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Equilibrium, kinetic and thermodynamic study for the efficient removal of malachite green dye onto untreated *Morus nigra* L. (mulberry tree) leaves powder and its biochar

Fairooz Ahmad Khan & Mazahar Farooqui*

Maulana Azad College Of Arts, Science & Commerce, Aurangabad 431 001, Maharashtra, India

E-mail address: mazahar_64@rediffmail.com

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In the present investigation the removal of malachite green (MG) dye from its aqueous solution by batch mode adsorption study onto mulberry leaves powder (MLP) and mulberry leaves biochar (MLB) has been proved to be efficient with removal percentage more than 95 % at 0.3 gm dose in 35.8 mg/L (20 ml) MG dye solution for 35 minutes. In this batch mode adsorption study various parameters like contact time, effect of dose, concentration, temperature and *p*H have been studied to investigate the effect of such parameters on MG dye adsorption process onto mentioned adsorbents. Among three commonly used isotherms (Langmuir, Freundlich, and Temkin), Freundlich isotherm model is best fit model with $R^2 = 0.99$. The kinetic study reveals that pseudo-second-order kinetic model is best followed having also $R^2 = 0.99$. Thermodynamically, the adsorption process of MG dye onto MLP is endothermic, exothermic onto MLB, and spontaneous in nature. Accordingly, the results obtained have shown that the adsorbents (MLP and MLB) can be used as low cost, rapid, and effective adsorbents for the removal of malachite green dye from its aqueous solution, thus also from wastewater.

Keywords: Biochar, Endothermic, Malachite green, Mulberry leaves powder, Spontaneous

Malachite green (MG) dye is one of the hazardous dyes and have been banned in many countries like US food and drug administration but still finds its use in some countries like India because of having low cost, easy availability and lack of proper alternative. This dye is used to colour textile, pharmaceutical, plastics, leather, printing, rubber, paint, pulp, cosmetic, food, and paper. The dye applied to the substrates is not fully utilized but a significant part of it gets lost into the wastewater during application. This lost part of dye finds its way to large water bodies having flora and fauna thus damaging them, therefore the need of its removal. There are three methods for the separation and removals of these dyes from wastewater namely biological, chemical, and physical. Some chemical and physical methods include coagulation, ion-exchange, reverse osmosis, precipitation, membrane filtration, adsorption, electrochemical techniques, and ozonation. Among various treatment methods adsorption is considered to be simple, efficient, low cost and environment friendly method for wastewaters and refining sources[1].

There are many different types of adsorbents used so for the removal of dyes from aqueous solutions and also from wastewaters. However, the preference has been given to the low cost, easy available and biodegradable adsorbents like agricultural wastes. In the literature different plant materials have been searched for the adsorption of dyes such as Carica papaya wood[2], Platanus orientalis (chinar tree) leaf powder and its biochar[3], Peltophorum pterocarpum fruit shells[4], Cladophora sp.[5], waste biomass[6], Algal biochar and its composites with Kombucha SCOBY[7], willow tree leaves powder and its respective biochar[8], mulberry leaves powder and its biochar[9] etc.

During investigation various parameters like contact time, concentration, temperature etc have been checked for MG dye adsorption onto MLP and MLB adsorbents. Both MLP and MLB have been characterized for FTIR[9]. The isotherms, kinetics and thermodynamics study was also performed to search the mechanism, rate and spontaneity of adsorption of MG dye onto our adsorbents. The plant (mulberry tree) leaves were selected as these trees are abundantly found in our region (first author) and the leaves of these trees fall in autumn season (deciduous trees) thus become waste. Secondly, the mulberry leaves contain functional groups showing attraction towards basic MG dye. Because of such reasons these leaves were selected and converted into adsorbents (MLP and MLB). The percentage removal of MG dye by MLP and MLB adsorbents shows almost similarity with the adsorbents found in literature like rice husk (95.7%) and wood apple shell (98.87%)[10,11].

