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Research Paper

Enhanced adsorptive removal of congo red from water using zinc oxide-graphene nanocomposite

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Abstract
Dye-laden effluents from the textile and related sectors pose serious dangers to the environment and human health, because of their toxicity, endurance, and visual pollution. Congo red (CR), a typical anionic azo dye, was removed from aqueous solutions using a zinc oxide–graphene oxide (ZnO@GO) nanocomposite, which was created and assessed as an effective adsorbent in this study. The effects of pH, contact time, initial dye concentration, adsorbent dosage, and temperature were examined in batch adsorption tests. Because of synergistic effects such increased surface area, π–π interactions, and electrostatic attraction, the ZnO@GO nanocomposite demonstrated a considerably higher adsorption capacity. While the kinetics followed a pseudo-second-order model, the Langmuir isotherm model best fit the adsorption equilibrium data, suggesting monolayer adsorption. The spontaneous and endothermic character of the adsorption process was validated by thermodynamic measurements. The findings show that the ZnO@GO nanocomposite is a promising, affordable, and effective adsorbent for treating wastewater that contains dyes.

Keywords: *Adsorption; Congo red; Endothermic; Nanocomposite.*

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I. Introduction

Water is essential to the survival of all living things. The availability of fresh water is essential to human civilization. Human metabolism, hygiene, household use, recreation, socioeconomic development, and the processes involved in the production of food, agriculture, and industry all depend on clean drinking water. Because water covers 71% of the planet's surface yet only 0.3% of it is pure enough for human consumption, the planet is known as the blue planet. This amazing resource has been abused by humans, leading to an unsustainable and quick evolution. Every year, 300–400 t of hazardous waste, heavy metals, hazardous chemicals, and solvents are released into rivers by industrial operations [1]. The textile industry is the second largest polluter behind the oil industry, accounting for almost 20% of global water pollution. The textile industry, which employs over 8000 plant species, utilizes more water and chemicals than any other business. Wastewater from the industry contains a large number of unbound pigments and colors. Approximately 10–15% of the 8 × 105 tons of dyes generated each year are discharged into the environment. At least 70% of the 1 × 104 synthetic dyes are azo dyes [2]. Azo dyes contain aromatic rings and one or more chromophore groups (−N_3N−). With the IUPAC designation disodium 4-amino-3-[4-[4-(1-amino-4-sulfonato-naphthalen-2-yl) diazenyl] phenyl] diazenyl-naphthalene-1-sulfonate, Congo red dye is a widely used azo dye for textile coloring. Because of the aromatic amine group in its composition, Congo red (CR) dye is carcinogenic. Because azo dyes have resonance and π-conjugated bonds, they are resistant to natural deterioration and extremely stable to light and the environment [3]. Additionally, commercial dyes with azo-based functional groups may produce toxic and carcinogenic pollutants during oxidation and hydrolysis, creating problems for the environment and the economy. These waste fluids contain toxic pigments that have a negative impact on the environment and sustainable growth, necessitating the use of innovative treatment techniques [4]. A variety of treatment techniques have been used to lessen the negative effects of azo dyes on human health and the environment. Adsorption, membrane filtration, and ion exchange are examples of physical techniques. Chemical methods such electrochemical oxidation, oxidation, and ozonation have also been used [5]. Additionally, microorganisms like algae, yeasts, fungi, and bacteria have been used to

generate biological processes like biodegradation, bioaccumulation, and biosorption [6]. The efficacy of conventional wastewater treatment technologies, which are costly and energy-intensive, is limited by issues with the chemical stability and degradation of dyes. Heterogeneous photocatalysis is one of the most promising and successful processes now in use for removing pollutants from wastewater [7].

Heterogeneous photocatalysts can be prepared in a variety of ways. Precise size, dimension, composition, and structure are the main benefits of chemically mediated nanoparticle creation. Chemical nanotechnology has improved many facets of life, but it has also caused nanopollution—pollution of the air and water. A plant-mediated synthetic approach is favored over traditional chemical and physical methods for the synthesis since it is easy to use, quick, inexpensive, environmentally friendly, and doesn't require high temperatures, pressures, or energies. Additionally, it does away with the use of dangerous chemicals [8].

