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# Methylene Blue Dye Removal from Aqueous Solution Using Activated Carbon Prepared from Corn Cob Stem: Kinetics, Isotherms and Mechanism Studies

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# ABSTRACT

ORIGINAL STUDY

In this study, a waste-to-wealth work has been proposed where activated carbon was synthesized from waste corn cob stems and used for methylene blue (MB) dye removal. Corn cob stems activated carbon (CCSAC) had been prepared in microwave technique using  $H_3PO_4$  as an activating agent where the impregnation ratio of corn cob stems to  $H_3PO_4$  was 1:4. Then CCSAC characterized by FTIR, BET, XRD, SEM, EDS, and Raman spectroscopy. The CCSAC showed the mesoporous with a good surface area of 54.61 m<sup>2</sup>/g. Around 150 mg adsorbent dose, 120 min contact time, pH 10, and 10 mg/L initial concentration, 98% of MB dye was eliminated in the aqueous environment. The Langmuir adsorption model gave a higher correlation coefficient of 0.90 and showed an adsorption capability of 129.77 mg/g. Again, the MB dye adsorption on the CCSAC surface was purely a chemical interaction as found from the pseudo-second-order kinetics model ( $R^2 = 0.95$ ). Three types of interaction i.e. pi-pi interaction, pore filling, electrostatic attraction and hydrogen bonding was occurred between MB dye and CCSAC adsorbent respectively. The analysis finding indicated that CCSAC is an effective material for MB adsorption from the aquatic environment.

Keywords: Corn cob stem, Dyes, Methylene blue, Adsorption and removal

# 1. Introduction

All living organisms from the micro bacteria to giant blue whales, require water for their existence, and water is also called another form of life [1]. The earth's surface covers one octillion liters of water, however, only 0.3% of fresh water is available for our day-to-day life activities [2]. However, the water quality is decreased by different water pollutants such as heavy metals [3, 4], agricultural waste products [5], pharmaceutical waste [6], and many harmful dyes [7, 8]. Out of all these pollutants, the organic dyes very badly affect the water systems [9]. Many industries such as paper, textiles, food, and printing are using dyes to coloring their products, which

later introduces into the water bodies cause water pollution by decreasing the quality of water [10– 12]. There are so many coloring dyes are presently available in the market i.e. methyl red, malachite green, methylene blue, methyl orange, and congo red [13]. Among all these dyes, methylene blue (MB), which has the chemical formula  $C_{16}H_{18}CIN_3S$  and a greater molar mass of 319.85 g/mol gains visible attraction recent days [14]. It is a dangerous one as it is very toxic, non-biodegradable, and carcinogenic [11]. It also causes allergies, skin diseases, irregular breathing, anemia, depression, discolored urine, and increased blood pressure in humans [15, 16]. Therefore, analysis should be done to eliminate this harmful substance from aqueous environment.

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Fig. 1. Nitrogen adsorption-desorption isotherm curves of CCSAC.

To remove MB dye from aqueous solution, several sophisticated techniques have been used, including ion exchange [17], adsorption [18], coagulation [19], ozonation [20], membrane separation [21]. precipitation [22] and flocculation [23]. Amongst all, adsorption is considered the best method in the removal process because of its eco-friendliness, suitability, and low operational cost [24]. There are several types of adsorbents applies for the removal of MB dye and among them some are metal oxide [25], graphene oxide [26], activated carbon [27], and polymer [28]. The surface area, huge high porosity, and greater binding sites, activated carbon is regarded as the best adsorbent [10]. For the last few years, most researchers have been interested in the agricultural byproduct residues, which act as an eco-friendly source, chief and available everywhere [29]. Many scientists have investigated the activated carbon preparation from agricultural byproducts like mango peels [30], mangosteen peels [31], palm shell [32], carrot [10], etc.

