

Efficient and Rapid Adsorption Characteristics of Templating Xanthan Gum-Graft-Poly (Aniline) and Silica Nanocomposite toward Removal of Toxic Methylene Blue Dyes

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Abstract—Conducting polymer composites with micro/nanostructures have attracted significant academic and technological attention because of their unique physical properties and potential applications in nanoelectronics, electromagnetics, and biomedical devices. This paper deals with application of sol-gel synthesis of xanthan gum-graft-poly (aniline)/silica (XG-g-PANi/SiO₂) hybrid nanocomposite toward the rapid removal of methylene blue dyes from aqueous solution. XG-g-PANi being act as a novel template for nanosilica formation. The detailed investigation of the adsorption isotherms of methylene blue dyes from aqueous solution showed that the dyes adsorb in accordance with a Langmuir adsorption isotherm. The results indicate that xanthan gum-graft-poly (aniline)/silica (XG-g-PANi/SiO₂) hybrid nanocomposite can be used as an effective adsorbent for removal of dyes from textile effluents.

Highlights ► Xanthan gum-graft-poly (aniline)/silica hybrid nanocomposite – an eco-friendly polymer matrix for the treatment of dye effluents. ► The nanocomposite decolorized 99% of dye bath effluent. ► The removal of dyes is largely depending on the solution pH. ► The removal process followed Langmuir isotherm.

Keywords—Biopolymer; polyaniline; template; nanocomposite; sol gel; adsorption; dye removal.

I. INTRODUCTION

Nowadays, synthetic dyes have been increasingly used in the textile, paper, rubber, plastic, cosmetics, pharmaceutical and food industries because of their ease of use, inexpensive cost of synthesis, stability and variety of colour compared with natural dyes [1,2]. Methylene blue and methyl orange are highly toxic, persistent, carcinogenic, and mutagenic in nature [3,4]. By virtue of their cationic/anionic as well as aromatic nature they are easily soluble in an aqueous/alcoholic medium and usually

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generate sulphur/nitric oxides at high temperature. As a result of the reduction process, these dyes reduce the dissolved oxygen, which modifies the properties as well as characteristics of aqueous fluids and can cause severe adverse health effects such as breathing difficulties, nausea, vomiting allergic dermatitis, skin irritation, cancer, and mutations [5]. Clean, safe and adequate freshwater is crucial to all living organisms and the normal functioning of ecosystems, communities and economies. Therefore, exploitation of safe water sources to overcome the scarcity of water has been a global challenge for many countries. The increasing demand of clean water has attracted much of the attention of government organizations and water industries to develop cost-effective technologies for water/wastewater treatment.

In this work, we have chosen methylene blue dye as a test probe for remedial experiments. Methylene blue dye can be used in many applications such as disinfectant in dye stuffs and a colouring material in paper, temporary hair, cottons, wools and other textile items but its harmful effects cannot be ignored. It is reported to have some harmful effects in human beings such as cyanosis, jaundice, vomiting, tissue necrosis, heartbeat imbalance etc. [6]. There are so many methods used for removal of dyes such as coagulation and flocculation, chemical reduction, advanced oxidative processes, ozonization, membrane separation, and ultra-filtration and electro-precipitation techniques [7, 8].

However these methods are not economical and are often unable to adequately reduce contaminants concentrations to desired levels. A search is on more effective and economic treatment techniques. The adsorption process provides an attractive alternative treatment, especially if the adsorbent is inexpensive and readily available [9-23].

Nanomaterials are playing a very important role in many fields such as sensor, drug delivery, catalysis, antimicrobial activity, wastewater treatments etc [24-36]. Thus Conducting polymer composites are nowadays used and proved to be more efficient towards dye removal [37]. Polyaniline coated onto sawdust (termed as PAN/SD) for removal of methylene blue dye from aqueous solutions [38]. Polyaniline–chitosan composite is used not only for fluoride ion adsorption but also for the removal of sulphonated dyes [39]. Polyaniline–silica composite has been used for removal of Acid green dyes from aqueous

solution [40]. Cellulose/PANI composite has been used for removal of Cr^{6+} [41].

