



Use of graphene substrates for wastewater treatment of textile industries

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Received: 22 September 2019 / Revised: 21 February 2020 / Accepted: 27 February 2020 / Published online: 21 April 2020
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Abstract

Dyes are among the main pollutants in the textile wastewaters, which, due to their complex molecular structure, are often poisonous, carcinogenic, and environmentally perilous. Therefore, the main objective of this study is to evaluate the removal efficiency of textile dye (tested on methylene blue, MB) from aqueous medium using graphene oxide (GO)-containing hydrogels due to their high surface area and ability to adsorb a large amount of water and water-soluble species. Therefore, a series of GO-based nanocomposites based on acrylamide and sodium alginate crosslinked by methylene bis-acrylamide (MBA) have been prepared to be used and evaluated as the starter. The effect of different parameters such as initial dye concentration, temperature, and contact time on the removal of MB is investigated as well as determination of the adsorption isotherm of MB on GO/hydrogel using the Langmuir model. Various concentrations, temperatures, and contact time have also been investigated on GO/hydrogels. The chemical structure of the GO/hydrogel is determined by scanning electron microscopy, FTIR, and X-ray diffraction. The results show that the GO/hydrogel can absorb MB by up to 95.76% efficiency in removal of MB from an aqueous media.

Keywords Graphene oxide · Hydrogel · Nanocomposite · Adsorption isotherm

1 Introduction

Dye-containing sewage produced from textile industries has toxic effects on aquatic ecosystems [1]. These dyes and products from their destruction have toxic effect on humans and microbial and aquatic species. Methylene blue as an azo color cationic, with molecular formula of $C_{16}H_{18}N_3SCl$ and molar mass of 319.85 g/mol, has extensive application in the paper industry and dyeing of cotton, wool, silk, leather, hair color, and so on. This dye can cause pains such as burning eyes, seizures, palpitations, shortness of breath, and skin irritation in humans. Therefore, removing dyes such as methylene blue from industrial wastewater has become one of the major environmental challenges [2]. Adsorption is one of the most widely used dye removal techniques

especially for non-separable colors [3]. Graphene oxide is a new type of carbon nanotube having various oxygen-containing functional groups with the high capability to remove ions and color compounds [4]. Hydrogels are classified into amorphous, semi-crystalline, hydrogen-bonded structures, super-molecular structures, and hydrocolloid aggregates. Hydrogels may also exhibit swelling behavior in the external environment [5]. GO has a lot of oxygenated groups, such as hydroxyl, epoxide, and carboxyl. As a result, they cause GO to be hydrophilic and easily dispersed in water and other solvents, and so it can be easily modified to improve the mechanical performance of the hydrogel with the polymer. One of the important characteristics of synthesized hydrogels is their sufficient strength so that they do not lose their structure after water adsorption and do not flow [3, 4]. Hydrogels have many applications in different fields, including cosmetic products, wastewater treatment, water preservatives, water adsorbents, dentistry, and food industry and in agriculture and horticulture to improve the capacity and maintenance of water and soil [6, 7].

The aim of this study was to synthesize hydrogel to remove cationic dye of methylene blue from aqueous

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solution. The chemical and structural properties of the hydrogel were identified using the infrared spectroscopy and X-ray diffraction pattern. The adsorption of methylene blue on the hydrogel was investigated under various conditions such as contact time, initial concentration, and temperature.

2 Experimental

2.1 Materials

Graphene oxide was purchased from US Research Nanomaterials, Inc. Ammonium persulfate (APS), acrylamide, methyl blue, N, and N-methylene-bis acrylamide (MBA) were supplied by Merck, Co. Sodium alginate was purchased from Alpha Co. All solutions were prepared by deionized water (DI).

2.2 Instruments

For the preparation and analysis of the samples, various equipments used such as magnetic stirrer for mixing (Heidolph MR Hei-Standard), spectrophotometer for reading absorbance (Hach DR 2800), and X-ray diffractometer analysis for XRD (siemens-d5000).

Fourier transfer infrared (FTIR) spectrophotometer (Bruker Tensor 27 FT-IR) was used for measuring the surface characteristics between 400 and 4000 cm^{-1} . Scanning electron microscopy (SEM, Cam Scan MV2300, UK) was also used to determine the surface morphology of the prepared samples.