Two methods are employed for producing activated carbon from various materials i.e. physical process

[33] and chemical process [34]. Studies observe that activated carbon produced from chemical methods using activating gents ZnCl<sub>2</sub> [35], HCl [36], H<sub>3</sub>PO<sub>4</sub> [37], KOH [38], K<sub>2</sub>CO<sub>3</sub> [39], and HNO<sub>3</sub> [40] in an inert environment shows very good results. Compared to other activating agents, H<sub>3</sub>PO<sub>4</sub> displays a remarkable effect in activated carbon surfaces with higher porous structure, good surface area, and a large number of binding sites [41]. Again, activated carbon synthesis using H<sub>3</sub>PO<sub>4</sub> has been successfully applied in heavy metals removal [42], dyes removal [37], organic compound removal [41], and supercapacitor application [43] respectively.

In our work, we used the corn cob stems for the synthesis of activated carbon, because it has high heat value, good carbon content amount and easily collected during corn grain harvest [44–47]. Every year, a large amount of about 1.85 billion metric tons of corn cob is produced and its stems are burned or go to the dustbin without any use [48]. Hence, the key goal in this study is to produce activated carbon using corn cob stems and applied to remove MB dye in the adsorption process. The structure, functional



Fig. 2. XRD patterns of CCSAC.

groups, morphology, surface area chemical structure and molecular interactions activated carbon was studied by different instrumental technique. To determine the ideal adsorption conditions, a number of batch adsorption experiments were conducted. The adsorption results were also well suited with various isotherm and kinetics simulations for the best understanding of the adsorption method. Lastly, an adsorption mechanism has been proposed for MB dye attachment on the surface of CCSAC.

# 2. Materials and methods

# 2.1. Materials

Himedia Chemicals, Mumbai, India supplied the sodium hydroxide (NaOH), orthophosphoric acid (H<sub>3</sub>PO<sub>4</sub>), and hydrochloric acid (HCl). The corn cob stems were gathered from the market in Gunupur, Odisha-765022, India. Methylene blue ( $C_{16}H_{18}ClN_3S$ ) dye was taken from Sigma-Aldrich Chemicals (Germany). Throughout the experimental analysis, double distilled water was utilized.

#### 2.2. CCSAC preparation

The activated carbon was prepared according to previously reported work [49, 50]. In order to remove contaminants and dust particles, the corn cob stems were first cut into small pieces, dried for seven days in sunlight and rinsed in water. Subsequently, the corn cob stems remained oven-heated for 2 days and then finely ground to powder form with a uniform size of 150  $\mu$ m, followed by another 2 days in an oven at 85° C. This powder was put in conc. H<sub>3</sub>PO<sub>4</sub> solution in the weight ratio of 1:4 for 24 h [49, 50]. Then, the acid-treated powder was rapidly passed through a washing process up to neutral pH, and it stayed at 85° C for 72h. Next, it was moved to the carbonization process in microwave technique at 900 W for 30 min under a nitrogen atmosphere. After that, the formed material treated rapidly with distilled water for several times and then heated at 80° C for 48 h in an air oven. Finally, the obtained dried powder was called corn cob stem activated carbon (CCSAC). The preparation of CCSAC is graphically presented in Scheme 1.



Scheme 1. Preparation of CCSAC from corn cob stems in microwave process.

# 2.3. Batch adsorption studies

In the 100 mL polylab plastic bottles, MB dye solution (50 mL) was added with prerequisite amount of adsorbent and stirred at 300 rpm in a rotary shaker. After that, with the help of the centrifugation method CCSAC was separated from the dye solution. The individual solution parameters of dose (10–200 mg), initial concentration (10–100 mg/L), pH (2–12), and time (0–180 min) were measured for MB dye removal using CCSAC adsorbent. All the batch experiments conducted in this work have been repeated three times to find the optimal conditions of MB dye adsorption. The removal percentage of MB and CCSAC adsorption capability were premeditated using the Eqs. (1) and (2).

$$R(\%) = \frac{Co - Ce}{Co} \times 100 \tag{1}$$

$$q_e = \frac{Co - Ce}{W} \times V \tag{2}$$



Fig. 4. Surface charge of CCSAC at different pH.

In the above equations, R and  $q_e = MB$  dye removal rate (%) and MB dye amount on the composite surface (mg/g); W = weight of composite (g) and and V = volume of MB dye solution (L); lastly C<sub>e</sub> and C<sub>0</sub> = equilibrium and initial concentrations (both mg/L) respectively.

