ARTICLE IN PRESS

Arabian Journal of Chemistry (2015) xxx, xxx–xxx

King Saud University

Arabian Journal of Chemistry

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ORIGINAL ARTICLE

Efficient treatment of lead-containing wastewater by hydroxyapatite/chitosan nanostructures

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Received 12 June 2014; accepted 14 December 2014

KEYWORDS

Adsorption isotherm;
Wastewater treatment;
Lead removal;
Hydroxyapatite;
Chitosan

Abstract The development of hydroxyapatite nanorods (nHAp) and hydroxyapatite/chitosan nanocomposite (nHApCs) was sought as potential sorbents for the removal of lead ions from aqueous lead-containing solutions in a batch adsorption experiment. The high resolution transmission electron microscopy, energy dispersive X-ray analysis, X-ray diffraction, Fourier transform infrared spectrophotometry and Zeta potential measurements were all combined to reveal the morphology, composition, crystal structure, functionality and stability of the prepared sorbents. The equilibrium concentration of Pb^{2+} ions was identified by the atomic absorption spectrophotometry. The kinetics of the sorption process was investigated together with the influence of initial lead ions concentration, sorbent dosage and solution pH on the sorption capacity. The sorption process followed pseudo-second-order kinetics, where 20 min was quite enough to attain equilibrium. Two models of adsorption isotherms (Freundlich and Langmuir) were employed to correlate the data in order to understand the adsorption mechanism. Interestingly, in one of the experiments, for a 200 mL solution (pH = 5.6) containing 100 ppm lead ions, a sorbent dosage of 0.4 g nHAp could achieve a complete removal for lead ions. However, typically, the sorption capacities of nHAp and nHApCs to lead ions were 180 and 190 mg/g respectively, which appear excellent for lead removal.

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Peer review under responsibility of King Saud University.

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1. Introduction

The violent spreading of kidney and liver failure, cancer as well as the common waterborne diseases such as the bacterial diarrhea, hepatitis, and typhoid fever has called for a sincere urgent treatment for wastewater emerged from sewages, industrial effluents and stormwater before draining into lakes, groundwater and/or rivers. Unfortunately, the existence of large dosages of heavy metals in these effluents represents

http://dx.doi.org/10.1016/j.arabjc.2014.12.016
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Please cite this article in press as: Mohammad, A.M. et al., Efficient treatment of lead-containing wastewater by hydroxyapatite/chitosan nanostructures. Arabian Journal of Chemistry (2015), http://dx.doi.org/10.1016/j.arabjc.2014.12.016

the greatest challenge in water purification, where exceeding the allowed concentration limit of these metals in the human body can end up with acute or chronic death. Of these heavy metals, lead is recognized as longstanding environment contaminant. It is released into the atmosphere from mining industries and fossil fuel burning, and has long been involved in the production of batteries, ammunition, metal products, and devices to shield X-rays (Mudipalli, 2007). Like other heavy metals, lead may contaminate surface and drinking water from industrial effluents, and the corrosion of household plumbing systems, respectively, which ultimately makes drinking risky (Hammer and Mark, 1996). Lead affects the central nervous system and inhibits the ability to synthesize red blood cells. Blood concentrations of lead above 40 μg/dL can produce observable clinical symptoms in domestic animals. It can also cause miscarriages and subtle abortions. According to the US environmental protection agency (EPA), a diet of 2–8 mg of lead per kilogram of bodyweight per day over a period of time will cause death (Mudipalli, 2007). Therefore, the development of proper treatments reducing the lead content in wastewater is essential in order to avoid lead poisoning.

Several approaches have been employed to remove heavy metal ions from wastewater such as the chemical precipitation, adsorption, cations-exchange, reverse osmosis, electrodialysis, and electrochemical reduction (Fu and Wang, 2011). However, generally, the adsorption process has been proved convenient in terms of cost, simplicity and flexibility (Rengaraj et al., 2002). In this regard, several sorbents have been recommended for the removal of Pb^{2+} ions, such as the activated carbon, and mesoporous and nanoporous materials as clays, zeolites, chitosan and apatite (Ziagova et al., 2007). Of these sorbents, calcium hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$, abbreviated as HAp), has shown a remarkable sorption efficiency for long-term containments (Sun et al., 2013). Recently, biological molecules such as chitin, chitosan, and lignin have also been recommended for the removal of toxic metals (Shen et al., 2013). Chitosan biopolymers (Cs), in particular, with their non-toxicity, hydrophilicity, biocompatibility, and biodegradability, exhibited effective sorption efficiency for heavy metals (Shen et al., 2013). In this investigation, artificially-made calcium hydroxyapatite nanorods (nHAp) and hydroxyapatite/chitosan nanocomposite (nHApCs) have been employed in the removal of lead ions from lead-containing aqueous samples using the adsorption technique. Several parameters including the adsorption time, initial Pb^{2+} concentration, adsorbent dosage, and pH have been investigated to evaluate their influence on the sorption efficiency.

