

Research Article

Kinetics, Equilibrium, and Thermodynamic Studies on Adsorption of Methylene Blue by Carbonized Plant Leaf Powder

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Carbon synthesized from plant leaf powder was employed for the adsorption of methylene blue from aqueous effluent. Effects of pH (2, 4, 6, 8, and 9), dye concentration (50, 100, 150, and 200 mg/dm³), adsorbent dosage (0.5, 1.0, 1.5, and 2.0 g/dm³), and temperature (303, 313, and 323 K) were studied. The process followed pseudo-second-order kinetics. Equilibrium data was examined with Langmuir and Freundlich isotherm models and Langmuir model was found to be the best fitting model with high R^2 and low χ^2 values. Langmuir monolayer adsorption capacity of the adsorbent was found to be 61.22 mg/g. From the thermodynamic analysis, ΔH , ΔG , and ΔS values for the adsorption of MB onto the plant leaf carbon were found out. From the values of free energy change, the process was found out to be feasible process. From the magnitude of ΔH , the process was found to be endothermic physisorption.

1. Introduction

Synthetic dyes in large variety and quantity are used by several industries for coloring their products [1–3]. Considerable amount of the dyes added in the process remain unconsumed and ultimately find their way to water bodies. Presence of dyes in water bodies poses serious threat to the environment as the color affects the nature of the water and makes it unsuitable for human consumption. Apart from being aesthetically displeasing, colored water also reduces photosynthetic action by preventing penetration of light [2]. Some dyes and/or their metabolites have carcinogenic, mutagenic and teratogenic effects on aquatic life and human beings [2, 4]. As adsorption is one of the most efficient techniques available for dye removal [5] and the commercial adsorbents like activated carbon are expensive [6], search is carried by several researchers for identifying low-cost adsorbents.

This has prompted many researchers to search for cheaper substitutes such as shells of bittim [7], wheat shells [1], bagasse fly ash [3], rice husk [8], acid treated rice husk [9], water hyacinth roots [10], saw dust [11], dead biomass [12, 13], *Thespesia populnea* bark [14], *Ananas comosus*

Activated carbon [15], peel of *Cucumis sativus* fruit [16]. Researchers have produced activated carbon from various low-cost materials [17–21] in an attempt to reduce the cost of carbon production. In addition, researchers have also explored the possibilities of using unburned carbon [6]. In the present investigation, application of carbon made from a low-cost agricultural waste, senescent plant leaf powder, for adsorption of methylene blue (MB) was examined.

2. Materials and Methods

2.1. Preparation of Carbon. Senescent plant leaves were collected from the local garden. Most of these leaves were mainly of teak (*Tectona grandis*) and Guava (*Psidium guajava*) trees. The collected plant leaves were dried in hot air oven at 100°C to complete dryness and then ground in a mixer. The powder was filled in self-sealed silica crucibles and heated at 500°C in a muffle furnace for one hour. Carbonized powder was cooled to room temperature in desiccators and screened using BSS (British Standard Screen) test sieves. Particles of mesh size –150 + 200 size (average particle size 90 μm) were collected and used for further studies. In the present investigation,

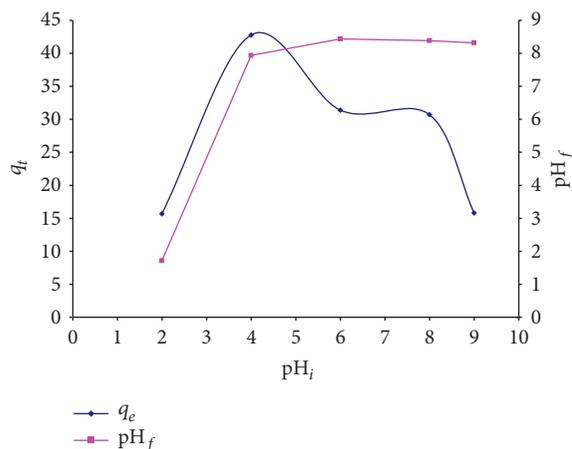


FIGURE 1: Effect of pH on adsorption of MB onto PLC (agitation speed = 200 RPM, $C_0 = 100 \text{ mg/dm}^3$, dosage = 2 g/dm^3 , $T = 303 \text{ K}$, particle size = $125 \mu\text{m}$).

