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# Efficient removal of coomassie brilliant blue R-250 dye using starch/poly(alginate-chitosan) nanohydrogel

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## ABSTRACT

In this study, we have synthesized a starch/poly(alginate-chitosan) nanohydrogel (ST/PL(AA-cl-AAm) NHG) by co-polymerization method which was used as an effective adsorbent for the removal of coomassie brilliant (CB) blue R-250 dye from the aqueous solution. The synthesized nanohydrogel was characterized by FTIR, SEM and TEM techniques. The effect of initial dye concentration, temperature, and agitation time on the adsorption ability of the nanohydrogel was studied comprehensively. The experimental outcomes exhibited that the prepared nanohydrogel had high adsorption tendency to remove the CB from the aqueous solution. The regression coefficient values for Langmuir (0.99), Freundlich (0.93) and Tempkin (0.98) recommended that adsorption process fit to Langmuir isotherm. The pseudo-second-order equation showed the better adsorption kinetics of the adsorption process. The feasibility of CB adsorption onto ST/PL(AA-cl-AAm) NHG was also studied thermodynamically and the results indicated that the adsorption was spontaneous and chemical in nature.

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## 1. Introduction

Due to the fast industrialization, the addition of various types of pollutants to the water system is one of the common problems worldwide. Addition of harmful metal ions, industrial dye molecules, phenols, fertilizers pollute the natural water bodies, which makes it unfavorable for the use by animals as well as by plants (Rivas and Munoz, 2010; Rao et al., 2010; Kyzas and Lazaridis, 2009; Sharma et al., 2015; Santhi et al., 2016; Kumar et al., 2016). Due to the toxic nature of water, it

can cause numerous problems such as jaundice, high heart beat rate, vomiting and skin problems. The presence of dyes and metal ions in the water can be carcinogenic to animals and humans (Sharma et al., 2017; Kumar et al., 2017). Various techniques have been applied for the removal of these pollutants from aqueous medium (Pathania et al., 2015; Naushad et al., 2016a,b,c). Adsorption is one of the best solutions for the removal of dyes from the wastewater due to its cost-effective nature (Alqadami et al., 2016; Bahram et al., 2011). Adsorption by the super adsorbing materials such as nano-sized hydrogels has made

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this field more important (Zhang et al., 2012; Meng et al., 2012; Patel and Patel, 2013; Kasgoz et al., 2008; Sun et al., 2012). Due to the small size and three-dimensional structures, nanohydrogels can be used in a number of applications such as drug delivery, water purification, adsorption, electrode making, or as smart materials. Their absorbing tendency can be easily tuned by adjusting the pore size and their chemical structure (Yang et al., 2013; Balakrishnan and Banerjee, 2011; Vlierberghé et al., 2011). Hydrogels are flexible in nature and have the capacity to retain water due to functional groups attached to the polymeric backbone. By changing the nature and chemistry of the precursor hydrogel, their mechanical properties can be improved efficiently (Anbia and Ghaffari, 2009; Hameed and Ahmad, 2009; Langmuir, 1916). Hydrogels can be categorized based on chemical and physical nature of crosslinked junctions. Different approaches have been used for designing of nanohydrogels, out of which first approach includes the physical attraction between the polymeric chains. Yu et al. has synthesized chitosan-based nanohydrogels via physical (electrostatic) attractions only. In contrast, the second approach includes the formation of nanohydrogels by chemical methods. For example, Bronich et al. synthesized a copolymer nanohydrogels by chemically crosslinking the constituting polymers (Strandman and Zhu, 2016; Phadke et al., 2012; Akay et al., 2013; Krogsgaard et al., 2013; Wei et al., 2014; Appel et al., 2012; Chirila et al., 2014). The third approach includes different polymerization techniques such as mini polymerization, reverse polymerization or precipitation polymerization.

Starch is a natural polymer and having various applications. It has substituted many conventionally used non-renewable materials and has occurred as an excellent base for the support of different compounds. Due to its inexpensive and biodegradable nature, it seems to be more favorable. The performance of starch can be effectively improved by the co-grafting and cross-linking approach (Puppi et al., 2010; Gomes et al., 2002; Neves et al., 2005; Schieker et al., 2006; Mano et al., 2004; Elvira et al., 2002; Reis and Cunha, 1995). This resulted in better mechanical properties and water stability of starch. It is also used in industry at a large scale as wrap sizing, since it increases the strength to a greater extent.

