

Methylene Blue Dye (MBD) and Safranin Dye (SD) Removal Using Activated Carbon Obtained From Rice Husk as Adsorbents

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Abstract

Dye pollution poses major threats to human health and aquatic ecosystems when it contaminates surface water. This study looks at the efficacy of removing cationic safranin dye (SD) and methylene blue dye (MBD) from synthetic wastewater using activated carbon produced from rice husk (ACRH) as a biowaste adsorbent. Before and after the adsorption procedure, the ACRH was produced and examined using several technologies. ACRH's microrough and heterogeneous surface was shown by SEM pictures, and functional groups such as amines, carboxylic, and hydroxyl groups were found by FTIR analysis. Adsorption studies were used to determine the ideal circumstances by examining the effects of factors such as starting dye concentration, pH, adsorbent dose, and contact duration on removal efficiency. When adsorbed onto ACRH, the removal efficiency for MBD and SD under these circumstances surpassed 99%. Three isotherm models were used: the Freundlich, Temkin, and Langmuir models. For both dyes, the Langmuir model suited the data the best. The adsorption process is shown to follow pseudo-second-order kinetics, according to kinetic studies. Additionally, thermodynamic parameters were computed, and regeneration investigations demonstrated that ACRH could sustain its effectiveness for a maximum of five cycles. This study demonstrates the potential of ACRH as a useful bio adsorbent that can be used to remove MBD and SD dyes from water while also providing a simple and effective manufacturing technique.

Keywords: Adsorption, removal of dye, Isotherm, Wastewater Treatment, Kinetics.

1. Introduction

Though only 0.03% of resources of water are used by human beings then also ecosystems have been severely affected by human activity and population growth worldwide, creating a danger to water safety (Akter et al., 2021). Untreated or inadequately treated industrial wastewater releases are the primary source of water contamination (Kadhum et al., 2021a). With a 17–20% contribution to industrial wastewater contamination, textile dyeing effluents are especially noteworthy. Aquatic life is negatively impacted by complex, difficult-to-degrade aromatic chemicals found in the wastewater of the textile and dyeing industries. In the paper, leather, and textile sectors, cationic dyes are widely utilized. It has become very important to remove these organic dyes because of their possible toxicity and the fact that they are present in surface water. Methylene Blue Dye (MBD) is a basic cationic dye that has two positive charges and is often employed in biology and medicine. Another extremely harmful cationic dye is safranin dye (SD) (Humelnicu et al., 2017). Many approaches have been used to address dye pollution: chemical approaches like ozonation, advanced oxidation, fenton reaction etc., biological processes like enzyme, algae degradation, adsorption by microbial mass etc. and physical processes like ion exchange, flocculation, coagulation etc. Because physical procedures are so easy to use and so successful, they are most often utilized

for color removal. Of these, adsorption is especially well-liked because to its ease of use, affordability, and excellent efficacy at low concentration of dye as well as at high concentration of dye. Various studies have been done to find out the efficiency of adsorbent from agricultural products such as shells of wheat, shells of ground nut (Bello et al., 2012), peels of potato, peels of orange fiber of oil palm and activated carbon (Namasivayam and Kavitha, 2002), in effectively removing dyes from various sources.

Rice husk is a particularly attractive option among these bio adsorbents because of their inexpensive cost and widespread availability. They work well as bio adsorbents for decolorization because of their chemical makeup, which consists of hemicellulose, cellulose, lignin, and pectin as well as different polar functional groups like hydroxyl, carboxylic, and phenolic acids. In order to ascertain rice husk's efficacy as a bioadsorbent for eliminating cationic MBD and SD, this study's biowaste material underwent a series of straightforward preparatory processes and analyses. This might be a new use for activated carbon from rice husk (ACRH) adsorbent in the removal of adsorbate like MBD and SD from aqueous solutions. To improve comprehension of the adsorption process, adsorption isotherms, kinetics, and thermodynamic parameters were also computed for both dyes.

2. Materials and Methodologies

The bio-adsorbent utilized in this investigation, rice husk, was procured from a nearby marketplace. Methylene blue dye (C₁₆H₁₈ClN₃S) and safranin dye (C₂₀H₁₉N₄Cl) were the dyes used. In addition, deionized water, concentrated sulfuric acid (H₂SO₄), and 0.01 M sodium hydroxide (NaOH) were utilized. The supplier of all chemical reagents was Sigma-Aldrich in Germany.

2.1 Rice husk from activated carbon (ACRH) Preparation

The enormous outer area of rice husk (RH) was used to manufacture activated carbon. Pressures up to 3.5 MPa and temperatures of 293K, 303K, 313K, and 323K are critical factors for rice husk activated carbon. RH was initially washed and dried in order to produce the activated carbon. After that, the dehydrated rice husk was carbonized in an atmosphere of nitrogen at 1273 K. Following the carbonization of RH, the material was grinded and potassium hydroxide was applied. For two hours, this combination was kept at a temperature 1073 K in a nitrogen atmosphere. Activated carbon (AC) was formed by vacuum drying the activated product at 393 K after it had been washed with distilled water on a regular basis.

Characterization of ACRH Using a Czech TESCAN VEGA III SEM, the surface topography, chemical content, and orientation of pre- and post-adsorption ACRH samples with MBD and SD were examined. Additionally, using an IRAffinity-1S spectrophotometer from Shimadzu, Japan, which covers the 4,000–400 cm⁻¹ spectral range, FTIR spectra of the ACRH samples were acquired both before and after the adsorption of MBD and SD.

