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Application of Magnetic Biochar@Alginate Composite as Adsorbent for Effective Removal of Methylene Blue from Aqueous Media

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Abstract: In recent years, magnetic biochar has been widely used for the removal of dyes in polluted water due to its magnetic separation abilities and has been used as composite adsorbents by incorporating different biomasses into its structure for the removal of such pollutants. In this study, a new composite magnetic biochar@alginate composite (mBC@Alg) was synthesized as an alternative adsorbent for the removal of methylene blue (MB) from aqueous solutions. The primary focus of this intensive preparation procedure is on the synthesis, characterization, and optimization of the mBC@Alg for enhanced MB adsorption capacity. The composite material was characterized using FT-IR analysis and scanning electron microscopy (SEM). Batch adsorption studies experiments were performed to determine the removal efficiencies, pH, adsorbent dose, contact time and initial concentration of MB molecules. Various adsorption isotherms such as Freundlich, Langmuir, Scathard and Dubinin-Radushkevich were used to describe the adsorption behavior at equilibrium. The Langmuir adsorption isotherm was identified as the most appropriate model to explain the observed adsorption phenomena, and the adsorption capacity of mBC@Alg for MB was determined to be 416.67 mg/g using this isotherm. Kinetic studies were carried out using pseudo-first-order and pseudo-second-order kinetic models and it was concluded that the experimental data fit well with the second-order kinetic model. The adsorbent properties of the mBC@Alg composite are particularly effective in removing MB molecules from aqueous solutions.

Keywords: Biochar, Alginate, Nano-Fe₃O₄, Methylene blue, Kinetics

Introduction

Dyes, and pigments from various human activities pose significant environmental risks, especially when discharged into aqueous medium. Due to their resistance to natural degradation, these substances threaten both human health and environmental safety. They can lead to adverse effects, including genetic mutations, allergic reactions, and cancer. Additionally, these contaminants harm aquatic ecosystems by lowering dissolved oxygen levels, reducing light penetration, and inhibiting photosynthesis.

The pollution of water bodies by organic dyes has become a pressing environmental concern, largely driven by industrial activities like textile dyeing, printing, and leather processing (Parlayıcı & Pehlivan, 2021). Among these dyes, methylene blue (MB) is widely utilized across multiple industries; however, its release into water systems poses serious health and ecological risks due to its toxicity, persistence, and potential for bioaccumulation. Consequently, developing efficient and sustainable methods for removing MB from aqueous environments is crucial to safeguard both environmental quality and public health (Jiang et al., 2018; Kasbaji et al., 2023). Adsorption has emerged as one of the most effective techniques for dye removal, given its simplicity, low energy requirements, and high efficiency. Recently, biochar (BC) — a carbon-rich adsorbent derived from biomass — has garnered attention as an eco-friendly adsorbent due to its porous structure, large surface area,

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and modifiable functional groups (Srivatsav et al., 2020; Vafakish et al., 2024; Parlayıcı et al., 2024; Suratman et al., 2024). However, conventional BC often lacks the magnetic properties needed for easy separation from treated water, which can complicate its practical application. To address this limitation, researchers have focused on enhancing biochar with magnetic nanoparticles, creating magnetic BC that can be easily separated via external magnetic fields (Feng etal., 2021; Yi et al., 2020).

Natural biopolymers (chitosan, cellulose, lignin, and alginate) demonstrated excellent affinity toward heavy metals and dyes through different mechanisms including, electrostatic attraction, complexation, chelation, and ion exchange (Doyo et al., 2023; Fouda-Mbanga et al., 2021; Kasbaji et al., 2024). In addition to magnetic modification, incorporating BC into a composite with biocompatible materials such as alginate can further enhance its adsorption capacity and structural integrity (Salem et al., 2023). Alginate, a natural polysaccharide derived from brown algae, is commonly used due to its non-toxicity, biodegradability, and gelling properties (Liu et al., 2023; Bahsaine et al., 2024; Aichour et al., 2024; Parlayıcı & Pehlivan, 2023). The combination of magnetic BC and alginate creates a robust, multifunctional composite that can effectively capture and remove pollutants like MB from water.

