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Removal of Methylene Blue Dye from Aqueous Solutions Using Modified Kaolin via Adsorption

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Modifiye Kaolin Kullanarak Sulu Çözeltilerden Metilen Mavisi Boyasının Adsorpsiyon Yoluyla Giderilmesi

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Abstract

In this research, the adsorption efficiency of nitric acid (HNO_3)-modified kaolin (MK) clay for the removal of methylene blue (MB) from aqueous solutions was examined. The influence of key operational parameters, including contact time and the initial concentration of MB, on the adsorption performance was systematically studied. Experimental findings indicated that the optimal conditions for MB elimination were achieved at an initial dye concentration of 350 mg/L and a contact duration of 80 minutes. Structural and compositional alterations on the adsorbent surface before and after adsorption were characterized using scanning electron microscopy (SEM), energy-dispersive spectroscopy (EDS), and Fourier transform infrared spectroscopy (FTIR) analyses. The adsorption data were found to be consistent with the Langmuir isotherm model, suggesting that both chemisorption and irreversible interactions played a role in the process. The maximum adsorption capacities of MB were determined to be 32.26, 34.13, and 35.46 mg/g at 25, 35, and 45 °C, respectively. Kinetic modeling demonstrated that the adsorption process followed the pseudo-second-order (PSO) model. Overall, the study highlights that modified kaolin is a promising, low-cost, and efficient adsorbent for the removal of methylene blue dye from wastewater.

Keywords: *Kaolin; Adsorption; Methylene Blue; İzoterm; Kinetic.*

Öz

Bu araştırmada, sulu çözeltilerden metilen mavisi (MB) giderimi için nitrik asit (HNO_3) ile modifiye edilmiş kaolin (MK) kilinin adsorpsiyon verimliliği incelenmiştir. Temas süresi ve MB'nin başlangıç konsantrasyonu gibi temel operasyonel parametrelerin adsorpsiyon performansı üzerindeki etkisi sistematik olarak incelenmiştir. Deney sonuçları, MB giderimi için optimal koşulların, 350 mg/L başlangıç boya konsantrasyonu ve 80 dakikalık temas süresi ile elde edildiğini göstermiştir. Adsorpsiyon öncesi ve sonrası adsorban yüzeyindeki yapısal ve bileşimsel değişiklikler, taramalı elektron mikroskobu (SEM), enerji dağılımlı spektroskopi (EDS) ve Fourier dönüşümlü kızılötesi spektroskopi (FTIR) analizleri kullanılarak karakterize edilmiştir. Adsorpsiyon verilerinin Langmuir izoterm modeliyle tutarlı olduğu bulunmuş ve bu da hem kimyasal adsorpsiyonun hem de geri dönüşümsüz etkileşimlerin süreçte rol oynadığını göstermiştir. MB'nin maksimum adsorpsiyon kapasitelerinin sırasıyla 25, 35 ve 45 °C'de 32,26, 34,13 ve 35,46 mg/g olduğu belirlenmiştir. Kinetik modelleme, adsorpsiyon sürecinin sözde ikinci derece (PSO) modelini izlediğini göstermiştir. Genel olarak, çalışma, modifiye kaolinin atık sudan metilen mavisi boyasının giderilmesi için umut verici, düşük maliyetli ve verimli bir adsorban olduğunu vurgulamaktadır.

Anahtar Kelimeler: *Kaolin; Adsorpsiyon; Metilen Mavisi; İzoterm; Kinetik.*

1. Introduction

With the rapid increase in industrialization and urbanization, environmental pollution caused by dyes poses a serious threat to all living things (Métivier-Pignon et al. 2003). These toxic dyes permanently pollute food sources, drinking water reserves, agricultural lands, and wastewater (Sayğılı et al. 2015; Kılınç et al. 2022; Hosny et al. 2022; Ekinci, 2023). Methylene blue (MB) carries significant risks for the environment and human health due to its complex aromatic structure (Sayğılı et al. 2015; Kılınç et al. 2023). The accumulation of MB in water resources inhibits photosynthetic activity by reducing light transmittance, thus disrupting the ecosystem balance and causing irreversible damage to aquatic plants and aquatic organisms. In addition, long-term exposure to high concentrations of MB can cause health problems such as respiratory disorders, weakening of the immune system, and metabolic imbalances and threaten human health with mutagenic and carcinogenic effects. Therefore, effectively removing MB from aqueous media is essential for environmental sustainability (Zhang et al. 2023).

