

UV-Spectroscopic Interaction Studies of Synthesized Graphene Oxide and Methyl Orange

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Abstract

Graphene oxide (GO) has become a popular material due to its valuable properties and wide range of applications in various fields. GO was synthesized using a modified Hummers' method and characterized through various techniques, including X-ray diffraction (XRD), and scanning electron microscopy (SEM). The effectiveness of GO in decolorizing methyl orange was assessed using UV-Vis spectrophotometry, with varying GO concentrations (0.05, 0.075, 0.1, 0.15) g and time (2–120 minutes) intervals. The study demonstrated that GO effectively adsorbs and removes MO from aqueous solutions. Specifically, at a GO concentration of 0.15 g, a 90.14% reduction in MO absorbance was observed after only 10 minutes, whereas at 0.05 g, 0.075 g, and 0.1 g GO, the reductions were 58.38% and 71.65%, and 80.61, respectively. At 120 minutes adsorption efficiency of 0.15 g is 99.57%. At 120 minutes the color of MO had become completely transparent. These results highlight the potential of GO as a highly efficient material for dye removal and provide insights into optimizing its use for environmental remediation applications.

Keywords: Graphene oxide, decolorization, methyl orange, dye removal, UV-Vis spectrophotometer

INTRODUCTION

Graphene oxide (GO) is a derivative of graphene that has attracted significant interest due to its functional groups, which make it versatile for various applications. GO is produced by oxidizing graphite, resulting in a material with a high surface area and numerous oxygen-containing functional groups such as epoxides, hydroxyls, and carboxyl.

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Hazardous environmental contaminants known as organic dyes are frequently detected in wastewater released from various industries, such as textiles, printing, dyeing, food and pharmaceutical processing, cosmetics, and paper products. These dyes are not biodegradable and are resistant to light and oxidants due to their extremely stable molecular structures [1–3]. Industries are extremely concerned about how to treat industrial effluent to make their methods more environmentally friendly [4]. Based on their advantages and disadvantages, several writers have employed various treatment modalities to reduce the number of dyes in the environment [5, 6]. Among the methods used to treat dye-contaminated water are coagulation, ion exchange, electrochemical treatment, and adsorption have been used recently. Adsorption is widely regarded as the most effective water treatment method due to its simplicity and universal application. Various adsorbents, from conventional to nanomaterials,

have been tested for pollutant removal [7]. One of the organic dyes, Methyl orange (MO) is poisonous and can occasionally be mutagenic, carcinogenic, and genotoxic, widely used of this dye in the textile sector. It is additionally employed in titrations as a pH indicator [8–10]. Graphene oxide has a large surface area and high porosity so, adsorbs organic dyes from water easily and most effectively.

This study presents the synthesis and properties of a novel carbon nanomaterial, GO. The prepared graphene oxide was used to remove the toxic dye listed above. In addition, researchers are investigating the UV study of removing methyl orange dye with time and different concentrations of GO.

EXPERIMENTAL

Chemicals and Reagents

Graphite powder (natural, >99% purity), sodium nitrate (NaNO_3), sulfuric acid (H_2SO_4), potassium permanganate (KMnO_4), hydrogen peroxide (H_2O_2), methyl orange (MO) purchased from Sigma-Aldrich. All chemicals were of analytical grade, purchased from a commercial supplier, with a concentration of 50 mg/L used in experiments, Deionized water, and other reagents as necessary for the experiment.

Preparation of Graphene Oxide

The modified Hummers' method was used to synthesize graphene oxide [11]. The steps are as follows:

In a 250 mL round-bottom flask, 2 g of graphite powder was mixed with 100 mL of concentrated sulfuric acid and 1 g of sodium nitrate while ice-cooling. The mixture was stirred for thirty minutes. Slowly, 6 g of potassium permanganate was added while keeping the temperature below 10°C . At room temperature, the response mixture was stirred for two hours. The addition of 200 mL of distilled water stopped the reaction. The temperature increased, and the solution turned yellow brown. After 30 minutes, 10 mL H_2O_2 was added to lighten the color even more. The GO suspension was then filtered and cleaned repeatedly with ethanol and deionized water until the filtrate became neutral. The filtered GO was dried in a vacuum oven.

Methyl Orange Solution Preparation

Stock Solution

25 ppm methyl orange working solution was prepared by dissolving 25 mg of methyl orange dye in 1 L of deionized water. The solution was stirred until completely dissolved.