#### 2.4. Analytical methods

MB dye concentration dye was assessed in the a UVvisible spectrophotometer (UV-Shimadhu 2450) at 663 nm. The phase structure of CCSAC was measured with XRD equipment (RIGAKU JAPAN/ULTIMA-IV) and supported to this, the Raman bands were recognized with Ranishawin Viarelex spectrometer. A quanta chrome surface area analyser was realistic for BET surface area and pore volume analysis. The surface structure with elemental composition was detected with an FESEM instrument (JSM-7800, Japan) coupled with an EDS energy dispersive spectrometer. The before and after adsorption CCSAC surface functional groups were detected with the FTIR (Perkin Elmer RX-I) instrument. Isoelectric point of CCSAC at different pH values was studied using the ZEN3690, Malvern UK, Zeta size Nano instrument.

Table 1. Details BET analysis of CCSAC.

Sample	BET surface	Average pore	Pore volume
	area (m <sup>2</sup> /g)	size (nm)	(cm <sup>3</sup> /g)
NL	54.61	3.1	0.031

# 3. Results and discussions

#### 3.1. Characterization of adsorbent

In adsorption study, the pore volume and surface area are essential. Fig. 1 shows the CCSAC's nitrogen adsorption desorption curves. A type IV isotherm curve was seen which agree with the FESEM images of mesoporous nature of the CCSAC respectively [51]. Table 1 shows the details of BET parameter deliberated from the instrument. The CCSAC adsorbent contained a defendable surface area of  $54.61 \text{ m}^2/\text{g}$ . This indicated that  $\text{H}_3\text{PO}_4$  activation brings good results in the surface area. Here.  $0.031 \text{ cm}^3/\text{g}$  was the pore volume and 3.1 nm was the pore diameter of the CCSAC respectively. MB dye size is about 1.19 nm [52]; henceforth, it could be easily deposited on the pores of CCSAC and effectively removed from the solution.



Fig. 5. FTIR bends of CCSAC (a) before and (b) after MB dye adsorption.

Fig. 2 depicts the XRD pattern of CCSAC. It could be seen that two major peaks were observed at 24.28° and 43.08° corresponding to miller indices 002 and 100 respectively [53]. The peak at 24.28° corresponded to the liquid phase associated with the graphite structure and the peak at 43.08° characterized the crystal plane of the graphite structure of activated carbon [54]. The peak observed at 24.28° was broad and this broadness confirms that the prepared CCSAC has a more disorder, that will ultimately increase the surface's adsorption binding sites. This would be very advantageous for the MB dye adsorption. Hence, this further confirmed that activated carbon has been successfully formed from corn cob stem.

Raman spectrum represents the inner intermolecular interaction and the consistent Raman bends of CCSAC are displayed in Fig. 3. The respective bends appeared on 1573 and 1355 cm<sup>-1</sup> agreeing of G and D bends in activated carbon respectively. This activated carbon has benzene rings with sp<sup>2</sup> and sp<sup>3</sup> hybridized carbon atoms. The D bends present in 1356 cm<sup>-1</sup> corresponded to C-C bond vibration of sp<sup>2</sup> hybridization and follow the  $E_{2g}$  Raman active vibration mode. Whereas the G bends at 1573 cm<sup>-1</sup> connected the graphitic C-C connection of sp<sup>3</sup> hybridization and it is a characteristic of  $A_{1g}$  Raman mode. This observation also confirms the successful formation of activated carbon from corn cob stems.

The surface charge density of adsorbent shows the major impact on adsorption process because of its association with pH. Fig. 4 demonstrations the charge density of CCSAC in different pH ranges. The isoelectric point (pH<sub>IEP</sub>) was found at 6.4. From this, we could say that greater percentage of MB dye (positively charged) could be removed at higher pH by electrostatic force of attraction with adsorbent (negatively charged) respectively. As seen in the figure, the CCSAC surface became more negative between pH 9, so maximum adsorption would be taking place nearly in this higher pH range respectively.

Fig. 5 presents the FTIR bends of fresh and MB dye absorbed CCSAC. From Fig. 3a, the peak at 3325 cm<sup>-1</sup> was attributed to –OH stretching vibrations of –COOH, -OH and phenol [53]. Other peak was seen in 1566 cm<sup>-1</sup> related to bending vibrations of -OH functional groups present on the CCSAC surface.



