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Synthesis, Characterization, and Application of Nanoadsorbent Derived from Prosopis Cineraria (Khejri)

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Abstract: ***The research paper discusses the preparation of plant-based nanoadsorbents through the chemical activation process, using various locally available plants and trees as sources of activated carbon. The study focuses on nanoadsorbents synthesized from the barks of Prosopis cineraria, commonly known as Khejri in the local language. The nanoadsorbent's characterization includes Attenuated total reflectance spectroscopy (ATR), Scanning electron microscopy (SEM), and X-ray diffraction (XRD), surface area analysis for assessing its capability to remove organic pollutants. The experiments were conducted using the batch adsorption technique, examining the pH effect and different adsorbent doses of activated carbon for removing the organic dye methylene blue from wastewater. The results demonstrated that the maximum adsorption of methylene blue occurred at pH 7, reaching adsorption equilibrium within 75 minutes at 100 mg/L MB concentration, with an adsorbent dose of 20 mg/50 mL for each sample. The removal efficiency of the MB dye increased with higher pH levels, with pH 7 showing the maximum removal capacity for all samples.***

Keywords: Nanoadsorbant, Prosopis cineraria, Methylene blue, Adsorption Isotherms

1. Introduction

The availability of clean water is essential for all living beings to survive on Earth [1]. However, water pollution, caused by industrialization, urbanization, and agricultural activities, has led to the degradation of surface water and groundwater resources [2-3]. One of the significant contributors to water pollution is the textile industry, which generates vast amounts of wastewater containing harmful organic compounds that are non-biodegradable and toxic [4]. Synthetic dyes used in the textile industry, including cationic azo dyes, vat dyes, anthraquinone dyes, anionic dyes, and reactive dyes, are major pollutants that contaminate aquatic systems and disrupt the aquatic food chain [5].

To address this issue, various dye reduction technologies have been introduced, including physical, chemical, and biological methods [6]. Among these, physical methods like adsorption have been favoured due to their lower cost, ease of operation, and eco-friendly nature. Nanoparticle-based adsorbents have gained attention recently due to their high adsorptive removal rate, characteristic surface properties, and compact size. Both conventional and non-conventional adsorbents, obtained from plant wastes and other sources, have been explored to meet the criteria of an ideal adsorbent [7-9].

Understanding the adsorption mechanisms and interactions is crucial for studying the kinetics of dye adsorption. Different isotherm models have been used to explain the contact time between adsorbents and pollutants in wastewater [10]. Nanoadsorbents possess unique characteristics, such as high selectivity and adsorption capacity, and imaging techniques like SEM and XRD help to study their effectiveness in dye removal.

The primary aim of the research discussed is to synthesize an eco-friendly and cost-effective nanoadsorbent using barks of Prosopis cineraria (Khejri) plant and apply it to remove methylene blue (MB) dye from wastewater. Prosopis cineraria are a species of flowering tree in the pea family, Fabaceae that is native to arid portions of Western Rajasthan [11]. It is also called Khejari, Shami and Janti [12]. The bark of the Prosopis cineraria (Khejri) tree, which is usually considered waste or discarded, can serve as a valuable source for preparing nanoadsorbents. By utilizing the bark in synthesizing nanoadsorbents, researchers can turn what was once considered waste into a useful and eco-friendly material for various applications, including removing contaminants such as dyes from wastewater.

2. Materials and Methods

2.1. Chemical used

Several chemicals and reagents were used in this study, including sulphuric acid (H_2SO_4), barium chloride ($BaCl_2$), hydrochloric acid (HCl), sodium hydroxide (NaOH), distilled water, and methylene blue dye ($C_{16}H_{18}ClN_3S$). Prosopis cineraria (Khejri) dead bark was also used in the experiment. A stock solution of methylene blue dye was prepared at a concentration of 1.0 g/L in double distilled water, and it served as the model pollutant for the study. Methylene blue has a molecular weight of 319.85 g/mol [13]. The chemicals and reagents employed in this study were of analytical grade. The structure of methylene blue is depicted in Figure 1.

