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A Comparative Study of Adsorption of Methylene Blue Dye onto Untreated *Platanus orientalis* (chinar tree) Leaves Powder and its Biochar - Equilibrium, Kinetic and Thermodynamic Study

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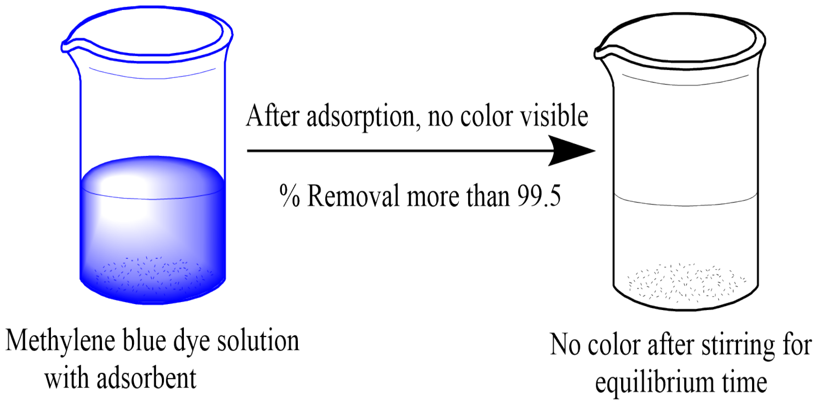
In the present investigation the adsorption behavior of methylene blue (MB) dye from aqueous solution onto untreated chinar leaves powder (CLP) and chinar leaves biochar (CLB) has been studied. To find out optimum conditions, adsorption process was carried out by varying different parameters such as contact time, adsorbent dose, concentration, temperature, salts, and pH. The equilibrium adsorption data were subjected to different popular isotherms (Langmuir, Freundlich, and Temkin) and kinetic models (pseudo-first order, pseudo-second order and intra-particle diffusion model). The thermodynamic study was also done on the adsorption process. The maximum monolayer adsorption capacities for MB dye onto given adsorbents are 1.53 mg/g (CLP) and 1.02 mg/g (CLB). Also the values of R_L and n suggest favourable adsorption process of MB dye onto CLP and CLB adsorbents. The pseudo-second order kinetic model is best obeyed by both the mentioned adsorbents for MB dye adsorption with R^2 value equal to 1. Thermodynamic study reveals that the adsorption process of MB dye is spontaneous, endothermic (CLB) and exothermic (CLP). At an equilibrium time of 20 min the uptake efficiency (> 99%) was recorded for both the low cost adsorbents (CLP and CLB) thus proved them fast and effective adsorbents for the removal of MB dye.

Graphical abstract



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1. Introduction

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Most of the dyes are synthetic in origin and are difficult to biodegrade because of having complex aromatic molecular structures. These dyes are widely used to colour various substances in industries such as textile, plastics, leather, paper, pharmaceutical, cosmetics, and food. During coloring these substances, a significant part of dyes is released through wastewater from these industries and finally find their path into fresh water bodies. The flora and fauna present in these water bodies get badly damaged as these dyes are toxic. The common effects are increase in chemical oxygen demand (COD) and reducing light penetration which have harmful effects on the photosynthetic phenomenon [1,2]. Among various dyes, methylene blue is one having wider applications like to colour cotton, wool, paper and hair. Although MB dye is not strongly hazardous but its acute exposure leads to harmful effects like increased heart rate, vomiting, jaundice, quadriplegia, tissue necrosis, and cyanosis [2]. Thus there is a need of its removal before its entry to water bodies.

There are several conventional methods for dye removal which include photo-degradation [3], chemical bleaching [4], biodegradation [5,6], photo-catalytic degradation [7-9], coagulation and flocculation [10], cation exchange membrane [11], electrochemical degradation [12,13], unicellular-enhanced ultra-filtration [14], fenton biological treatment [15], and adsorption [16-18]. Among these removal methods, adsorption on low cost biomaterials is an attractive alternative method, thus batch mode adsorption is selected for the present investigation.

There are various low cost biomaterials which have been investigated for the removal of dyes from aqueous solutions. Some among them are Platanus orientalis (chinar tree) leaf powder and its biochar [19], populous leaves [20], Buxus sempervirens leaf powder [21], cereal chaff [22], rice husk [23], giant duckweed [24], wheat shell [25], green peas shell [26], Morus nigra L. (mulberry tree) leaves powder and its biochar[27], Salix alba L. (Willow tree) leaves powder and its respective biochar[28], spines of Taxus bacata [29], cajanuscajan (red gram) seed husk [30], husk of green gram (Phaseolus aureus) seed [31], Cassia siamea (CS), Albizia labbeck (AL), Nerium indicum (NI), Durauta erecta (DE), and potato husk (PH) [32], mulberry tree leaves powder [33], mulberry tree leaves biochar[34], and guava leaf powder [35].

