



Original Research Article

Fast removal of methyl green from aqueous solution by adsorption onto new modified chitosan Schiff base

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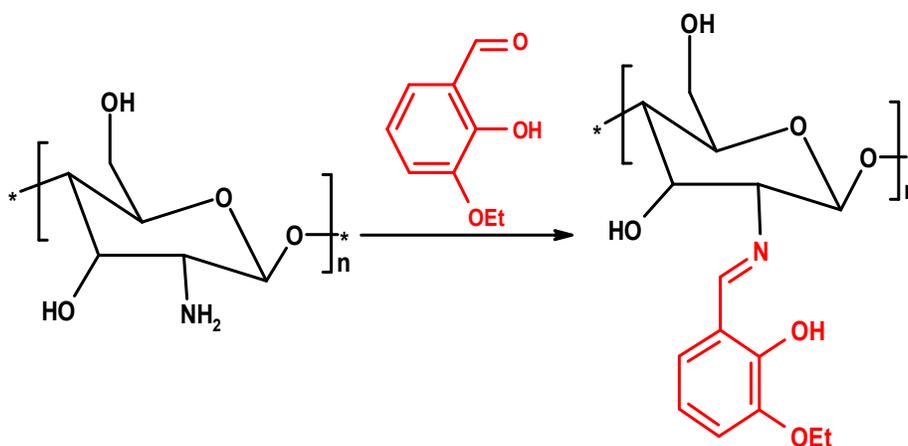
Methyl green

Removal

ABSTRACT

New modified chitosan Schiff base (3E-2H-BCs) was prepared via a condensation reaction between 3-ethoxy-2-hydroxybenzaldehyde and chitosan. The title compound 3E-2H-BCs was characterized by various techniques such as FT-IR, UV-Vis, XRD, SEM and TG-DTA. The results show that the title compound 3E-2H-BCs was successfully prepared. In addition, the title compound 3E-2H-BCs applied as an efficient adsorbent for methyl green (MG) removal from aqueous solution and the effect of contact time and adsorbent dosage has been studied. The equilibrium of dye adsorption by 3E-2H-BCs was reached at 1 min contact time at the presence of MG. The dye adsorption results show that the title compound 3E-2H-BCs is as an efficient adsorbent for other dye removal from wastewater.

GRAPHICAL ABSTRACT



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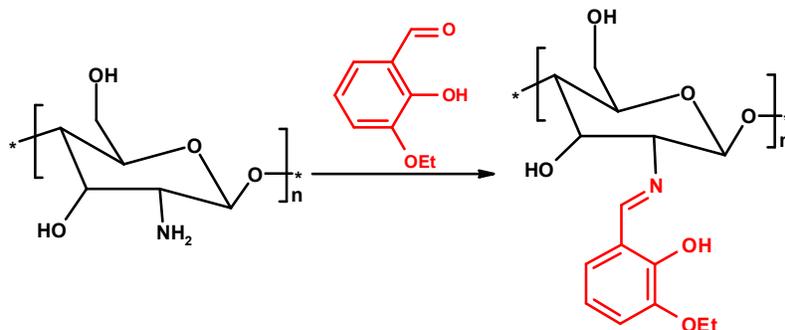


INTRODUCTION

Synthetic and natural dyes are widely used in various industries such as textiles, leather and plastics [1]. About 15% of the dyes used in factories remain in the wastewater. Improper discharge of wastewater and their entry into the environment causes great harm to humans, animals and plants [1,2]. Therefore, before discharging the wastewater of factories, contaminants such as different colors as well as metal ions must be removed [3,4]. Organic dyes are generally very stable under natural environmental conditions, so they do not decompose easily [1-4]. There are many methods to remove them such as oxidation [5], chemical coagulation [6], biodegradation [7] and adsorption process [8-14]. Most chemical and biological methods of dye removal are expensive or very complex. Among these methods, the adsorption method has received much attention for many reasons such as process simplicity, cheapness, and high efficiency, use of various synthetic and natural adsorbents as well as adsorption and reuse [8-14]. So far, many adsorbents have been used to remove different cationic and anionic dyes [8-20]. Chitosan (natural polymer) and its derivatives, which are biodegradable, environmentally friendly and non-toxic and have many applications in various fields [21-24], have been used as adsorbents in the removal of transition metal ions [25-33] and various organic dyes [1-4, 34-40]. Its chitosan