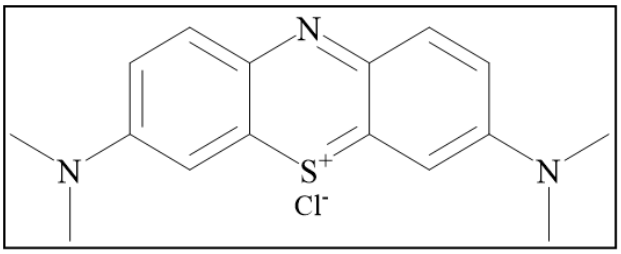

Figure 1: Structure of Methylene blue (MB) dye

2.2. Adsorbent Material

The barks of P. cineraria, obtained from western Rajasthan, underwent an initial cleaning step using distilled water to eliminate water-soluble adherent impurities. Subsequently, the cleaned barks were dried in an oven at 80 °C for 6 hours to eliminate moisture and other volatile impurities. After this drying process, the barks were ready for further processing.

2.3. Carbonization and chemical activation of dead Prosopis cineraria bark

The barks were subjected to a treatment of 10% v/v sulphuric acid in a 1:1 ratio. After the treatment, they were placed in an oven at 150 °C for 24 hours. The treated barks were then filtered and washed repeatedly with distilled water to eliminate any remaining sulphuric acid (confirmed by testing the washings with two drops of barium chloride solution). Subsequently, the barks were dried and thermally activated at 300 °C for 3 hours. Once cooled to room temperature, the material was transformed into a powder form, sieved, and stored in an airtight container for further characterization. This prepared material served as the adsorbent for studying the adsorption of dye contaminants at different pH levels.

2.4. Instrumentation

In the study, the crystallinity, phase purity, and particle size of the prepared activated carbon (AC) were analyzed using X-ray diffraction (XRD). The XRD pattern was obtained using a PANalytical X'Pert PRO diffractometer, which operated with Cu Kα radiation (λ = 1.5406 Å) at 40 mA and 40 kV, within the 2θ range of 10 to 70 degrees.

To analyze the presence of various functional groups and bonding types, ATR (Attenuated Total Reflectance) spectra were acquired using a Bruker Alpha ATR spectrometer provided by Bruker, Germany. The ATR technique allows for examining samples in their native form without extensive sample preparation, making it a valuable tool for characterizing the molecular composition of the material under study [14].

The microstructural and textural properties of the synthesized adsorbent were analyzed using the scanning electron microscopy (SEM) technique. The study of the surface morphology of the adsorbents was conducted using the Carl Zeiss Evo 18 scanning electron microscope, which is manufactured by Carl Zeiss in Germany. SEM is a powerful imaging tool that provides detailed information about the surface features, particle size, and shape of the adsorbent material, enabling a comprehensive understanding of its structure and texture [15].

Absorbance measurements were carried out using an ECIL-made PC-based double-beam UV-Vis spectrophotometer UV 5704 SS. The spectrophotometer was equipped with matched quartz cells for precise measurements. The absorbance of methylene blue was measured at a wavelength of 655 nm. This analysis allowed for the determination of the concentration of methylene blue in the samples and facilitated the evaluation of its adsorption characteristics [16].

2.5. Methylene blue adsorption experiment

In this study, batch adsorption experiments were conducted to evaluate the adsorption capacity of the prepared adsorbent samples using an aqueous solution of MB dye. The adsorption process was examined by varying parameters, such as the adsorbent amount, initial dye concentration, pH, and contact time.

To create different solutions with varying concentrations, the stock solution of MB dye was diluted accordingly. The adsorption process was carried out in a 250 mL conical flask, containing 50 mL of the MB dye solution, under optimized conditions and at room temperature. The flask was placed on a thermostatic shaking platform for agitation. During the experiments, the dye concentration and adsorbent dose were varied within the ranges of 100-250 mg/L and 3-8 g/L, respectively, while the contact time varied from 15 to 105 minutes. The pH of the solution was adjusted using 0.1 M NaOH and HCl solutions, and the pH range examined was from 5 to 9.

To determine the effectiveness of adsorption, the remaining concentration of MB dye in the solution was measured by analyzing the absorbance at 665 nm using a UV-visible spectrophotometer (Perkin Elmer, LAMBDA-750). The equilibrium adsorption capacity (qe), percentage of dye removal, and adsorption capacity (qt) were calculated using the following equations (1)-(3):

$$q_e = \left(\frac{C_o - C_e}{m}\right) xV \qquad (1)$$

$$\text{Percentage removal} = \left(\frac{C_o - C_e}{C_o}\right) xV \qquad (2)$$

$$q_t = \left(\frac{C_o - C_e}{m}\right) xV \qquad (3)$$

here, Co and Ce mg/L) represents the initial concentration and equilibrium concentration of MB dye, respectively. V is the volume of the MB solution (L), and m is the mass (g) of the adsorbent [17].