Experiment Section

Materials

All the chemicals which were used in the present search were of analytical grades. Malachite green (MG) (1% aqueous solution) Loba Chemie, purity > 99 %, *p*H of the MG dye solution was adjusted by either 0.1 M NaOH solution or 0.1 M HCl solution. Mulberry leaves from which adsorbents (MLP and MLB) were prepared were collected from locality (Dungdara Dawlatpora Kreeri Baramulla Kashmir India) were large number of mulberry trees exist.

Instrumentation

UV/Vis. spectrophotometer (Systronics single beam 118.), grinder machine (Usha Mixer Grinder (MG-3576) 750 Watt.), water distillation plant, hot plate stirrer (Macro Scientific Works (R),10A/UA, Jawahar Nagar, Delhi- 110007.), *p*H meter (Equip - Tronics EQ-610), centrifuge (eltek multispin TC 650 F.), electric muffle furnace (Shiva DC – 42A) and analytical balance were used while performing experimental investigations.

Preparation of adsorbents (MLP and MLB)

Mulberry leaves were collected from locality and were first washed with tap water to get rid from undesired materials from their surface. The washed leaves were then sun dried for four days and was then crushed into powder form in a grinder named as mulberry leaves powder (MLP). A part of the leaves powder was converted into biochar in limited supply of air in an electric muffle furnace at 500°C with 31% yield and was named as mulberry leaves biochar (MLB). Both MLP and MLB adsorbents were again and again washed with distilled water to remove colour and dirt particles if any. After washing both the adsorbents were dried under sun for four days and then in an oven at 60°C to attain constant weight. For further use in experiments, these adsorbents were stored in air tight containers.

Adsorption process

The biosorption tests were conducted by preparing a stock solution (1 g/L) from which other desired concentrations were prepared. A definite volume (20 mL) of dye solution to which fixed amount of adsorbent dose was added in a 100 mL capacity beaker and stirred for fixed time, temperature, concentration, and *p*H on a magnetic stirrer. However, *p*H of MG dye solution was not studied in basic medium as base (NaOH) discolourise it and was therefore studied only in acidic medium at 4.2, 3.6 and 3.3 *p*H values. The supernatant solution obtained was then centrifuged for five minutes to settle down the biomass and was then transferred to the cuvette of spectrophotometer for checking its absorption values at λ_{max} (620 nm). Using following three equations 1, 2, and 3 the percentage removal of MG dye (% R), amount of MG dye adsorbed at equilibrium (q_e) and amount of MG dye adsorbed at time t (q_t) respectively can be calculated.

$$\% \mathrm{R} = \frac{C_o - C_t}{C_0} \quad \ldots (1)$$

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

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

Where, q_e and q_t stands for amount of MG dye (mg/gm) adsorbed onto adsorbents at equilibrium and at any time t respectively. C_o (mg/L), C_t (mg/L), C_e (mg/L), W (g) and V (L) are the initial concentration, concentration at time t, equilibrium concentration, mass of adsorbent and volume of MG dye solution.

Results and Discussion

The MG dye solution was treated with MLP and MLB adsorbents and the parameters were varied over a wide range to find out the optimum conditions for its maximum removal. The parameters those were studied for the present investigation include contact time, adsorbents dosage, concentration, salt treatment, temperature and *p*H. The results obtained for these parameters are represented in their respective graphs.

Effect of contact time

Contact time study is important for the evaluation of kinetics of adsorption process. Figure 1 (a) represents the role of contact time on the adsorption of MG dye by MLP and MLB adsorbents. For this study all the parameters like concentration (35.85 mg/L), adsorbent dose (0.10 g), temperature (10°C) and volume (20 mL) were kept constant keeping changing only of contact time. From figure it can be observed that the adsorption process can be divided into two steps: (1) a rapid adsorption (10 min) in which more than 85 % dye removal had taken place for both the adsorbents because of available vacant

