



# Effective removal of Congo red dye from aqueous solution using modified xanthan gum/silica hybrid nanocomposite as adsorbent



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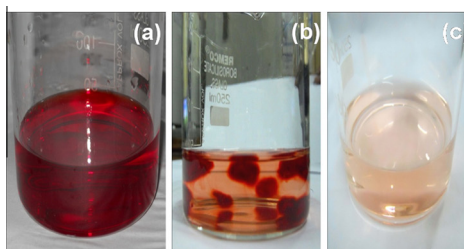
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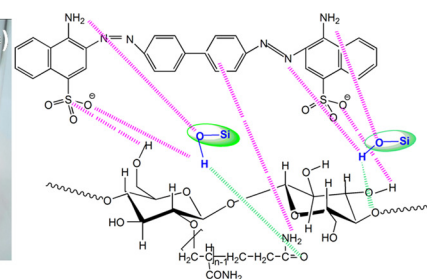
## HIGHLIGHTS

- XG-g-PAM/SiO<sub>2</sub> nanocomposite exhibits excellent CR dye adsorption capacity.
- The adsorption efficacy of dye depends highly on pH.
- The nanocomposite based adsorbent demonstrates good recyclability.

## GRAPHICAL ABSTRACT



(a) Before adsorption; (b) dye loaded nanocomposite after 150 min; (c) after complete adsorption.



Interaction between dye and the nanocomposite.

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## ABSTRACT

The aim of this work is to study the feasibility of XG-g-PAM/SiO<sub>2</sub> nanocomposite towards its potential application as high performance adsorbent for removal of Congo red (CR) dye from aqueous solution. The surface area, average pore size and total pore volume of the developed nanocomposite has been determined. The efficiency of CR dye adsorption depends on various factors like pH, temperature of the solution, equilibrium time of adsorption, agitation speed, initial concentration of dye and adsorbent dosage. It has been observed that the nanocomposite is having excellent CR dye adsorption capacity ( $Q_0 = 209.205 \text{ mg g}^{-1}$ ), which is considerably high. The dye adsorption process is controlled by pseudo-second order and intraparticle diffusion kinetic models. The adsorption equilibrium data correlates well with Langmuir isotherm. Desorption study indicates the efficient regeneration ability of the dye loaded nanocomposite.

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## 1. Introduction

The discharge of dye effluents into environment is currently one of the world's major environmental problems from both toxicological and inventive standpoint (Ahmaruzzaman, 2009). Out of different industries (like textile, printing and dyeing, cosmetic, petroleum and leather), a massive amount of dyes is discharged by textile industries because of improper processing (Sharma et al., 2009; Ma et al., 2012). The discharged dye effluents are highly toxic even

at very low concentration (Sharma et al., 2009). Hence removal of toxic dyes from wastewaters before discharge into water bodies emerges a major challenge from environmental point of view (Feng et al., 2012).

Congo red (CR) dye [1-naphthalenesulfonic acid, 3,3'-(4,4'-biphenylene bis (azo) bis (4-amino-) disodium salt] is a benzidine-based anionic bisazo dye. This dye is known to metabolize to benzidine, a known human carcinogenic and mutagenic, is a potential danger of bioaccumulation and exposure to this dye can create allergic problems (Oladoja and Akinlabi, 2009; Chatterjee et al., 2010). Here, it has been chosen as model dye because of its complex chemical structure, limited biodegradability and stability towards light as well as high solubility in aqueous solution.

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Adsorption is one of the most efficient techniques for removal of dyes from aqueous solution. Many polymeric material based adsorbents have been developed for treatment of dye containing wastewater (Chatterjee et al., 2011; Zhu et al., 2012). Among various types of polymeric adsorbents, modified and unmodified natural polymers are preferred now-a-days, because of their low cost, easy availability and biodegradable nature (Crini and Badot, 2008; Singh et al., 2009a,b). However, modified polysaccharides as adsorbent are having certain limitations like lower hydrodynamic volume, poor thermal stability as well as low surface area. Because of these limitations, of late modified polysaccharide based nanocomposites are used extensively for the removal of toxic dyes from aqueous solution (Janaki et al., 2012). Because of strong synergistic effect between the inorganic nanofiller and organic polymer matrix, the hybrid nanocomposites show crucial change in mechanical and thermal properties as well as higher surface area and hydrodynamic radius in comparison with pure organic polymer (Singh et al., 2009a,b; Wang et al., 2008).

The most common procedure for the synthesis of hybrid materials is based on sol–gel process, for the formation of inorganic network onto the surface of polymer matrix (Shchipunov and Karpenko, 2004). Recent years, attention has been devoted to synthesis of monodispersed silica nanoparticles because of their uniform size, shape, composition and functional properties as well as their effectiveness as surface modified substrate (Nozawa et al., 2005).

