

Biomaterials and Nanotechnology: Green Tea (Waste) – Aluminium Nanoparticles (Gt-Al Nps) For Methylene Blue Adsorption

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Abstract

This study examines the adsorption of Methylene Blue (MB) dye from aqueous solutions using Aluminium Nanoparticles synthesized with green tea powder as a biological reducing agent. Methylene Blue, an aromatic phenothiazine dye, is widely applied in medical diagnostics and various industrial processes, but its uncontrolled release into water bodies leads to environmental pollution due to its intense colour, stability, and potential toxicity. Therefore, identifying an efficient and eco-friendly adsorbent for Methylene Blue removal is essential. In this study, Green Tea–Aluminium (GT–Al) Nanoparticles were prepared and used as a low-cost adsorbent. The results collectively highlight the potential of GT–Al nanoparticles as a sustainable alternative to conventional chemical adsorbents. Green tea contains natural polyphenols and biomolecules that assist in reducing and stabilizing aluminium ions during nanoparticle formation. The synthesized nanoparticles were characterized using SEM, EDX, and TGA analysis to determine surface morphology, elemental composition, and thermal stability. These characterizations confirm the successful formation and stability of GT–Al nanoparticles. Adsorption experiments were performed by varying key parameters such as contact time, adsorbent dosage, pH, initial dye concentration, and temperature to determine conditions that maximize dye removal efficiency. The equilibrium data were evaluated using Langmuir, Freundlich, and Temkin isotherm models, providing insight into adsorption behavior and surface interaction mechanisms. Structural changes in the nanoparticles before and after adsorption were analyzed using FTIR spectroscopy, which helped identify functional groups participating in dye binding. The adsorption kinetics were studied using pseudo-first order and pseudo-second-order models to understand the rate of MB uptake. The dye concentration was accurately measured using a UV–Visible Spectrophotometer.

Keywords: Adsorption, FTIR, green synthesis, isotherms, kinetics, nano particles, thermodynamics

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INTRODUCTION

Synthetic dyes are widely used in the textile, food, and cosmetic industries, but their release into water bodies poses environmental and health risks [1–3]. Methylene Blue dye is mainly utilized in the plastic, paper, textile, printing, pharmaceutical industries, and for medical applications [4, 5]. Several adsorbents, including activated carbon, clays, and biosorbents, have been reported [6–8]. However, these often suffer from regeneration and cost issues.

Nanotechnology offers highly efficient adsorbents due to their large surface area, tunable properties, and reactivity [9, 10]. Green synthesis of nanoparticles using plant waste extracts is a sustainable method [11, 12]. Tea waste contains

polyphenols and flavonoids that reduce and stabilise nanoparticles, making it an attractive precursor [13–15]. In this study, aluminium nanoparticles were synthesised using green tea waste extract and evaluated for Methylene Blue adsorption [16, 17].

Methylene Blue dye is a cationic water-soluble dye poses danger to human and aquatic life when excessive amount and exposure (properties and structure as indicated in Table 1, Figure 1).

MATERIALS AND METHODS

The biological waste (Green tea powder) is collected from the organic bin.

Chemicals Required

Chemicals

Aluminium sulfate (Al_2SO_4)₃, Hydrochloric acid (HCl), and Sodium hydroxide (NaOH).

Table 1. Properties of methylene blue.

Properties	
Chemical formula	$\text{C}_{16}\text{H}_{18}\text{N}_3\text{SCl}$
Molar mass	319.85 g/mol
Appearance	Dark green powder
Melting point	100 to 110 °C
Solubility in water	43.6 g/L (25 °C)

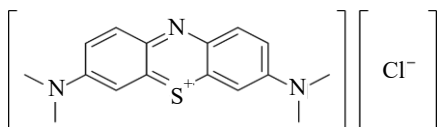


Figure 1. Structure of methylene blue dye.

Equipment

pH meter, UV spectrophotometer, Heating mantle, Centrifuge, and Glassware, etc.

Dyes

Methylene Blue.

Preparation of Standard Solutions

Solutions are prepared using distilled water, and all of the chemicals employed were of laboratory reagent quality.

Preparation of Aluminium Sulfate (Al_2SO_4)₃ Solution

120mg of Aluminium sulfate was diluted in 1000 ml of distilled water in a volumetric flask with a tightly fitting stopper.

Preparation of Green Leaves Extract

The Green Tea powder was initially boiled in water to acquire the extract, followed by separating, drying, powdering, and mixing.

Preparation of Extract from Green Leaves

To create Green Tea – Aluminum Nanoparticles (GT-Al NPs), 8 g of green tea extract, 100 ml of $\text{Al}_2(\text{SO}_4)_3$, and 5 ml of 10% NaOH (pH 10.5) were combined, agitated at 303K, centrifuged, and oven-dried (Figure 2).

Preparation of Dye Solutions

Melting 10 mg of Methylene Blue dye in a stock solution dissolved with a 1000 mg/L concentration. By gradually diluting the original solution with a suitable amount of distilled water, the dye concentration (10 – 100 mg/L) was achieved.

Calibration Curve

According to Beer's and Lambert's laws, a UV-Vis calibration curve at 650 nm demonstrated linearity with Methylene Blue concentrations (0–50 mg/L). Path length and concentration were correlated with absorbance (Figure 3). To guarantee precise measurement, samples outside of this range were diluted.