Because of their nanoscale size, high surface area-to-volume ratio, strong reactivity, exceptional chemical and thermal durability, broad availability, and promise for nanoscale catalysis, scientists are intensively investigating carbon-based nanomaterials.16 There are various kinds of carbon-based nanomaterials, including carbon nanotubes (CNTs), graphene oxide (GO), reduced graphene oxide (rGO), fullerenes, carbon dots (CDs), nanodiamonds, carbon nanohorns (CNHs), and carbon nanofibers (CNFs). GO is one of the most promising carbon-based compounds because of its easier synthesis and useful multifunctional qualities. In the nanocomposite, it serves as an adsorbent, conductive carrier, photosensitizer, photostabilizer, photocatalyst, and cocatalyst. Similarly, as compared to pure metal oxide, the GO-metal oxide nanocomposite materials show greater adsorption capabilities. By adorning the ZnO semiconductor, it is envisaged that the GO's adsorption efficiency can be further increased. Therefore, in this investigation, the environmentally friendly produced nanoadsorbent ZnO@GO was used in a batch manner with different experimental parameters to remove CR dye. Furthermore, the dye removal data were evaluated using nonlinear isotherms, kinetics models, and thermodynamics study that was also carried out.

II. Methodology

Synthesis of graphene oxide (GO)

Graphene oxide (GO) was created using a modified Hummer's process. In short, 2.5 g of $NaNO_3$ and 120 mL of H_2SO_4 were introduced to a 1000 mL conical flask. Then, 4.5 g of graphite powder was added gradually to the liquid. 15 g of $KMnO_4$ was added after the mixture had been stirred for 20 minutes at 20 °C and cooled to 0 °C using an ice bath. Stirring the mixture continuously for 60 minutes was followed by the addition of 250 mL of DI water. A few minutes later, the combination solution was gradually supplemented with 20 milliliters of 30% H_2O_2. After then, the temperature was increased to room temperature, and for the next two hours, stirring was continued. The precipitate was then collected by centrifuging the resultant solution for 10 minutes at 6000 rpm. It was then washed twice with DI water and 10% HCl. After being cleaned and baked for 48 hours at 72 °C, the precipitate sample was recovered as graphene oxide (GO).

Preparation of zinc oxide (ZnO) nanoparticles

First, 10 g of zinc acetate dehydrate was dissolved in 250 mL of DI water at 25 °C. A 3 M NaOH solution was then added gradually while being magnetically agitated. The addition of NaOH was halted when the pH of the reaction hit 12.0, but stirring was maintained for the following half hour to produce a homogeneous mixture. After an hour, the mixture was allowed to form a precipitate, which was then periodically rinsed with ethanol and DI water to get rid of any leftover compounds. To get rid of any last bits of residue, it was finally left to air dry at 45 °C for ten hours. The baked sample was calcined in a muffle furnace for four hours at 400 °C in order to produce ZnO nanoparticles.

Synthesis of ZnO@GO nanocomposites

Synthetic ZnO nanoparticles were immobilized on nano GO via a relatively simple ultrasonication process. In a typical preparation process, 0.24 g of ZnO was progressively added after 0.08 g of GO had been ultrasonically sonicated in DI water for 30 minutes. To create a uniform dispersion solution, the mixture was stirred for three hours at 60 °C. Centrifugation was used to gather the precipitate after the temperature cooled to room temperature. After that, it was frequently cleaned with DI water and dried for ten hours at 45 °C in an air oven.

