

Electrochemical removal of lead ions using paper electrode of polyaniline/attapulgitite composites



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ABSTRACT

Polyaniline/attapulgitite composites (PANI/ATP) were facilely synthesized via in situ oxidative polymerization of aniline on the surface of ATP. The structure of the resulting composites was characterized by scanning electron microscopy (SEM) and FT-IR spectra. The PANI/ATP was coated on a piece of stiff paperboard to construct a paper electrode, and the electrosorption of Pb(II) was carried out by polarizing the PANI/ATP paper electrode at the potential of -0.3 V. The removal of Pb(II) was mainly attributed to the synergistic effect of physisorption, chemisorption (chelation) and electrostatic interaction, and the effect of electrostatic interaction and chelation is more significant than that of physisorption. Batch experiments were conducted to investigate the effect of applied potential, mass ratio of aniline to ATP, initial concentration, and contact time on the removal of Pb(II). The Freundlich isotherm model fitted the experimental data significantly, and the adsorption kinetics was well described by the pseudo-second-order model. Moreover, electrodesorption experiments were performed to reveal the feasibility for the regeneration of the PANI/ATP paper electrode. All the results indicated that the PANI/ATP could be a promising material for Pb(II) removal.

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1. Introduction

Heavy metals are nonbiodegradable and persistent environmental contaminants that are detrimental to the environment and public health [1]. Pb(II), one of the high priority toxic pollutants, can reach the environment from natural geochemical processes as well as from various process industries, such as acid battery manufacturing, metal plating and finishing, tetraethyl lead manufacturing and ceramic and glass industries [2,3]. As a consequence, it is often found in waters and wastewaters arising from these processes. Therefore, the removal of Pb(II) from effluents appears to be an important and urgent task. Till now, many methods have been proposed for eliminating Pb(II) contaminant, such as chemical precipitation, reverse osmosis, ion exchange, solvent extraction, liquid membrane transport and adsorption methods [4–6].

Recently, electrosorption has been regarded as a novel technique for removing dissolved inorganic contaminants such as radionuclides [7], metal ions [8], and anions [9] from aqueous

solutions. It is generally a potentially induced adsorption onto the surface of charged electrodes, and the charged ions in the aqueous solutions can move toward the surface of the oppositely charged electrode by imposing an electric field, i.e., electrostatic interaction plays a key role during the electrosorption process. After the electric field is removed, the ions will soon withdraw from the electrode surface to the bulk solution, and this provides a possible strategy for electrode regeneration. Various carbon materials of good electrical conductivity and high specific surface area (SSA) are the conventional electrodes for electrosorption, such as activated carbon [10], carbon nanotubes and carbon nanofibres composite [8,11], and carbon aerogel [9,12]. Especially, carbon aerogel is an excellent electrode material studied extensively for electrosorption on account of its high porosity and SSA, low electrical resistivity, and controllable pore size distribution [13–15].

As a kind of functional material, conducting polymers (CPs) have attracted more and more attention because of their unique properties, such as ease of synthesis, high stability under ambient conditions, and variable conductivity [16–18]. In particular, the heteroatoms on the chains of CPs, e.g., N atom in polyaniline (PANI) and polypyrrole, S atom in polythiophene, and so on, play an important role in the removal of heavy metal ions by chelation [19,20]. Recently, the electrosorption of Cu(II) onto

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poly(*m*-phenylenediamine) (PmPD) paper electrode for wastewater purification has been reported in our group [21]. The results indicate that the PmPD is an excellent electrode material, and the equilibrium adsorption capacity (q_e) of the PmPD paper electrode can reach high up to 123 mg g^{-1} , which is much higher than that of the activated carbon electrode. Furthermore, the PmPD electrode can undergo ten successive electrosorption/electrodesorption cycles without the loss in the ability of Cu (II) removal.

As we know, CPs usually have quite small SSA, and thus it is a pity that physisorption contributes little to the electrosorption of toxic metal ions by CPs, i.e., only electrostatic interaction and chelation are involved in this process. It is reasonable that if materials with large SSA are introduced into CPs to construct a composite electrode, a synergistic effect of physisorption, chelation and electrostatic interaction is expected in the removal of heavy metal ions using the composite electrode. Attapulgite (ATP), also known as palygorskite, is a natural cationic clay mineral with a large SSA (determined to be $119 \text{ m}^2 \text{ g}^{-1}$ in our group) [22], and it has drawn increasing attention as a high-efficient adsorbent for the adsorption of heavy metal ions [23–25]. In fact, the adsorption of Cu(II), Ni(II), Cd(II) and Cr(VI) with the CPs/ATP composites has been investigated in our group [26,27]. However, as far as we are aware, the electrosorption of heavy metal ions with the CPs/ATP composites has never been reported.