Characterization of Aluminium and Green Tea Aluminium Nanoparticles

Scanning Electron Microscope (SEM)

High-energy electron beams are used in scanning electron microscopy (SEM) to produce signals that show the structure, content, and surface morphology of a material. Preparation is necessary for non-conductive samples to avoid static accumulation. The ~200 nm-sized GT-Al NPs are photographed at 100.00 KX magnification in Figure 4, offering excellent resolution and steady probe currents for in-depth surface investigation.

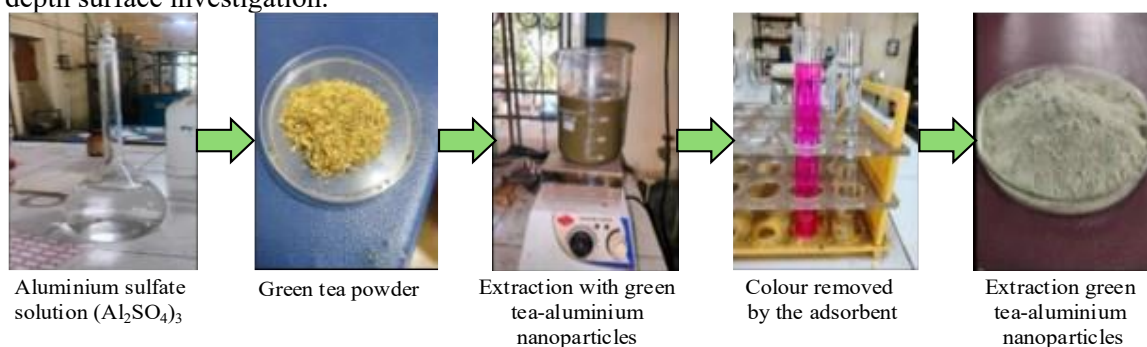


Figure 2. Flow sheet for the colour removed by GT-Al Np's.

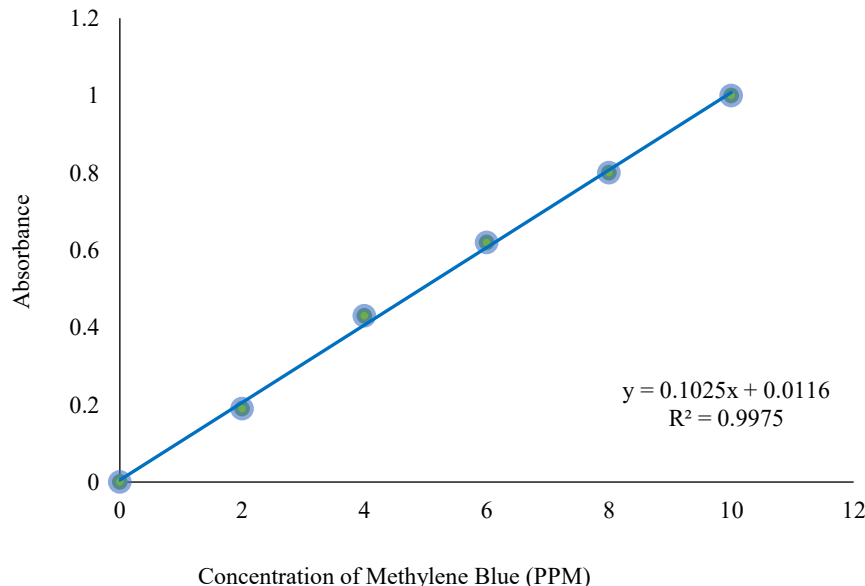


Figure 3. Concentration of methylene blue vs absorbance.

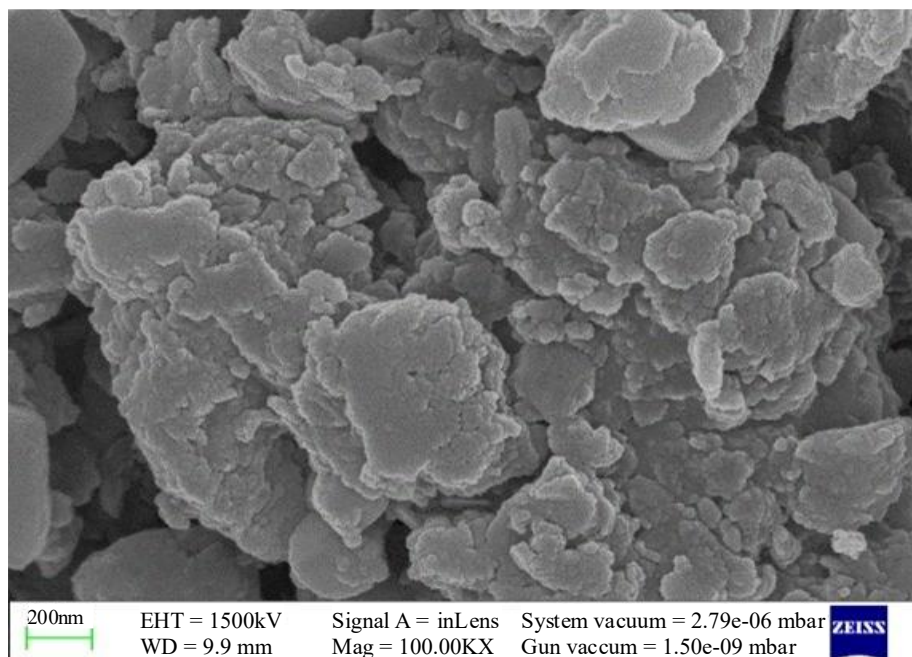


Figure 4. SEM analysis of GT-Al Np's.