Methyl Orange Working Solution

100 ml solution of 25 ppm is used in different concentrations (0.05, 0.075, 0.1, 0.15) g of graphene oxide.

Characterization of Graphene Oxide

An X-ray generator 40 kV, 30 mA fitted with a $\text{Cu}_K\text{-beta}_1$ radiation source ($\lambda = 1.5406 \text{ \AA}$) was used by Japan Rigaku, Smart Lab to conduct an X-ray diffraction (XRD) analysis. With a step size of 0.02 and a scan speed of $10.00^\circ/\text{min}$, XRD data were gathered in the 2θ range, spanning from 5° to 90° . Using the Scherrer equation, lattice constants and crystallite size were determined. The morphology of the prepared graphene oxide was analyzed using a JEOL JSM-IT500 SEM with energy-dispersive X-ray spectroscopy

Adsorption Studies

A double-beam spectrophotometer was used for the adsorption study of graphene oxide with different concentrations in methyl orange aqueous solution using the UV–visible spectrophotometer set at 464 nm, the concentration of dye in the supernatant solution was observed. Every test was performed analyzed twice, and data using the average values.

RESULTS AND DISCUSSION

X-ray Diffraction (XRD)

One of the most popular techniques for examining the structural characteristics of graphene oxide samples is X-ray diffraction scattering. By using X-ray phase analysis, the structural characteristics of graphene oxide produced using the Hammer method were examined, and corresponding carbon peaks were seen in the spectrum. The spectrum shows two distinct peaks. The smaller particles can account for the dispersion of these peaks. The XRD pattern of GO showed a broad peak at around 9.9° , corresponding to the (001) plane of GO, indicating an expansion of the interlayer spacing due to the introduction of oxygen-containing groups.

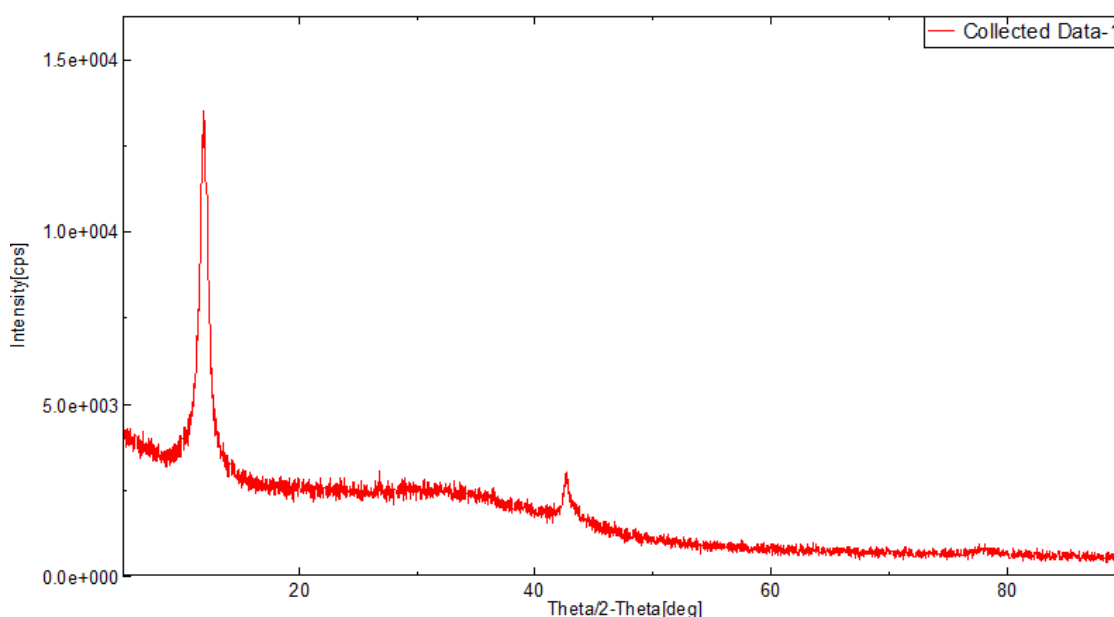


Figure 1. XRD spectrum of graphene oxide.