To the best of our knowledge, this is the first time that such type of polymer composite based metal oxide will be employed for the removal of methylene blue from aqueous solution. The novelty of the work is in the achievement of almost 100% adsorption of methylene blue onto this newly synthesized nanocomposite. The synthesis of xanthan gum-graft-poly (aniline)/silica nanocomposite affords electrostatic charges that result in an increased hydrodynamic volume and enhanced electrostatic attraction with cationic dyes, resulting in a significantly higher adsorption efficiency. The nanocomposites obtained show rapid adsorption and excellent adsorption efficiencies for uptake of methylene blue dye from aqueous solution, which improves the performance beyond the state of the art reported in the literature.

II. MATERIALS AND METHOD

A. Materials

The biopolymer, XG from *X. campestris* (G1253, Sigma), monomer, aniline ($\geq 99.5\%$, Sigma-Aldrich; 242284), initiator, APS ($\geq 98.0\%$, Sigma-Aldrich; 248614), solvent, 1-methyl-2-pyrrolidone (NMP) (Merck; 806072), hydrochloric acid (32% Merck, SA; 100319), Ammonium hydroxide solution (32.0%, Sigma-Aldrich; V000637), Tetraethylorthosilicate (98%, Sigma-Aldrich; 131903), ammonium hydroxide (30%, NH_3 , Merck, 105423), sodium hydroxide (Merck, SA; 106469) and ethanol (99.9% pure, Merck, SA; 102428) and the dyes MB (λ_{max} , 662 nm, Merck, SA; 159270) were used. MB is a heterocyclic aromatic chemical compound has the molecular formula $\text{C}_{16}\text{H}_{18}\text{N}_3\text{S}\text{Cl}$ and the molecular structure is shown in Fig. 1.

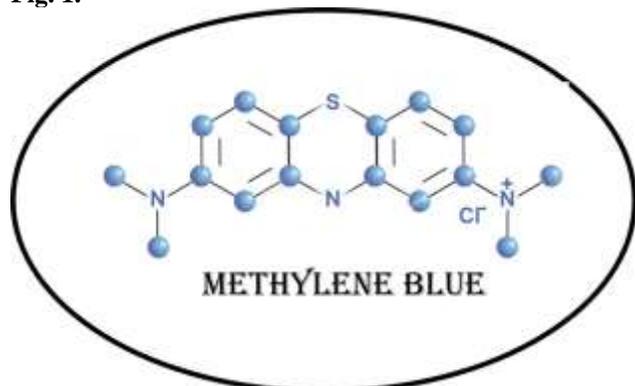


Fig 1. Shows the molecular structure of methylene blue dye

B. Graft copolymerization method for synthesis of mwXG-g-PANi composite

XG-g-PANi was used as synthesized earlier [32], where XG (2g/L) was dissolved in 25 mL deionized (DI) water. A 0.05M amount of aniline (ANi) and 0.15M of hydrochloric acid (HCl) solutions were included in the beaker. Further catalytic amount (0.045M) of ammonium peroxydisulphate (APS) was added in order to initiate the reaction of graft copolymerization. Further, the reaction mixture was exposed to microwave irradiation at microwave power (80%) and exposure time (50s). After desired time period, the grafted sample was precipitated by pouring the

reaction mixture into the NMP. After sought time period, the copolymer were dried in a vacuum oven at 60 °C and weighed to obtain the XG-g-PANi of 172 %G.

C. Sol gel method for the synthesis of mwXG-g-PANi/SiO₂ nanocomposites

The nanocomposites were synthesized in situ by sol gel method. Three separate solutions were prepared as follows: XG-g-PANi (0.5 g) was dissolved in 20 mL of distilled water, TEOS (2.5 mL) was dissolved in ethanol (1.25 mL) and a third solution incorporating 0.85 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 12 hours at 40 °C in order to develop monodisperse SiO₂ particles inside of the biopolymer/modified biopolymer medium. The resulting blend was then dissipated in air at 60 °C for 3 h and then 80 °C for about 2 h until a dry material, XG-g-PANi/SiO₂ nanocomposites was formed.

