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HARNESSING BIOGENIC ZINC OXIDE NANOPARTICLES FOR ECO-FRIENDLY ADSORPTIVE DETOXIFICATION OF METHYLENE BLUE DYE

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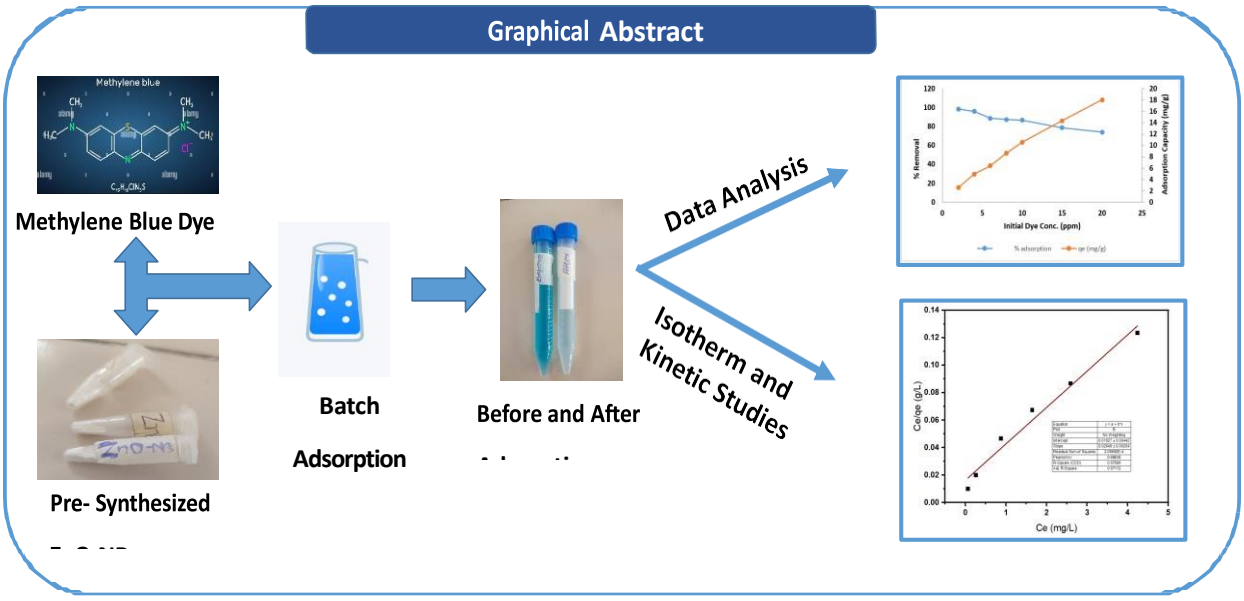
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Abstract

Excessive release of synthetic dye into the environment has been reported by researchers and poses a major problem for water quality; thus, it is important to investigate inexpensive and less harmful strategies for treatment. In this study, pre-synthesized ZnO nanoparticles prepared by plant-mediated green method were used as an adsorbent to remove methylene blue (MB) from aqueous medium. Batch Experiments showed that the extent of methylene blue removal varied significantly with pH (3-12), dye content (5-30 ppm), contact duration (0-40 min), and adsorbent dosage (5-20 mg). A fast uptake in the beginning was followed by a steady region when the surface became saturated. The kinetic plots fitted best to the pseudo-second order model ($R^2 \approx 0.99$), whereby implies that the rate is mainly controlled by surface interaction rather than mere diffusion. The precision of the kinetic model was additionally evaluated through statistical parameters such as mean squared error (MSE) and root mean squared error (RMSE), which demonstrated the superior correlation and fitting accuracy of the PSO model. Among the equilibrium models tested, the Freundlich isotherm described the empirical findings more suitably, pointing towards a heterogeneous adsorption surface in contrast to a monolayer. Under the optimum conditions, the ZnO nanoparticles removed MB efficiently, suggesting that such leaf-extract-derived nanomaterials can serve as a simple and greener option for dye-laden wastewater.



1. INTRODUCTION:

In recent years, because of the severe ecological effects and ensuing financial burden, the problem of contaminated water has attracted a lot of attention globally. One of the main pollutants found in water bodies are artificial dye chemicals, which inhibit light penetration in aquatic environments, restricting photosynthetic activity and hindering the growth of aquatic life [1,2]. In reality, one of the predominant contributors is the textile industry, which uses over ten thousand different synthetic dyes annually and colors most of them in water. Therefore, wastewater containing heavy metals and harmful chemicals is released into the environment during the residual dye bath is disposed of, which is why these effluents are regarded as a major hazard [3,4].

