



Sol-gel derived xanthan gum/silica nanocomposite—a highly efficient cationic dyes adsorbent in aqueous system



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ABSTRACT

A sol-gel synthesis of nanocomposite adsorbent materials by dehydroxylation condensation of tetraethoxysilane (TEOS) in the presence of xanthan gum (XG) using base catalyst and ethanol as co-solvent is discussed. The synthesized nanocomposite material was evaluated for their ability to adsorb cationic dyes—methylene blue (MB) and bismarck brown Y (BB) from an aqueous solution. The XG/SiO₂ nanocomposite shows the ordered macro- and meso-porous structures and also exhibited large surface area, excellent chemical/mechanical stability and high binding capability for MB and BB dyes. Different parameters such as pH, adsorbent dose and initial concentration were studied. The adsorption behaviour of the nanocomposite was investigated by performing both kinetics and equilibrium studies under batch conditions. The maximum adsorption capacity (Q_{max}) of the nanocomposite for MB and BB was found to be significantly high (432.90 mg g⁻¹ and 448.43 mg g⁻¹, respectively). The time taken for the removal of 99.4% of MB and 87.2% of BB from 100 mg L⁻¹ of dye solutions is only 35 min. Thus, the proposed XG/SiO₂ nanocomposite is a very promising adsorbent for the fast and efficient removal of dye from aqueous solutions.

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1. Introduction

The release of industrial effluents containing pollutants such as dyes into the environment has a destructive impact on living organisms, and it constitutes a global environmental concern [1]. The discharged dyes from different textile industries into water bodies influences the symbiotic processes by affecting photosynthetic activity [2]. Hence, the treatment of polluted, dye-containing water using economically feasible and eco-friendly techniques before being released into the environment is necessary. Of the several available physical, chemical and biological techniques, adsorption is considered as the most effective method for the uptake of dyes from aqueous solution [3,4]. Methylene Blue dye (MB) and bismarck brown Y dye (BB) are selected as our model system due to their similarity i.e both are positively charged azo dyes. MB is a methylthionium chloride azo dye [5] having molecular formula C₁₆H₁₈ClN₃S and molecular weight 319.85 g mol⁻¹. MB is used as a stain for human cheek cells. BB consist of two azo bonds (–N=N–) with a chemical formula and molecular weight of C₁₈H₁₈N₈·2HCl and 419.31 g mol⁻¹ respectively. Like MB, BB is also for biological

analysis. It should be noted that azo dyes constitute over 60% of all dyes produced per year and thus MB and BB are a good representative [6]. Both MB and BB dyes are known to be toxic to humans and ecosystems [4,6,7]. So the treatment of wastewaters containing MB, BB and related dyes has become essential.

It is well known that polysaccharides-based adsorbents are being utilized for the treatment of wastewater from dye industry [8–11]. However, due to the low surface area, thermal stability and hydrodynamic volume as well as their limited ability to form bonds with dyes, the use of polysaccharides-based adsorbents is limited [9]. To improve the extraction properties of the polysaccharides-based adsorbents, nanocomposites-based polysaccharides have been used extensively as an adsorbent in order to produce a synergistic effect of an inorganic filler and polysaccharides matrix [12,13]. In this study, a nanocomposite was synthesized by using xanthan gum (XG) as a template for the in situ formation of the silica nanocomposite. The reaction proceeded via an interaction between the hydroxyl groups present in the XG and the silica surface [14,15]. It is believed that XG works as self-template for the generation of silica nanoparticle through H-bonding and electrostatic interactions, which leads to the formation of xanthan gum/silica nanocomposite (XG/SiO₂).

Recently, Pal et al. [16,17] used polyacrylamide grafted onto the xanthan gum as templates to generate silica nanocomposite for the removal of dyes. However, the monomer modification of the xan-

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than gum in their study increased the cost of synthesis since it is more economical to directly make use of xanthan gum as templates to produce silica nanocomposite for the adsorption of dyes. In this work, we synthesized via a sol-gel method, a XG/SiO₂ nanocomposite adsorbent for the efficient removal of MB and BB dyes. A literature survey has revealed that very few studies have been carried out on the removal of BB dye from aqueous media [18–21]. A high adsorption capacity of XG/SiO₂ nanocomposite for the BB dye, which is due to the electrostatic attraction between the XG/SiO₂ and the dye was achieved.

2. Materials and methods

2.1. Materials

Tetraethylorthosilicate (98% TEOS) was purchased from Sigma–Aldrich. NH₄OH (30% NH₃), xanthan gum, ethanol, sodium hydroxide, hydrochloric acid, methylene blue and bismarck brown Y were purchased from Merck (South Africa), and utilized without further purification.