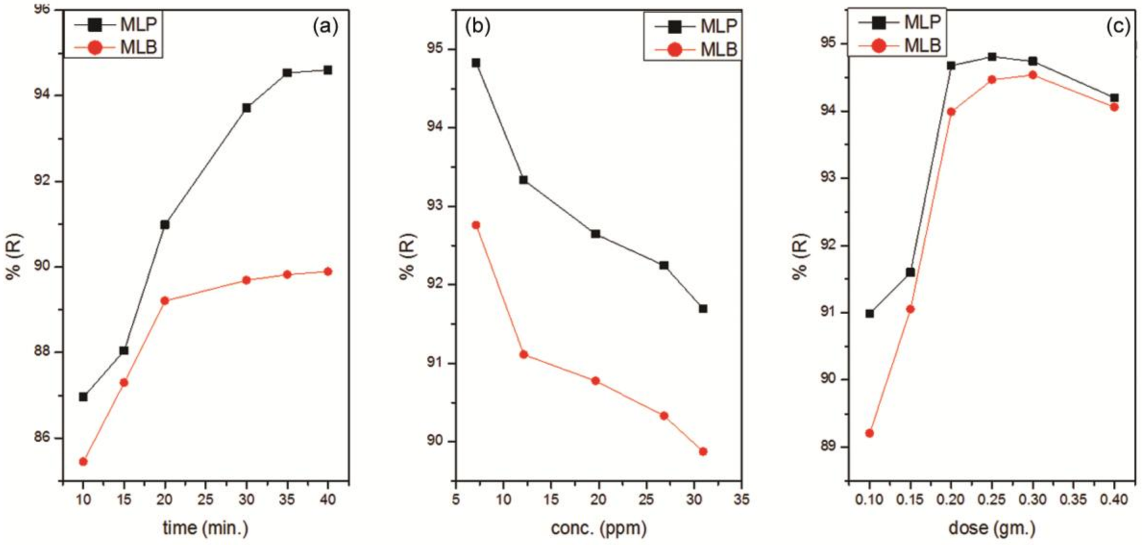

Fig. 1 — (a) Effect of contact time on adsorption of MG dye with MLP &MLB (conc. = 37.7 ppm, dose = 0.10 g, temp. = 10 ^{o}C, volume = 20 ml) (b) Effect of conc. on adsorption of MG dye with MLP & MLB (contact time = 35 min., dose = 0.20 g, temp. = 10 ^{o}C, vol. = 20 ml) (c) Effect of dose on adsorption of MG dye with MLP & MLB(conc. = 35.8 ppm, contact time = 35 min., temp. = 10 ^{o}C, vol. = 20 ml).

sites on both the adsorbents (MLP and MLB); and (2) slow adsorption process from 10 min to 35 min where equilibrium was attained and percentage removal of MG dye reached up to 94.5 % (MLP) and 89.8 % (MLB) at equilibrium. This slow process of adsorption was shown because of unavailable sufficient vacant sites on the adsorbents.

Effect of initial dye concentration

Concentration variation plays an important role in studying the adsorption isotherms. This study was carried out at constant contact time (35 min), adsorbent dose (0.2 g), temperature (10°C) and volume of dye solution (20 mL) while varying only initial concentration of MG dye solution. It can be observed from Fig. 1 (b) that the percentage removal of dye onto given adsorbents decreases with increase in concentration and this decrease may be due to the saturation of vacant sites of adsorbents[12]. However, the amount (mg/g) of MG dye adsorbed increased with increase in initial dye concentration as initial concentration of dye provides an essential driving force for adsorbate transfer between solution phase and adsorbent surface[13].

Effect of dose of adsorbent

The experiments for this parameter were carried out at constant contact time, adsorbent dose, temperature and volume as in concentration parameter with 35.85 mg/L concentration of MG dye. The results present in Fig. 1 (c) shows increase and then gradual decrease in percentage removal of MG dye from its aqueous solution while increasing adsorbents dose. The increase in removal percentage (90.9-94.8 %) for MLP and 89.2-94.5 % for MLB may be because of increase in unoccupied sites on the adsorbent surfaces and then gradual decrease may be hidening of vacant sites because of aggregation of adsorbent particles in specified volume at higher dose[14].