Fallen chinar leaves causing environmental problem in the community could be an alternative biomaterial from which low cost and efficient adsorbents like CLP and CLB can be prepared for the removal of methylene blue dye from aqueous solution, thus also from waste water. So the aim of this present investigation was to clean environment from undesirable fallen leaves and clean waste water containing MB dye by utilizing these chinar leaves. Although the adsorption study of MB dye onto Platanus orientalis (chinar tree) leaves powder have been found in the literature [36] but in the present investigation different conditions are taken for the comparative study of adsorption onto untreated Platanus orientalis (chinar tree) leaves powder and its biochar.

2. Material and Methods

2.1. Materials

The analytical grades of chemicals were used in the present investigation. Methylene blue (MB) (99.9 %) qualikems, fallen chinar leaves from which adsorbents (CLP and CLB) were prepared were collected from the local village (Putukhah Muqam Sopore Baramulla Kashmir India).

2.2. Instrumentation

Grinder machine (Usha Mixer Grinder (MG-3576) 750 Watt.), water distillation plant, pH meter (Equip - Tronics EQ-610), centrifuge (eltek multispin TC 650 F.), UV/Vis. Spectrophotometer (Systronics single beam 118.), hot plate stirrer (Macro Scientific Works (R),10A/UA, Jawahar Nagar, Delhi- 110007.), and analytical balance were used during the experimental investigations.

2.3. Preparation of Leaf powder and Biochar

Fallen chinar leaves were collected from local village Putukhah Muqam Sopore Baramulla Kashmir India where huge quantity of such fallen leaves exist in autumn season. These fallen leaves were collected and washed again and again to remove dirt from them. After washing the leaves were dried under sun for four days. The dried leaves were converted into powder (CLP) form with the help of grinder. A part of this powder was then converted into biochar CLB) in limited supply of air. Both the prepared adsorbents (CLP and CLB) were then extensively washed with distilled water to remove colour and dirt particles. Dirt and colour free adsorbents were first dried under sun for four days and then in an oven at 60 °C to attain constant weight. The adsorbents (CLP and CLB) were then stored in air tight voils for further use in experiments.

2.4. Adsorption process

The adsorbents (CLP and CLB) were used as such without any chemical treatment and with the help of UV/VIS. spectrophotometer the concentrations of methylene blue (MB) dye from its aqueous solutions were determined at 665 nm (λ_{max}.). The experimental MB dye solutions were prepared from aqueous stock solution (1 g/L) in 250 mL volumetric flask. During investigation 0.10 g of adsorbent dose was added to 20 mL MB dye solution in a 100 mL beaker capacity in all the parameters except in dosage variable (0.05-0.40 g). After stirring the solution containing fixed amount of adsorbent the supernatant solution was then taken in a cuvette of UV/VIS spectrophotometer to check its absorption values at λ_{max}. The percentage removal (% R), amount adsorbed at equilibrium (q_e) and amount adsorbed at time t (q_t) of the MB dye onto CLP and CLB adsorbents were calculated from initial and final absorbance values with the help of equation 1, 2 and 3 respectively.

$$\% \, R = \frac{C_o - C_t}{C_o} \qquad (1)$$

$$q_e = \frac{(C_o - C_e)V}{W} \qquad (2)$$

$$q_t = \frac{(C_o - C_t)V}{W} \qquad (3)$$

Where, C_o (mg/L), C_e (mg/L), C_t (mg/L) and V (L) are the initial concentration, equilibrium concentration, concentration at time t and volume of MB dye. W (g) is mass of adsorbent used in each reading.

2.5. Characterization

Both the adsorbents were already characterized in our previous literature by different techniques like Fourier transform infrared spectroscopy (FT-IR), scanning electron microscope (SEM), thermogravimetric analysis (TGA) and Carbon, hydrogen, nitrogen and sulphur (CHNS) analyser [19]. SEM reveals that CLB has greater porosity thus greater surface area because of thermal activation, therefore shows higher percentage removal of MB dye than that of CLP supporting experimental results.

3. Results and Discussion

The structure of MB dye is given in Figure 1 and results obtained are represented with the help of graphs. The experimental parameters were changed over a wide range to find out optimum conditions for the maximum removal of MB dye from its aqueous solution. These parameters include contact time, methylene blue (MB) dye solution concentration, amount of dose, salt treatment, temperature, and the pH.

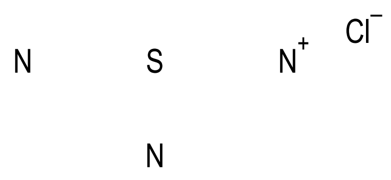

Fig. 1. Methylene blue structure.

