



## Performance of novel biopolymer-based activated carbon and resin on phosphate elimination from stream



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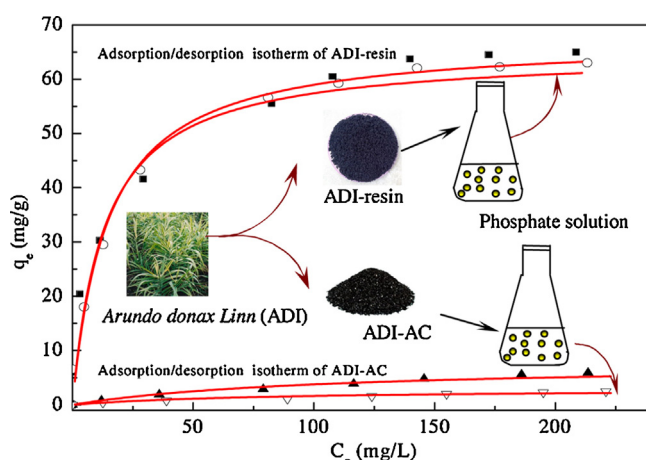
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### HIGHLIGHTS

- Novel biopolymer activated carbon and resin were prepared from *Arundo donax* Linn.
- Adsorption capacity of stratified column for phosphate was evaluated.
- Adsorption and desorption isotherm of phosphate by the biosorbents were determined.
- Raman spectra were used to evaluate the phosphate-laden samples.

### GRAPHICAL ABSTRACT



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### ABSTRACT

*Arundo donax* Linn-based resin (ADI-resin) and *Arundo donax* Linn-based activated carbon (ADI-AC) were prepared for phosphate elimination from stream. Characteristics (BET surface area, Zeta potential, SEM, FTIR, and Raman spectra) of the biosorbents indicated that some required functional groups have been grafted onto the ADI-resin. The regenerated ADI-resin achieved 92–94% of adsorption capacity even after seven cycles of adsorption and desorption. However, only about 15–25% of phosphate could be desorbed from the ADI-AC at the same desorption conditions. A stratification-packed column with different particle sizes of ADI-resin/ADI-AC was also designed. When the stratified resins with different particle sizes were introduced, the bed utilization of ADI-resin was greatly enhanced. In contrast, the stratified ADI-AC packed in column was not effective as the performance of stratified ADI-resin.

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## 1. Introduction

Eutrophication is considered as the most important water quality problem in freshwater and coastal ecosystems [1–3]. Both phosphate and nitrate are the emerging pollutants in agricultural, municipal, industrial, and mining wastewaters [4]. They can stimulate eutrophication, such as blue and green algae growth, and deterioration of water bodies, which leads to short-term and long-term environmental and esthetic problems [2,5,6]. It had been found that some blue–green algae have the ability of biological fixation of nitrogen, and planktonic  $N_2$ -fixing cyanobacteria bloom in freshwaters when phosphorus is abundant while nitrogen availability is low [2,4]. As a result, phosphorus control is more important for eutrophication control.

Various techniques, such as chemical precipitation, ion exchange, adsorption, biological methods, reverse osmosis, membrane, and constructed wetlands, have been studied for the removal of phosphate from wastewater [6–9]. These conventional techniques have their own inherent limitations such as less efficiency, sensitive operating conditions, production of secondary pollution. Of all these methods, adsorption and ion-exchange technologies are currently the most effective and commonly used technologies for phosphate removal from water [10,11]. A wide variety of commercial activated carbons and strong basic anion exchange resins with high selectivity of phosphate have been developed [12–14]. Although commercial activated carbons and resins are well tested and show the high capacity for phosphate removal in water, there are still some major obstacles to their applications in the field, such as high cost, no renewability, requirement of pre-concentration of anions, and disposal after use. Since 1990s, the utilization of low cost renewable organic materials for adsorption of phosphate has been explored [15,16]. Recently, attention has been diverted toward the economic and eco-friendly biomaterials such as byproducts or the wastes from large scale industrial operations and agricultural waste materials [17–19]. The major advantages of these biomaterials for the adsorption of anionic species include: unique chemical composition, availability in abundance, renewable, and low in cost [19].

The unique chemical compositions in these biomaterials have made them attractive in developing some novel biosorbents. Most of these biomaterials are the lignocellulosic materials containing large amounts of functional groups (e.g.,  $-OH$ ,  $-CHO$ ). As a result, some functionalization reactions (e.g., condensation, etherification, and polymerization) have been commonly applied for introducing some functional polymers [19–22]. In addition, carbon contributes 35–47% of contents in cellulose, hemicellulose, and lignin. As a result, carbonization process is also proceeded to enrich carbon content in the biomaterials by eliminating non-carbon species and produce the porous materials for adsorption [23].