Effect of salt

Varying amounts of two salts (KCl and $CaCl_2$) were added to the fixed volume (20 mL) of aqueous MG dye solution (35.8 mg/L) keeping all other parameters constant. The effect of these two salts can be observed graphically from Figs 2 (a) and 2 (b). From figure the percentage removal of MG dye decreases with increase in KCl amount for MLP but the removal percentage increases (93.9-98.4 %) up to a particular amount of KCl for MLB. However, by the addition of $CaCl_2$ the percentage removal decreases for both the adsorbents (MLP and MLB) as shown in figure. The decrease in removal percentage of MG dye may be due to screening of positively charged ions of these salts and increase in percentage removal up to particular amount of KCl may be due to the dominance of activation of biochar over its screening factor.

Effect of temperature

Temperature is an essential parameter as in industries where dyes are used release warmer effluent containing waste dyes. So the adsorbents

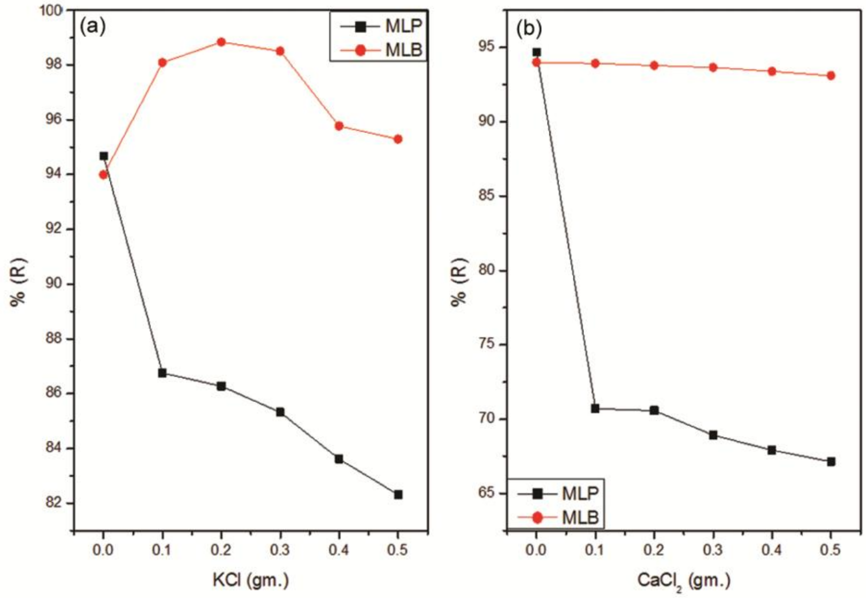

Fig. 2 — (a) Effect of KCl on adsorption of MG dye with MLP & MLB (conc. = 35.8 ppm, dose = 0.20 g, temp. = 10 °C, vol. = 20 ml) (b) Effect of $CaCl_2$ on adsorption of MG dye with MLP & MLB (conc. = 35.8 ppm, dose = 0.20 g, temp. = 10 °C, vol. = 20 ml).