This study examines the use of a mBC@Alg composite as an adsorbent for removing MB from aqueous solutions. The research includes the synthesis, characterization, and evaluation of the mBC@Alg composite's adsorption performance, focusing on its adsorption capacity, kinetics, and the influence of factors such as pH, contact time, and initial dye concentration. The results provide insights that support the development of efficient, reusable, and eco-friendly adsorbents for water purification, presenting a promising approach to mitigating water pollution.

Method

Materials

All analytical-grade chemicals were used as received, without any further purification. MB was obtained from Acros Organics (New Jersey, USA). Sigma-Aldrich was the supplier of both the sodium alginate powder and the iron oxide (Fe_3O_4) nanopowder. Hydrochloric acid (HCl) and sodium hydroxide (NaOH) were purchased from Merck.

Preparation of Adsorbent



Figure 1. Synthesis scheme of mBC@Alg composite.

The pomegranate was obtained from a local market in Turkey. The peel and fruit parts were separated. The peels were washed with pure water and dried in an oven for 24 hours. The dried seeds were heated at 600 °C in a Magma THERM MT1210/B2 muffle furnace for 3 hours and converted to BC in the presence of low oxygen (Amalina et al., 2022). The BC, cooled in a desiccator, was ground into a powder. Particles of 125 μ m in size, obtained by sieving, were used in the preparation of the composite.

For the preparation of mBC@Alg, sodium alginate was first dissolved in 100 ml (5% w/v) distilled water. It was mixed on a magnetic stirrer for about 4 hours to obtain a homogeneous gel. Meanwhile, a suspension of 2.5 g of nano-Fe₃O₄ in 20 ml of distilled water and a suspension of 2.5 g of BC in 20 ml of distilled water were prepared. These suspensions were mixed individually at 300 rpm for 2 hours. The prepared suspensions were then added to the prepared alginate gel and mixed on a magnetic stirrer for 6 hours until a homogeneous solution was formed. Spheres were created by adding the mixture dropwise into a 0.5 M CaCl₂ solution using an injector, and the spheres formed were mixed on a magnetic stirrer at 100 rpm for about 3 hours. The beads were left in this solution overnight, then collected by filtration and thoroughly washed with water until a neutral pH was achieved. Finally, the beads were filtered out and dried at 60 °C for 24 hours (Figure 1).

Characterization



Figure 2. SEM image mBC@Alg composite (a) and after MB adosorption (b).

The morphology of mBC@Alg before and after the adsorption of MB ions was examined using SEM imaging. A thorough understanding of the structure and morphology of the mBC@Alg is necessary to evaluate its efficacy and potential interactions with MB. The adsorption efficiency of the composite is directly impacted by the surface properties, particle size, porosity, and overall structural integrity that may be shown by scanning electron microscopy (SEM). According to SEM pictures, the mBC@Alg has a porous surface structure with different levels of homogeneity and roughness, which is crucial for increasing adsorption sites and surface area. It appears that the magnetic characteristics were successfully incorporated into the composite because the magnetic particles contained in the BC matrix are clearly distributed. When Figure 2a. is examined, the roughness of the surface draws attention. As can be seen from Figure 2b., MB ions were coated on the pores and accumulations were seen on the surface. This showed that MB ions were adsorbed to the mBC@Alg surface.

Important information about the chemical structure and functional groups of the mBC@Alg composite can be obtained from its Fourier Transform Infrared (FT-IR) spectrum. Characteristic peaks in the FT-IR analysis show the presence of functional groups in the matrix of the alginate and biochar components (Thakur et al., 2016, Kusuktham et al., 2014). These peaks are frequently observed in areas that correlate to C=O stretching, O–H stretching, and C–O–C vibrations. Iron oxide (FeO₄) nanoparticles, which may exhibit unique peaks associated with Fe–O stretching, are commonly used to add magnetic characteristics. According to the FT-IR spectrum of mBC@Alg, it was observed that the stretching vibration peak of O–H is represented in the range of 3270 cm⁻¹. The peak at 1590 cm⁻¹ is caused by carboxyl –C=O stretching vibrations. The vibration peak at 1420 cm⁻¹ is aromatic C=C stretching vibrations. The stretching at 1016 cm⁻¹ corresponds to the ether –C–O and alcohol –C–O stretching. The characteristic absorption bands associated with Fe₃O₄ nanoparticles are observed at 535 cm⁻¹, which is related to the stretching modes of the Fe–O bond.



