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Alginate-based attapulgite foams as efficient and recyclable adsorbents for the removal of heavy metals

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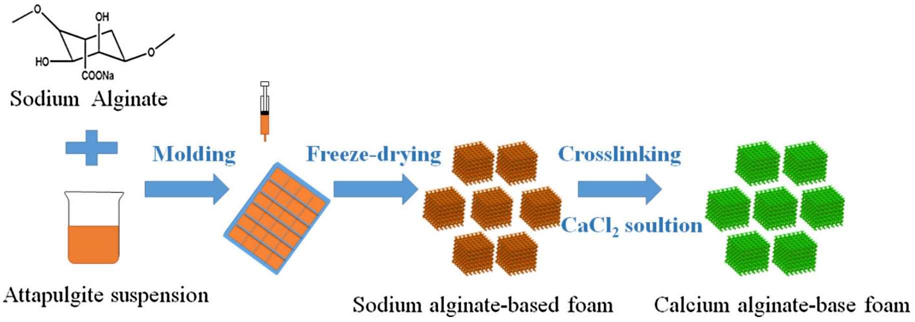
GRAPHICAL ABSTRACT



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ABSTRACT

Floatable and porous foam adsorbents constructed by encapsulating attapulgite (ATP) in sodium alginate (SA) were fabricated via a freeze-drying and post cross-linking method, and both attapulgite and sodium alginate possessed adsorptive sites. These adsorbents were characterized by XRD, FTIR, and SEM to investigate their crystal structures, surface properties, size and morphology. In the adsorption tests, the adsorption capacity was derived from the Langmuir isotherm model, and the maximal adsorption capacity of as-prepared adsorbents was 119.0 mg g^{-1} for Cu(II) and 160.0 mg g^{-1} for Cd(II). In addition to the remarkable adsorptive performances, these adsorbents presented strong chemical stability and were readily recyclable because of their floatability in water solution. These aforementioned advantages highlight that the alginate-encapsulated attapulgite foams are potential scalable adsorbents for heavy metal ions removal from polluted water, and such a structure design could intrigue the development of novel adsorptive materials.

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1. Introduction

Water contaminants including heavy metal ions severely jeopardize animals, plants and human beings due to their non-biodegradability and bioaccumulation [1,2]. Various methods have been exploited to remove harmful metal ions through such as membrane separation [3], ion-exchange [4], adsorption [5–7] and chemical precipitation [8]. Among these applied methods, adsorption is a promising technique because of its high efficiency and easy operation [9]. For instance, Cui et al. developed polyaniline/attapulgite composites to remove Hg(II) with the maximum adsorption capacity being over 800 mg g^{-1} [10]. Kim et al. fabricated zeolite-like materials of AMH-3 and they exhibited the maximal adsorption capacity of 93.4 mg g^{-1} for Cu(II), 182.2 mg g^{-1} for Cd(II), and 238.7 mg g^{-1} for Pd(II) [11]. Although these composite adsorbents exhibited high adsorption capacities, they are powdery and thus difficult to separate and recycle in practical applications.

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Moreover, they could cause secondary pollution due to leaching or leakage especially when applied in continuous-flow streams. To combat these problems, floatable and porous attapulgite/polyethersulfone beads were developed in our previous study for the removal of Cu(II) and Cd(II), and the maximum adsorption capacity was 25.3 mg g^{-1} for Cu(II) and 32.7 mg g^{-1} for Cd(II) [6]. However, the adsorption capacity is difficult to further improve owing to the chemical inertness of the polyethersulfone employed, and thus novel adsorbent structures or adsorptive materials are highly demanding.

Sodium alginate (SA) is a renewable natural polysaccharide containing abundant functional groups (e.g. $—COO^-$) [12,13], SA has been applied as green adsorbents for heavy metal ions removal [14–16]. Chen et al. prepared Cu(II)-imprinted SA-based porous film adsorbents for Cu(II) removal and the maximal uptake of 79.3 mg g^{-1} was achieved [17]. Wang et al. synthesized phosphate-embedded calcium alginate beads for the adsorption of Cd(II) and the maximum capacity was 82.6 mg g^{-1} [18]. However, the mechanical weakness [19] and the high cost of pure SA (compared with natural clay) [20] significantly limit large-scale utilization of SA for treating wastewater. To enhance the mechanical strength and reduce the cost, attapulgite (ATP) was utilized to combine their both advantages as adsorbents. ATP is a typical phyllosilicate that consists of two Si—O tetrahedron layers and one (Al, Mg, Fe)—O—OH octahedron layer [21]. Moreover, it is abundant, nontoxic, low-cost and has cation exchange properties and large surface area [6,22], and thus has been used for metal ions and dyes removal in the last decade [23,24]. However, dye adsorption is irreversible due to the interaction between dye molecules and clay with an extraordinarily high stability against acids, alkalis, organic solvents, and UV irradiation [25–27]. Introducing ATP into sodium alginate polymer matrix would exert the capacity of both components, and reduce the cost of adsorbent and minimize the loss of attapulgite in the practical application.

