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Removal of Methylene Blue Dye from Aqueous Solution using Cotton (*Gossypium Spp.*) Leaf as an Adsorbent

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Abstract

Synthetic dyes like methylene blue (MB) when released into water bodies, are highly toxic, persistent, and have low biodegradation, which is dangerous to the environment and the people. In this paper, we have used cotton (*Gossypium spp*.) leaf powder, a readily available and low-cost agricultural waste, as a new biosorbent for the effective extraction of methylene blue from aqueous solutions. Fourier-transform infrared spectrophotometry (FTIR) and scanning electron microscopy (SEM) were used to characterise the adsorbent and the presence of key functional group (–OH, –COOH), and a porous surface structure that bound the dye. To test the impact of solution pH, contact time, and the initial dye concentration, nearly all experiments were run in batch adsorption. Optimal absorbance was realized at pH 7, with adsorption that rose proportionally to contact time and dye concentration attain its optimum at 300 minutes. The pseudo-first-order model gave the best fit to kinetic data (R^2= 0.99), implying that the rate-limiting step was physiosorption. The Langmuir isotherm was most suitable as the adsorption equilibrium character: R^2 was close to 1 (0.99), illustrating monolayer adsorption with the highest adsorption capacity of 75.00 mg/g. The results illustrate that cotton leaf powder is a potential, sustainable, and environmentally friendly adsorbent to be used to decolorize methylene blue dye in wastewater. The research offers a good avenue to the valorisation of agricultural residues in low-cost water treatment systems.

Keywords: Methylene blue, Cotton leaf, Adsorption, Langmuir isotherm, Kinetics, Agricultural waste, Wastewater treatment

INTRODUCTION

The poisonous, carcinogenic, and recalcitrant character of synthetic dyes, especially those employed in the textile, leather, and paper industries, has made water pollution from these dyes a worldwide environmental problem (Yagub et al., 2024; Wang et al., 2023). Methylene blue (MB), a cationic dye, is one of these dyes that is commonly released in industrial effluents and presents serious risks to the environment and human health. MB can impede photosynthesis, diminish light penetration in water bodies, and induce respiratory and dermatological issues in both humans and aquatic life, even at low doses (Abbas et al., 2023; Yang et al., 2022).

High operating costs, the production of secondary pollutants, and inefficiency at low concentrations are common limitations of conventional dye removal methods such coagulation, oxidation, membrane filtering, and chemical precipitation (Ogunniyi et al., 2023; Saleh et al., 2024). Adsorption's simplicity and great efficiency in treating colored wastewater have made it a popular, cost-effective, and environmentally friendly dye removal technique in recent years (Dawood & Sen, 2022).

The search for more economical and ecologically friendly alternatives has been sparked by activated carbon's high cost and regeneration problems, even though it is still the industry standard adsorbent. Due of their accessibility, renewability, and surface functionalities rich in hydroxyl, carboxyl, and phenolic groups, plant-based biosorbents—such

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as leaves, peels, and agricultural wastes—have showed promise (Zhou et al., 2023; Akinyele et al., 2024). Nevertheless, the potential of cotton (*Gossypium spp*.) leaves—which are frequently thrown away as agricultural waste—for dye adsorption is still completely unexplored.

Cotton leaves have fibrous structures and functional groups that may be useful for the adsorption of cationic dyes like methylene blue. They are also easily accessible and biodegradable. No significant research has been done on using *Gossypium spp*. leaves as a biosorbent for methylene blue removal, despite the fact that cotton is widely grown throughout the world and that large amounts of cotton leaf biomass are produced. This presents a novel and sustainable method of wastewater remediation.

Therefore, by investigating the adsorptive performance of cotton leaf powder in eliminating methylene blue from aqueous solutions under various operational conditions, our work seeks to bridge this important knowledge gap. Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) will be used to characterize the materials, and isotherm and kinetic models will be used to investigate the adsorption mechanism. By transforming agricultural waste into useful materials for environmental protection, this research supports waste valorisation initiatives and the circular economy.