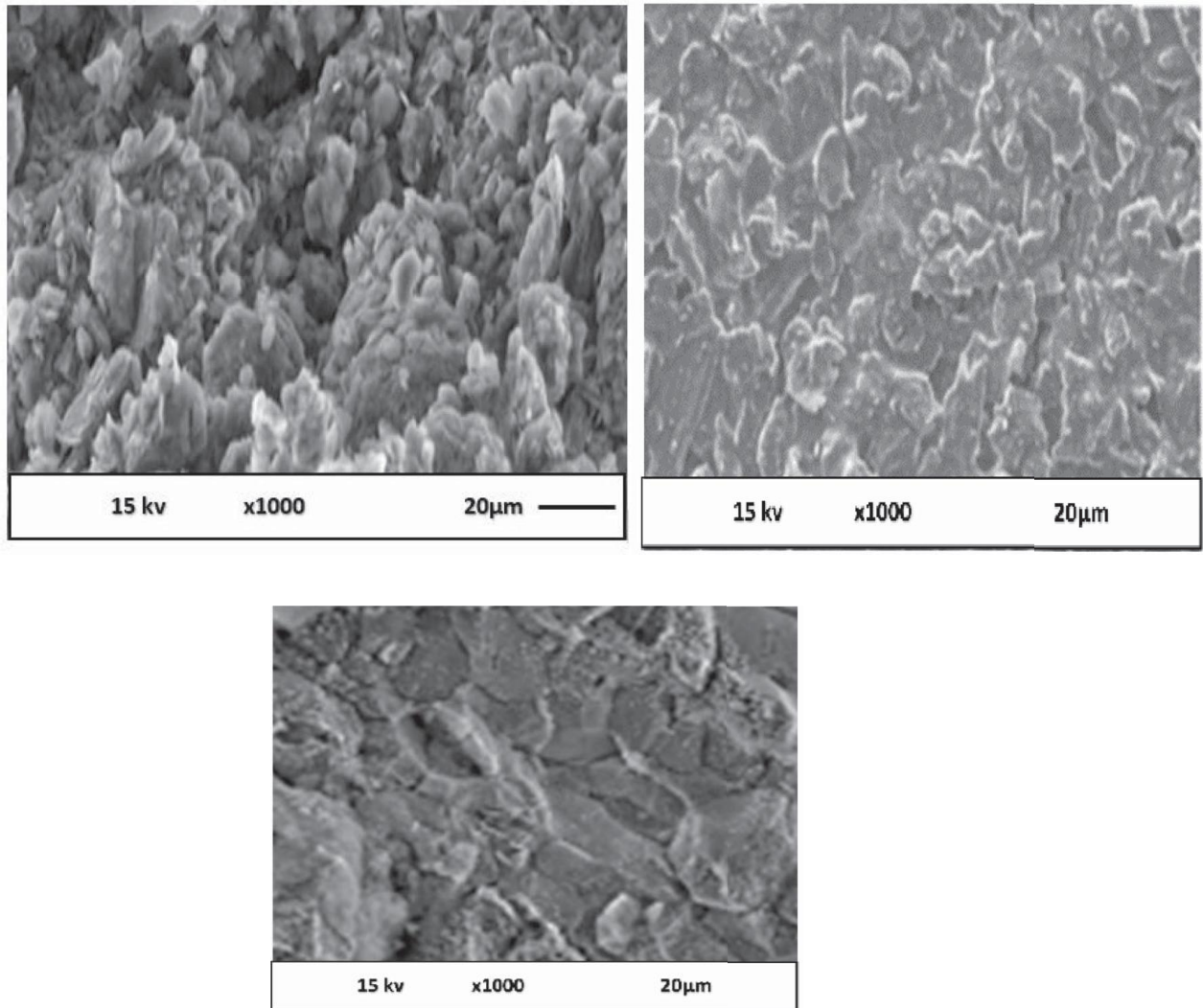


Fig 1: SEM images of ACRH: (a) prior adsorption; (b) Post adsorption of MBD; and (c) Post adsorption of SD

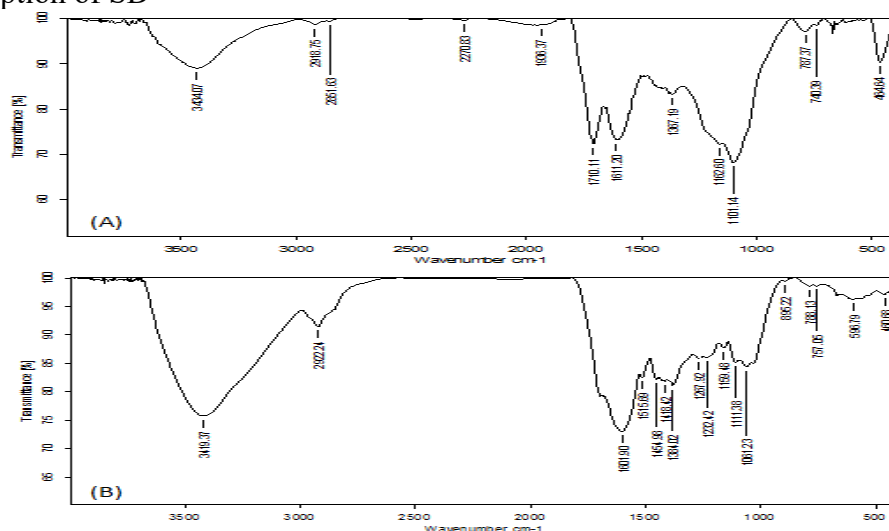


Fig 2. FTIR analysis of RHAC : (a) Prior to adsorption; and (b) Immediately after adsorption

2.2 Batch Adsorption

The capacity of the synthesized ACRH in adsorbing cationic dyes (MBD and SD) and extracting them from the solution was evaluated by batch adsorption experiments. Every test was run in a 1-liter beaker. Synthesized solutions with predefined concentrations of MBD or SD and deionized water were mixed with a certain dosage of ACRH. Either 0.1N NaOH or 0.1N H₂SO₄ was used to change the pH of each solution. With a magnetic stirrer set at 250 rpm, the solutions were stirred. Following agitation, syringe filters with 0.45 µm hole sizes were used to filter the samples. UV spectroscopy was used to measure the absorbance in order to determine the amounts of MBD and SD in the solutions.

The capacity of removal of the dyes was calculated by comparing the initial as well as final concentration of dye in the solution. Removal rate % = $\frac{C_0 - C_t}{C_0} \times 100$ -----1

Where,

C₀ = concentrations of dyes at the times zero

C_t = concentrations of dyes at the times t

t=time.

2.3 Models for Isotherm

Adsorption models for isotherm give important insights into the kinetics of the adsorption process, surface properties, and adsorbent effectiveness (Kadhum et al., 2021b).

The equilibrium adsorption of dye molecule by ACRH is described by the Langmuir model, which focuses on monolayer adsorption, in which an adsorbate molecule single-layer develops on the adsorbent surface. The linear version of the Langmuir formula is (Langmuir 1916).

$$\frac{C_{eq}}{q_e} = \frac{1}{q_m} C_{eq} + \frac{1}{K_L q_m} \dots\dots\dots 2$$

Langmuir isotherm's basic properties can be elucidated by the separation factor RL, given by:

$$R_L = \frac{1}{1 + K_L C_0} \dots\dots\dots 3$$

where K_L is the Langmuir adsorption constant (L/mg).