Figure 3. FT-IR spectra of MB before adsorption by mBC@Alg (a) and after adsorption by mBC@Alg (b).

Results and Discussion

Effect of pH on MB adsorption

One important aspect affecting the effectiveness of dye removal from wastewater is the impact of pH on the adsorption of MB by a composite adsorbent (Liu et al., 2022). The ionization state of the adsorbent and the adsorbate are both significantly influenced by the pH of the solution. The attraction between positively charged MB molecules and negatively charged sites on the composite can be strengthened in acidic environments by protonating functional groups on the surface of the BC (Xu et al. 2024). The deprotonation of these groups, on the other hand, may decrease the adsorption capability in alkaline circumstances because of increased electrostatic repulsion. Therefore, maximizing the effectiveness of the mBC@Alg composite in wastewater treatment applications requires an understanding of the ideal pH range for MB adsorption.

The remaining MB concentrations measured at pH levels 2, 3, 4, 5, 6, 7, and 8 after the 60-minute equilibrium period were analyzed to determine how pH affected the adsorption of MB ions by mBC@Alg composite. The adsorption process is further affected by the interaction between pH and the structural characteristics of the mBC@Alg composite. Variations in pH can influence the surface charge, porosity, and availability of active sites on the composite material. Research indicates that hydrophobic interactions between MB and the BC

matrix might be enhanced at certain pH levels, resulting in improved adsorption efficiency. Thus, it is crucial to thoroughly investigate how pH impacts adsorption kinetics and equilibrium to develop effective treatment strategies. The optimal pH range for enhancing the removal efficiency of MB is between pH 6 and pH 8 (Figure 4.).



Figure 4. Effect of pH on MB adsorption.

Effect of Contact Time on MB Ions Adsorption

The effect of contact time on MB adsorption was studied at various intervals (5, 15, 30, 60, 120, 180, and 240 minutes) with an adsorbent dosage of 2 g/L, at pH 6, and a temperature of 25 °C. An optimal contact time of 60 minutes was selected for further experiments. A key factor influencing the effectiveness of pollutant removal from aqueous solutions is the role of contact time in the adsorption of MB onto a mBC@Alg composite. Typically, the adsorption capacity increases with longer contact times due to enhanced interactions between the MB and the surface functional groups of the composite material. Initially, adsorption occurs rapidly as accessible sites on the mBC@Alg composite surface are filled. However, as these sites become occupied, the adsorption rate diminishes, leading to a state of equilibrium (Figure 5). An optimal contact time of 60 minutes was selected for further experiments. Understanding this relationship is essential for optimizing the design of mBC@Alg composite in wastewater treatment, as it helps determine the optimal contact time required to achieve maximum removal efficiency of MB.



Figure 5. Effect of contact time on MB adsorption.

Adsorbent Dose Effect

The effect of adsorbent dosage (Figure 6.) on the removal of MB ions was investigated through adsorption experiments at various doses (0.25, 0.50, 1.0, 1.25, 1.50, and 2.0 g/L). The optimal adsorbent dosage was determined to be 1.25 g/L.



Figure 6. Effect of adsorbent dosage on MB adsorption.

Effect of Initial Concentration on MB Adsorption

The starting concentration of MB in a solution significantly influences the adsorption capacity of mBC@Alg composite. As the initial concentration rises, the force driving mass transfer between the liquid phase and the solid adsorbent intensifies, leading to increased adsorption rates. When concentrations are low, the available sites on the mBC@Alg composite are not fully engaged, resulting in lower removal efficiencies. However, as the concentration increases, more MB ions interact with the surface functional groups of the composite, thereby enhancing the overall adsorption capacity. This relationship underscores the importance of initial concentration in assessing the effectiveness of the adsorbent material. The adsorption capacity was evaluated by varying the initial MB concentration (25, 50, 100, 150, 200, 250, 300, 400, and 500 ppm).