In the present paper, we have synthesized starch/poly(alginate-chitosan-covalently crosslinked acrylamide) nanohydrogel which was efficaciously used for the removal of coomassie brilliant blue R-250 dye from aqueous medium due to its high adsorbing ability. The equilibrium removal efficiency was studied using Langmuir, Freundlich and Temkin isotherm models.

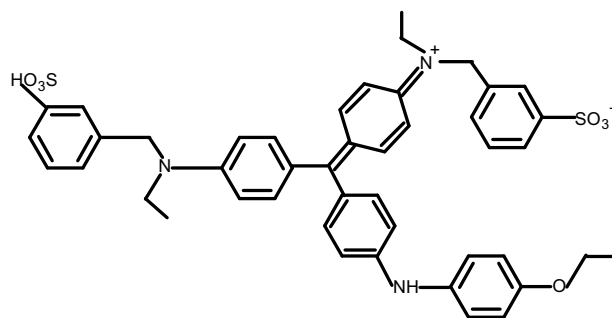
## 2. Experimental protocol

### 2.1. Reagents and instruments

The main reagents were alginate (LobaChemie), acrylamide (LobaChemie), starch (LobaChemie), ammonium persulphate (CDH Pvt., Ltd., India), *N,N'*-methylene bis-acrylamide (S-D Fine Chem. Ltd.). Various instruments used were shaker (INCO Instrument and Chemical Private Limited), centrifuge machine (INCO Instrument and Chemical Private Limited.), double beam UV spectrophotometer (INCO Instrument and Chemical Private Limited), FTIR (Agilent Technologies Cary 630 FTIR), Scanning electron microscopy (JEOL Model, JSM 610), Transmission electron microscopy (Techni G2 20 S-Twin).

### 2.2. Synthesis of starch/poly(alginate-chitosan-covalently crosslinked acrylamide) nanohydrogel

The ST/PL(AA-cl-AAm)NHG was prepared by co-graft polymerization method. The solution of starch (5%) was prepared in double distilled water with constant stirring using magnetic stirrer. To the above prepared starch gel, 0.1 M alginate (AA) and 0.1 M acrylamide (AAm) solutions were added drop wise in the presence of initiator, APS (5%) and cross linker, *N,N'*-



**Scheme 1 – Structure of coomassie brilliant (CB) blue R-250 dye.**

methylenebisacrylamide (MBAAm) (7%). The reaction mixture was vigorously stirred for 4 h at 65 °C using thermostat. The ST/PL(AA-cl-AAm)NHG was collected after 4 h, washed with acetone and dried at 50 °C in hot air oven. The reaction yield was calculated using formula:

$$\% \text{yield} = \frac{W_R - W_P}{W_R} \times 100, \quad (1)$$

### 2.3. Characterization

FTIR spectra for ST/PL(AA-cl-AAm)NHG and dye adsorbed ST/PL(AA-cl-AAm)NHG was recorded by Fourier-transform infrared spectrophotometer using KBr disk method. The FTIR spectrum was recorded in the wave number range from 400 to 4000  $\text{cm}^{-1}$ . Microphotographs of nanohydrogels were recorded by using scanning electron microscope (SEM). The two samples were placed on a carbon tape with silicon adhesive and mounted on an aluminum stub. The microphotographs were obtained at different magnifications. The particle size of ST/PL(AA-cl-AAm)NHG was carried out using transmission electron microscope (FEI 200 kV, Technai). The particle dimension and morphology were determined by preparing the suspensions of prepared nanohydrogel in ethanol and positioned on carbon copper grid.

### 2.4. Adsorption of hazardous coomassie brilliant blue R-250 (CB)

Adsorption of toxic coomassie brilliant blue R-250 (CB) dye (Scheme 1) from aqueous phase was studied by batch technique. 50 mL of CB solution with varying initial dye concentrations was shaken with a known amount of adsorbent in 100 mL Erlenmeyer flask at 30 °C. The experimental flasks were taken out at fixed time interval until the adsorption equilibrium was achieved. The dye solution was separated by centrifugation operated at 3500 rpm for 5 min. The supernatants were then filtered to ensure that solutions were free from ST/PL(AA-cl-AAm)NHG before measuring the residual CB concentration. The remaining dye in the filtrate was analyzed by UV-visible spectrophotometer (Shimadzu UV-1601) at 555 nm. The effect of agitation time, initial CB dye concentration, adsorbent amount, pH and temperature were studied and optimized for the maximum adsorption of dye. The experiments were conducted in the duplicate and negative controls were simultaneously carried out. The % removal or amount of dye adsorption per unit mass of ST/PL(AA-cl-AAm)NHG at equilibrium were evaluated as:

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

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