Methylene blue is extensively utilized to dye fabrics such as cotton, silk and at times wood. Ingestion leads to symptoms such as irritation of the throat, and it may also cause nausea, vomiting, sweating, and abrupt chills; eye exposure to this dye can cause severe eye pain which may lead to permanent loss of vision [5]. Therefore, wastewater contaminated with MB pollution should be monitored and effectively treated prior to being discharged into the environment. However, most conventional processes for wastewater treatment cannot effectively remove these compounds.

Coagulation, membrane filtration, oxidation, and biological techniques are traditional methods used for dye removal, which can be highly expensive in operation, have poor decolorization potential, and cause excessive sludge generation. Adsorption has been regarded as a relatively budget-friendly, rapid, and highly efficient technology with high sorption capacity, which does not generate secondary pollution in comparison to other classical methods [6].

Although a lot of the potential that nanomaterials (in the range 1–100 nm) can offer is yet to be exploited, this field has witnessed various technological developments at an accelerated pace. These novel materials possess outstanding attributes, including high adsorption and photocatalytic activity in the eradication of wastewater contaminants resulting from their unique physicochemical features, large surface area (SA), and quantum size effects [7, 8]. Zinc oxide is known to be the best choice of metal oxide nanoparticles because of less cost in preparation, easy synthesis, and more surface reactivity. It is very effective to treat water severely contaminated with organic or inorganic pollutants [9].

Metal oxide nanoparticles, including nanomaterials themselves and metal oxides of diverse valencies, have excellent adsorption capability resulting from their large specific surface areas, high reactivity with solvents in

the environment, and easy control of surface chemistry. Although these nanoparticles can be synthesized by physical as well as chemical approaches, the use of toxic reagents like sodium borohydride and hydrazine raises concerns on safety and environmental issues, urging the need for cleaner, more sustainable, environmentally benign alternative methods for the synthesis of bio-nano-materials [10].

Non-toxic and eco-friendly characteristics of green synthesis are a healthier technique for the production of nanoparticles than traditional chemical methods. Plant extracts that are high in phytochemicals like phenolics, flavonoids, and terpenoids act as both stabilizing as well as reducing agents at the same time, allowing for controlled growth and improved stability of the final nanoparticles [11]. For Example, Debnath and Mondal [12] reported the biosynthesis of spherical ZnO nanoparticles (~100 nm) from *Hibiscus rosa-sinensis* leaves, which removed Congo red with 95.55% efficiency. In a study by Ghosh *et al.* [13], *Justicia adhatoda*-mediated magnetic iron oxide nanoparticles were developed and successfully applied to eradicate methylene blue from polluted water.

In the current work, pre-synthesized zinc oxide (ZnO) nanoparticles obtained employing a biogenic synthesis route using leaf extracts of *Syzygium cumini* and *Mangifera indica* (1:1 ratio) were employed for the adsorption of methylene blue dye from aqueous medium. Structural and morphological characterization of these ZnO nanoparticles was previously confirmed by FTIR, XRD, SEM, BET, and UV–Vis analyses, which verified the formation of hexagonal wurtzite-phase ZnO having spherical morphology and a mean particle size of approximately 41 nm [14]. Based on current knowledge, this is the first study to employ these ZnO nanoparticles obtained from this dual plant-extract system to eliminate a cationic dye (methylene blue). Batch mode adsorption experiments were executed to determine the impact of multiple operational parameters, including initial dye concentration, pH, contact duration, and dose of adsorbent, on dye removal efficiency. In addition, the adsorption mechanisms and equilibrium characteristics were described by kinetic and adsorption isotherm models. This investigation suggests the application of bio-synthesized ZnO Nanoparticles to industrial dye removal and environmental protection as an environmentally friendly and sustainable technology for wastewater treatment.



Fig. 1. Production of nanoparticles and elimination of dyes [15]

2. Experimental Work

2.1. Materials and Chemicals

Every reagent utilized in this study was of analytical quality (Sigma-Aldrich, USA) and used without additional purification. The chemicals included Methylene Blue dye ($C_{16}H_{18}ClN_3S$), 0.1M solutions of NaOH and HCl. Methylene Blue (analytical grade, molecular weight 319.8 g/mol) was utilized as a model dye contaminant in

this experiment. ZnO nanoparticles employed as adsorbents in this study were sourced from an earlier publication with the same research group [14], where they had been synthesized via a green route utilizing *Mangifera indica* and *Syzygium cumini* leaf extracts.

2.2. Stock Solution Preparation

A precisely weighed amount of MB was dissolved using distilled water as a solvent, to make a stock solution with a concentration of 1 g/L (1000 ppm). The resulting solution has been subsequently diluted tenfold to attain a 100 ppm working solution. A series of standard Methylene blue dye solutions with levels ranging from 2, 5, 10, 15, 20, 25, and 30 ppm was prepared to construct the calibration curve. A UV–Vis spectrophotometer (Shimadzu UV-1800, Japan) has been employed to assess absorbance at the maximum wavelength (λmax = 664 nm) intended to
determine the dye concentrations. To ascertain the relationship for quantitative analysis, a calibration curve has been generated between absorbance and concentration.