Recently, a novel biodegradable nanocomposite based on polyacrylamide grafted xanthan gum and silica (XG-g-PAM/SiO<sub>2</sub>) has been developed which has been found to be an effective adsorbent for removal of Pb(II) ion from aqueous solution (Ghorai et al., 2012). Keeping in view of the toxic effects of dye, this study highlights the application of the developed nanocomposite for efficient removal of CR dye from aqueous solution.

## 2. Methods

### 2.1. Reagent and materials

Xanthan gum and tetraethylorthosilicate (98% TEOS) were purchased from Sigma–Aldrich, Germany. Acrylamide was procured from Merck, Mumbai, India. Potassium persulphate was supplied by Qualigens Fine chemicals, Mumbai, India. Analytical grade acetone and hydroquinone were supplied by S.D. Fine chemicals, Mumbai, India. CR dye (The formula, molecular weight and  $\lambda_{\max}$  of the CR dye were C<sub>32</sub>H<sub>22</sub>N<sub>6</sub>Na<sub>2</sub>O<sub>6</sub>S<sub>2</sub>, 696.66 g/mol and 497 nm, respectively) used in this study as a model anionic azo dye, was purchased from Loba Chemicals, India. All the chemicals were used as received without further purification. For all the experiments, double distilled water was used.

### 2.2. Synthesis of polyacrylamide grafted xanthan gum/silica nanocomposite (XG-g-PAM/SiO<sub>2</sub>)

The graft copolymer of polyacrylamide and xanthan gum has been synthesized by free radical polymerization technique in nitrogen atmosphere using potassium persulphate (KPS) as initiator. By varying the reaction parameters, various grades of graft copolymers were synthesized and optimized the best one with respect to its higher% grafting efficiency (%GE), hydrodynamic radius, and intrinsic viscosity. Afterwards, the nanoscale Stöber silica (sol–gel process using Stöber method) was incorporated *in situ* on the surface of the graft copolymer by hydrolysis and condensation of TEOS in a mixture of ethanol, water and ammonia as reported earlier (Ghorai et al., 2012).

### 2.3. Characterization

The N<sub>2</sub> adsorption–desorption isotherm was measured at liquid nitrogen temperature 77 K using NOVA 3200e instrument (Quantachrome, USA). The specific surface area was calculated by the Brunauer–Emmett–Teller (BET) equation. The total pore volume was estimated from the amount of nitrogen adsorbed at a relative pressure of about 0.99. Pore size distribution curves were obtained by the Barrett–Joyner–Halenda (BJH) method from the desorption branch. The zeta potential of the nanocomposite was measured using ZETASIZER Nano-ZS90 (Malvern, UK).

### 2.4. Adsorption experiments

The adsorption experiments of aqueous solution of CR dye were performed on a thermostated orbital shaker (Rivotek, Kolkata, India). The experimental parameters such as solution pH, temperature, contact time, agitation speed, initial concentration of dye and adsorbent dosage were varied to obtain the best adsorption condition. A stock solution of 1000 mg L<sup>-1</sup> CR dye was prepared in double distilled-deionised water. In all typical batch experiment, required amount of adsorbent was thoroughly mixed with 50 mL of dye solution, whose concentration was known previously. The samples were withdrawn from the shaker at predetermined time intervals and the dye solution was separated from the adsorbent by centrifugation (Centrifuge, Make: REMI; Model – R-24) at 10000 rpm for 10 min and the absorbance was recorded using a UV–Vis spectrophotometer (Make: Shimadzu, Japan; Model: UV 1800) at 497 nm corresponding to a maximum absorbance of CR dye. The percentage adsorption of dye was calculated using Eq. (1):

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

And the equilibrium uptake was calculated using Eq. (2):

$$q_e = (C_0 - C_e) \times \frac{V}{W} \quad (2)$$

where  $q_e$  is the equilibrium capacity of dye on the adsorbent (mg g<sup>-1</sup>),  $C_0$  is the initial concentration of dye solution (mg L<sup>-1</sup>),  $C_e$  is the equilibrium concentration of dye solution (mg L<sup>-1</sup>),  $V$  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.

### 2.5. Desorption experiments

To determine the reusability of the nanocomposite as adsorbent, four successive adsorption–desorption cycles were performed. The desorption experiment was carried out with 100 mg L<sup>-1</sup> of CR dye solution mixed with 50 mg of nanocomposite for 2 h. The dye loaded samples were separated by filtration and the samples were dried completely. Afterwards, it was used for desorption study. Three different stripping solutions having pH: 2.5, 7.5 and 10 were used to determine the maximum desorption percentage of dye from the nanocomposite.

## 3. Results and discussions

The nanocomposite has been prepared *in situ* by incorporation of nano scaled Stöber silica onto the surface of XG-g-PAM, which was previously synthesized by free radical polymerization technique using potassium persulphate as initiator. It has been proposed that H-bonding interaction is predominating between the hydroxyl and amide groups of modified polysaccharides and the silanol groups of silica particles (Ghorai et al., 2012). By changing the reaction parameters, four grades of nanocomposites have been

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