III. Result and Discussion

Batch study

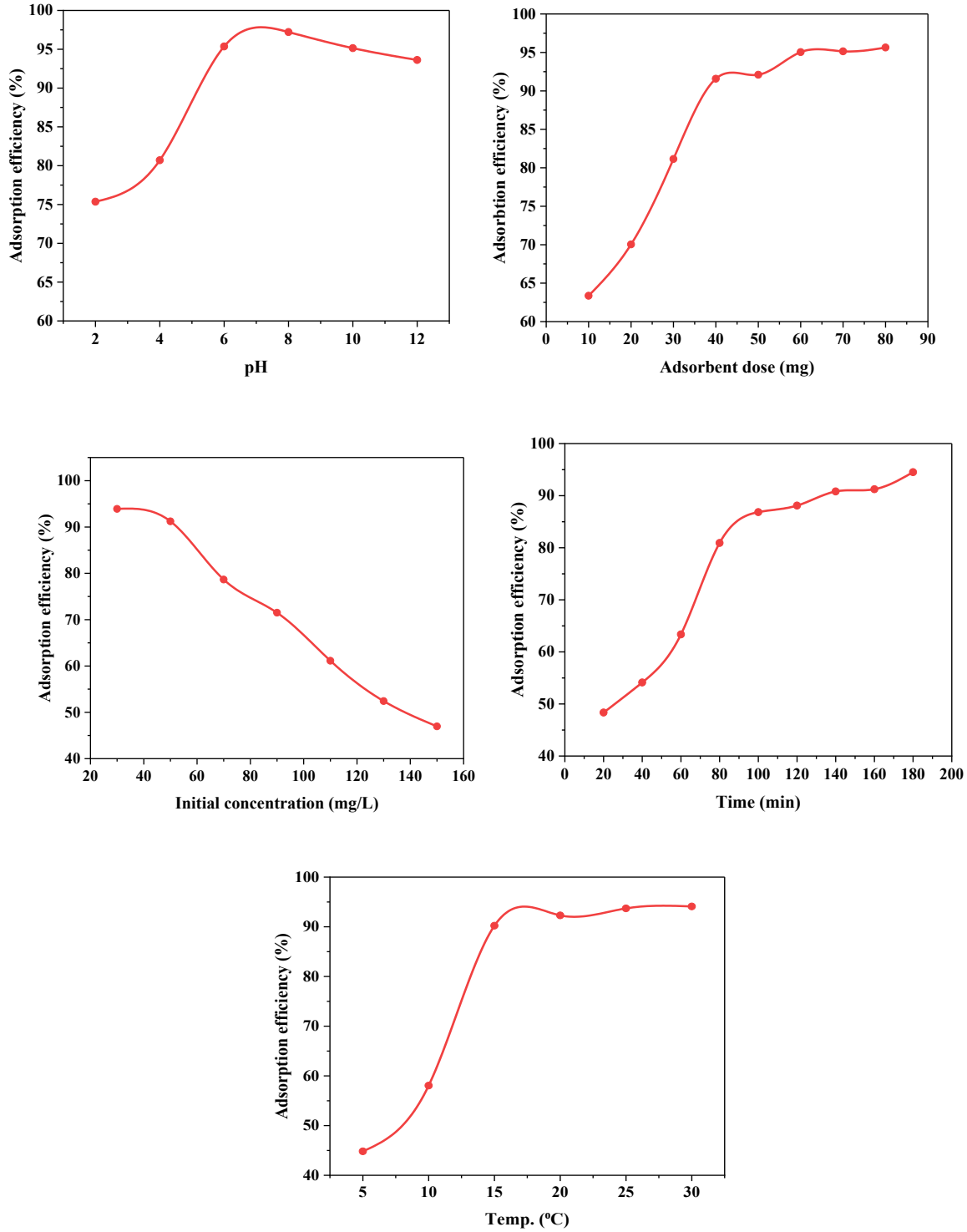

Fig: 1. Effect of pH, adsorbent dose, initial ion concentration, time and temperature.