2.3. Batch Experiment

In the current work, ZnO nanoparticles previously biosynthesized from *Mangifera indica* and *Syzygium cumini* leaf extracts had been employed in batch adsorption trials owing to their advantageous structural traits and excellent adsorptive capability. The Erlenmeyer flask (100 mL) with 20 mL aliquot of dye solution was used throughout the tests. The initial pH has been changed while being tracked using a pH paper by utilizing HCl and NaOH (0.1M). In order to maintain a consistent suspension of nanoparticles, adsorption studies were conducted in an orbital shaker at room temperature (about 30 ± 2 °C) while stirring continuously. The study assessed how the adsorption performance was affected by a number of operational parameters, particularly pH conditions (3-12), exposure duration (0-40), dose of adsorbent (5-20 mg), and dye content (5-30 ppm). The nanoparticles were removed by centrifugation at ~3000 rpm and samples were taken after the given contact time for 40 min. A UV-Vis spectrophotometer was assessed to quantify the dye remaining in the supernatant at maximum absorption wavelength (664 nm) for MB dye, each measurement was made twice under the same circumstances. The adsorption of dye was estimated with the help of the following equation:

$$\% \text{ Removal} = (C_o - C_e)/C_o * 100 \quad (1)$$

The concentration at equilibrium is Ce, and the initial concentration is Co. The adsorption capacity of the dye, qe, was obtained using the following formula. In this equation, V stands for the volume used in liters, and W for the adsorbent's total mass in grams

$$q_e = (C_o - C_e / \text{M}) * \text{V} \quad (2)$$

2.4. Adsorption Isotherm study

Adsorption isotherms are theoretically crucial approaches for evaluating the equilibrium properties of adsorption processes that take place across the liquid-solid interface. Assuming that adsorption takes place uniformly at particular, similar spots on the surface, the Langmuir adsorption isotherm is employed to assess the adsorption effectiveness of adsorbents. It talks about monolayer
adsorption, in which the adsorbate has an identical affinity for each site. The following is a mathematical expression for the Langmuir isotherm:

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \quad (3)$$

Where, KL is the Langmuir constant that indicates the adsorbent–adsorbate affinity, qm and KL are obtained by creating a plot of Ce/qe versus Ce [16]. The correlation coefficient (R^2) obtained from the plotted data can be used to assess the model's appropriateness. Adsorptive interaction on the adsorbent surface is well explained by the Freundlich isotherm, which depicts multilayer adsorption. The following is a numeric expression for the Freundlich isotherm:

$$\log q_e = \log k_F + \frac{1}{n}\log C_e \qquad (4)$$

Here, the Freundlich constants KF and n stand for the adsorption capacity and adsorption intensity, respectively, which reflect surface heterogeneity [17]. Adsorption favorability is indicated by the metric 1/n; values above 1 denote unfavorable behavior, whereas values falling within 0-1 are considered favorable adsorption. The linearity and adsorption appropriateness of the model are assessed using a plot of log qe vs log Ce.

2.5. Kinetic Analysis of Adsorption

To elucidate the kinetics and interaction route of dye removal by the pre-synthesized ZnO, pseudo first order (PFO) and pseudo second order (PSO) models were applied to estimate the equilibrium time and kinetic constants for MB adsorption. The PFO is standardly applied to interpret solute adsorption from solution, and its better fit typically implies that the mechanism operates under physisorption. According to the PSO model, the uptake rate is influenced by bulk adsorbate concentration and the availability of active surface sites. A superior PSO fit ($R^2 \approx 1$) highlights that chemisorption predominates over the PFO mechanism. The linearized integral formulations of these kinetic models are given in equations below:

$$\ln(q_e - q_t) = lnq_e - K_1 t \qquad (5)$$

$$\frac{t}{q_e} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} \qquad (6)$$

The dye uptake at time t is represented by qt (mg g^{-1}). The PFO rate constant, k_1 (min^{-1}), is derived from the slope of the plot of log (qe − qt) vs t. Additionally, k_2 is the PSO rate constant, which is found by calculating the slope of the t/qt vs t linear plot. The mean squared error (MSE) and root mean squared error (RMSE) were determined using the following equations to assess the accuracy and reliability of the model fit.

$$MSE = \frac{1}{n}\sum_{i=1}^{n}(q_{e,exp,i} - q_{e,cal,i})\text{^}2 \qquad (7)$$

$$RMSE = \sqrt{\frac{1}{n}\sum_{i=1}^{n}(q_{e,exp,i} - q_{e,cal,i})\text{^}2} \qquad (8)$$