D. Adsorption Studies.

Methylene blue sorption investigations were performed by the batch method. Adsorption examinations were carried out using XG-g-PANi/SiO₂ nanocomposites as adsorbents on a temperature controlled incubator shaker set at 180 rpm kept up at 325K for 45 min. Here, known measures of adsorbents were completely mixed with 30 mL of individual methylene blue 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):

$$qe = (Co - Ce) \times \frac{V}{W} \dots \dots \dots (1)$$

where q_e is the equilibrium capacity of dye on the adsorbent (mgg^{-1}), C_o denotes the initial and the C_e denotes the equilibrium concentrations (mgL^{-1}) of methylene blue, 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, Korea) domestic microwave oven having 2450 MHz microwave frequency and a power output from 0 to 900 W was used for synthesis of mwXG-g-PANi composite. 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). The powder X-ray diffraction patterns of the samples (XG, of mwXG-g-PANi, mwXG-g-PANi /SiO₂ and MB loaded of mwXG-g-PANi /SiO₂) were examined on Rigaku Ultima IV diffractometer utilizing Cu K α radiation (1.5406 Å) operated at 45 kV. 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. The concentration of the dye was determined using Shimadzu UV-1208 model UV-vis spectrophotometer.

IV. RESULTS AND DISCUSSION

A. Characterization

Sol-gel method for the synthesis of mwXG-g-PANi/SiO₂ nanocomposites take place according to the established sequences of (i) the initial hydrolysis of TEOS, (ii) the condensation of silanol groups to afford oligomers assembled as sol particles and finally (iii) the cross linking of sol particles to a sol-gel transition. The precursor medium accelerates the sol-gel process in the presence of mwXG-g-PANi. Here, XG-g-PANi acts as a template for the nucleation and growth of a SiO₂ shell because of the H-bonding between the -COO⁻/-CONH₂ groups of the surface of modified XG and hydroxyl groups at the SiO₂ nanoparticle surface.

Figure.2 illustrates the XRD patterns of mwXG-g-PANi/SiO₂ nanocomposites. XG demonstrates a typical amorphous pattern (Figure not shown), while on account of the mwXG-g-PANi, the XRD pattern demonstrates the semi crystalline structure (Figure not shown)[33]. In the XRD pattern of mwXG-g-PANi, Bragg diffraction peak at $2\theta = 16.30^\circ$, 20.12° and 25.56° correspond to the (011), (020) and (200) crystal planes of orthorhombic crystalline PANI in its emeraldine salt form, respectively [42]. The peak at 20.12° is related to the repeat units of the polyemeraldine chain and the periodicity parallel to the polymer chains of PANI. The peak at 25.56° is owed to the periodicity in the direction perpendicular to the polymer chain [43]. These typical diffraction peaks confirm that the PANI is highly crystalline. By and large polymers are thought to be amorphous however PANI is indicating crystalline structure due to its fiber nature and planar nature of benzenoid and quinoid functional groups. While in case of mwXG-g-PANi/SiO₂ nanocomposites shown in **Figure.2**. It can be seen clearly that the silica possesses a broad diffraction peak at about 21.9° , indicating that the mesoporous silica is amorphous. Furthermore, there is no diffraction peak of mwXG-g-PANi, emerging in the patterns of mwXG-g-PANi/SiO₂ nanocomposites, confirming that the PANI is also amorphous and has been encapsulated in the pores and channels of the silica; and the crystallization of PANI is impeded owing to the confinement of silica framework.

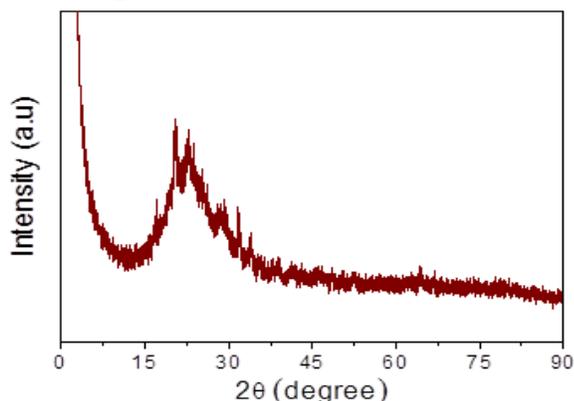


Fig.2 X-ray diffraction pattern of mwXG-g-PANi/SiO₂ nanocomposites.

Scanning electron microscopy is widely used to study the morphological features and surface characteristics of adsorbent. XG has smooth regular surface morphology (Figure not shown). The internal pores is generated and roughness of surface is increased after the formation of silica particle by modify XG in case of mwXG-g-PANi/SiO₂ nanocomposites (**Figure 3**). SEM images of mwXG-g-PANi/SiO₂ nanocomposites at two different magnification (500x and 1kx) are shown in Figure. 3.