The RL values provide insight into the process of adsorption. If the value of RL is greater than unity then it indicates unsuitable process of adsorption, if the value is equal to zero then it suggests the process as irreversible, if the value is equal to unity, it implies that the form of adsorption is linear and if the value of RL is less than unity then it means that the process of adsorption is suitable

The Freundlich isotherm, proposed by Freundlich in 1926, is an empirical equation used to characterize surface heterogeneity.

$$\ln q_e = \ln k_f - \ln C_{eq} \dots\dots\dots 4$$

By examining the linear connection between ln q_e and ln C_{eq}, which stand for the slope and intercept, respectively, one may determine the Freundlich constants (n and K_f).

As Dada et al. (2012) pointed out, the Tempkin model includes a parameter to assess the interdependency of dye molecule and ACRH particles. According to this concept, adsorption heat reduces with coverage in a linear rather than a logarithmic manner. The Temkin model's use was noted in.

$$q_e = \beta \ln \alpha + \beta \ln C_e \dots\dots\dots 5$$

The slope and intercept of the plot of q_e vs ln C_e are used to compute the values of α and β, respectively.

Table 1: Isotherm constants for sorption onto ACRH

Isotherm Model	Constants	MBD	SD
Langmuir	q_m(mg/g)	202.8	214.5
	b (L/mg)	4.245	6.915
	R_L	0.219	0.307
	R²	0.997	0.998
Freundlich	K_F (mg/g)	10.56	12.96
	N	5.34	6.78
	1/n	0.213	0.209
	R²	0.984	0.9984
Temkin	B_T	0.292	0.324
	K_T (L/mg)	54.678	57.141
	R²	0.985	0.993

2.4 Adsorption Kinetics

Mathematical models of adsorption and desorption rates are included in adsorption kinetics. Usually, these models fall into two categories: diffusion models and adsorption reaction models.

According to Lagergren, a pseudo-first-order kinetic model that illustrates the process adsorption of a substance onto an adsorbent via a first-order process is given by:

$$\ln(q_e - q_t) = \ln q_e - K_1 t \dots \dots \dots 6$$

The second-order model posits that the adsorption rate is constrained by the availability of adsorption sites on the adsorbent.

$$\frac{t}{q_t} = \frac{1}{K^2 q_e^2} + \frac{t}{q_e} \dots \dots \dots 7$$

The sorption diffusion kinetics are explained by the intraparticle diffusion model. It determines whether the rate of sorption processes is governed by intraparticle or external transport.

$$q_t = K_p t^{0.5} + C \dots \dots \dots 8$$

Where K_p (mg/g min^{0.5}) represents the constant rate of intraparticle diffusion, and c denotes a constant. The value of K_p is calculated from the slope of the linear correlation between the quantities of q_t and $t^{0.5}$.

Table 2: Kinetic constants for sorption onto ACRH

Model Constants		
	MBD	SD
Pseudo 1st order		
q _{e,calc} (mg/g)	34.3	101.4
K _f (min ⁻¹)	0.0084	0.0154
R ² (linear)	.983	.983
Pseudo 2nd order		
q _{e,calc} (mg/g)	121.7	189.3
h (mg/g/min)	1.426	1.378
K _s (min ⁻¹)	0.493	0.436
R ² (linear)	1	1

Intra Particle Diffusion		
K_{ip}	3.78	4.56
C_i	80.2	99.8
R^2	0.935	0.975

2.5 Thermodynamic Study

The spontaneity of the process of adsorption may be given by the thermodynamic characteristics of adsorption.

$$\ln K_D = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \dots\dots\dots 9$$

The slope and intercept of the linear connection between $\ln K_D$ and $1/T$ may be used to get ΔG° , whereas the equation can be used to derive ΔH° and ΔS° .

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \dots\dots\dots 10$$

Table3. Thermodynamic properties

Dye	T(K°)	ΔG°	ΔH°	ΔS°	R^2
MBD	298	-4.53	83.3	0.296	.985
	308.	-7.42			
	318	-10.42			
SD	298	-3.68	78.5	0.277	.993
	308	-6.48			
	318	-9.25			

2.5 Regeneration Study

To investigate the reusability of adsorbent (ACRH), batch tests were carried out with an adsorbent dosage of 0.5 g and a dye concentration (MBD or SD) of 50 mg/L, with agitation set at 250 rpm and a temperature of 25°C. After being stirred, the material was centrifuged, filtered, and then cleaned using distilled water and 0.1 M NaOH. The adsorbent was then dried after that. There were six iterations of this entire procedure.

Results and Discussion

3. Characterization of ACRH

As shown in Fig. 1, SEM was used to analyze the surface texture, content, and structure of activated carbon from rice husk (ACRH) both before and after the adsorption of methylene blue dye (MBD) and safranin dye (SD). The form of the ACRH particles was found to be uneven in the SEM picture [Fig. 1a)], suggesting a heterogeneous surface with a microrough roughness that may improve adsorption effectiveness. Significant alterations were seen following the adsorption of MBD and SD. As seen in Figs. 1(a) and 1(b), the surface of ACRH had a smoother appearance with fuzzy pores. Similar findings of different colors adhering to rice husk have been documented.

Materials of all kinds, including organic, polymeric, and even inorganic compounds, may be identified via FTIR analysis. The surface functional groups of ACRH are visible in the FTIR spectra, which are shown in Fig. 2, both before to and following the adsorption of MBD and SD. The regions between 3,365 and 3,391 cm^{-1} were found to include the strongest and broadest peaks in Figures 2(a) and 2(b).