Adsorption is a low-cost, efficient, and environmentally friendly method for removing toxic dyes such as methylene blue (MB) from aqueous solutions (Zhang et al. 2023). Since it has a more straightforward application process than alternative treatment techniques and offers high removal capacity, it is widely used to treat industrial wastewater. In order to minimize the negative effects of MB on the environment and human health, various

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natural materials such as clay minerals, activated carbon, and plant wastes have been evaluated as adsorbents (Hosny et al. 2022). Studies have shown that both natural and surface-modified adsorbents show high efficiency in removing dye pollution. The large surface area, high porosity, and good adsorption capacity of these materials contribute to reducing environmental pollution by facilitating the retention of pollutants. Furthermore, these adsorbents are biodegradable, recyclable, and sustainable, offering an environmentally friendly alternative in industrial wastewater treatment. In this context, the development and widespread use of natural and modified adsorbents is essential in reducing environmental pollution and protecting water resources (Tan et al. 2008; Canpolat et al. 2022). Clays are natural, eco-friendly materials characterized by their large specific surface area and excellent cation exchange capacity. Kaolin, a commonly found clay mineral, is recognized for its strong ability to adsorb harmful and toxic dyes from aqueous environments (Robinson et al. 2001; Ravi et al. 2005). This property can be improved by incorporating inorganic and/or organic substances into the interlayer structure of kaolin and changing the selectivity and mechanism of kaolin-dye interactions. The adsorption capacity of the clay surface can be enhanced by acid or base treatment or functionalization with certain additives, thereby strengthening the interactions on the surface and enabling more efficient removal of dyes (Ghosh and Bhattacharyya, 2002; Wu and Tseng, 2008).

In this study, the capacity of modified kaolin (MK) to remove methylene blue (MB) from aqueous solutions was investigated in detail. Various experimental tests and analyses were carried out to evaluate the efficiency of MK in MB adsorption. In line with the data obtained, many experimental parameters were comprehensively analyzed to determine the adsorption efficiency of MK for MB. In particular, the factors that maximize the interaction between MB and MB in solution were revealed by examining the effect of initial MB concentration on adsorption efficiency. Analyzing these parameters allowed the determination of the optimal conditions for the effective removal of MB. In addition, the study evaluates the kinetics and isotherms of the adsorption process, ultimately determining the adsorption capacity of MB.

2. Materials and Methods

Kaolin-type clay used in this study was obtained from quarries in Şenkaya district of Erzurum province. In preparing acid-modified kaolin, raw kaolin was first dried at a specific temperature to remove the moisture content. Then, it was ground to obtain a homogeneous particle size distribution and sieved to remove particles below 100 µm. After these treatments, a 1 M nitric acid (HNO_3) solution was prepared for modification. Next, 250 mL of a 1 M HNO_3 solution was added to a 500 mL flask containing 20 g of kaolin. The mixture was stirred continuously for 24 hours. After stirring, the kaolin was filtered to remove acid residues and washed with deionized water to bring the pH of the filtration to neutralize. To finish the modification procedure, the cleaned kaolin was dried at 105 °C in an oven. A 500 mg/L stock solution of methylene blue (MB) was prepared for adsorption experiments, and MB solutions with different concentrations were prepared with this stock solution. All solutions used in the experiments were freshly prepared diluted solutions. The Sigma-Aldrich business supplied the chemicals utilized in the investigation.

The binding patterns of functional groups were examined using Fourier Transform Infrared (FTIR) spectroscopy (SHIMADZU IRXross) on the modified kaolin (MK) surface before and after adsorption. This technique provides an effective tool to detect chemical changes and functional group interactions occurring on the surface of MK. In addition, A scanning electron microscope (SEM) (JEOL JSM-6010LA) equipped with energy dispersive X-ray spectroscopy (EDS) was used to examine the surface morphology and composition in detail. While EDS gave details on the elemental composition and distribution of MK, SEM photos showed changes in the surface morphology of kaolin. This combination offers an essential contribution to understanding adsorption processes and a detailed evaluation of the changes on the adsorbent surface.