In the present work, PANI/ATP composites are facilely synthesized via in situ oxidative polymerization of aniline on the surface of ATP, and the as-prepared PANI/ATP is coated on a piece of stiff paperboard to construct a paper electrode for the electrosorption of Pb(II). The PANI/ATP combines the advantages from the individual components (PANI and ATP), and therefore, physisorption, chelation and electrostatic interaction can all contribute to the removal of Pb(II). The important parameters influencing the electrosorption of the PANI/ATP paper electrode as well as adsorption isotherms and adsorption kinetics are discussed in detail in the present work. Also, the structure variations of the PANI/ATP before and after its polarization are revealed by the FT-IR spectra.

2. Experimental

2.1. Materials and reagents

Aniline (>99.5%), polyvinyl alcohol (PVA) and ammonium persulfate (APS) of analytical grade were purchased from Sinopharm Chemical Reagent Company (Shanghai, China). Aniline was distilled under reduced pressure before use. ATP was supplied by Jiangsu NDZ Technology Company (Changzhou, China) and washed with doubly distilled water to remove impurities and dried at room temperature. All the other reagents were of analytical grade.

2.2. Fabrication of PANI/ATP paper electrode

The PANI/ATP composites were synthesized via in situ oxidative polymerization as reported previously [26,28]. Here, ATP was adopted as the substrate for in situ polymerization of aniline. The specified synthetic procedures were as follows: a certain amount of ATP and 7.3 mL of aniline were dispersed in 100 mL doubly distilled water and 100 mL of 2.0 mM HCL aqueous solution was added in the resulted dispersion. The mixture was continuously stirred for 30 min. Then 100 mL of 0.4 M APS aqueous solution was added into the mixture dropwise, and the polymerization reaction was carried out in an ice-water bath for 4 h with agitation. The same amount of APS solution was added dropwise again, and the reaction was done for another 4 h. The mixture was placed overnight after the stirring was stopped, and then filtered. The resulting dark green PANI/ATP composites were washed with

anhydrous ethanol and doubly distilled water several times, respectively, until the filtrate became colorless. Finally, the product was dried at 60°C for 24 h and then it was stored in a desiccator ready for standby application. For comparison, PANI was synthesized according to the same procedures without ATP addition.

1.0 g of the PANI/ATP composites was dispersed in 4.0 mL of 4.0 wt.% PVA aqueous solution under ultrasonication, and then 50 mg of the resulting dispersion was coated on both sides of a piece of stiff paperboard about $400 \mu\text{m}$ in thickness of $35 \text{ mm} \times 8 \text{ mm}$ size (500 g m^{-2} of basic weight, supplied by General Mills Co., Ltd. (China)). PVA acts as a binding agent in this work, and the final product was dried at 60°C for 4 h to construct the PANI/ATP paper electrode.

2.3. Electrosorption experiments of Pb(II)

A stock solution of Pb(II) (2.5 mM) was prepared from analytical grade $\text{Pb}(\text{NO}_3)_2$ using doubly distilled water and was serially diluted to prepare solutions of varying initial concentrations for the experimental purpose. Electrosorption of Pb(II) was carried out by a CHI 660D Electrochemical Workstation (Beijing, China) in a conventional three-electrode system. The PANI/ATP paper electrode and a platinum foil was used as the working electrode (WE) and the counter electrode (CE), and a saturated calomel electrode (SCE) was used, respectively as the reference electrode (RE) to keep the stability of the applied potential. The experiments were performed at room temperature. The sketch diagram of the experimental setup is shown in Fig. 1.

2.4. Characterization

The surface morphologies of ATP, PANI and PANI/ATP were recorded by a JSM-6360LA scanning electron microscope (SEM) (JEOL, Japan). The FT-IR spectra of different PANI/ATP samples were measured on a Nicolet FTIR-8400S spectrophotometer (Shimadzu, Japan) using KBr pellets. The concentration variation of Pb(II) was monitored by using a digital DDS-11A conductivity meter (Shanghai Rex Xinjing Instrument Co., Ltd., China). The relationship between conductivity and Pb(II) concentration was obtained by a calibration plot made prior to the experiment. The efficiency of electrosorption can be calculated by using the following equation: $\%R = C_0 - C_e / C_0 \times 100$, where $\%R$, C_0 and C_e are the removal ratio, the initial and the equilibrium concentration of Pb(II), respectively.

3. Results and discussion

3.1. Characterization of PANI/ATP

The SEM images of purified ATP, PANI and PANI/ATP are shown in Fig. 2. As can be seen, ATP presents a nano-fibrillar appearance, and PANI bears a porous and cauliflower-shaped microstructure. The PANI particles with a diameter of about $0.5 \mu\text{m}$ aggregate compactly. However, the morphologies of the PANI/ATP composites are quite different from that of ATP or PANI. As shown in

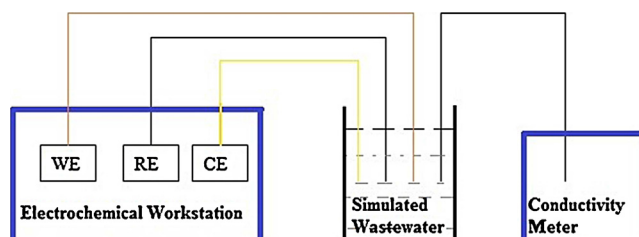


Fig. 1. The sketch diagram of the experimental setup.

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