Fig. 6. FESEM with corresponding EDS image of CCSAC before (a, b) and after (c, d) adsorption.

Again, the  $1219 \text{ cm}^{-1}$  and  $1373 \text{ cm}^{-1}$  peaks was attributed to the benzene rings C=C vibrations [55]. One more bend was seen in the position of  $1104 \text{ cm}^{-1}$  was contributing the vibrations of C-O in alcohol, phenol, and acid respectively [56]. After adsorption (Fig. 3b) of MB dye, several shifting and less intense bends were identified in the FTIR spectrum of CCSAC. Respective peak of  $3325 \text{ cm}^{-1}$  diminished completely and the peak on  $1566 \text{ cm}^{-1}$  was moved to  $1540 \text{ cm}^{-1}$ . Therefore, these shifting and removal of bends ultimately established the MB dyes attachment on the surface of CCSAC and effectively eliminated from the aqueous solution.

Fig. 6 explains the surface structure and element composition of fresh and MB dye loaded CCSAC. From Fig. 1a, CCSAC possessed good porous structure with numerous small holes which ultimately helpful for MB dye adsorption. Fig. 1b shows the elemental composition of CCSAC and it was observed that the K, O, and C had been seen with a mass percentage ratio of 10.66:22.12:67.23. After adsorption, substantial variations were observed in the morphology of CCSAC as shown in Fig. 1c. This alternation in CCSAC morphology was mainly because of MB dye attachment. Fig. 1d shows the presence of N, S, and Cl along with the C, O, and K, which confirmed that dye adsorbate was attached on the adsorbent. The MB loaded CCSAC had elemental ratio of 69.40:21.00:6.28:2.72:0.38:0.21 for C: O: K: N: S: K respectively.

# 3.2. Application for MB dye removal

#### 3.2.1. Adsorbent dose studies

The removal rate in an adsorption process is highly affected by the adsorbent dose as it directly affects the adsorption process. Keeping other parameters like pH, concentration, and time constant, adsorbent dose



Fig. 7. Effect of adsorbent dose (a), initial concentration (b), contact time (c), and pH (d), on the MB dye removal.

batch study was carried out. The MB dye clearance rate rise significantly from 15% to 93% when the CCSAC dosage was changed from 10 mg to 200 mg (Fig. 7a) [57]. The number of active binding sites is closely correlated with the removal tendency. Therefore, as the dosages of the adsorbent are increased, the quantity of binding sites on the CCSAC surface rises, leading to an enhanced the tendency for dye removal. The optimum adsorbent dose was 150 mg as after this a straight line was obtained and no significant changes in MB dye removal efficiency.

#### 3.2.2. MB dye concentration analysis

This dye concentration analysis was done in 150 mg of adsorbent dose with different concentrations of MB dye range from 10 mg/L to 100 mg/L (Fig. 7b). Here, the MB dye removal rate progressively decreased from 91% to 12% as the dye concentration rose from 10 mg/L to 100 mg/L. As the adsorbent dose was fixed (i.e. 150 mg), a constant quantity of binding locations was available on CCSAC surface. As the concentration changed from lower to higher, in the solution the

amount of molecules of MB dye also increased. Therefore, the experimental outcomes revealed that the MB dye removal percentage declines with increasing in concentration, whereas the surface's binding sites accessibility of CCSAC was higher at lower MB concentration. The optimum MB dye concentration was chosen at 10 mg/L for further analysis.

#### 3.2.3. Contact time studies

Time is the foremost parameter that adversely affects the adsorbent's adsorption capability. It was shown from the Fig. 7c that the rate of removal of MB dye climbed with time, reaching a certain period and after that, it showed a steady graph. With change in time from 10 to 120 min, CCSAC was able to remove from 23% to 97% and after that, it remained constant up to 180 min. This indicated that first, the MB dye molecules adsorbed on the pores and then diffused on the surface, and after complete covering of the surface, no further adsorption took place and remained constant. The optimum contact time for this analysis was 120 min, which was taken for further analysis.



