



Preparing of novel fibrous ligand exchange adsorbent for rapid column-mode trace phosphate removal from water



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ABSTRACT

We developed a potentially high-performance adsorbent for sustainable treatment of soluble inorganic trace phosphate from water by zirconium(IV) loaded bifunctional fibers. In the presence of common Cl^- and SO_4^{2-} , phosphate adsorption was not adversely affected but slightly enhanced due to co-ion and Donnan invasion mechanism. Trace phosphorus (0.0143 mM) was also removed in presence of relatively high amounts of competing anions at high feed flow rate (850 h^{-1}). In competitive arsenate and phosphate adsorption, this novel adsorbent slightly preferred phosphate to arsenate. The adsorbent is reversible and keeps remaining functionality to further reuse in many cycles.

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1. Introduction

Eutrophication of water bodies such as rivers, lakes and inland seas is one of the most important environmental problems and phosphate is considered to be a key factor leading to this problem [1–3]. Consequently, phosphate removal from water bodies is an important issue to control eutrophication and conserve the natural aquatic environment. Therefore, recommended maximum permissible limit of phosphorus (P) in wastewater is $10\text{--}50 \mu\text{g/L}$ to prevent such problem for living organisms [4]. On the other hand, phosphorous is a key nutrient element for living organisms but the excess amount adversely affected.

Many methods have been investigated for phosphate removal from wastewater namely physical and coagulation methods [5], chemical precipitation by using ferric, calcium or aluminum salts [6–8], biological removal [9], and adsorption [10–15]. However, physical methods are expensive and inefficient for phosphate removal from wastewater [5]. Chemical precipitation and biological processes have been widely used but they suffered from high costs, sludge handling and operational difficulties with low efficiency for trace level of phosphate removal [16,17]. Many

researchers have devoted their efforts in inventing new techniques to remove toxic anions and cations to ensure the safety of water for human consumption and to preserve the natural aquatic environment. Recently, ligand immobilized mesoporous adsorbent have attracted much attention for their accurate and rapid detection/removal/extraction of target ions [18–20]. Moreover, these are cost effective, easy to use, and show promise in capturing of metal ions. Adsorption is the most attractive technique based on economic and high efficiency approach for phosphate removal, particularly for low phosphate concentration in wastewater. Such features have shown intensive interest to many scientists as a suitable treatment technology [2,12,13,21]. Several types of phosphate adsorbents have been investigated such as natural and abundant wastes [10,11,14], primary amine containing fibrous anion exchangers [22,23], metals loaded ligand exchange chelating resins and fibers [17,24–26], aluminum/ferric oxide or hydroxide [27], fly ashes and activated carbon [28]. Many adsorbent have preferred to phosphate even in the presence of high concentration foreign anions but slow kinetics [13]. Several metals oxides adsorbents are unable to reuse and leaked the immobilized metal ion during phosphate adsorption or elution or regeneration operations [24,29].

In the case of metal loaded fibrous adsorbents, the metal ion is strongly retained on functional groups even for a large pH variation interval during adsorption and regeneration processes [17,30]. Moreover, these adsorbents have exhibited high kinetic performances with excellent high phosphate adsorption efficiency and capacity because the fibrous adsorbent possess short diffusion

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path and were able to remove phosphate at high feed flow rate [17]. In the monofunctional group such as phosphonic or phosphonate chelating resins/fibers, the metal ions immobilization is not high because of intraligand hydrogen bonding mechanism [31] and this behavior causes relatively low removal efficiency of arsenate and phosphate from water [32]. Intraligand cooperation is playing an important role for enhancing metal-ion affinities to polymer matrix phase. Introducing a highly hydrophilic sulfonate group into the polymer matrix can eliminate intraligand hydrogen bonding [32]. Therefore, several resins and fibers have been prepared containing bifunctional groups based on specific preparation procedure and thermal stability [17,33,34]. In the present study, we have developed bifunctional cation exchange fibers having both phosphonate and sulfonate groups where the precursory fiber was grafted with chloromethylstyrene only for high immobilization of Zr(IV) onto fiber phase [33]. We have successfully immobilized the high amount of Zr(IV) immobilization for arsenate removal [30]. However, the present study is completely different based on fiber characterization and experimental conditions. The sulfonate group has enhanced the accessibility of metal ions into polymer matrix and consequently higher metal immobilization [17,24,32].