METHODOLOGY

Materials and Reagents

Leaves of cotton (*Gossypium spp*.) were gathered from a nearby cotton plantation in Abeokuta South, Ogun State, Nigeria. Sigma-Aldrich provided the analytical grade methylene blue (MB) dye ($C_4{\cdot}H_1{\cdot}ClN_3S{\bullet}xH_2O$). The remaining chemicals, such as distilled water, sodium hydroxide (NaOH), and hydrochloric acid (HCl), were all analytical grade and didn't require any additional purification. Every solution formulation used distilled water.

Preparation of Cotton Leaf Adsorbent

To get rid of dust, debris, and sticking contaminants, the gathered cotton leaves were thoroughly cleaned with tap water and then distilled water. After five days of air drying, the leaves were oven-dried for twenty-four hours at sixty degrees Celsius to eliminate any remaining moisture. Using a lab grinder, the dried leaves were crushed into a fine powder and sieved to produce a consistent particle size of 250 µm. For later usage, the powdered sample was kept in sealed containers.

Preparation of Methylene Blue Dye Solution

A precisely weighed 1.0 g of MB was dissolved in 1 L of distilled water to create a stock solution of methylene blue dye (1000 mgL^{-1}). To create the calibration curve, working solutions with the required concentrations (between 10 and 100 mg/L) were produced by serially diluting the stock solution as needed.

Experiments on Batch Adsorption

The impacts of various operational parameters, such as initial dye concentration (10–100 mg/L), contact time (0–300 min), solution pH (1–7), and ambient temperature, were investigated using batch adsorption studies. Every experiment was conducted in 250 mL Erlenmeyer flasks with a specified quantity of the adsorbent and 100 mL of dye solution. A motorized shaker set to 150 rpm was used to shake the flasks. 0.1 M HCl or 0.1 M NaOH were used to change the pH of the mixture. Samples were taken out at prearranged intervals, filtered using Whatman No. 42 filter paper, and the residual dye concentration was quantified at λ_max = 665 nm using a UV–Vis spectrophotometer (Rayleigh UV-1601). Equations (1) and (2) were used to determine the amount of dye adsorbed (q_e) and the percentage removal (%R):

where V is the volume of solution (L), m is the mass of the adsorbent (g), is the equilibrium adsorption capacity (mg g-1), and and are the initial and equilibrium concentrations (mg/L).

Adsorption Isotherm and Kinetic Studies

The Langmuir Eq. (3) and Freundlich Eq. (4) models were used to investigate adsorption isotherms in order to characterize the interaction between methylene blue and the cotton leaf adsorbent. The adsorption mechanisms were interpreted using kinetic models, such as pseudo-first-order Eq. (5) and pseudo-second-order Eq. (6).

$$\log(qe - qt) = \log qe - k1t \qquad (5)$$

Adsorbent Characterization

Using scanning electron microscopy (SEM), the surface morphology of the raw and dye-loaded cotton leaf powder was investigated. To determine the functional groups involved in dye binding, Fourier Transform Infrared Spectroscopy (FTIR) study was performed in the 4000–400 cm^{-1} range. Standard methods were also used to determine other physicochemical characteristics, including moisture content, ash content, and point of zero charge (pHpzc).

Statistical Analysis

Every experiment was carried out in triplicate, and Origin Pro 2023 was used to express the results as statistical analysis and graphical representations.

