

Xanthan gum-cl-poly (acrylic acid) / Reduced Graphene Oxide Hydrogel Nanocomposite as Adsorbent for Dye Removal

Edwin Makhado, Sadanand Pandey* , Philiswa Nomngongo ' and James Ramontja

Abstract— In the present work, we have synthesized Xanthan gum-cl-poly (acrylic acid) / Reduced Graphene Oxide hydrogel nanocomposite as adsorbent via microwave assisted copolymerization of acrylic acid (AA) onto xanthan gum (XG) backbone. MBA and APS were used as crosslinker and initiator, respectively. Reduced Graphene Oxide (rGO) was incorporated into the hydrogel matrix during the grafting reaction. The infrared spectra (FTIR) and Scanning electron microscopy (SEM) were used to verify the adsorbent formed under optimized reaction conditions. XG-cl-pAA/rGO hydrogel nanocomposite was used for adsorption of methylene blue (MB) from aqueous solutions. The factors influencing adsorption capacity of the adsorbents such pH of the initial dye solution, dose of adsorbent, contact time, and initial dye concentration were investigated via a batch adsorption system. The maximum adsorption capacity (qm), calculated based on the Langmuir isotherm for MB was 526.3 mg g⁻¹ at 30°C. The adsorption kinetics and isotherms were found to follow Pseudo-second-order kinetic model and Langmuir model, respectively.

Keywords— Xanthan gum, hydrogel composite, Microwave irradiation, Adsorption; Isotherm

I. INTRODUCTION

With nanotechnology, a large set of materials and improved products rely on a change in the physical properties when the feature sizes are shrunk. These nanomaterials are used in many applications such as sensors, water purification, antimicrobial, catalysis, biomedical, agricultural etc. [1-16] One of the most important application of nanomaterials in water purification are discuss in the present article. Due to the steady expansion of industrialization, the excessive release of various types of pollutants into water sources is one of the common problems global. For example, organic dyes are one of the most hazardous pollutants in industrial effluents that are discharged from

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various industries such as textiles, leather, food, pharmaceuticals, cosmetics and paper.

The presence of organic dyes water systems can be carcinogenic to animals and humans [17, 18]. A number of techniques have been applied for the removal of organic dyes from aqueous medium [19-21]. The adsorption is globally recognized as the most promising technique for the removal of organic dyes from the wastewater due to its cost-effective nature [22-24]. In this direction, the use of a low cost hydrogel based on biopolymer as an adsorbent for adsorption of organic dyes received widespread attentions.

Xanthan gum (XG) is one of the natural polymers that have attracted considerable attention both in academic world and industrial. XG is efficient polyelectrolyte due to the presence of tunable hydroxyl (-OH) groups. It has been used widely in pharmaceutical and cosmetic industry as suspending agent and as additive in food, where it serves as a thickening agent [25]. XG is an anionic polysaccharide derived from *Xanthomonas campestris*. The structural unit of XG consists of backbone of β -(1 \rightarrow 4)-linked- D-glucofuranose glucan and alternate glucoses have a short branch consisting of a glucuronic acid sandwiched between two mannose units. The backbone of the XG is similar to the chemical structure of cellulose. The side chains are consists therefore of β -(1 \rightarrow 3)- α -linked D-mannopyranose-(1 \rightarrow 2)- β -D-glucuronic acid-(1 \rightarrow 4) β -D- mannopyranose on alternating residues. The terminal D-mannose contains a pyruvic acid residue linked via keto group to the 4 and 6 positions. The D-mannose unit linked to the main chain contains an acetyl group at position O-6 [26].The molecular structure of xanthan gum repeating unit (C₃₅H₄₉O₂₉,) is displayed in (figure 1).

Hydrogels are regarded as as hydrophilic, three-dimensional (3D) network with ability to swell and retain a significant fraction of water within its structure without dissolving in water. Biopolymer-based hydrogels have attracted intense interest in wastewater treatment owing to their high adsorption property, recovery capacities and regeneration for repeated recycles. However, hydrogels crosslinked by the conventional crosslinking agents have poor mechanical strength. To enhance the mechanical, sorption capacity and other properties of hydrogels an inorganic component such carbon-based, montmorillonite, polymeric, ceramic, and metallic nanomaterials [27-31] are incorporated in the hydrogel

polymeric matrix. For example, Huang et al., reported the incorporation of graphene oxide (GO) into polymer hydrogels to enhance their ability to retain a large amount of water [32]. In another study, Sui et al., synthesized rGO-based hydrogels and the prepared rGO hydrogel exhibit excellent mechanical and electrical properties [33]. To the best of our knowledge, no substantial work has been reported related to the adsorption performance of hydrogels with rGO can be found in literature so far. The aim of this study was to investigate the efficiency of the microwave-assisted XG-cl-pAA/rGO hydrogel composite in the removal of MB from the aqueous solutions. The equilibrium removal efficiency was studied using isotherm models and kinetic parameters of the adsorption process.