3. Results and Discussion

3.1. Morphological Features of ZnO Nanoparticles Utilized for Adsorption

The morphological, structural, and chemical profile of the ZnO nanoparticles utilized in this study were previously described by Inam et al [14]. The nanoparticles were manufactured through a green route via a combined aqueous extract of *Syzygium cumini* and *Mangifera indica* leaves. The production of crystalline ZnO with a hexagonal wurtzite structure was validated by XRD examination (JCPDS 00-005-0664), while FTIR spectra verified characteristic Zn–O stretching vibrations, indicating successful nanoparticle formation. UV–Vis absorption showed a strong band around 378 nm, and SEM imaging revealed nearly spherical particles having an average size of
~41 nm, consistent with DLS and BET analyses. These well-characterized ZnO nanoparticles were therefore employed in the present work as efficient adsorbents regarding the eradication of MB dye from a water-based solution.

3.2. Adsorption Parameters and Calibration Curve

A calibration curve (Figure 2) was established utilizing a UV–Vis spectrophotometer by evaluating the absorbance of methylene blue standard solutions at known concentrations ranging from 2 to 25 ppm. The recorded absorbance values were plotted to generate a linear relationship between
concentration and absorbance. The concentration of MB dye in each aqueous sample has been assessed spectrophotometrically at its maximum absorption wavelength (λ_{max}) of 664 nm.

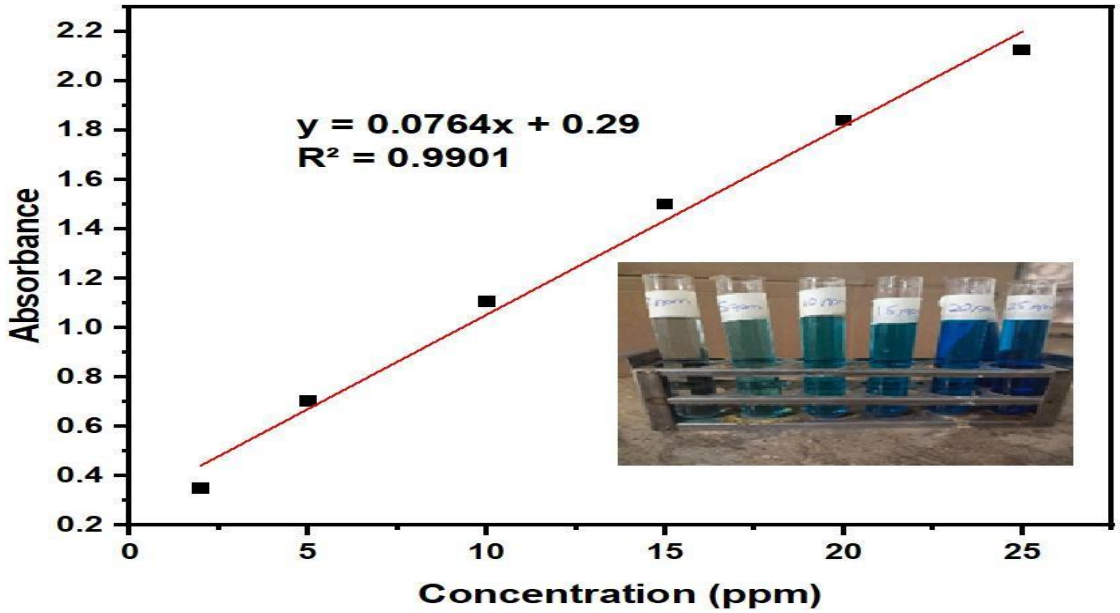


Fig. 2 Calibration Curve of MB at 664 nm

3.2.1. Exploring pH Dynamics and Mechanism of Adsorption

The percentage of MB dye removed via ZnO nanoparticles was predominantly influenced by the pH of the solution. With an initial MB dose of 5 ppm and a constant temperature of 30 °C, experiments were executed across pH values from 3-12 with the adsorbent dosage of 15 mg. The pre-synthesized ZnO nanoparticles exhibited the point of zero charge at 7.6, meaning that the pH
> 7.6 the surface carries a negative charge, and at pH < 7.6 the surface is positively charged. Methylene blue is a cationic species (MB^+) in aqueous media; reduced adsorption results from electrostatic repulsion in acidic environments as a result of competitive interaction among H^+ ions and MB^+ for adsorption sites. Contrarily, the surface of ZnO bears a negative charge at alkaline conditions, usually between 8 and 11, which favors electrostatic interaction with MB^+ ions and increases dye uptake. A higher density of OH^- groups builds up on the ZnO surface as pH rises, which promotes chemical precipitation and implies that the adsorption of MB is affected by hydrogen bonding amongst the dye's cationic (=N^+) moieties and ZnO's oxygen functions [18]. As
the pH elevated from 3 to 8, the decolorization of methylene blue increased from 6% to 98%, which is similar to the reported results [19]. This enhanced interaction improves removal efficiency. Changes in the surface charge of ZnO nanoparticles and structural alterations in MB molecules are responsible for the variance in removal effectiveness over the studied pH range, demonstrating that the adsorbent's adsorption ability is highly pH-dependent.