Energy Dispersive X-ray (EDX)

Using X-ray emission, Energy Dispersive X-ray Analysis (EDX) determines the elemental composition. It is a non-destructive method that permits analysis of the material in its original state with little sample preparation. The elements found in Green Tea – Aluminum nanoparticles are shown by peaks in the EDX spectra displayed in Figure 5. For industrial forensic investigations and contaminant detection, EDX works well.

Thermogravimetric Analysis (TGA)

Thermogravimetric Analysis (TGA) tracks the weight variation of a sample as it is heated in a furnace, with the weight recorded by an external analytical balance. As shown in Figure 6, TGA is widely used in pharmaceutical, food, environmental, and petrochemical fields to assess thermal stability and composition of materials.

Effect of Various Parameters

Dye concentration, contact time, pH, dosage, and temperature were all modified in equilibrium testing. After adding GT-Al NPs to dye solutions, samples were periodically examined with a UV spectrophotometer.

RESULTS AND DISCUSSION

Fourier Transform Infrared Spectroscopy (FTIR)

Functional groups on GT-Al NPs were identified by FTIR analysis, which detected infrared absorption at specific frequencies. By comparing the absorbance spectrum, which is a molecular fingerprint, with reference spectra, the chemical structure and surface groups of the adsorbent were determined (Figure 7).

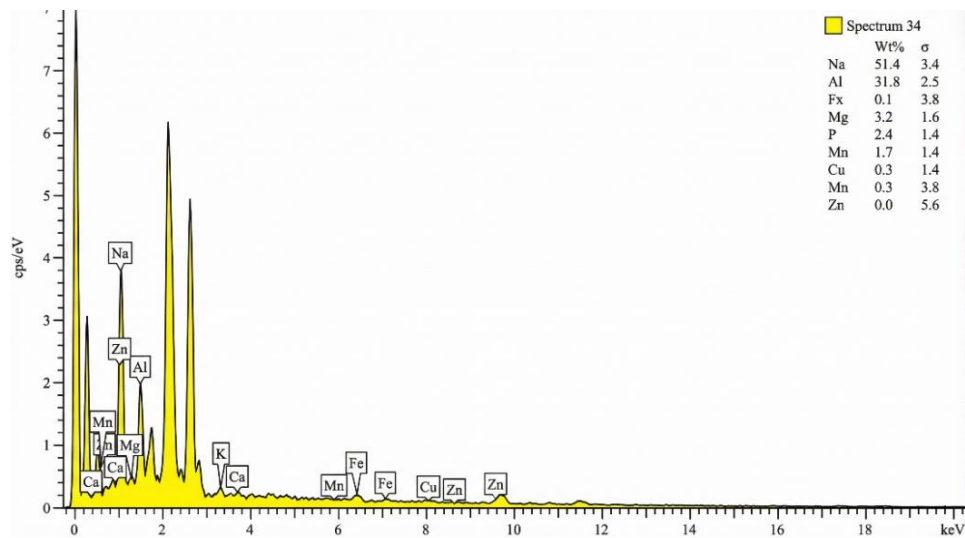


Figure 5. EDX analysis of GT-Al Np's.

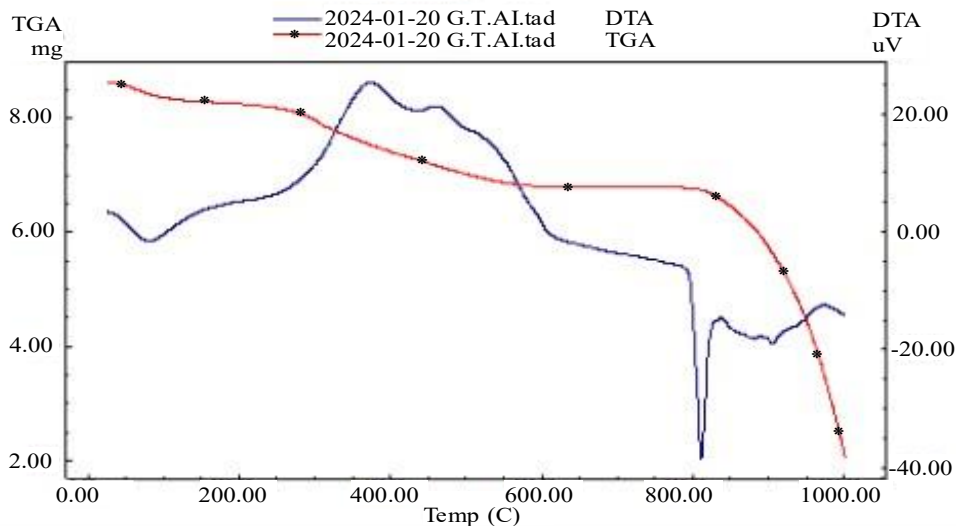
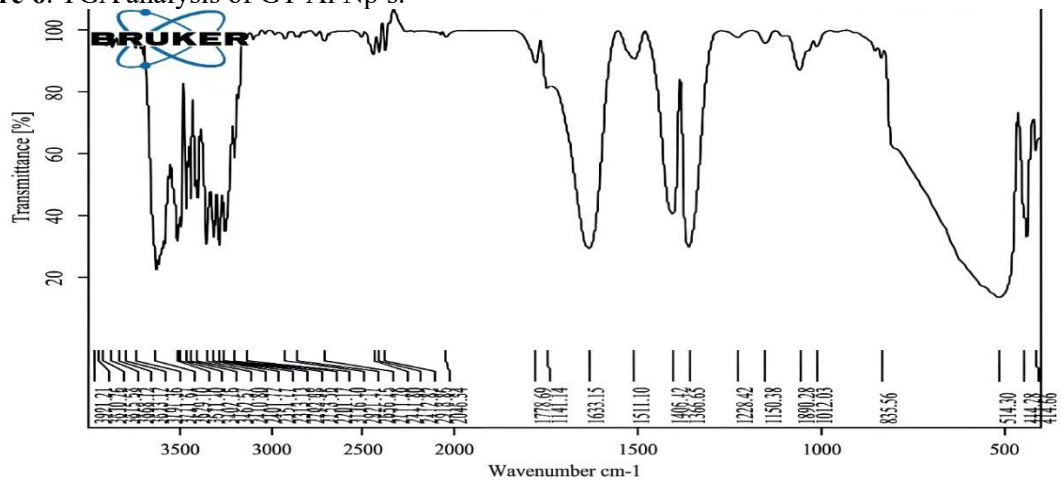
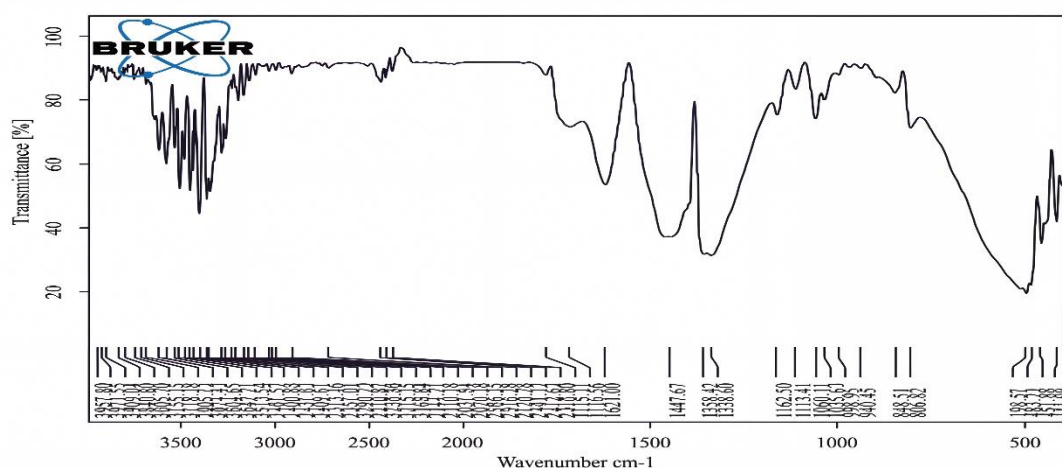


