-RESEARCH PAPER-

International Journal of Mining and Geo-Engineering

IJMGE

IJMGE 59-3 (2025) 267-272

DOI: 10.22059/IJMGE.2025.384404.595203

Investigation of an optimized mineral adsorbent modified with polydopamine for the removal of methylene blue contaminants from industrial wastewater

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Article History:

Received: 12 February 2025.

Revised: 22 April 2025.

Accepted: 22 July 2025.

ABSTRACT

Wastewater generated by the textile industry leads to significant color pollution and the release of hazardous substances. The adsorption process provides an effective method for mitigating organic pollutants, such as dye-laden wastewater. This study aimed to develop a Zeolite@Polydopamine composite that serves as a highly efficient material for the elimination of methylene blue dye from aqueous solutions. To attain maximum adsorption capacity, different effective parameters, such as temperature, contact time, dye concentration, adsorbent weight, and solution pH, were optimized. The optimal conditions for the removal process were established with a dye concentration of 10 mg/mL and an adsorbent weight of 30 mg. Under these conditions, the ideal contact time was achieved to be 30 min, with a pH level of 7 and a temperature maintained at 25°C. Evaluations of isotherm models and adsorption kinetics revealed that the adsorption process is best defined by the pseudo-second-order kinetic model in conjunction with the Freundlich isotherm. Additionally, the maximum adsorption capacity, as calculated by the Langmuir model, was found to be 70.92 mg/g. The negative values observed for both enthalpy and entropy suggest that this process of adsorption becomes more desirable at lower temperatures.

Keywords: *Adsorption isotherm, Adsorption, Polydopamine, Zeolite, Methylene blue, Synthetic adsorption.*

1. Introduction

The growth and industrialization of human societies result in the generation of substantial amounts of wastewater, which is often discharged into freshwater sources. This practice presents a considerable threat to human health because of the harmful effects of these pollutants. As a result, water quality serves as a critical indicator of the overall quality of life in various communities [1]. One of the major threats to freshwater resources is the release of organic dyes and other harmful substances found in industrial effluents, which adversely impact water quality and pose serious health risks to humans, leading to significant environmental challenges [2, 3]. Even at low concentrations, these dyes can harm aquatic ecosystems and human health by remaining in moving water for long durations. The primary sources of these dyes include factories involved in the production of food, cosmetics, pharmaceuticals, paper, rubber, plastics, leather, and textiles [4, 5]. Methylene blue, a widely used cationic dye, is known for its high stability, solubility, and biotoxicity in water. Its presence in wastewater creates a dark blue coloration that disrupts photosynthesis, blocks sunlight penetration, and hampers re-oxygenation in aquatic systems, ultimately posing negative effects on human health [8]. Direct exposure to methylene blue can inflict permanent damage to the skin and eyes in both animals and humans, and inhalation may lead to respiratory difficulties, as well as adverse effects on the central nervous system, along with potential harm to reproductive health, brain function, and organs, such as the liver and kidneys [8]. For eliminating hazardous pollutants from wastewater, several treatment techniques are available, including membrane filtration, oxidation, and electrochemical treatment. Various methods can be employed for this purpose, including ion exchange and adsorption. Of these methods, adsorption has gained popularity for removing colored pollutants from industrial wastewater on account of its low initial cost, minimal energy requirements, ease of implementation, and the ability to regenerate and reuse the spent adsorbent. A variety of adsorbents with high surface activity have been explored for this purpose, including activated carbon, zeolites, polymers, and zero-valent metal oxides. Notably, natural zeolites are abundant in nature and are extensively utilized in water purification processes because of their unique properties, such as high porosity, cation exchange capacity, and molecular sieve characteristics. Zeolites are aluminosilicates characterized by a regular crystal structure, selective adsorption capabilities, specific surface area, thermal stability, and a high net negative charge, which enables them to effectively adsorb heavy cations. They can also serve as excellent supports for nano-metal oxides in adsorption applications. Clinoptilolite is one of the most prevalent and accessible natural zeolites, widely used across various industries, including lightweight concrete production, refining, and chemical fertilizers [9]. A variety of natural zeolites demonstrate distinct ion exchange capacities for cations, including ammonium and heavy metal ions. Moreover, certain zeolites are proficient at adsorbing anions and organic compounds from aqueous solutions. To improve their adsorption capabilities for organic substances and anions, natural zeolites can undergo several modification techniques, including acid purification, ion exchange, and surfactant functionalization. These enhancements significantly increase their effectiveness in