Peaks measured at 2,926.11, 2,862.46, 1,735.99, and 1,612.45 cm^{-1} were linked to the following reactions: stretching of COO^- anions, bending of OH, C–H and C=O stretching in carboxylic acids or esters, and C–H stretching in alkanes (Memon et al., 2008). The peaks seen at 1,064–1,247 cm^{-1} are linked to C–O stretching of ethers or esters in both situations, both before and after adsorption. Significant changes in several of the peaks in Fig. 2(b) are probably caused by the chemical interactions that form between the surface functional groups on ACRH and MBD or SD (Sartape et al., 2017). ACRH has a significant concentration of hydroxyl and carboxyl groups, which are probably from cellulose, as well as amines from hemicellulose, according to FTIR spectra investigation (Oyekanmi et al., 2019). The composite character of ACRH and its potential as an efficient bioadsorbent are highlighted by the occurrence of discrete peaks linked to different functional groups (Dahiru et al., 2018). As seen in Fig. 5, the pH_{pzc} for ACRH was found to be 5.6. The surface charge of ACRH becomes negative as the solution's pH rises over pH_{pzc}. Thus, when pH values rise (>5.6), the adsorption of cationic dyes (such MBD and SD) onto ACRH becomes more intense because of the stronger electrostatic interaction between them.

Batch Experiments

2.2.1 Time Effect

Over the course of 60 minutes, the contact time effect on the effectiveness of methylene blue dye (MBD) and safranin dye (SD) removal was examined. The starting conditions were pH 5, 500 mg/L of ACRH, and 50 mg/L of initial dye concentration, as shown in Fig. 3. The graph shows that during the first ten minutes, the removal rate increased quickly. Furthermore, there is an obvious positive association between the dye clearance rate and contact time. The start of adsorption equilibrium was shown by the fact that after 30 minutes, the clearance rate did not significantly rise further. Both dyes exhibited the same behavior, with very little variation in their elimination rates. This finding makes sense since, beyond this point in time, there are much fewer potential vacant binding sites (Albayati et al., 2017).

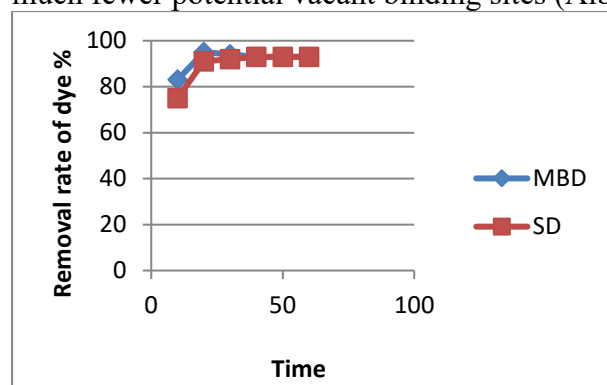


Fig3: Contact time effect

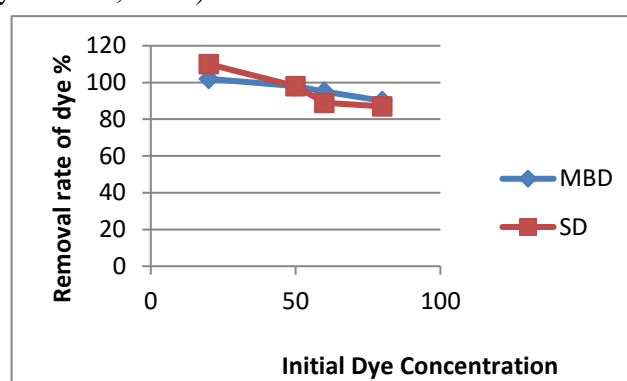


Fig4: Initial dye concentration effect

2.2.2 pH effect

Because it affects the adsorbent's surface characteristics, the parameter pH of a solution is one of the utmost crucial factors that affects the capacity of adsorbent. Examining the adsorption throughout a pH range of 4 to 9, with beginning circumstances set at a dye concentration of 50 mg/L, ACRH dosage of 500 mg/L, and contact period of 30 minutes, allowed us to identify the ideal pH values. The effects of the solution's initial pH on the adsorption of safranin dye (SD) and methylene blue dye (MBD) onto ACRH are shown in Fig. 6. Notably, the adsorption of dyes increased as pH levels rose, peaking at pH 9 for MBD and between pH 7 and pH 9 for SD. This phenomenon may be explained by the fact that, in acidic circumstances (high H^+ ion concentration), cationic dye molecules compete with H^+

ions to attach to the adsorbent surface, which lowers adsorption effectiveness. On the other hand, the ACRH surface becomes negatively charged at higher pH values (low H^+ ion concentration), which increases the electrostatic interaction between the ACRH surface and cationic MBD and SD molecules. As a result, adsorption improves in efficiency, increasing the rate at which dye is removed.