Figure 6. TGA analysis of GT-Al Np's.





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Figure 7 (a, b). FTIR analysis of GT-Al Np's before and after adsorption.

Effect of Various Parameters

Temperature, dye concentration, adsorbent dosage, pH, and contact time were all changed in equilibrium investigations. In a 250 mL flask, 100 mL of a 10 ppm Methylene Blue solution was mixed with green tea-aluminum nanoparticles and agitated. Periodically, samples were taken, and the dye concentration was measured using UV-spectrophotometry (Table 2).

Effect of Time

Using 100 mL of room-temperature, 10 ppm Methylene Blue at 5-minute intervals for 30 minutes, adsorption was investigated. Following centrifugation and filtering, UV-Vis was used to evaluate the samples. Figure 8 shows that equilibrium was reached after 20 minutes.

Effect of Adsorbent Dosage

The adsorbent dosage effect was studied by treating 100 mL of 10 ppm Methylene Blue with 0.02–0.12 g/L GT-Al NPs at room temperature and a 20 minute contact time. Dye removal efficiency increased with dosage up to 0.1 g/L, which was determined as the ideal dosage (Figure 9).

Table 2. Experimental parameters.

Variables	Symbols	Units	Range	
			From	To
Contact time	T	Min	5	30
Adsorbent dosage	W	g/L	2	12
pH	pH	--	5	9
Initial concentration	Co	Ppm	10	100
Temperature	T	K	303	318

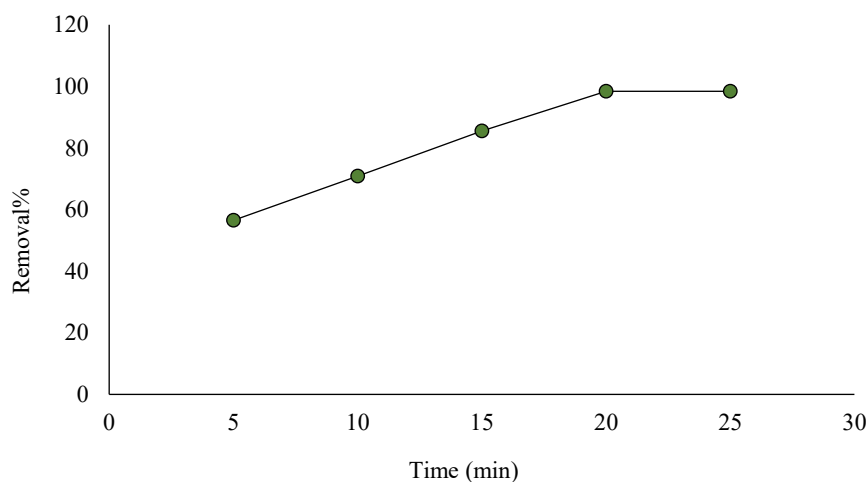


Figure 8. Effect of time on the removal of MB dye by GT-Al Np's.

