Characterization and Adsorption Studies of *Cocos nucifera L.* Activated Carbon for the Removal of Methylene Blue from Aqueous Solutions

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Characterization and adsorption characteristics of *Cocos nucifera L.* activated carbon (CNAC) have been investigated. The activated carbon used in this study was prepared from the coir of *C. nucifera L.*, commonly known as “coconut”. The activated carbon was characterized for surface groups and other parameters and was analyzed for pore size distribution. The surface texture of the adsorbent, the pore structure, and the number and position of the functional groups available on its surface for binding methylene blue (MB) were investigated by Fourier transform infrared spectra. X-ray diffraction studies confirmed the amorphous nature of the adsorbent. The activated carbon developed has been used for the removal of MB. Batch adsorption experiments were carried out for the removal of MB onto the activated carbon. The effect of contact time and initial concentration, adsorbent dose, and temperature on the removal of the dye was investigated. A maximum removal of almost 100 % was achieved at 60 $\text{mg}\cdot\text{L}^{-1}$ of dye concentration by increasing the adsorbent dose from (3 to 7) $\text{g}\cdot\text{L}^{-1}$. Kinetic data were found to fit the first- and second-order kinetic equations, and the values of the rate constants were found to be $0.55\cdot10^{-2}$ min^{-1} and 0.082 $\text{g}\cdot(\text{mg}\cdot\text{min})^{-1}$, respectively. The resultant equilibrium data were fitted to various isotherm equations. The adsorption capacity of the adsorbent was found to be 15.59 $\text{mg}\cdot\text{g}^{-1}$ at room temperature (30 °C).

Introduction

The industrial revolution resulted in an improving lifestyle, raising the standard of living of people, but also resulted in an indiscriminate exploitation of natural resources. Pollution in general and water pollution in particular has attracted the attention of scientific workers the world over. Out of a number of industries, the textile industry is a major source of colored effluent discharge to water bodies.[1] Effluents from the dyeing and finishing processes of the textile industry are known to add a number of pollutants like dissolved and suspended solids, heavy metals, and a large amount of surfactants and colors to the water sources. Dyes have been reported to be toxic, carcinogenic, and mutagenic. The colored water contains $-\text{C}{=}\text{C}$-bonds and $-\text{N}{=}\text{N}$-bonds, and heterocyclic and aromatic rings occur at the time of reduction of the cleavage of these groups.[2] Cationic dyes make the brightest class of the known dyes and are water-soluble. Several methods like flocculation, coagulation, and membrane separation have been regularly reported for the treatment of dyestuff wastewaters,[3,4] but most of these methods are either expensive or pose the problem of sludge disposal. The adsorption process provides an attractive alternative for the treatment of dyestuff wastewaters. This process is easy to handle, requires less maintenance, and produces smaller amounts of sludge. Coconut coir (*Cocos nucifera L.*) is an agricultural waste biomaterial and is light and fluffy. The estimated production of coconut coir in India is about 7.5 million tonnes per year.[5] Part of it is used in different applications, and the remaining coconut coir is disposed.

The objective of this work is to prepare activated carbon from coconut coir (CNAC) and then to investigate its adsorption characteristics for the removal of methylene blue (MB). MB is an organic dye and has applications in different processes in different industries. Acute exposure to MB has been reported to be toxic for aquatic biota and human beings. On exposure to MB, human beings are reported to develop allergic reactions in the eyes, vomiting, dizziness, cyanosis, and increased blood pressure.[6] One of the major advantages of this method is that during removal of the dye, no toxic byproducts are generated. The prepared CNAC has been characterized by sophisticated instruments, and the effect of various parameters on the removal of MB has been carried out. Kinetic and equilibrium studies have also been reported for the process of removal of the dye.