3.1. Effect of contact time

The adsorption of MB dye adsorbed onto CLP and CLB as a function of time was investigated to find out the equilibrium time for maximum adsorption. 0.10 g of each adsorbent was added to 20 mL of MB dye aqueous solution having concentration 1.171 ppm at 298.15 K keeping all other variables constant. The effect of contact time was investigated from 5- 30 minutes, however the equilibrium was observed after 20 minutes at the given concentration. At equilibrium the percentage removal was more than 99.5 % showing highly efficient and rapid adsorption as shown in Figure 2.

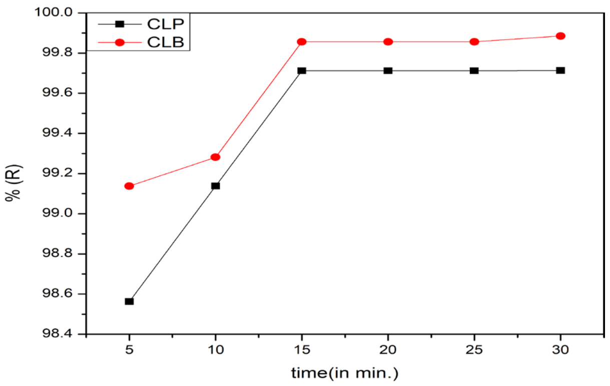

Fig. 2. Effect of contact time on adsorption of MB dye with CLP & CLB (conc. = 1.17 ppm, dose = 0.10 g, temp. = 25 °C, volume = 20 mL).

3.2 Effect of initial dye concentration

The initial concentration of MB dye was varied from 1.17 ppm to 7.12 ppm at constant temperature (298.5 K), dose (0.10 g), volume (20 mL) and contact time (20 min.). The results are shown in figure 3 which show that the percentage removals of MB dye decreases with increase in concentration. However, the amount of MB dye uptake (mg/g) increased with increase in MB dye concentration as the initial dye concentration provides an essential driving force to overcome the resistance of mass transfer between aqueous phase and solid phase [37].

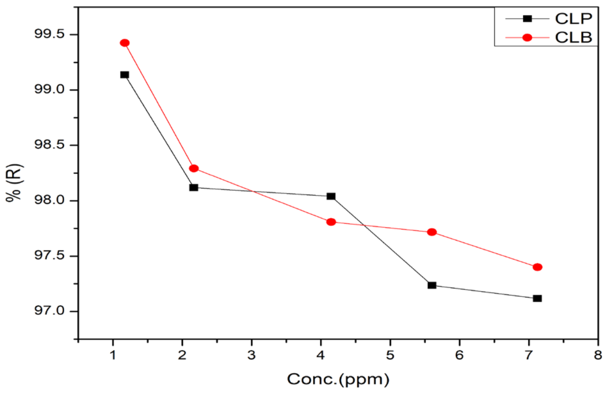

Fig. 3. Effect of conc. on adsorption of MB dye with CLP & CL (contact time = 20 min., dose = 0.10 g, temp. = 25 °C, vol. = 20 mL).

3.3 Effect of dose of adsorbent

To find out the suitable amount of adsorbent dose for the maximum removal of MB dye from its aqueous solution, all other parameters were kept constant as in contact time study and only amount of dose was changed for equilibrium time (20 min.). It can be observed in Figure 4 that the removal percentage increases with increase in adsorbent dose and reaches up to 99.7 % (CLP) and 99.9 % (CLB) for 0.20 g/ 20 mL. The increase in percentage removal is because of increase in surface area and therefore also adsorbent sites. However after 0.20 g/ 20 mL the removal percentage slowly decreases and may be due to hidening of adsorbent sites in this specified volume.

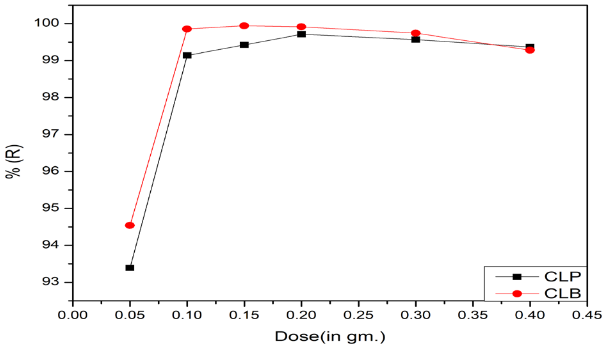

Fig. 4. Effect of dose on adsorption of MB dye with CLP & CLB (conc. = 1.17 ppm, contact time = 20 min., temp. = 25 °C, vol. = 20 mL).