properties can be improved by modifying the surface of chitosan with transition metal oxides nanoparticles to prepare various composites [1, 36, 39] and or in a condensation reaction to form chitosan Schiff bases [34]. Obeid et al. used chitosan/maghemite composite as a magsorbent to remove methyl orange dye from aqueous solution. The highest amount of methyl orange dye removal under optimal conditions reported 799 mg/g [39]. Haldoraj and Shim used chitosan/MgO composite to remove methyl orange in aqueous solution [36]. In the best conditions (pH 5, use of 0.5 g of adsorbent and time 5 minutes) the maximum amount of methyl orange dye adsorption was 9.90%. Ke et al. modified the chitosan using quaternary ammonium salts and used it as an adsorbent to removal of methyl orange from wastewater [3]. The maximum capacity of this adsorbent under optimal conditions was 9.98%. Foroughnia et al. prepared a new chitosan Schiff base and its Fe₂O₃ nanocomposite and studied their antibacterial and methyl orange dye removal properties [40].

In his paper, we prepared new chitosan Schiff base (3E-2H-BCs) via a condensation reaction between 3-ethoxy-2-hydroxybenzaldehyde and chitosan (Scheme 1) and characterized by FT-IR, UV-Vis, XRD, SEM and TG-DTA. In addition, the adsorption of methyl green (MG) from aqueous solution was studied.



Scheme 1. Synthesis reaction of 3E-2H-BCs

EXPERIMENTAL

Materials and methods

Chitosan, 3-ethoxy-2-hydroxybenzaldehyde, glacial acetic acid, and ethanol were purchased from Sigma-Aldrich and Merck Co. and were used as received without further purification. Perkin-Elmer FT-IR spectrophotometer instrument was used for the recording FT-IR (KBr disks, 4000–400 cm^{-1}) spectra. DSC analyses were recording DSC analyzer Model 60A, Shimadzu, Japan. XRD patterns were determined by Bruker AXS-D8 X-ray diffractometer ($2\theta = 10\text{-}80^\circ$), and SEM images were recorded on the TESCAN Vega Model scanning electron microscope. The Perkin-Elmer TGA analyzer was used for the thermogravimetry analysis in air atmosphere at a flow rate of 20 $^\circ\text{C}/\text{min}$ (25-700 $^\circ\text{C}$). UV-Vis spectra were carried out with UV-Visible spectrophotometer (Perkin-Elmer).

Synthesis of 3-ethoxy-2-hydroxybenzaldehyde chitosan Schiff base (3E-2H-BCs)

Chitosan (2 g) was suspended in 100 mL of ethanol-glacial acetic acid (95:5 v/v) and stirred. After 10 min, 10 mL ethanolic solution of 3-ethoxy-2-hydroxybenzaldehyde (2 g) was slowly added and the reaction mixture was kept at 80 $^\circ\text{C}$, up to a yellow-orange solid precipitated. The mixture was cooled and the solid was filtered off, washed with cold ethanol and dried at room temperature for several days.

Adsorption experiments

All adsorption experiments were carried out in a series of 50 mL Erlenmeyer flasks containing 25 mL of dye solution with various adsorbent (0.005, 0.01 and 0.02 g) and the effect of contact times (0-120 min) were investigated. The solutions were the shaken and at various contact time, then adsorbent (3E-2H-BCs) was separated by centrifuged and the solution was analyzed by using UV-Vis spectrophotometer.