The prominent white pieces may also be a clump of electrons. Given that the GO X-ray diffraction peak is at 9.9° , the interlayer distance (d) will be around 0.90 nm. This peak is assigned an index (001). Afterward there have shown one another peak situated at 43.1° , as shown in Figure 1, has inter layer ratio is 0.334 nm, therefore, is distinguished by miller index (002).

SEM/EDS Analysis of Graphene Oxide

SEM images displayed a crumpled, paper-like morphology of GO sheets.

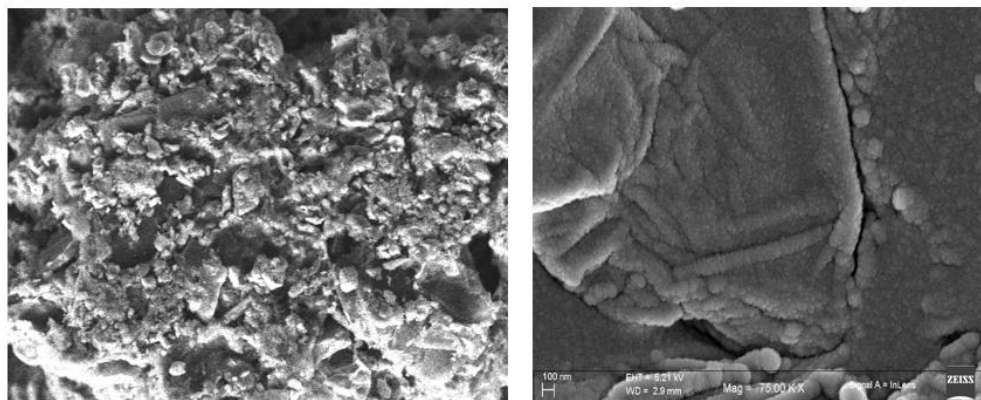


Figure 2. SEM image of graphene oxide.

SEM images of graphene oxide and EDS spectrum are shown in Figures 2 and 3, respectively. A voltage of 5.21 KV and a magnification of 7500 KX are being used to capture an image. The GO sample and the optical lens were isolated through 2.9 mm. The findings show that the typical surface morphology of graphene oxide has been well observed, revealing a porous interlinked three-dimensional graphene layer. In this SEM image, we can see some bends that indicate wrinkles and bulges.

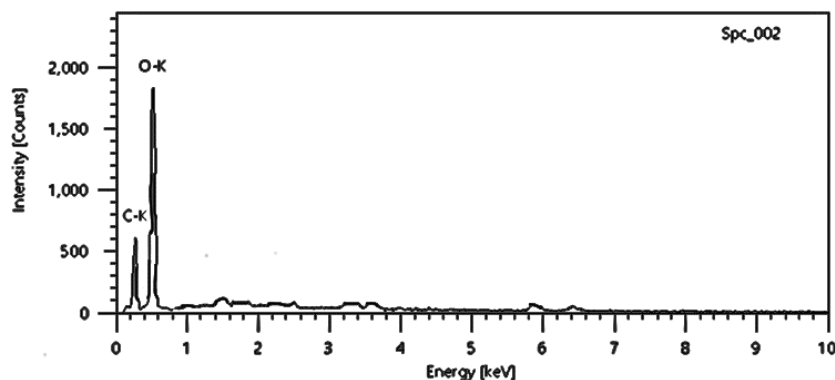


Figure 3. EDS spectrum of graphene oxide.

The elemental analysis result from the EDS analysis illustrates that carbon makes up most of the compound's content, accounting for $68 \pm 2\%$ of its mass, followed by oxygen at $31 \pm 4\%$, proving the existence of graphene oxide. As predicted by the oxidation process, this composition verifies the existence of functional groups containing oxygen in the graphene oxide. Literature shows graphene is optimally oxidized, its carbon to oxygen (C:O) atomic ratio falls between 2.1 and 2.9. This indicates the degree of graphene oxidation and evaluates the nature of oxygen functionalities [12].

ADSORPTION STUDY

Adsorption Behavior of Methyl Orange on Graphene Oxide with Time

The decolorization of methyl orange aqueous solution by graphene oxide (GO) powder was evaluated by varying the amount of GO and the duration of treatment. The initial concentration of methyl orange was 25 mg/L (25 ppm), and the experiments were conducted at room temperature. Table 1 shows absorption parameters of methyl orange using different concentrations (0.05–0.15) g GO at varying times from 2 min to 120 min.