2.3 Methods

2.3.1 Preparation of three-component GSP composite hydrogels

Hydrogels were prepared by a simple mixture and solution polymerization using initial solutions consisting of monomers (GO, SA, and AM), a cross-linking agent (MBA), and an initiator (APS). At first, 150 mg of GO was dispersed in 10 mL of water by sonication for 3 h. Then, 1 g of sodium alginate and 2 g of acrylamide were dissolved in 10 mL of GO dispersion and magnetically stirred for 3 days at room temperature. The GO/SA/AM solutions were kept at 0 °C and then 0.02 g of ammonium persulfate and 0.03 g of methylene bisacrylamide were added. After magnetic stirring for 2 h at 0 °C, the homogeneous solutions were transferred to a glass tube and stored for 2 h at 80 °C to form a hydrogel. After the hydrogel formation, the three-component GSP composite hydrogels were taken as a

cylindrical shape from a glass tube and placed in deionized water to remove impurities in the hydrogel. Water was renewed every 8 h for 1 week.

2.3.2 Preparation of GO/AM/SA mixture

The preparation of this solution is in accordance with the synthesis of the hydrogel. The difference is that the cross-linking agent is less used [8, 9]. First, 150 mg of graphene oxide was stirred in 10 ml of water for 3 h. Then 2 g acrylamide and 1 g sodium alginate were added to aqueous graphene magnesium oxide aqueous solution for 3 days at room temperature. A solution of graphene oxide/acrylamide/sodium alginate mixture was placed in an ice bath. A total of 2 g of ammonium sulfate as a primer and 1 g methylene bis acrylamide was added to the grid for 2 h. Here, because the amount of grid is less than the previous method, the hydrogel does not form.

2.3.3 Hydrogel swelling

There is a fundamental relationship between the swelling of a polymer in the solvent and the nature of the polymer and solvent. Hydrogel swelling in distilled water was calculated using Eq. 1:

$$SR = \frac{(W_2 - W_1)}{W_1} \quad (1)$$

in which, w_1 and w_2 are the weights of the dried sample (g) and swollen hydrogel (g), respectively. Also, SR is the amount of swelling of the samples (g/g) [10].

2.3.4 Preparation of methylene blue dye solution

A mother solution with a concentration of 100 mg/L was prepared from methylene blue dye, and then the required concentration was prepared by diluting its certain volume [11].

2.3.5 Dye adsorption by adsorbent and percentage of dye removal

The desirable amounts of the dried hydrogels are first placed in deionized water to achieve equilibrium swollen states. The equilibrated swollen hydrogels were added, and dye adsorption experiments were studied at 25 °C for 1 week until the final equilibrium is reached. Before and after adsorption, the concentration of dye was checked using UV-Visb spectroscopy at $\lambda = 667$ [12, 13].

The equilibrium adsorption capacity, Q_e (mg/g), was calculated using Eq. 2:

$$q_t = \frac{(C_0 - C_t)V}{C_0} \quad (2)$$

in which C_0 and C_t (in mg/mL unit) are initial and final concentrations of dye solution, respectively. Also, V (mL) is the volume of dye solution and adsorbent mass, respectively.

The efficiency of dye removal by adsorbent is determined by the percentage of removal. To determine the percentage of removal (% RE) of this dye from the solution using the synthesized samples, Eq. 3 was used:

$$R(\%) = \frac{(C_0 - C_t)}{C_0} \times 100 \quad (3)$$

in which C_0 is the initial concentration of dye solution and C_t is the concentration per unit time obtained from spectrophotometer [12, 14].

2.3.6 Study on adsorption behavior

Adsorption behavior was investigated by Langmuir's adsorption isotherm model, which describes the single-layer adsorption on a homogeneous system. In the Langmuir adsorption isotherm, it is assumed that the adsorption process is dynamic. In equilibrium, the number of molecules that are adsorbed per unit time is equal to the number of molecules that are desorbed. Langmuir isotherm model is expressed by Eq. 4:

$$q_e = \frac{q_0 K_L C_e}{1 + K_L C_e} \quad (4)$$

In Eq. 4, q_e is the amount of adsorbed component per unit mass of the adsorbent (mg/g), q_0 is methylene blue adsorption capacity per unit hydrogel mass (mg/g), K_L is Langmuir isotherm constant (mg/L), and C_e is equilibrium concentration of the adsorbed substance (methylene blue) in the solution after adsorption (mg/L). The separation parameter (R_L) is shown in Eq. 5:

$$R_L = \frac{1}{1 + K_L C_0} \quad (5)$$

In Eq. 5, R_L is the separation parameter, which is a feature of the Langmuir isotherm, and C_0 is the initial concentration of methylene blue (mg/L).