RESULTS AND DISCUSSION

Characterization

To identify the functional groups on the modified chitosan, we used FT-IR spectroscopy. The FT-IR spectrum of 3E-2H-BCs was shown in Fig. 1. The broad peaks at 3445 and 2917 cm^{-1} represents the symmetrical stretching vibration of OH groups and the symmetric stretching of C-H bond of -Schiff base and pyranose ring [41]. The weak peak at 1714 cm^{-1} predict the unreacted aldehyde is also present in the title compound 3E-2H-BCs [26]. The sharp peak appeared at 1633 cm^{-1} assigned to the imine C=N that prepared by condensation of carbonyl group (-CHO) of 3-ethoxy-2-hydroxybenzaldehyde and amine (-NH₂) group of chitosan [29,30,34]. The peak at 1068 cm^{-1} corresponded to the stretching vibration of C-O bonds [38, 42].

XRD pattern of 3E-2H-BCs was shown in Fig. 2. A broad peak at about 20.61 $^\circ$ in the XRD pattern of 3E-2H-BCs assigned to the chitosan and showed that it was amorphous [13,36]. Also, observed a small peak appeared at about 14.86 $^\circ$. This might be due to the preparation of Schiff base from the reaction of 3-ethoxy-2-hydroxybenzaldehyde and amine (NH₂) group of chitosan.

To investigate of the morphology of the title compound, SEM image was performed and the result is shown in Fig. 3. SEM image show that the surface of 3E-2H-BCs is wrinkling similar to the surface of H₂SO₄ crosslinked chitosan [2] due to the addition and condensation of 3-ethoxy-2-hydroxybenzaldehyde with chitosan to prepared chitosan Schiff base compound [44-45]. While the surface of chitosan is smooth [2,43].

Thermal stability of 3E-2H-BCs has been determined by thermogravimetric study. The TG/DTA curves of 3E-2H-BCs are shown in Fig. 4. TGA curve reveals the mass loss of 3E-2H-BCs during the heating process and exhibited two main decomposition stages. The first one start as at around 100 to 280 $^\circ\text{C}$ and the second stage start at around 280 to 360 $^\circ\text{C}$ [13].