3.4. Effect of salt

The salts KCl and $CaCl_2$ were used to check their impact on adsorption process of MB dye onto given adsorbents at 298.15 K, concentration 1.51 ppm, volume 20 mL, contact time 20 minutes and dose 1.10 g. Both the salts show negative effect on adsorption process and may be due to screening of surface charges as shown graphically in Figure 5 and Figure 6.

3.5. Effect of temperature

The effect of temperature for the adsorption of MB dye onto CLP and CLB was investigated from 293.15 K to 353.15 K keeping all other parameters constant as in contact time for equilibrium time. The two adsorbents (CLP and CLB) show two contrasting phenomenon i.e decrease and increase in adsorption capacities as shown in figure 7. The decrease in percentage removal with increase in temperature may be due to weakening of force of attraction between dye molecules and adsorbent (CLP) [38]. The increase in percentage removal for CLB adsorbent with increase in temperature may be due to dominance of increasing mobility and diffusion of MB dye molecules across the boundary layer and internal pores of adsorbent. Furthermore, with increasing temperature the viscosity of solution decreases and also causing swelling effect because of which the pore size increases enables large dye molecules free entry [39].

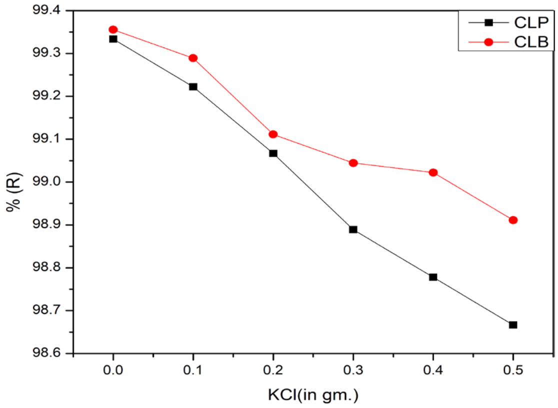

Fig. 5. Effect of KCl on adsorption of MB dye with CLP & CLB (conc. = 1.5 ppm, dose = 0.10 g, temp. = 25 °C, vol. = 20 mL).

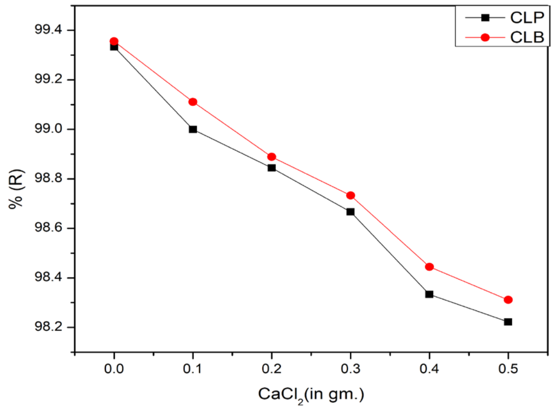

Fig. 6. Effect of $CaCl_2$ on adsorption of MB dye with CLP & CLB (conc. = 1.5 ppm, dose = 0.10 g, temp. = 25 °C, vol. = 20 mL).

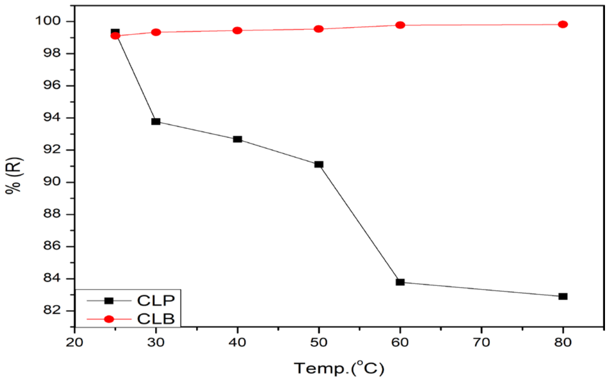

Fig. 7. Effect of temp. on adsorption of MB dye with CLP & CLB (conc. = 1.5 ppm, dose = 0.10 g, time. = 20 min., vol. = 20 mL).

3.6. Effect of pH

As in contact time study the parameters like concentration of MB dye (3.4 ppm), volume (20 mL), temperature (283.15 K), adsorbent dose (0.10 g), and contact time (20 min.) were kept constant changing pH values only. With increasing pH values the percentage removal also increases for both the adsorbents as shown in figure 8. This is because at lower pH (acidic) the H^+ ions compete with the positively charged MB dye molecules and thus reduces the adsorption capacity of adsorbents for MB dye molecules. Also the percentage removal increases by increase in pH values (basic) because the surface of adsorbents becomes more and more negatively charged attracting positively MB dye molecules by electrostatic force of attraction [1].