Materials and Methods

Materials. All reagents used in the present experiments were of analytical grade and were obtained from Merck, Mumbai, India. The stock solution of MB was prepared by dissolving the dye in distilled water. The chemical formula of MB is $C_{16}H_{18}C\ N_3S\cdot3H_2O$, with a molecular weight of 373.91 $\text{g}\cdot\text{mol}^{-1}$ and $\lambda_{\max}$ of 663 nm (C.I. No. 52015). The raw material (coconut coir) was collected from nearby Durgamandir, Varanasi, Uttar Pradesh, India.

Methods

Preparation of Activated Carbon. The raw material was dried in sunlight, and then the pith was chopped into small pieces. The pieces were subjected to carbonization at 700 °C for 1 h using a tubular muffle furnace. A constant flow of 150 $\text{mL}\cdot\text{min}^{-1}$ of high-purity (99.99 %) nitrogen was maintained throughout the process of carbonization. The final product obtained was primary carbon. It was cooled to room temperature,

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and for better cleaning[5] of the product was washed with hot distilled water and 0.50 N HCl until the pH of carbon sample reached 7.0. The final product was dried in a hot air oven at 110 °C for 12 h.

The primary carbon was impregnated with 20 % zinc chloride ($ZnCl_2$) solution for 24 h. Zinc as such does not participate in adsorption. Zinc chloride has been used as a catalyst which enhances the activation process resulting in an increase of surface area in the activated carbon. In this solution, the ratio of primary carbon and zinc chloride solution was 1:1 (mass fraction). The mixture was dehydrated in an oven at 110 °C for 12 h to remove moisture and was activated under the same conditions as those for carbonization but to a final temperature of 700 °C for 2 h. The activated product was cooled under nitrogen gas flow to room temperature and was subsequently washed with hot distilled water and 0.10 N hydrochloric acid until the pH of washing solution also reached 7.0. The activated carbon was dried in a hot air oven at 110 °C, ground and sieved to obtain desired particle size (150 μm), stored in desiccators, and used for the adsorption studies.

Analysis of the Adsorbent Sample. The textural characteristics of the adsorbent were determined from a nitrogen adsorption isotherm at 77 K. The BET (Brunauer−Emmett−Teller) surface area of the CNAC was determined using a Micromeritics ASAP 2020 surface area analyzer, and the pore size distribution was determined by using the BJH model.

Some changes, both physical and chemical, in the lignocellulosic material of coconut coir, are expected to occur upon carbonization and activation. These changes were verified by Fourier transform infrared (FTIR) spectral absorption studies, which were carried out on infrared spectrometer (Simadzu/8400S) to determine the presence of different functional groups in the CNAC. The spectral wavelength was covered from (0 to 4500) cm^{-1}.

X-ray diffraction (XRD) analysis of the adsorbent was carried out using an X-ray diffractometer (Scifert and Co., model ID 3000) to determine the phase characterization of the CNAC. A copper target with nickel as the filter medium was used for XRD. The powder sample for the XRD was prepared by putting a thin layer of powder smeared over a glass plate. The XRD was maintained at 1 $deg \cdot min^{-1}$, and orientation was kept from (0 to 100) deg. The CNAC crystallite size was determined by using the XRD result and using the diffraction peaks from the Scherrer formula:

$$X_s = 0.9\lambda/\text{fwhm} \cos \theta \qquad (1)$$

where X_s is the crystallite size (nm), λ is the wavelength of the monochromatic X-ray beam (nm), (λ is 0.154056 nm for Cu Kα radiation), fwhm is the “full width at half-maximum” for the diffraction peak under consideration (rad), and θ is the diffraction angle (deg).