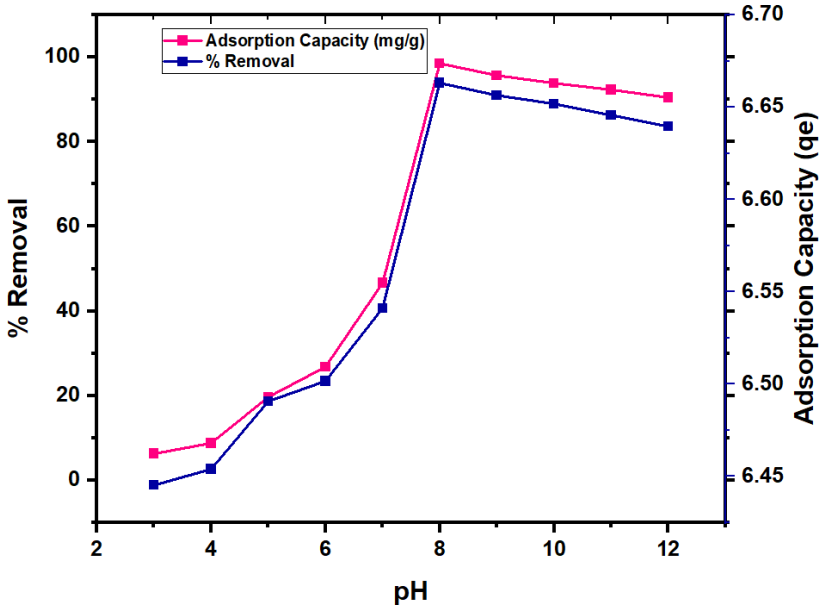


Fig.3 Optimization of pH

3.2.2. Influence of Adsorbent Dosage

The amount of adsorbent is a pivot factor in determining the adsorption process, because it directly impacts the rate and effectiveness of adsorption. Furthermore, by adjusting the dose from 2 to 20 mg of a 5 ppm MB solution, contact time of 40 min at pH 8, the impact of adsorbent dosage on methylene blue eradication by ZnO nanoparticles was examined. As the dosage of ZnO nanoparticles elevated from 2-20 mg, the adsorption efficiency escalated from 31% to 98% (Figure 4). Greater homogeneity of ZnO nanoparticles in the aqueous media may be the cause of the increased removal effectiveness at lower adsorbent quantities. As a result, more active surface sites can be exposed, thereby rendering it simpler for dye molecules to reach more adsorption

sites [20]. Resulting from diminished concentration profile or driving force between the solution and the adsorbent surface, the equilibrium adsorption capacity (qe, mg/g) in the same context significantly decreased as more adsorbent was introduced. Because there are more adsorption sites accessible at greater dosages, there is aggregation and less dye uptake, which causes this reduction [21].