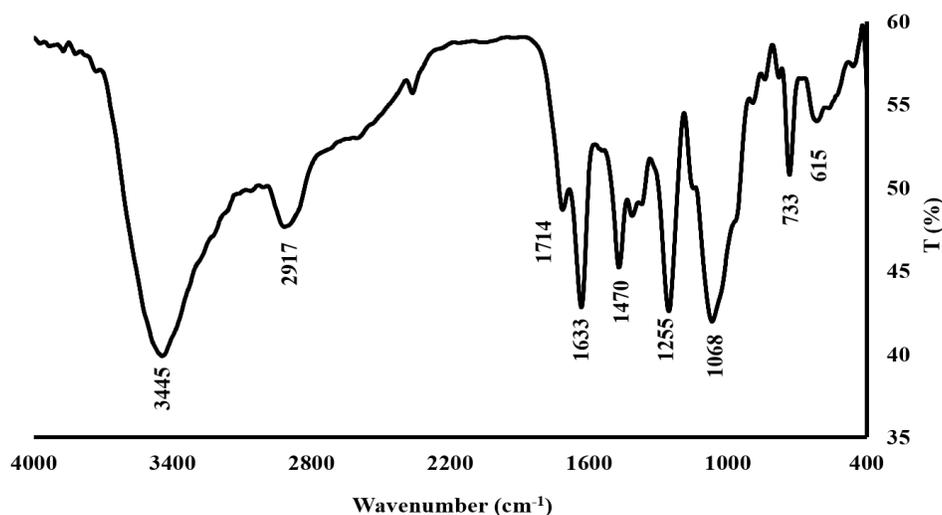


Figure 1. FT-IR spectrum of 3E-2H-BCs

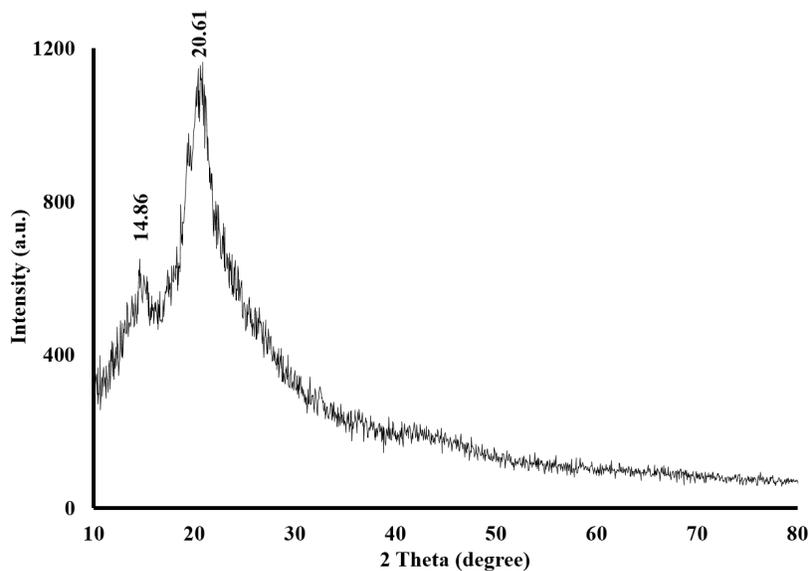


Figure 2. XRD pattern of 3E-2H-BCs

In the first stage, the 4% loss of 3E-2H-BCs was related to the evaporation of the absorbed ethanol solvent [43]. In the second state, the 36% loss of 3E-2H-BCs was related to the decomposition of the Schiff base groups of 3E-2H-BCs [43]. By heating the compound from 360 to 830 °C, about 18% of the mass of the compound is

decomposed. Finally, at 830 °C, about 42% of the mass of the compound remains [13]. According to the DTA, the main exothermal thermal decay temperature of 3E-2H-BCs was determined to be 310 °C [45], is more than similar Schiff bases of chitosan [43].

