An Electrospun Zein/Graphene Oxide Nanofibrous Composite: Typical Application as a New Biopolymeric Adsorbent in Removal of Methylene Blue and Malachite Green Dyes from Aqueous Media

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ABSTRACT

In this study, for the first time, an electrospun nanofibrous (Nfs) composite was prepared from Zein biopolymer and graphene oxide (Ze-GONfs). The effective parameter in electrospining preparation of zein-GO nanofiber was the amount of zein. Highly mechanical stable membranes were obtained using 30% w/v in glacial acetic acid. The fiber diameter distribution was in the range of 41–50 nm (zein), 31–40 nm (0.5% GO), 29–39 nm (1.0% GO), and 28–37 nm (1.5% GO). The structural morphology of the nanofibrous composites were characterized using Fourier transform infrared, scanning electron microscope (SEM) and X-ray diffractometer. As an application of the prepared biopolymeric nanofibrous, the removal of malachite green (MG) and methylene blue (MB) (as cationic industrial dyes) from aqueous medium using Ze-GONfs was investigated as a model. The effect of various parameters such as solution pH, adsorbent dosage, dye concentration, time and temperature were evaluated by the Ze-GONfs. Detailed analysis of the adsorption kinetics showed that the adsorption process followed a pseudo-second-order model. The adsorption isotherm was best fitted by the Langmuir model. The thermodynamic results showed that MB and MG adsorption onto the Ze-GONfs was endothermic and spontaneous. Prog. Color Colorants Coat. 14 (2021), 55-65© Institute for Color Science and Technology.

1. Introduction

At present, water contamination has been the main problem as a serious public and scientific concern. Effluents come from different industries like wastewater from textile, pharmaceutical, printing, cosmetics, rubber, and food industries [1, 2]. The presence of dyes in water inhibit the penetration of sunlight and oxygen, generating numerous problems for ecosystem wastewater [3]. Additionally, most of the dyes are considered toxic and carcinogenic and are difficult to remove because of their complex chemical structure [4]. Thus, removing toxic dyes from wastewater represents a challenge for the researchers and the environment [3, 4]. There are several methods for the treatment of wastewater, including ozonation, coagulation flocculation, advance oxidation, reverse osmosis, membrane ultrafiltration, and photo degradation [5]. However, these methods have higher operating costs in dye wastewater treatment, while
adsorption process is highly interested due to its efficiency, simplicity and flexibility of design as it can eliminate different types of pollutants more efficiently. Over the years, various types of nanoadsorbents such as carbon-based materials, mineral oxides, organic polymer materials and biosorbents have been reported for removing pollutants from industrial wastewater [6]. Among them, organic polymers have been increasingly used to remove organic pollutants from wastewater due to their good elasticity, high adsorption capacity and low density [7].

Graphene oxide (GO) is prepared from natural graphite, which is a promising adsorbent material for the removal of cationic dye molecules from aqueous solutions [8, 9]. The GO has large surface area and contains carboxyl, hydroxyl, carbonyl, and epoxide groups. These functional groups in graphene enhance the negative charge density on the layer in basic medium [10].

Electrospinning is a simple method for producing nanofibers and nonwovens mats as compared to regular fibers [11]. Nanofiber offers some promising properties such as high porosity, light weight, and large surface area to volume ratio which are suitable for adsorption. Nowadays, electrospinning has become more popular as new class of materials notable for nanofiber below 100 nm in diameter [12, 13]. These properties make the electrospun membrane very suitable for water treatment. Electrospun polymer fiber has been successfully used in drug delivery [14], tissue engineering scaffolds [15] electronic devices [16], water filtration [17], and so on.

Zein is an inexpensive byproduct fraction of corn protein that contains more than 50% non-polar amino acids [18]. Zein, as a water insoluble natural biopolymer with high biodegradable and biocompatible properties, can be used as an organic material adsorbent in environmental cleanup as well as drug delivery in medicinal sciences. Recently, nanofibers of Ze/GO have been used as an efficient compatible adsorbent for in vitro drug delivery of tetracycline hydrochloride [19].