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environmental applications [10,11]. Recently, polymeric adsorbents, such as an anionic polysaccharide, sodium alginate, and polydopamine have garnered significant attention for their advantageous adsorption and regeneration properties, along with their mechanical strength and cost-effectiveness. Polydopamine is synthesized through the self-polymerization of dopamine in an aerobic environment under alkaline conditions. This process yields a material that retains various functional groups, including catechol, amine, and imine. These characteristics enhance the utility of polydopamine in environmental remediation applications [12]. This unique composition allows Polydopamine to be easily applied to almost any solid surface, altering its surface characteristics and microstructure. Research has demonstrated that both pure Polydopamine and materials modified with Polydopamine are highly effective adsorbents for various pollutants in wastewater [13-15]. Furthermore, modifying materials with Polydopamine can significantly enhance their selectivity for the adsorption of cationic species. Motivated by the properties of these materials, this study presents the development of an efficient composite by modifying zeolite with Polydopamine to adsorb the color pollutant methylene blue from aqueous solutions. The composite's structure was analyzed through FESEM and FTIR. To enhance adsorption efficiency, essential factors including adsorbent weight, pH, contact time, temperature, and concentration were optimized. Furthermore, the research explored adsorption isotherms, kinetics, and thermodynamics to gain deeper insights into the adsorption mechanism.

2. Experimental

2.1. Materials

Zeolite, methylene blue dye, and Tris were acquired from Merck Company, while dopamine was received from Caspian Pharmaceutical Company. Deionized water was utilized throughout all experimental procedures.

2.2. Characterization

The measurement of absorbance of solutions containing dyes was done by a Perkin Elmer spectrophotometer LAMBDA 25 made in America. Maximum absorbance of methylene blue (λmax) was studied at 665. To analyze the stretching and bending vibrations of compounds, FTIR (Tensor-27, BRUKER) was used. Examination of the morphology of the prepared composite was done using FE-SEM.

2.3. Synthesis of Polydopamine@Zeolite composite

First, disperse 800 mg of zeolite in 100 mL of Tris buffer solution (0.12 mg/mL) with the help of ultrasonication for 20 minutes (the pH was adjusted to 8.5 with the help of 1 M hydrochloric acid). Then, 300 mg of dopamine was added to the above stirring solution. After 24 hours, the suspension was filtered and wash three times with distilled water. The obtained product was dried in the oven for five hours at a temperature of 50 degrees Celsius.

3. Results and Discussion

3.1. Effect of concentration

The impact of methylene blue concentration, varying from 10 to 50 mg/L, was assessed while keeping contact time and pH constant. Figure 1 illustrates that an increase in the initial concentration of methylene blue corresponds to a decrease in the percentage of dye removal. This decline is attributed to the equilibrium being reached on the adsorbent surface at elevated concentration levels of methylene blue.

Generally, at lower concentrations, the ratio of adsorbed molecules to available active sites is low, making the adsorption process relatively independent of the initial concentration. However, at elevated concentrations, access to the adsorption sites is limited, resulting in a greater dependency of color removal on the initial concentration of methylene blue.

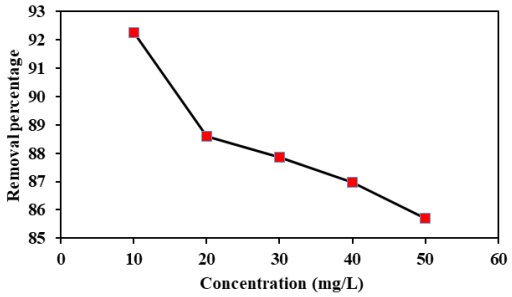

Figure 1. The impact of concentration on the removal efficiency of methylene blue.