Although higher initial concentrations generally improve adsorption, this trend has its limits as shown in Figure 7. At elevated concentrations, the mBC@Alg composite may reach a saturation point where most adsorption sites are filled. Beyond this saturation level, the adsorption rate may decline, leading to diminishing returns in MB removal efficiency. Additionally, factors such as competition among MB ions for available sites and possible aggregation of the composite can hinder effective uptake at very high concentrations.



Figure 7. Effect of initial concentration on MB adsorption.

Adsorption Isotherms

Understanding the effectiveness of mBC@Alg composite as adsorbent for MB treatment requires an understanding of adsorption isotherms. These isotherms provide insights into how pollutants interact with the composite material at various concentrations, enabling researchers to analyze and predict the adsorption behavior of contaminants, including organic dyes. Key factors like maximal adsorption capacity and surface heterogeneity can be found by analyzing the data using different models, such as Freundlich, Langmuir, Scathard and D-R isotherms (Langmuir, 1917, Freundlich, 1906, Scatchard, 1949, Dubinin and Radushkevich,

1947) (Figure 8.). These parameters are crucial for improving the design and use of these composites in practical situations (Parlayici & Pehlivan, 2024).

The appropriateness of the Langmuir isotherm model in characterizing the adsorption behavior of contaminants on mBC@Alg composite suggests a monolayer adsorption mechanism, in which each adsorption site accommodates only one molecule of the adsorbate (Huangfu et al., 2023). This model is based on the premise of uniform adsorption sites and a limited number of available locations, which corresponds well to the properties of these composites, especially when their surface functionalization is tailored for specific contaminants (Qi, et al., 2018). The maximum adsorption capacity, denoted as q_e , is a vital parameter derived from the Langmuir isotherm, indicating how effectively the composite can bind pollutants. q_e was calculated as 416.7 mg/g. A higher value q_e reflects an increased ability of the adsorbent to capture contaminants, underscoring its potential for effective use in wastewater treatment. Therefore, the Langmuir model not only facilitates predictions of adsorption performance but also serves as a guide for optimizing the design of mBC@Alg composite to maximize MB removal efficiency.





Model	Equation	Paramete	ers for dye		
Langmuir	$\frac{\overline{C_e}}{\overline{q_e}} = \frac{\overline{C_e}}{\overline{A_s}} + \frac{1}{\overline{K_b}A_s}$	q _e 416.7	К ь 0.0232	R ² 0.984	R _L 0.177
Freundlich	$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e$	K _f 12.96	n 1.37	R ² 0.965	
D-R	In $q_e = \ln q_m - \beta \epsilon^2$	X _m 0.008	K 0.006	E 9.13	R² 0.984
Scathard	$qe/C_e = Q_s K_s - q_e K_s$	Q s 475.22	K _s 0.019	R ² 0.947	

Table 1. Adsorption isotherm parameters for removal of MB.

Adsorption Kinetic Modeling

Understanding the rate and mechanism of contaminant uptake in dye treatment applications involves kinetic modeling of MB adsorption onto mBC@Alg composite. Kinetic models such as pseudo-first-order and pseudo-second-order provide information on the factors that influence the rate at which MB molecules adsorb onto the composite surface. The pseudo-first-order model typically captures the initial rapid adsorption phase in which many adsorption sites are rapidly filled. In contrast, the so-called second-order model usually provides a better fit over longer time periods, suggesting that the adsorption process may be chemically controlled rather than entirely governed by diffusion.



Figure 9. Graphs of pseudo-first-order and pseudo-second kinetic models for MB adsorption.

 Table 2. Comparison of the pseudo-first-order, pseudo-second-order adsorption rate constants and calculated and experimental qe values obtained at different initial MB concentrations.