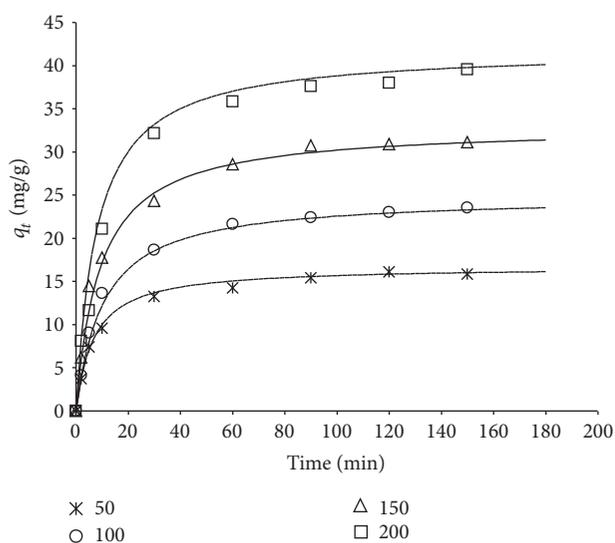


FIGURE 2: Effect of Concentration on adsorption of MB onto PLC. (agitation speed = 200 RPM, dosage = 2 g/dm^3 , $T = 303 \text{ K}$, particle size = $125 \mu\text{m}$).

TABLE 1: Effect of pH on MB adsorption onto PLC.

pH	$k_2 \text{ (g mg}^{-1} \text{ min}^{-1})$	$q_{m,pre} \text{ (mg g}^{-1})$	R^2
2.0	0.0009	20.42	0.936
4.0	0.0030	43.81	0.998
6.0	0.0060	31.40	0.995
8.0	0.0030	31.34	0.990
9.0	0.0039	16.20	0.977

the carbon obtained was used as it was, without further activation. The product was designated as PLC (plant leaf carbon).

2.2. Preparation of Dye Solution. The dyes used in this study were of commercial grade, obtained from a nearby textile

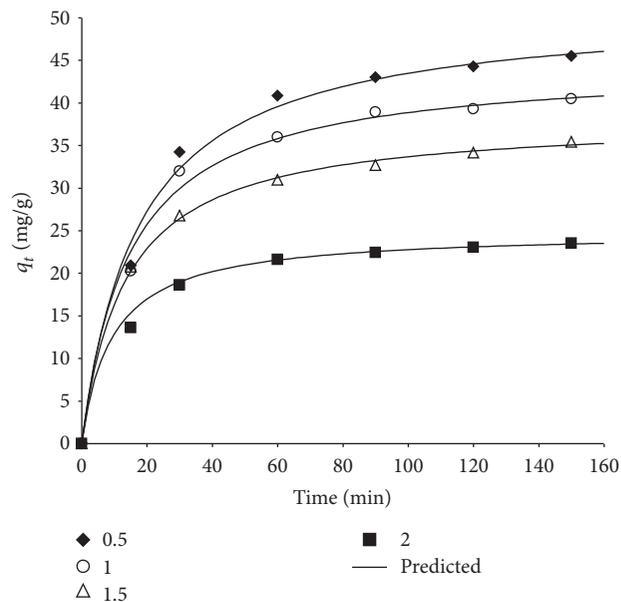


FIGURE 3: Effect of adsorbent dosage (agitation speed = 200 RPM, dye concentration = 100 mg/dm^3 , $T = 303 \text{ K}$, particle size = $125 \mu\text{m}$).

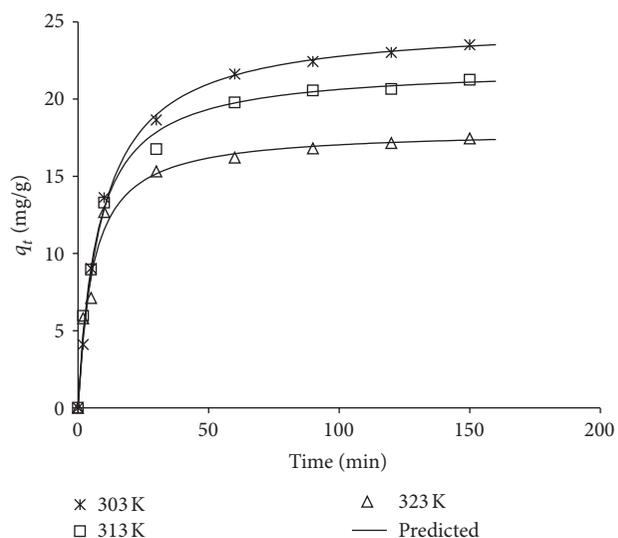


FIGURE 4: Effect of temperature (agitation speed = 200 RPM, dosage = 2 g/dm^3 , dye concentration = 100 mg/dm^3 , particle size = $125 \mu\text{m}$).

unit, and used without further purification. One gram of MB was dissolved in double distilled water to prepare stock solution (1000 mg/dm^3). Stock solution was later diluted to required concentrations. Methylene blue is selected in this study for it is (i) widely used by textile industries, (ii) well known for its adsorption characteristics, and (iii) toxic.