In this study, Ze-GONFs composite was produced by electrospinning technique and used for the removal of methylene blue (MB) and malachite green (MG) (Figure 1) from aqueous medium. The physical and chemical properties of the nanofibrous composites were studied using scanning electron microscopy (SEM), Fourier transform infrared (FTIR), and X-ray diffractometer (XRD) techniques. The dye removal efficiency and adsorption capacity of the adsorbent was studied using the UV–Vis spectrometer.

2. Experimental

2.1. Materials

All the chemicals used in this study were of analytical reagent grade. Graphite was purchased from Fluka (Buchs, Switzerland). Zein, MB (C_{16}H_{18}ClN_{3}, M.W. 373.91, ≥ 97%), and MG (C_{23}H_{25}ClN_{2}, M.W. 364.91, ≥ 99%) were purchased from Sigma-Aldrich (St. Louis, MO, USA). Potassium permanganate (99%), hydrogen peroxide (30%), and hydrochloric acid (30%) were purchased from Merck Co. (Darmstadt, Germany). All the reagents were used as received without further purification.

![Figure 1: Chemical structure of malachite green (MG) (A) and methylene blue (MB) (B)](image-url)
2.2. Synthesis of graphene oxide (GO)

GO was synthesized by the modified Hummers method [20]. In this method, 270 mL sulfuric acid (H\textsubscript{2}SO\textsubscript{4}) and 30 mL phosphoric acid (H\textsubscript{3}PO\textsubscript{4}) (volume ratio 9:1) were mixed and stirred for several minutes. Briefly, 3 g graphite powder was added into the solution under stirring in a 500 mL reactor for 20 min in an ice bath to obtain a homogeneous solution. Then, 9 g of KMnO\textsubscript{4} was added slowly under continuous stirring at temperatures below 20 °C. Then, the suspension was stirred for 6 hours until the solution became dark green at 40 °C. To eliminate the excess KMnO\textsubscript{4}, 20 mL of H\textsubscript{2}O\textsubscript{2} (30 wt%) was added and the solution was vigorously stirred for 20 min. Then, the mixed solution was cooled at room temperature. The obtained product was centrifuged and washed consecutively with 250 mL of 10% HCl solution and 1 L deionized water. Then, the samples were centrifuged five times in deionized water to obtain a solution with a pH of 6. After that, the suspension was dried at 60 °C. The GO yield was about 60%.

2.3. Preparation of Ze/GONfs composite

Ze-GONfs composite was prepared using partial modified electropinning technique according to the method reported by Asadi et al. [19]. Briefly, different masses of GO (0.0, 0.02, 0.04, 0.06 g) were separately dispersed in 5 mL ethanol using an ultrasonic probe device at 100 W for 30 min to obtain GO-dispersed mixtures with various concentrations. Then, 1.0 g zein biopolymer was added to the GO dispersion mixture and stirred until the solution became homogeneous. Therefore, the zein concentration used for fabricating the nanofibers was fixed at 25% w/v and weight concentrations of GO in fabricated mats were 0, 0.5, 1.0 and 1.5 % w/v. Subsequently, the obtained solutions were immediately loaded in a 1 mL syringe with 20-G stainless steel needle in order to prevent phase separation. Electrospinning (Fanavaran NanoMeghyas, Iran) was performed using a voltage of 12 kV, a working distance of 15 mm, and a flow rate of 0.1 mL/h. The collected nanofibers were then dried under vacuum for 24 h at 40 °C to remove any residual solvent.

2.4. Adsorption and kinetic experiments

Aqueous stock solutions of MB and MG (1000 mg/L) were prepared and further diluted to the required concentrations of 20, 40, 50, 60, 80, 100 and 120 ppm for the adsorption studies. The pH of the MB and MG dye solutions was adjusted using 1 M HCl or NaOH solution. In adsorption experiments, Ze-GONfs were individually treated with desired concentration of dye solution. All experiments were carried out at room temperature with continuous shaking (120 rpm). In all experiments, the amount of MB and MG was measured before and after exposure to the adsorbent at maximum absorption wavelength of 663 and 624 nm, respectively.