Adsorption Studies. Adsorption experiments were carried out by agitating 0.25 g of CNAC in 50 mL of dye solution of desired concentrations [(60 to 100 $mg \cdot L^{-1}$)] in 250 mL stoppered conical flasks at 150 rpm, at 30 °C in a thermostatted water bath shaker to attain equilibrium. The dye solution was separated from the adsorbent by centrifugation at 10 000 rpm for 10 min. The concentration of dye in the supernatant solution was measured spectrophotometrically by monitoring the absorbance at 663 nm (λ_{max}) using a UV−visible spectrophotometer (Spectronic 20, Bausch & Lomb, USA). Langmuir and Freundlich isotherms were employed to study the adsorption capacity of CNAC. The removal (%) of MB and equilibrium adsorption uptake in the solid phase, q_e ($mg \cdot g^{-1}$), were calculated using the following relationship:

$$q_e = (C_o - C_e)V/w \qquad (1a)$$

where C_o and C_e (both in $mg \cdot L^{-1}$) are the initial and equilibrium concentrations of dye in the solution, V is the volume of the solution (L), and w is the mass of the activated carbon (g). The kinetic data for removal of MB onto CNAC were fitted to pseudofirst-order and second-order rate equations.

Table 1. Physical Characteristics of Activated Carbon

BET surface area ($m^3 \cdot g^{-1}$)	205.27
total pore volume, V_p ($cm^3 \cdot g^{-1}$)	0.0246
micropore surface area s_{mic} ($cm^3 \cdot g^{-1}$)	181.0
mesopore sueface area s_{mes} ($cm^3 \cdot g^{-1}$)	24.0
mean pore diameter, D (Å)	41.24

Results and Discussion

Characterization of the Adsorbent. Pore Size Distribution of Adsorbent. Textural characterization of the prepared activated carbon, CNAC, was carried out. The surface area and pore size distribution of the activated carbon was determined and is given in Table 1. The textural characterization, however, has been reported in Table 2. The surface area and pore size distribution were determined by the volumetric adsorption of nitrogen by the activated carbon at 77 K and have been reported in Figures 1 and 2. The system provides data for the determination of the monolayer adsorbed amount, apparent specific surface area, pore volume, and pore size distribution.[7] The sample (activated carbon) was degassed for 12 h under vacuum at a temperature of 350 °C prior to analysis to remove any impurities. The adsorption of nitrogen under different partial pressures was determined. The micropores and mesopores can be defined by the hysteresis during adsorption at relatively high relative pressures (P/P_o). The calculations of the surface area, microporosity, mesoporosity, and pore volume were carried out by standard methodologies.[8]

FTIR Studies. Infrared spectra of both the coconut coir (raw material) and the CNAC have been presented in Figure 3a,b. The IR spectrum of the CNAC gives information on the chemical structure and surface functional groups of the samples. Before carbonization, the material was a typical lignocellulosic material, which is clear from Figure 3a. After carbonization and activation, some changes were observed in the material because of the elimination of other elements in the form of water and other species.[9] These changes were evident in the formation of structures containing multiple carbon−carbon bonds, as well as the elimination of the originally present oxygen and hydrogen atoms. The intense absorption band with a maximum at about 2929.99 cm^{-1} can be attributed to the stretching vibrations of C—H bonds in alkane and alkyl groups where carbon is bonded with hydrogen bonds. The band at 2328.75 cm^{-1} corresponds to vibration in alkenes and alkyl groups, whereas the band near 1532.94 cm^{-1} is attributed to the vibration in aromatic groups. At 1094.32 cm^{-1}, the band is less intense ν(C—O) and is related to the ν(C—O) stretching vibration of the bonds in ester, ether, or phenol groups. The weak absorption band at 639.32 cm^{-1} corresponds to the λ(O—H) vibration in the benzene ring.

XRD Analysis. XRD patterns for CNAC after thermal degradation in nitrogen or in air media were recorded. The sample was amorphous, although a broad diffused peak was observed (figure not shown). The diffraction peak of crystalline carbon was not observed. The X-ray diffractograms (figure not shown) confirmed that the activated carbon possesses a heterogeneous surface.