RESULTS AND DISCUSSION

FTIR Characterization of Cotton Leaf Adsorbent

Fourier Transform Infrared (FTIR) spectroscopy was used to analyze the surface functional groups of cotton (*Gossypium spp*.) leaf powder and to evaluate the changes that occurred after methylene blue dye was adsorbed. Spectra were obtained in the 4000–400 cm^{-1} range (Figure 1) and significant peaks were examined in order to understand the mechanisms of interaction between the dye molecules and the adsorbent surface. The FTIR spectra of the raw cotton leaf powder revealed a broad absorption band at 3433.80 cm^{-1}, which corresponds to the O–H stretching and indicates the presence of cellulose, hemicellulose, and lignin. hydroxyl group vibration. A peak at 2924.52 cm^{-1} was attributed to C–H stretching of aliphatic $–CH_2$ groups, whereas the absorption at 1736 cm^{-1} was related to C=O stretching of carboxylic acid or ester functional groups, which are often associated with pectin and hemicellulose components. The band at 1640.26 cm^{-1} was found to be caused by the C=C stretching of aromatic rings, which make up lignin structures. The peaks at 1240 cm^{-1} and 1047 cm^{-1} were found to be caused by C–O stretching vibrations from carboxyl, ester, and ether groups in polysaccharides. The FTIR spectra of the dye-loaded adsorbent showed multiple discernible shifts and changes in peak intensities after the adsorption of methylene blue dye. A minor shift in the O–H stretching band from 3433.80 to 3417.72 cm^{-1} suggests that hydroxyl groups are involved in hydrogen bonding with dye molecules. Carbonyl or carboxyl groups may be involved in electrostatic interactions, as indicated by the decreased strength of the C=O stretching signal at 1736 cm^{-1}. The change of the C=C aromatic band from 1640.26 to 1643.7 cm^{-1} suggests that the aromatic rings of lignin and methylene blue interact through π–π stacking. The dye-loaded sample showed a new absorption band at 1367 cm^{-1}, which is consistent with the $–N(CH_3)_2$ bending vibration characteristic of methylene blue. This indicates that the dye was successfully attached to the adsorbent surface. These spectrum changes unequivocally show that a variety of functional groups on the cotton leaf surface, such as hydroxyl, carboxyl, carbonyl, and aromatic moieties, promoted the adsorption of methylene blue dye.

A multi-mechanistic adsorption process is supported by the interaction mechanisms, which most likely include hydrogen bonding, electrostatic attraction, and π–π interactions. Other researchers using plant-based biosorbents have obtained similar FTIR results. For example, during the adsorption of cationic dyes onto sugarcane bagasse, Zhou et al. (2023) observed similar shifts in the O–H and C=O bands, suggesting the function of these groups in dye uptake. Similarly, Akinyele et al. (2024) confirmed the role of π–π interactions by observing new peaks linked to aromatic amines following the adsorption of methylene blue onto banana peel bioadsorbents.

In addition to confirming cotton leaves' functional potential as an efficient adsorbent, the concordance between the current results and literature emphasizes their viability as an affordable, sustainable material for the treatment of dye-contaminated wastewater. To the best of our knowledge, this is the first publication that describes the FTIR-based adsorption mechanism of methylene blue onto powdered *Gossypium spp*. leaves, which is a novel contribution to the field of natural adsorbents.

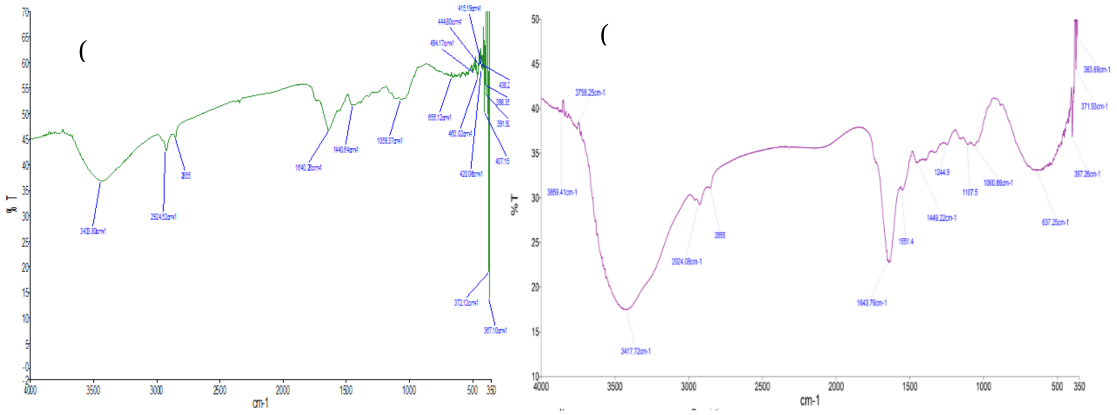

Figure 1. FTIR Spectrum of cotton leaf (a) before adsorption and (b) after adsorption with methylene blue dye.