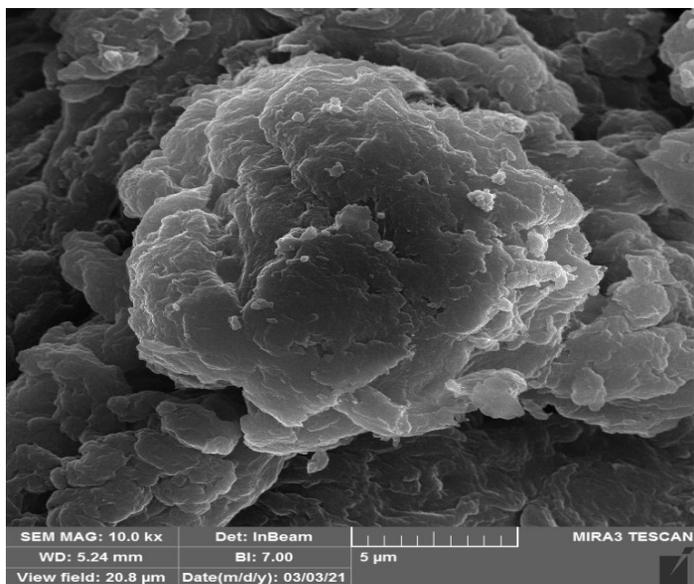


Figure 3. SEM image of 3E-2H-BCs

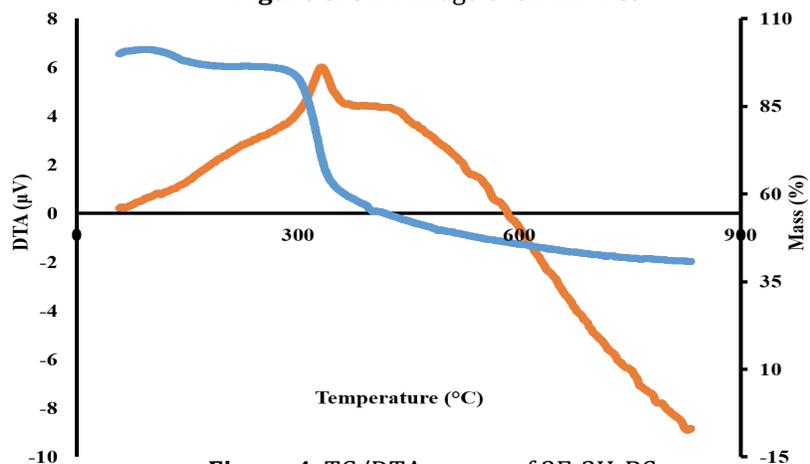


Figure 4. TG/DTA curves of 3E-2H-BCs

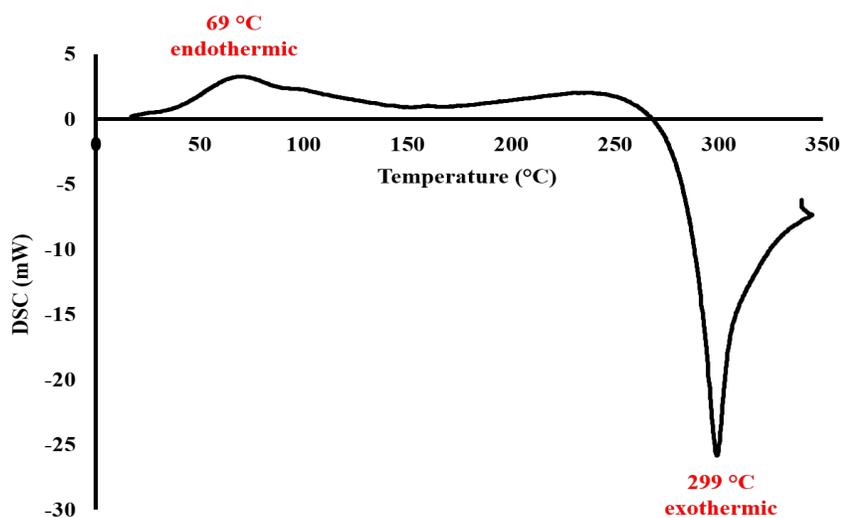
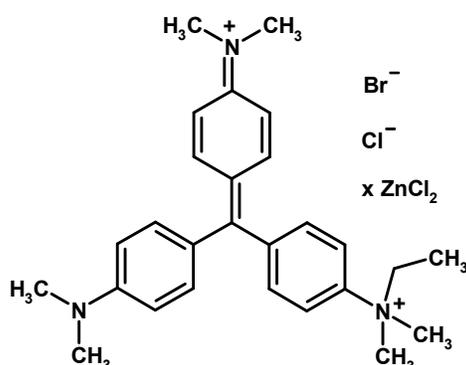


Figure 5. DSC curve of 3E-2H-BCs

In the DSC curve of 3E-2H-BCs a broad weak endothermic peak observed at 69°C, corresponding the evaporation of the absorbed ethanol solvent and a sharp exothermic peak observed at 299 °C, correspond the decomposition of Schiff base groups [40].

Investigation of the MG removal ability

Methyl green is a cationic dye (Scheme. 2) and used as change solution color in biology and medicine [46] and various adsorbent used to its removal from aqueous solution [11,46-52].



Scheme 2. Molecular structure of methyl green

According to the results in the previous work [48] on the removal of methyl green at various pH, we selected and studied the MG removal in the pH of 8. As seen in Fig. 6, there are three aromatic phenyl group in the structure of MG and also two N⁺ group. Therefore, the electrostatic interaction of them with active groups of adsorbent cause sorption of MG onto the adsorbent. At low pHs we will have competition between H⁺ ions and dyes to interact with the active groups on the adsorbent. Because of its small size and lightness of H⁺, it is easier to interact with the adsorbent and by disabling the active groups of the adsorbent, it does not allow the methyl green to interact with the adsorbent. The adsorption of MG onto the 3E-2H-BCs as adsorbent has been studied at pH = 8 with 40 mg/L as initial concentration of MG dye and the effect of contact times and the adsorbent dosage have been considered (Fig. 6). Fig. 6 shows the UV-Vis