It was reported that 20 mL of solution at different concentration ranges (between 100 and 500 mg/L) were used in adsorption trials in 100 mL conical flasks and agitated in a water bath shaker at a rotation rate of 100 cycles per minute. After the adsorption process, the amount of unadsorbed MB remaining in the aqueous medium was measured by UV-Vis Spectroscopy (Shimadzu-1900 model device) at a wavelength of λ = 664 nm. MB's initial concentration and contact time were thoroughly examined and used to determine the optimum parameters to maximize the adsorption efficiency. Furthermore, the effect of thermal conditions on the adsorption process was evaluated at controlled temperatures of 25, 35, and 45 ºC. Equation 1 was used to determine the amount of adsorbed MB (Canpolat, 2023)

$$q_e = \frac{(C_0 - C_e)}{W} V \quad (1)$$

The mass of the adsorbent in this study is represented by W(g), and the volume of solution used is represented by V(mL). C_o and C_e (mg/L) were the MB solution's starting and equilibrium concentrations, respectively.

3. Results and Discussion

3.1. Characterization studies

The binding process of MB dyestuff was examined and possible adsorption functional groups on the MB surface were identified using FTIR spectroscopy. FTIR spectra were recorded and compared before and after the adsorption of MB, and these data are presented in Figure 1. When the IR spectrum of MB is analyzed, the peaks at 1005 and 1114 cm^{-1} reflect the stretching vibrations of Si-O-Si groups in the tetrahedral structure. The peak at 685 cm^{-1} shows the vibration band associated with the silica compound, and the peaks at 458 and approximately 529 cm^{-1} show the vibrations of the Si-O-Si and Al-O-Si bonds, respectively. After the adsorption of MB dyestuff, significant shifts in these characteristic peaks and decreases in the vibration bands were observed. These spectral changes indicate that the functional groups on the MK surface directly interact with MB molecules and actively participate in the adsorption process. In particular, the decreases in peak intensities suggest that the functional groups are partially saturated or subjected to chemical binding during MB adsorption. Furthermore, the shifts in wavelengths indicate the presence of different adsorption mechanisms, such as electrostatic interactions, hydrogen bonding, and possible covalent bond formation. These findings show that functional groups on the MK's surface are crucial for MB adsorption and that these interactions have a significant impact on the adsorption capacity.

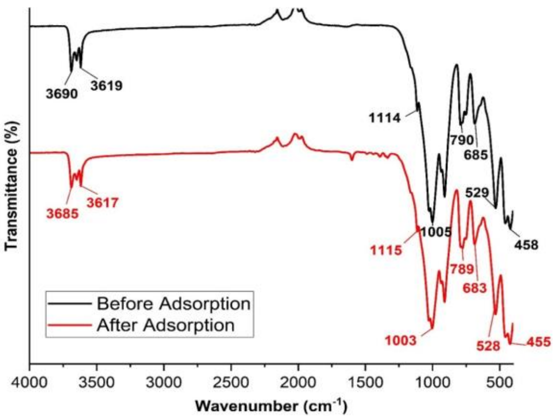

Figure 1. FTIR spectrum before and after adsorption

The surface morphology and chemical composition of MK were examined using SEM and EDS both before and after adsorption. The SEM map obtained before adsorption (Figure 2a) shows that MK is heterogeneous and has different colors and region densities on the surface. The SEM map obtained after MB adsorption (Figure 2b) reveals the presence of significant color changes on the surface of the MK, indicating the changes in the surface and the effect of MB on the adsorption process on the surface of the MK. In addition to SEM analysis, EDS analysis revealed remarkable changes in the surface chemical composition of MB before and after adsorption. An increase in the ratios of carbon (C), sulfur (S), and nitrogen (N) elements was observed on the surface after MB adsorption, confirming the binding of MB molecules to the surface. In addition, changes in the relative densities of some key components (e.g., silicon (Si) and aluminum (Al)) have occurred, suggesting that new chemical interactions and possible complex formation have happened on the surface. These morphological and chemical changes indicate that during MB adsorption, new compounds are formed on the MB surface, and the chemical properties of the surface are transformed. The initiation of a dissolution-precipitation mechanism characterizes the adsorption process due to physicochemical interactions between the surface and MB molecules. These findings, supported by EDS data, reveal that the changes occurring on the surface of MB after adsorption are essential both at morphological and chemical levels.