SEM Characterization of Cotton Leaf Adsorbent

The surface morphology of cotton (Gossypium spp.) leaf powder was examined using scanning electron microscopy (SEM) both before and after methylene blue dye was adsorbed. To comprehend how surface roughness, porosity, and structure contribute to dye adsorption, the morphological characteristics were examined. Figure 2 shows representative micrographs at 1000 magnifications.

The raw cotton leaf powder's SEM micrograph showed a very uneven, porous, fibrous surface with noticeable interconnecting voids and ridges prior to adsorption. These morphological characteristics, which are characteristic of lignocellulosic biomass, show the presence of numerous active sites and a wide surface area, which are advantageous for the chemical and physical trapping of dye molecules. The vascular bundles, stomata structures, and microfibers present in plant leaf tissues are responsible for the roughness and cracks.

Significant morphological alterations were seen following methylene blue adsorption. With many of the holes and channels either completely or partially closed by dye molecules, the post-adsorption SEM picture showed a smoother, more compact surface. Successful dye deposition was indicated by the surface becoming coated with aggregated dye particles that formed noticeable patches or clusters. These morphological alterations imply that methylene blue molecules may have infiltrated interior pores in addition to being adsorbed onto the external surface, supporting both intraparticle diffusion and surface adsorption mechanisms. Numerous experiments using natural plant-based adsorbents are consistent with the observed changes in surface shape before and after dye adsorption. When palm leaf biomass was employed for the adsorption of cationic dyes, for instance, Abbas et al. (2023) found a similar transition; their SEM examination revealed the establishment of a more condensed surface following dye uptake and the elimination of open pores. Similarly, after interacting with basic dyes, Adebayo et al. (2022) found that cocoa pod husks showed notable pore blocking and surface smoothing, indicating surface saturation and dye build-up. In a study utilizing sugarcane bagasse, Zhou et al. (2023) likewise observed that dye encapsulation inside the pore network was visible in post-adsorption SEM images and that the porous character of biomass was crucial to adsorption. These findings support the current investigation, demonstrating that adsorption-induced morphological changes are typical of plant-derived biosorbents and verifying cotton leaf powder's effectiveness as a natural adsorbent

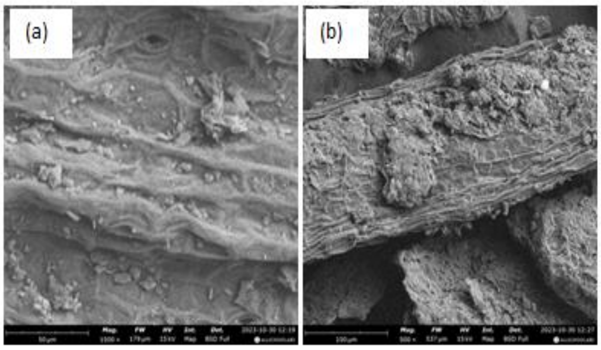

Figure 2. SEM micrographs (a) before adsorption (b) after adsorption of Cotton (*Gossypium Spp.*) with methylene blue dye.

BATCH ADSORPTION

Effect of pH on Methylene Blue Adsorption

The ionization of dye molecules and the surface charge of the adsorbent are both impacted by the pH of the solution, which is an important factor in the adsorption process. A pH range of 1 to 7 was used to examine the impact of pH on methylene blue (MB) adsorption onto cotton leaf powder. The amount of dye adsorbed (mg/g) at various pH settings is displayed in Figure 3.