3. Results and Discussion

3.1. Surface morphology of nanofibers

The SEM technique was used for the characterization of the nanofibers diameter. Figure 2 (a–d) shows SEM images of Ze-GONfs prepared from 0.0, 0.5, 1.0, and 1.5 wt% GO blended with Zein. The figure shows that the fibers were randomly distributed in the nanofiber composite. The fiber diameter was in the range of 41–50 nm (zein), 31–40 nm (0.5% GO), 29–39 nm (1.0% GO), and 28–37 nm (1.5% GO). From these results, it could be concluded that the diameter of the fibers decreased by increasing the amount of GO in the mixture.

XRD patterns of the GO, Zein and Ze-GONfs samples are shown in Figure 3. Pure zein nanofibers showed a peak at 2\(\theta\) = 9° and a wide peak at 2\(\theta\) = 20°. XRD patterns of GO nanofibers showed a characteristic peak at 11.1, which is in good agreement with the literatures [21]. Three diffraction peaks at 2\(\theta\) = 11.2°, 9.1°, and 20.4° could be indexed to the (111), (200), and (222) planes in Ze-GONfs.

FTIR spectra of the Zein, GO and Ze-GONfs was shown in Figure 4. In the case of GO, the presence of carbonyl group at 1720 cm\(^{-1}\) (C=O stretching vibration) and the epoxy groups at 1220 cm\(^{-1}\) revealed the existence of oxygen-containing functional groups on the GO surface. The broad peak from 2500 to 3500 cm\(^{-1}\) suggests the presence of COOH group. The zein fiber showed typical absorption peaks at 1649 cm\(^{-1}\) and 1538 cm\(^{-1}\) which represent the characteristic vibrational bands of pure zein known as amide I and II. The presence of amine groups in zein biopolymer is confirmed by the appearance of two peaks at 1449 and 1612 cm\(^{-1}\) related to N-H bending vibration. Meanwhile, the broad peak at 3300 cm\(^{-1}\) is attributed to the hydroxyl stretching vibration. The spectrum of the Ze-GONfs is almost similar to that of the zein except that the peak at 1712 cm\(^{-1}\) belongs to the stretching vibration of carbonyl functional group in carboxylic acid moiety of GO which proves the presence of GO in the nanofibers composite.
Figure 2: SEM images of electrospun (a) Zein, (b) Ze-1 GONfs, (c) Ze-2 GONfs and (d) Ze-5GONfs samples (10000X).

Figure 3: The XRD patterns of the GO, ZeNfs and Ze-GONfs samples.
3.2. Adsorption studies

3.2.1 Batch adsorption experiments

Batch adsorption experiments were conducted to investigate MB and MG adsorption by varying pH, adsorbent dosage, initial MB concentration, contact time and temperature. In a typical experiment, 0.01 g of adsorbent was added to a beaker containing 10 mL of 20 mg/L MB or MG solution. The pH of the solution was then adjusted with 0.1 mol/L HCl and NaOH and test tube was fixed to a shaking table (120 rpm) controlled at a desired temperature (285-308 K). The adsorption capacity ($q_e$, mg/g) and the sorbent removal percentage were calculated by the following equations.

$$ q_e = \frac{V(C_0 - C_e)}{m} $$  

(1)

Removal(%) = $\frac{100(C_0 - C_e)}{C_0}$  

(2)

where $q_e$ is the quantity of adsorbate per unit weight of Ze-GONFs (mg/g), $C_0$ and $C_e$ are the initial and equilibrium concentration (mg/L) of MB and MG dyes in solution after adsorption procedure, respectively. $V$ (L) is the volume of solution (L) and $m$ is the mass (g) of the sorbent added.

3.2.2. Effect of pH

The surface charge of the adsorbent, the dissociation of functional groups on the active sites of the adsorbent, the degree of ionization of the adsorbent and the structure of the dye molecule can be changed with the pH of solution [22]. The point of zero charge ($pH_{PZC}$) is an important property identifying the surface electrical neutrality of the adsorbent. In order to determine $pH_{PZC}$, the difference between the final and initial pH values ($\Delta pH = pH_f - pH_i$) was plotted versus the initial pH values ($pH_i$) and the $pHzpc$ was found to be ~5.75. The results are depicted in Figure 5. It can be concluded that at pH values above $pH_{PZC}$, the Ze-GONFs surface is considered to have a predominantly negative charge, more likely for the impregnation of the cationic dyes.