3.2. Investigating the impact of adsorbent dosage

The dosage of adsorbent is a critical factor in the process of adsorption, as it significantly influences the adsorbent's capacity to uptake dye from the solution. In this study, the weight of the adsorbent was varied between 5 and 40 mg to assess its impact on the removal percentage of methylene blue dye. As depicted in Figure 2, increasing the weight of the adsorbent leads to a higher average efficiency of dye removal. This improvement is ascribed to the greater availability of adsorption sites on the adsorbent's surface relative to the constant number of dye molecules present. However, this efficiency does not increase indefinitely. As the adsorbent weight continues to rise, factors such as concentration gradients between the adsorbent and dye molecules, the length of the diffusion path, mass transfer resistance, and the blocking of some permeation pathways can limit the effectiveness of surface adsorption. Additionally, some internal surfaces and cavities within the adsorbent may remain unused. Based on the findings, an adsorbent weight of 30 mg was determined to be optimal for maximizing dye removal under the given conditions.

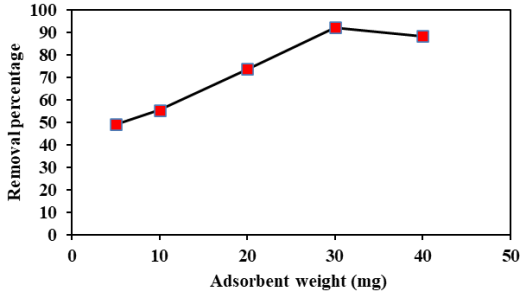

Figure 2. The impact of adsorbent weight on methylene blue adsorption efficiency.

3.3. Investigating the impact of pH

The pH of the solution significantly impacts the elimination of dye pollutants by altering the surface charge of the adsorbent. This study evaluated the impact of pH on the adsorption efficiency of methylene blue within a range of 2 to 10. The outcomes, illustrated in Fig. 3, demonstrate that the highest percentage of removal occurs at pH 7. Consequently, subsequent experiments were conducted with the pH maintained at this optimal level to ensure effective adsorption.

3.4. Investigating the Impact of Contact Time on Adsorbent Efficacy in Methylene Blue Removal

Fig. 4 illustrates the influence of contact time between the adsorbent and the sample solution on the removal efficiency of methylene blue dye at optimal pH levels. The data clearly indicate a positive correlation between contact time and percentage color removal, suggesting that extended contact allows more dye molecules to be adsorbed by the adsorbent. However, beyond 50 minutes, there is a negligible change in adsorption capacity, indicating that equilibrium has been reached. This

observation supports a two-stage adsorption process: an initial rapid adsorption phase occurring at the surface of the adsorbent, followed by a slower phase characterized by internal mass transfer. Based on these findings and to optimize testing durations, a contact time of 30 minutes was determined as optimal for effective methylene blue removal.

3.5. Investigating the effect of temperature

To evaluate the removal of methylene blue dye, experiments were performed at various temperatures 25, 30, 40, and 50 °C under optimal conditions. The outcomes depicted in Fig. 5 indicate that as the temperature of the solution increases, the percentage of methylene blue removal decreases. This trend suggests that the adsorption reaction is exothermic, meaning that higher temperatures inhibit the efficiency of dye removal.

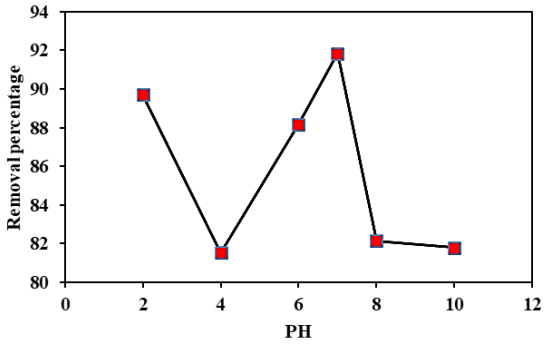

Figure 3. The effect of pH on the adsorption efficiency of methylene blue.

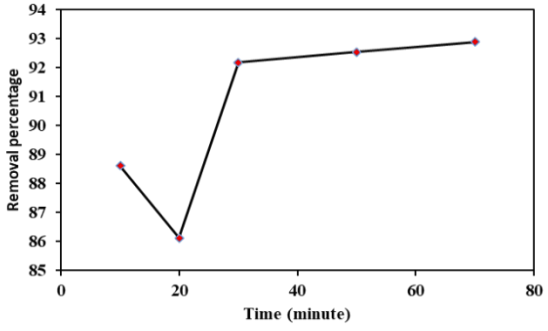

Figure 4. The impact of adsorbent and solution contact time on methylene blue removal efficiency.