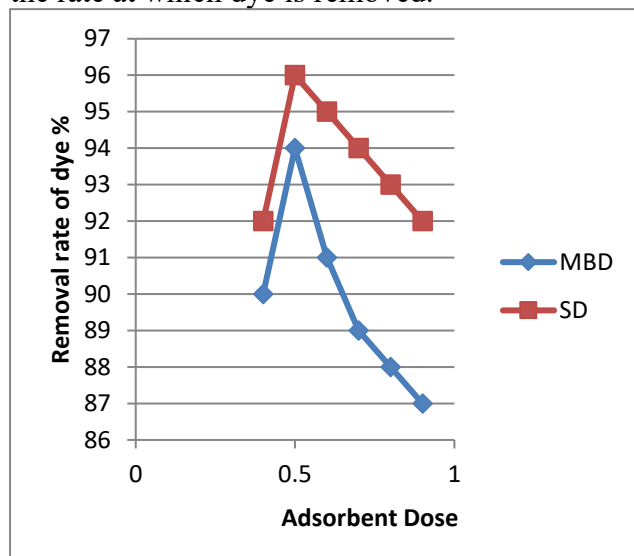


Fig5:Effect of adsorbent dose

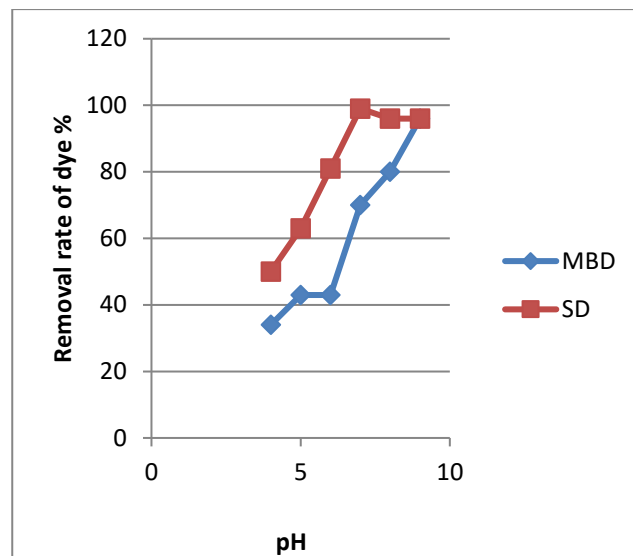


Fig6:Effect of pH

2.2.3 ACRH (Adsorbent) Dose Effect

ACRH dose ranging from 0.4g/L to 0.9g/L, the effect of activated carbon obtained from rice husk (ACRH) dosage for the ability of adsorption of methylene blue dye (MBD) and safranin dye (SD) onto ACRH was investigated. The batch trials started with a pH of 9, a contact period of 30 minutes, and a dye concentration of 50 mg/L. When the adsorbent dose was less than 0.5 g/L, as shown in Fig. 5, the removal rate showed a positive connection with the dose. But when the dose above 0.5 g/L, an inverse link was seen. These results might be explained by any or both of the following factors. First off, larger adsorbent dosages may cause particle aggregation, which would decrease the adsorbent's specific surface area (ACRH) and lengthen the diffusion channel. Secondly, unsaturated adsorption sites could occur at high ACRH dosages and low dye concentrations. when a result, the equilibrium adsorption and reduction capacity decrease when the adsorbent (ACRH) dosage approaches a certain threshold (Allabaksh et al., 2010).

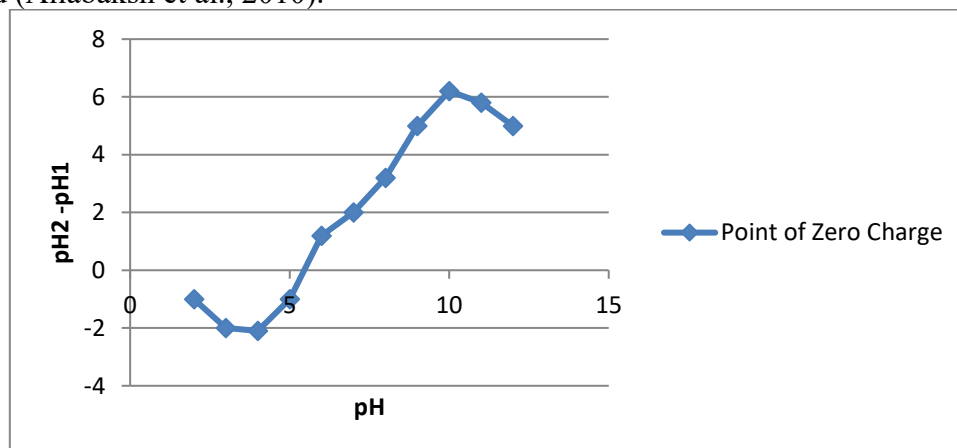


Fig 7:Point of zero charge of ACRH

2.2.4 Effect of concentration of dye (initial)

The concentration of adsorbate used in the beginning of the process is a crucial consideration when assessing adsorbents in research. The efficacy of 20–80 mg/L dye concentrations at the beginning stage was examined using adsorbent dose of 0.5 g/L; contact time was taken as 30 min and pH was considered 9, as seen in Fig. 4. As the dye concentration was increased from 20 mg/L to 80 mg/L, the removal effectiveness of safranin dye (SD) and methylene blue dye (MBD) was found to be decreased from 99% to 91% and 99.5% to 88% respectively using ACRH.

2.3.1 Models for Adsorption Isotherm

Isotherm models showing the adsorption of safranin dye (SD) and methylene blue dye (MBD) onto activated carbon derived from rice husk (ACRH) are shown in Figures 8–10. We used the Freundlich, Temkin, and Langmuir models to find the model that best matches the experimental results. Table 1 lists the parameters for each of the three models. The table clearly shows that the Langmuir isotherm for ACRH for both dyes has a greater correlation coefficient (R^2) than the Freundlich and Temkin isotherms. This shows that the Langmuir isotherm may fit the adsorption data well, suggesting a monolayer adsorption on the sorbent with a homogeneous activity distribution (Pathania et al., 2017).

The fact that the RL value in Table 1 is between 0 and 1 suggests that the adsorption of MBD and SD onto ACRH is advantageous. For MBD and SD, the Freundlich constant n is more than 1, indicating considerable adsorption properties (Yao et al., 2011).

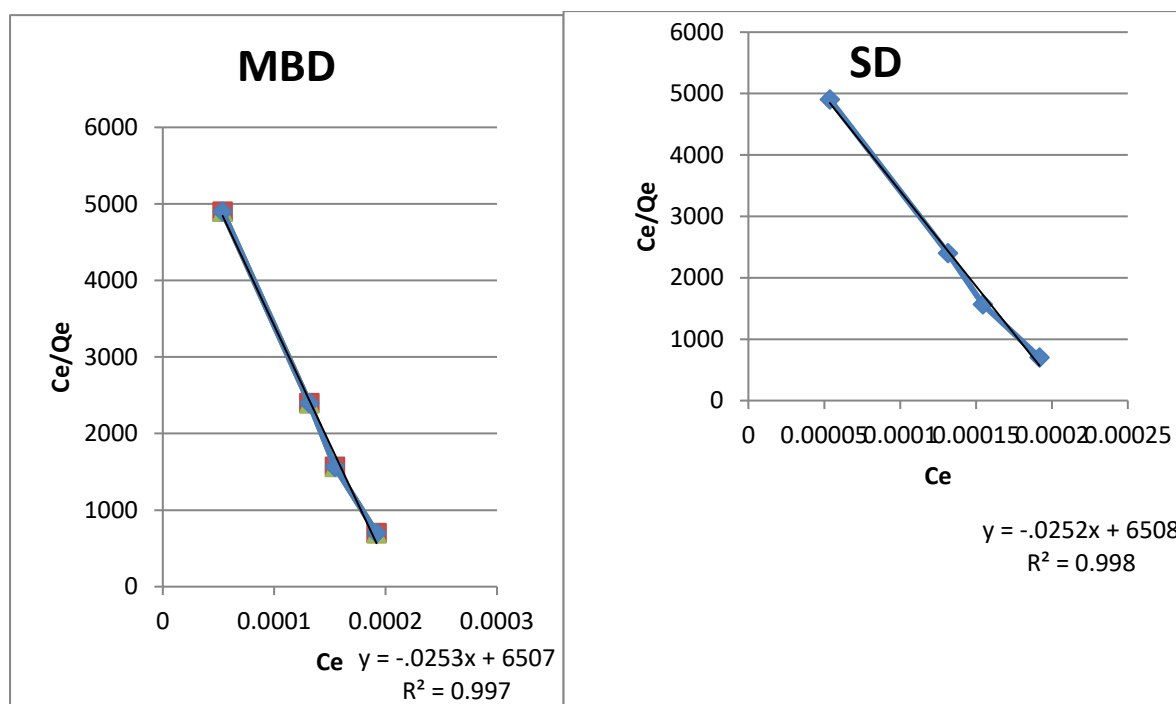


Fig 8: Langmuir isotherm models for (a) MBD; and (b) SD.

