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SCHIFF BASE-MODIFIED CHITOSAN/ACTIVATED CARBON COMPOSITE FOR SIMULTANEOUS DYE REMOVAL AND DISINFECTION OF WASTEWATER

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ABSTRACT

A composite adsorbent material was developed by embedding activated carbon into a chitosan polymeric matrix cross-linked with epichlorohydrin and modified via Schiff base formation for the effective methylene blue dye removal and disinfection of wastewater. Characterisation of the adsorbent included FTIR, BET, FE-SEM, and pHpzc analyses. Adsorption experiments were conducted to evaluate and optimise the composite's capacity for methylene blue removal, considering process variables, namely, solution pH (2-11), adsorbent dosage (0.08-0.46 g), temperature (303-323 K), and contact time (5-60 min). Kinetic and isothermal models were applied to analyse the adsorption process, and the results showed that the composite exhibited high adsorption efficiency, fitting to the Langmuir adsorption isotherm and pseudo-second-order kinetics, and remained effective after four reuse cycles. Also, the composite was subjected to antibacterial tests against *Escherichia coli* and *Staphylococcus aureus*. The results demonstrated significant antimicrobial properties, suggesting dual functionality in both dye removal and disinfection of wastewater microorganisms.

Keywords: Methylene Blue, Epichlorohydrin, Chitosan Schiff Base, Activated Carbon, Adsorption.

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INTRODUCTION

Water pollution caused by synthetic dyes remains a serious global environmental issue. Among them, methylene blue (MB), a stable cationic dye, poses significant health and environmental hazards due to its toxic, carcinogenic, and mutagenic nature.[1] With millions of tons of dye-containing wastewater released annually, addressing dye contamination has become an urgent challenge in achieving sustainable water management. Adsorption is a preferred, cost-effective dye removal method, known for its simplicity and potential for adsorbent reuse.[2-4] Biopolymer-based composites are effective adsorbents for wastewater treatment due to their biocompatibility, functional group richness, high adsorption capacity, sustainability, biodegradability, chemical stability, antimicrobial properties, and non-toxicity. As a result, recent research focuses on synthesising and optimising such composites using biosorbents as core materials.[5,6] Chitosan (CH), a porous biopolymer, is notable for its reactive $–NH_2$ and –OH groups that enhance adsorption via electrostatic and hydrogen bonding interactions.[7-9] However, native CH suffers from low mechanical strength and chemical instability, especially in acidic conditions.[10,11] Modifications such as crosslinking, Schiff base formation, and combining with materials like activated charcoal have improved its properties.[12-15] In this study, CH was modified and combined with activated carbon (AC) from sugarcane bagasse to create a composite adsorbent for MB removal. This work aligns with Sustainable Development Goal 6 (Clean Water and Sanitation) and Goal 12 (Responsible Consumption and Production) by promoting eco-friendly materials for wastewater treatment. Adsorption efficiency was

tested under varying conditions, and kinetics, thermodynamics, and antimicrobial properties were evaluated to determine its multifunctional potential.

EXPERIMENTAL

Material and Methods

CH (deacetylation >75%) and 5-bromothiophene-2-carboxaldehyde were obtained from Sigma Aldrich (India), while other reagents, including H_3PO_4, CH_3COOH, epichlorohydrin, NaOH, HCl, acetone, and MB dye, were from Spectrochem (India). Sugarcane bagasse was collected locally in Mangalore, Karnataka.

Preparation of the Adsorbent

AC: Sugarcane bagasse was first rinsed with tap water, followed by distilled water, dried at 105°C for about 24 h, and impregnated with 30% H_3PO_4(ratio 1.5) for 6 h at 30°C. The impregnated bagasse was then dried, carbonised at 500°C for 45 min, washed to pH 6-7, and dried again.

Crosslinked CH Schiff Base (CCHSB): As per a reported method[16], 1.0 g CH was dissolved in 5% CH_3COOH (100 mL) at 30°C for 1h, crosslinked with 1mL epichlorohydrin, then reacted with 0.99g 5-bromothiophene-2-carboxaldehyde at 60°C for 6 h and stirred for 24 h.