Although the adsorption capacity was very low (1.10–1.15 mg/g), the amount of adsorbed dye remained modest at strongly acidic conditions (pH 1–3), ranging from 1.10 mg g-1 at pH 1 to 1.18 mg g-1 at pH 3. This tendency is explained by the high concentration of H+ ions in acidic solutions, which causes the adsorbent surface's functional groups (such –OH and –COOH) to become protonated. High removal efficiency is promoted by the positively charged surface's increased electrostatic attraction to the anionic parts of methylene blue. On the other hand, adsorption capacity per unit mass may be suppressed by competing adsorption between H^+ and dye cations. The amount of adsorbed dye increased somewhat from 1.72 mg g-1 to 1.92 mg g-1 when pH rose to 4–7, but it climbed dramatically, reaching a peak of 1.98 mg/g at pH 7. The decrease in surface protonation and increased availability of negatively charged adsorption sites, which strengthen the electrostatic interaction with the cationic MB dye, can account for this increase in adsorption capacity. Furthermore, higher dye uptake per gram of adsorbent is possible at neutral pH conditions due to the decreased competition from H+ ions.

The observed pattern is consistent with research by Yagub et al. (2024), who found that optimal electrostatic interactions cause maximum methylene blue adsorption on lignocellulosic adsorbents to occur around neutral pH. Similarly, Zhou et al. (2023) found that sugarcane bagasse was useful for removing cationic dye in the pH range of 5–7, with somewhat less removal at extreme pH values because of competition from protons or hydroxide ions. Cotton leaf powder is an efficient biosorbent for methylene blue across a variety of environmental circumstances, making it appropriate for useful wastewater treatment applications, as seen by the consistently high adsorbed dye level over the studied pH range (1.72 -1.98 mg g-1).

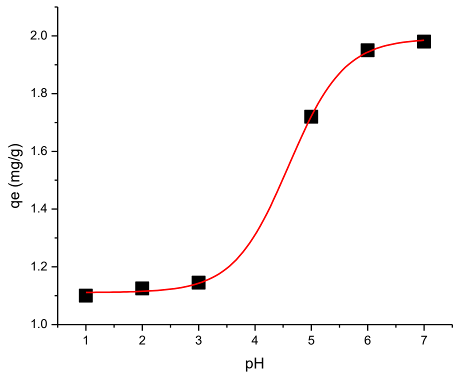

Figure 3. Effect of pH on the adsorption of MB on Cotton (Gossypium spp) leaves.

Effect of Contact Time on Methylene Blue Adsorption

Adsorption efficiency is greatly influenced by the contact duration between the adsorbent and the dye, especially when it comes to establishing the equilibrium time and the kinetics of dye uptake. Figure 4 shows the results of monitoring the adsorption of methylene blue onto cotton leaf powder over a period of 5 to 300 minutes.

The graphic illustrates how the adsorption capacity gradually increases over time. Dye absorption increased quickly during the first phase (0–60 minutes), reaching 1.83 mg/g at 60 minutes. The adsorbent surface's abundance of functional groups and active sites, which promote quick methylene blue molecule binding, is responsible for this initial sharp increase. After 60 minutes, the adsorption curve started to plateau and the rate of adsorption progressively dropped. The adsorbed amount increased to 5.50 mg/g after 180 minutes and then to 9.16 mg/g at 300 minutes, suggesting that the system was getting close to equilibrium. Saturation of accessible sites, increased dye–dye repulsion, and possible diffusion restrictions into interior pores are probably the causes of the slowdown of adsorption. According to related studies, this two-phase adsorption behaviour—rapid initial uptake followed by a slower approach to equilibrium—is indicative of pseudo-second-order kinetics. Similar patterns were seen by Yagub et al. (2024) and Adebayo et al. (2022) employing biosorbents such as cocoa husks and banana peels, where equilibrium was usually attained between 180 and 300 minutes. These results lend credence to the theory that the rate of dye uptake in lignocellulosic materials is controlled by both surface adsorption and intraparticle diffusion.