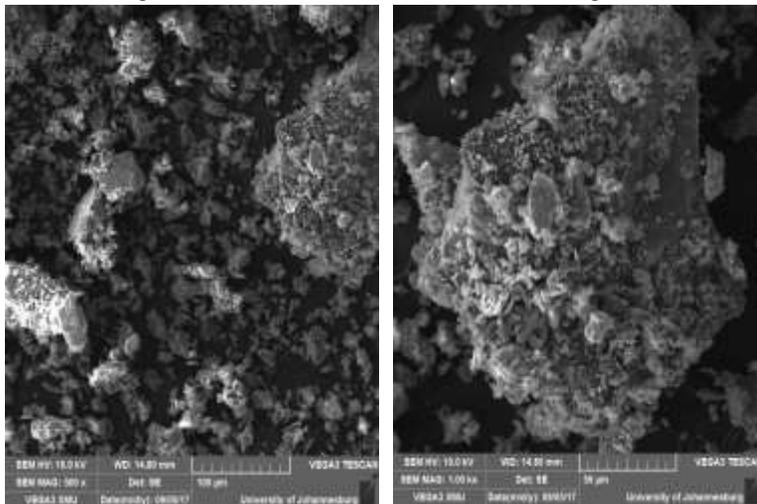


Fig.3 SEM image of mwXG-g-PANi/SiO₂ nanocomposites at two different magnification (500x and 1kx)

B. Sorption of Methylene blue dye by mwXG-g-PANi/SiO₂ Nanocomposites

Effect of pH

A series of experiments has been performed to optimize the adsorption conditions for removal of methylene blue dye using the mwXG-g-PANi/SiO₂ nanocomposites. The pH of an aqueous medium is an important factor that may influence the uptake of the many adsorbates such as dyes, so the influence of pH on dye adsorption by the mwXG-g-PANi/SiO₂ nanocomposites adsorbent was studied. The other condition such as methylene blue dye concentration: 300 ppm, reaction volume: 30mL, adsorbent dose: 0.04g, contact time: 45min, adsorption temp= 25°C was kept constant.

It was observed that increasing solution pH increases the extent of dye removal. Lower adsorption percentage of MB on mwXG-g-PANi/SiO₂ nanocomposites at highly acidic conditions (pH 2) is probably due to the presence of high concentration of H⁺ ions on the surface of adsorbent competing with methylene blue (a cationic dye) for adsorption sites in the adsorbent. With an increase in the solution pH 9, the electrostatic repulsion between the positively charged methylene blue and the surface of adsorbent is lowered. Consequently removal efficiency is increased.

Effect of adsorbent dose

For investigating the effect of adsorbent mass on the adsorption of methylene blue dye, a series of adsorption experiment was carried out with different adsorbent dosages (0.010-0.045 g). The results follow the expected pattern, in which the percentage sorption increased from 42% to 99% as the sorbent dose was increased over the range 0.01 – 0.04 g

(figure not shown). This is as a result of increased surface area and availability of more adsorption sites.

Effect of contact time

The effect of period of contact on the removal of methylene blue dye by the adsorbents was determined by keeping other conditions (particle size, initial concentration, dosage and pH) constant at the optimum. The effect of contact time was investigated by treating 0.04 g of the adsorbents mwXG-g-PANi/SiO₂ nanocomposites and with 30 mL of 300 mg L⁻¹ methylene blue dye solution at pH value of 9. The mixture was agitated in a mechanical shaker for different periods of contact time (5-50 minutes). It was observed that the rate of removal of MB dye increases from 64% to 99% with increase in contact time from 5 min to 45 min to some extent (figure not shown). Further increase in contact time does not increase the uptake due to deposition of dyes on the available adsorption site on adsorbent material. As the data show the sorption process was rapid for mwXG-g-PANi/SiO₂ nanocomposites.

C. Equilibrium Models

Langmuir isotherm

The Langmuir isotherm theory infers monolayer coverage of adsorbate over a homogenous adsorbent surface [44]. 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 4). Langmuir isotherm can be given as Equation. (2) as follows

$$q_e = \frac{q_m b C_e}{1 + b C_e} \dots \dots \dots (2)$$

When linearized, Equation (3) becomes:

$$\frac{C_e}{q_e} = \frac{1}{q_m b} + \frac{1}{q_m} C_e \dots \dots \dots (3)$$

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 25 °C are summarized in (Table.1).