One of the characteristics of the Langmuir equation is the dimensionless parameter of the separation coefficient, R_L , shown in Eq. 5 by which the type of adsorption process can be determined. If $R_L > 1$, the adsorption process is undesirable, if $R_L = 1$, the adsorption process is linear, if $0 < R_L < 1$, the adsorption process is desirable, and if $R_L = 0$, the adsorption is irreversible. Langmuir equation is consistent with empirical experiments when the adsorption sites are the same and the surface is uniform.

3 Results and discussion

3.1 FTIR spectra of adsorbents (hydrogel GSA, GO/AM/SA, GO)

Figure 1 shows the FTIR spectra of adsorbents (Hydrogel GSA, GO/AM/SA mixture and GO), which are taken to determine functional groups of GSA hydrogel samples, GO/AM/SA mixture, and GO. As is seen from Fig. 1, for hydrogel sample, there is a broad spectrum of superficial functional groups. A strong peak is observed at 3600 cm^{-1} , which is related to the stretching NH_2 group of acrylamide and sodium alginate hydrogels. The peaks observed at the wavelength of $3400\text{--}3500 \text{ cm}^{-1}$ for all three adsorbents are related to the stretching vibrations of OH groups.

The absorption of N-H in the region of $3300\text{--}3400 \text{ cm}^{-1}$ is covered due to the presence of a broad acidic O-H absorption peak. The new peaks appear at 2885 cm^{-1} , which are attributed to the C-O group of in hydrogel, GO, GO/AM/SA, and the carbon of carbonate species as a result of the interactions between AM and GO nanosheets. The peaks around 1620 and 1640 cm^{-1} are vibrations due to symmetric and asymmetric stretching of COO^- groups. The peak at 1600 cm^{-1} is related to C=C stretching vibrations which can also be found in graphene oxide. At the wavelength of 2900 cm^{-1} , stretching CH_2 band is observed for both polymers. The vibrations at $1000\text{--}1500 \text{ cm}^{-1}$ in hydrogel, graphene oxide, and GO/AM/SA are related to the COC and COH functional groups [15, 16].

3.2 Investigation of hydrogel and adsorbent textures by SEM

The synthesized hydrogel is shown in Fig. 2. For graphene oxide (Fig. 2a), the layered structure is crumbled and appears to be wrinkled due to the oxidation process [17]. SEM micrographs images of the GO/AM/SA mixture are presented in Fig. 2b. As is seen from Fig. 2b, the polymers are less bonded and have a dispersed structure. This structure is not layered

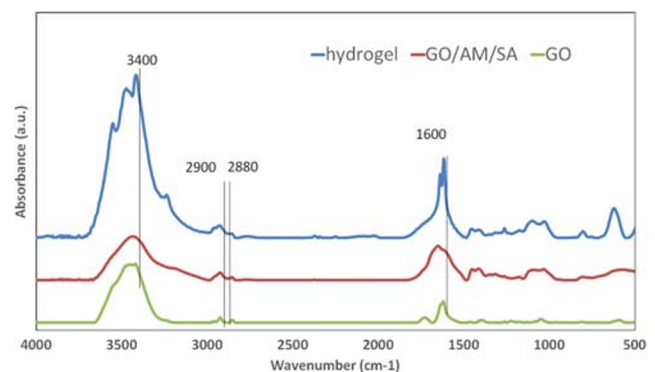


Fig. 1 FTIR spectra for different adsorbents (GSA hydrogel, GO/AM/SA mixture, and GO)