3. Results and Discussions

3.1 Structural characterization of the prepared nanoadsorbent

Characterization of prepared activated carbon of Prosopis cineraria (AC-PC) nanoadsorbent by the following methods has been done:

➢ **XRD pattern**

The XRD analysis of the synthesized activated carbon derived from Prosopis cineraria (AC-PC) dead bark confirmed the presence of an amorphous structure. The XRD pattern displayed distinc peaks at 23.13° and 26.40°. These peaks are characteristic of activated carbons, indicating the stacking of a few graphene-like layers [18].

The average particle size of the activated carbon was determined using Debye Scherrer's equation (Eq. 4), where D represents the particle size, λ (1.540 Å) is the wavelength of Cu Kα radiation, β is the full width at half maximum (FWHM) of the peaks, and θ is the Bragg's angle. The calculated average crystalline size using Debye-Scherrer's equation is approximately 2 nm for the activated carbon.

$$D = \frac{0.9\lambda}{\text{Cos}\,\theta\,\beta} \qquad (4)$$

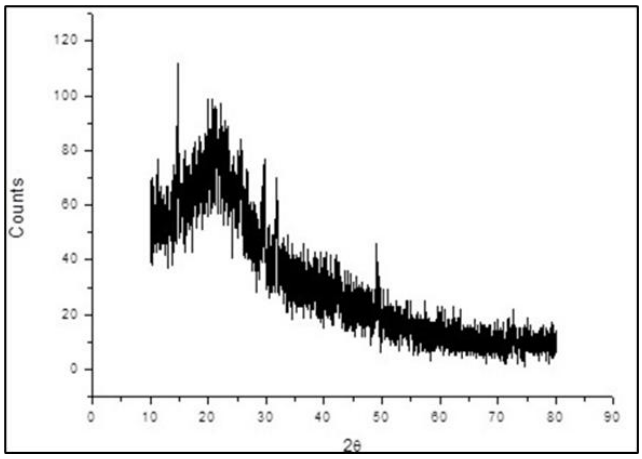

Figure 2: XRD spectra of AC-PC nanoadsorbent

➢ **ATR spectra**

The ATR spectra of the activated carbons derived from the dead bark of Prosopis cineraria (Khejri) (Fig.3) exhibit various functionalities that are commonly observed in other carbons produced through sulphuric acid activation of lignocellulosic precursors. One notable absorption band appears in the range of 3700-3600 cm−1, with a maximum of approximately 3400-3340 cm−1 [19]. This band corresponds to the stretching vibration of O-H groups. Another peak around 1683 cm−1 is attributed to C=O stretching vibrations, which can be linked to ketones, lactones, aldehydes, or carboxyl groups. However, the relatively minor intensity of this peak suggests a lower content of carboxylic groups compared to other oxygen-containing groups in the carbon adsorbents [20] Furthermore, a broad band in the primary fingerprint spectral region, cantered at 1143 cm−1, indicates C–O stretching in various groups, such as acids, phenols, alcohols, ethers, and esters. A peak is observed at 960 cm−1, which can be attributed to the stretching vibration of C–C or C–H groups or the presence of the C=O group. Additionally, the region between 1240-1000 cm−1 indicates the presence of phenolic and alcoholic groups, while the bands at 900-600 cm−1 point to the existence of aromatic ring structures. A specific peak at 593 cm-1 is credited to the in-plane ring deformation [21].

Overall, the ATR spectra reveal the diverse functional groups present in the AC-PC nanoadsorbent, suggesting their potential suitability for various adsorption applications.