Fig. 8. Nonlinear Langmuir and Freundlich isotherm plot of MB dye removal.

#### 3.2.4. pH analysis

An important component that adversely affects the adsorbent's affect the adsorbent's removal tendency is pH. Keeping other parameters like adsorbent dose 150 mg, time 120 min and concentration 10 mg/L constant, pH analysis was conducted in the pH range 2 to 12 respectively. From Fig. 7d, it was noticed that at pH 2, the CCSAC showed minimum adsorption of MB dye. The tendency to remove increased from 16% to 95% when the pH value was raised from 2 to 12. The  $pH_{IEP}$  of the CCSAC was 6.4 as obtained from Fig. 5, below this pH value CCSAC surface has positively charged and repelled positively charged MB dye molecules, resulting in the decreased removal rate [39]. However, at upper pH levels, interaction of the positively charged MB dye with electrostatic molecules and the surface that is negatively charged had been occurred as a result of which the MB removal rate increased up to the pH 10 and after that it became constant.

#### 3.3. Isotherm study

The experimental data was fitted to a variety of isotherm models to better understand the Methylene Blue (MB) dye adsorption mechanism on the CCSAC surface. Among all the isotherms, in this removal study, two familiar isotherms i.e. Freundlich isotherms and Langmuir isotherms were used. Adsorbate is formed as a single monolayer on the adsorbent surface molecules in the Langmuir isotherm model [58]. In other words, the surface of the adsorbent contains a specific quantity of active binding sites, and each binding site only absorbs one molecule of the adsorbate. The heterogeneous adsorbent surface is indicated as the multiple layers forming on the surface of the adsorbent, as shown by the Freundlich isotherm equation [59]. The Freundlich and Langmuir isotherm model's nonlinear equations can be written as follows;

$$q_e = \frac{q_m b C_e}{1 + b C_e} \tag{3}$$

$$q_e = K_F C_e^{1/n} \tag{4}$$

 $q_m$  and qe represent the adsorbent's equilibrium and maximum absorption capabilities (mg/g); The equilibrium concentration is Ce, while the heterogeneity factor, Freundlich constant, and the constants n, b and K<sub>F</sub> represent the Langmuir coefficients. Fig. 8 shows how the model was applied to the MB dye removal data, and Table 2 lists the obtained isotherm parameters. It was discovered that the Langmuir model suited the experimental data more accurately than the Freundlich model. The value of the correlation coefficient for the Langmuir model



Fig. 9. Nonlinear first and second-order kinetics of MB dye removal.

 Table 2. Nonlinear Langmuir and Freundlich isotherms parameter.

Langmuir isotherm		Freundlich isotherm			
q <sub>m</sub> (mg/g)	129.77	K <sub>f</sub> (mg∕g)	2.82		
b(L/mg)	0.01	n	1.43		
R <sub>L</sub>	0.86				
R <sup>2</sup>	0.90	$\mathbb{R}^2$	0.86		

was maximum i.e.  $R^2 = 0.90$  and the calculated  $R_L$  value was 0.86, this again confirmed suitable fitted data to the Langmuir model. Therefore, a monolayer of adsorbate was identically spread on the equivalent binding sites of the CCSAC surface [56]. The highest amount of MB dye that can be adsorbed on CCSAC surface has been found to 129.77 mg/g respectively. The capacities for adsorption of other waste materials prepared activated carbon to our case study are compared in Table 3.

#### 3.4. Kinetics study

To ascertain the rate of the adsorption process, several kinetics models are employed in adsorption analyses. The elimination of MB dye was evaluated using two well-known kinetic models: pseudo first order and pseudo second order. It defines the pseudofirst-order reaction as a bimolecular reaction that is

Table 3. Uptake capacities of different activated carbon prepared from waste materials.