The R_L a dimensionless constant referred to as separation factor. R_L is calculated using the following Equation (4):

$$R_L = \frac{1}{1 + b C_0} \dots \dots \dots (4)$$

The R_L values found in the present study were in the range of 0.2557-0.0467 indicating that adsorption of methylene blue dye by mwXG-g-PANi/SiO₂ nanocomposites was favorable (0 < R_L < 1).

The 1.1 plot of C_e/q_e versus C_e Fig. 4 gave straight lines for all the concentrations, implies that the adsorption for adsorbent well fitted to Langmuir isotherm. The Langmuir adsorption capacity was found to be 1250 mg/g for methylene blue dye onto mwXG-g-PANi/SiO₂ nanocomposites at 25°C. The high correlation coefficient obtained for mwXG-g-PANi/SiO₂ nanocomposites (R²= 0.99) indicates high affinity between adsorbent surface and methylene blue dye which plays the major role in the adsorption mechanism.

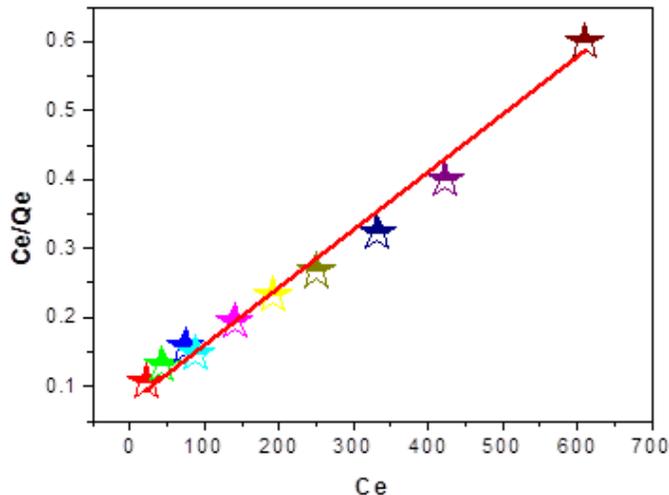


Fig. 4 Graph of Langmuir adsorption Isotherm

Freundlich isotherm

The Freundlich equilibrium isotherm equation [45] is used for the description of multilayer adsorption with interaction between adsorbed molecules. The Freundlich isotherm is generally expressed as Equation (5) as follows:

$$q_e = K_F C_e^{1/n} \dots \dots \dots (5)$$

The linear expression takes the following form Equation (6)

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \dots \dots \dots (6)$$

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 25 °C and the results were illustrated (Table.1).

TABLE 1. PARAMETERS FOR METHYLENE BLUE DYE ADSORPTION BY BY MWXG-G-PANI/SIO₂ NANOCOMPOSITES TO DIFFERENT EQUILIBRIUM MODELS.

Langmuir isotherm constants			
qm (mg/g)	R _L	b	R ²
1250	0.2557-0.0467	0.0097	0.99

Freundlich isotherm constants		
n	K _F	R ²
1.93	50.35	0.95

From the high correlation coefficient obtained for mwXG-g-PANi/SiO₂ nanocomposites (R²= 0.99) indicates high affinity between adsorbent surface and methylene blue. It could be concluded that the adsorption isotherm of methylene blue using mwXG-g-PANi/SiO₂ nanocomposites give a better fit to the Langmuir model.

V. CONCLUSIONS

In summary, we have successfully fabricated the mwXG-g-PANi/SiO₂ nanocomposites via a simple, green and industrially feasible approach. The adsorption experiments indicated that adsorbent used in this paper was effective in removing methylene blue from aqueous solution. The adsorption isotherm is well fitted by Langmuir isotherm model,

and the maximum adsorption capacity is about 1250 mg/g. The correlation coefficients in this case were found in 0.99 and 0.95 for Langmuir model and Freundlich model respectively. The currently introduced adsorbents are both simple and cost effective and might have successful application for treatment of textile wastewaters in near future technology.

VI. CONFLICTS OF INTEREST

The authors declare no conflict of interest.

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