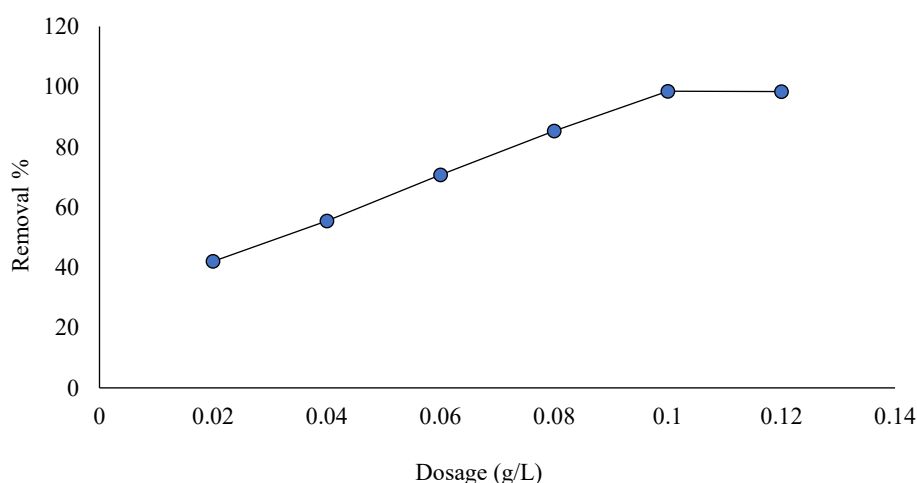


Figure 9. Effect of dosage on the removal of MB dye by GT-Al Np's.

Effect of pH

The impact of pH (5–9) on Methylene Blue adsorption was investigated using 0.1 g/L GT-AlNPs in 100 mL of 10 ppm dye for 20 minutes at 150 rpm. Using 1N HCl/NaOH, the pH was adjusted. The highest adsorption was seen at pH 8 by UV-Vis measurement (Figure 10).

Effect of Initial Dye Concentration

Using 100 mL solutions at room temperature with pH 8, 0.1 g/L adsorbent, and a 20-minute contact duration, the impact of the initial Methylene Blue concentration (10–100 ppm) on adsorption was investigated. Centrifugation, filtration, and UV-Vis analysis were used to measure the amount of residual dye. At 10 ppm, the adsorption efficiency peaked (Figure 11).

Effect of Temperature

0.1 g/L GT-Al NPs in 100 mL of 10 ppm dye at pH 8 with continuous agitation and a 20 minute contact duration were employed to examine the temperature effect (303–318 K) on Methylene Blue adsorption. 1N HCl or NaOH was used to alter the pH. The samples underwent centrifugation, filtration, and UV-Vis analysis. At 313 K, uptake peaked (Figure 12).

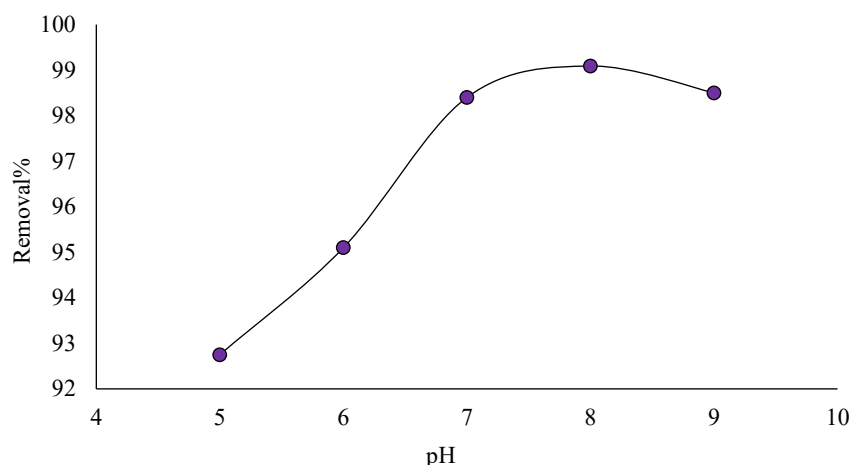


Figure 10. Effect of pH on the removal of MB dye by GT-Al Np's.

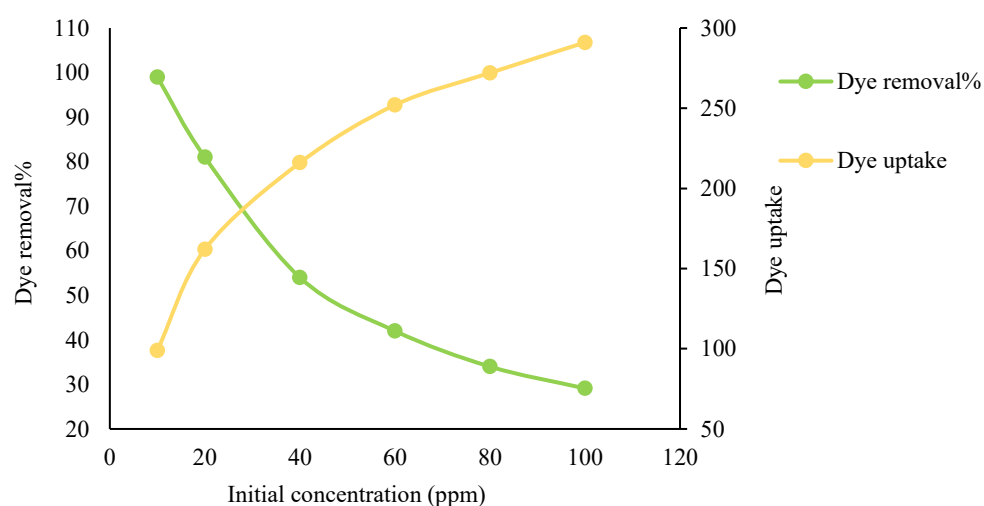


Figure 11. Effect of initial dye concentration for the removal of MB dye by GT-Al Np's.