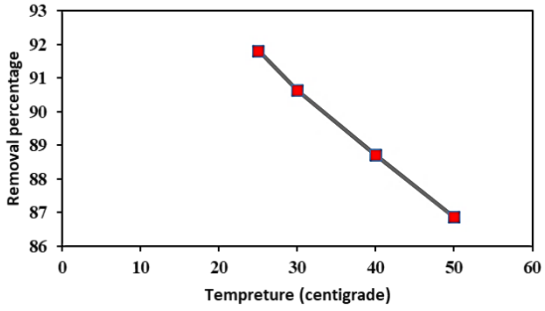

Figure 5. The impact of temperature, contact time of adsorbent and solution on the removal efficiency of methylene blue.

3.6. Investigating adsorption isotherms

Isotherms of adsorption play a critical role in evaluating and characterizing the capacity of an adsorbent, which is vital for assessing the feasibility and designing effective adsorption systems. In this study, dye solutions with various concentrations of 10, 20, 30, 40, and 50 mg/L were prepared. Each concentration underwent extraction under optimal conditions specifically, pH = 7, an adsorbent weight of 30 mg, a solution volume of 25 mL, and a contact time of 50 minutes. The equilibrium adsorption behavior of methylene blue onto the adsorbent was analyzed using the Langmuir, Freundlich, and Temkin isotherms at a controlled temperature of 25±1°C (as presented in Table 1). The findings demonstrated that the Freundlich isotherm offered the most accurate representation of the data collected in this research

3.7. Langmuir isotherm

It is used to characterize the adsorption of solutes from aqueous solutions. This model is based on the concept of monolayer adsorption taking place on a uniform adsorbent, where all adsorption sites are identical and possess equal energy. According to this model, once a site is filled, no additional adsorption can occur at that particular site. The equation representing the adsorption of a saturated monolayer can be expressed as follows in Equation 1:

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

Where:

- (qe) refers to the quantity of adsorbate per unit mass of adsorbent at equilibrium, expressed in milligrams per gram (mg/g).
- (Ce) signifies the equilibrium concentration of the adsorbate, measured in milligrams per liter (mg/L).
- (q[ma}) indicates the maximum adsorption capacity, also expressed in milligrams per gram (mg/g).
- (K_L) represents the Langmuir constant, with units of liters per milligram (L/mg).

Figure 6-1 shows the Langmuir isotherm for methylene blue dye. Langmuir constants were calculated using the slope and intercept from the origin of the lines and according to the form of the Langmuir equation. According to the figure, the adsorption of methylene blue by the synthesis adsorbent does not match the Langmuir isotherm.

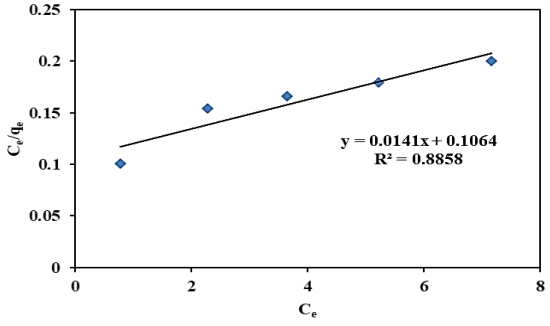

Figure 6-1. Langmuir isotherm.

3.8. Freundlich isotherm

It serves as an empirical model for understanding adsorption on heterogeneous surfaces, where the energy of adsorption varies from one site to another on the adsorbent. This model is particularly relevant in scenarios where adsorption does not take place uniformly across the surface. The equation representing Freundlich adsorption [17] can be articulated as follows:

$$q_e = K_f C^{1/n} \quad (2)$$

- The amount of solute adsorbed per unit weight of adsorbent at equilibrium, denoted as qe, is measured in mg/g.
- The equilibrium concentration of the solute in the solution is represented by Ce and is expressed in mg/L.
- K_F refers to the Freundlich constant ((mg/g) $(L/mg)^{1/n}$), which implies the capacity of adsorption.