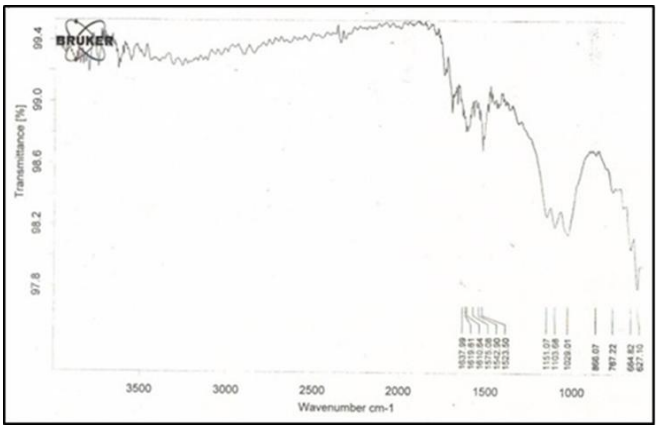

Figure 3: ATR spectra of AC-PC nanoadsorbent

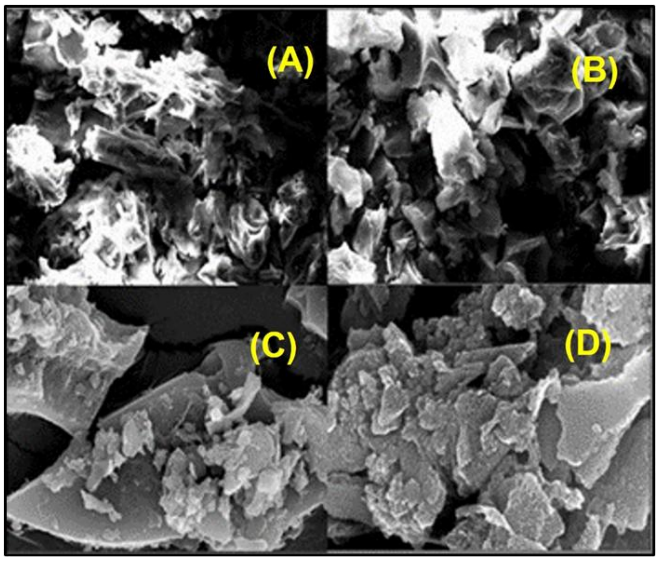

Figure 4 (A-D): SEM images of AC-PC nanoadsorbent

➢ **SEM images**

The morphological studies of activated carbon were carried out using scanning electron microscopy (SEM) analysis. The SEM images at different magnifications are shown in Figure 4(A-D). The micrographs show the rough surface of AC-PC nanoadsorbenr. On the surface, there are irregular channels present for the adsorption of the dye and other pollutants [22].

3.2 Adsorption Parameters

Various adsorption parameters have been studied to understand the adsorption efficiency of AC-PC nanoadsorbent:

➢ **Effect of Contact Time**

The graphical representation in figure 5 illustrates the impact of varying contact times on the percentage removal of methylene blue dye by AC-PC nanoadsorbent. As the contact time increases, the uptake of methylene blue dye also increases, indicating that the adsorption process becomes more efficient with a longer exposure time. However, after 75 minutes, the percentage removal of the dye remains constant. This plateau suggests that the adsorption sites on the AC-PC nanoadsorbent are well exposed, and equilibrium is achieved at this point. Based on this observation, the effective contact time, also known as the equilibrium time, is determined to be 75 minutes. Beyond this duration, there is no significant increase in dye removal, indicating that the adsorption process has reached its optimal capacity.

