



Preparation and adsorption properties of nano magnetite chitosan films for heavy metal ions from aqueous solution

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ABSTRACT

Nano magnetite chitosan (NMag-CS) film was prepared and characterized with different analytical methods. X-ray diffraction (XRD) patterns confirmed the formation of a pure magnetite structure and NMag-CS nanocomposite. TEM image of the film, revealed the uniform dispersion of magnetite nanoparticles inside chitosan matrix. The adsorption properties of the prepared film for copper, lead, cadmium, chromium and nickel metal ions were evaluated. Different factors affecting the uptake behavior by the composite films such as time, initial pH and film dose were investigated. The adsorption equilibrium attained using 2 g/L of the film after 120 min of reaction. The equilibrium data were analyzed using Langmuir and Freundlich models. The adsorption kinetics followed the mechanism of the pseudo-second-order equation for all metals. The metals regenerated from films with an efficiency greater than 95% using 0.1 M ethylene diamine tetra acetic acid (EDTA) and films were successfully reused for adsorption.

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1. Introduction

Heavy metals are highly toxic at low concentrations and can accumulate in living organisms, causing several disorders and diseases [1]. The main techniques that have been used for metal content reduction from industrial waste are chemical precipitation, ion exchange, membrane filtration, electrolytic methods, reverse osmosis, solvent extraction, and adsorption [2,3]. However, these methods are limited by high operational cost and/or may also be efficient in the removal of some toxic metal ions, mainly at trace level concentrations [4]. Adsorption has increasingly received much attention in recent years because the method is simple, relatively low-cost and effective in removing heavy metal ions from water [5]. Nanoparticles, often characterized by large specific surface area, have attracted great interest because of their unique properties and potential applications. Adsorption procedure combined with magnetic separation has been used extensively

in water treatment and environmental cleanup [6,7]. Iron oxide NMs are promising for industrial scale wastewater treatment, due to their low cost, strong adsorption capacity, easy separation and enhanced stability [8]. The adsorption of heavy metal ions onto magnetic nanoparticles has many advantages, such as easy recycling, high reusability and large surface area of nanoparticles [9,10].

Attention has recently been focused on chitosan and its derivatives as bioadsorbents. Chitosan (CS) is one of the most abundant biopolymers in nature [11] and has been classified as one of the potentially low-cost sorbents with high adsorption capacity [12]. It is characterized by its ability to uptake several metal ions and its dissolution of was decreased through crosslinking treatments. The cross-linking procedure may be performed by reaction of chitosan with different cross-linking agents such as glutaraldehyde [13,14]; glyoxalin [15] and ethylene glycol diglycidyl ether [16]. Using pure chitosan as an adsorbent have some disadvantages such as low surface area [17].

Recently, many researchers have reported the application of magnetic chitosan for removing metal ions [18,19]. Magnetic chitosan have several advantages as the strong metal chelating capability due to presence of the amine and hydroxyl groups in chitosan chain. Also, it can easily be separated from the sorption system using magnetic field due to magnetic properties. In the

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present study, nano magnetite–chitosan films were prepared and were further used for the removal of heavy metals from aqueous solution.

2. Experiment

2.1. Materials

Ferric chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$), ferrous chloride tetrahydrate ($\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$), ammonium hydroxide, nitric acid, copper sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$), lead nitrate ($\text{Pb}(\text{NO}_3)_2$) and the chloride salts of Cd(II) and Ni(II) were of analytical grade and obtained from Aldrich, Germany. Chitosan with acetylation degree of 90% obtained according to methodology previously described by Abdou et al. [20] and Tawfik et al. [21]. All aqueous solutions were prepared with de-ionized water. The working solutions of metal ions were prepared by appropriate dilutions of the stock solution immediately prior to their use.

2.2. Nano magnetite–chitosan films (NMag–CS) preparation

Magnetite nanoparticles prepared by co-precipitation method obtained according to methodology previously described in other work [22] with average size 2–5 nm.

The NMag–CS films were prepared by casting methodology. Magnetite nanoparticles were dispersed in chitosan solution using ultrasound radiation (chitosan was solubilized in 7% of acetic acid solution). The mass ratios between chitosan and magnetite were fixed in 1:1 mass ratio, with the addition of the crosslinker solution (25% glutaraldehyde aqueous solution). The resultant solution was poured into round-plate shape petri dishes. The films were dried at room temperature and peeled off petri dishes and immediately washed with NaOH aqueous solution (0.01 M); purified in distilled water then dried and stored at room temperature.