- A dimensionless parameter termed n relates to the intensity of adsorption; when n exceeds 1, it signifies that adsorption is favorable.

The values of adsorption capacity and adsorption intensity were obtained from the logarithmic plot of q_e versus C_e. The results of examining the Freundlich isotherm model are given in Fig 6-2. According to the diagram, the Freundlich constants were obtained from the intercept and the slope of the line. This diagram shows that the adsorption of methylene blue dye by the synthesized adsorbent corresponds to the Freundlich isotherm.

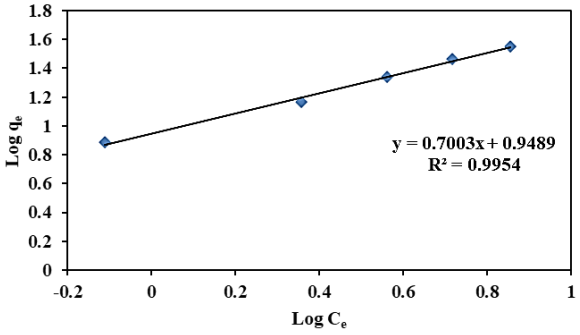

Figure 6-2. Freundlich isotherm.

3.9. Temkin adsorption isotherm

The Temkin isotherm is another model used to express adsorption processes, particularly in systems where the heat of adsorption decreases with increasing coverage [18]. In this model, the amount of adsorbed material is proportional to the logarithm of the equilibrium concentration, reflecting the interactions between adsorbed molecules. The linear form of the Temkin isotherm can be expressed as:

$$q_e = K_1 Ln(K_2) + K_1 Ln(C_e) \quad (3)$$

- The equilibrium amount of solute adsorbed per unit weight of adsorbent, denoted as qe, is expressed in mg/g.
- The concentration of the solute at equilibrium in the solution is represented by Ce and measured in mg/L.
- K1 refers to the Temkin constant that corresponds to the maximum adsorption capacity, with units of mg/g.
- K2 signifies the Temkin constant associated with the heat of adsorption (KJ/mol) and also indicates the affinity between the adsorbent and the solute (L/g).

The examination results of the Temkin isotherm model are given in Figs. 3-6. Using the slope and intercept from the origin of the graph, the constants of the Temkin isotherm were calculated. The correlation coefficient resulting from this plot displayed that the methylene blue adsorption by the corresponding adsorbent does not follow the Temkin isotherm.

A valuable approach for identifying the optimal fit of experimental data to isothermal adsorption models involves analyzing regression coefficients, which gauge the degree of alignment between the data and each model. In this investigation, the adsorption behavior of methylene blue onto a fabricated adsorbent was evaluated using three isotherm models. The outcomes of these analyses are presented in Table 1, which details the R^2 values along with pertinent constants for each isotherm. The calculated R^2 value for the Freundlich isotherm was 0.9954, indicating a strong fit and suggesting that the adsorption process exhibits heterogeneous characteristics. Conversely, the Langmuir model yielded an R^2 value of 0.8858, while the Temkin model produced an R^2 value of 0.9286. These findings imply that although there is some correlation with both Langmuir and Temkin models, it is evident that the Freundlich model more accurately captures the overall behavior of adsorption. From these correlation coefficients, we can infer that methylene blue adsorption primarily occurs at specific sites on the adsorbent surface, indicating a monolayer formation in a uniform manner. Furthermore, according to calculations from the Langmuir model, the maximum adsorption capacity (q_m) for methylene blue was obtained to be 92.70 mg/g of adsorbent. This result underscores the potential effectiveness of this synthesized adsorbent in dye removal applications.

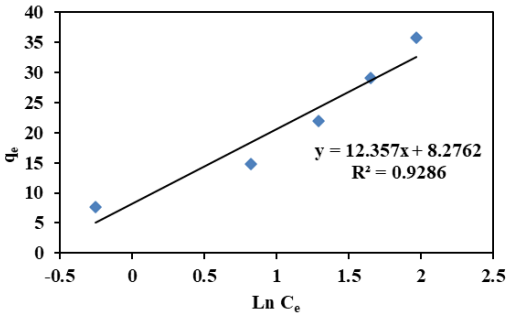

Figure 6-3. Temkin isotherm.