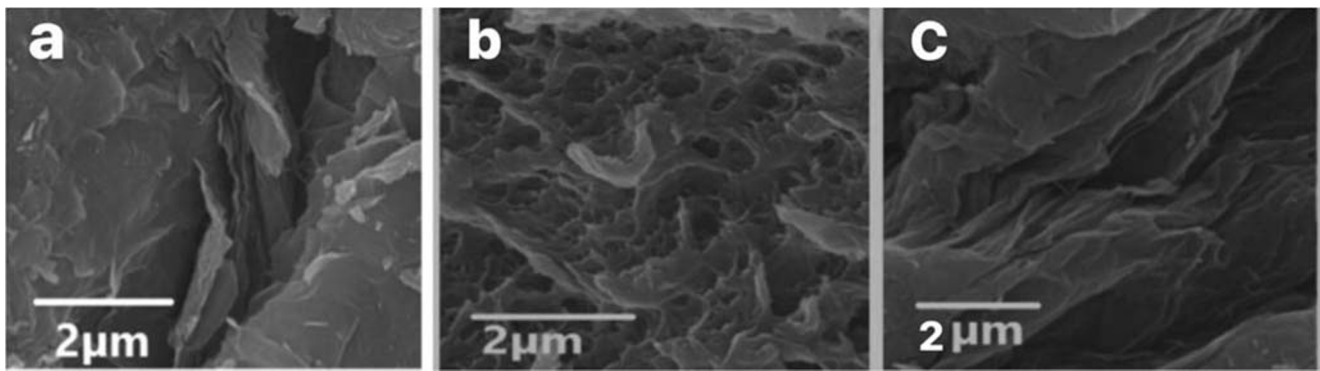


Fig. 2 SEM micrograph of GO (a), GO/AM/SA (b), and GSA hydrogel (c)

and continuous. Due to less *N,N'*-methylenebisacrylamide, network structure is not widely formed. Therefore, it has less adsorption than the hydrogel [18].

It is clear that hydrogel in Fig. 2c samples has a sponge-like structures with micrometer-sized porosities. Hydrogel holes are distributed uniformly on a large scale. For GO/AM/SA composite hydrogels, interconnected holes inside and outside of the sample are visible. These structures are in response with the participating GO, and therefore it is placed inside the hydrogel networks and leads to a compressed structure. The hydrogel has a uniform structure with numerous and contracted holes that are joined together. The porosity of the structure leads to increase in the surface area of the hydrogels and, as a result, faster water absorption. *N,N'*-methylenebisacrylamide improves structural integrity and also increases the swelling. The high porosity of hydrogel surfaces is a major factor for its high adsorption capacity [17, 19].

3.3 XRD analysis

XRD is an effective method to determine the orderly and irregular dispersion of graphene in a polymeric composite field. Since the average distance between diffraction planes can be calculated from Bragg's law, therefore, the diffusion of polymer between the graphene layers can be studied using Eq. 6:

$$d = \frac{\lambda}{2 \sin \theta} \quad (6)$$

In this respect, d is the distance between the bilayers, λ is the wavelength of the X-ray that is 1.54056 nm, and θ is the angle of radiation. To investigate the structure of GSA, GO/AM/SA, and GO hydrogels, X-ray diffraction (XRD) test in the range of 10 to 90 in 2θ was used. Figure 3 shows XRD patterns for the GSA hydrogel and the GO/AM/SA mixture and GO.

Graphene oxide (GO) has a strong sharp peak at $2\theta = 11^\circ$, indicating the structure of graphene with oxygen-containing groups, which have been successfully deposited on the graphene surface that was created by a strong oxidation reaction of graphene. It is observed that when the composite is formed, GO is shifted from the angle of 11° to 23° , which is equivalent to change the distance between GO plates from 0.743 to 0.386 nm. Hence, it can be stated that GO can disperse in a solution of the SA and AM mixtures and form a uniform solution. As is seen from Fig. 3, GO nanosheets are uniformly distributed in three-component nanocomposite hydrogels [20].