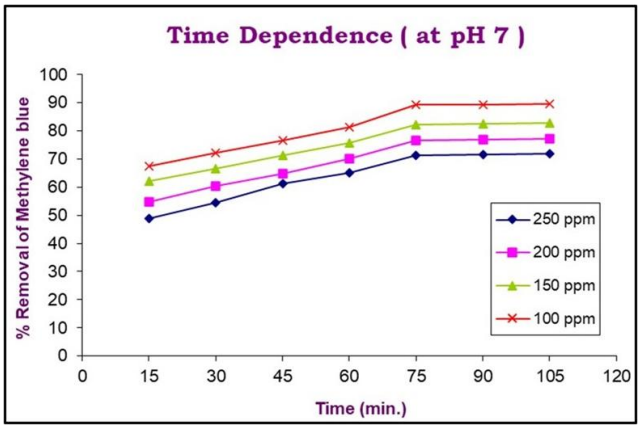

Figure 5: Effect of contact time on MB adsorption onto AC-PC nanoadsorbent

➢ **Effect of initial dye concentration**

Figure 6 displays the effect of varying initial MB dye concentrations at the optimum pH of 7 on the percentage removal by AC-PC nanoadsorbent. At a lower initial dye concentration of 100 mg/L, the removal efficiency is notably higher, reaching 89.3% for AC-PC nanoadsorbent. However, as the initial concentration of the dye increases, the removal efficiency decreases. This trend can be explained by considering the competition among the adsorbate species for the available binding sites on the adsorbent. At lower initial concentrations, there are fewer adsorbate species, allowing for a more efficient adsorption process. As the initial concentration rises, more dye molecules are introduced into the solution, increasing competition for binding sites on the AC-PC surface. Consequently, the removal efficiency decreases because there are fewer active sites available to accommodate all the adsorbate species. At higher dye concentrations, the lack of available active sites on the adsorbent leads to some adsorbate species remaining unadsorbed in the solution, contributing to decreased removal efficiency. This phenomenon is commonly observed in adsorption processes, where the adsorbent's capacity becomes saturated with increasing adsorbate concentration [23].

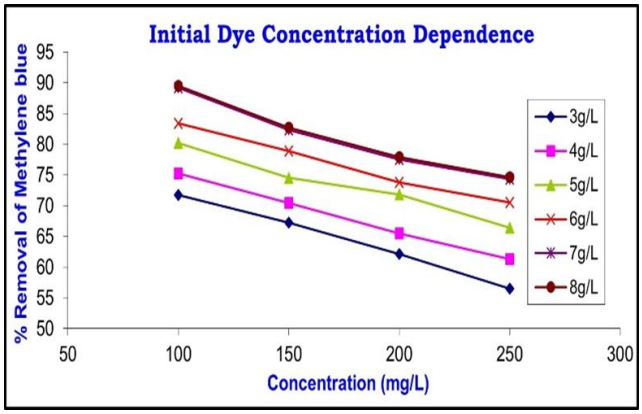

Figure 6: Effect of initial dye concentration on removal by AC-PC nanoadsorbent

➢ **Effect of dose of AC-PC nanoadsorbent**

Figure 7 illustrates the impact of varying the dose of AC-PC nanoadsorbent on the percentage removal of methylene blue dye. As the dose of AC-PC increases, the removal of methylene blue dye also increases, and this trend continues up to a dose of 7 g/L. After reaching this dose, the increase in removal becomes relatively small, indicating that the adsorption process becomes less efficient at higher doses. Therefore, the effective dose of AC-PC is determined to be 7 g/L at pH 7. The reason behind this behavior lies in the greater availability of exchangeable sites or surface area at higher doses of the adsorbent. With an increased dose, more active sites on the AC-PC become available for the adsorption of dye molecules, leading to more significant removal of the dye from the solution.

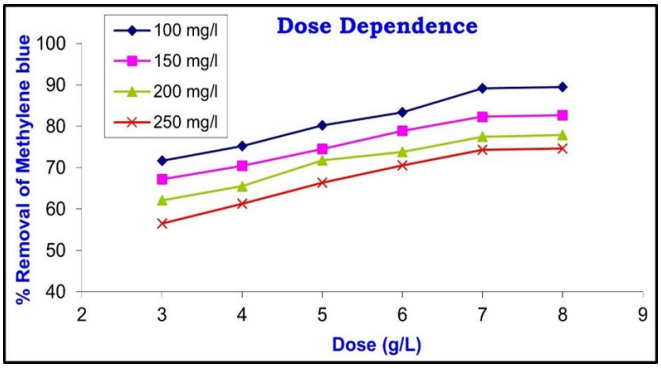

Figure 7: Effect of adsorbent dose of AC-PC nanoadsorbent for MB dye removal

➢ **Effect of pH**

The effect of pH variation on the percent removal of MB dye by AC-PC nanoadsorbent is graphically represented in Figure 8. At low pH the adsorption percentage of MB dye is small. In between pH values 5-7 the percentage increase sharply up to 89.2 %, then it decreases with an increase in pH. The lower the pH, the more H+ ions compete with dye for adsorption sites, thus reducing their adsorption. On the other hand, the higher the pH, the less the H+ ions compete with dye for adsorption sites, thus increasing their adsorption.