2.3. Batch Adsorption Studies. For each run, 100 mL of dye solution of desired concentration was taken in 250 mL conical flasks. pH of the solution was adjusted by addition

TABLE 2: Effect of concentration, adsorbent dosage, and solution temperature on adsorption of MB onto PLC.

C_0 (mg/dm ³)	D (g/dm ³)	T (K)	k_2 (g mg ⁻¹ min ⁻¹)	$q_{e,pre}$ (mg/g)	h (mg g ⁻¹ min ⁻¹)	R^2
50	2.0	303	0.0088	16.73	0.1472	0.999
100	2.0	303	0.0043	24.88	0.1070	0.999
150	2.0	303	0.0036	32.97	0.1187	0.999
200	2.0	303	0.0023	41.86	0.0963	0.999
100	0.5	303	0.0011	51.07	0.0562	0.998
100	1.0	303	0.0015	44.55	0.0668	0.998
100	1.5	303	0.0020	38.18	0.0764	0.999
100	2.0	313	0.0063	22.10	0.1392	0.998
100	2.0	323	0.0099	17.99	0.1781	0.999

of 0.1 N HCl or 0.1 N NaOH as needed. Definite quantity of PLC was added to the solution and the mixture was kept in an incubated orbital shaker at 200 RPM. Samples were drawn from the mixture using a fine tip syringe at regular time intervals for analysis. Samples were centrifuged and concentrations of supernatant solutions were determined by measuring absorbance at λ_{max} (662 nm) using UV-Vis spectrophotometer (Systronics 2201). When the absorbance exceeded 1.0, the solution was suitably diluted with doubled distilled water and concentration of diluted solution was determined.

Specific uptake of dye at any time “ t ” was calculated using the following equation:

$$q_t = \frac{(C_0 - C_t)}{D} \text{ mg/g}, \quad (1)$$

where q_t = specific uptake (amount of dye adsorbed/amount of adsorbent used, mg/g) at time “ t ” min, D = amount of adsorbent used (g/dm³), and C_0 and C_t are aqueous phase concentration of MB at time $t = 0$ and $t = t$ min, respectively (mg/dm³).

2.4. Adsorption Kinetics. Pseudo-second-order kinetic model [27–29] was used to test the kinetics in this work. Pseudo-second-order is expressed in linear form as follows:

Pseudo-second-order kinetic model

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}, \quad (2)$$

where q_t = dye uptake at time t (mg/g), q_e = equilibrium dye uptake at time t (mg/g), t -time (min), k_2 -pseudo second order rate constant (g mg⁻¹ min⁻¹).

Values of k_2 and q_e were estimated by linear regression.

2.5. Equilibrium and Thermodynamic Analysis. Most frequently employed isotherm models, namely, Langmuir [30] and Freundlich [31] were used in this study for equilibrium analysis.

Langmuir isotherm is given by the following expression:

$$q_e = q_m \frac{K_L C_e}{1 + K_L C_e}, \quad (3)$$

where C_e = equilibrium dye concentration in liquid phase (mg/dm³), K_L = Langmuir adsorption constant (dm³ mg⁻¹), q_m = Langmuir isotherm parameter, and maximum dye adsorbed/unit mass of adsorbent (mg g⁻¹). A dimensionless separation factor R_L expressed as a function of K_L is often employed as indicator of the feasibility of any adsorption process. It is defined as $R_L = 1/(1 + K_L C_0)$. Adsorption process is considered to be feasible if value of R_L lies between 0 and 1.

Freundlich isotherm is given by the following expression:

$$q_e = K_F C_e^{1/n}, \quad (4)$$

K_F = Freundlich constant (mg g⁻¹) (dm³/mg)^{1/n}, $1/n$ = Freundlich isotherm parameter.