The removal percentage of MB and MG over a pH range from 2 to 10 is plotted in Figure 6. For MB at pH > $pH_{PZC}$, the surface of the adsorbent was negatively charged and the adsorption of MB was increased [23]. At pH of 6.0 and contact time of 60 min at 25 mg/L dye, the maximum adsorption was found to be 94%
through the electrostatic interaction between adsorbent functional groups and the cationic dye. At low pH values, the H$^+$ ions compete with positively charged MB ions for the adsorption sites of the Ze-GONFs. So, the removal efficiencies are diminished [24]. A gradual increase in uptake capacity was observed with the increase in pH from 2.0 to 5.0. A similar result was observed for the adsorption of MG. By increasing pH values from 2 to 7, the adsorption of MG increases rapidly from 76 to 89% due to the electrostatic interaction between the dye and the negatively-charged surface of the Ze-GONFs. The pHzpc of Ze-GONFs was ~ 5.75 which means that at pHs < pHzpc, the sorbent surface was positively charged and repulsion occurred, while at pHs > pHzpc, the surface of the adsorbent was negatively charged, so it favored the adsorption of cationic MG dye [25].

Figure 5: Plot of $\Delta$pHs ($\Delta$pH = pH$_{f}$ − pH$_{i}$) values vs. initial pH values (pH$_{i}$) to determine the point of zero charge of Ze-GONFs.

Figure 6: Effect of pH on the removal of MB and MG by Ze-GONFs (initial dye concentration = 25 mg/L, agitation speed = 120 rpm, adsorbent dose = 0.01 g, contact time = 60 min, temperature = 298 K).
3.2.3. Effect of adsorbent dosage

Adsorbent dose is an important parameter influencing the adsorption processes since it determines the adsorption capacity of an adsorbent. The effect of Ze-GONFs dosage on removal of MB and MG was studied at adsorbent dosages ranging from 1 to 14 mg. Figure 7 shows the removal percentage of MB and MG at various mounts of the sorbent. As can be seen, the removal efficiencies increase from 34 to 93% for MB and from 24 to 86 % for MG by increasing the sorbent mass. This fact is attributed to the increase in the adsorptive surface area and the volume of the binding sites [26]. Based on these results, the optimal amounts of Ze-GONFs for the removal of MB and MG from 25 mL of 20 mg L\(^{-1}\) dye solution are 8 and 10 mg, respectively.

3.2.4. Adsorption isotherm

The isotherms show the relationship between the concentration of adsorbate in solution and the amount of adsorbate adsorbed by the adsorbent at equilibrium. The influence of MB and MG concentration on the adsorption capacity was studied at concentrations ranging from 2 to 100 mg L\(^{-1}\) on Ze-GONFs. Figure 8a shows that adsorption of MB and MG decreased from 2 to 50 mg/L and then decreased slowly when initial concentration was reached to 100 mg/L. This behavior may be attributed to the restriction of identical sites in Ze-GONFs occupied by MB and MG molecules. Figure 8b shows the effect of dye concentration on the adsorption capacity of Ze-GONFs. As can be seen, increasing the initial dye concentration enhances the adsorption capacity of Ze-GONFs and the maximal adsorption capacity (83.6 and 73.75 mg/g) is obtained under the initial MB and MG concentration of 100 mg/L.

The effect of initial adsorbate concentration of MB and MG on adsorption capacity of Ze-GONFs was explained by analyzing different adsorption isotherms. Adsorption isotherm describes adsorbate molecule distribution in the solid-liquid interface at equilibrium and adsorption capacity. Herein, experimental adsorption of MB and MG on Ze-GONFs was correlated with Langmuir and Freundlich as conventional model [27]. The Langmuir and Freundlich equations can be illustrated as:

\[
\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{K_L q_m}
\]

(3)

\[
logq_e = \frac{1}{n} logC_E + LogK_F
\]

(4)

**Figure 7:** The effect of adsorbent dosage on the removal of MB and MG by Ze-GONFs.
where $q_m$ is the maximum adsorption capacity (mg/g), $C_e$ is the equilibrium solution phase concentration, and $K_L$ (L/mg) is the Langmuir adsorption equilibrium constant. $K_L$ and $q_m$ values can be obtained from the intercept and slope of $C_e/q_e$ plot vs $C_e$, respectively.