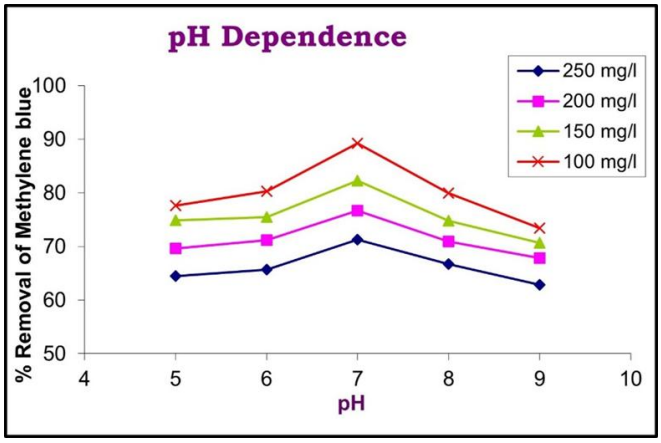

Figure 8: Effect of pH on MB dye removal by AC-PC nanoadsorbent

➢ **Kinetic study of adsorption**

The study of adsorption dynamics is essential to understand the rate at which the adsorbent material takes up the solute. This rate plays a crucial role in determining the residence time of the adsorbate at the solid-solution interface. In this study, the kinetics of MB adsorption on AC-PC nanoadsorbent was analyzed using the pseudo-second-order model. The pseudo-second-order model is a widely used kinetic model in adsorption studies, and it describes the rate of adsorption as being directly proportional to the square of the amount of unadsorbed solute and the amount of available adsorption sites on the adsorbent surface [24]. To assess the conformity between the experimental data and the model-predicted values, correlation coefficients (R2 values) were calculated. A relatively high R2 value, close to 1, indicates a strong agreement between the experimental data and the model, suggesting that the pseudo-second-order model effectively describes adsorption kinetics for the AC-PC system. In the adsorption kinetics measurements, dye solutions with concentrations of 100, 150, 200, and 250 mg/L were used at the optimum pH value. These different concentrations allowed for a comprehensive understanding of the adsorption dynamics and provided valuable information on how the adsorption rate varies with varying initial dye concentrations.

Pseudo second order equation:

$t/qt = 1/ho + 1/ (qe)t$

Where:

ho is the initial adsorption rate (mg/g min.);
qe is the amount of dye adsorbed at equilibrium (mg/g);
qt is the adsorbed at time t (mg/g).

The initial adsorption rate, ho, as $t \rightarrow 0$ is defined as:

$h = K_2 qe^2$

Where: K_2 is the Pseudo second-order rate constant for the adsorption process (g/mg min.). The initial adsorption rate ho, the equilibrium adsorption capacity, and the pseudo second-order rate constant, K_2, were determined from the slope and intercept plot of t/qt against t [25].

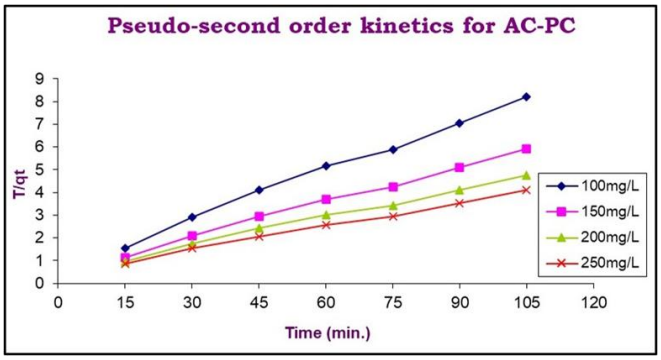

Figure 9: Pseudo-second order kinetics model for MB dye removal

Table 1: Pseudo-second order kinetic model and other statistical parameters for MB dye at pH 7 by AC-PC nanoadsorbent

Parameters	100 mg/L	150 mg/L	200 mg/L	250 mg/L
K_2	3.69 x 10^{-3}	2.18 x 10^{-3}	1.74 x 10^{-3}	1.10 x 10^{-3}
R^2	0.9918	0.9892	0.9907	0.9797
q_e	14.64	21.41	26.52	32.47
H	0.7917	1.0012	1.2223	1.1649

➢ **Adsorption Isotherm Study**

From the graphs, the values of the Freundlich and Langmuir constants were determined for AC-PC and are summarized in Tables 2 and 3. The Freundlich constant "n" for MB was found to be 1.9716 by AC-PC at the effective dose and contact time. This value of "n" suggests a good adsorption potential of the adsorbent, indicating favorable adsorption characteristics. The values of R_L (separation factor) and R^2 (correlation coefficient) further confirm the favorable nature of the adsorption process. A value of R_L for the 100 mg/L MB dye solution was determined to be 0.3578 by AC-PC, which indicates favorable adsorption conditions for the given concentration. Overall, the obtained values from the Freundlich and Langmuir isotherms, along with R_L and R^2, demonstrate the favorable and efficient adsorption potential of AC-PC for removing methylene blue dye from aqueous solutions.