homogenously dissolve in a 100 mL open beaker containing DI. Calculated amount of AA, MBA were added and APS was added in order to initiate the reaction of graft copolymerization. Then 80 mg rGO was dispersed in 5 mL of deionized water, and then sonicated for 5 min by using ultrasonicator, the rGO solution was added to the graft copolymerization reaction in a 100 mL open beaker. The beaker was exposed under fixed microwave power for a definite time period in a domestic microwave oven with a microwave frequency of 2450 MHz. After desired time period, the XG-cl-pAA/rGO hydrogel composite was precipitated by pouring the reaction mixture into a large quantity of acetone and washed well to remove adhered homopolymer, if any is present along with graft copolymers. The precipitated copolymer was filtered and the copolymer samples obtained were finally dried under vacuum at 60 °C for >24

C. Adsorption Studies

MB sorption investigations were performed by the batch method. Adsorption examinations were carried out using XG-cl-pAA/rGO hydrogel composite as adsorbents on a temperature controlled incubator shaker set at 180 rpm kept up at 30 °C for 50 min. Here, known measures of adsorbents were completely mixed with 20 mL of individual MB solutions, whose concentrations and pHs were beforehand known. After the PE plastic bottles were shaken for the desired time, the suspensions were filtered through 0.45 μm PVDF syringe filters. The concentration of the unadsorbed dye left behind in each solution was analyzed using a UV/vis spectrophotometer (Shimadzu UV-1208 models) at the λ_{max} of 662 nm for MB. The equilibrium uptake was calculated using Equation. (1):

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

where q_e is the equilibrium capacity of dye on the adsorbent (mgg^{-1}), C_0 denotes the initial and the C_e denotes the equilibrium concentrations (mgL^{-1}) of MB, respectively. V is the volume of dye solution used (L) and W is the weight of adsorbent (g) used. All the batch experiments were carried out in triplicate and results represented here are the average of three readings.

III. CHARACTERIZATION

LG (Model No. MS-283MC; 1300 W, Made: Korea) domestic microwave oven having 2450 MHz microwave frequency and a power output from 0 to 900 W was used for synthesis of hydrogel and hydrogel nanocomposite. The pH of the reaction mixture was adjusted using HCl or NaOH (0.1 M). The pH measurements were made with HI 9811-5/HI 1285-5 (Romania). FTIR spectra were recorded on a Spectrum-100 Perkin Elmer, USA, in the spectral range of 4000 to 400 cm^{-1} with a resolution of 4. The samples were compressed into pellets using spectroscopic grade KBr (Sigma-Aldrich, South Africa). The surface morphologies of the samples were examined by a scanning electron microscopy (SEM), (TESCAN, VEGA SEM) under a 20 kV electron acceleration voltage. To avoid charging these samples were coated with carbon.

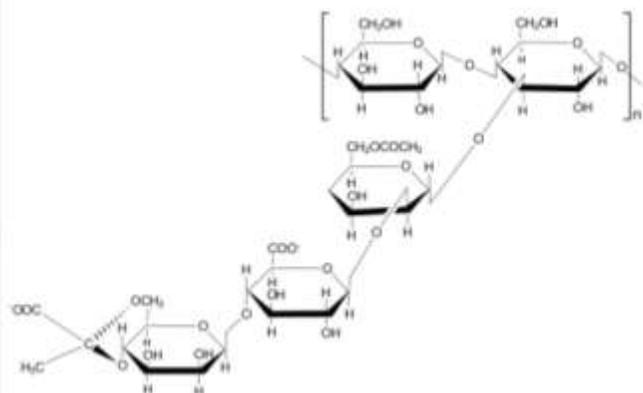


Fig. 1. The molecular structure of xanthan gum.