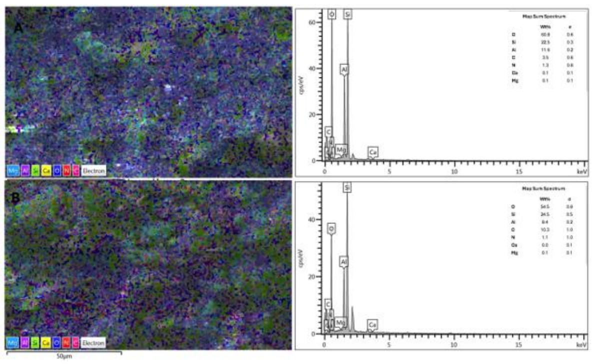

Figure 2. SEM/EDS spectrum before (A) and after (B) adsorption

3.2. Batch Experiments

3.2.1 Effect of initial concentration

The efficiency of the adsorption process is largely dependent on the solution's starting concentration. The findings obtained in this study show that the adsorption rate varies depending on both contact time and initial concentration, and this relationship can be explained by kinetic parameters, (Altunkaynak et al. 2024). To evaluate this effect, 0.2 g of MK was used in the specified experimental conditions and mixed with 20 mL of MB solution at 25, 35, and 45°C for 120 min. The initial concentrations of the MB solutions were systematically varied between 100 and 500 mg/L. The amount of MB that remained in the solution phase following adsorption was measured using a UV-Vis spectrophotometer.

The data presented in Figure 3 show that MB extraction from aqueous medium by MK varies depending on the initial concentration. The findings revealed that MB adsorption on the MK's surface increased up to a certain concentration threshold. Still, after exceeding this threshold, the adsorption capacity remained constant. The adsorbent surface's active site saturation by MB molecules can explain this. Furthermore, as shown in Figure 3, the experiments executed at 25, 35, and 45 °C showed an increase in the amount of MB adsorbed when the temperature rose, indicating that MB's adsorption capacity is positively correlated with temperature. In the later stages of the study, 350 mg/L concentrations of MB solutions were used with MK based on the data obtained.

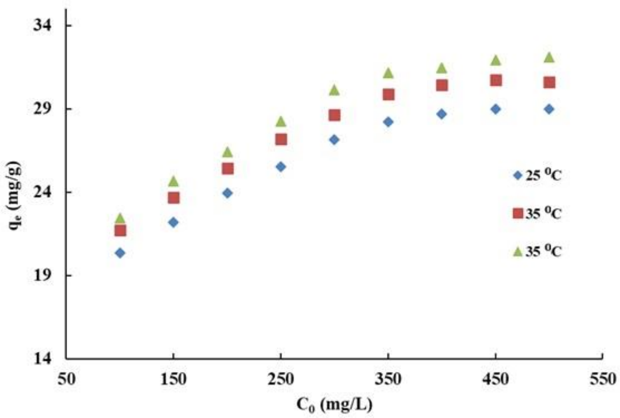

Figure 3. Impact of starting concentration on MK's ability to remove MB (Adsorbent mass of 0.2 g and V = 20 mL)