CCHSB-AC composite (CCHSBAC): 1.0 g AC was introduced to the reaction mixture consisting of CCHSB, stirred gently for 6 h, precipitated with acetone, followed by washing, filtration, and drying at 60°C for about 24 h.

Detection Method

FTIR spectrum of CCHSBAC were obtained using a PerkinElmer Spectrum Two spectrometer. FESEM-EDX (Zeiss Ultra 55) analysed surface morphology and elemental composition. Thermal stability was tested via TGA (PerkinElmer TGA 2000) from 0-600°C at 10°C min^{-1}. BET analysis (BELSORP MINI X) measured pore volume and surface area at 77 K. Antibacterial activity was assessed using the broth dilution method.

RESULTS AND DISCUSSION

Characterisation of CCHSBAC

FTIR Analysis

The FTIR spectrum of CCHSBAC (Fig.-1) shows a broad band at 3311.04 cm^{-1} for –OH and $–NH_2$ stretching, and a weak band at 2875.58 cm^{-1} for $–CH_2$. The peak at 1652.41 cm^{-1} indicates Schiff base formation, while bands at 1372.47, 1020.21, and 895.80 cm^{-1} correspond to C-N, C-O-C, and CH bending in the chitosan ring.

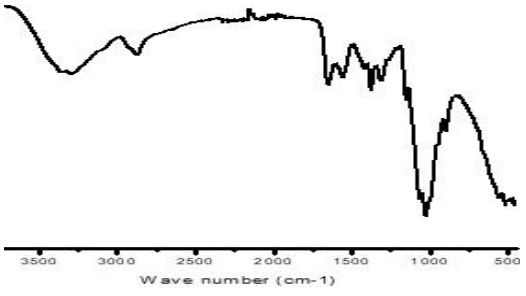

Fig.-1: FTIR Spectrum of CCHSBAC

Nitrogen Sorption Analysis

Samples were nitrogen-degassed at 110°C for 3 hours to remove moisture. The Type I isotherm indicates microporosity (<2 nm). Table-1 shows that CH modification and AC addition improve microporosity and dye uptake.

Table-1: Surface Properties of CH and CCHSBAC Measured from N_2 Adsorption-Desorption Isotherm Data

Sample	BET surface area (m^2 g^{-1})	Total volume of the pore (cm^3 g^{-1})	Avg. diameter of the pore (nm)	V_m (cm^3 g^{-1})
CH	8.7803	0.002171	0.989	2.0173
CCHSBAC	102.9	0.051489	1.438	23.641

SEM and EDX Analysis

The surface morphology and composition of AC, CH, and CCHSBAC were studied using FESEM-EDX. SEM images (Fig.-2a–c) show that AC has cavities from phosphoric acid evaporation, CH has a rough, poreless surface, and CCHSBAC exhibits an irregular, agglomerated morphology, indicating AC integration. EDX analysis confirms bromine in CCHSBAC, supporting Schiff base formation.