II. MATERIALS AND METHOD

A. Materials

The biopolymer, xanthan gum (XG) from *Xanthomonas campestris* was supplied by Sigma-Aldrich (South Africa). Poly acrylic acid (pAA, 99%) monomer was obtained from Sigma-Aldrich (Netherlands) and acetone was procured from Merck (South Africa) and was used without further purification. Initiator ammonium persulfate (APS) ($\geq 98\%$; 248614), the cross linker N, N'-methylene bis-acrylamide (MBA), 99%, Methylene blue (MB) cationic dye, were obtained from Sigma-Aldrich (South Africa) and used without further purification. Sodium hydroxide (NaOH) and Hydrochloric acid (HCL) were procured from Merck (South Africa). All reagents used were of analytical grade. For all the experiments, deionized (DI) water was used. The stock solution of MB (1000 mg L^{-1}) was prepared by dissolving an appropriate amount of dye in 1 L of deionized water, and the stock solution was further diluted for batch experiments.

B. Synthesis of XG-cl-pAA/rGO Hydrogel Composite

Reduction of graphene oxide (rGO) was adapted from [34]. A polymer matrix composed of xanthan gum-cl-poly (AA) was prepared by using MBA as crosslinker and APS as initiator in a domestic microwave. The grafting of poly acrylic acid (pAA) onto (XG) in the presence of MBA cross linker by free radical co-polymerization technique. XG (0.1 g) was dissolved was

IV. RESULTS AND DISCUSSION

A. Characterization of XG and XG-cl-pAA/rGO

FTIR-spectroscopy

The structural changes of XG and XG-cl-pAA/rGO hydrogel composite were confirmed by FTIR spectroscopy. As shown from (figure 2a) the characteristic bands at 3246 cm^{-1} , 2932 cm^{-1} , and 1404 cm^{-1} due to the characteristic stretching vibration of both primary and secondary O-H bonds, -CH stretching of alkyl group, and at 1404 cm^{-1} due to CH bending of methyl group in XG, respectively. Additional characteristic absorption bands of the polysaccharide appear at 1023 cm^{-1} due to stretching of the C O bond. In the cases of XG-cl-pAA/rGO hydrogel composite, the intensity of characteristic band at 1404 cm^{-1} was increased, which is attributed to the symmetric stretching mode of the carboxylate group confirms the formation of XG-cl-pAA/rGO hydrogel composite. Furthermore, a hump at 3142 cm^{-1} appeared in a broad absorption peak of XG after the surface-modification with AA. These results suggest that the copolymer of AA had been successfully grafted onto the XG. Additional peaks at 1537 cm^{-1} related to the C=O asymmetric stretching of the carboxylate anions in the AA units were observed.

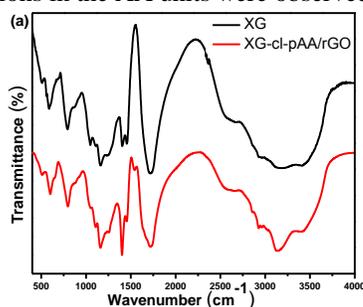
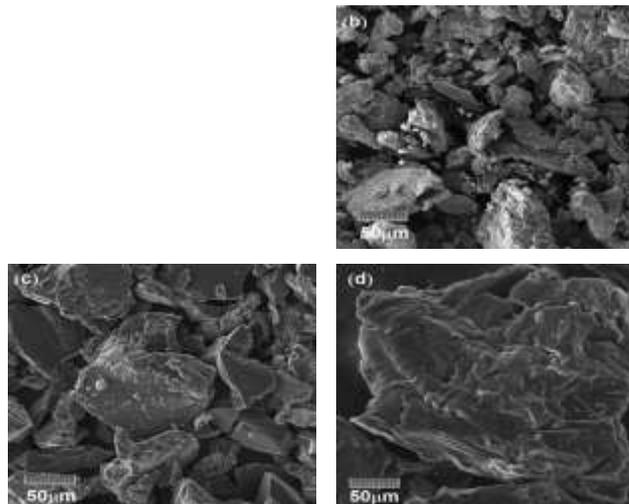


Fig 1. (a) Shows the FT-IR spectra of the XG and XG-cl-pAA/rGO; SEM image resolution at 600x of (b) XG; (c) XG-cl-pAA/rGO and (d) MB loaded XG-cl-pAA/rGO.