Pseudo First-order			Pseudo Second- order				
C _o (ppm)	q _{e exp}	\mathbf{k}_1	q _e	\mathbf{R}^2	k ₂	q _e	\mathbf{R}^2
100	76.30	0.0167	34.50	0.836	0.0006	82.64	0.992
200	149.01	0.0236	120.71	0.978	0.0002	169.49	0.996
300	217.04	0.0209	116.56	0.853	0.0002	243.90	0.989

By analyzing the kinetic parameters derived from these models, including rate constants and equilibrium times, they can optimize operational conditions to achieve maximum MB removal efficiency. The applicability of the pseudo-second-order rate mechanism for MB adsorption kinetic modeling on mBC@Alg composite highlights the complexity and efficiency of the adsorption process. This model is particularly relevant as it indicates that the rate of MB adsorption is primarily influenced by chemical interactions between the pollutant and the active sites on the composite surface, rather than merely by physical diffusion. The pseudo-second-order model provided a better fit for experimental data over extended time periods, suggesting that adsorption sites become increasingly occupied as equilibrium is approached (Figure 9). This behavior is crucial for practical applications, as it implies that the adsorbent can effectively bind higher concentrations of MB, making it a promising solution for dye treatment.

Thermodynamic Parameters

The thermodynamic parameters associated with MB adsorption on mBC@Alg composite provide critical insights into the nature of the adsorption process and its feasibility under varying temperature conditions. The effect of temperature on MB adsorption was investigated over a temperature range of 25 °C, 35 °C, and 45 °C (Figure 10.) and thermodynamic parameters were calculated. These parameters such as the change in Gibbs free energy (ΔG°), enthalpy (ΔH°), and entropy (ΔS°) can be derived from equilibrium data given in the Table 3., offering a comprehensive understanding of the energetic and disorder aspects of the adsorption process. A negative ΔG° value indicates that the adsorption is spontaneous, while positive ΔH° values suggest an endothermic process, implying that higher temperatures favor the adsorption of MB. Conversely, a positive ΔS°

value reflects an increase in randomness at the solid-liquid interface during the adsorption, which is indicative of favorable interactions between the composite material and the dye molecules. The value of ΔG° (Gibbs free energy change) becomes more negative as the temperature rises (Table 3), indicating that the process becomes more thermodynamically favorable at higher temperatures. This suggests that the uptake efficiency is greater at higher temperatures, where the more negative ΔG° values signify a stronger tendency for the process to occur spontaneously (Bahrami et al., 2024).



Figure 10. Ln K_c -1/T plot

Table 3. Thermodynamic parameters for MB adsorption

ΔS^{o}	ΔH^{o}		ΔG^{o}	$(J \text{ mol}^{-1})$	
J K ⁻¹ mol ⁻¹	J mol ⁻¹	T=298.15K	T=308.15K	T=318.15K	R^2
87.85	20934.24	-5254.98	-6133.37	-7011.76	0.934

Conclusion

The study demonstrated that mBC@Alg composite is an effective and environmentally friendly adsorbent for removing MB from aqueous media. By integrating BC with alginate and magnetic particles, the composite combines high adsorption efficiency, easy recovery, and reusability, providing a sustainable approach to wastewater treatment. The experiments demonstrated that optimal conditions for effective MB removal included an equilibrium time of 60 minutes, an initial pH of 6, an initial dye concentration of 200 ppm, and a composite dosage of 2 g/L. The adsorption process was shown to follow Langmuir isotherm and pseudo-second-order kinetic models, indicating a monolayer adsorption mechanism with strong interaction between MB and the mBC@Alg composite. The pseudo-second-order kinetics model was selected for time-dependent studies to accurately describe the adsorption process and predict the rate of MB removal over time. Furthermore, the magnetic properties of the composite allowed facile separation from the solution, making it practical for realworld applications. The reusability studies revealed that mBC@Alg composite could maintain substantial adsorption capacity over multiple cycles, reinforcing its economic and environmental viability. In conclusion, mBC@Alg composite is a promising adsorbent for the removal of MB from water systems, highlighting its potential application in industrial wastewater management and environmental remediation efforts. Future research could explore its application for various other pollutants and optimization in industrial-scale setups to fully realize its potential in diverse environmental applications.

Scientific Ethics Declaration

The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM Journal belongs to the authors.

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