3.4 Influence of swelling behavior of graphene oxide/acrylamide/sodium alginate at different temperatures

Swelling behavior of hydrogels at different temperatures (25, 50, 70, and 90 °C) was investigated according to the Eq. 1. According to Fig. 4, it is found that at 90 °C, the swelling is in the maximum and at 50 °C is minimum. The reason for this can be attributed to the fact that high temperatures weakened or even destroyed hydrogen bonding between water

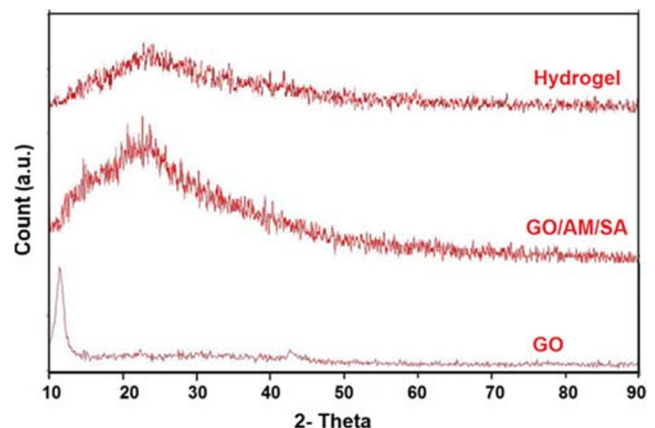


Fig. 3 XRD patterns of GSA hydrogel, GO/AM/SA mixture, and GO

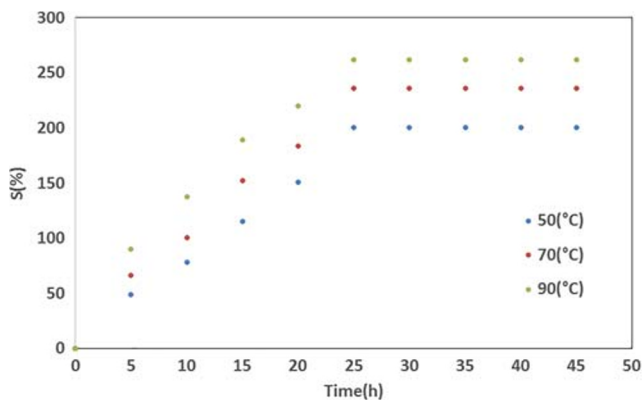


Fig. 4 Swelling percent of the hydrogel at different temperatures

molecules, acrylamide, and sodium alginate. But at low temperatures such as 25 and 50 °C, the hydrogen bonding and the intramolecular and intermolecular complexes between water molecule, acrylamide, and sodium alginate rigidify the mixture and inhabit higher water diffusion into the hydrogel. Therefore, the percentage of swelling at lower temperatures is lower. After 25 h, the amount of swelling remained constant and the percentage of swelling remained unchanged, indicating that after 25 h, the amount of water absorbed by the hydrogel was constant [12, 21].

3.5 Effect of contact time on methylene blue dye adsorption

Adsorption of methylene blue dye was investigated with three different adsorbents (GSA hydrogel, GA/AM/SA mixture, and graphene oxide) at different times (195–155 min). As shown in Fig. 5, adsorption of methylene blue increased with increasing contact time. The highest removal rate was at the first 150 min. At first, the adsorption rate of methylene blue was high and continued until adsorption sites occupied so that the highest rate of adsorption occurred during the first 150 min then the adsorption rate decreased simultaneously with the occupancy of the adsorption sites and finally became constant. The reason for having no significant changes in adsorption after 150 min by all three adsorbents can be due to the

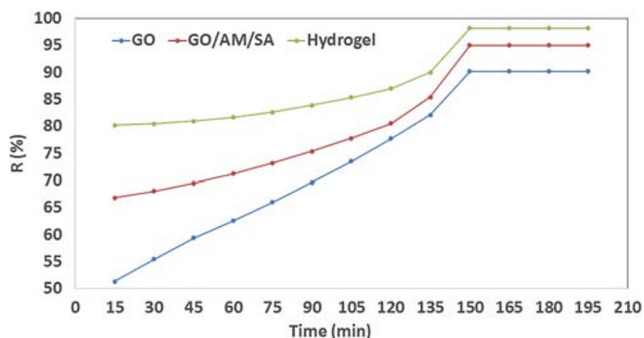


Fig. 5 Effect of contact time on the dye adsorption process

occupancy of active sites of adsorbent or methylene blue resorption, while, after reaching the time of equilibrium, the resorption rate is equal to the adsorption rate and there is no change in the removal efficiency. The rapid adsorption of methylene blue dye by GSA hydrogel adsorbent can be attributed to the high adsorption level, and since the number of active sites for dye adsorption in a given system is limited, over time and the saturation of active sites, the adsorption increases more rapidly. By comparing the removal percentage of the three adsorbents, according to the figure, the methylene blue removal percentage by GSA hydrogel adsorbent is more than GO/AM/SA and graphene oxide, respectively [22].

