



One pot eco-friendly synthesis of highly dispersed alumina supported alginate biocomposite for efficient chromium(VI) removal



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ABSTRACT

An eco-friendly biocomposite namely alumina/alginate (AlAlg) composite was synthesized by uniformly dispersing alumina (Al) over alginate (Alg) polymeric matrix by one-pot method. The structure, surface morphology and elements present in the biocomposite was characterized using instrumental techniques like FTIR, SEM and EDAX analysis respectively. Batch process was adopted to investigate the potential of the composite toward chromium(VI) removal. The various adsorption influencing factors like contact time, pH, competing anions, initial chromium concentration and temperature were investigated. The maximum Cr(VI) sorption capacity (SC) of AlAlg composite was found to be 17.45 mg/g and shows enhanced SC than CaAlg composite which possesses the SC of 9.45 mg/g. The experimental data were fitted to various isotherms and kinetic models. The nature of chromium sorption was explained by the calculated values of thermodynamic parameters and the feasible chromium sorption mechanism was also established.

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1. Introduction

Heavy metal pollution is a growing concern due to its prolonged and pronounced toxicity. [1]. Compared with organic pollutants, heavy metals accumulate more easily into the food chain of the living beings [2]. Among the heavy metals, chromium is one of the top sixteen toxic pollutants due to its carcinogenic nature [3]. The natural sources of chromium are volcanic dusts, rocks and soil, whereas the man-made sources are the effluents released from assorted industries like electroplating, leather tanning, mining, etc. Chromium exists as Cr(VI) as anion and Cr(III) as cation in the aqueous solution. Anionic hexavalent chromium crosses the cell membrane and cause DNA damage on the other hand trivalent chromium is an essential nutrient for maintaining protein metabolism and diabetes. In view of the fact, that chromium exposure leads to lung cancer, ulceration, hemorrhage, etc., its removal has become the need of the hour [4].

The removal of carcinogenic hexavalent chromium can be achieved through numerous ways viz., precipitation [5], reduction [6], ion-exchange [7], adsorption [8] and electro-chemical methods [9]. Adsorption is the efficient among the reported

methods due to its low cost, easy handling and its selectivity towards chromium. Adsorption mainly relies on the nature of the adsorbent material subjected in the process which might be of mineral, organic or biological origin. Pioneer researchers have tried the variety of adsorbents namely graphene [10], alumina [11], zeolites [12], clays [13], biopolymers [14,15], alumina/chitosan composite [16], nano composite [17], membranes [18,19] etc., for the removal of chromium. Alumina is one such inorganic material that can be used as adsorbent for chromium removal, but the main drawback of using alumina as powdered form is that it would create pressure drop during field studies. Such type of the technical problems can be surmounted by developing polymeric composites by dispersing alumina in the polymeric matrix.

The non-toxic and abundance nature of biopolymers have made it as an imperative adsorbent among the documented [20]. However, these biopolymers also have their drawback of possessing low stability and low sorption capacity. To overcome such tailbacks, biocomposites were synthesized by dispersing inorganic materials over biocompatible biopolymers. Such biocomposites were subjected for chromium removal and found to possess enhanced sorption capacity [21–23]. The most abundant polysaccharide in living organisms is alginate, which is a linear copolymer obtained from marine brown algae and it is non-toxic, biocompatible and inexpensive [24]. Similar to other biopolymers, alginate is also unstable and possesses low sorption capacity. To overcome these

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limitation, various alginate supported composites were synthesized and subjected for toxic ions removal [25–28].

Based on the literature study, the present investigation focuses on the eco-friendly synthesis of the biocomposite by uniformly dispersing alumina particles over the alginate polymeric matrix (AlAlg composite). The synthesized biocomposite was characterized using FTIR, SEM and EDAX analysis. The optimization studies for the maximum Cr(VI) sorption was achieved by varying the contact time, pH, competing anions, initial chromium concentration and temperature. The equilibrium data was fitted with various isotherm and kinetic models. The thermodynamic parameters viz., ΔG° , ΔH° and ΔS° have also been calculated to predict the nature of sorption and a feasible sorption mechanism was established for chromium removal.

2. Materials and methods

2.1. Materials

Sodium alginate with a molecular weight of 70,000–80,000 was purchased from Himedia (India). Alumina, potassium dichromate, 1,5-diphenyl carbazide, calcium chloride and all other chemicals used were of AR grade and purchased from Merck (Germany). Double distilled water was used to prepare the standard solutions.

2.2. Synthesis of alumina/alginate (AlAlg) composite

About 20 g of alumina (Al) was immersed in 30 mL of distilled water to make it a slurry. To it about 2 g of sodium alginate dissolved in distilled water was added with constant stirring for 12 h. To the above slurry mixture, 2% of CaCl_2 solution was added dropwise, stirred vigorously for 2 h and was left for 24 h to undergo complete cross-linking reaction. The obtained AlAlg biocomposite was filtered, washed with distilled water and dried at 60 °C for 24 h in hot air oven. The biocomposite was grinded to fine powder using ball mill (IKA, Germany). The synthesized AlAlg biocomposite was then used for chromium sorption studies after sieving to get uniform sized particles.