In this work, the aquatic plant, *Arundo donax Linn*, was used as the starting materials for preparing two kinds of biosorbents, *Arundo donax Linn*-based resin (ADI-resin) and *Arundo donax Linn*-based activated carbon (ADI-AC). The physicochemical characteristics of ADI-resin and ADI-AC were measured by BET surface area, zeta potential, SEM, FTIR, and Raman spectra. The pH effect and adsorption/desorption isotherm on phosphate adsorption were evaluated. In addition, regeneration of the saturated ADI-resin and ADI-AC at different conditions was conducted. The column adsorption of phosphate by different particles sizes of ADI-resin and ADI-AC in stratified columns was also determined.

## 2. Materials and methods

### 2.1. Materials

*Arundo donax Linn* was obtained from Nansi Lake (Shandong, China). All chemicals were of analytical reagent grade and were

purchased from Tianjing Kermel chemical reagent Co., (China). The phosphate stock solution (1000 mg/L) was prepared by dissolving 1.4315 g of  $KH_2PO_4$  into 1 L of deionized water.

### 2.2. Preparation of ADI-resin and ADI-AC

The ADI-AC was prepared according to the method mentioned by Sun [24]. Dried *Arundo donax Linn* was mixed with 45%  $H_4P_2O_7$  at an impregnation ratio of 3:4 ( $H_4P_2O_7$ : *Arundo donax Linn*), followed by activating at 400 °C at a heating rate 10 °C/min. After cooling down to room temperature, the activated carbons were washed with tap water, hydrochloric acid, and distilled water for removal of residual  $H_4P_2O_7$ . The wet ADI-AC was then dried at 105 °C for 24 h and sieved.

The ADI-resin was prepared by a cross-linking reaction (Fig. 1). A total of 10 g of natural *Arundo donax Linn* was mixed with 40 ml of epichlorohydrin and 40 ml of N,N-dimethylformamide in a 500 ml of three-neck round bottom flask at 90 °C for 1 h. Thereafter, 20 ml of catalyst (pyridine) was added and the mixture was stirred for 1 h at 90 °C, followed by adding 30 ml of 99% triethylamine (w/w) with stirring for 3 h (90 °C). The primary product was washed with 500 ml of distilled water and then was dried at 60 °C for 12 h and sieved to particles with diameter less than 5000  $\mu m$ .

### 2.3. Physicochemical measurement of ADI-AC and ADI-resin

The pore structure of the samples, including the surface area ( $S_{BET}$ ), the total pore volume ( $V_{tot}$ ), the average pore diameter ( $D_p$ ) were determined from nitrogen adsorption/desorption isotherms at 77 K by using a surface area analyzer (JW-BK122 W, China). The number of the acidic and basic functional groups was calculated under the assumption that NaOH neutralizes acidic functional groups and HCl neutralizes basic functional groups.

The surface morphologies of the two biosorbents were characterized using a scanning electron microscope (Hitachi S4800, Japan). The natural and spent activated carbons/resin samples as well as solid  $Na_2HPO_4$  were analyzed with FTIR spectroscopy (PerkinElmer “Spectrum BX” spectrometer). The samples were mixed with powdered KBr and pressed into pellet for analysis. FTIR spectra were recorded from 4000 to 400  $cm^{-1}$ .

The zeta potentials of clean and phosphate-loaded ADI-AC and ADI-resin were measured at different pH conditions using a microelectrophoresis apparatus (JS94H, Shanghai Zhongchen Digital Technical Apparatus Co., Ltd, China). The clean samples were prepared in 50 ml suspensions containing 0.1% NaCl and 0.2 g powdered ADI-AC or ADI-resin. The initial pH of the suspensions was adjusted between 2.0 and 12.0 with mixing for 24 h. Thereafter, the zeta potentials of the suspensions and final pH of the suspensions were measured. The phosphate-loaded adsorbent samples were prepared by mixing 0.2 g powdered ADI-AC or ADI-resin with 15 or 100 mg/L of phosphate solutions (50 ml, pH: 2.0–12.0). The phosphate uptake capacities and equilibrium pH at respect pH conditions were also evaluated.

### 2.4. Adsorption tests

In the batch equilibrium experiments, 0.2 g ADI-AC/ADI-resin were added to a set of 100 ml Erlenmeyer flasks containing 50 ml phosphate solutions (pH: 6.0) with concentrations range of 10–250 mg/L for ADI-AC, and 50–800 mg/L for ADI-resin. The residual phosphate was determined at the wavelengths of UV-maximum ( $\lambda_{max}$ ) at 700 nm by ammonium molybdate spectrophotometric method through a UV-visible spectrophotometer (model UV754GD, Shanghai). After the adsorption isotherm experiments, desorption isotherm was also studied according to the method mentioned by Yoon [22]. It was conducted by removing the clear

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