2. Materials and methods

2.1. Sorbent preparation

All chemicals used in this investigation were of analytical grade and used without further purification. Calcium hydroxyapatite nanorods (nHAP) were synthesized by the sol gel method according to the procedure described earlier (Salah et al., 2014). Typically, 100 mL 0.479 M H_3PO_4 (Sigma–Aldrich No. 04107) was dropwisely added (3 mL/min) to a 100 mL 0.8 M solution of $Ca(NO_3)_2{\cdot}4H_2O$ (Sigma–Aldrich No. 31218) in order to achieve a Ca/P stoichiometric ratio of 1.67. The pH was adjusted at 10 ± 0.05 using ammonia solution (33%). The following equation represents the reaction involved therein at room temperature.

$$10Ca(NO_3)_2 + 6H_3PO_4 + 20NH_4OH \rightarrow Ca_{10}(PO_4)_6(OH)_2 \downarrow + 20NH_4NO_3 + 18H_2O \quad (1)$$

The suspension was left under moderate stirring for 16 h, and after 24 h of aging at room temperature, the precipitate was rinsed with deionized water and dried at 80 °C under vacuum.

Similarly, the hydroxyapatite/chitosan nanocomposite (nHApCs) was prepared by adding 0.479 M H_3PO_4 dropwisely to 0.8 M solution of $Ca(NO_3)_2{\cdot}4H_2O$ containing 0.25 g chitosan (Cs, Sigma, No. 48165) in order to keep the stoichiometric ratio of Ca/P at 1.67, and similar procedure was followed up but the precipitate drying was achieved at 60 °C. By the way, Cs was initially dissolved in acetic acid (HAc) before mixing with calcium nitrate solution.

$$\text{In acid medium} \quad Cs-NH_2 + HAc \rightleftharpoons Cs-NH_3^+ + Ac^- \quad (2)$$

$$\text{In Basic medium} \quad Cs-NH_3^+ + OH^- \rightarrow Cs-NH_2 \downarrow + H_2O \quad (3)$$

2.2. Materials characterization

The morphology, crystal structure, functionality and stability of the prepared sorbents were revealed using a set of advanced techniques. The high resolution transmission electron microscope (HR-TEM, Tecnai G20, FEI, Netherland) supported with an energy dispersive X-ray (EDX) unit was used for the sake of imaging and crystal structure revelation. Two different modes of imaging were employed; the bright field at 200 kV using LaB_6 electron source gun and the diffraction pattern imaging. Before imaging, the HAp particles were deposited from a dilute aqueous suspension onto Cu grids with the support of a carbon film. The crystal structure of the prepared sorbents was identified using X-ray diffraction (XRD) (X'Pert PRO PANalytical, Netherland), which operated at 45 kV and 30 mA using filtered Cu Kα radiation (λ = 1.5406 Å) in the 2θ range from 5° to 80° and high score plus software. In order to investigate the sorbent' functionality, Fourier transform infrared spectroscopy (FTIR, Jasco 6100 − Japan) was conducted in KBr pellet at room temperature over the range from 400 to 4000 cm^{-1}. Zeta potentials (Zetasizer Nano S, Malvern Instruments, UK) of nHAp and nHApCs aqueous suspensions of different pH (2–11) were measured to evaluate the point of zero charge (PZC) of these colloidal suspensions. Before measurements, nHAp and nHApCs were dispersed in deionized water (Milli-Q Millipore, Billerica, MA, USA) and the pH was adjusted using NaOH and HCl, and the suspension was left under sonication (Ultrasonicator, SB-120DTN, Taiwan) for 10 min.

2.3. Sorption experiment

2.3.1. Determination of the sorption capacity

The batch equilibrium technique was employed at room temperature. Lead ions solutions with different concentrations were prepared by dissolving $Pb(NO_3)_2$ (Eastern fine chemicals, Italy) in deionized water. Typically, a certain amount of nHAp or nHApCs is added to 200 mL of the Pb^{2+} solutions after adjusting the pH. Next, the mixture was shaken (Shaker, Lab-line 4625-1CE, USA) at a speed of 300 rpm for a given

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