The congo red dye was adsorbed using the produced ZnO@GO nanocomposite. Batch analysis was utilized for dye optimization of parameters. pH, dosage, concentration, time, and temperature were among the many characteristics that were employed. By changing one variable while holding the others constant, the effects of all these parameters were examined. Because it controls the adsorbent charge and the extent of adsorbate ionization in solution, pH is one of the factors that significantly affects adsorption capacity. The impact of solution

pH on CR dye adsorption was investigated in the range of 2.0–12.0, while all other parameters were kept constant. Fig. 1 shows how pH affects the dye's rate of adsorption onto the nanocomposite surface. The removal capacity for CR dye increased from 2.0 to 8.0 due to the protonation of the hydroxyl, SiO, and amino groups on the nanoparticle's surface, but it then decreased when the pH of the solution increased further. The greatest adsorption capacity of CR dye was attained at pH 8.0. As a result, CR-dye's optimal pH for additional adsorption tests was found to be 8.0. At low pH levels, there is a strong electrostatic repulsion between the dye molecules and the hydrogel film surface. The adsorption capacity is constrained by the competition between dye and H+ ions for the available adsorption sites. At the proper pH, dyes' active sites enable them to attach to the hydrogel film and achieve a better adsorption efficiency. The adsorption effectiveness falls as the original dye concentration rises.
One of the elements that greatly influences sorption capacity is the dosage of the adsorbent; it offers a binding site and a substantial surface area for the dye to be adsorbed on the adsorbent. The effects of dose were examined in the range of 10.0 mg to 80.0 mg in 50.0 mL of adsorbate solutions, while the initial dye concentration (20 mgL^{-1}), contact time (90 minutes), and pH 8.0 were maintained constant. The adsorption capability for CR adsorption rises with increasing adsorbent dosage, as seen in Fig 1. Because there were more exchangeable sites available at greater adsorbent doses or surface areas, some adsorption sites were left empty, which led to a drop in dye by an adsorbent-mass unit.

An important component of an adsorption experiment that directly affects the kinetics of adsorption is contact time. At a constant pH of 8.0 for CR dye, dose of 20.0 mg, and temperature of 25°C, the effects of contact duration (20–180 min) on CR adsorption onto the adsorbent surface were examined. The adsorption process began extremely rapidly because the nanocomposite surface included a large number of active sites. Dye molecules had to contend for the available adsorption sites when equilibrium was achieved. The CR adsorption equilibrium was achieved after 100 minutes. Once equilibrium has been reached, the adsorption capacity remains constant over time. Consequently, the optimal time for CR dye adsorption was determined to be 100 minutes.

Figure 1 illustrates how temperature affects congo-red elimination between 5°C and 30°C. The increased temperature improves the mobility of congo-red molecules, increasing the clearance percentage. Furthermore, a stronger chemical connection between the nanocomposite and congo-red is encouraged by a higher temperature, which improves removal efficiency. At higher temperatures, molecules have more kinetic energy and move more swiftly. By enhancing the collision between congo red and the nanocomposite, this rapid transit promotes better adsorption. The removal efficiency is at its highest around 20˚C. Therefore, 20˚C is the optimal temperature for congo red elimination. Equilibrium is obtained when the removal efficiency remains constant after reaching 20˚C. At higher temperatures, however, a constant clearance rate is attained and more active sites become available.

Isotherm, kinetics and thermodynamics study

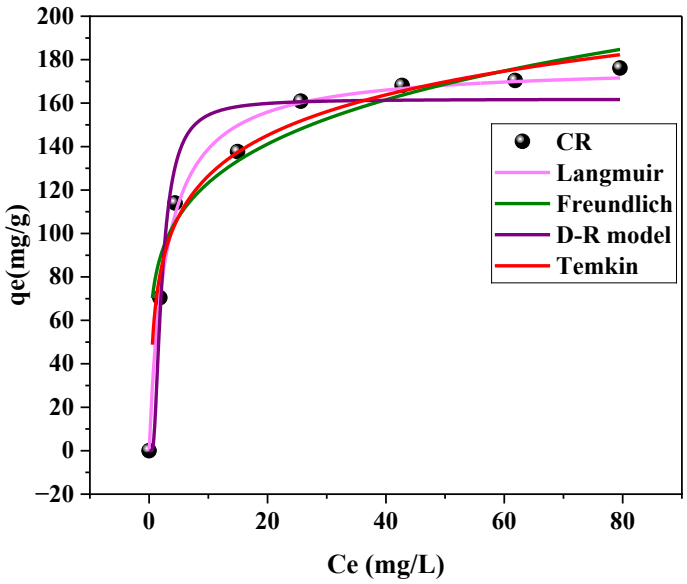

Fig: 2. Isotherm study for congo red dye using ZnO@GO.