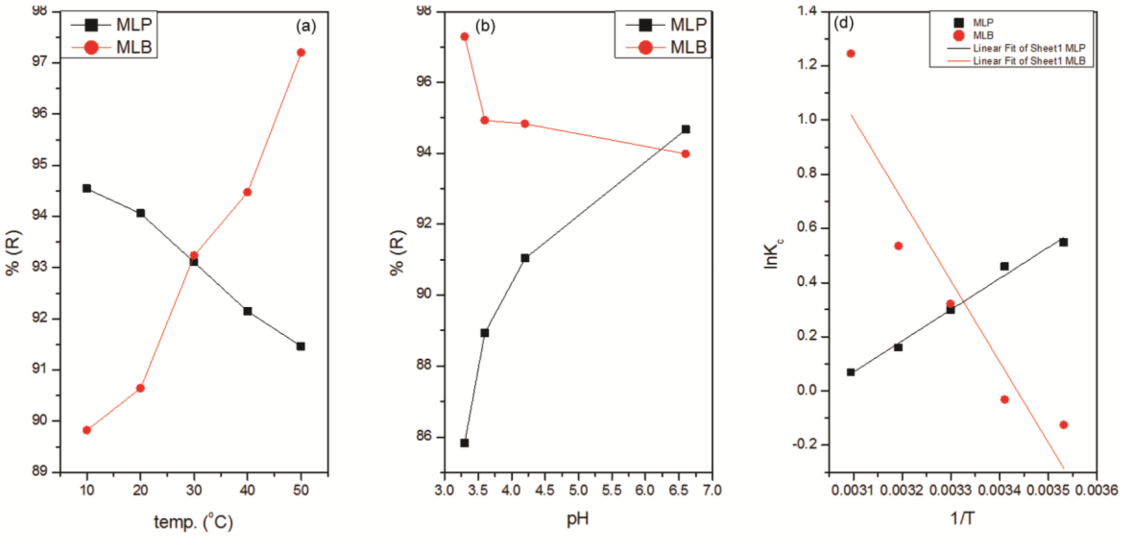

Fig. 3 — (a) Effect of temp. on adsorption of MG dye with MLP & MLB (conc. = 35.8 ppm, dose = 0.20 g, time. = 35 min., vol. = 20 ml) (b) Effect of pH on adsorption of MG dye with MLP & MLB (conc. = 35.5 ppm, dose = 0.20 g, temp. = 10 °C , vol. = 20 ml) (c) Van't Haff plot for MG dye adsorption onto MLP & MLB.

showing adsorption at higher temperatures are considered good ones for such effluent. Our adsorbents (MLP and MLB) for MG dye proved to be good at higher temperature especially MLB showing endothermic adsorption process. In studying this parameter only temperature was varied keeping all other parameters constant like contact time (35 min.), adsorbent dosage (0.20 g), concentration (35.8 mg/L) and volume (20 mL). It can be seen from Fig. 3 (a) that removal percentage of MG dye increases from 89.8-97.1% for MLB by increasing temperature from 10-50°C. However, there is little decrease in percentage removal (94.5-91.4%) of MG dye by MLP while increasing the same temperature indicating exothermic adsorption process.

Effect of pH

The adsorption of MG dye onto MLP and MLB was studied in acidic pH only as base (NaOH) discolourise the MG dye because of breaking conjugation. The MG dye percentage removal decreased by decreasing the *p*H for MLP, and increased by decreasing *p*H for MLB, as shown graphically in Fig. 3 (b). The decrease in percentage removal may be due to potonation[15] of large number of functional groups present on MLP surface as given by its FTIR. Also, there increase the competition between positively charged dye molecules and protons. However, the increase in percentage removal onto MLB by decrease in *p*H may be due to activation of adsorption sites which dominates the protonation as its most of the functional groups get escaped through volatile substances during its preparation from MLP.

Adsorption isotherms

The adsorption isotherms provide an essential tool for interpreting the distribution of adsorbate (dye) molecules between the adsorbent surface and solution phase at equilibrium. Adsorption isotherm also helps us to understand the adsorption capability of adsorbent as it studies the interaction between adsorbate and adsorbent. In this search three important isotherm models have been employed namely Langmuir[16,17], Freundlich[18], and Temkin[19]. Figure 4 (a) depicts the plot of Langmuir isotherm and its physical parameters are listed in Table 1 (a). The linear form of Langmuir isotherm is:

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

where, b (L/mg) and q_o (mg/gm) are Langmuir constants indicating adsorption rate and adsorption capacity respectively. q_e (mg/gm) is the amount of MG dye adsorbed at equilibrium and C_e (mg/L) is the equilibrium concentration. From intercept ($1/q_o$) and slope ($1/q_o b$) of linear plot between $1/q_e$ and $1/C_e$, the values of Langmuir parameters can be calculated. The computed dimensionless separation factor (R_L) indicates whether the adsorption process is favourable ($0 < R_L < 1$), unfavourable ($R_L > 1$), linear ($R_L = 1$) or irreversible ($R_L = 0$)[20]. The equation for the calculation of R_L is:

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

where, C_o is the highest initial MG dye concentration. The value of R_L for the present investigation lies between 0 and 1 indicating favourable adsorption process.