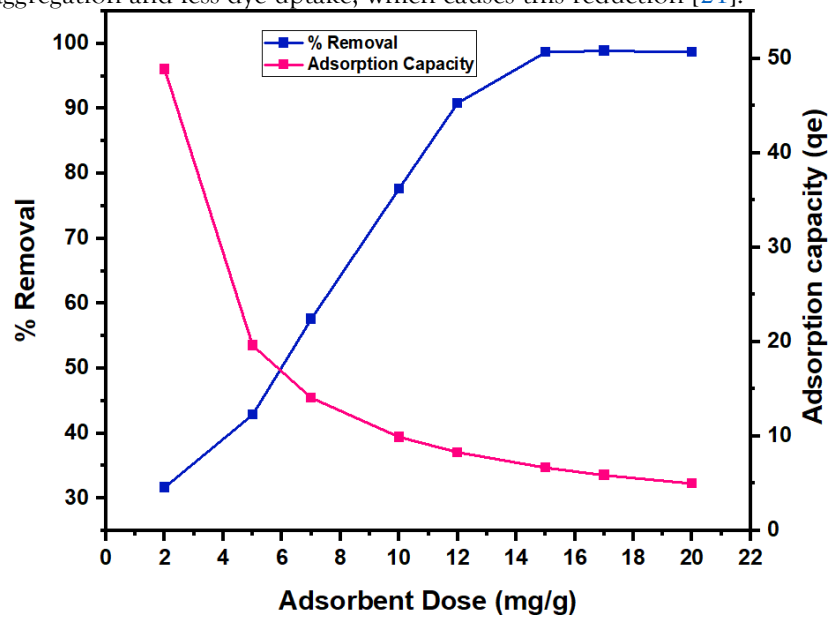


Fig. 4 Impact of Adsorbent Dose

3.3.3. Impact of Dye Content

The initial concentration of the adsorbate, which acts as the catalyst for the transfer of dye molecules onto the surface of the adsorbent, has a decisive impact on the adsorption efficiency. The adsorption of MB onto ZnO nanoparticles was examined across the range of concentrations (5–30 mg/L) at 30 °C with optimal pH and adsorbent dosage in order to evaluate this impact. Until equilibrium conditions were reached, the dye concentration in each solution was continuously monitored. Because of the increased mass transfer driving force prior to achieving adsorption–desorption equilibrium and the greater availability of MB molecules close to the surface of the adsorbent, the adsorption capacity (qe) escalated as the initial dye concentration increased. With higher initial MB concentrations, as illustrated in Figure 5, the adsorption capacity of ZnO nanoparticles rose from 6.57 mg/g to 34.35 mg/g, while the percentage removal reduced from 98% to 81%, most likely as a result of the restricted availability of active adsorption sites at higher dye levels.

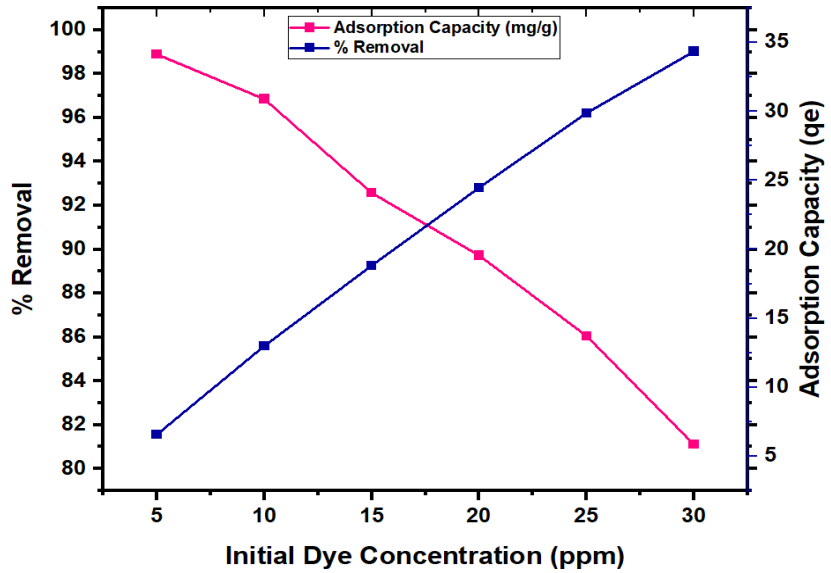


Fig. 5 Impact of Initial Dye Concentration

3.2.4. Optimization of Contact Time

Adsorption increased substantially in the initial 20 minutes, leveled off, and attained equilibrium at 30 minutes, as seen in Figure 6. After this, no more removal took place, suggesting that the available adsorption sites were saturated and that there was no improvement with longer contact times. The percentage removal peaked at the ideal contact time at an initial MB concentration of 5 ppm; after this, no further adsorption was seen, indicating that the available active sites had been saturated and that longer contact times had no effect on uptake. The simple colonization of several empty sites on the ZnO-NPs surface by MB molecules is responsible for the quick initial absorption, but later interactions amongst the dye and the adsorbent prevent additional adsorption onto the remaining sites.

