



Removal of copper using novel amidoxime based chelating nanohybrid adsorbent

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ABSTRACT

Novel poly(acrylonitrile) grafted sepiolite nanohybrid material (RGS) has been synthesized by using simultaneous radiation grafting technique. The grafting yield was measured by varying the absorbed dose and monomer concentration in the mixture. The acrylonitrile group present in nanohybrid material was chemically modified into amidoxime. Both the grafting yield and degree of conversion of acrylonitrile to amidoxime was examined by gravimetric analyses. The structural and morphological investigations of these nanohybrid materials were performed by FT-IR, XRD and FESEM. Batch adsorption studies were carried out for copper uptake onto amidoximated nanohybrid adsorbents to envisage the effects of pH, adsorbent dose, contact time and initial concentration. Removal of copper ion attained equilibrium within 30 minutes following pseudo-second order kinetics. The equilibrium process was well described by the Langmuir isotherm model and the maximum adsorption capacity (Q_m) was found to be 278 mg/g for 5 kGy irradiated sample. This environmental friendly and cost effective adsorbent can be used for the removal of copper ion from aqueous solutions.

1. Introduction

The organic-inorganic nanohybrid materials have received approbation by the engineering minds in the last few decades [1]. These nanohybrid materials have demonstrated their applications in almost every field of life due to their synergistic properties [2]. Clays contribute an imperative function as a support for organic materials, owing to their structural, morphological and textural features which are the key components for the preparation of a wide range of innovative nanohybrid materials [3].

Among clays, sepiolite holds special position due to its morphological inimitability [4]. It is 2:1 phyllosilicate having two tetrahedral silica sheets sandwiching an octahedral central sheet of magnesium oxide-hydroxide. The periodic inversion of the silica sheets results in the discontinuity of the tetrahedral sheets. As a result silanol groups (Si-OH) are present at the peripheral surface of the sepiolite fibers. The silanol groups are utilized to incorporate different organic species including coupling agents and polymers [5]. The surface area and density of silanol groups of sepiolite are about 320 m²/g and 2 Si-OH groups/nm² respectively which is highest among all the clay minerals [6]. Sepiolite has good sorption capacity due to its high surface area, porosity as well as the unusual needle-like particle shape that make it a valuable material for a wide range of applications.

Grafting is an appealing process to transform the surface properties of base material according to target applications [7]. This technique is being used by many researchers to incorporate the organic moiety to enhance its compatibility and considered the best way to achieve hybrid material [8].

Radiation-induced graft polymerization (RIGP) is one of the most remarkable techniques for transforming the physico-chemical

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properties of natural and synthetic polymers by free radical and/or ionic polymerization mechanisms [9,10]. It has been known as a simple, efficient and environmentally amicable method in modifying polymeric materials. Nasef and Guven [9] reviewed status, challenges and future directions for radiation grafted copolymers used for separation and purification purposes. Badawy et al. [10] synthesized crosslinked polyacrylonitrile granules by RIGP. Using this technique for grafting a monomer on modified clay is a rare concept in literature.

The natural environment is contaminated by the release of large quantities of heavy metals. Toxic metals are responsible for the cause of various diseases and ailments when exceed specific limits. They are associated with tendency to accumulate in living organisms and consequent amplification in the food chain augmenting their ferocity. Among heavy metals, copper is a frequently used material resulting in many actual or potential sources of copper effluence. The USEPA has determined that drinking water should not contain more than 1.3 mg L^{-1} of Cu(II). Minimization and elimination of Cu(II) from aqueous media is therefore a vital area of study. The adsorption is considered the most promising method for the removal of toxic metals. The adsorbent should have strong attraction for the target metal ions binding them irreversibly under ambient conditions [11]. Gao et al [12] successfully prepared poly(amidoxime)/SiO₂ chelating materials for adsorption of heavy metal ions especially for Cu(II) ion. Mahdavinia et al [13] synthesized magnetic amidoximated nanocomposites based on a combination of chitosan and magnetic laponite and examined the removal of Cu(II). Clays have been extensively studied for the adsorption of metal ions because of their structural morphology and abundance [14].

The objective of this work is to synthesize novel nanohybrid material by simultaneous radiation grafting technique. In this work sepiolite is first functionalized with coupling agent vinyltriethoxysilane to obtain vinyl modified sepiolite. Gamma radiation is used to graft acrylonitrile monomer on vinyl modified sepiolite. The acrylonitrile grafted nanohybrid is chemically modified into amidoxime chelating adsorbent which is used for Cu(II) uptake studies.

2. Experimental

2.1. Materials and chemicals

Sepiolite, acrylonitrile (AN), vinyltriethoxysilane (VTES), acetone, methanol, copper sulphate and hydrochloric acid were purchased from Sigma Aldrich. Isopropanol from Daejung, dimethyl formamide (DMF) from Lab-Scan, hydroxylamine hydrochloride from Pancreac, anhydrous sodium carbonate from Honeywell and sodium hydroxide from Merck were used in this study. All chemicals were of analytical grade and used without further purification.

2.2. Synthesis

2.2.1. Purification and vinyl modification of sepiolite

Sepiolite (SP) was washed and vinyl modified according to the reported method [15]. Briefly, sepiolite (10 g) was mechanically dispersed in 1 L water for 24 h. It was filtered, dried and ground into fine powder. This fine powder was dispersed in isopropanol in a glass reactor and hydrolyzed VTES (0.12 mol) was added drop wise. This mixture was stirred for 2 h at 60 °C, filtered, washed with methanol and dried under vacuum. The modified sepiolite (MS) was pulverized into fine powder.

2.2.2. Simultaneous radiation grafting

Acrylonitrile monomer and MS (61.0 mmol of AN/g of MS) were added in glass reactor. Prior to this addition, the glass reactor was first evacuated and purged with dried nitrogen. This mixture was stirred vigorously for 8 h under inert environment to form a stable-in-time suspension. Then it was irradiated in gamma cell at different doses (2.5, 5.0, 7.5 and 10.0 kGy) with dose rate of 5.84 kGy/h at ambient temperature, at Nuclear Institute of Food and Agriculture (NIFA), Peshawar. After irradiation, the resulting nanohybrids (denoted as RGS 2.5, RGS 5, RGS 7.5 and RGS 10) were ball milled using FRITSCH pulverisette 6 Ball Mill at revolution speed of 200 rpm for 1 h. The powder was washed with acetone and Soxhlet extracted with DMF for 8 hours to remove unreacted monomer and homopolymer. Samples were vacuum dried till constant weight. Grafting percentage (G %) was calculated by the following formula

$$\text{Grafting (\%)} = \frac{W_g - W_0}{W_0} \times 100 \quad (1)$$

where W_0 and W_g are the initial and grafted weights of MS and RGS respectively.

2.2.3. Amidoximation of radiation grafted sepiolite

This step converts nitrile group ($-\text{C}\equiv\text{N}$) present on RGS into amidoxime group (AO, $-\text{C}(\text{NH}_2) = \text{NOH}$). RGS was immersed in a solution of hydroxylamine hydrochloride (10 g) dissolved in water-methanol ($v/v = 1:1$) system containing sodium carbonate (7.5 g). The pH of the above solution was maintained at 7.0 ± 0.5 and gently refluxed at 80 °C. After 6 h of stirring, the mixture was washed with deionized water to remove unreacted substances. The final product was vacuum dried at 60 °C and denoted as AO-RGS 2.5, AO-RGS 5, AO-RGS 7.5 and AO-RGS 10. The conversion ratio of CN to AO (AO %) and group density of AO (AO-GD) were calculated as follows:

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