Isotherm parameters were estimated by nonlinear regression using chi-square as an error estimate.

Thermodynamic parameters, namely, free energy, enthalpy, and entropy changes of adsorption were estimated using Vant Hoff's equation stated as follows [1]:

$$\ln K_L = -\frac{\Delta G^0}{RT} = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT}, \quad (5)$$

where ΔG^0 is free energy of adsorption (J mol⁻¹), ΔH^0 is change in enthalpy (J mol⁻¹), and ΔS^0 is change in entropy (J mol⁻¹ K⁻¹).

3. Results and Discussion

3.1. Effect of Initial Solution pH. One of the variables that most influence the adsorption process is pH. Thus, effect of pH on adsorption of MB on PLC was studied by varying the pH. Equilibrium uptakes of PLC at various initial solution pH are shown in Figure 1 along with corresponding final pH. However, earlier reports on adsorption on basic dyes on activated carbon suggest that equilibrium dye uptake increase with increasing initial solution pH. In the case of activated carbon, adsorption was due to the electrostatic attraction between the dye molecules and the adsorbent. As the activated carbon is protonated at low pH, adsorption of cationic dyes is favored by increase in initial solution pH. However, in this paper, uptake was low at both high (9) and low (2) pH. This suggests that electrostatic forces may

not be solely responsible for the dye adsorption on PLC. Since PLC used in this study had not been activated (does not contain active sites), this may be possible. However, this observation suggests that more detailed study is required. Estimated pseudo-second-order kinetic parameters are listed in Table 1. High R^2 value confirms the suitability of pseudo-second-order model to explain the kinetics of MB adsorption onto PLC. However, the remaining studies were conducted at pH 7 to avoid possible operational difficulties involved in the use of low pH (like 4).

3.2. Effect of Dye Concentration. Effect of concentration on adsorption of MB onto PLC was studied by varying concentration from 50 to 200 mg/dm³. Second-order kinetic parameters are shown in Table 2. It could be noticed that adsorption rate constant k_2 decreased with increase in concentration of the dye. Meanwhile, maximum specific uptake increased with increase in initial concentration. These observations were consistent with earlier reports [3, 32–37]. In order to predict the kinetic parameters k_2 in terms of initial dye concentration following models was proposed [38]:

$$k_2 = \frac{a_1 C_0}{a_2 C_0 + a_3}. \quad (6)$$

Regression coefficients (a_1, a_2 , and a_3) and coefficient of determinations (R^2) were determined using nonlinear regression. Substituting the regression coefficients, the following empirical equation was obtained:

$$k_2 = \frac{0.0187C_0}{7.49C_0 - 269.83}, \quad R^2 = 98.25\%. \quad (7)$$

Comparison of predicted pseudo-second-order kinetics with the experimental data is shown in Figure 2.

3.3. Effect of Adsorbent Dosage. Figure 3 illustrates the effect of adsorbent dosage on adsorption of MB. Dye uptake was found to decrease with increase adsorbent dosage. This was due to the fact that at smaller adsorbent dosages fraction of active sites saturated with dye increases. This was consistent with previous reports [29]. It was evident from this figure that initial rate of adsorption was much higher when the adsorbent dosage was higher. The values of kinetic parameters along with initial adsorption rate are given in Table 2. Initial rate of adsorption is defined as the product of kinetic rate constant and square of equilibrium uptake ($h = k_2 q_e^2$, mg g⁻¹ min⁻¹) [29].

3.4. Effect of Temperature. Effect of temperature is shown in Figure 4. Pseudo-second-order model fits the experimental data very well and the kinetic parameters are shown in Table 2 along with the regression coefficients. Uptake decreases with increase in temperature while kinetic constant increase with increase in temperature. Temperature dependency of the rate constant was exponential and follows Arrhenius law [38]. A plot of $\ln k_2$ versus $1/T$ was a straight line (plot not shown here). Activation energy of the process was determined from the slope of the Arrhenius plot. It was found to be 33.95 kJ/mol.