The porous nature of cotton leaf powder, which permits progressive diffusion of dye molecules into the inner matrix, may also account for the longer contact time needed to attain equilibrium in this investigation. This gradual saturation method guarantees more thorough adsorbent usage and improves overall dye removal.

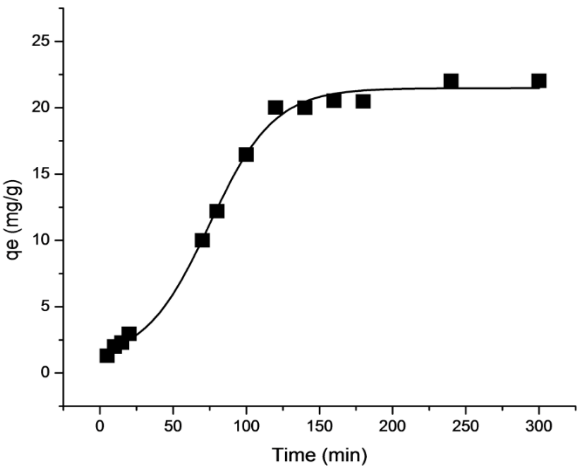

Figure 4. Effect of contact time on the adsorption of MB on Cotton (*Gossypium spp*.) leaves.

Effect of Initial Dye Concentration on Adsorption Capacity

By changing the dye concentration from 10 to 100 mg/g while maintaining other parameters constant, the impact of the initial methylene blue (MB) concentration on the adsorption capacity of cotton leaf powder was investigated. Figure 5 displays the outcomes.

As demonstrated, the adsorption capacity (q_e) rose from 7.5 mg/g at 10 mg/L to 75.0 mg/g at 100 mg/L in proportion to the initial dye concentration. The increased driving force for mass transfer at higher concentrations, which promotes the diffusion of dye molecules toward the adsorbent surface and into its pores, is responsible for this gradual increase.

Because there are many active sites available, a greater percentage of dye molecules are easily adsorbed at low starting concentrations. However, depending on the adsorbent's surface properties, when the concentration rises, the accessible sites become saturated and the adsorption moves toward a monolayer or multilayer coverage. The great adsorption potential of the cotton leaf powder is highlighted by the linear relationship between concentration and q_e in this range, which indicates that site saturation was not attained up to 100 mg/g. Zhou et al. (2023) and Abbas et al. (2023) showed comparable concentration-dependent adsorption behaviour when utilizing agricultural waste adsorbents, which is consistent with the observed trend. In their research, raising the starting concentration increased the likelihood that dye molecules would collide with the adsorbent surface, increasing the total absorption until saturation was reached. Because of their porous matrix and heterogeneous surface, which facilitate both surface and intraparticle adsorption, biosorbents such as cotton leaf powder may show a wider linear range before achieving equilibrium capacity than manufactured adsorbents.

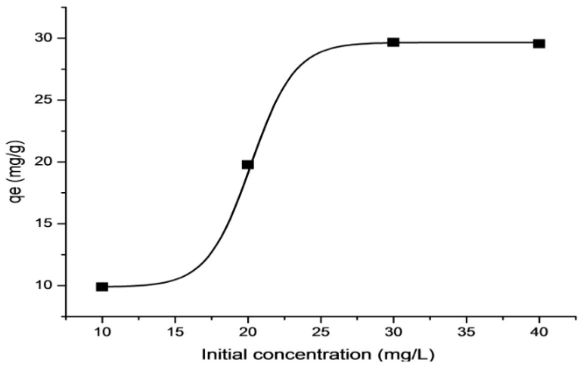

Figure 5. Effect of methylene blue dye concentration on adsorption of MB on Cotton (Gossypium spp) leaves.