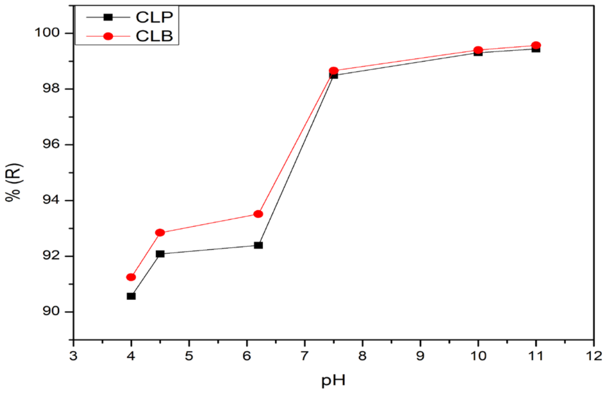

Fig. 8. Effect of pH on adsorption of MB dye with CLP & CLB. (conc. = 3.4 ppm, dose = 0.10 g, temp. = 10 °C , vol. = 20 mL).

3.7. Adsorption isotherms

The adsorption isotherms tell about the distribution of adsorbate molecules between liquid phase and solid phase at equilibrium. It is important to fit experimental data to different isotherms to find out the suitable isotherm model for design purpose. Three isotherm models were used for the present investigation namely Langmuir, Freundlich, and Temkin isotherm model. The linear equation for the Langmuir model [40,41] is given in equation 4 and graphically represented in figure 9 for the adsorption of MB dye by CLP and CLB adsorbents.

$$\frac{1}{q_e} = \frac{1}{q_o} + \frac{1}{q_o b C_e} \qquad (4)$$

Where, q_e (mg/g) is the amount of MB dye adsorbed onto CLP and CLB, C_e (mg/L) is the equilibrium concentration of MB dye in aqueous solution, q_o (mg/g) and b (L/mg) are Langmuir constants representing adsorption capacity and adsorption rate respectively. When $1/q_e$ was plotted against $1/C_e$, straight lines were obtained with slopes $1/q_o b$ and intercepts $1/q_o$. The important characteristics of Langmuir isotherm can be expressed by a dimensionless constant known as separation factor or equilibrium factor (R_L) and is given by [42].

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

Where, C_o is the highest initial dye concentration (mg/L) and b is Langmuir constant. The values of R_L indicate the favorability of adsorption process with respect to Langmuir isotherm model. The adsorption process having R_L value greater than 0 and less than 1 ($0 < R_L < 1$) is considered to be favorable adsorption process while as $R_L > 1$ (unfavourable), R_L

= 1(linear), and R_L = 0 (irreversible) [28]. The values of R_L for MB dye adsorption onto CLP and CLB lies between 0 and 1 indicating favourable adsorption process under given sets of conditions. These calculated R_L values and Langmuir constants are given table 1.

Freundlich isotherm model [43] assumes heterogeneous adsorption sites and its linear form is:

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \qquad (6)$$

Where, q_e (mg/gm) is the amount of MB dye adsorbed, C_e is equilibrium concentration of MB dye, K_F [mg/g $(L/g)^{1/n}$] is Freundlich constant which represents the adsorption capacity of adsorbent and n is also a constant giving an idea about how the favourable adsorption process is. By plotting a graph between $\ln q_e$ and $\ln C_e$, a straight line with slopes 1/n and intercepts $\ln K_F$ were obtained as shown in figure 10 and the values of Freundlich constants are listed in table 2. The value of slope 1/n represents adsorption intensity or surface heterogeneity and its value closer to 0 represents more heterogeneous surface [44].

Temkin isotherm [45] is represented by the following linearized form of equation:

$$q_e = B \ln A_T + B \ln C_e \qquad (7)$$

$$B = \frac{RT}{b_T}$$

Where, q_e and C_e have usual meanings as in Langmuir and Freundlich isotherms, A_T is known as equilibrium binding constant and gives maximum binding energy and B is also a constant related to adsorption heat. A graph plotted between q_e and $\ln C_e$ gives slopes equal to the value of B and intercepts $B \ln A_T$ as shown in figure 11 and the Temkin parameters are listed in Table 3.

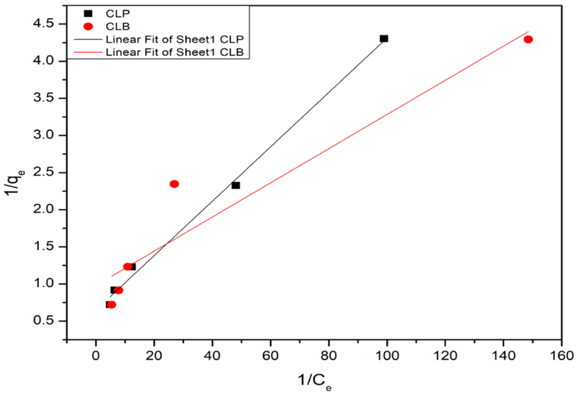

Fig. 9. Langmuir isotherm plot of MB dye on CLP & CLB.