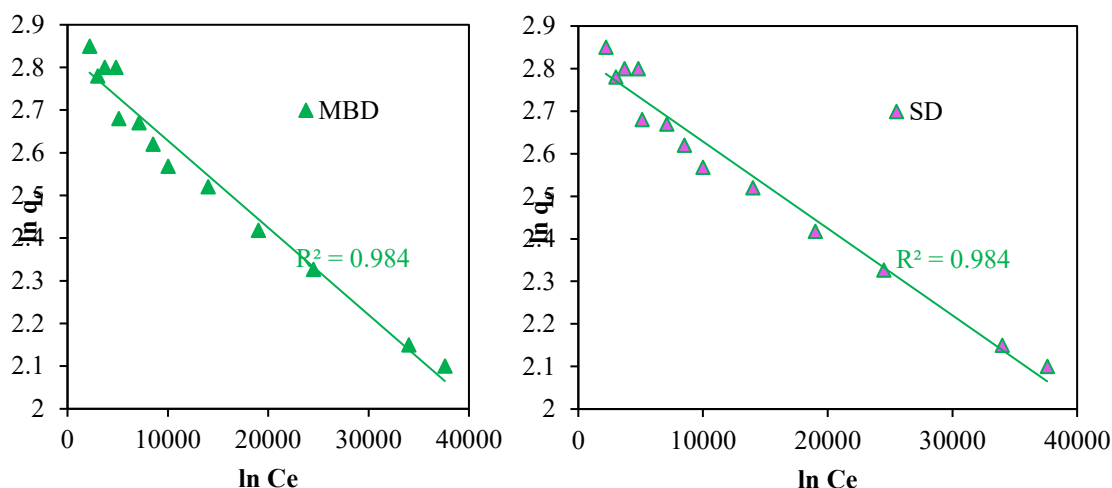


Fig 9: Freundlich isotherm models for (a)MBD;and(b)SD.

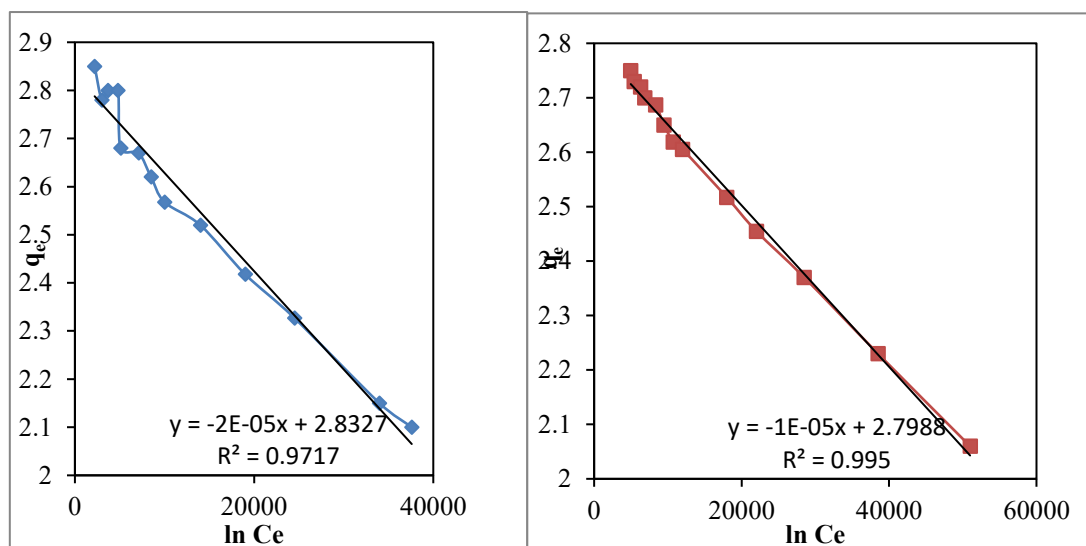


Fig 10: Temkin isotherm models for (a)MBD;and(b)SD.

2.4.1 Adsorption Kinetics

As indicated in Figs. 11–14, respectively. The several parameters and coefficients of correlation (R^2) for both 1st and 2nd order kinetic model and intraparticle diffusion models are given in Table 2. The table shows that for both dyes, the pseudo-second-order model's R^2 value is 1, which is higher than the R^2 values of the other two models. This demonstrates that the pseudo-second-order model more closely matches the adsorption data of cationic dyes (MBD and SD) onto ACRH. The fitting of pseudo 2nd order kinetic model confirms that the rate-limiting phase involves chemisorptions.

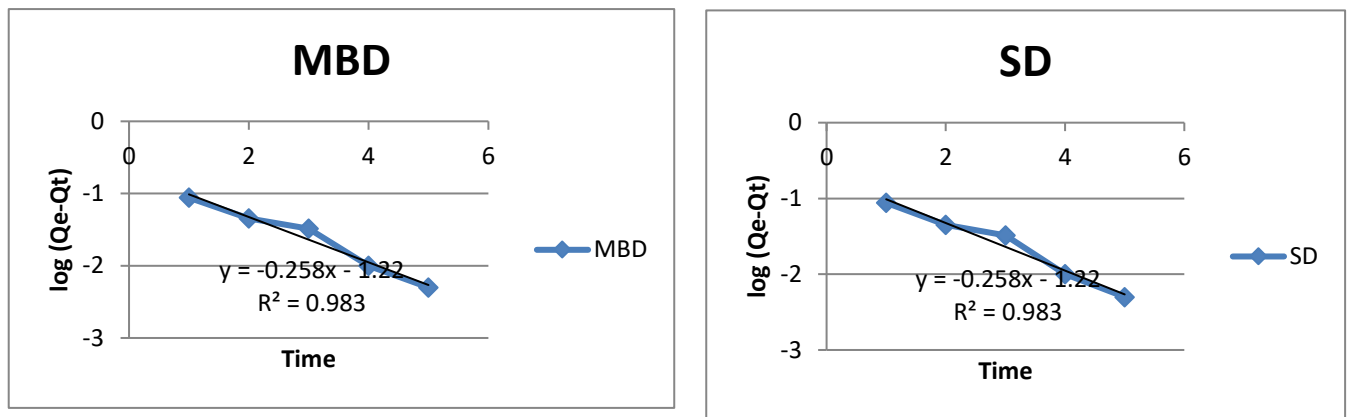


Fig11:a) Pseudo first-order kinetic model for adsorption of MBD (b)Pseudo first-order kinetic model for adsorption of SD.

