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Synthesis of Guar gum/bentonite a novel bionanocomposite: Isotherms, kinetics and thermodynamic studies for the removal of Pb (II) and crystal violet dye

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ABSTRACT

In the present study, the performance of Guar gum/bentonite bionanocomposite was evaluated for the sequestration of Pb (II) and crystal violet (CV) dye from aqueous solution. The bionanocomposite was characterized by SEM, EDX, FT-IR, XRD, TGA, BET and TEM techniques. The bionanocomposite showed porous, crystalline nature with particle size ranging between 3 and 11 nm and increased thermal stability. The point of zero charge was found to be 2.2. The maximum adsorption was observed at pH 5.1, 7.6 and contact time 240 min, 300 min for Pb (II) and CV, respectively. The kinetic study revealed that the adsorption process of Pb (II) and CV onto the bionanocomposite followed the pseudo-second order model, confirming chemisorption as a rate determining step. The adsorption isotherm was in good agreement with the Freundlich for Pb (II) and Langmuir for CV isotherm model, respectively. Thermodynamic studies showed that the process was physiosorption, spontaneous and endothermic with increased in randomness. The desorption study of Pb (II) and CV exhibited the excellent regenerative efficacy (upto fifth cycle) of bionanocomposite using 0.1 M HCl. The bionanocomposite has also been successfully applied for the removal of heavy metals from electroplating, battery manufacturing and medical wastewater before and after spiking CV dye solution to it.

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1. Introduction

With increasing urbanisation and industrialisation, the quality of water is being deteriorated. The major source of water pollution is the disposal of toxic industrial effluents into freshwater [1,15]. Hence, the accessibility of potable water and cleanliness is the most insistent problem distressing the living creature [15]. The dyeing and heavy metal effluents have harmful impact on the environment including flora and fauna. Therefore, considerable efforts have been devoted for the treatment of dye and heavy metal-laden effluents using various techniques such as coagulation, flocculation, reverse-osmosis, photo-degradation processes and ion-exchange. Among the numerous techniques, adsorption is the most preferred technique due to its simplicity, efficiency, convenience, ease of operation and cost ineffective nature [14].

Pb (II) is a toxic heavy metal that causes serious health issues such as cardiovascular deficiencies, lungs problem, bone injuries, kidney, liver and central nervous system disorders, hypertension and cancer on long exposure [4,10]. Crystal violet belongs to a cationic nature of dyes that is more toxic, mutagenic and carcinogenic than anionic and non-ionic dyes and are included in triarylmethane dye category. It causes various eyes related issues like irritation, conjunctiva and cornea injury. In severe cases, it can cause respiratory and kidney failure, permanent blindness and cancer. This dye is a potential biohazard as it has been proved to be a mitotic poisoning agent [9,16]. Therefore, it is essential to treat these toxic and carcinogenic pollutants using eco-friendly adsorbent.

With the advent of nanotechnology and practicability of green chemistry, bionanocomposites have attracted great attention for the removal of pollutants from wastewater [15]. The natural biopolymers are gaining interest because of their abundance, non-toxic, safe in use, inexpensive, renewable and biodegradable nature [19]. The adsorption capacity of the biopolymers can be improved drastically by introducing nanoscale fillers on the surface of polysaccharides [15]. Guar gum has been proved to be a prospective candidate owing to its high molecular weight, long polymeric chain and wide accessibility as compared to other biopolymers [6]. The derivatives of Guar's have high stability at high temperature and pH environments. Guar gum is a hetero polysaccharide of a mannose backbone with galactose side groups [6]. Being water miscible, guar gum cannot be exploited as an adsorbent in aqueous system. Therefore, modification of guar gum with the nanofiller clay-bentonite have resulted in water insoluble, porous and multifunctional adsorbent. Bentonite has been selected as the nanoscale filler due

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to its non-toxicity, high-cation exchange capacity, low cost, rich intercalation chemistry and ease of availability [7]. Bentonite is a 2:1 type of clay, consisting of two tetrahedral sheets (silicon/oxygen) separated by an octahedral sheet (aluminium/oxygen/hydroxyl) [1]. Guar gum is a good choice to modify bentonite for potential applications. Its intercalation would enhance the interlayer space of bentonite and also, the intercalated guar gum contains many hydroxyl groups that allow the easy uptake of pollutants by bentonite. The present paper reports a facile and simple, yet efficient synthesis of Guar gum/bentonite bionanocomposite (GG/Bent) as resourceful adsorbent for the rapid sequestration of toxic and carcinogenic pollutants-Pb(II) and crystal violet dye from aqueous media. The synthesized GG/Bent bionanocomposite has been analyzed by SEM-EDX, TEM, FTIR, BET, TGA and XRD techniques. The effect of various adsorption experimental parameters viz., pH, contact time, dose, initial concentration and temperature were studied to investigate the adsorption efficiency of GG/Bent bionanocomposite for Pb(II) and crystal violet dye. The kinetics, isotherm and thermodynamic studies of adsorption process have also been performed. In order to make the process more economical and feasible, the desorption with regeneration studies were also carried out for Pb(II) and crystal violet dye. The present bionanocomposite has also been proved to show excellent practical utility in treating electroplating, battery manufacturing and medical wastewater.

2. Materials and methods

2.1. Materials

Guar gum and bentonite were purchased from Sigma-Aldrich (USA). CH_3COOH, KCl, HCl and NaOH used in the experiments were of analytical grade. The nitrate salts of lead, copper and nickel (99% pure) were obtained from Merck, India. The stock solution of metal ion and dye (1000 mgL^{-1}) were prepared by dissolving the requisite amount of metal nitrate salts and dye in double distilled water.

