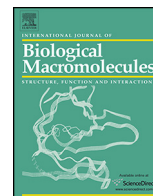




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Short communication

Carboxymethyl cellulose-g-poly(2-(dimethylamino)ethyl methacrylate) hydrogel as adsorbent for dye removal

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ABSTRACT

A novel adsorbent was prepared via crosslinking graft copolymerization of 2-(dimethylaminoethyl)methacrylate (DMAEMA) onto carboxymethyl cellulose (CMC) backbone. Ethylene glycol dimethacrylate and potassium persulphate were used as crosslinker and initiator, respectively. CMC-g-PDMAEMA hydrogel was used to remove methyl orange (MO) from aqueous solutions. The adsorption kinetics and isotherms were found to follow Pseudo-second-order kinetic model and Langmuir model, respectively. The high maximum adsorption capacity (1825 mg/g) implied that CMC-g-DMAEMA can be used as promising adsorbent for the synthetic dyes removal from wastewater.

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1. Introduction

Chemical industries consumed annually more than 7×10^5 t of dye [1]. However, it is reported that 2% of dyes produced annually are discharged in water resources during industrial processing causing adverse effects on aquatic organisms and human health [2,3]. Moreover, the complex aromatic structure makes synthetic dyes more stable and difficult to remove [4]. Different techniques like membrane separation [5], oxidation or ozonation [6], electro-coagulation [7] and adsorption [3,8] have been used for waste water treatments [9]. Recently, hydrogels obtained from bioresources, such as sodium alginate [10], starch [11] cellulosic materials [12,13] and chitosan [2,14,15] are emerging as a potential alternative adsorbents for metal ions and synthetic dyes from aqueous solutions. CMC has gained increasing interest in the recent years as a bio-adsorbent for dyes capture from aqueous solutions due to its biocompatibility, biodegradability and anionic properties [16,17].

This work aims to develop a novel high-loading adsorbent based on CMC for methyl orange (MO) removal from aqueous solutions. In terms of monomer selection, methacrylates carrying tertiary amino groups, in particular 2-(dimethylamino)ethyl methacrylate (DMAEMA), is of increasing interest for many applications such as membranes [18], drug delivery systems [19] and dye removal [20]. It has been reported [21] that DMAEMA was successfully grafted onto several substrates producing compounds with specific

properties, such as thermo- and pH-sensitivity [22]. The effect of contact time, solution pH and initial dye concentration on the adsorption of MO onto CMC-g-PDMAEMA hydrogel was systematically studied. In addition, adsorption isotherm and kinetic models for MO adsorption was also evaluated.

2. Experimental

2.1. Materials

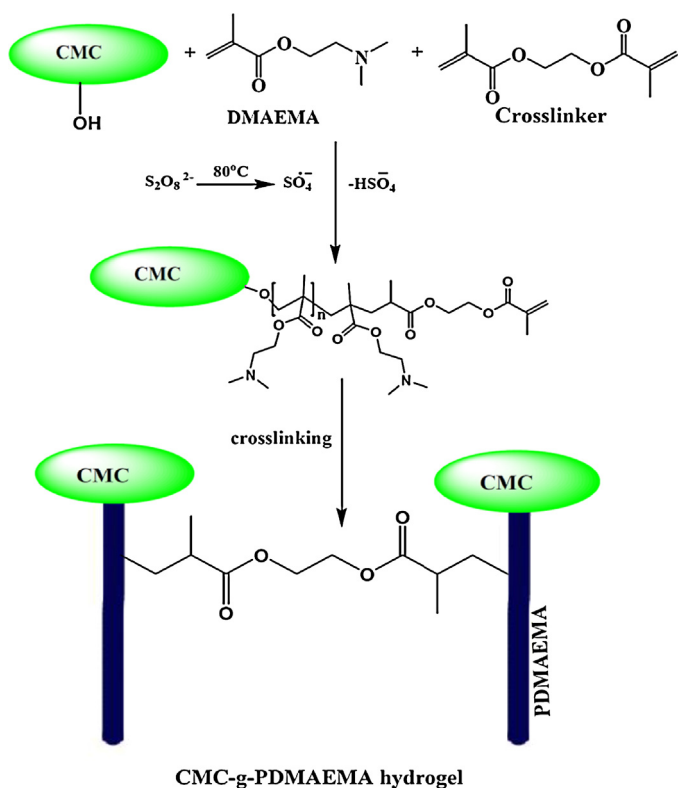
Carboxymethyl cellulose sodium salt (>99.5%) with high viscosity was purchased from Fluka Biochemika. The viscosity of 4% CMC in water at 25 °C is 1000–1500 mPa s. 2-Dimethylaminoethyl methacrylate monomer (DMAEMA) from Merck and ethylene glycol dimethacrylate as crosslinker from Sigma-Aldrich were used without any purification. MO was purchased from S D Fine-Chem Limited (India). Other chemicals were of pure analytical grade and used as received.

2.2. Preparation of CMC-g-PDMAEMA hydrogel

Superabsorbent hydrogel was prepared by adding 2 g CMC powder to 100 mL distilled water in a reactor to prepare 2% CMC solution. The solution was stirred until complete CMC dissolving then placed in a water bath adjusted at 70 °C and purged with nitrogen for 30 min. The initiator (0.017 mol/L) followed by the monomer DMAEMA (0.4 mol/L) then by the crosslinker (0.007 mol/L) were added to the reaction mixture and stirred until complete gelation. For removing the unreacted molecules, the gel

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Scheme 1. Proposed mechanism for synthesis of CMC-g-PDMAEMA hydrogel.

product was chopped to small pieces and allowed for completely swelling in excess amounts of distilled water for several times. The dewatered gel particles were filtered and dried in oven (at 50 °C) for 48 h. Proposed free radical mechanism for the synthesis of CMC-g-PDMAEMA was presented in [Scheme 1](#).