Table 1. Absorption parameters of methyl orange using different concentrations (0.05–0.15) g GO.

S.N.	Time	Concentration of Graphene Oxide			
		0.05 g	0.075 g	0.1 g	0.15 g
1	Without GO	1.6837*	1.6837	1.6837	1.6837
2	2 min	1.3277	1.0329	0.7547	0.418
3	4 min	1.2584	0.9434	0.5228	0.3755
4	6 min	0.9857	0.7513	0.4511	0.2764
5	8 min	0.8163	0.5787	0.3644	0.1996
6	10 min	0.7007	0.4773	0.3264	0.1659
7	15 min	0.621	0.4569	0.2847	0.1449
8	30 min	0.5351	0.4517	0.1995	0.1313
9	60 min	0.3907	0.3418	0.1355	0.0728
10	90 min	0.3621	0.1882	0.09	0.0505
11	120 min	0.3588	0.0976	0.0641	0.0071

*Absorbance value of Methyl orange (A.U.) at 464 nm

Using the following formula, the removal efficiency (E%) of the graphene oxide as an adsorber on MO was determined:

$$E\% = \frac{(C^{\circ} - C)}{C^{\circ}} * 100$$

where C° denotes the starting concentration of a solution and C denotes its ending concentration of a solution.

The efficiency% of different concentrations of graphene oxide is shown in Table 2.

Table 2. Efficiency (%) of different concentrations of graphene oxide.

S.N.	Time	Efficiency % GO			
		0.05 g	0.075 g	0.1 g	0.15 g
2	2 min	21.14	38.65	55.17	75.17
3	4 min	25.25	43.96	68.94	77.69
4	6 min	41.45	55.37	73.20	83.58
5	8 min	51.51	65.62	78.35	88.14
6	10 min	58.38	71.65	80.61	90.14
7	15 min	63.11	72.86	83.09	91.39
8	30 min	68.21	73.17	88.15	92.20
9	60 min	76.79	79.69	91.95	95.67
10	90 min	78.49	88.82	94.65	97.00
11	120 min	78.68	94.20	96.19	99.57

First, without adding graphene oxide to 25 ppm of methyl orange, its absorption is found to be 1.6837 when checked with a UV spectrophotometer. Then after 2 minutes of adding 0.05 gm of graphene oxide in 100 ml solution, its absorbance seems to change. The adsorption efficiency of graphene oxide is 21.14% at 2 min. worse, with increasing time the absorption decreases or the adsorption efficiency of graphene oxide increases. At 120 min the absorption at 0.05% is 0.3488 or the adsorption efficiency of GO is 78.68%. Figure 4 illustrates UV- Vis absorption spectra for absorption of methyl orange dye in the presence of 0.05% w/v graphene oxide.