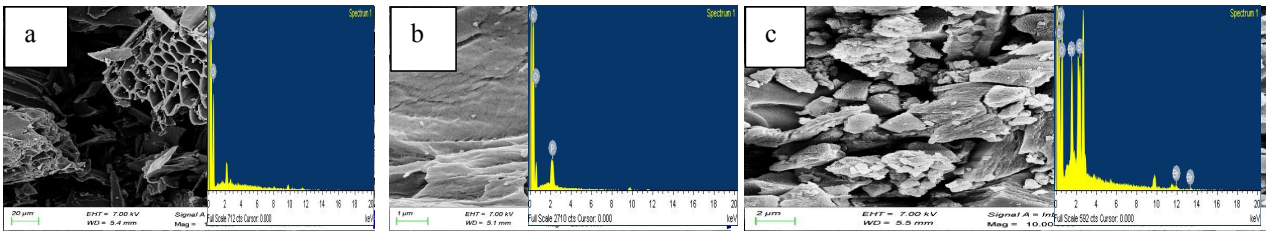

Fig.-2: SEM images and EDX Spectra of (a) AC, (b) CH, and (c) CCHSBAC

Batch Adsorption Study

Effect of Adsorbent Dose and Solution pH

The effect of CCHSBAC dosage on MB removal was evaluated using 0.08-0.46 g under fixed conditions (MB 100 mg L^{-1}, 100 mL, pH 8.0, 60 min, 303 K). As shown in Fig.-3(a), removal efficiency increased from 53.5% to 98.27% up to 0.24 g due to more active sites, with negligible improvement beyond this dose; thus, 0.24 g was chosen as optimal. The pH effect (2-11) ~~was~~ studied at 0.24 g adsorbent dose showed enhanced dye removal from pH 2 to 6 and maximum adsorption at pH ~8.0 (Fig.-3b). The point of zero charge (pHpzc) was found to be 5.74 (Fig.-3c); below this, the surface repels MB dye, while above it, negative charge enhances adsorption via electrostatic attraction.

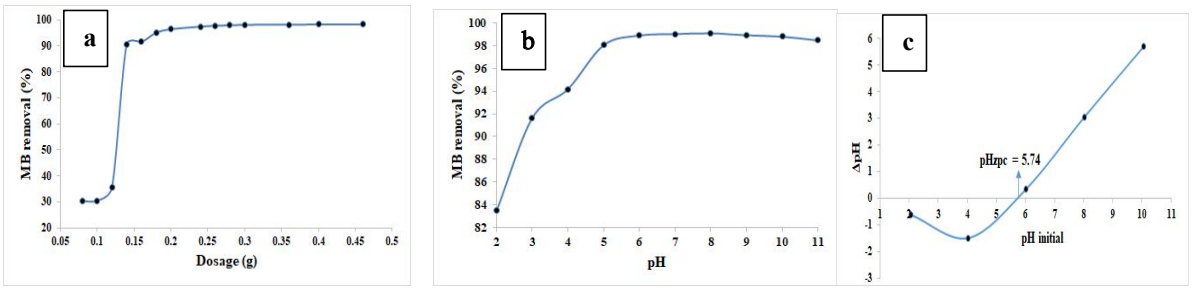

Fig.-3: (a) Effect of CCHSBAC Dosage; (b) Effect of pH of the Solution on MB Percentage Removal; (c) pHpzc of CCHSBAC

Influence of Temperature and Contact Duration

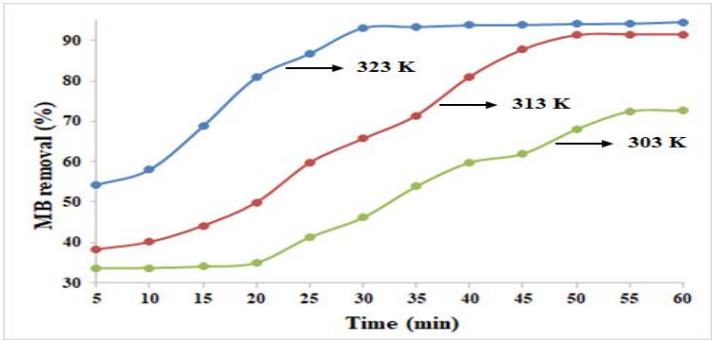

Fig.-4: Effect of Time of Contact on MB Adsorption at Various Temperatures

The impact of temperature (303–323 K) on MB adsorption by CCHSBAC was examined at pH 8.0, 100 mg L^{-1} adsorbate, and 0.24 g L^{-1} adsorbent. Adsorption rose from 65% to 95% with temperature, indicating an endothermic process. As shown in Fig.-4, rapid uptake occurred in the first 30 minutes, reaching saturation by 40 minutes.