spectra of MG with contact of 3E-2H-BCs in different contact times (0-60 min) and adsorbent dosages (0.005, 0.01 and 0.02 g). The sharp peak at 632 nm is the characteristic peak of MG. After addition of adsorbent and stirring of mixture, this peak was started to shift downwards with increase of contact time [37]. The MG uptake increased rapidly within the first 1 min, and almost 95.35%, 95.30% and 96.15% MG can be removed for adsorbent dosages of 0.005, 0.01 and 0.02 g, respectively. After that, the MG remove has been slow. Finally, after 60 min we can see that there is no additional enhancement in adsorption capacity [37]. The removal rate R (%) and adsorption capacity q_t (mg/g) of MG onto the 3E-2H-BCs were calculated on the basis of the following equations [1-3,34, 36-38]:

$$R = \{(C_i - C_t) \times 100\} / C_i \quad q_t = V \times \{(C_i - C_t) / M\}$$

where C_i (mg/L) is the initial and C_t (mg/L) is the final concentration of MG, V (L) is the volume of solution and M (g) is the mass of 3E-2H-BCs.

The contact time is one the important parameters to remove of the organic dyes in an aqueous solution. Similar to previous reports on the influence of contact time on the adsorption process [1-3,34,36-38], it can be seen that the removal rate (Fig. 7a) and adsorption capacity (Fig. 7b) of MG increased with the increasing of contact time (60 min) [2], however the removal rate R (%) is very fast at the beginning of the adsorption process, 76.8%, 83.15% and 92.62% by the addition of 0.005, 0.01 and 0.02 g of 3E-2H-BCs, respectively [4,36]. Fig. 7b showed the adsorption equilibrium had been reached at 1 min contact time, with adsorption capacity of 153.6, 83.15 and 46.31 mg/g by the addition of 0.005, 0.01 and 0.02 g of 3E-2H-BCs, respectively. This result confirmed the adsorption of MG onto the surface of 3E-2H-BCs as adsorbent is very fast due to electrostatic interaction and H-bonds between the active groups of MG and 3E-2H-BCs, such as NH₂, OH, N⁺(CH₃)₂ and also maybe for π-π interaction of aromatic phenyl rings of MG and 3E-2H-BCs (Fig. 8).