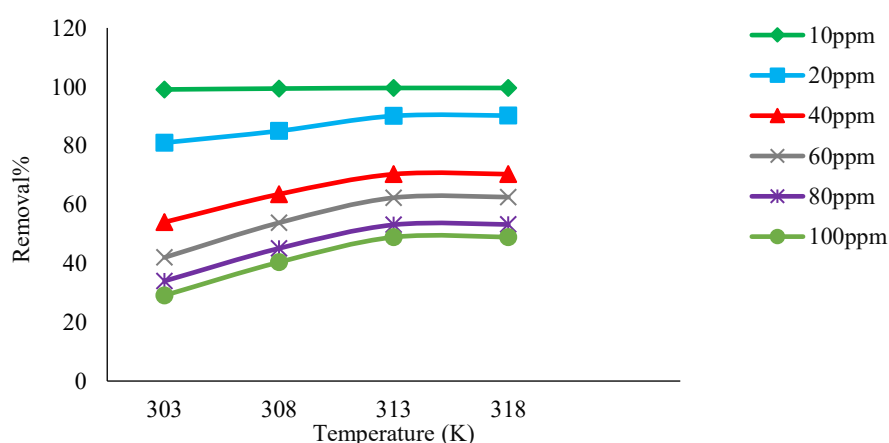


Figure 12. Effect of temperature on the removal of MB dye by GT-Al Np's.

Equilibrium Studies

Selecting the appropriate adsorption isotherm model is crucial for adsorbent design. At constant pH and temperature, isotherms display the capacity, efficiency, and interactions between the adsorbent and the pollutant. In this study, 0.1 g/L GT-Al NPs, 10 ppm Methylene Blue, pH 8, 313 K, and a 20-minute contact time were used.

Langmuir Isotherm

As per the Langmuir adsorption theory, adsorption takes place at specific homogeneous sites on the adsorbent surface. Once occupied, these sites can contain a single dye molecule and stop further adsorption. This is the linear form of the Langmuir isotherm:

$$\frac{C_e}{q_e} = \frac{1}{bq_{max}} + \frac{1}{q_{max}}C_e \quad (7)$$

At 313 K, the Langmuir plot (C_e/q_e vs. C_e) for Methylene Blue adsorption showed a strong fit ($R^2 = 0.9906$), with a Langmuir constant (K_L) of 0.283 L/mg and maximum capacity (q_{max}) of 294.1 mg/g, indicating favorable adsorption as seen in Figure 13 onto GT-Al NPs following Langmuir isotherm behavior.

Freundlich Adsorption

Assuming heterogeneous surface adsorption, Freundlich isotherm shows that adsorption energy falls exponentially with increasing surface coverage, with high-energy sites being filled first. This is how the model is expressed:

$$q_e = K_f C_e^{1/n} \quad (8)$$

The Freundlich isotherm can be represented as a straight line.

$$\log q_e = \log K_f + \frac{1}{n} \ln C_e \quad (9)$$

With a correlation coefficient of 0.9896, the Freundlich constants for Methylene Blue were calculated from the plot of $\log q_e$ vs $\log C_e$ (from Eq. (9)) at 313 K (Figure 14): $K_f = 139.25 \text{ (mg/g)(L/g)}^n$ and $1/n = 0.1639$. According to these findings, multilayer adsorption on a heterogeneous surface is confirmed by a strong fit to the Freundlich model.

Temkin Isotherm

Due to adsorbate-adsorbent interactions, this model assumes uniform binding energies with a maximum, where adsorption heat reduces linearly as coverage increases. The Temkin isotherm equation is expressed as

$$q_e = \frac{RT}{b} \ln (A_T C_e) \quad (10)$$

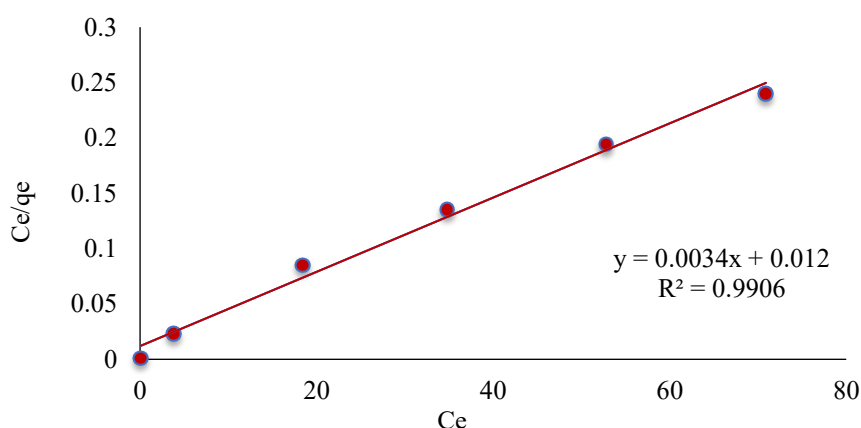


Figure 13. C_e versus C_e/q_e for MB.

