACF was subsequently immersed into zirconium oxychloride octahydrate and ferric nitrate mixed solution. After 24 h of reaction, the obtained zirconium-iron-modified ACF medium was combined with a 1 M NaOH solution for 2 h to precipitate the mixed ions. The product was filtered and washed repeatedly with distilled water to lower the pH below 8.0. After drying at 105 °C for 24 h, the zirconium-iron-modified ACF (ACF-ZrFe) adsorbent was obtained.

### 2.3. Characterization

The morphology of the sample was characterized using a scanning electron microscope (SEM, JSM-6400F). The FT-IR spectrum of ACF-ZrFe adsorbent before and after phosphate adsorption were measured by a Nicolet 6700 FT-IR spectrometer. XPS analysis was conducted using an ESCALAB 250Xi X-ray photoelectron spectroscopy equipped with a monochromatized Al Kα X-ray source, all the binding energies were referenced to the C 1s peak at 284.8 eV of the surface adventitious carbon.

### 2.4. Batch sorption experiments

All the phosphate adsorption tests were carried out at room temperature in 250 mL conical flasks. Phosphate solution of 100 mL was used for each experiment with varying dose of ACF-ZrFe. Batch adsorption experiments were carried out at different initial pH values ranging from 2.0 to 12.0. The solution pH was adjusted by adding HCl (0.10 M) or NaOH (0.10 M) solutions. Considering that abundant salt additives are often present in natural waters, it is important to examine the performance of ACF-ZrFe under high ionic strength, thus, Na<sub>2</sub>SO<sub>4</sub>, NaCl, NaF or NaNO<sub>3</sub> solutions were added as sources of the competing anions when necessary. The mixture was shaken at a thermostatic shaker with certain

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