Table 2. BJH Adsorption Pore Distribution Report of the Adsorbent

pore diameter range	average diameter	incremental pore volume	cumulative pore volume	incremental pore area	cumulative pore area
Å	Å	$\mathrm{cm^3 \cdot g^{-1}}$	$\mathrm{cm^2 \cdot g^{-1}}$	$\mathrm{m^2 \cdot g^{-1}}$	$\mathrm{m^2 \cdot g^{-1}}$
399.0 to 178.0	211.1	0.004811	0.004811	0.912	0.912
178.0 to 115.2	132.6	0.002454	0.007265	0.740	1.652
115.2 to 84.5	94.7	0.001646	0.008911	0.695	2.347
84.5 to 66.5	73.1	0.001445	0.010355	0.790	3.137
66.5 to 54.3	59.0	0.001223	0.011579	0.830	3.967
54.3 to 45.4	48.9	0.001533	0.013112	1.253	5.220
45.4 to 38.6	41.4	0.001178	0.014290	1.139	6.360
38.6 to 33.2	35.4	0.001400	0.015689	1.582	7.942
33.2 to 28.7	30.5	0.001703	0.017393	2.232	10.173
28.7 to 24.8	26.4	0.002085	0.019478	3.160	13.333
24.8 to 21.4	22.8	0.002246	0.021724	3.945	17.278
21.4 to 18.1	19.4	0.003634	0.025358	7.499	24.777

Effect of Various Parameters on the Removal of MB by CNAC. The effect of various important parameters, namely, contact time, initial dye concentration, adsorbent dose, and temperature, on the removal rate of dye was investigated. The reaction acquired equilibrium in 100 min, and the maximum removal (93.6 %) was achieved at an initial dye concentration of 60 $\mathrm{mg \cdot L^{-1}}$. The removal of dye increased from (74.2 to 93.6) % by decreasing its concentration from (100 to 60) $\mathrm{mg \cdot L^{-1}}$ at 30 °C. An obvious increase in removal rate was achieved by increasing the adsorbent dose from (3.0 to 7.0) $\mathrm{g \cdot L^{-1}}$. The removal increased from 65.3 % to almost 100 % by increasing the adsorbent dose. A higher removal was obtained at increasing temperatures, indicating the process of removal to be endothermic, and the removal was found to be 98 % at 50 °C.

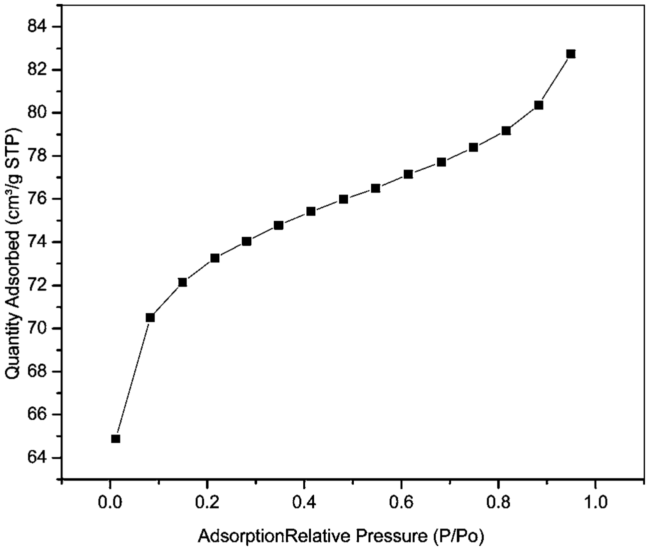

Figure 1. Nitrogen adsorption isotherm of CNAC at 77 K.

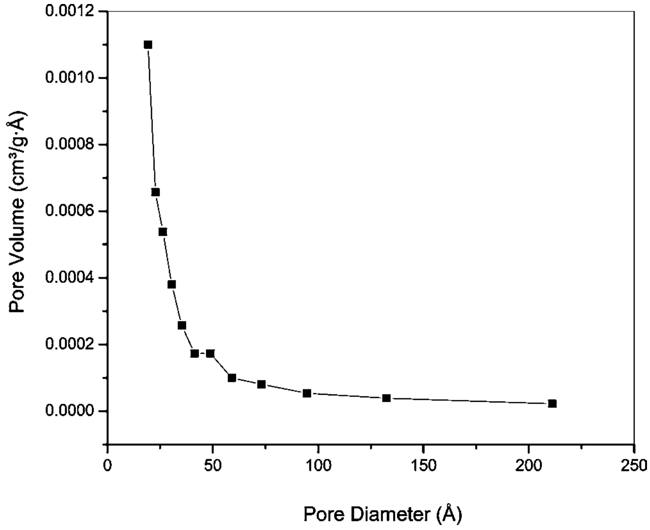

Figure 2. Pore size distribution of CNAC.