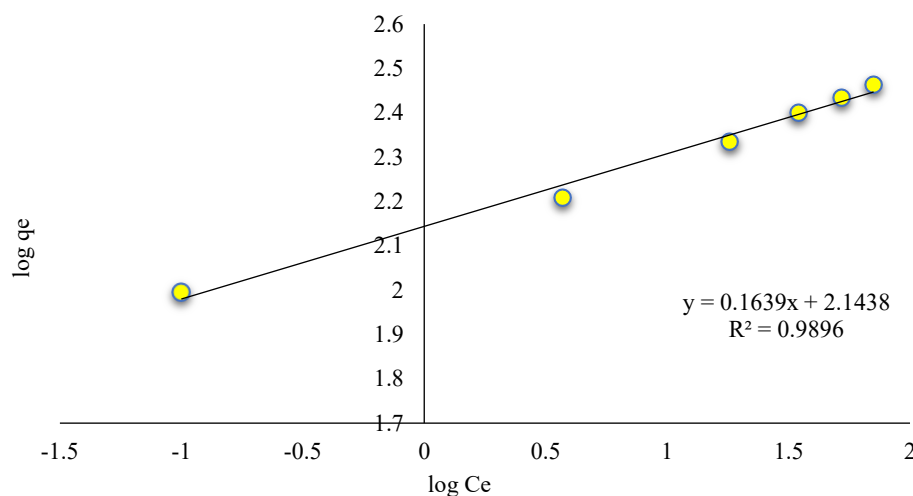


Figure 14. $\ln C_e$ versus $\ln q_e$ for MB.

The linear form of the Temkin equation is

$$q_e = B_T \ln A_T + B_T \ln C \quad (11)$$

By using equation (11), the values of the isotherm constants A_T and b can be determined as 5.199 and 28.585 through plotting q_e against $\ln C_e$. Figure 15 displays the q_e plotted against the natural logarithm of C_e for the adsorption of Methylene Blue dye onto the adsorbent, including the parameter values. $R^2 = 0.9383$.

Kinetic Studies

Adsorbate retention at the interface is impacted by the solute adsorption speed, which is described by adsorption kinetics. This process is frequently studied using pseudo-first and pseudo-second order models.

First-Order Kinetic Model

Adsorption is described by the pseudo-first-order model, which postulates that the rate is proportional to the number of vacant sites. It is said as follows:

$$\frac{dq}{dt} = k_1 (q_e - q) \quad (12)$$

The linear form is obtained by integrating under the boundary constraints $q=0$ at $t=0$ and $q=q$ at $t=t$.

$$\ln(q_e - q) = -K_1 t + \ln q_e \quad (13)$$

q is adsorption at time t , K_1 is the rate constant (min^{-1}), and q_e is the equilibrium capacity (mg/g). Plotting $\ln(q_e - q)$ vs t under ideal conditions (20 min, 0.1 g/L, 40 °C, pH 8, 10 ppm) showed a moderate fit with $R^2 = 0.8678$, as shown in Figure 16.

Second-order kinetic model

According to the pseudo-second-order kinetic model, adsorption comes after second-order chemisorption. It is said as follows:

$$\frac{dq}{dt} = k_2 (q_e - q)^2 \quad (14)$$

$$\frac{dq}{(q_e - q)^2} = K_2 dt \quad (15)$$

The linear form is obtained by rearranging and integrating Eq. (15):

$$\frac{t}{q} = \left(\frac{1}{q_e}\right)t + \left(\frac{1}{K_2 q_e^2}\right) \quad (16)$$

q and q_e represent dye adsorbed at time t and equilibrium (mg/g), respectively; t is contact time (min), and K_2 is the rate constant (g/mg·min). Plotting t/q versus t under ideal conditions (20 min, 0.1 g/L, 40 °C, pH 8, 10 ppm) showed a high model fit ($R^2 = 0.9927$), with slope and intercept used to calculate K_2 (Figure 17).

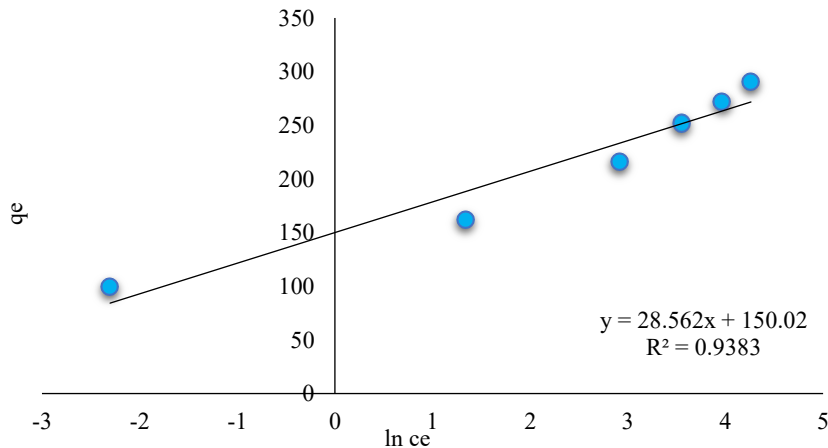


Figure 15. q_e versus $\ln C_e$ for MB.

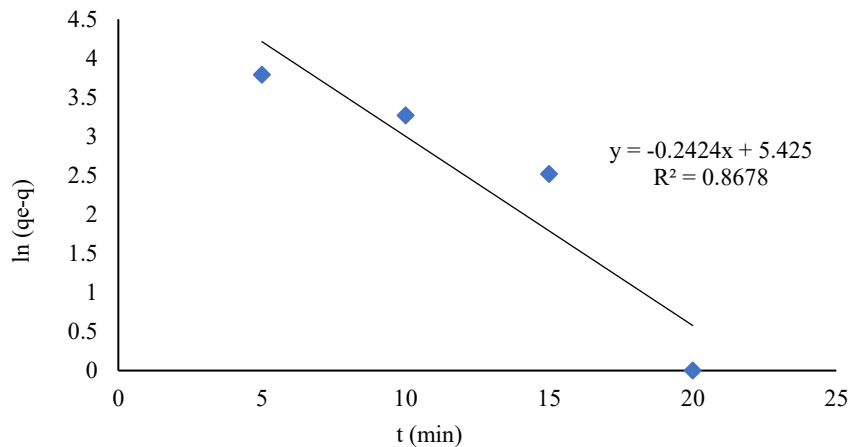


Figure 16. Time versus $\ln(q_e - q)$ for MB.