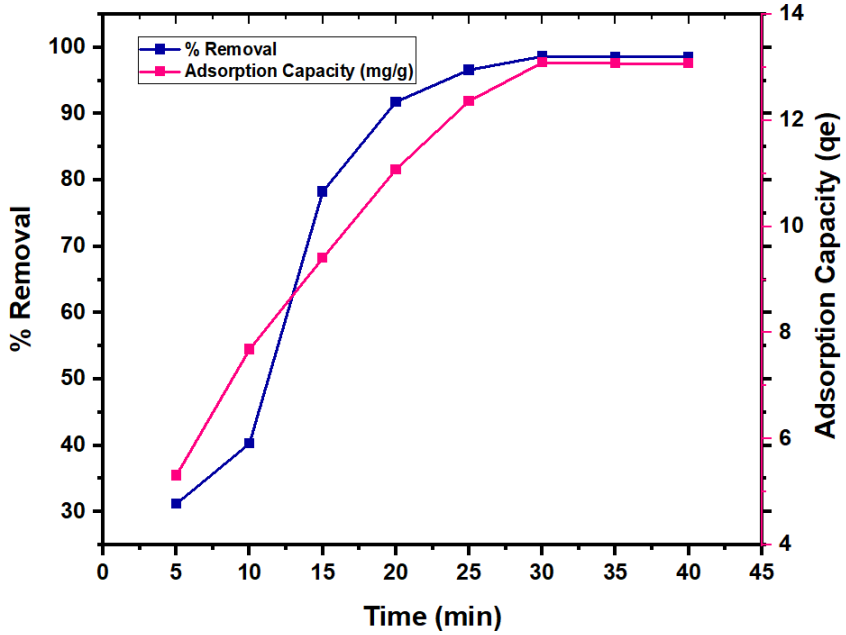


Fig. 6 Influence of Contact Time

3.3. Adsorption Isotherms

The results of equilibrium adsorption provide significant insights into the mechanistic aspects that govern the interaction between ZnO NPs and methylene blue (MB). Langmuir and the Freundlich isotherms model were implemented to fit the adsorption outcomes based on the parameters evaluated, as well as the correlation coefficient. The model fitting of Langmuir was verified by the graph of Ce/qe versus Ce for ZnO nanoparticle concentrations amongst 5-30 mg/L, that were a straight line closer to a slope equal to 1/qmax (Figure 6(a)). Higher interaction of MB was observed on ZnO NPs, with an increase in KL to be 1.62 (Langmuir constant/L), indicating that a relatively strong adsorption binding affinity among the adsorbent and the adsorbate. Depending on the slope and intercept of the linear plot, the derived constants, qmax and KL, are shown in Table 1. Adsorption on uneven surfaces is justified by the Freundlich isotherm model, which postulates that the adsorbate concentration affects the adsorption capacity. Plotting ln qe versus ln Ce produced a linear relationship, as depicted in 6(b), from which the slope (1/n) and intercept (KF) were calculated. Stronger adsorption capability is indicated by 1/n value, which is 0.39, as favorable adsorption is indicated by a 1/n value between 0 and 1 [22]. The adsorption mechanism mostly takes place on a heterogeneous surface employing multilayer adsorption, as evidenced by the fact

that the Freundlich model (R^2 = 0.99) showed a greater correlation for MB adsorption than the Langmuir model, having R^2 = 0.97. These outcomes are in agreement with earlier research in which the Freundlich isotherm model was also assessed to describe the adsorption of methylene blue [13]. The maximum adsorption capacity (qmax) has been demonstrated to be 37.7 mg/g, confirming the strong affinity between MB and the adsorbent surface. The outcomes of the isotherm study, including the computed parameters, are summarized in Table 1.

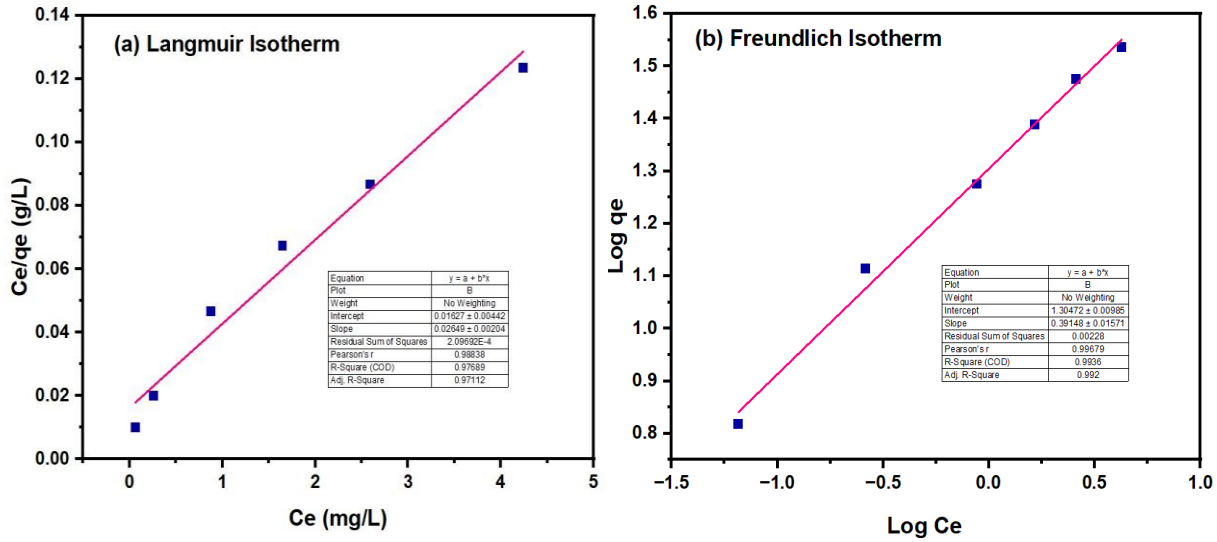


Fig. 6 Linear plots of (a) Langmuir and (b) Freundlich isotherms

Table 1. Computed Isotherm profile for MB adsorption on ZnO

Isotherms	Parameters	
Langmuir	KL	1.62
	qmax (mg/g)	37.7
	R^2	0.972
Freundlich	1/n	0.39
	KF	0.26
	R^2	0.99