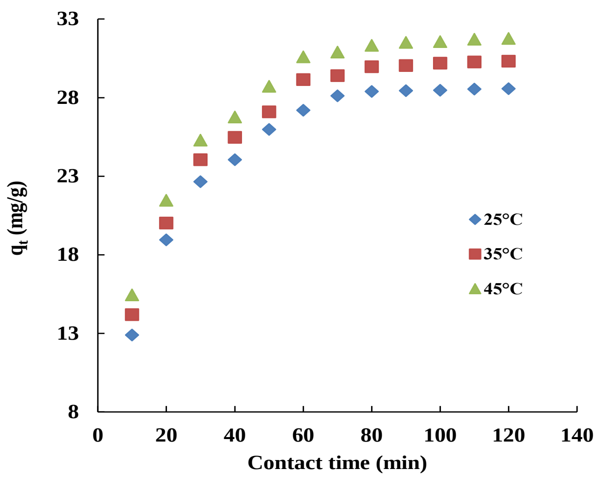

Figure 4. Impact of contact duration on MB adsorption with MK

3.2.2 Effect of contact time

In an aqueous solution, the adsorbent and MB's interaction period is one of the main parameters determining the efficiency of adsorption processes (Altunkaynak and Canpolat, 2023). Figure 4 shows the MB removal effectiveness changes depending on MK's contact time. The adsorption tests were conducted at temperatures of 25, 35, and 45 °C using 0.2 g adsorbent and MB solution at a 350 mg/L concentration. According to the data presented in Figure 4, it was determined that the amount of MB adsorbed (q_t) was plotted against time using the values measured in the period between 10 and 120 minutes, and the time of balance in the adsorption process was achieved in approximately 80 minutes. At the initial stage, the amount of MB removal showed a continuous increase during the first 60 minutes. However, a plateau trend was observed in the MB removal rate beyond this time because the saturation of the active sites on the adsorbent surface. This leads to the adsorption capacity remaining constant at a certain level despite increasing contact time.

3.2.3 Kinetic Study

Kinetic studies were carried out to understand the mechanism of MB adsorption on MB and to determine the rate-limiting step of the process. Contact time is a crucial parameter in the process of adsorption, and modeling of nonlinear kinetic was applied to investigate the kinetic properties of this process in more detail. Based on experimental data, pseudo-first-order (PFO) and pseudo-second-order (PSO) kinetic models were examined in the assessment of adsorption data. Equations 2 and 3 present the PFO and PSO models' nonlinear kinetic equations (Canpolat, 2023; Altunkaynak and Canpolat, 2023).

$$\ln(q_e - q_t) = lnq_e - k_1 t \qquad (2)$$

$$\frac{t}{q_t} = \frac{t}{q_e} + \frac{1}{k_2 q_e^2} \qquad (3)$$

Here, q_e represents the equilibrium adsorption capacity, and qt represents the amount of adsorbed MB at a given time t. The PFO and PSO kinetic model's rate constants are represented by k_1 and k_2, respectively.

The parameters obtained from the nonlinear fitting of the kinetic models are shown in Figures 5 and 6 and Table 1.

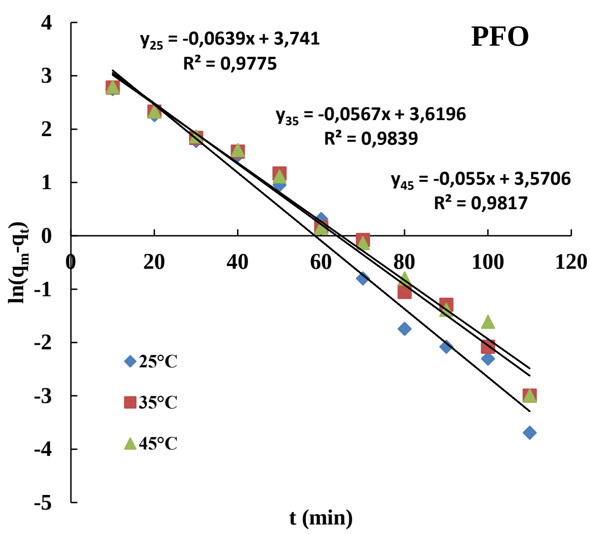

Figure 5. PFO model plot for MB adsorption on MK

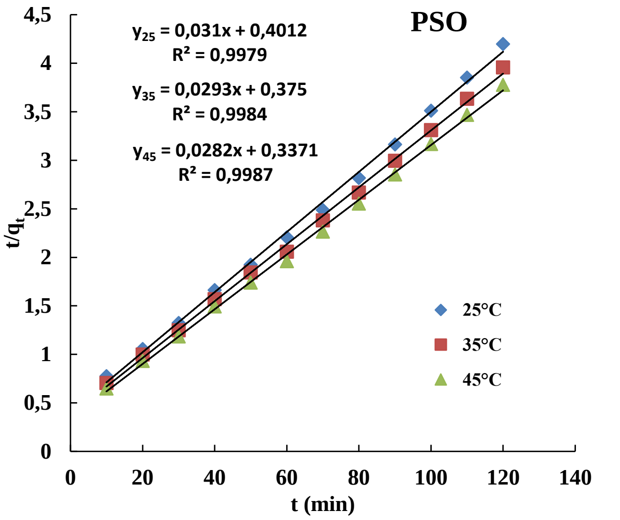

Figure 6. PSO model plot for MB adsorption on MK

Table 1. Kinetic factors for MB removal on MK at various temperatures

PFO Model	25 °C	35 °C	45 °C
q_e (mg/g)	42.14	37.32	35.54
k_1 (min^{-1})	0.064	0.057	0.055
R^2	0.9775	0.9839	0.9817
PSO Model			
q_e (mg g)	32.26	34.13	35.46
k_2 (g/mg min)	0.0024	0.0023	0.0024
R^2	0.9979	0.9984	0.9987

