



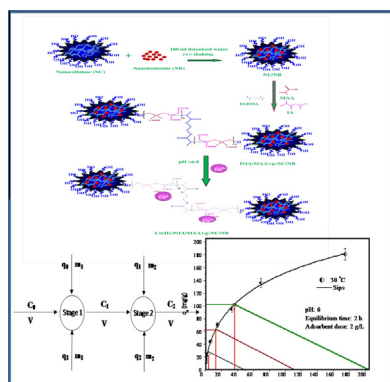
Nanocellulose/nanobentonite composite anchored with multi-carboxyl functional groups as an adsorbent for the effective removal of Cobalt(II) from nuclear industry wastewater samples



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GRAPHICAL ABSTRACT



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ABSTRACT

A novel adsorbent, poly(itaconic acid/methacrylic acid)-grafted-nanocellulose/nanobentonite composite [P(IA/MAA)-g-NC/NB] with multi carboxyl functional groups for the effective removal of Cobalt(II) [Co(II)] from aqueous solutions. The adsorbent was characterized using FTIR, XRD, SEM-EDS, AFM and potentiometric titrations before and after adsorption of Co(II) ions. FTIR spectra revealed that Co(II) adsorption on to the polymer may be due to the involvement of $-\text{COOH}$ groups. The surface morphological changes were observed by the SEM images. The pH was optimized as 6.0. An adsorbent dose of 2.0 g/L found to be sufficient for the complete removal of Co(II) from 100 mg/L at room temperature. Pseudo-first-order and pseudo-second-order models were tested to describe kinetic data and adsorption of Co(II) follows pseudo-second-order model. The equilibrium attained at 120 min. Isotherm studies were conducted and data were analyzed using Langmuir, Freundlich and Sips isotherm models and best fit was Sips model. Thermodynamic study confirmed endothermic and physical nature of adsorption of the Co(II) onto the adsorbent. Desorption experiments were done with 0.1 M HCl proved that without significant loss in performance adsorbent could be reused for six cycles. The practical efficacy and effectiveness of the adsorbent were tested using nuclear industrial wastewater. A double stage batch adsorption system was designed from the adsorption isotherm data of Co(II) by constructing operating lines.

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1. Introduction

Toxic heavy metals such as Cadmium, chromium, cobalt, lead and mercury are major concern in the field of water pollution [1,2]. Cobalt, a natural element present in certain ores of the Earth's crust, is essential to life in trace amounts, as it is the major constituent in vitamin B₁₂ [3,4]. But if it is in higher concentration it is very harmful. Application of Cobalt(II) [Co(II)] in galvanization, battery recharging, metal plating, paints and pigment industries, mining operations, nuclear power plant grinding wheels and tanneries produce various liquid and solid wastes rich in Co(II), soil surrounding many military bases are also contaminated [5]. The effects of acute Co(II) poisoning in humans can cause, diseases like asthma, lung irritations, diarrhea, pneumonia, weight loss, vomiting and it adversely affect thyroid gland and liver. Cobalt inside the body in excess amount can cause gene mutation and cancer [6–8]. Exposure to ionizing radiation is associated with an increased risk of developing cancer. Some isotopes of cobalt do emit ionizing radiation. Recovery of cobalt is very important as cobalt and its salts are used in nuclear medicine, enamels and semiconductors, grinding wheels, painting on glass and porcelain, hygrometers and electroplating; as a drier for lacquers, varnishes and paints, and as a catalyst for organic chemical reactions [9,10].

For reducing toxicity of industrial effluents various methods have been developed for the effective reduction of concentration of toxic metals in aqueous solutions. Even though various technologies were developed for the recovery of cobalt from the aqueous solution, ion exchange, and adsorption are considered as basic purification methods for the treatment of industrial wastewater due to simplicity in practical application, high efficiency and economic nature [4]. Recent adsorption techniques, based on the nano-composite/polymer adsorbent, has been hot spot due to significant higher surface area to volume ratio resulting higher adsorbent efficiency and spontaneous removal of toxic metal ions [11,12]. The adsorption properties of native cellulose can be increased by converting it to the nanostructure [13]. The physical and chemical properties of nanocellulose [NC] can be improved by incorporation of nanobentonite [NB] to the polymer chain of NC [14]. Grafting of synthetic polymers on to solid nano composite and multi-carboxyl fictionalization is reported to be effective for the removal of metal cations from the aqueous solution [15]. The proposed profitability of the chemically modified polymeric bio-material/clay composite is not only the availability of functional moiety covalently bonded to the inorganic matrix for binding metal ions but also utilization of properties eco friendly composite materials [16].