3.10. Investigation of adsorption kinetics

Kinetic studies are essential in understanding the adsorption process, particularly how contact time between the adsorbent and the adsorbate affects the number of species adsorbed. These studies provide insights into the speed of the adsorption process and the mechanisms involved in surface adsorption, including both adsorption and desorption rates. This information is critical for designing and evaluating adsorption systems. In this investigation, laboratory data were examined employing three kinetic models: pseudo-first-order [19], pseudo-second-order [20], and intraparticle diffusion [21]. The experiments were conducted under optimal conditions with a methylene blue solution at a concentration of 10 mg/L, pH 7, an adsorbent dosage of 30 mg, and a solution volume of 25 mL. The contact times varied from 10 to 70 minutes. For the pseudo-first-order kinetic model, the rate of change in the removal of dissolved species over time is directly proportional to the difference between the saturation concentration and the amount removed at that time. The linear form of the pseudo-first-order kinetics can be defined as follows in Equation 4:

$$\log(q_e - q_t) = \log(q_e) - \frac{K_1}{2/333}\, t \quad (4)$$

The equation 5 demonstrates the linear form of pseudo-second-order kinetics:

$$\frac{t}{q_t} = \left(\frac{1}{K_2 q_e^2}\right) + \left(\frac{1}{q_e}\right) t \quad (5)$$

- (q_e) denotes the amount of solute adsorbed at equilibrium, expressed in milligrams per gram (mg/g).
- (Qt) indicates the amount of solute adsorbed at a specific time, also measured in milligrams per gram (mg/g).
- (k1) and (k_2) represent the rate constants for pseudo-first-order adsorption kinetics, expressed in grams per milligram per minute (g/(mg·min)).
- (t) refers to the contact time, measured in minutes.
- (K[diff]) signifies the intraparticle diffusion rate constant, expressed in milligrams per gram per minute raised to the power of -1/2 (mg g^{-1} $min^{-1/2}$).

Figs. 7-1 to 7-3 illustrate the values derived from the kinetic variables of methylene blue adsorption on the adsorbent. Table 2 presents the correlation coefficients (R^2) and the constants associated with the adsorption kinetics as determined from the kinetic diagrams. Notably, the determined capacities of the adsorption from both the pseudo-first-order model and the intraparticle penetration model differ from the experimental values, suggesting that the adsorption of methylene blue on the adsorbent does not conform to these kinetic models. Conversely, the pseudo-second-order kinetic model exhibits a high correlation coefficient of 0.9996, indicating that the process of the adsorption is predominantly governed by chemisorption.

Table 1. The obtained parameters of isotherm models for methylene blue adsorption.

Langmuir				Freundlich			Temkin		
q_m	K_L	R^2	R_L	K_F	n_F	R^2	K_2	K_1	R^2
70.92	0.13	0.885	0.1	8.89	1.43	0.995	1.95	12.357	0.928

Table 2. Parameters for various kinetic models for methylene blue adsorption.

P- F-order			P-S-order			I-P-D		
q_e	K_1	R^2	q_e	K_2	R^2	K_{dif}	C	R^2
1.02	0.073	0.7822	7.86	0.12	0.999	0.09	7.03	0.5978

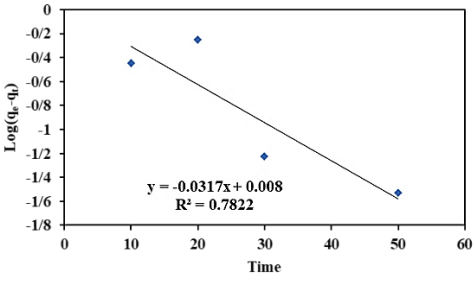

Figure 7-1. Kinetic model of the pseudo-first order.