2.3. Characterization of adsorbent

2.3.1. X-ray diffraction spectrometry (XRD)

The structural characterization of the prepared films was conducted by powder X-ray diffraction spectrometry (XRD) using Bruker D8 advance instrument between 5 and 80° (2θ) at a scanning rate of 4°/min.

2.3.2. Transmission electron microscopy (TEM)

High Resolution transmission electron microscope (HR TEM-JEOL 2100, Japan) was used to determine the morphology of the nanoparticles. Nanoparticles solutions were sonicated to produce better dispersion for particle and preventing the particle agglomeration on the copper grid.

2.3.3. Brunauer–Emmett–Teller (BET) analysis

Gas adsorption analyzer with Brunauer–Emmett–Teller (BET) method (Quanta chrome NOVA automated gas sorption system-sorb-1.12) was used for the surface area determination where N_2 gas was used as adsorbate at 77 K.

2.3.4. FTIR analysis

The functional groups of different adsorbents were identified by Fourier Transform Infrared Spectroscopy (FTIR) analysis using FTIR-6100 (JASCO-Japan) instrument via the KBr pressed disc method, in a range starting from 400 to 4000 cm^{-1} wavenumbers.

2.3.5. Magnetic characterization

Magnetic properties were measured in the solid state using a Vibrating Sample Magnetometer (VSM). The saturation

magnetization value was determined from the plateau region of the magnetic flux density of a solid sample at 8000.

2.4. Adsorption studies

The adsorption behavior of the prepared film for metal ions (Cu^{2+} , Pb^{2+} , $\text{Cr}(\text{VI})$, Cd^{2+} and Ni^{2+}) was investigated by means of the batch experiments at room temperature. A known amount of the film was mixed with a synthetic solution of metal ions at 20 mg/L for each. Experiments were conducted using different doses of the adsorbent that ranged from 0.1 to 4 g/L. The adsorption of metal ions by prepared film was investigated in an initial pH range of 4–9. The solution pH was adjusted by 0.1 M NaOH and 0.1 M HNO_3 . The bottles were shaken in a rotary shaker at 200 rpm at different contact times from 5 to 140 min.

The equilibrium adsorption capacity, q_e (mg/g), of metal was calculated using the mass balance, according to the following equation:

$$q_e = (C_0 - C_e)V/m \quad (1)$$

where V is the sample volume (L), m is the mass of the adsorbents (g), C_0 is the initial metal ion concentration (mg/L), and C_e is the equilibrium concentration of metal ion in the solution (mg/L).

2.5. Analytical methods

The concentration of metal ions in the solution was determined according to APHA [23] using Atomic Absorption Spectrometer (Varian Spectra AAS 220) with graphite furnace accessory and equipped with deuterium arc background corrector. The precision of the metal measurement was determined by analyzing samples in triplicate and for each series of measurements an absorption calibration curve was constructed.

2.6. Desorption of metal ions and reusability of NMag–CS films

In this experiment, desorption of metals from metal-loaded nanoadsorbent was performed using ethylene diamine tetraacetic acid (EDTA 0.1 M) with the contact time of 2 h at 200 rpm of agitation. After desorption, the nanoadsorbent was separated by filtration and metal concentration measured. To test the reusability of the film, 10 mL of 20 mg/L metals solution was mixed with 20 mg of film for 2 h and then desorbed with the addition of 10 mL of EDTA (0.1 M) with stirring for 2 h. After each cycle of adsorption–desorption, the adsorbent was washed thoroughly with distilled water to neutrality, then dried and reconditioned for adsorption in the succeeding cycle.

3. Results and discussion

3.1. Characterization of NMag–CS films

3.1.1. XRD analysis

In the X-ray powder diffraction (XRD) pattern of the prepared magnetite nanoparticles (not shown here), six characteristic peaks ($2\theta = 30.1, 35.5, 43.1, 53.4, 57.0, \text{ and } 62.6^\circ$) were obtained and the characteristic peak was at position of 2θ of 35.5° [22]. Fig. 1 showed XRD patterns of NMag–CS film.

The composite has amorphous nature and the characteristic peaks for magnetite shifted to 34.7° indicating that the magnetite nanoparticles were successfully coated by amorphous chitosan. Chitosan film showed characteristic peaks around 2θ of 20.48° with an amorphous structure of chitosan. The structure of chitosan is strongly dependent on its processing treatment, such as dissolving, precipitation, and drying.

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