Adsorbents	q <sub>m</sub> (mg/g)	References
Pineapple waste	9.61	[60]
Pine cone	60.97	[61]
Coriandrum Sativum	94.9	[62]
Lychee seed	124.5	[14]
CCSAC	129.77	Present study

made to behave like physical adsorption [63]. Furthermore, chemical adsorption is the foundation of the pseudo-second-order kinetics model [64]. The nonlinear equations for the pseudo first and secondorder kinetic models are expressed as follows:

$$q_t = q_e \left( 1 - e^{-k_1 t} \right) \tag{5}$$

$$q_t = \frac{q_e^2 k_2 t}{1 + q_e k_2 t} \tag{6}$$

In this context, t denotes time; the first-order equilibrium constant is denoted by  $k_1$ ;  $k_2$  signifies the second-order equilibrium constant; at equilibrium,  $q_e$ represents the uptake capacity, while  $q_t$  refers to the uptake capacity at time t. Fig. The first and second order kinetics curves shown in Fig. 9, and Table 4 represents the corresponding kinetics factors. The



Fig. 10. Possible adsorption mechanism of MB dye on CCSAC adsorbent surface.

Table 4. Kinetic parameters and correlation coefficients (R<sup>2</sup>) for pseudo-first-order and pseudo-second-order kinetic models.

Pseudo-first-order			Pseudo-second-order		
q <sub>t</sub> (mg/g)	K <sub>1</sub>	R <sup>2</sup>	q <sub>t</sub> (mg/g)	K <sub>2</sub>	R <sup>2</sup>
37.57	0.027	0.943	46.45	0.006	0.953

pseudo-second-order model's R<sup>2</sup> value (0.953) was greater than the pseudo-first-order model (0.943). It confirmed that the MB dyes the removal of aqueous solution on the surface of CCSAC was occurring by chemical adsorption process [65]. Similar types of chemical adsorption have been observed by other researchers to remove metal ions and dye from aqueous solution respectively [66, 67].

#### 3.5. Adsorption mechanism

Fig. 10 represents the likely interaction between MB dye and CCSAC. From FTIR studies (Fig. 3), it was found that the alcohol, phenol, ether and acid present on the CCSAC surface have peak of –OH functional groups, which was shifted after adsorption and frolicked a significant part in MB removal studies.

Isoelectric point or zero surface charge of the CCSAC was 6.4. Above this pH, the negative surface of CC-SAC interacted with the cationic dye i.e., MB by the force of electrostatic attraction. MB dye molecules have electronegative N and S atoms were forming strong hydrogen bonds with the CCSACA's -OH and -COOH groups. The pi-pi interaction was occurred between the  $\pi$ -bond of the fused hexagons layer of CCSAC with the  $\pi$ -bond of benzene rings present in MB dye respectively. Lastly, the CCSAC has numerous mesopores which would directly fill up by MB dye molecules and be removed from the solutions. Hence, four types of mechanisms were occurring i.e. hydrogen bonding, electrostatic force of attraction, pi-pi interaction and pore filling. Similar types of observation have been obtained by other scientists confirming the above-suggested mechanism of our work respectively [30, 68].

# 4. Conclusion

In our work, we have prepared the activated carbon from corn cob stems by thermal heating microwaves technique using  $H_3PO_4$  as an activating agent. The formed CCSAC has a mesoporous structure having the surface area of 54.61 m<sup>2</sup>/g. Our adsorption process was again well established by different batch adsorption experiments and 98% of MB dve was eliminated under batch conditions with a 10 mg/L of dye concentration, pH 10, 150 mg of adsorbent dosage, and 120 minutes of contact duration. The data from the adsorption experiment aligned effectively considering the Langmuir isotherm model, revealing an adsorption capability of up to 129.77 mg/g. Furthermore, with an R<sup>2</sup> value of 0.953, our adsorption finding was consistent with pseudo-second-order kinetics. The process by which the CCSAC absorbs MB dye showed that there was an electrostatic force of attraction, hydrogen bonding,  $\pi$ - $\pi$  attraction and pore filling respectively. Given the above analysis, our prepared CCSAC is an excellent, productive, and well-synthesized material to remove the MB dye from the polluted water.

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# **Ethical approval**

Not applicable.

#### **Conflict of interests**

The authors declare no conflict of interest.

# **Data availability**

Not applicable.

# Author contributions

Gopi Narasimha Murthy: Investigation, Formal analysis, Data curation and Writing original draft, Uttam Kumar Sahu: Writing, review & editing original draft.

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