The values of q_e and q_t given in Table 1 are based on calculations performed at 25, 35, and 45 °C. The low R^2 coefficients of determination of the PFO model and the discrepancy between experimental (qexperimental) and theoretical ($q_{theoretical}$) adsorption capacity indicate that this model is unsuitable for explaining the adsorption process of MB. In contrast, the PSO model better represents the adsorption kinetics of MB by MK, supported by the high agreement between experimental and theoretical adsorption capacities and relatively high R^2 values.

3.2.4 Isotherm Study

The equilibrium adsorption characteristics of MK were assessed using the Langmuir and Freundlich isotherm models. These isotherm models describe the relationship between the adsorbent surface's characteristic properties and the adsorbate's adsorption capacity in solution. The Langmuir isotherm model assumes that MB adsorbs homogeneously and as a monolayer on the surface of MB. According to this model, adsorption is predicted to occur over a specific and limited quantity of active surfaces. Equation 4 presents the Langmuir isotherm's linear equation (Sayar and Canpolat, 2024; Altunkaynak and Canpolat, 2025).

$$\frac{Ce}{qe} = \frac{1}{qmax.K_L} + \frac{Ce}{qmax} \tag{4}$$

The maximum adsorption capacity is denoted by q_m, while C_e indicates the equilibrium concentration of MB, and q_e refers to the amount of MB adsorbed at equilibrium. The equilibrium constant associated with the adsorption process is represented by K_L.

The adsorbent surface's heterogeneous structure, multilayer adsorption, and molecular interactions between the adsorbent and adsorbate are all taken into account by the Freundlich isotherm model. This model explains the dynamic nature of the system by reflecting the complex interactions during the process of adsorption. Equation 5 represents the Freundlich isotherm model's linear version (Sayar and Canpolat, 2024; Altunkaynak and Canpolat, 2025).

$$logq_e = logK_F + \left({}^{1}/_{n}\right)logC_e \tag{5}$$

In this case, the adsorbent's adsorption capacity and the degree of surface heterogeneity are represented by the constants K_F and n in the Freundlich isotherm model, respectively.

The adsorption equilibrium data of the Freundlich and Langmuir isotherm models are presented visually in Figures 7 and 8. In addition, the calculated isotherm constants and q values obtained from the models are summarized in Table 2. Based on the comparison of the R^2 values and the agreement between the experimental and calculated q_e values, it was determined that the Langmuir isotherm model best describes the MB adsorption process on MK. This result indicates that the adsorption follows the Langmuir equation, suggesting monolayer adsorption on a homogeneous surface and implying that the adsorption mechanism is mainly

