



Lead sorption onto acrylamide modified titanium nanocomposite from aqueous media



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ABSTRACT

The purpose of this study was to investigate the adsorption of lead (Pb(II)) onto acrylamide (AM) doped TiO₂ nanocomposites (Ti-AM) using batch techniques for evaluation of isothermal and kinetic properties. Chemical, structural and textural characteristics of the material were determined by FTIR, XPS, XRD, SEM and EDAX analysis. XPS results showed that a change in oxidation state occurred due to lead adsorption. The adsorption conditions for the adsorbent were optimized by varying several experimental parameters, i.e., contact time, initial lead concentration, adsorbent dose, pH, and electrolyte amount of the solution. The adsorption data were modeled using both the Langmuir and Freundlich isotherms. The maximum adsorption capacity using Langmuir isotherm (q_{max}) for the nanocomposite was found to be 476.19 mg g⁻¹. Adsorption showed pseudo-second-order kinetics with a rate constant of 8.7×10^{-4} and 1.2×10^{-4} g mg⁻¹min⁻¹ at 100 and 500 mg L⁻¹ initial Pb(II) concentrations, respectively. Acrylamide concentration in nanocomposite synthesis up to 1 g had greater influence on the sorption of lead. The most favorable pH for the adsorption was pH 5.5. With increasing concentrations of three electrolytes (NaCl, Na₂SO₄ and Na₂CO₃) from 0.01 to 1.0 M Pb(II), the lead removal decreases from 76.3 to 32.8 mg g⁻¹, from 97.4 to 68.7 mg g⁻¹, and from 98.8 to 72.5 mg g⁻¹, respectively. Further, the Ti-AM nanocomposite is amenable to efficient regeneration by a 0.05 N HCl solution for repeated (up to six cycles) use without any significant capacity loss, making this approach very economical.

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1. Introduction

Adsorption behavior of lead ions from aqueous media has become of great importance in the last decade. Among the heavy metal ions, Pb(II) is of particular interest since it is toxic and not biodegradable, tends to accumulate in vital organs, and causes various diseases and disorders in humans and animals as a result of long-term exposure (Benhimaa et al., 2008; Jiang et al., 2012). Lead is frequently used in industrial processes such as electroplating, mining, extracting, printing, painting, textile and battery manufacturing, tanning, metallurgy activities, metal processing and others (Pimentel et al., 2008; Googerdchian et al., 2012). The high toxicity of lead ions leads to serious ecological damage, thus, there is an increasing necessity to develop processes to eliminate or at least to greatly reduce the concentration in wastewater prior to release into the environment.

As per the World Health Organization (WHO), the maximum allowed concentration value for lead is 0.05 mg/l (Singh et al., 2007)

in drinking water and other types of waters. Many physicochemical treatment processes have been developed to remove heavy metal ions from industrial effluents. These include reverse osmosis, ion exchange, electrodialysis, evaporation, chemical precipitation, flocculation, membrane separation and chelation. Most often, these methods have high reagent requirements, generate secondary pollutants or are simply inefficient and particularly require high cost in removing toxic metal ions in trace quantities from solutions (Fu and Wang, 2011; Ghorai et al., 2012). Above all the various methods, adsorption has been recognized as one of the most economical (as compared to ion exchange, extraction or electrodialysis methods) and effective methods for the removal of the lead from aqueous media due to flexibility in design and operation (technically easy, as compared chemical precipitation or reverse osmosis methods) offered by the adsorption process (Okoye et al., 2010; Hao et al., 2012).

Adsorption using different polymeric materials and metal oxide sorbents is the method of choice in many wastewater treatment processes for removing metals from chemical industries in some developed countries (Akkaya and Ulusoy, 2008; Pan et al., 2009; Ulusoy and Akkaya, 2009; Ngah and Fatinathan, 2010). Many researchers have reported on the use of hydrous metal

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oxides as adsorbents for the removal of lead ion from aqueous media (Hua et al., 2012). Titanium dioxide has been intensively investigated as a potential sorbent due to its high chemical stability in the pH range 2–14 and because the process is simple with a fast rate of adsorption and desorption (Samarghandi et al., 2007; Li et al., 2010a,b; Tehrani et al., 2011). In addition, nano-size titanium dioxide possesses many unique features, such as high surface area, more surface atoms, high surface reactivity, unique catalytic activity and high suspension stability when compared to larger size particles (Jia et al., 2009). Titanium dioxide nanoparticles (Hu and Shipley, 2012) and amorphous titanium phosphate (Jia et al., 2008) have high potential for lead ion adsorption. Therefore, research has been conducted recently to include surface modification of TiO₂ to enhance the adsorption capacity of TiO₂. Modification of the TiO₂ surface with polymer, due to the combined characteristics of titanium and polymer, is potentially an even more attractive candidate to be used as an adsorbent, as their combined properties represent an improvement upon the thermal, mechanical, and porosity properties as compared to the homologous characteristics of the individual organic and inorganic components (Sanchez et al., 2001). Polymers such as, cellulose microspheres, polystyrene, polyethylene oxide (Nagaoka et al., 2002; Zan et al., 2004; Sung et al., 2006) and chitosan (Chena et al., 2012) used as supports would have some advantages including low cost and easy separation from reaction solution. Acrylamide (AM) is a well known water soluble polymer containing large numbers of amide groups; the hydrophobically associating cationic polyacrylamide, one kind of water-soluble hydrophobically associating copolymers, polymerized by cationic monomer, hydrophobic monomer and acrylamide (Yang et al., 2010). Also, AM has been widely grafted to various adsorbent materials; polymerization occurs only in supersaturated waters with respect to silica (Demadis and Stathoulopoulou, 2006). Acrylamide based materials are efficient adsorbents for the removal of toxic metal ions (Rivas et al., 2001; Li et al., 2005) and organic pollutants (Meng et al., 2005). The adsorption efficiency of materials can be greatly enhanced after modification with AM. The properties of the functional groups (i.e., active site types, and amide group, etc.) are very important for the adsorption capacity of adsorbents. In order to evaluate and to compare the effects of functional groups and active site type on the adsorption capacity of materials, the same content of functional groups on the surfaces of the adsorbent are required.