Adsorption Kinetics

The pseudo-first-order (PFO)[17] and pseudo-second-order (PSO)[18] kinetic models were applied in a time-dependent study to best describe MB adsorption on the CCHSBAC surface. Equations-1 and 2 show the non-linear mathematical formulas of the PFO and PSO kinetic models, respectively.

$$q_t = q_e(1 - e^{-k_1 t}) \qquad (1) \qquad q_t = \frac{q_e^2 k_2 t}{1 + q_e k_2 t} \qquad (2)$$

Here, q_t and q_e (mg g^{-1}) denote dye uptake on CCHSBAC at time t and at equilibrium, respectively, while k_1(min^{-1}) and k_2 (g mg^{-1} min^{-1}) are rate constants of the PFO and PSO models. The adsorption kinetics of MB dye onto CCHSBAC fit the PSO model better than the PFO, with a higher R^2 of 0.999 (Fig-5) versus 0.734, indicating chemisorption (k_2= 0.186 g mg^{-1} min^{-1}) as the main mechanism.

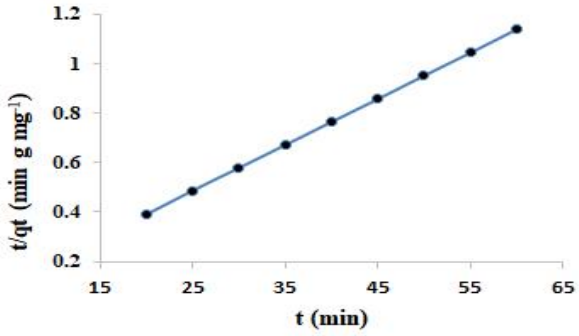

Fig.-5: PSO kinetic Plot for the Adsorption of MB Dye on CCHSBAC

Adsorption Isotherms

To estimate the nature of the interactions between the dye molecules and the CCHSBAC adsorbent, Langmuir[19] and Freundlich[20] isotherm models were applied in non-linear mathematical expressions as shown in equations 3 and 4, respectively.

$$\frac{C_e}{q_e} = \frac{1}{q_m} C_e + \frac{1}{q_m b} \qquad (3)$$

Where q_e (mg g^{-1}): quantity of adsorbed dye at equilibrium (adsorption capacity of CCHSBAC) and C_e (mg L^{-1}): residual MB concentration at equilibrium after adsorption. b (L mg^{-1}) and q_m (mg g^{-1}): Langmuir constant and maximum adsorption capacity of CCHSBAC, respectively.

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

K_F (mg g^{-1}) and n are constants that give an idea of the degree of adsorption and the nonlinearity between the adsorption process and dye concentration. A value of *n* between 1 and 10 is considered as favourable adsorption.

A constant separation factor, R_L describes characteristics of the Langmuir isotherm model, quantified from eqn.-5.

$$R_L = \frac{1}{1 + bC_0} \qquad (5)$$

Where C_o (mg L^{-1}): initial dye concentration and b: Langmuir constant (L mg^{-1}).

The value of R_L predicts the nature of the isotherm:

$R_L = 0$ irreversible; R_L=1 linear; $R_L > 1$ unfavorable adsorption; $0 < R_L < 1$ favorable adsorption

The Langmuir isotherm plot (Fig.-6) and data (Table-2) show a strong fit (R^2 = 0.999), suggesting adsorption occurs in a single molecular layer. The estimated maximum adsorption capacity, q_m, reached 39.99 mg g^{-1}, and the R_L value of 0-1 confirms favourable adsorption.

Desorption and Regeneration of CCHSBAC

The regeneration process was repeated four times, and removal percentages over the cycles are shown in Fig.-7. Desorption efficiencies for the 1st to 4th cycles were 83.8%, 80.8%, 71.3%, and 63.0%, respectively. Although efficiency fell below 75% after the second cycle, it remained above 60% through the fourth cycle.

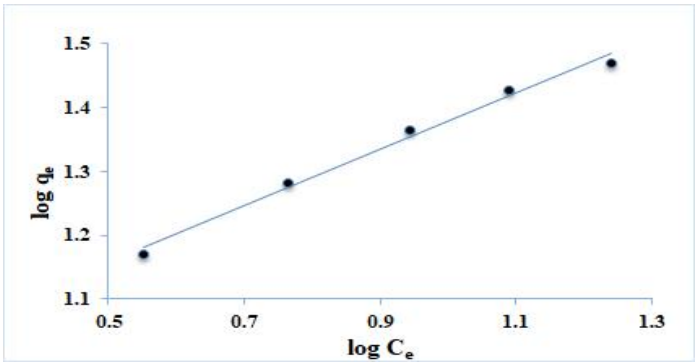

Fig.-6: Langmuir Adsorption Isotherm Plots for MB Dye Uptake onto CCHSBAC

Table-2: Isotherm Data for the Adsorption of MB Dye on CCHSBAC

Adsorption models	Langmuir	q_m (mg g^{-1})	39.99
		b (L mg^{-1})	0.00641
		R_L	0.06
		R^2	0.999
	Freundlich	K_F (mg g^{-1})	8.677
		n	2.276
		R^2	0.989