Kinetic Studies. A kinetic study of the removal of MB was taken up by using different rate equations. The kinetic data were found to fit Lagergren's model[10,11] which confirmed the process of removal to be pseudofirst order:

$$\log(q_e - q) = \log q_e - (K_{ad}/2.303)t \tag{2}$$

where q_e and q (both in $\mathrm{mg \cdot g^{-1}}$) are the amounts of MB adsorbed at any time and at equilibrium, respectively, and K_{ad}

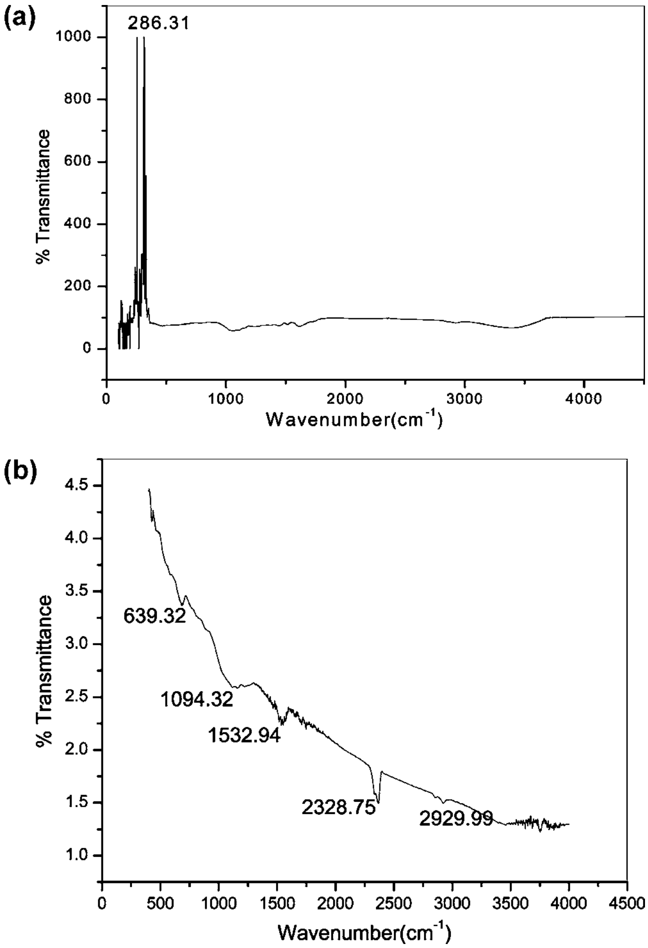

Figure 3. (a) FTIR spectrum of the raw material. (b) FTIR spectrum of CNAC.