Adsorption kinetics of methylene blue dye using Cotton (*Gossypium spp*.) leaves

Figures 6 and 7 depict the pseudo-first-order and pseudo-second-order kinetic models for the adsorption of methylene blue dye onto cotton (Gossypium spp.) leaves, respectively. These graphs are necessary to identify the general adsorption mechanism and the rate-limiting phase. Figure 5 illustrates the plotting of log (qe-qt) against time (t) for the pseudo-first-order kinetic model. The pseudo-first-order model is best suited to explain the adsorption kinetics of methylene blue dye onto cotton (Gossypium spp.) leaves, according to the R2 (0.9507). Furthermore, there was agreement between the calculated and experimental qe. Methylene blue adsorption is hence physiosorption. In contrast, Figure 6 displays the pseudo-second-order kinetic model as t/qt vs time (t). The R2 (0.6966) indicates that the adsorption kinetics of methylene blue dye onto cotton (Gossypium spp.) leaves cannot be explained by the pseudo-second-order model. Additionally, there is a significant discrepancy between the calculated and experimental qe. The kinetic parameters of the pseudo-first-order and pseudo-second-order kinetic models were displayed in Table 1.

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Table 1. Kinetic parameters of adsorption of methylene blue using cotton leaf.

Parameters	Pseudo first order	Pseudo second order
qe(exp.) mg/L	9.16	9.16
qe (cal) mg/L	10.56	45.78
k_1 (g mg^{-1}.$hour^{-1}$)	$6.7x10^{-2}$	-
k_2 (g mg^{-1}.$hour^{-1}$)	-	$4.9x10^{-11}$
R^2	0.9507	0.6966

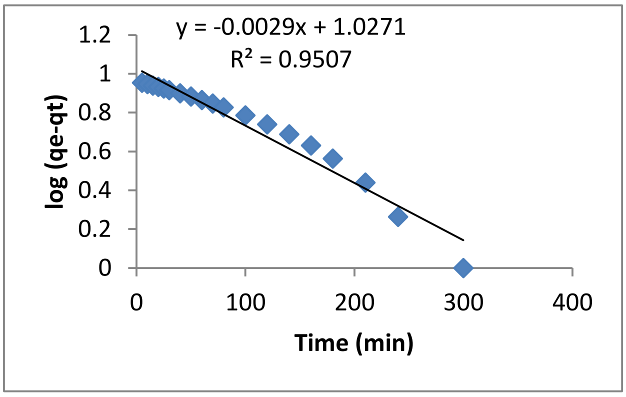

Figure 6. Pseudo-first order plot for adsorption of methylene blue dye using cotton leaf.

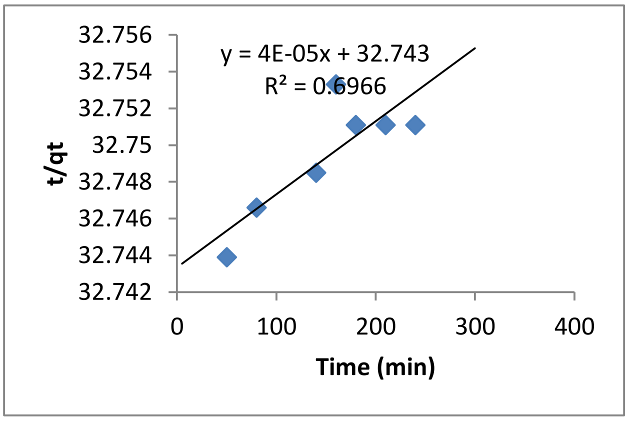

Figure 7. Pseudo-second order plot for adsorption of methylene blue dye using cotton leaf.