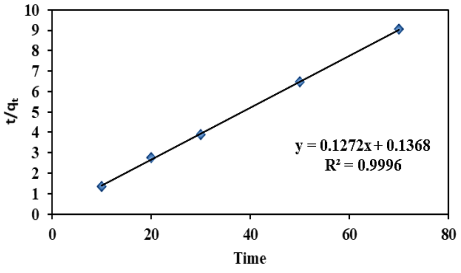

Figure 7-2. Kinetic model of the pseudo-second order.

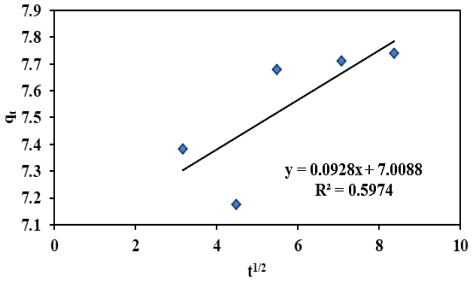

Figure 7-3. Kinetic model of intraparticle diffusion.

3.1. Investigating the thermodynamics of the reaction

To explore the thermodynamics of the process at temperatures of 25, 30, 40, and 50 degrees Celsius, the following equations were employed [22]:

$$K_d = \frac{q_e}{C_e} \quad (6)$$

$$\Delta G = -RTLnK_d \quad (7)$$

In these relationships, K_d is the equilibrium constant, q_e is the concentration of methylene blue adsorbed on the adsorbent at the time of equilibrium (C_e, mg/L), the equilibrium concentration of methylene blue (R, mg/L) represents the global gas constant (J/mol). K is 8.314 and T is the absolute temperature in Kelvin.

Enthalpy (ΔH°) and entropy (ΔS°) parameters related to the adsorption process are obtained through the following equation [22]:

$$\text{Ln } K_d = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \quad (8)$$

As the ambient temperature rose from 25 to 50 °C, the efficiency of methylene blue adsorption diminished, suggesting that the reaction is exothermic. Furthermore, as indicated in Table 3, the negative values of the ΔG parameter confirm that the adsorption of methylene blue is thermodynamically favorable and can occur spontaneously. Additionally, the negative value of ΔH° reinforces that the process is exothermic. It is evident that entropy plays an unfavorable role in the adsorption of methylene blue, with the process being predominantly influenced by enthalpy.

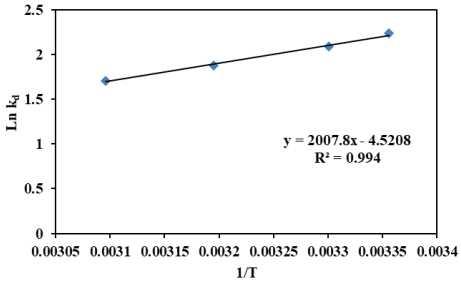

Figure 8. Linear diagram of Ln kd in relation to 1/T to determine thermodynamic parameters.

Table 3. Calculated thermodynamic parameters for methylene blue adsorption.

Temperature(°K)	ΔG° (J mol^{-1})	ΔH° (J mol^{-1})	ΔS °(J mol^{-1} K^{-1})
25	-5538.69	16692.8	-37.59
30	-5261.45	-	-
40	-4893.38	-	-
50	-4585.24		

4. Conclusion

This study introduces an innovative composite material specifically engineered for the efficient elimination of methylene blue dye from aqueous solutions. A comprehensive examination of the composite's morphology and structural properties was conducted utilizing advanced techniques. Adsorption experiments indicated that optimal conditions for dye removal were achieved under the following parameters: an adsorbent mass of 30 mg, a solution pH of 7, an initial dye concentration of 10 mg/L, a temperature of 25°C, and an adsorption duration of 30 minutes. The equilibrium data revealed that the adsorption of methylene blue is consistent with the Freundlich isotherm model, indicating a heterogeneous surface for adsorption. Furthermore, the maximal capacity of adsorption, as calculated using the Langmuir isotherm model, was determined to be 70.92 mg/g. Kinetic analysis revealed that the pseudo-second-order kinetic model most accurately represents the adsorption mechanism, implying that chemical adsorption predominantly drives the process. Moreover, thermodynamic evaluations demonstrated that the adsorption of methylene blue onto the composite is both thermodynamically spontaneous and endothermic. These findings underline the composite's effectiveness in applications aimed at dye removal.

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