Among three mentioned isotherm models the Langmuir yielded best fit model for MB-CLP adsorbate adsorbent system with R^2 = 0.99, however for MB-CLB adsorbate adsorbent system the Freundlich model is best obeyed with R^2 = 0.98.

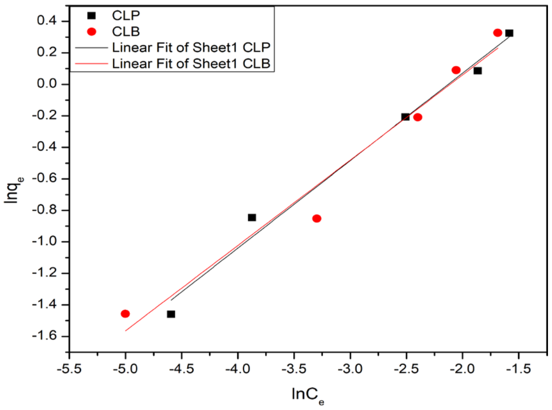

Fig.10. Freundlich isotherm plot of MB dye on CLP & CLB.

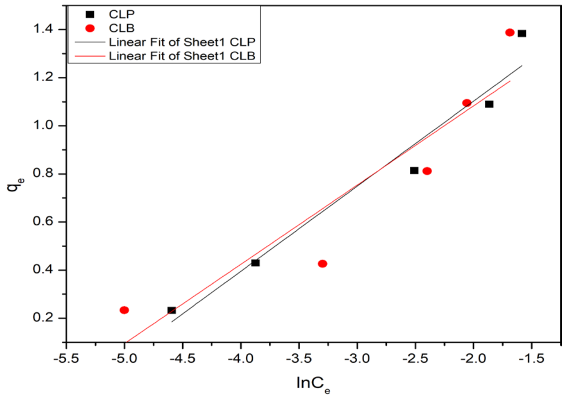

Fig.11. Temkin plot of MB dye on CLP & CLB.

Table 1. Langmuir isotherm data for CLP & CLB.

CLP	**Q_o (mg/g)**	**b (L/mg)**	**R_L**	**R^2**
	1.53	17.72	0.007	0.99
CLB	**Q_o (mg/g)**	**b (L/mg)**	**R_L**	**R^2**
	1.02	49.00	0.003	0.88

Table 2. Frundlich isotherm data for CLP & CLB.

CLP			
1/n	**n**	**K_f**	**R^2**
0.55	1.80	3.26	0.98
CLB			
1/n	**n**	**K_f**	**R^2**
0.54	1.84	3.13	0.95

Table 3. Temkin isotherm data for CLP & CLB.

CLP	**A_T**	**B**	**b_T**	**R^2**
	168.59	0.35	7022.15	0.94
CLB	**A_T**	**B**	**b_T**	**R^2**
	198.69	0.32	7534.40	0.80

3.8. Kinetic studies

The kinetic study is an important aspect as it helps us to investigate the mechanism of adsorption process. Two popular kinetic models namely pseudo-first-order and pseudo-second-order kinetic model have been investigated in the present study for MB dye adsorption onto CLP and CLB adsorbents. The linear form of pseudo-first-order kinetic model [46] given by Lagergren is:

$$\log(q_e - q_t) = \log q_e - \frac{K_1}{2.303} t \qquad (8)$$

Where, q_t and q_e are the amounts of MB dye adsorbed at time t (minutes) and at equilibrium respectively. K_1 (min.$^{-1}$) is rate constant obtained from the value of slope when $\log(q_e - q_t)$ against t is plotted as shown in figure 12. However, from the values of intercepts q_e can be calculated. The parameters for Lagergren model are given in table 4 which indicates that MB dye adsorption onto CLP and CLB do not follow this model as R^2 values are very low and calculated q_e values do not match with the experimental values.