The objective of the present work is to develop a polymer composite emphasis to the pH sensitive composite polymer based on nanocellulose/nanobentonite [NC/NB], Itaconic acid [IA], Methacrylic acid [MAA] as the adsorbent for the removal of the Co(II) metal ions from the aqueous solutions considering the fact that IA could be degraded by microorganism in the environment in which the polymer is applied for the removal of the metal ion, and the rest of the polymer will be destroyed hence the environmental adverse impact would be minimal.

Poly(itaconic acid)-poly(methacrylic acid)-grafted-nanocellulose/nanobentonite composite [P(IA/MAA)-g-NC/NB], was synthesized from NC/NB. NC was synthesized using cellulose extracted from cotton by previously described procedure [17]. Polymer synthesis involves ionic cross linking of NC/NB with IA is followed by radical polymerization of MAA and cross linking with Eethylene glycol dimethacrylate [EGDMA]. The adsorbent before and after Co(II) adsorption was characterized using FTIR, SEM-EDS, XRD and potentiometric titrations. To achieve maximum efficiency, the adsorption conditions such as pH, adsorbent dose, contact

time, initial metal ion concentrations by batch method. The results obtained from kinetic and equilibrium studies imply that P(IA/MAA)-g-NC/NB can be used as efficient adsorbent for the removal of Co(II) from aqueous solutions.

2. Materials and methods

2.1. Materials

Analytical grade chemicals were used for the investigation. MAA, IA, KPS and EGDMA were obtained from E-Merk, India Ltd. Cellulose was extracted from cotton. Bentonite and Co(NO₃)₂·6H₂O were supplied by Fluka Chemie (Switzerland). Working solution of Co(II) concentration ranging between 10 and 500 mg/L was prepared by diluting the stock solution (1000 mg/L of Co(NO₃)₂·6H₂O) with distilled water.

2.2. Equipments and methods of characterization

The FTIR spectra of the adsorbent materials were taken with Perkin Elmer 1800 model IR operating a frequency range from 600 to 4000 cm⁻¹ in transmission mode. The pH measurements were made on systronics μ pH meter (Model 361). X-ray diffraction (XRD) patterns of the samples were examined using Siemens D5005 X-ray unit. Cu K_α (λ = 1.54064 Å) radiation generated a voltage of 40 kV and current of 40 mA was used as X-ray source. Scanning electron microscopy/Energy Dispersive X-ray Spectroscopy or EDS (SEM/EDS) analyses were done using Nova Nano SEM NPEP 252 High resolution FE-scanning electron microscope/Bruker. Atomic force microscope (AFM) images were recorded on Buker DIMENSION edge with SCAN ASYST instrument by taping mode. Temperature controlled water bath shaker (Lab line instruments Pvt Ltd Kochi, India) with a temperature tolerance ±1 °C was used for performing batch mode kinetic and isotherm experiments. To measure Co(II) concentration in the aqueous solution before and after adsorption, GBC atomic absorption spectrometer, (AVANTA, Australia) was used.

2.3. Preparation of adsorbent

Cellulose was extracted from cotton using acid hydrolysis method [18] and it was converted to NC [19]. NB prepared from bentonite by stirring with ethanol for 48 h maintained at 170 °C. The NC/NB composite was prepared by shaking 1.0 g each of NB and NC in a beaker containing 100 mL of distilled water for 12 h [20]. The functionalized NC/NB polymer composite was prepared by radical polymerization and cross linking of NC/NB with EGDMA as cross-linker IA and MAA as monomers.

2.4. Adsorption experiments

Adsorption experiments were conducted using thermostatic water bath shaker at 30 °C with a shaking speed of 200 rpm with 100 mL Erlenmeyer flasks. Batch experiments were performed by equilibrating 0.05 g of adsorbent with 25 mL Co(II) solutions of predetermined initial concentrations. By adding NaOH and HCl of different concentrations, initial pH of the solutions was maintained at the required pH value. The suspensions were shaken for 3 h. The solid and the liquid phases were separated by centrifugation at 6000 rpm for 15 min. The Co(II) concentrations in liquid phase were determined by AAS. The amount of Co(II) adsorbed in (mg/g) at equilibrium (q_e) was calculated from the following mass balance equation.

$$q_e = \frac{(C_0 - C_e)V}{W} \quad (1)$$

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