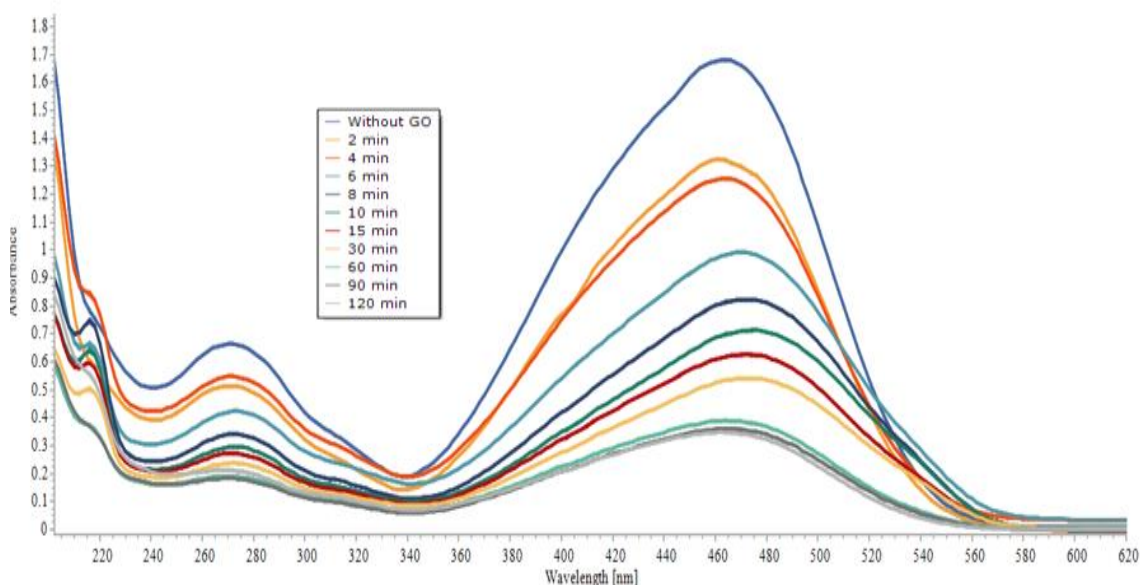


Figure 4. UV-Vis absorption spectra of methyl orange in the presence of 0.05 g graphene oxide.

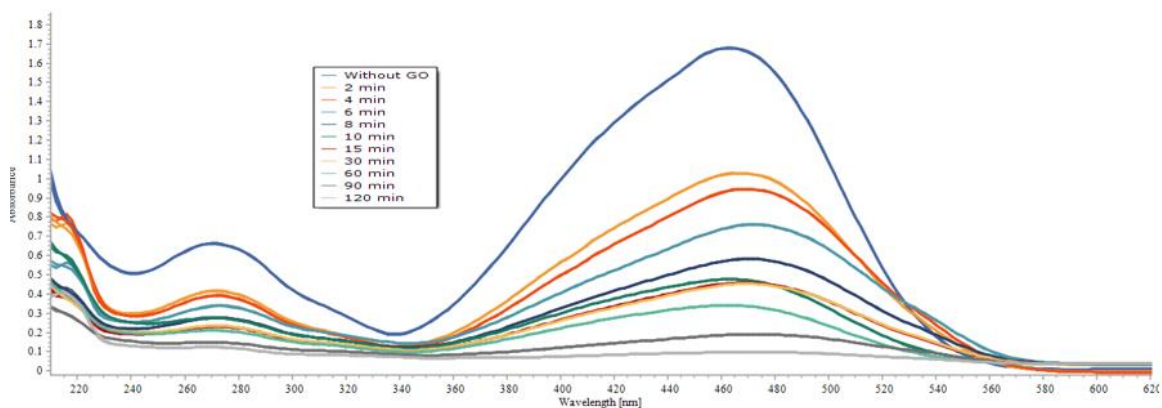


Figure 5. UV-Vis absorption spectra of methyl orange in the presence of 0.075 g graphene oxide.

With 0.075 g of GO, the efficiency rose from 38.65 % at 2 minutes, 73.17% at 30 minutes and 94.20% at 120 min (Figure 5). The rate of decolorization is faster compared to 0.05 g of GO, suggesting that more GO provides a greater number of active sites, allowing for quicker removal of the dye.

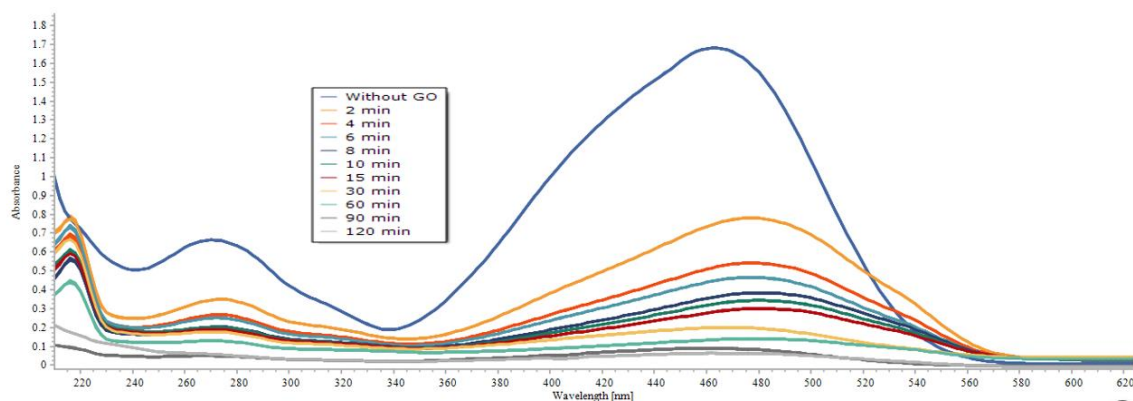


Figure 6. UV-Vis absorption spectra of methyl orange in the presence of 0.1g graphene oxide.