The major aim of the present study is to explore the adsorption behavior of Pb(II) cation from aqueous media onto acrylamide-TiO₂ using IR, XRD, XPS and SEM/EDX analysis. In particular, this study is aimed to examine the effects of operational parameters, such as contact time, initial pH of the ionic solution and initial concentration of ions to optimize the adsorption capacity. Also, the equilibrium adsorption data have been evaluated by adsorption isotherms (Langmuir and Freundlich), and kinetics parameters are determined.

2. Materials and methods

2.1. Materials

All the chemicals used in this study were of analytical reagent grade and were utilized as received without further purification. Titanium (IV) isopropoxide in the liquid form and potassium persulfate as radical initiator for the synthetic reaction of acrylamide (AM) were obtained from Daejung Chemical and Metals Co. LTD., South Korea. Milli-Q deionized water was used throughout the study. Aqueous solutions of lead nitrate were prepared by dissolving Pb(NO₃)₂ (Merck) in deionized water.

2.2. Synthesis of Acrylamide-TiO₂ (Ti-AM) composites

Nano titanium was synthesized by sol-gel method (Kallel et al., 2009). In-situ doped acrylamide titanium nanocomposite (Ti-AM) was synthesized by a sol-gel process, where acrylamide monomer was doped into nano titanium during the sol-gel process. Afterward, the reaction was completed with initiator by polymerization and the products were characterized as described earlier. Acrylamide monomer (0.1, 0.2, 0.5, 1, 2 g) was dissolved in distilled water (120 mL) at ambient temperature. The typical synthesis procedures for nano TiO₂ and acrylamide doped nano-TiO₂ are as follows. Titanium(IV) isopropoxide (5.58 mL) was hydrolyzed using glacial acetic acid (10.74 mL) at 35 °C. To this solution 120 mL acrylamide/water was added drop-wise under vigorous stirring for 30 min. Acrylamide-TiO₂ solution was stirred and heated to 70 °C in a water bath for 15 min and initiator potassium peroxydisulfate (0.1 g) was added. The mixture was kept stirring for 1 h longer until a clear solution of acrylamide-TiO₂ nanocrystals was formed. The prepared solution was kept in the dark for the nucleation process for 14 h. After this period, the solution was placed in an oven at a temperature of 85 °C for a period of 14 h for the gelation process. The gel was then dried at 120 °C and subsequently the nanocomposite was crushed into fine powder.

2.3. Characterization of the composites

The experimental data were corrected for the background absorption of the solvent. Infrared spectra (Perkin-Elmer infrared spectrophotometer) were carried out on Nicolet Nexus 470 FTIR spectrometer in the region 3800–500 cm⁻¹. For this, the samples were prepared in the form of pellets with KBr at ambient conditions and were measured in transmission mode. Moreover, nano-TiO₂ and acrylamide-TiO₂ surfaces were characterized by X-ray photoelectron spectroscopy (XPS) using a Thermo Scientific K-Alpha XPS spectrometer to determine the superficial change on adsorbents before and after adsorption at pH 5.5 when the lead adsorption amount was relatively higher than nano titanium. The X-ray diffraction (XRD) patterns were recorded on a Bruker AXN in the 2θ range of 10–80° using Cu-Kα radiation (λ = 1.5418 Å). The diffraction data at a 0.02° step were collected at room temperature (T = 296 K). The accelerating voltage and the applied current were 40 kV and 30 mA, respectively. Semi-quantitative analyses were performed on an energy-dispersive X-ray (EDX) spectrometer connected to a Hitachi S-4700 scanning electron microscope (SEM).

2.4. Lead quantification

Dissolved lead was determined using a Varian AA240 Atomic Absorption Spectrophotometer (AAS) using an air-acetylene flame at a wavelength of 283.3 nm using a slit width of 0.7 nm. Experimental samples were filtered using Whatman 0.45 mm filter paper and the filtrates after suitable dilutions were analyzed. This is water sample so no need to acid digestion because during adsorption pH is 5.5 (Sing et al., 2007). However AAS analysis is used to calculate the initial and final concentration of the lead ions from the aqueous medium. Control experiments showed that no sorption occurred on either glassware or filtration systems. All assays were carried out in triplicate and only mean values are presented.

2.5. Adsorption in batch system

Adsorption experiments were carried out under optimized conditions using the composite as an adsorbent in a temperature controlled incubator shaker set at 120 rpm maintained at 30 °C for 1.5 h.

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