In this work, floatable and porous alginate-based ATP foams were developed by a freeze-drying and post cross-linking method. SA was utilized as supports to immobilize ATP and to minimize its loss, and ATP is uniformly dispersed in the sodium alginate polymer matrix, which is favorable to improve mechanical properties of polymer [28]. In the adsorption tests, the low density of the porous foams makes them floatable and thus convenient to recycle. The adsorption performances as to heavy metal ions were investigated under various conditions, including different ratios of ATP/SA and pH values, and the adsorption process and mechanism were also explored by adsorption isotherm and kinetics.

2. Experimental section

2.1. Chemicals

Sodium alginate (SA, analytical reagents) and cadmium diacetate dihydrate ($Cd(CH_3COO)_2 \cdot 2H_2O$, chemical reagents) were purchased from Sinopharm Chemical Reagent Co. Ltd., China. Attapulgite was obtained from Xuyi, Jiangsu, China. Anhydrous calcium chloride ($CaCl_2$, analytical reagents) was purchased from Xilong Scientific Co., Ltd., China. Sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$, analytical reagents) was obtained from Shanghai Xinbao Fine Chemical Factory. Hydrochloric acid (HCl, analytical reagents) and sodium hydroxide (NaOH, analytical reagents) were obtained from Nanjing Chemical Reagent Co. Ltd., China.

2.2. Preparation of alginate/attapulgite foams

Alginate-based ATP foams were prepared, following the process as shown in Fig. 1. 0.25–0.5 g of ATP was dispersed in 25 ml of deionized water to form clay suspension under vigorous stirring. 1 g of SA was then added into the clay suspension. After complete dissolution of SA, the mixed slurry was injected into a square mould with the size of $1 \times 1 \times 1$ cm^3 by a syringe, followed by freeze-drying treatment overnight. The as-obtained foams (SP-0.25(Na) and SP-0.5(Na)) were impregnated in 40 ml of $CaCl_2$ solution (5 wt%) and kept for 4 h to crosslink completely at ambient temperature owing to excessive Ca^{2+} [29]. The resulting foams were obtained after washing and then dried in an oven at 55 °C. The samples prepared with different weight ratios of ATP/SA were denoted as SP-0.25, SP-0.5. Pure SA foams (SP-0(Na) and SP-0) were prepared by the same method using no ATP.

2.3. Characterizations

X-ray diffraction (XRD) patterns of the samples were recorded using a Rigaku Ultima IV diffractometer (Rigaku, Japan) with Cu Kα radiation at a scan rate of 5°/min from 5 to 50° (2θ). Fourier transform infrared spectra (FTIR) was used to detect the surface functional groups by a FTIR spectrophotometer (Thermo Electron Nicolet-360, USA) using the KBr wafer technique. The morphology of the composite materials was examined by scanning electron microscopy (SEM) utilizing a JSM-7600F (JEOL Ltd., Japan) with an operating voltage of 30 kV. Elemental analysis was performed using energy-dispersive X-ray spectroscopy (EDX) attached to the microscope. pH meter (PHS-3E, Shanghai INESA Scientific Instrument CO., Ltd) was used to measure pH values. The concentration of Cu(II) and Cd(II) was determined using an atomic absorption spectrometer (PinAAcle 900F, American PerkinElmer).

2.4. Adsorption experiments

The concentration of metal ions was determined by an atomic absorption spectrometer. The adsorption capacity of SP-0, SP-0.25 and SP-0.5 was calculated according to the Eq. (1):

$$q_e = (C_0 - C_e)V/m \tag{1}$$

where q_e is the adsorption capacity (mg g^{-1}) at equilibrium, C_0 is the initial concentration of Cu(II) or Cd(II) (mg g^{-1}) and C_e is the concentration of Cu(II) or Cd(II) at equilibrium (mg L^{-1}). V is the volume of solution (mL), and m is the mass of the adsorbent: SP-0, SP-0.25 and SP-0.5 (mg). The pH value of the metal ions solution was adjusted by 0.1 mol L^{-1} HCl or 0.1 mol L^{-1} NaOH aqueous solution.

The adsorption isotherm study was carried out by adding 40 mg of SP-0.25 to 40 L of Cu(II) or Cd(II) solution, with the initial concentration of metal ions being 100–500 mg L^{-1}. The experiments were carried out at ambient temperature with the magnetic stirring speed of 250 rpm. In order to further understand the adsorption process, the experimental adsorption data were simulated according to the Langmuir (Eq. (2)) and Freundlich (Eq. (3)) isotherm model:

$$C_e/q_e = C_e/q_m + 1/(K_L q_m) \tag{2}$$

$$\ln q_e = \ln K_F + (1/n)\ln C_e \tag{3}$$

where q_e (mg g^{-1}) represents the adsorption capacity at equilibrium, q_m (mg g^{-1}) stands for the maximum adsorption capacity, C_e (mg L^{-1}) is the aqueous concentration in Cu(II) and Cd(II) at equilibrium. K_L (L mg^{-1}) and K_F (mg g^{-1}) are the Langmuir equilibrium and Freundlich equilibrium constants, respectively. The value of n is related to adsorption intensity [20].

The adsorption kinetics experiments were performed as well. Typically, 40 mg of SP-0.25 was added in 40 ml of the tested solution with the initial Cu(II) or Cd(II) concentration of 250 mg L^{-1}, and the contact time varied from 0.5 to 24 h. The kinetics results

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