2.2. Instrument analysis

The FTIR characterization of XG, XG/SiO₂, MB loaded XG/SiO₂ and BB loaded XG/SiO₂ were carried out using Perkin–Elmer PE 1600 FTIR spectrophotometer in KBr pellets. The powder X-ray diffraction patterns of the samples (XG, XG/SiO₂, MB loaded XG/SiO₂ and BB loaded XG/SiO₂) were examined on Rigaku Ultima IV diffractometer utilizing Cu K α radiation (1.5406 Å) operated at 45 kV/40 Ma. A thermogravimetric analyzer (Perkin–Elmer TGA 4000) was used to evaluate the thermal properties of XG and XG/SiO₂. Thermal studies were performed in a nitrogen atmosphere and temperature range of 30–900 °C. The morphology of XG, XG/SiO₂, MB loaded XG/SiO₂ and BB loaded XG/SiO₂ was investigated using a scanning electron microscopy (TESCAN, VEGA SEM) with a 20 kV electron acceleration voltage after the carbon coating of samples. The Micromeritics, ASAP 2020, surface area and porosity analyzer was utilized to determine the pore size, pore volume and specific surface area (BET) of XG and XG/SiO₂ via nitrogen adsorption–desorption measurements. The concentration of the dye was determined using Shimadzu UV-1208 model UV–vis spectrophotometer. The zeta potential of XG/SiO₂ was measured using Zetasizer Nano-ZS, ZEN3600 (Malvern Instruments Limited).

2.3. Synthesis of nanocomposites: xanthan gum-silica nanocomposite (XG/SiO₂)

Three separate solutions were prepared as follows: XG (0.5 g) was dissolved in 10 mL of distilled water, TEOS (2.5 mL) was dissolved in ethanol (2.5 mL) and a third solution incorporating 1.75 mL of 12 N ammonium hydroxide was prepared. The three solutions were thereafter mixed together in a reaction glass flask and kept under tender blending for more than 18 h at room temperature in order to develop monodisperse SiO₂ particles inside of the biopolymer/modified biopolymer medium. The resulting blend was then dissipated in air at 40 °C (4 h), 60 °C (5 h), 70 °C (3 h) and 80 °C for until a dry material, XG/SiO₂ nanocomposite was formed.

2.4. Preparation of control silica oligomer

The method for the synthesis of control silica oligomer is similar to XG/SiO₂ nanocomposite but the control silica oligomer was prepared in the absence of XG.

2.5. MB and BB dye removal study

A batch adsorption method was used to perform the dye adsorption experiments. Adsorption studies were performed by using 100 mL glass bottles immersed in a thermostat water bath shaken at 200 rpm. Adsorption reaction parameters, i.e. pH (2–11) and adsorbent dose (10–70 mg), were optimized for the adsorption of MB and BB using XG/SiO₂ as the adsorbent at 25 °C. The initial dye concentration of 100 mg L⁻¹ and dye solution of 50 mL were used for optimization experiments. Either 1 M NaOH or 0.1 M HCl was used to adjust the pH of the dye solutions. In a typical experiment, 40 mg of XG/SiO₂ was added in a 50 mL dye solution. The samples were shaken on a water bath for a predetermined period of time, filtered and analyzed using a UV–vis spectrophotometer at λ_{max} of 663 nm and 457 nm of MB and BB respectively. The adsorption percentage of the dye was computed by using the following equation

$$\% \text{removal} = \frac{C_0 - C_e}{C_0} * 100 \quad (1)$$

where C_e and C_0 are the respective equilibrium and initial concentrations of dye solution in mg L⁻¹.

Adsorption isotherm experiments were carried out using different concentrations of the dye solution at temperature 25, 35, and 45 °C. Adsorption kinetic studies were operated with two different dye concentrations at 25 °C. The adsorption capacity of XG/SiO₂ for MB and BB was calculated by using the following Eq. (2):

$$q_e = \frac{C_0 - C_e}{m} * V \quad (2)$$

where q_e is equilibrium dye adsorption by XG/SiO₂ (mg g⁻¹); V is the volume of dye solution used (L) and m is the weight of XG/SiO₂ used.

3. Results and discussion

3.1. Synthesis of XG/SiO₂ nanocomposite

The in-situ formation of XG/SiO₂ nanocomposite essentially involves two important steps; the initial step begins with the hydrolysis of TEOS, and the condensation of the resulting silanols (Si–O–H) to form oligomers that resulted in sol particles [22–24]. The sol particles were cross-linked through sol–gel transition in the presence of co-solvent (ethanol) and base catalyst. In this step, the anionic-XG increases the rate of the sol–gel procedure and also acts as a template for the nucleation and growth of SiO₂ nanoparticles (NPs) [22]. Furthermore, the silanol groups is hydrolyzed and thereafter take part in the poly-condensation reaction to create nanoscale silica particles on the biopolymer surface through electrostatic interactions between the polymer and the SiO₂ fillers [23]. In this manner, the anionic-XG helps the formation of SiO₂ NPs by increasing the rate of the sol–gel process.

3.2. FTIR characterization

The infrared spectra of XG, XG/SiO₂, MB loaded XG/SiO₂ and BB loaded XG/SiO₂ are presented in Fig. 1. In the spectrum of XG, the peaks appearing at 2911 and 3427 cm⁻¹ correspond to stretching vibrations of C–H and O–H respectively [25]. The two peaks at 1617 cm⁻¹ and 1413 cm⁻¹ are assigned to stretching vibrations of the COO⁻ group [26,27]. The presence of a peak at 1053 cm⁻¹ is attributed to the acetal group and the peak at 1734 cm⁻¹ is due to the C–O axial deformation. The ??-glycosidic linkages are evidenced by the presence of the peak at 887 cm⁻¹ [26]. The formation of silica can be identified from the peak at 469 cm⁻¹, which corresponds to the vibration of the Si–O–Si bonds. The peak at 800 and 625 cm⁻¹ are due to vibrations of tetrahedrons SiO₄ [27], which

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