The experimental data were further analyzed by using pseudo-second-order kinetic model [47] represented by its linear form as:

$$\frac{t}{q_t} = \frac{1}{K_2 \times q_e^2} + \frac{1}{q_e} \times t \qquad (9)$$

Where, q_t and q_e has its usual meanings as in pseudo-first-order kinetic model while as K_2 [g/(mg min)] is pseudo-second-order rate constant. The values of q_e and K_2 were obtained from the slopes and intercepts of linear plot between t/qt versus t (figure 13). This model is best fit model for our adsorbate-adsorbent systems as R^2 values are 1 for both the adsorbents (CLP and CLB). The experimental values of q_e also agree with the calculated values of q_e, thus the adsorption of MB dye onto given adsorbents is best described by this model having high correlation coefficients (R^2). The parameters related to pseudo-second-order kinetic model are listed in table 5. With the help of K_2, another parameter v_o (initial sorption rate) was calculated at t → 0.

$$v_o = K_2 \times q_e^2 \qquad (10)$$

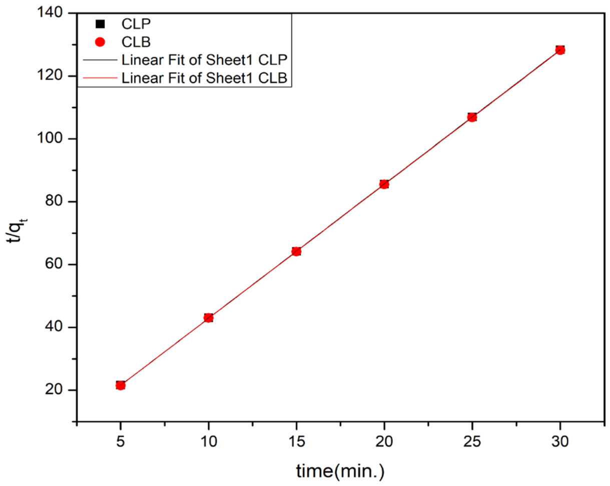

Fig.12. Pseudo first order kinetic model of MB dye on CLP&CLB.

Table 4. Pseudo-first order kinetic model data.

CLP	R^2	K_1	q_e
	0.68	-0.56	0.03
CLB	R^2	K_1	q_e
	0.68	-0.19	0.004

Table 5. Pseudo-second-order kinetic model data.

CLP	R^2	q_e	K_2	V_0
	1	0.23	64.59	3.54
CLB	R^2	q_e	K_2	V_0
	1	0.23	79.01	4.34

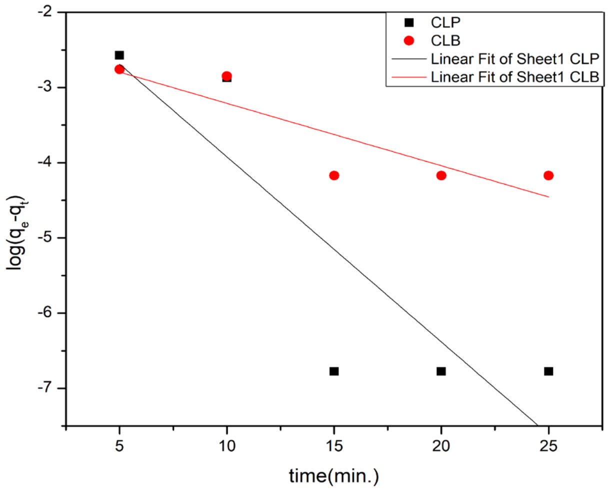

Fig.13. Pseudo second order kinetic model of MB on CLP&CLB.

3.8.1. *Intraparticle diffusion model*

To investigate the Intraparticle diffusion mechanism, Weber and Morris [48] plot was used.

$$q_t = K_{id} t^{0.5} + I \qquad (11)$$

Where, K_{id} represent intra-particle diffusion rate constant (mg/g min$^{0.5}$) and can be obtained from the slopes of linear plot of qt versus $t^{1/2}$ (figure 14). The intercepts I (mg/g) give an idea about the thickness of the boundary layer. Both K_{id} and I values are listed in table 6. Applying this model to our adsorbate-adsorbent systems, the linear plots do not pass through the origin indicating that the Intraparticle diffusion model is not the sole rate controlling step. similar results were also observed in the literature [49].

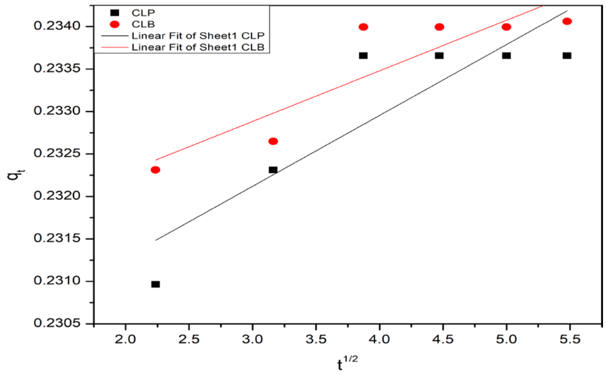

Fig.14. Intraparticle diffusion model of MB on CLP&CLB.

Table 6. Intraparticle diffusion model data.