Table 2: Values of Langmuir isotherm constants for adsorption of MB dye by AC-PC nanoadsorbent.

Adsorbent dose (g/L)	Qm (mg/g)	b (L/mg)	R_L	R^2
3	71.42	0.0179	0.3578	0.9997
4	59.52	0.0182	0.3545	0.9979
5	51.02	0.0221	0.3113	0.9908
6	43.1	0.0272	0.2682	0.9913
7	34.36	0.0459	0.1787	0.9752
8	30.03	0.0475	0.1737	0.9765

Table 3: Values of Freundlich isotherm constants for adsorption of MB dye by PV-AC nanoadsorbent

Adsorbent dose (g/L)	Freundlich Constants		
	K_f	n	R^2
3	4.5019	1.9716	0.9873
4	3.5546	1.912	0.997
5	3.4498	1.9379	0.993
6	3.4769	2.0136	0.9981
7	4.7654	2.4576	0.9947
8	4.2648	2.4752	0.9953

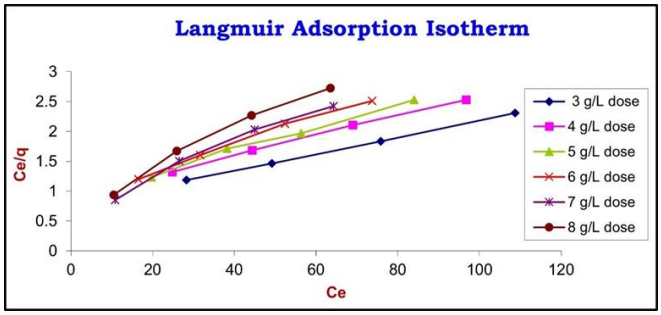

Figure 10: Langmuir isotherm plot for MB dye adsorption by AC-PC nanoadsorbent

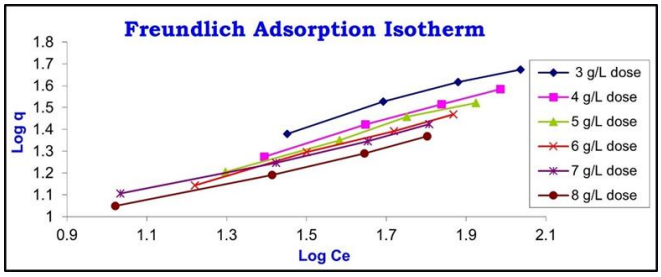

Figure 11: Freundlich isotherm plot for MB dye adsorption by AC-PC nanoadsorbent

4. Conclusions

The results and conclusions from the adsorption studies present a new perspective and enrich our understanding of this broad and fascinating field. Based on the findings of this research, the nanoadsorbent prepared from the barks of the plant Prosopis cineraria proves to be highly effective in removing methylene blue dye. The abundance of Prosopis cineraria in the local environment makes it a highly economical choice for adsorption purposes, as it is much cheaper compared to commercial activated carbon. This cost-effectiveness is a significant advantage, especially for large-scale applications and water treatment processes. To further minimize the running cost of the treatment process, it is recommended to optimize the contact time as a cost-limiting factor in batch processes. By keeping the contact time as short as possible while ensuring the required efficiency, the operational costs can be reduced. The adsorption kinetics of methylene blue dye on the AC-PC nanoadsorbent revealed that equilibrium was achieved within 75 minutes. This indicates a relatively fast and efficient adsorption process, making it suitable for various practical applications. Moreover, the nanoadsorbent exhibited an excellent removal rate of 89.3% for MB dye, underscoring its high effectiveness in removing organic pollutants from water. In conclusion, the research highlights the promising potential of the AC-PC nanoadsorbent as a cost-efficient and effective means for removing methylene blue dye from wastewater, offering valuable insights for practical applications and environmental remediation efforts.

Acknowledgment

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