Surface Morphologies Characterization

The surface morphologies of the XG and XG-cl-pAA/rGO are depicted in (figure 2b-c). SEM micrographs of the XG at 600x (figure 2b) shows the granular morphology which suggests the amorphous nature of the biopolymer [35]. The SEM of XG-cl-pAA/rGO exhibits peeled smooth combined with rough uneven surface morphology shown in (figure 2c). The peeled smooth and rough uneven surface morphology observed for XG-cl-pAA/rGO hydrogel nanocomposite seem to have been covered by MB molecules after the adsorption MB onto the XG-cl-pAA/rGO hydrogel nanocomposite (figure 2d).



B. Optimization of MB onto XG-cl-pAA/rGO hydrogel nanocomposite

Effect of pH on the Adsorption of MB

Figure 3a shows the effect of pH value of dye solution on the adsorption capacities of the XG-cl-pAA/rGO hydrogel nanocomposite. It was found that as the pH values increased from 1 to 5, the adsorption capacities of the XG-cl-pAA/rGO hydrogel nanocomposite sharply increased from 53.4 to 99.8 mgg^{-1} , and then tend to leveling off with further increase in pH. Increasing the pH of solution, deprotonation of XG-cl-pAA/rGO derivative is realized and strong attractive forces, between the positive charged dye and negatively charged XG-cl-pAA/rGO, which is favorable to increase the absorption for MB.

Effect of Adsorbent Dose

Figure 3b demonstrates the effect of XG-cl-pAA/rGO hydrogel nanocomposite dosage on the removal of MB from aqueous solution. The percentage adsorption of MB was studied at various amounts of hydrogel and hydrogel nanocomposite samples in the range of 10 to 90 mg with an increment of 20 mg. This result can be explained on the basis of higher surface area of the adsorbent as well as availability of more adsorption sites.

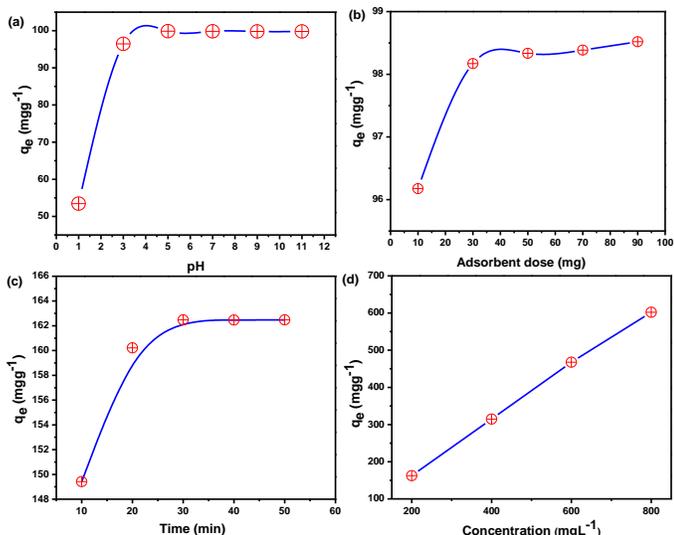


Fig. 3. Effect of (a) pH; (b) adsorbent dose; (c) time on MB dye adsorption; and (d) Concentration on MB dye adsorption capacity in (mg g⁻¹).

C. Kinetic Models

Pseudo-first-order and the pseudo-second order equations MB removal by XG-cl-pAA/rGO hydrogel nanocomposite as a function of contact time was measured and the results are shown in (figure 3c). The kinetic parameters obtained at 200 mgL-1 concentration of MB are illustrated in (Table 1) The pseudo-first-order kinetic model of Langergren (Equation.(2)) [36] and the pseudo-second order kinetic (Equation.(3))[37] models were most often used to govern the rate constant and to examine the mechanism of the adsorption process. Their linear forms can be expressed as:

$$\frac{\log(q_e - q_t)}{q_e} = \log q_e - \frac{k_1}{2.030} t \tag{2}$$

$$\frac{t}{q_t} = \frac{1}{k'q_e^2} + \frac{t}{q_e} \tag{3}$$

Where q_e (mg g⁻¹) is the adsorption capacity of XG-cl-pAA/rGO hydrogel nanocomposite in equilibrium; K₁ (min⁻¹) is the rate constant of the pseudo-first-order model; and K' (g mg⁻¹ min⁻¹) is the rate constant of the pseudo-second-order model. The experimental data in were fitted linearly by using Equation (2) and (3). These results indicate that the pseudo-second-order kinetic model gave a better correlation for the adsorption of MB on XG-cl-pAA/rGO hydrogel nanocomposite compared to the pseudo-first-order model. Furthermore, this result suggests that the chemical interaction between the MB and the XG-cl-pAA/rGO hydrogel nanocomposite surfaces is dominated the adsorption process.