3.6 Effect of temperature on the adsorption of methylene blue dye

Adsorption of methylene blue dye with three different adsorbents (GSA hydrogel, GA/AM/SA mixture, and graphene oxide) under the constant concentration of 60 mg, 150 min equilibrium time, 0.01 g adsorbent at various temperatures (20 °C to 120 °C) was checked in the hot water bath. The temperature rises to 120 °C since it has accomplished in a thermodynamically closed system. Temperature can play an important role in the adsorption process. The results show that with increasing the solution temperature, the adsorption capacity of the hydrogel GSA becomes higher than other adsorbents. According to Fig. 6, at higher temperatures, more active sites are available for adsorption of cationic dyes, which due to increased cationic dye mobility, the amount of adsorption has increased [23, 24].

3.7 Effect of methylene blue dye concentration on the adsorption

Effect of methylene blue concentration on the adsorption capacity of adsorbents by changing the initial concentration of dye (from 20 to 120 ppm) was studied. The results are shown in Fig. 7. As indicated in this figure, with increasing the initial dye concentration from 20 to 120 mg/L, the adsorption capacity is increased. As shown in Fig. 7, which is depicted for a

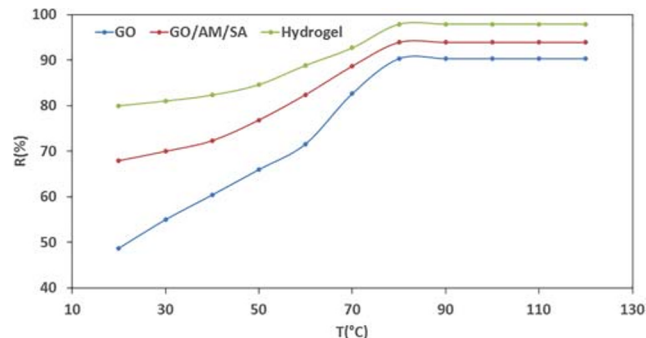


Fig. 6 Effect of temperature on adsorption process by three adsorbents

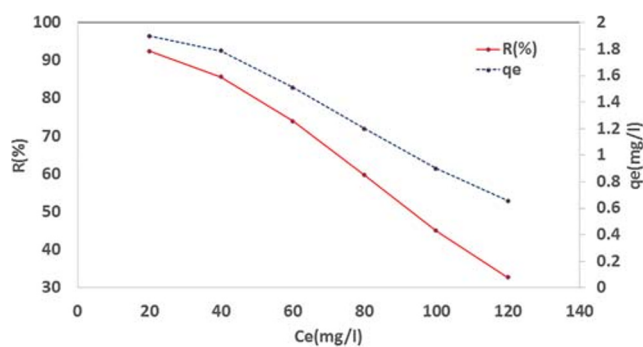


Fig. 7 Effect of concentration on adsorption capacity and removal percent of GSA hydrogel

hydrogel adsorbent, it is found that by increasing the initial concentration of the dye, a higher amount of dye interacts with the adsorbent surface and causes increasing the adsorption capacity. The similar studies have been done by researchers to study the initial concentration for the removal of methylene blue dye. By increasing the initial concentration of dye, its adsorption capacity increases. On the other hand, at the higher concentration of dye, its adsorption capacity decreases. By increasing the concentration from 20 to 120 mg/L, adsorption percent of methylene blue dye decreases from 95.76% to 35.989%. In Fig. 8, concentrations are investigated with various adsorbents that we find that the hydrogel has the highest percentage removal at lower concentrations [25].

3.8 Adsorption isotherm of methylene blue

The experimental adsorption equilibrium data from the Langmuir isotherm model as Eq. 4 were used to describe the state of adsorbed component between the solid and fluid phases.