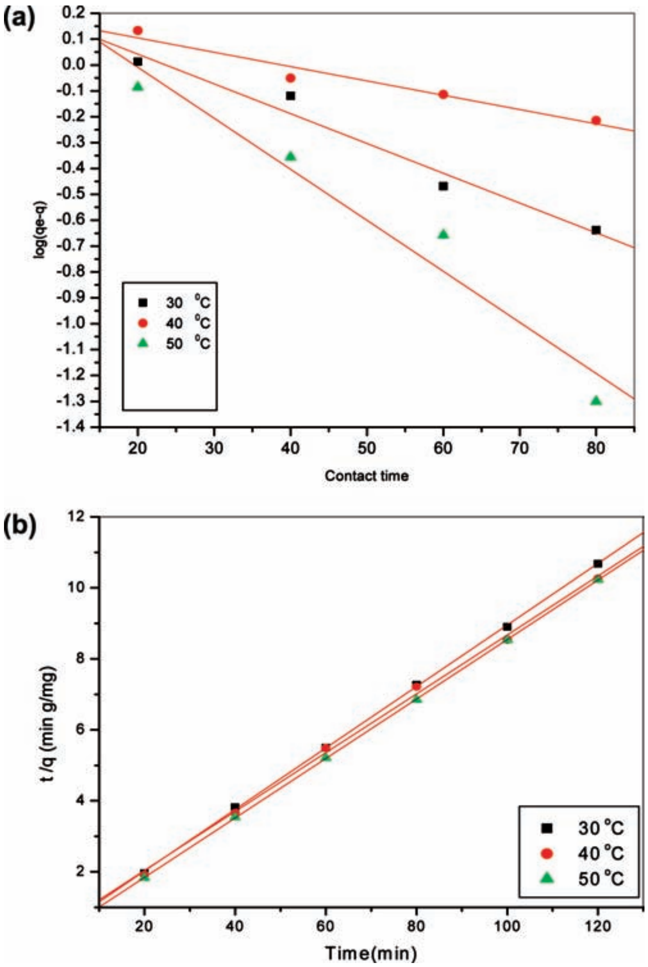

Figure 4. (a) Lagergren's plot for kinetic modeling of the adsorption process of MB on CNAC. (b) Second-order kinetic model of the adsorption or MB on CNAC.

Table 3. Values of Rate Constant of Adsorption for the Removal of MB by Adsorption on CNAC

temperature	K_{ad} ($\cdot 10^{-2}$)		k_2	
°C	min^{-1}	R^2	$\text{g}\cdot\text{mg}^{-1}\cdot\text{min}^{-1}$	R^2
30	0.55	0.9698	0.082	0.9996
40	1.15	0.9534	0.083	0.9999
50	1.97	0.9515	0.086	1.0

(min^{-1}) is the rate constant of adsorption. Straight line plots of "log ($q_e - q$) versus t" (Figure 4a) confirm the validity of the model and that the process of removal is governed by first-order kinetics. Values of the rate constant, K_{ad}, for the removal of MB by adsorption on CNAC at different temperatures were determined by the slopes of the graphs of Figure 4a.

The second-order kinetic model was also used to investigate the removal of MB by CNAC. The kinetic data for the removal of MB was assessed by using second-order kinetic model using the following equation:[12]

$$t/q = 1/k_2 q_e^{\,2} + t/q_e \tag{3}$$

where k_2 ($\text{g}\cdot\text{mg}^{-1}\cdot\text{min}^{-1}$) is the rate constant of the second-order kinetic equation. The values of k_2 and q_e were calculated from the slope of plot t/q versus t (Figure 4b). Values of q_e were also calculated at this stage and were almost similar to the experimental values. Values of the rate constants of first-order and second-order reactions are given in Table 3, and the values of the rate constants were found to be $0.55\cdot 10^{-2}$ min^{-1} and 0.082 $\text{g}\cdot\text{mg}^{-1}\cdot\text{min}^{-1}$, respectively.

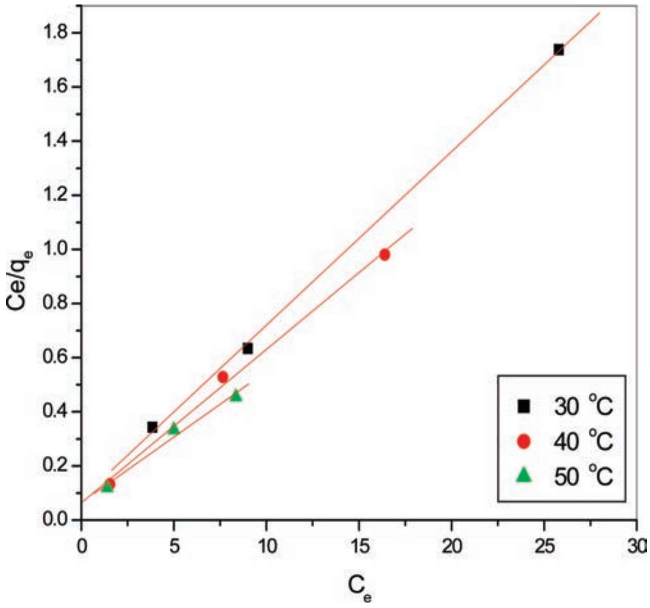

Figure 5. Langmuir isotherm plot for the adsorption of MB on CNAC.