CLP	R^2	Intercept (I)	Slope (K_{id})
	0.74	0.22	8.34
CLB	R^2	Intercept (I)	Slope (K_{id})
	0.75	0.23	5.95

3.9. Thermodynamic studies

The thermodynamic equations 12, 13 and 14 are used to calculate the thermodynamic parameters (ΔG°, ΔH° and ΔS°) by plotting a graph between $\ln K_c$ versus 1/T (figure15 and equation 14). To calculate such parameters the temperature was varied from 293.15 K to 353.15 K. The thermodynamic equations are:

$$\Delta G^o = -RT\ln K_c \qquad (12)$$

$$\Delta G^o = \Delta H^o - T\Delta S^o \qquad (13)$$

Using Van't Hoff equation Δ*H*° and Δ*S*° were calculated for the adsorption process of MB dye onto CLP and CLB.

$$\ln K_c = \frac{\Delta S^o}{R} - \frac{\Delta H^o}{RT} \qquad (14)$$

Where, K_c is equilibrium constant, R is gas constant and T is absolute temperature. From the linear plot of $\ln K_c$ versus 1/T, Values of slope ($\Delta H^o/R$) and intercepts ($\Delta S^o/R$) give ΔH^o and ΔS^o values respectively. However, K_c values were obtained from the following relation:

$$K_c = \frac{q_e}{C_e} \qquad (15)$$

Where, C_e (mg L^{-1}) is equilibrium concentration of MB dye solution after stirring in a batch mode adsorption and q_e (mg/gm) is amount of adsorbate adsorbed onto the adsorbents.

At all the considered temperatures the Δ*G*° values were negative indicating favorable and spontaneous adsorption process. The negative value of Δ*G*° decreases with increase in temperature for CLP and vice versa for CLB as shown in table 7. Adsorption of MB dye onto CLP is exothermic process as ΔH^o is negative and endothermic for CLB having positive ΔH^o. The ΔS^o values are also positive and negative indicating overcoming of hydration energy for CLB and adsorption of MB dye molecules for CLP respectively.

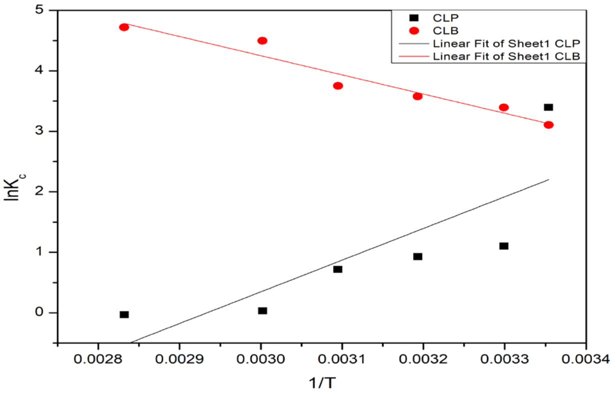

Fig. 15. Van't Haff plot for MB dye adsorption onto CLP & CLB.

Table 7. Thermodynamic parameters for CLP & CLB.

Temperature (k)	CLP	CLB				
	ΔG=-RTlnKc	**ΔG=-RTlnKc**	**CLP**	**R^2**	**ΔS**	**ΔH**
				0.57	-0.13	-43.54
293.15	-8.41	-7.69	**CLB**	**R^2**	**ΔS**	**ΔH**
303.15	-2.78	-8.55		0.92	0.11	26.38
313.15	-2.41	-9.31				
323.15	-1.93	-10.08				
333.15	-0.089	-12.45				
353.15	0.092	-13.86				

4. Conclusions

Chinar leaves powder (CLP) and chinar leaves biochar (CLB) proved to be very effective and low cost adsorbents for the adsorption of methylene blue dye from its aqueous solution having percentage removal (> 99%) at the given concentration. CLB adsorbent shows more percentage removal of MB dye than that of CLP which can be observed in graphs. Both the adsorbents are rapid and obeyed dominantly pseudo-second-order kinetic model. The equilibrium data were analyzed by using Langmuir, Freundlich and Temkin models. CLP followed Langmuir isotherm model while as CLB shows better agreement with Freundlich isotherm model for MB dye. Thermodynamic study revealed that the adsorption process of MB dye onto the CLP and CLB adsorbents is feasible and spontaneous. Also CLB adsorbent has better efficiency at higher temperature than that of CLP adsorbent for the same dye. Lastly, increase in pH value towards basic increases the removal percentage of MB dye by the mentioned adsorbents.

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Author Contributions

Fairooz Ahmad Khan: Investigation, Formal analysis, Writing-original draft; Mushtaq Ahmad Bhat and Pathan Mohd Arif: Formal analysis, Software; Mazahar Farooqui: Conceptualization, Supervision, Writing- review & editing.

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