In Eq. (4), q_e is the amount of adsorbed component per unit mass of adsorbent (mg/g), q_0 is the methylene blue adsorption capacity per hydrogel mass unit (mg/g), K_L is the Langmuir isotherm constant (L/mg), and C_e is equilibrium concentration of the adsorbed substance (methylene blue) in the solution

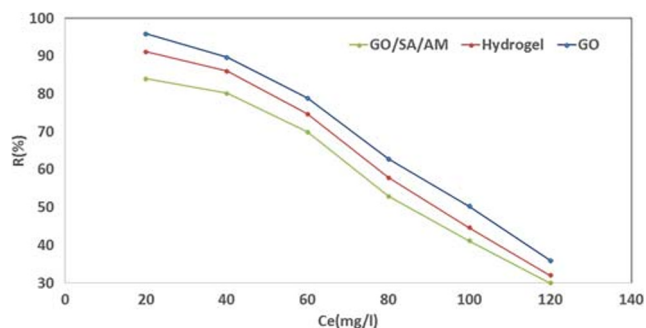


Fig. 8 Effect of initial dye concentration on dye adsorption process

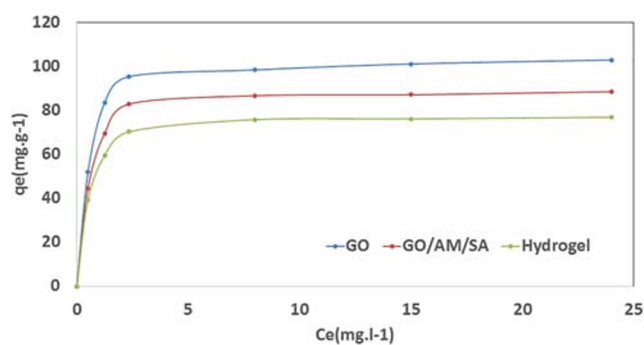


Fig. 9 Langmuir isotherm for methylene blue adsorption by different adsorbents (GSA hydrogel, GO/MA/SA, and GO)

after adsorption (mg/L). The R_L separation parameter is shown in Eq. 5.

The adsorption isotherms provide the distribution of molecules between the liquid and solid phases in the adsorption process under equilibrium conditions. Study on the adsorption isotherm provides valuable information about hydrogel structure. In Fig. 9 and Table 1, the results of the Langmuir isotherm are shown for samples of methylene blue adsorption. As shown from the table, the adsorption capacity for the sample prepared by the GSA hydrogel (110.2 mg g^{-1}) is higher than GO/AM/SA (99.877 mg g^{-1}) and graphene oxide (GO) (86.121 mg g^{-1}). Increasing the adsorption capacity of the GSA hydrogel in comparison with GO/AM/SA and graphene oxide (GO) suggests that hydrogel has a significant ability to create micro-porosity in its structure and has been able to successfully improve micro-active sites that are most often occurred therein. The R_L dimensionless coefficient (separation coefficient) is less than 1 for all three samples, which indicates adsorption is desirable. The adsorption capacity for hydrogel, GO/AM/SA, and graphene oxide are 110.2, 99.897, and 86.121, respectively, indicating the high adsorption capacity for adsorbents [25].

4 Conclusions

The aim of this study was to obtain useful information for the development and evaluation of the effectiveness of one of the newest technologies to remove textile dyes from the aqueous solution. In this study, the GSA hydrogel containing sodium alginate and graphene oxide was synthesized. The GSA hydrogel structure and

Table 1 Langmuir isotherm parameters for the used adsorbents

Absorbent	$q_0(\text{mg g}^{-1})$	$K_L(\text{L mg}^{-1})$	R^2	R_L
Hydrogel GSA	110.2	2.21	0.942	0.022
GO/AM/SA	99.877	1.45	0.899	0.033
GO	86.121	1.23	0.833	0.038

the GO/AM/SA mixture and graphene oxide (GO) were investigated by Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and X-ray diffractometer analysis (XRD). The swelling behavior of the hydrogel at temperatures of 25, 50, 70, and 90 °C was investigated. The higher the temperature, the higher the amount of swelling.

Adsorption experiments were carried out to remove methylene blue from aqueous solution. The effects of various parameters such as initial dye concentration, contact time, and temperature were investigated. The results of the experiments showed that for the adsorption of methylene blue dye, the optimum adsorbent is 0.01 g and the optimum time is 150 min. The results obtained from the methylene blue adsorption isotherm on GSA hydrogel showed that this hydrogel had an acceptable efficiency (95.76%) for dye removal against magnetic hydrogels with the adsorption (91%) from the aqueous solution.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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