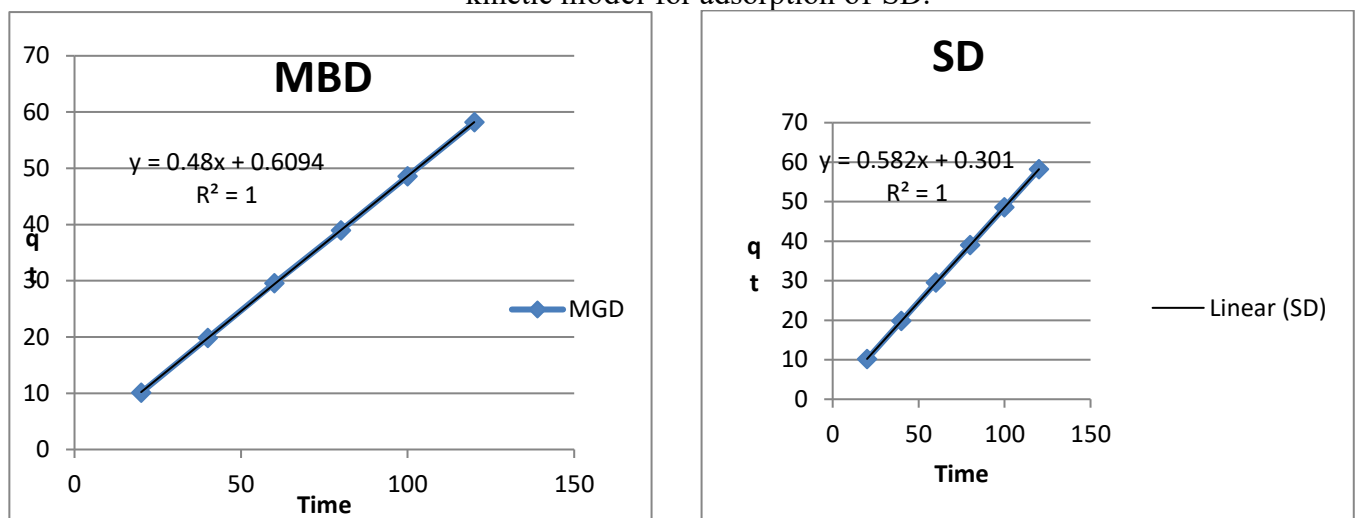


Fig 12:a) Pseudo second-order kinetic model for adsorption of MBD(b)Pseudo second-order kinetic model for adsorption of SD.

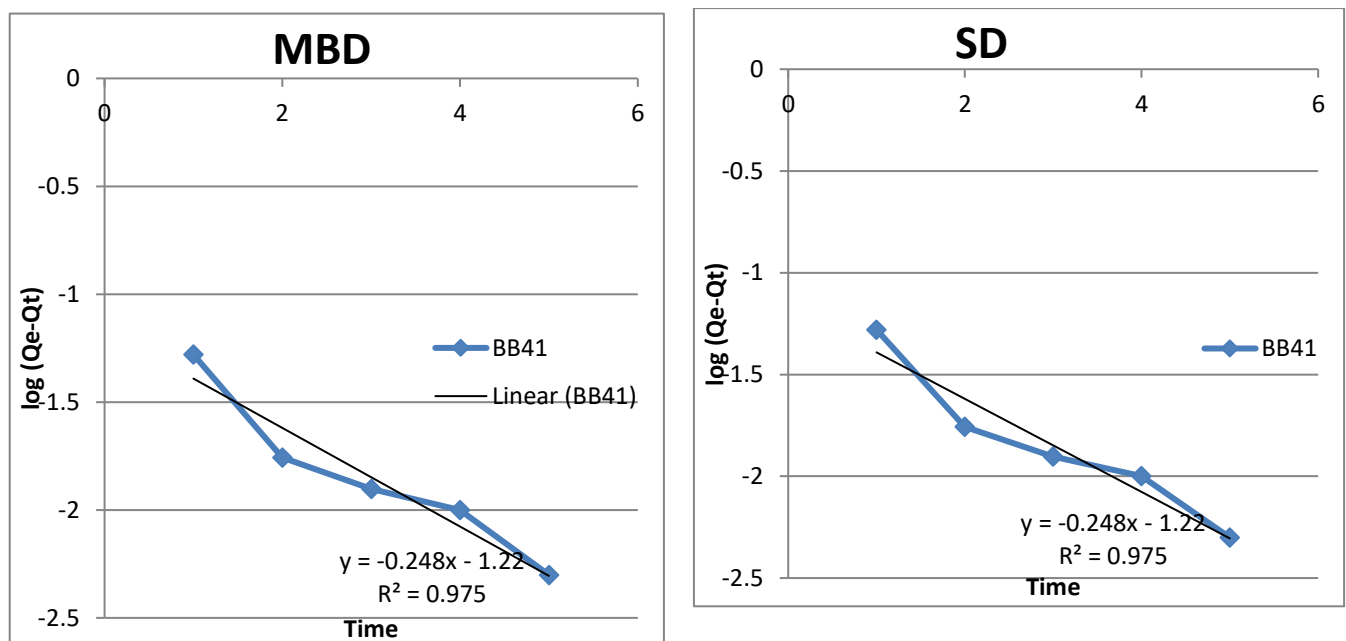


Fig 13:Intraparticle diffusion model for the adsorption of (a)MBD;and(b)SD.