This work examined the adsorption qualities using the Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich isotherms. The evaluations were conducted using non-linearized versions of the isotherms in OriginPro 2025B. Figure 2 and Table 1 display the plots of the isotherm models and isotherm parameters,

respectively. The corelation coefficient (R^2) values were used to choose the best model for the adsorption of CR dye onto ZnO@GO. The Langmuir isotherm from Table 1 with the highest R^2 value (0.99) among the isotherms analyzed is thought to be the best fit for the adsorption of CR dyes onto ZnO@GO. Adsorption for CR dye takes place on a homogenous surface subsequent to monolayer coverage. Based on the R^2 values for CR, the isotherm is Langmuir > Temkin > Freundlich > Dubinin-Radushkevich in order of fittness. The qmax (mg/g) for CR (176.33) indicates that the adsorbent has a much higher maximum monolayer adsorption capacity for CR, exhibiting greater affinity and surface coverage. The adsorbent surface aggregating due to the attraction between particles with different charges may be the cause of the heterogeneity. Favorable adsorption is indicated when n is bigger than 1. The ZnO@GO nanoparticles' high Kf values (77.12 for CR) demonstrated their remarkable attraction for dye molecules. It suggests that the dye molecules have a high probability of adhering to the ZnO@GO NPs' surface. The binding equilibrium constant is significantly greater for CR, suggesting stronger binding site availability, as indicated by the Temkin Isotherm CR (25.98) and Kt value of 11.19 for CR. The high correlation coefficient (CR = 0.97) indicates that Temkin also fits dye adsorption.

Table 1: Isotherm parameters for CR adsorption onto ZnO@GO NPs

Isotherm model	Parameter	CR
Langmuir	q_{max} (mg/g)	176.33
	R^2	0.99
	R_L	0.34
Freundlich	N	0.19
	K_f (mg/g)	77.12
	R^2	0.92
Temkin	B	25.98
	K_t (L/g)	11.19
	R^2	0.97
Dubinin–Radushkevich	Q_{DR}	161.79
	K_{DR}	5.08
	R^2	0.87

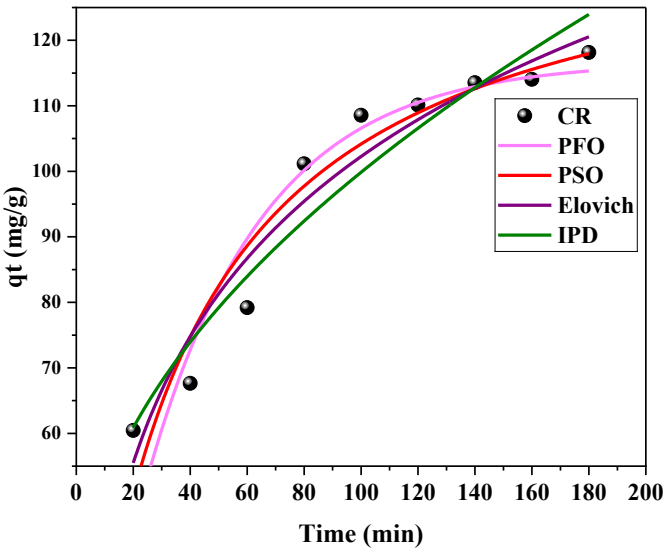

Fig: 3. Kinetic study for congo red dye using ZnO@GO adsorbent.

The findings show that the pseudo-second-order kinetic model outperforms the pseudo-first-order model in terms of fitting the experimental data as shown in Fig. 3 and table 2. The preponderance of the pseudo-second-order mechanism in the adsorption process was confirmed by the non-linear kinetic modeling model with the greatest R^2 value among the evaluated models. The kinetic models fit the experimental data in the following order: pseudo-second-order > Elovich > IPD > pseudo-first-order kinetic. The low value of k (0.02 for CR) suggested a slow rate of adsorption. The reported results could be due to intraparticle diffusion, the rate-determining phase in a multi-step adsorption process.