2.3. Chromium sorption experiments

Batch equilibrium method was performed to determine the extent of chromium adsorption by the composite. The initial Cr(VI) solution (100 mg/L) was prepared by dissolving 0.2828 g of AR grade potassium dichromate in 1000 mL distilled water. About 0.1 g of the composite was added to the flask containing 50 mL of the solution with 100 mg/L chromium concentration and placed in a thermo stated shaker at 200 rpm. After attaining equilibrium, the samples were filtered and the final chromium concentration was determined. Various influencing factors on sorption process viz., contact time, pH of the medium, initial chromium concentration and the presence of other competing anions in water were all investigated. The influence of pH on the sorption of chromium was studied by varying the pH range from 2–10 using 0.1 M HCl/NaOH solution. The effect of temperature was studied at three different temperatures 30, 40 and 50 °C by taking 50 mL of chromium solution at different initial concentrations viz., 80, 100, 120 and 140 mg/L and sorbent mass of 0.1 g. The filtrate was then used for residual chromium ion determination.

2.4. Qualitative analysis

The chromium estimation was done by using UV–vis spectrophotometer – Spectroquant – Pharo 300 (Merck, Germany) by adding 1,5-diphenyl carbazide as complexing agent and measuring the color intensity at 540 nm. The standard methods were adopted

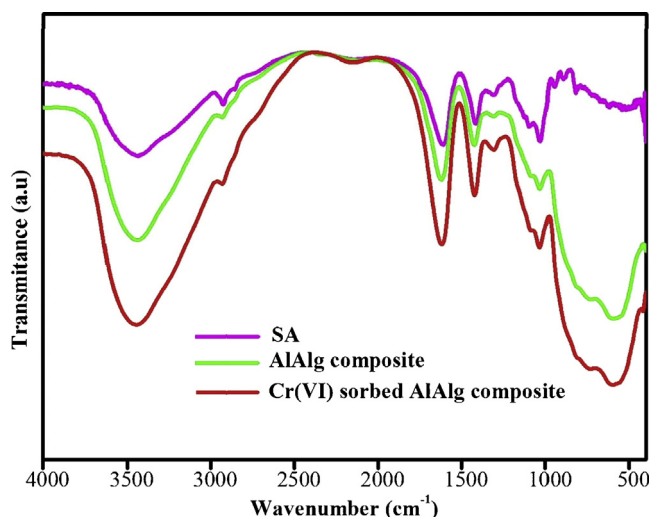


Fig. 1. FTIR spectra of sodium alginate, AlAlg composite and Cr(VI) sorbed AlAlg composite.

for investigating other water quality parameters [29]. The pH of the solution was measured using Thermo Orion Benchtop multiparameter kit (VERSA STAR 92) using pH electrode. The pH at zero point charge (pH_{zpc}) of the composite was determined by pH drift method [30].

2.5. Instrumental analysis

For structural investigation of the composite, Fourier transform infrared (FTIR) studies were carried out using JASCO-460 plus spectrophotometer in the range of 400–4000 cm^{-1} using KBr pellets. Scanning electron microscopy (SEM) with JSM 6390 was used to study the surface morphology of the composite. The SEM facilitates the direct observation of the surface microstructures of the fresh and Cr(VI) sorbed composite. To explore the localization of elements on the biocomposite, energy dispersive X-ray analysis (EDXA - JSM 6390) was carried during SEM observations.

2.6. Statistical tools

Microcal Origin (Version 8) was used for the computational works. The dependability of the fit and the best model were discussed by adopting the error bar plot, regression correlation coefficient (r), standard deviation (sd) and chi-square analysis (χ^2).

3. Results and discussion

3.1. Characterization of the composite

FTIR spectra of sodium alginate (SA), AlAlg composite and Cr(VI) sorbed AlAlg composite are shown in Fig. 1. The bands observed at 3443, 1614 and 1423 cm^{-1} in IR spectrum of SA were due to the stretching vibrations of -OH, asymmetric and symmetric stretching vibrations of -COO groups respectively [31]. In the FTIR spectra of AlAlg composite, a new band at 560 cm^{-1} was observed due to the incorporation of alumina in the alginate polymatrix. Interestingly among all the major bands in the FTIR spectra of chromium sorbed AlAlg composite, a new band at around 625 cm^{-1} is due to the metal-oxygen stretching mode. The shifted bands observed at 1628 and 1435 cm^{-1} suggest that chromium sorption occurs on AlAlg composite. The broadening of band at 3443 cm^{-1} and shifting of other bands to higher frequencies in the chromium treated

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