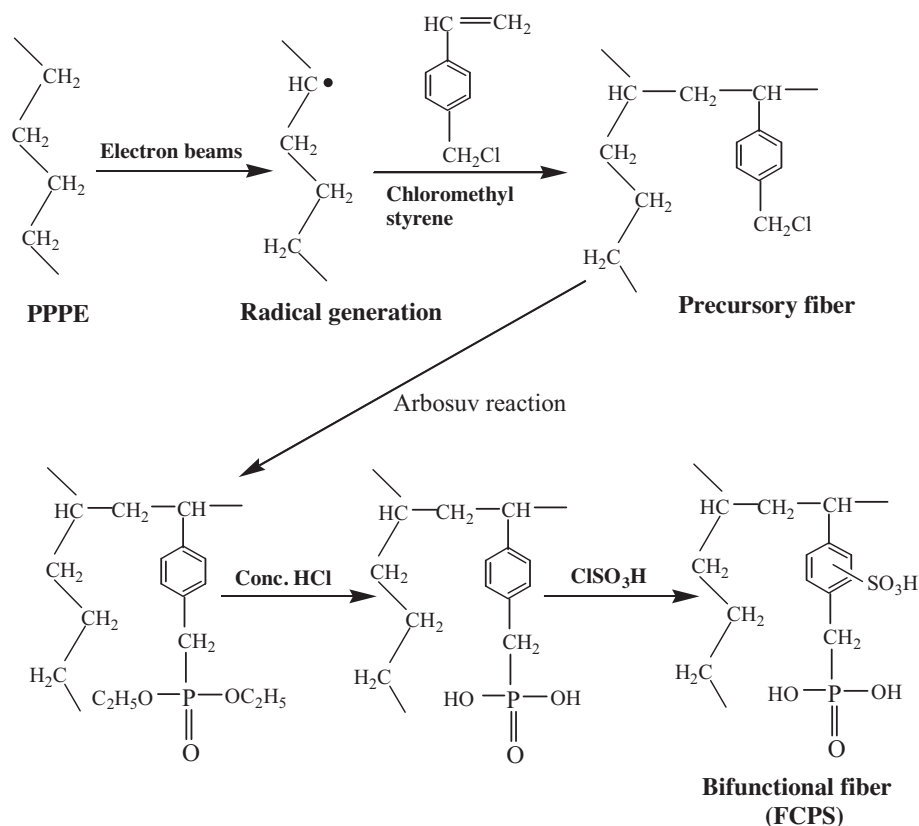
The bifunctional fiber has high affinity to hard Lewis acid cations such as Ti(IV), Fe(III) and Zr(IV). For example, Zr(IV) strongly binds to phosphonate group and make a very stable bond which remains in strong acid solution. It has been shown that Zr(IV) loaded bifunctional fiber can be efficiently used as a novel ligand exchanger for toxic anions removal from water [17,24]. Several researchers reported that Zr(IV) is ideal for ligand exchange adsorption for anions than Fe(III) [24,29]. The fixed-bed column adsorption processes are ideal candidates for reduction of dissolved phosphate to near-zero level and provide information on the long-term phosphate removal performance of the adsorbent. The main

objectives of this study were following folds: (i) to investigate high immobilization of Zr(IV) onto fiber phase for developing novel adsorbent to remove phosphate with high adsorption efficiency and capacity, (ii) to study the high kinetic performances by dynamic flow tests of column packed fibrous adsorbent to investigate the effect of pH, selectivity to phosphate over competing anions, initial concentration, feed flow rates, extremely high feed flow rate for trace phosphate removal, (iii) to evaluate competitive adsorption of arsenate and phosphate, (iv) to test the regeneration and the reuse of the synthesized adsorbent.

2. Materials and methods

2.1. Materials

All materials and chemicals were of analytical grade and used as purchased without further purification. Ultra-pure water prepared by a Milli-Q Academic-A10 (Nippon Millipore Co., Tokyo, Japan) was used throughout. $Zr(SO_4)_2 \cdot 4H_2O$, disodium hydrogenphosphate ($Na_2HPO_4 \cdot 2H_2O$) and disodium hydrogenarsenate ($Na_2HASO_4 \cdot 7H_2O$) are guaranteed grade and were obtained from Wako Pure Chemical Industries Ltd., Osaka, Japan. Sulfuric acid, sodium hydroxide, nitric acid and perchloric acid were also purchased from Wako Pure Chemical Industries Ltd., Osaka, Japan. Chloromethylstyrene was grafted onto polyethylene coated polypropylene precursory fiber by electron irradiation graft polymerization technique. The chloromethylstyrene was only grafted in this preparation from the stand point of high phosphorus content which may be suggested for high Zr(IV) immobilization and subsequently for high phosphate uptake. The major steps in the preparation of phosphonate and sulfonate group containing bifunctional cation exchange fiber (FCPS) are electron irradiation technique and followed by Arbusov reaction, chlorosulfonation



Scheme 1. Synthesis of bifunctional fiber containing both phosphonate and sulfonate groups (FCPS).

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