3.4. Adsorption Kinetics and Error Analysis

The linear PFO plot ln(qe−qt) versus time as depicted in Figure 8(a) for MB adsorption on ZnO nanoparticles showed poor agreement across computed and experimental qe and lower R^2 values, indicating that the process deviates Pseudo-first-order kinetics. In contrast, the PSO plot (t/qt vs. t) illustrated in Figure 8(b) exhibited higher R^2 and close agreement with experimental qe [23], confirming that the adsorption of methylene blue onto ZnO nanoparticles follows a PSO kinetic model governed by chemisorption. The PSO parameters k_2 and qe (Table 2) were assessed to describe MB adsorption, where qe represents adsorption capacity and k_2 the kinetic rate constant [24]. From the PSO plot, the k_2 rate constant was obtained and the calculated qe (13.54 mg/g) was near to the experimental qe (17.48 mg/g) shown in Table 2; similar studies reported comparable agreement under PSO with $R^2 \approx 0.99$ [25]. The suitability and accuracy of PFO and PSO kinetic models were evaluated using regression coefficients (R^2) along with statistical error parameters, including mean squared error (MSE) and root mean squared error (RMSE). The PSO model exhibited lower MSE and RMSE values compared to the PFO model, as presented in Table 2, indicating its superior fit to the experimental data.

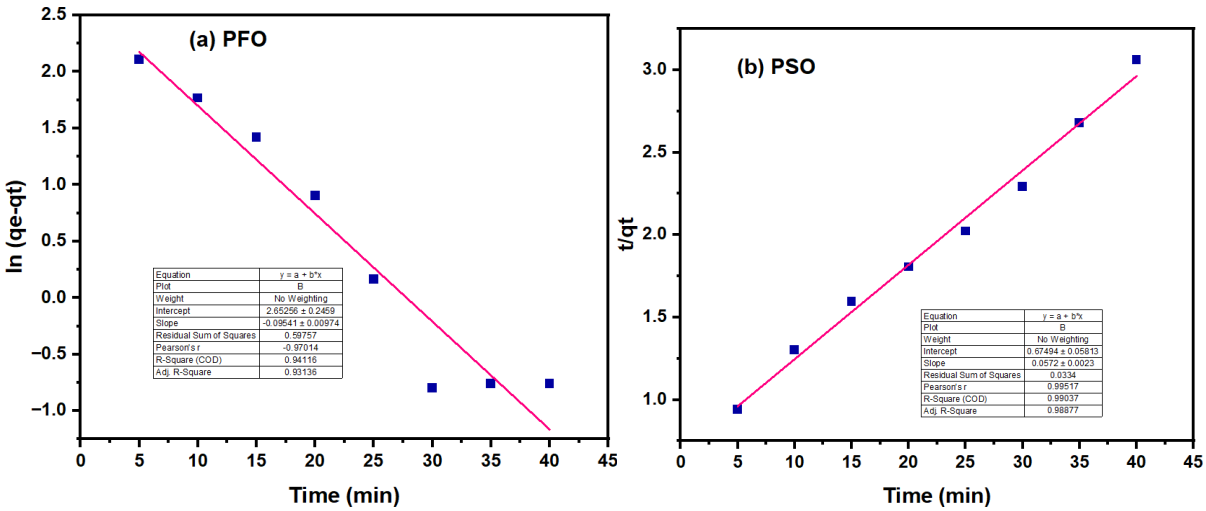


Fig. 7 Kinetic fitting of MB uptake on ZnO-NPs using (a) PFO (b) PSO

Table 2. Kinetic parameters and Error Analysis

Models	Parameters	
PFO	K1(min^{-1})	-0.002385
	qe (mg/g), exp	13.54
	qe (mg/g), cal	7.788
	R^2	0.94
	MSE	4.135
	RMSE	2.033
PSO	K2(min^{-1})	0.004848
	qe (mg/g), exp	13.54
	qe (mg/g), cal	17.48
	R^2	0.99
	MSE	1.94
	RMSE	1.32

Conclusion

The present study confirms that previously green synthesized ZnO nanoparticles can effectively remove methylene blue (cationic dye) from aqueous solutions under mild batch conditions. The adsorption mechanism was predominantly chemisorptive, occurring on a heterogeneous surface, as evidenced by the strong conformity to the PSO kinetic model and the Freundlich isotherm. These results indicate that the adsorption process proceeds via non-ideal multilayer uptake rather than simple monolayer coverage, providing adaptability across varying concentration ranges. The demonstrated efficiency along with the low adsorbent dose, highlights ZnO-NPs as a potential material for large-scale dye removal. In future studies, the results can be extended by carrying out actual wastewater system's performance and adsorbent regenerating (to investigate long-term reusability).

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