Table- 2: Kinetics model for CR dye adsorption study

Kinetics model	Parameters	CR
Pseudo second order	k_2 (g/mg min^{-1})	1.89
	q_e (cal) (mg g^{-1})	142.14
	R^2	0.97
Intra-particle diffusion model	K_{id} (mg g^{-1} $min^{-1/2}$)	1.29
	C (mg g^{-1})	7.28
	R^2	0.90
Elovich	A	142
	β	0.32
	R^2	0.93

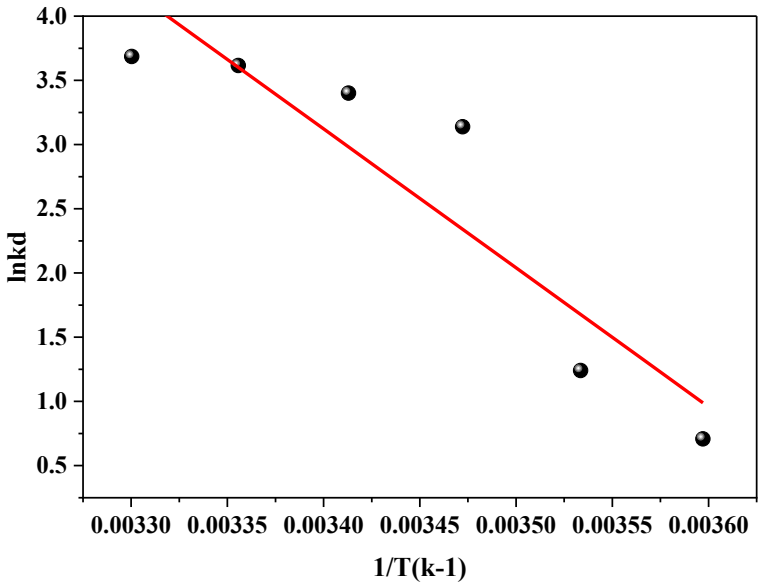

Fig: 4. Thermodynamic study for adsorption of CR onto ZnO@GO.

Table-3. The parameter of thermodynamic study for adsorption of CR onto ZnO@GO.

ΔG^O (kJ mol^{-1})	
T (K)	**CR**
278	-2.26
283	-3.90
288	-5.52
293	-7.19
298	-8.32
303	-10.12
ΔH^O (kJ mol^{-1})	87.94
ΔS^O (J/(mol^{-1})	329.25

Important insights into the mechanism of adsorption, ideal conditions, spontaneity, and practicality can be gained from thermodynamic studies. Understanding thermodynamic parameters facilitates the successful design, control, and optimization of adsorption-based systems. Figure 4 illustrates the relationship between 1/T(K−1) and ln Kd for CR dye adsorption on ZnO@GO NPs. The values of the thermodynamic parameters obtained by extrapolating the slope and intercept of the plot are shown in Table 3. Given that a negative value of ΔG° was observed at all temperatures, the table indicates that CR dye adsorption onto ZnO@GO NPs was spontaneous, feasible, and advantageous. The positive values of 87.94 kJ/mol observed for ΔH^0 show that the adsorption process is endothermic. The fact that the percentage elimination rose with temperature served as

confirmation of this. For ΔS^o, a positive value of 329.25 J/mol/K was found, indicating that as disorder and entropy grow, so does the process. Additionally, the process is spontaneous and thermodynamically more favorable as temperature rises, in accordance with an endothermic, entropy-driven mechanism, as ΔG is negative and gets more negative at higher T.

IV. Conclusion

The effective synthesis of a ZnO@GO nanocomposite revealed outstanding adsorption capabilities toward Congo red dye. High adsorption capacity, quick kinetics, and good stability were the outcomes of the synergistic interaction between ZnO and graphene oxide. The work emphasizes how ZnO@GO nanocomposites can be used as effective adsorbents for the treatment of wastewater contaminated with dyes.

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