The efficiency for 0.1 g of GO increased from 55.17% at 2 minutes, 88.15% at 30 minutes to 96.19% at 120 minutes. The rate of decolorization is faster compared to both 0.05 g of GO and 0.075 g of GO. Figure 6 shows UV-Vis absorption spectra for absorption of methyl orange dye in the presence of 0.1 g GO.

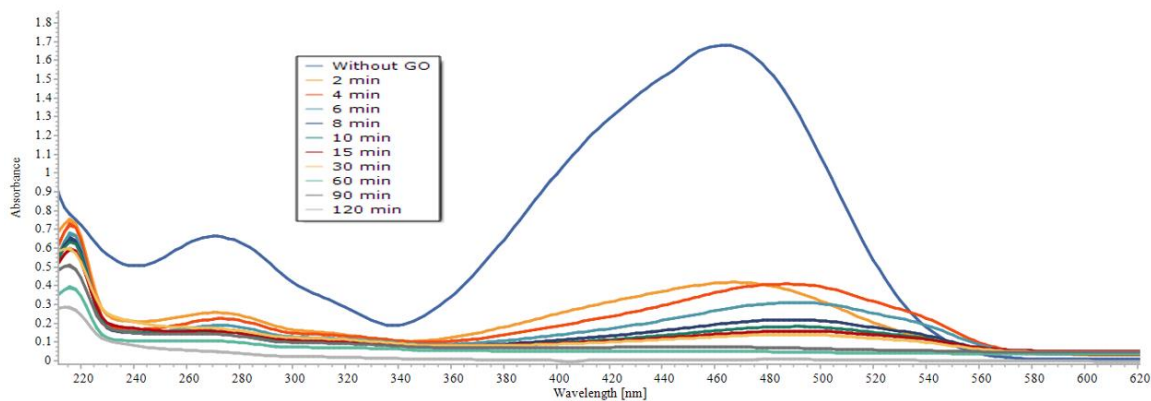


Figure 7. UV-Vis absorption spectra of methyl orange in the presence of 0.15 g graphene oxide.

Decolorization efficiency of methyl orange at various GO concentrations over time. At lower concentrations of graphene oxide (0.05 g and 0.075 g), the decolorization rate of methyl orange was relatively slow. Concentrations of 0.1 g or higher result in a significant increase in decolorization efficiency. Table 1 shows the time-dependent absorption values for all graphene oxide concentrations. The maximum decolorization rate occurred with 0.15 g of graphene oxide, reaching approximately 99.57% removal within 120 minutes, implying that adding 0.15 g of GO completely removes color from the aqueous solution after 120 minutes. Figure 7 depicts the graph of 0.15 g GO.

The ability to estimate the maximum adsorption capacity for a given initial adsorbate concentration makes the adsorption amount a crucial parameter. Table 1 summarizes review studies on MO organic dye adsorption, including extraction conditions and adsorption capacity values for nanocarbon materials and composites. The increased amount of GO powder generally leads to higher decolorization efficiency, which can be attributed to the increased number of active sites and greater surface area available for adsorption and possible catalytic reactions [13].

CONCLUSION

This study successfully synthesized graphene oxide (GO) by the hummers method and characterized their physical morphology through XRD and SEM-EDS. XRD will observe the inter-layer distance between graphene oxide layers (d) equal to 0.905 nm. SEM shows that its morphology is like a sheet of paper. Synthesized graphene oxide is used to decolorization of methyl orange dye in aqueous solution. its properties to evaluate its effectiveness in the decolorization of MO dye. Through systematic experimentation using UV-Vis spectrophotometer, we established that GO could effectively facilitate the decolorization process, with the efficiency significantly influenced by the different concentrations (0.05, 0.075, 0.1, 0.15) g of GO and the duration of decolorization. The UV-Vis spectrophotometric analysis revealed a clear correlation between the concentration of GO and the rate of dye removal, showcasing the material's potential as a versatile and efficient adsorbent for dye degradation. The absorbance of methyl orange decreased more significantly at a higher concentration (0.15 g) of GO, suggesting higher adsorption and decolorization efficiency. These findings imply that raising the GO concentration speeds up the decolorization process while also improving dye removal efficiency. These results add to the increasing corpus of research on the environmental applications of graphene-based materials and provide insights into the conditions that should be optimized for efficient dye removal.

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