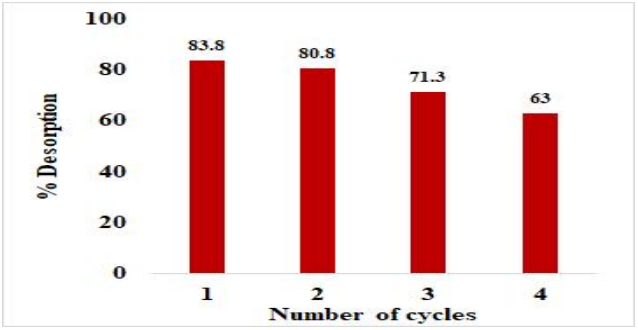

Fig.-7: Desorption of MB Dye Molecules from MB Adsorbed CCHSBAC

Antibacterial Susceptibility Testing by the Method of Broth Dilution

The bacterial inhibition activity of AC and CCHSBAC was assessed in vitro against *Staphylococcus aureus* and *Escherichia coli* using the broth dilution method in Mueller-Hinton broth (25–200 µg/mL). In 96-well plates, 100 µL each of MH broth (OD 0.5 McFarland) and samples were maintained at 37°C for 24 h. Optical density at 570 nm was used to determine the minimum inhibitory concentration (MIC). Figures-8(a) and (b) indicate that CCHSBAC showed stronger antibacterial activity than AC, with lower MIC values for both strains.

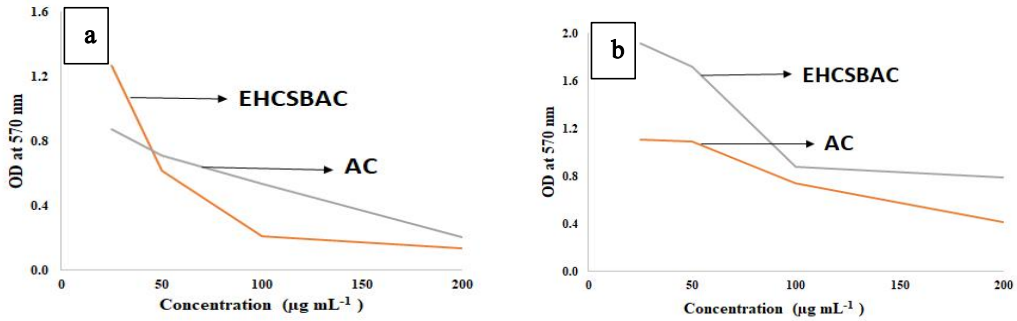

Fig.-8: (a) and (b): Antibacterial Activity of CCHSBAC and AC against *Escherichia coli* and *Staphylococcus aureus,* respectively

CONCLUSION

Epichlorohydrin cross-linked CH-Schiff base/AC composite was synthesised for MB dye removal, showing high adsorption efficiency. The adsorption process adhered to pseudo-second-order kinetic behaviour and confirmed the Langmuir adsorption model, exhibiting a maximum adsorption capacity of 39.99 mg g^{-1}, indicating monolayer adsorption. The addition of AC, crosslinking and modification via Schiff base formation enhanced surface area and active sites of the adsorbent. The composite adsorbent also exhibited potent antimicrobial activity against both Gram-positive and Gram-negative strains, highlighting its dual function in treating dye and microbe contaminated wastewater. Furthermore, the outcomes of this study contribute to SDG-6 (Clean Water and Sanitation) by enabling efficient wastewater purification.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-6: Clean Water and Sanitation*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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