In Freundlich model, $K_F$ (L/mg) is the Freundlich constant, $n$ is the empirical parameter indicating heterogeneity factor obtained from the slope and intercept of log$q_e$ vs. log $C_e$, respectively. The obtained experimental data for adsorption of MB and MG on Ze-GONFs based on Langmuir and Freundlich isotherm models is represented in Figure 8c and Figure 8d. Also, Table 1 shows the isotherm parameters and regression coefficient ($R^2$) values. Ze-GONFs satisfies Langmuir adsorption isotherm due to the high correlation coefficient value. This attributed that monolayered and homogeneous adsorption of MB and MG has occurred in Ze-GONFs. The maximum adsorption capacity obtained is 81.3 and 86.95 mg/g for MB and MG, respectively.

![Figure 8: Effect of initial concentrations of MB and MG (a) and adsorption isotherm plots of Langmuir (b, c), Freundlich (d) (pH = 7.0 and 6 for MB and MG, respectively, T = 298 K).](image)

**Table 1: Adsorption isotherms constants.**

<table>
<thead>
<tr>
<th>Dye</th>
<th>$q_{(\text{max})}$ (mg g$^{-1}$)</th>
<th>$(K_L, q_{(\text{max})}^{-1})$</th>
<th>$R^2$</th>
<th>$K_F$</th>
<th>$n$</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>MB</td>
<td>81.30</td>
<td>0.15</td>
<td>0.99</td>
<td>12.01</td>
<td>1.92</td>
<td>0.96</td>
</tr>
<tr>
<td>MG</td>
<td>86.95</td>
<td>0.14</td>
<td>0.99</td>
<td>23.41</td>
<td>2.69</td>
<td>0.88</td>
</tr>
</tbody>
</table>
3.2.5. Adsorption kinetic

The adsorption mechanism of MB and MG onto Ze-GONfs was analyzed by two linear kinetic models including Lagergren pseudo-first-order model (PFO) and pseudo-second-order model (PSO). The pseudo-first-order model was described by Lagergren as (Eq. 5).

\[
\log(q_e - q_t) = \log q_e - \frac{kt}{2.303}
\]

The pseudo-second-order kinetic model is expressed as Eq. (6):

\[
\frac{t}{q_t} = \frac{1}{kq^2} + \frac{t}{q_e}
\]

where \(q_e\) (mg/g) is the amount of dye adsorbed at equilibrium; \(q_t\) (mg/g) is the amount of dye adsorbed at time \(t\) (min); \(k_1\) (min\(^{-1}\)) is the rate constant of the PSO adsorption process (min\(^{-1}\)), and \(k_2\) is the rate constant of the PSO model of adsorption (g mg\(^{-1}\) Min\(^{-1}\)).

The constants can be calculated from the intercepts and slopes of the linear plots of \(\log(q_e - q_t)\) versus \(t\) (Eq. (5)) and \(t/q_t\) versus \(t\) (Eq. (6)), respectively, as shown in Figure 9 (a, b). The adsorption kinetic parameters and correlation coefficients are listed in Table 2. It is clear that the calculated \(q_e\) values (\(q_{e,\text{cal}}\)) from the PSO model is close to the experimental values (\(q_{e,\text{exp}}\)), whereas the values calculated by the PFO models differed significantly. Furthermore, the \(R^2\) value of the PSO kinetic model is higher than that of PFO model, indicating that the kinetics of MB adsorption follow the PSO kinetic model.