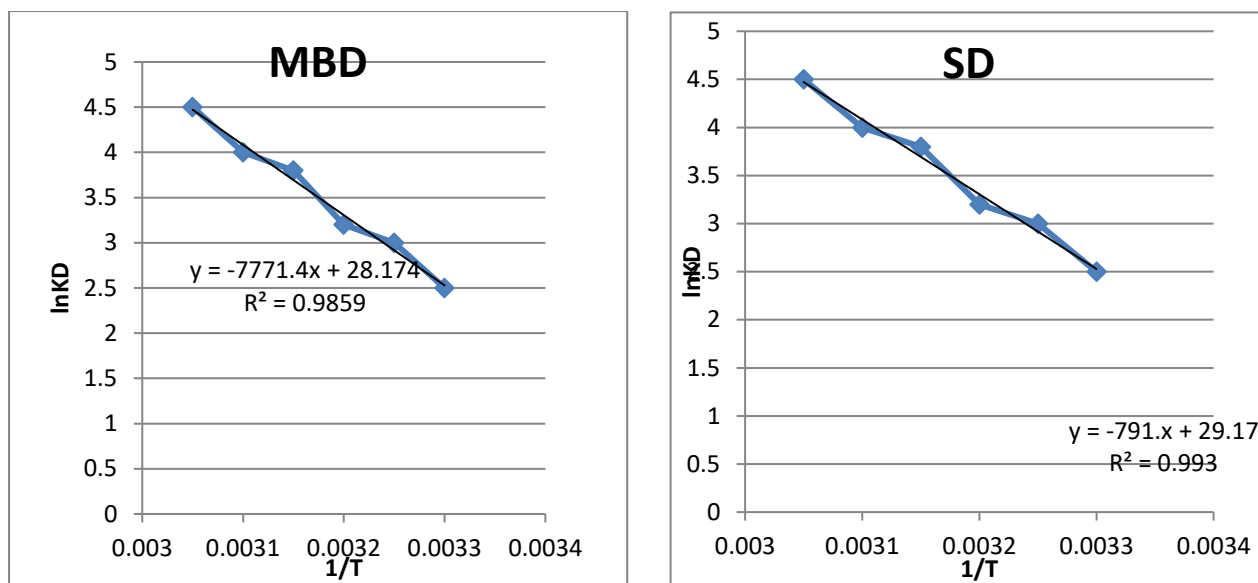


Fig 14. Van't Hoff relation of (a)MBD;and(b)SD.

2.5.1 Thermodynamics

At 25, 35, and 45 degrees Celsius, the thermodynamic parameters for the adsorption of safranin dye (SD) and methylene blue dye (MBD) onto activated carbon from rice husk (ACRH) were computed. Fig. 15 shows the linear connection between $\ln KD$ and $1/T$. Table 3 provides a summary of the determined thermodynamic parameters. The results of the investigation show that MBD and SD adsorb endothermically on ACRH. Furthermore, the negative values of ΔG° imply that both dyes' adsorption processes are spontaneous.

4. Adsorption Mechanism

In general, the adsorption system belongs to one of two categories: chemisorption or physisorption. The adsorption of methylene blue dye (MBD) and safranin dye (SD) onto activated carbon from rice husk (ACRH) showed chemical adsorption, according to the findings of the adsorption kinetics investigation. The primary mechanism for MBD and SD adsorption from aqueous solution with ACRH is the active surface functional groups of ACRH, which include carboxylic acid, aromatics, hydroxyl, and phenols. FTIR spectrum research has established the existence of these functional groups. The surface functional groups of ACRH, MBD, and SD are anticipated to interact in a number of ways during the adsorption process. The might involve hydrogen bonding, electrostatic attraction the reaction between ACRH and dye molecule, and π - π stacking.

5. Comparative Study

The results of this study demonstrate the exceptional adsorption capacity of rice husk activated carbon (ACRH) against methylene blue dye (MBD) and safranin dye (SD). With respect to MBD and SD, ACRH is among the best-performing adsorbents, exhibiting high adsorption capabilities, according to the data in Table 4. ACRH has a number of benefits over other adsorbents, such as being less expensive, simpler to prepare, and environmentally friendly.

Table4.Comparative study of the performance of adsorption of MBD and SD by several biosorbents

Dye	Adsorbent	Capacity(mg/g)	Reference
MBD	Oxide of Graphite	29.42	Farghali et al.(2013)
	Oxide of Graphite	28.5	Sharma et al.(2014)
	Bamboo	15.5	Atshan(2014)
	Activated carbon from Brachy chiton populneus shells fruit	67.93	Rida et al.(2020)
	Activated carbon from Cryogenic crushed waste tires	71.43	Belgacem etal.(2022)
	Rice Husk	41.2	Present study
SD	Shale of Bituminous Stems of Rice	55	Müftüoglu et al. (2003)
	Husk of Rice	17.7	Faraji et al.(2015)
		22.6	
		24.4	
		34.6	Karaj(2016)
	Seed of Salvia	19.16	Hamzezadeh etal.(2020)
	shell of Pistachio	21.83	Şentürk and Alzein(2020)
	Rice husk	39.06	Present study

5. Conclusions

This work demonstrates the effective removal of cationic dyes, methylene blue dye (MBD) and safranin dye (SD), from synthetic effluent using biowaste, especially rice husk. The surface structure of activated carbon derived from rice husk (ACRH) is microrough and heterogeneous, as evidenced by the analysis of SEM pictures. ACRH's FTIR spectra also revealed the presence of amines from hemicellulose and a significant quantity of carboxylic and hydroxyl groups from cellulose. These tests conducted in the lab have demonstrated that ACRH has qualities that make it a perfect adsorbent.

Adsorption equilibrium was attained after 30 minutes, according to the adsorption experiment results, with a pH of 9 being ideal for MBD and a range of 7 to 9 for SD. The ideal ACRH dosage was discovered to be 0.5 g/L, while the pH_{pzc} of ACRH was found to be 5.6. Furthermore, the rate at which MBD and SD were eliminated by ACRH decreased as dye concentration rose. Under optimal working conditions, ACRH was able to achieve a clearance rate of more than 99% for both MBD and SD. The Langmuir isotherm model was shown to be the most effective model due to its higher correlation coefficient (R^2) value. The adsorption of MBD and SD onto ACRH is fully described by the pseudo-second-order kinetic model. Furthermore, it was discovered that the adsorption process was spontaneous and endothermic.

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