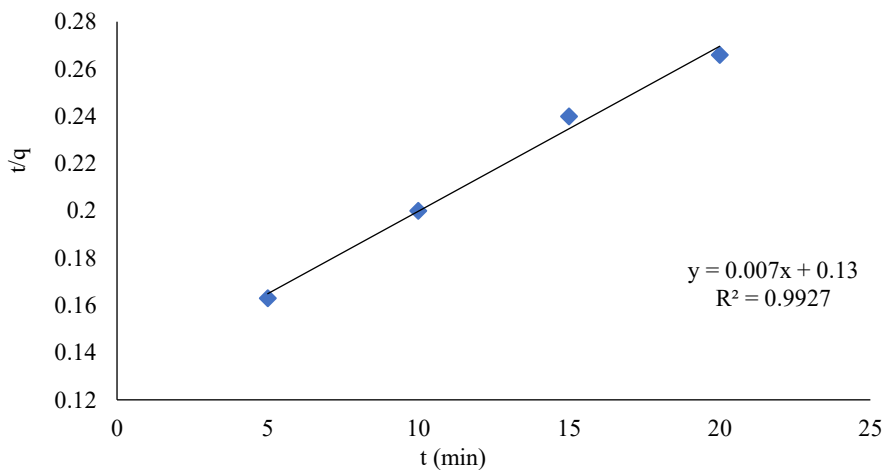


Figure 17. Time versus $\frac{t}{q}$ for MB.

Thermodynamic Studies

The thermodynamic parameters ΔG° , ΔH° , and ΔS° were calculated from K_c using the Van Hoff plot ($\ln K_c$ vs. $1/T$). In tests, spontaneous, endothermic adsorption was shown at pH 8, 40°C, 0.1 g/L adsorbent, 10 ppm dye, and a contact time of 20 minutes (303–318 K). Table 3 and Figure 18 show that the activation energy (E_a) was 862.71 kJ/mol.

Table 3. Thermodynamic characteristics for the adsorption of MB and Green Tea-Aluminum Nanoparticles (GT-Al NPs) adsorbent.

Temperature (K)	ln K	ΔG° (J/mol)	ΔH° (KJ/mol)	ΔS° (J/mol.K)	Van't Hoff Equation
303	6.1	-15366.7	.172	4.365	$\ln(K) = -862.71(1/T) + 8.9446$
308	6.18	-15825.2			
313	6.19	-16108.1			
318	6.24	-16497.6			

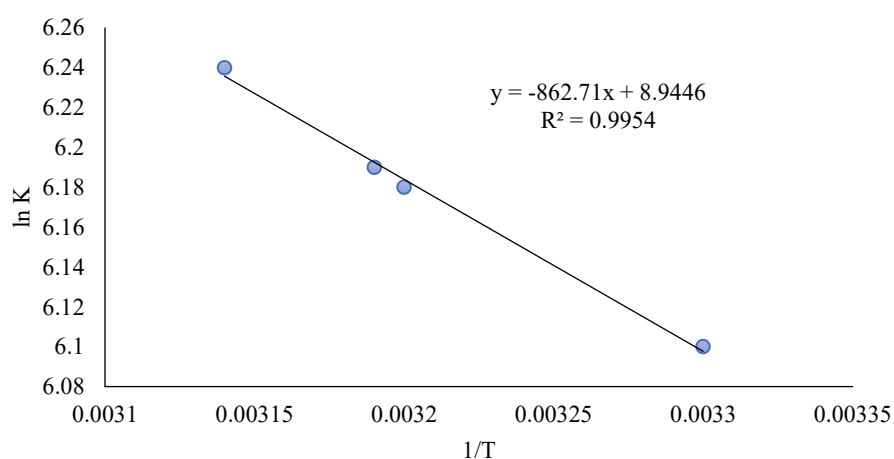


Figure 18. $1/T$ versus $\ln K$ for MB.

CONCLUSIONS

Eco-friendly green tea – aluminum nanoparticles (GT-Al NPs) were created to remove Methylene Blue. SEM, FTIR, EDX, and TGA characterization revealed a size of about 200 nm and validated the elemental composition and functional groups. High GT-Al NP performance was demonstrated by the maximum removal efficiency of 99.6% under ideal conditions, which included 20 min contact, 0.1 g/L dose, pH 8, 10 ppm dye, and 313K.

Kinetic analysis showed that the pseudo-second-order model suited the data, with an adsorption capacity (q_e) of 88.8 mg/g and R^2 of 0.9927. Equilibrium data were examined using Langmuir, Freundlich, and Temkin isotherms. The Freundlich model provided the best fit ($K_f = 139.25$, $R^2 = 0.9896$), with the highest capacity of 294.1 mg/g. Temkin constants ($AT = 5.199$) further supported the adsorption tendency. Thermodynamic analysis indicated an endothermic process with positive entropy ($\Delta S^\circ = 74.365$ J/mol·K) and enthalpy ($\Delta H^\circ = 7.172$ kJ/mol), suggesting increased system randomness without structural changes in the adsorbent.

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