Further, the kinetic data for the present system were tested for both pseudofirst-order and pseudosecond-order kinetic equations. Perusal of Table 3 shows that the values of R^2 are greater for k_2 as compared with K_{ad}, and this indicates that pseudosecond-order kinetic model is more suitable for the kinetic data of the present system.

Equilibrium Modeling. Equilibrium modeling for the process of the removal of MB by CNAC was carried out using the Langmuir and Freundlich isotherm models. Several mathematical models have also been applied for describing equilibrium studies for the removal of pollutants by adsorption on solid surfaces. The selection of an isotherm equation depends on the nature and type of the system.

Langmuir Model. The Langmuir model assumes that uptake of MB ions occurs on a homogeneous surface by monolayer adsorption by the following equation:[13,14]

$$C_e/q_e = 1/Q^o b + C_e/Q^o \tag{4}$$

where C_e ($\text{mg}\cdot\text{L}^{-1}$) is the equilibrium concentration of the solute, q_e is the amount adsorbed at equilibrium ($\text{mg}\cdot\text{g}^{-1}$), and $Q°$ ($\text{mg}\cdot\text{g}^{-1}$) and b ($\text{L}\cdot\text{mg}^{-1}$) are constants related to the adsorption capacity and energy, respectively. A plot of C_e/q_e versus C_e (Figure 5) gives a straight line. These straight line plots confirm the validity of the Langmuir model for the present system.

For the investigation of whether adsorption is "favorable" or "unfavorable", the Langmuir model can be classified by a term R_L, a dimensionless constant separation factor:[15,16]

$$R_L = 1/(1 + KC_o) \tag{5}$$

where K is the Langmuir constant ($\text{L}\cdot\text{g}^{-1}$) and C_o is the highest initial dye concentration ($\text{mg}\cdot\text{L}^{-1}$). The values of R_L indicate whether the isotherm is unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($R_L < 1$), or irreversible ($R_L = 0$). The values of R_L at different temperatures, (30, 40, and 50) °C, in the experiments were 0.020, 0.018, and 0.021. The values of R_L are greater than zero and less than unity. It further shows that the Langmuir isotherm was favorable for the adsorption of MB on CNAC. The values of $Q°$ and b were determined by the slopes and intercepts of Figure 5 and are given in Table 4.

Table 4. Langmuir and Freundlich Isotherm Model Parameters for Adsorption of MB on CNAC

isotherm	temperature (K)	parameters		
Langmuir		$Q°$ ($\text{mg} \cdot \text{g}^{-1}$)	b ($\text{L} \cdot \text{mg}^{-1}$)	R^2
	303	15.59	0.81	0.8982
	313	17.62	0.90	0.9985
	323	20.62	0.75	0.9761
Freundlich		K_f ($\text{L} \cdot \text{g}^{-1}$)	$1/n$ ($\text{L} \cdot \text{g}^{-1}$)	R^2
	303	0.98	0.142	0.8655
	313	1.03	0.140	0.9879
	323	1.02	0.239	0.9964

Freundlich Model. Adsorption data for the removal of MB on CNAC were fitted to the linear form of the Freundlich isotherm:

$$\log x/m = \log K_f + 1/n \log C_s \tag{6}$$

where x/m is the amount adsorbed per unit mass of the adsorbate, C_s is the equilibrium concentration, and $1/n$ and K_f are the Freundlich constants. The constant K_f is related to the degree of adsorption, and n provides an approximate estimation of the intensity of the adsorption:

The values of the constants, K_f and n, were determined by the slopes and intercepts of Figure 6 and have been given in Table 4. It may be noted that the value of K_f and n increase with an increase in temperature for all of the concentrations of MB indicating that adsorption is favorable at higher temperatures. The dye having the greater value of K_f has a high affinity toward the adsorbent as compared to other having low K_f values.[17,18] Further, the values of R^2 for the two isotherm models are higher for the Langmuir model at (30 and 40) °C, and at 50 °C, the values of R^2 are greater for the Frendulich model. Thus, for (30 and 40) °C, the Langmuir model fits the equilibrium data better, and at 50 °C, the Freundlich isotherm is better.

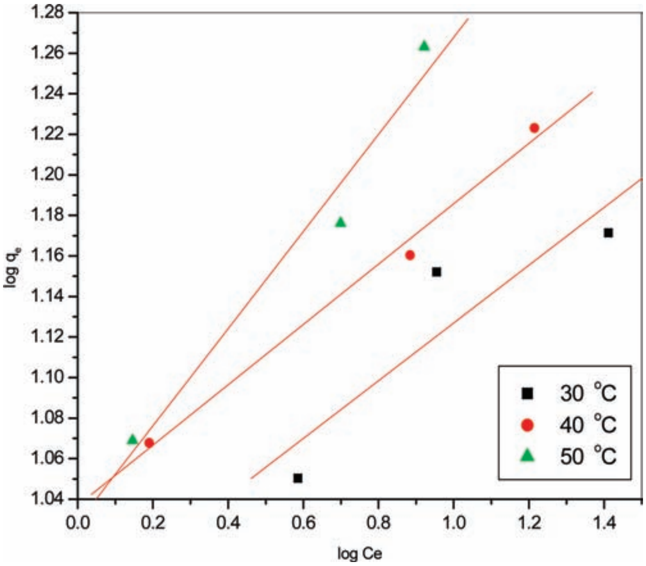

Figure 6. Freundlich adsorption isotherm plot for MB on CNAC.

Table 5. Comparison of Adsorption Capacities of Different Adsorbents for Dye Removal

adsorbent (activated carbon)	q_m ($\text{mg} \cdot \text{g}^{-1}$)	ref
pineapple stem	119.05	Hameed et al., 2009[19]
jackfruit peel	285.71	Hameed, 2009[20]
coconut husk	434.78	Tan et al., 2008[21]
rattan sawdust	294.14	Hameed et al., 2007[22]
spent tea leaves	300.05	Hameed, 2009[23]
rice husk	312.26	McKay et al., 1999[24]
cotton waste	277.78	McKay and Ho, 1999[25]
coir pith	5.87	Kavitha and Namasivayam, 2007[26]
CNAC	15.59	this study

Table 5 presents a vivid picture of the comparison of adsorption capacities for the removal of MB by different adsorbents. On perusal of this table, it is clear that coconut husk displays the maximum adsorption capacity followed by rice husk. Further, the adsorption capacity of CNAC, the adsorbent for the present system, is also significant, justifying the suitability of CNAC for the removal of MB in particular and that of dyes in general. However, the values of q_m for activated carbons prepared from most of the other raw materials are higher than that of the CNAC, but probably coconut coir seems to be most abundant in most parts of the world, so it makes sense to use *C. nucifera L.* as a raw material for the preparation of activated carbon.

Conclusions

Cocos nucifera L. coir, an agricultural waste material that is plentiful in most parts of the world, has been successfully used to derive activated carbon (CNAC). The FTIR of the CNAC shows the position of functional groups available for binding MB onto its surface. The pore size distribution of the activated carbon showed that it had both micropores and mesopores. XRD of the adsorbent showed it to be amorphous in nature. The removal of the dye was dependent on the physicochemical characteristics of the adsorbent. Kinetic and isotherm studies were carried out. The sorption was analyzed using pseudofirst-order and second-order kinetic models. Though a detailed cost analysis of the preparation and subsequent application of the CNAC is to be carried out, it seems that the entire process may be cost-effective.

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