2.3. Adsorption studies

The adsorption studies were performed using batch method. In brief, 0.05 g of dried adsorbent was soaked in an aqueous buffer for 1 h before adding dye solutions. After ending the adsorption process at desired conditions, the solution was separated from the adsorbents with a syringe filter and the dye concentration was measured using UNICO UV-2000 spectrophotometer at 464 nm. Data are representative of at least three experiments, and standard deviations are less than 8.0%.

2.4. Characterization

Attenuated total reflection-Fourier transform infrared spectroscopy (ATR-FTIR) was done from 500 to 4000 cm^{-1} on a Thermo Nicolet FT-IR Nexus 470. Scanning electron microscopy was done on a JEOL JXA-840A Electron probe microanalyzer with tungsten filament (30 kV).

3. Results and discussion

3.1. FTIR-spectroscopy

The structural changes of CMC and CMC-g-PDMAEMA hydrogel were confirmed by FTIR spectroscopy. As shown in [Fig. 1A](#) the characteristic bands at 1623, 2890, and 3441 cm^{-1} are related to carboxyl group, asymmetric C-H stretching and O-H stretching vibration in CMC, respectively. However, [Fig. 1B](#) shows new and strong bands at 1727 and 1149 cm^{-1} which are attributed to ester

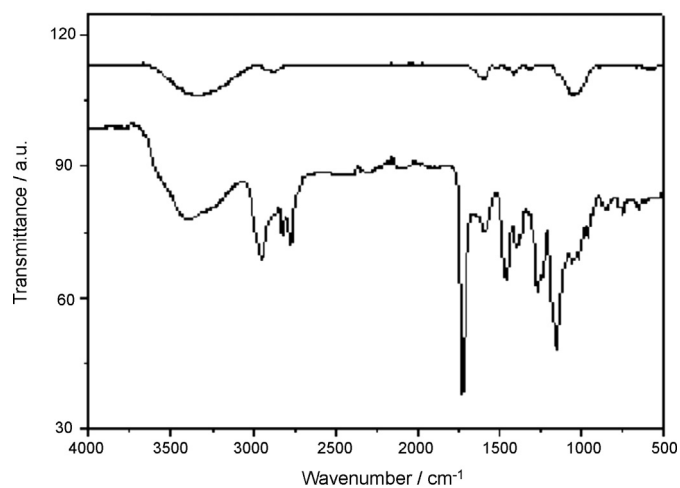


Fig. 1. FTIR spectra of CMC (A) and CMC-g-PDMAEMA hydrogel (B).

stretching carbonyl and C-N stretching. In addition, the characteristic peaks at 2770 and 2821 cm^{-1} may be attributed to $-\text{CH}_2-$ groups which are adjacent to the nitrogen atom [23]. The presence of these new bands confirms the graft copolymerization of PDMAEMA onto CMC backbone.

3.2. MO uptake study

3.2.1. Effect of pH on MO adsorption

[Fig. 2](#) shows the effect of pH value of dye solution on the adsorption capacities of the hydrogel. It was found that as the pH value increased from 2.0 to 6.0, the adsorption capacities of the hydrogel sharply decreased from 193 to 135 mg/g, and then tend to leveling off with further increase in pH. These results may be interpreted as follows: most of the tertiary amino groups of the adsorbent are ionized and interact with the dye molecules via strong electrostatic interaction, which is favorable to increase the absorption for MO. In addition, the amino groups presented in PDMAEMA can also be protonated to form NH_3^+ at lower pH, which increase the number of fixed ionized groups and generate the electrostatic repulsion forces

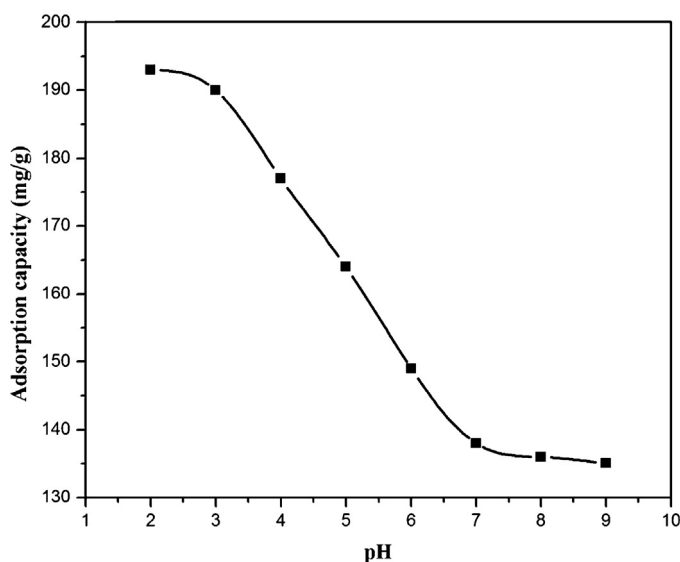


Fig. 2. Effect of the pH values on the adsorption capacities of CMC-g-PDMAEMA for MO. Adsorption experiments: dye concentration: 100 mg/L; sample dose: 0.05 g/100 mL and equilibrium time: 60 min.

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