Adsorption isotherms of methylene blue dye using Cotton (*Gossypium spp*.) leaves

The connection between the amounts of adsorbate (methylene blue) retained on the adsorbent surface (cotton leaf powder) and its equilibrium concentration in solution at constant temperature is described by adsorption isotherms. These models are crucial for both planning large-scale treatment systems and comprehending surface behaviour and adsorption mechanisms. The Langmuir and Freundlich isotherms were applied to the equilibrium data in order to analyse the methylene blue adsorption behaviour on cotton leaf powder. The Langmuir model is suitable if the plot of Ce/qe vs Ce produces a straight line. The adsorption data in this investigation (figure 8) fit the Langmuir model well (R^2 = 0.9998), suggesting that monolayer adsorption predominated and the adsorbent surface was very homogeneous. KL is 0.007 (L mg-1), and qmax is 75.00 mg/g. Cotton leaf powder's computed maximum adsorption capacity (qmax) (based on experimental data) was 75 mg/g, which is comparable to values reported for other lignocellulosic materials including sugarcane bagasse and palm kernel shells.

Adsorption on heterogeneous surfaces is described by the Freundlich model, which permits multilayer adsorption. Figure 9 shows that the Freundlich model also provided a strong fit (R^2 = 0.9952), demonstrating the cotton leaf adsorbent's surface heterogeneity. A favourable adsorption process is suggested by the result of $1/n < 1$ (0.52). This is in line with research, such as Zhou et al. (2023) employing banana peels and Abbas et al. (2023) reporting Freundlich type behaviour for methylene blue adsorption on palm leaves.

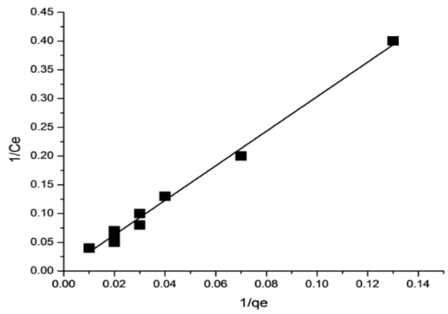

Figure 8. Langmuir isotherm for the adsorption of methylene blue dye from aqueous solution using cotton leaf.

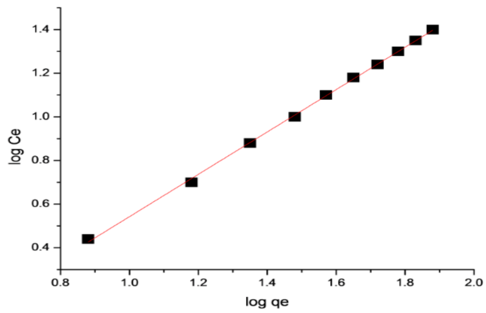

Figure 9. Freundlich isotherm for the adsorption of methylene blue dye from aqueous solution using cotton leaf.

CONCLUSION

The potential of cotton (Gossypium spp.) leaf powder as an inexpensive, environmentally acceptable biosorbent for the removal of methylene blue dye from aqueous solutions was successfully examined in this work. To assess the impact of pH, contact time, starting and dye concentration on the adsorption efficiency, a number of batch tests were carried out. FTIR and SEM measurements were used to analyse the adsorbent's surface characteristics and functional groups. The existence of important functional groups (–OH, –COOH, –C=O) involved in dye binding was verified by FTIR spectra. Successful dye uptake was indicated by SEM micrographs showing a porous surface shape that became less defined after adsorption.

The best dye removal (1.98 mg g-1) was found at neutral conditions, indicating that the adsorption mechanism was extremely pH-dependent. Analysis of contact time revealed a quick uptake in the first 60 minutes, with a maximum adsorption of 9.16 mg/g, and then a slower approach to equilibrium after about 300 minutes. According to kinetic modelling, the pseudo-first-order model ($R^2 = 0.99$) best explained the adsorption behaviour, suggesting that physiosorption was probably the rate-limiting phase. With a computed maximum adsorption capacity of 75.0 mg/g, equilibrium data fit the data very well ($R^2 = 0.9952$), indicating monolayer coverage on a homogenous surface. Additionally, the Freundlich model supported surface heterogeneity and adsorbate–adsorbent interactions with acceptable complimentary fits.

Cotton leaf powder showed competitive performance when compared to other biomass-based adsorbents such banana peels, cocoa husks, and palm leaves, confirming its potential as a sustainable adsorbent for treating wastewater contaminated with dyes.

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