2.2. Synthesis of GG/Bent bionanocomposite

The solution intercalation method was utilized for the synthesis of bionanocomposite reported elsewhere [20]. 5 g of bentonite were dispersed in 100 mL of double distilled water using a magnetic stirrer (800 rpm) at 313 K for 24 h with continuous stirring. Subsequently, the guar gum solution (5 g in 100 mL) was added to the bentonite solution and was left for ultrasonication for 48 h at 313 K. Finally, the bionanocomposite was washed thrice using water, then dried at 323 K for 5 h and grounded well using a mortar for subsequent studies.

2.3. Characterization

The elemental analysis and morphology of the bionanocomposite, Pb(II) and CV loaded bionanocomposite were examined by scanning electron microscope (SEM) equipped with energy dispersive X-ray spectrometer (EDX) (model JSM 6510LV, JEOL, Japan) with samples coated with gold. The size of bionanocomposite was examined using a transmission electron microscope (TEM, JEM 2100, JEOL, Japan) with samples placed on carbon coated copper grid. FTIR measurements were recorded in the range of 400–4000 cm^{-1} with a Nicolet iS50 FTIR spectrometer from samples in KBr pellets. XRD pattern of samples were analyzed using Bruker AXSD8 Advance X-ray diffractometer with Cuα radiation ($\lambda = 1.542\ A^0$) and scanning angle (0–80°). The Thermogravimetry analysis (TGA) of samples were carried out by using Perkin Elmer, STA 6000 analyzer under nitrogen flow at 10 °C/min heating rate in the range of 30–800 °C. The Brunauer-Emmette-Teller) BET surface area of bionanocomposite was evaluated using Nova Station 0 instrument recorded at liquid N_2 temperature. The pore diameter and pore volume were estimated from the adsorption curve of N_2 adsorption-desorption isotherm by BJH method (Barret-Joyner-Halenda) using Nova Station 0 instrument. Atomic absorption spectrophotometer (GBC 902, Australia) was used for examining the concentration of metal ion. The absorbance of the crystal violet solutions were measured by (T70 UV/VIS Spectrometer PG Instruments Ltd., U.K.) UV–vis spectrophotometer working at λ_{max} of 582 nm. The point of zero charge (pH_{PZC}) was determined using solid addition method. 20 mL of 0.1 M KCl solution were placed in a series of 100 mL conical flasks. The initial pH (pHi) of solutions was adjusted to 1–12 by using 0.5 M NaOH or 0.5 M HCl. 0.01 g of the adsorbent were added to the series of different pHi solutions and the suspensions were left to equilibrate for 24 h. The final pH (pHf) of the supernatant liquids were noted. The difference between pHf-pHi (ΔpH) was plotted against pHi. The point of intersection of the curve at $\Delta pH = 0$, gave the value of pH_{PZC}.

2.4. Batch adsorption studies

The batch adsorption experiments were conducted using 0.01 g of adsorbent treated with 20 mL of initial Pb (II) concentration (10–-100 mgL^{-1})/initial CV concentration (5–50 mgL^{-1}), contact time for Pb (II) and CV-(5–360 min), adsorbent dose (0.01–0.05 g), pH for Pb (II) and for CV-(1–7) and temperature - 293–313 K(±3) in a series of conical flasks. The concentration, pH, contact time and temperature were varied in each experimental study. The influence of pH, contact time, dose and temperature were studied. The initial pH of the adsorbate solutions were adjusted using 0.5 M HCl and 0.5 M NaOH solution. After treatment of adsorbate solution with adsorbent, the solution was filtered using Whatman Filter paper 1. The concentration of adsorbate solutions were then determined using AAS for Pb (II)) and UV–Vis spectrophotometer- for CV ($\lambda_{max} = 582$ nm).

The % removal of adsorbate was evaluated by using Eq. (1).

$$\% = \frac{C_i - C_e}{C_i} X100 \tag{1}$$

The amount of adsorbate per unit mass of the adsorbent was evaluated by using Eq. (2).

$$q_e = \frac{(C_i - C_e)}{m} XV \tag{2}$$

where, q_e (mgg^{-1}) was the adsorption capacity of the adsorbent, C_i and C_e were the initial concentration and concentration at equilibrium t (mgL^{-1}), m was the mass of the adsorbent (g) and V (L) was the initial volume of the adsorbate solution, respectively [2].

2.4.1. Selectivity study

The selective nature of bionanocomposite were studied for Pb (II), Cu (II), Ni (II), Cd (II) and crystal violet dye. The % removal for these pollutants were found to be in the order as follows: Pb (II)-(97%) > Crystal violet dye-(94%) > Cu (II)-(92%) > Ni (II)-(65%) > Cd (60%). Therefore, Pb (II) and crystal violet dye were selected for the detailed experimental study.

2.5. Desorption and regeneration studies

Desorption study is helpful in elucidating the regenerating capability of the bionanocomposite. The eluents both acidic and basic (0.1 N HCl, 0.1 N CH_3COOH and 0.1 N NaOH) were tested for desorption. But the best result for desorption was observed for 0.1 N HCl. This could be explained as, there is high concentration of H_3O^+ (hydronium ion) at low pH that allows the easy removal of Pb(II) and CV dye from the surface of the bionanocomposite by an ion exchange mechanism. The eluent (20 mL) was treated with adsorbate loaded bionanocomposite at room temperature for 24 h. The adsorption-desorption cycles were repeated several times for Pb (II) and CV.

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