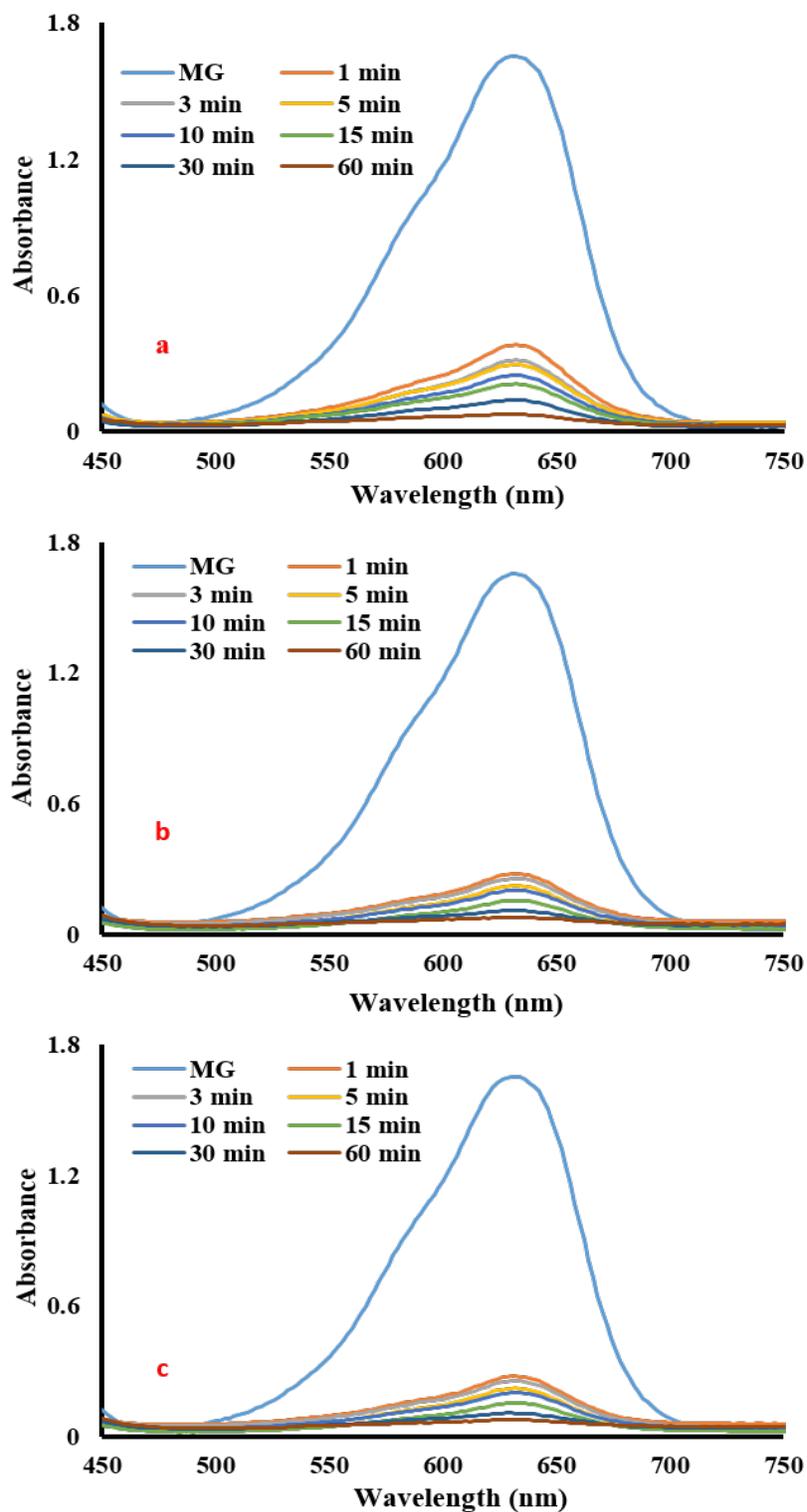


Figure 6. UV-Vis spectra of MG in different contact times and different adsorbent dosages a) 0.005, b) 0.01 and c) 0.02 g