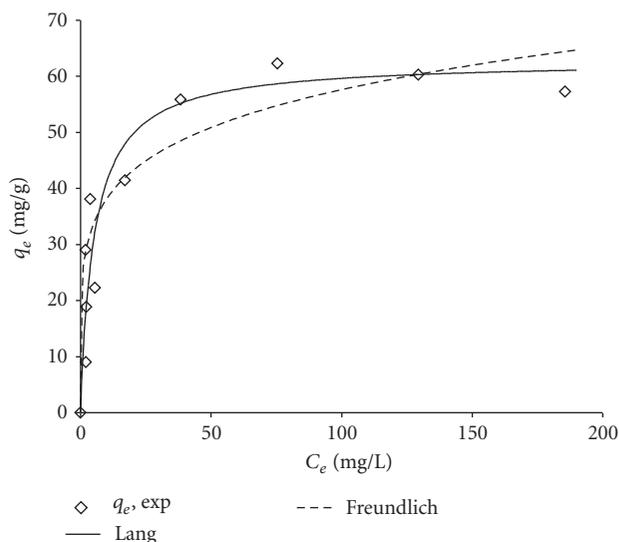


FIGURE 5: Equilibrium curve for adsorption of MB onto PLC at 303 K (agitation speed = 200 RPM, dosage = 2 g/dm³, particle size = 125 μm).

TABLE 3: Equilibrium parameters for adsorption of MB onto PLC.

	Langmuir	Freundlich	
K_L , dm ³ /mg	0.205	K_F , (mg/g) (dm ³ /mg) ^{1/n}	20.36
q_{max} , mg/g	61.22	N	4.41
χ^2	11.24	χ^2	12.87
R^2	94.33	R^2	92.29

3.5. Equilibrium. Best fitting isotherm model was found by using least square method and by minimizing nonlinear error function χ^2 [39–41]. It was found that Langmuir model fit the data well with high R^2 and low χ^2 values when compared to that of Freundlich model. The Langmuir and Freundlich isotherm parameters at 303 K are given in Table 3 along with R^2 and χ^2 values [39–41]. The comparison of experimental data with the predicted values at 303 K is given in Figure 5. Dimensionless parameter R_L suggests that adsorption of MB onto PLC is a feasible process. A comparison of adsorption potential of PLC with other adsorbents reported recently in the literature is given in Table 4. It could be seen that adsorption of potential of PLC was low compared to that of various activated carbons. This is expected and logical as the PLC reported in this study is not activated. However, the adsorption potential of PLC was better than that of many natural/unmodified alternate adsorbents reported in the literature.

3.6. Thermodynamic Analysis. The Langmuir parameter K_L was used to determine the thermodynamic parameter Gibbs free energy change which is an indicator of feasibility of the adsorption process. Meanwhile, a plot of $\ln K_L$ versus $1/T$ (figure not shown here) gives other thermodynamic parameters, namely, enthalpy and entropy changes of adsorption process. These values are shown in Table 5. Negative values

TABLE 4: Comparison of adsorption capacities of low cost adsorbents.

Adsorbent	Adsorption potential (mg/g)	Reference
Grass waste (GW)	457.640	[22]
Jackfruit peel (JFP)	285.713	[23]
Rejected tea (RT)	147–156	[24]
PLC	61.22	Present study
Wheat shell	16.56–21.50	[2]
<i>Posidonia oceanica</i> (L.) fibres	5.56	[25]
Pretreated dead <i>Streptomyces rimosus</i>	6.93–9.86	[26]

TABLE 5: Thermodynamics parameters for adsorption of MB onto PLC.

T (K)	ΔG (J/mol)	ΔH (J/mol)	ΔS (J/mol K)	R^2
293	-21650.0			
303	-23323.6	23146.4	153.1	0.9957
313	-24748.1			
323	-26270.6			

of ΔG indicate the feasibility of the process. Positive value of other ΔH indicates that the process is endothermic. Positive ΔS indicated increased disorder of the dye molecules on the solid surface after adsorption. Low value of ΔH also suggests that the adsorption may be due to physical binding forces. The earlier observation, increasing dye uptake with increasing temperature, is also consistent with this finding.

4. Conclusion

Adsorption of MB onto carbon derived from plant leaves (PLC), an agro-waste, was studied. Effects of variables like initial solution pH, initial dye concentration, amount of adsorbent used, and solution temperature were studied in batch mode. Adsorption kinetics was well described by pseudo-second-order model. Using Arrhenius equation, activation energy of MB adsorption on PLC was estimated to be 33.35 kJ/mol. Langmuir monolayer adsorption capacity (q_m) was determined to be 61.22 mg/g. From the thermodynamic parameters, it can be concluded that the process was spontaneous endothermic physisorption. From the results of the study, it could be concluded that PLC can be effectively used for the removal of methylene blue from aqueous solutions.

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