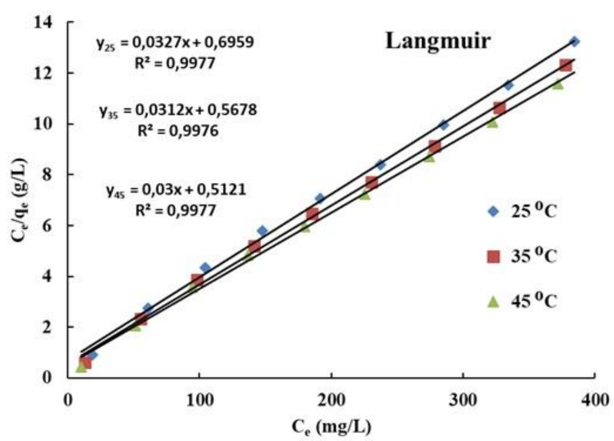

Figure 7. Plot for MB adsorption on MK using the Langmuir model

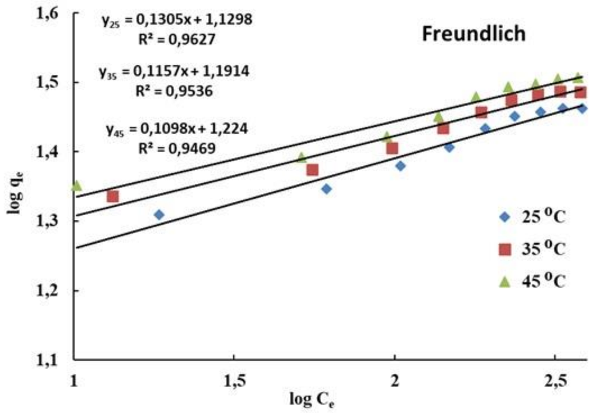

Figure 8. Plot for MB adsorption on MK using the Freundlich model

Table 2. Kinetic factors for MB removal on MK at various temperatures

Langmuir Model	**25 °C**	**35 °C**	**45 °C**
q_{max} (mg/g)	30.58	32.05	33.33
K_L (L/mg)	0.047	0.055	0.059
R^2	0.9977	0.9976	0.9977
Freundlich Model			
K_f (mg g)	13.48	15.54	16.75
n	7.66	8.64	9.11
R^2	0.9627	0.9536	0.9469

Table 3. Comparing the ability of several adsorbents to remove MB

Adsorbent	Adsorption capacity (mg/g)	Reference
Raw kaolin	15.26	Rida et al. 2013
Cold plasma modified kaolin	23	Yavuz and Saka 2013
HCl treated kaolin	27.13	Boukhemkhem and Rida 2017
Chitosan cross-linked zeolite	252.5	Hidayat et al. 2022
Zeolite-activated carbon from oil palm ash	285.71	Khanday et al. 2017
Mesoporous-activated carbon (CSAC) from chitosan flakes	143.5	Marrakchi et al. 2017
Clay-chitosan-alginate hydrogel beads	303.03	Bellaj et al. 2026
Sodium alginate-kaolin beads	188.7	Marzban et al. 2021
Chitosan-containing organoclay	138	Kabbadj et al. 2019
NaOH treated clay	16.3	Ghosh et al. 2002
Modified Kaolin	32.26 (25 °C) 34.13 (35 °C) 35.46 (45 °C)	This work

As shown in Table 3, the adsorption capacity obtained in this study is comparable to that of modified natural mineral-based adsorbents reported in the literature. Although some polymer-mineral composites exhibit higher adsorption capacities, their preparation generally requires more complex and costly processes. Therefore, the developed modified kaolin can be considered a promising adsorbent for the removal of methylene blue due to its simple preparation and satisfactory adsorption performance.

4. Conclusion

This study investigated using kaolin-type clay modified with HNO_3 as a successful adsorbent for the removal of MB from wastewater. The results revealed that MK removed MB dyestuff with high efficiency (32.26, 34.13, 35.46 mg/g at 25, 35, 45 °C, respectively). The Langmuir model best explains the adsorption process, according to equilibrium isotherm analysis. Kinetic modeling results showed that MB removal was fitted to the PSO. FTIR and SEM-EDS analyses confirmed that notable modifications occurred in the chemical and morphological structure of MB before and after adsorption. These changes and sedimentation mechanisms are thought to contribute significantly to removing MB from aqueous solutions. When the obtained data were evaluated, it was concluded that kaolin-derived clay offers a sustainable and economically viable alternative for MB removal in treatment of wastewater due to its wide availability, strong adsorption ability, and economical efficiency. The findings obtained in this study show that kaolin-derived clay is an effective adsorbent for MB removal. However, applying different chemical and physical modification methods may help increase the adsorption capacity. In particular, the effect of acidic and basic activation processes on surface properties should be investigated in detail. Furthermore, the sustainability of kaolin-based adsorbents can be increased by investigating their reusability and regeneration processes. Conducting field studies to determine their effectiveness in industrial wastewater is essential. In addition, analyzing the adsorption capacity of kaolin-derived adsorbents against different organic and inorganic pollutants can expand the potential of this material as a versatile treatment agent.

Declaration of Ethical Standards

The authors declare that they comply with all ethical standards.

Credit Authorship Contribution Statement Author

Author 1: Research, Methodology, Experiment, Writing
Author 2: Research, Methodology, Experiment, Writing

Declaration of Competing Interest
The authors have no conflict of interest to declare regarding the content of this article.

Data Availability
All data generated or analyzed during this study are included in this published article.

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