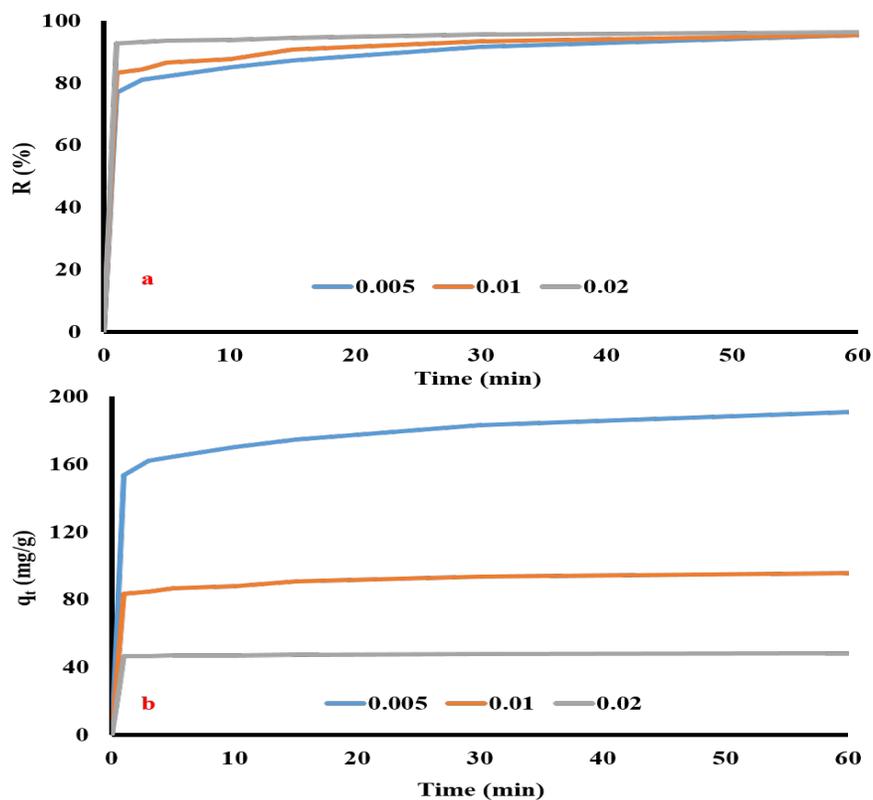


Figure 7. The effect of time and adsorbent dosage on a) removal rate and b) adsorption capacity

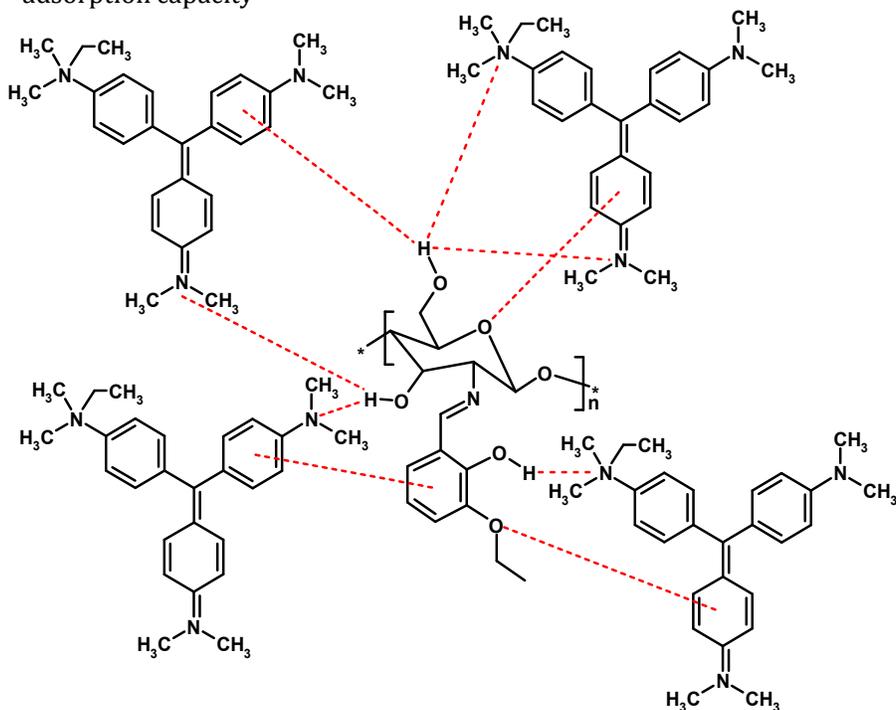


Figure 8. Schematic diagram of the interaction between MG and 3E-2H-BCs

CONCLUSION

In this paper, the new modified chitosan Schiff base (3E-2H-BCs) was successfully synthesized by simple condensation method from the reaction of chitosan and 3-ethoxy-2-hydroxybenzaldehyde and characterized. In addition, the title compound 3E-2H-BCs was used as adsorbent to the remove of MG from aqueous solution. Amount of $\approx 95\%$ of MG was adsorbed within 60 min from 40 mg/L MG at pH = 8 and at room temperature. The results showed good adsorption capacity, removal maximum of MG is 190.7 mg/g using 0.005 g of 3E-2H-BCs as adsorbent.

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