TABLE I: KINETIC PARAMETERS FOR MB ADSORPTION BY XG-CL-PAA/RGO HYDROGEL NANOCOMPOSITE.

Pseudo first order		
qm (mg/g)	k1 (min ⁻¹)	R ²
12.08	0.0138	0.957

Pseudo second order		
qe (mg/g)	k2 (g (mg min) ⁻¹)	R ²
166.66	6.92x10 ⁻³	0.999

D. Equilibrium models

Langmuir isotherm

The Langmuir isotherm theory infers monolayer coverage of adsorbate over a homogenous adsorbent surface [38]. The equilibrium adsorption data were generally interpreted using Langmuir and Freundlich isotherm models. The isotherm constants for these models were calculated by linear regression method and given in (figure 3d). Langmuir isotherm can be given as Equation. (4) as follows

$$q_e = \frac{q_m b C_e}{1 + b C_e} \tag{4}$$

When linearized, Equation (5) becomes:

$$\frac{C_e}{q_e} = \frac{1}{q_m b} + \frac{1}{q_m} C_e \tag{5}$$

Where C_e is the equilibrium concentration (mg L⁻¹) and q_e the amount adsorbed at equilibrium (mg g⁻¹). The Langmuir constants q_m (mg g⁻¹) represent the monolayer adsorption capacity and b relates the heat of adsorption. The linear plots of C_e/q_e versus C_e at 30 °C are summarized in (Table.2).

The R_L a dimensionless constant referred to as separation factor. R_L is calculated using the following Equation (6):

$$R_L = \frac{1}{1 + b C_0} \tag{6}$$

The R_L values found in the present study were in the range of 0.0957-0.0258 indicating that adsorption of MB by XG-cl-pAA/rGO hydrogel nanocomposite was favorable (0 < R_L < 1).

Freundlich isotherm

The Freundlich equilibrium isotherm equation [39] is used for the description of multilayer adsorption with interaction between adsorbed molecules. The Freundlich isotherm is generally expressed as Equation (7) as follows:

$$q_e = K_f C_e^{1/n} \tag{7}$$

The linear expression takes the following form Equation (8)

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \tag{8}$$

Where, q_e is the adsorbed amount at equilibrium (mol g⁻¹), K_f the Freundlich equilibrium constant (mol g⁻¹)/(mol L⁻¹)^{1/n}, n is indicative of the energy or intensity of the reaction and suggests the favourability and capacity of the adsorbent/adsorbate system. To determine the constant K_F and n, may be used to plot ln q_e against ln C_e at 30 °C and the results were illustrated (Table.2). On the basis of the obtained results, the adsorption process was well described Langmuir isotherm model which assumes the mono-layer adsorption of the MB on the surface of XG-cl-pAA/rGO hydrogel.

TABLE II: PARAMETERS FOR MB ADSORPTION BY XG-CL-PAA/RGO HYDROGEL NANOCOMPOSITE ACCORDING TO DIFFERENT EQUILIBRIUM MODELS.

Langmuir isotherm constants			
qm (mg/g)	R _L	b	R ²
769.23	0.0957-0.0258	0.047	0.995
Freundlich isotherm constants			
n	K _F	R ²	
2.08	1.8935	0.989	

V. CONCLUSIONS

In the present study, XG-cl-pAA/rGO hydrogel nanocomposite was successfully synthesized by microwave irradiation method. This hydrogel nanocomposite was effectively used for the removal of MB dye from the aqueous medium. The adsorption was immensely dependent upon various parameters such as pH, adsorption dose, concentration, and time. On the basis of the obtained results, the adsorption process was well described by Pseudo second-order kinetic model and by Langmuir isotherm model, showing the chemical complexation between MB ions and -COO⁻ was mainly responsible for high adsorption capacity. The maximum adsorption capacity of MB onto XG-cl-PAA/rGO hydrogel nanocomposite was 769.23 mg g⁻¹ at 30°C. This study revealed that XG-cl-pAA/rGO hydrogel nanocomposite is a potential candidate for the removal of MB dye from aqueous medium.

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