3.2.6. Effect of temperature

The study of temperature dependence during adsorption process gives valuable information on the enthalpy and entropy changes accompanying adsorption processes. The effect of temperature on the adsorption of MB and MG at 50 mg L\(^{-1}\) onto Ze-GONfs as a function of temperature is presented in Figure 10. Thermodynamic parameters such as enthalpy (\(\Delta H\)), entropy (\(\Delta S\)) and Gibb’s free energy (\(\Delta G\)) were determined by Eq. (7) and (8) [28].

\[
\ln K_c = \frac{\Delta S}{R} - \frac{\Delta H}{R} \cdot \frac{1}{T}
\]

\[
\Delta G = \Delta H - T \Delta S
\]

where \(K_c\) is the equilibrium constant, \(C_e\) is the equilibrium concentration (mgL\(^{-1}\)) of dyes in solution, \(q_e\) is the amount of dyes adsorbed at equilibrium (mg g\(^{-1}\)), \(R\) is the gas constant (8.314 Jmol\(^{-1}\)K\(^{-1}\)) and \(T\) is the temperature (K). \(\Delta H\), \(\Delta S\) and \(\Delta G\) are changes in enthalpy (kJmol\(^{-1}\)), entropy (Jmol\(^{-1}\)K\(^{-1}\)) and Gibb’s free energy (kJmol\(^{-1}\)), respectively. The equilibrium constant, \(K_c\), can be defined as:

\[
K_c = \frac{C_{ads}}{C_e} = \frac{C_e - C_{e,\text{eq}}}{C_e}
\]

Table 2: Kinetic parameters for MB and MG adsorption onto GONfs.

<table>
<thead>
<tr>
<th>Dye</th>
<th>(q_{(max)}) (mg g(^{-1}))</th>
<th>(k_1)</th>
<th>(q_{e,\text{cal}}) (mg g(^{-1}))</th>
<th>(R^2)</th>
<th>(k_2)</th>
<th>(q_{e,\text{cal}}) (mg g(^{-1}))</th>
<th>(R^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MB</td>
<td>85.13</td>
<td>0.069</td>
<td>31.18</td>
<td>0.88</td>
<td>0.0075</td>
<td>86.2</td>
<td>0.99</td>
</tr>
<tr>
<td>MG</td>
<td>155.8</td>
<td>0.079</td>
<td>43.71</td>
<td>0.91</td>
<td>23.41</td>
<td>149.25</td>
<td>0.99</td>
</tr>
</tbody>
</table>

Figure 9: a) Pseudo-first-order kinetics and b) second-order kinetics for adsorption of MB and MG using Ze-GONfs.
Table 3: Thermodynamic parameters for MB and MG adsorption onto Ze-GONfs.

<table>
<thead>
<tr>
<th>Dye</th>
<th>∆H (kJmol(^{-1}))</th>
<th>∆S (Jmol(^{-1})K(^{-1}))</th>
<th>∆G(^0) (kJmol(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>298 K</td>
<td>308 K</td>
<td>313 K</td>
</tr>
<tr>
<td>MB</td>
<td>70.12</td>
<td>19.631</td>
<td>17.910</td>
</tr>
<tr>
<td>MG</td>
<td>33.70</td>
<td>5.992</td>
<td>4.062</td>
</tr>
</tbody>
</table>

where \(C_0\) (mg/L) and \(C_e\) (mg/L) represent the initial and equilibrium concentrations of adsorbate, respectively.

The values of ∆H and ∆S were determined from the slopes (∆H /R) and intercept (∆S /R) of the plots of Ln (\(K_c\)) vs. 1/T. The ∆G values were calculated using Eq. (8). The values of thermodynamic parameters are presented in Table 3. Negative values of ∆G indicated that the adsorption process was feasible and spontaneous in nature. Negative values of ∆H suggested that the exothermic nature of adsorption and negative values of ∆S described the randomness at the adsorbent-solution interface decreased during the adsorption.

4. Conclusions

This study showed that Ze-GONfs act as a good adsorbent for the removal of MB and MG from aqueous solutions. The adsorption characteristics of MB and MG in aqueous solution were shown to be dependent on solution pH, adsorbent concentration, initial dye concentration, and contact time. Isotherm modeling revealed that the Langmuir equation could better describe the adsorption of the dyes onto the Ze-GONfs as compared to other models. Kinetic studies have shown that adsorption may follow a second-order kinetic model, although the correlation coefficients of first-order quasi-kinetic model were high. The thermodynamic parameters indicated that the adsorption was spontaneous, exothermic and physical in nature.

5. References

6. G. N. Hlongwane, P. T. Sekoai, M. Meyyappan, K.


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