Freundlich isotherm model assumes multilayer adsorption that takes place on to the heterogeneous surface of an adsorbent. The stronger binding sites are occupied first and the binding strength depends upon concentration of an adsorbate at equilibrium[21]. The linear equation for Freundlich isotherm is:

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

where, K_F [mg/g (L/gm)$^{1/n}$] is the Freundlich constant giving an idea about bonding energy, adsorption capacity and quantity of adsorbate (MG) adsorbed at equilibrium. Slope 1/n value represents adsorption intensity or surface heterogeneity from which n value can be calculated giving favorability of adsorption

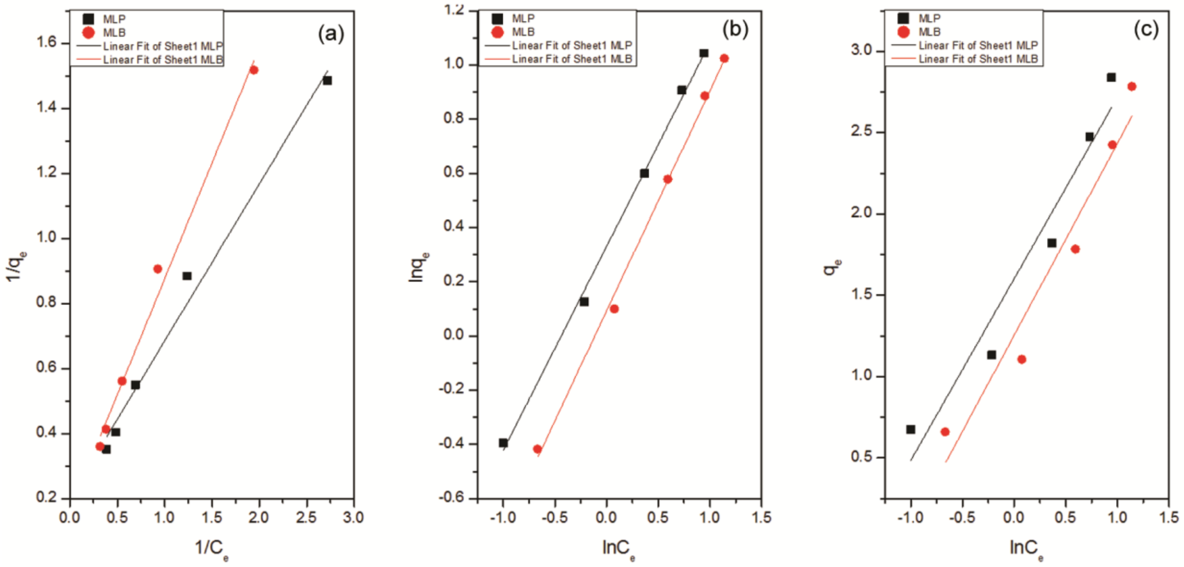

Fig. 4 — (a) Langmuir isotherm plot of MG dye on MLP & MLB (b) Freundlich isotherm plot of MG dye on MLP & MLB (c) Temkin plot of MG dye on MLP & MLB.

process[22]. The value of n from 1–10 is considered as favorable adsorption process. The linear plot of Freundlich isotherm ($\ln q_e$ vs. $\ln C_e$) is given in Fig. 4 (b) and its parameters are listed in Table 1 (b).

The Temkin isotherm describes the indirect adsorbate- adsorbate interactions and suggests that heat of adsorption decreases linearly with coverage because of these interactions[23]. The following equation 7 presents linear form of Temkin isotherm.

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

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

where, A_T is equilibrium binding constant giving maximum binding energy, B is also a constant that gives adsorption heat. The linear plot (figure 4c) of q_e against $\ln C_e$ gives slope B and intercepts $B\ln A_T$ from which value of B and A_T can be calculated. The values of these Temkin parameters are listed in Table 1 (c).

Kinetic studies

Kinetic study helps us to predict the mechanism of adsorption process and describes the adsorption capability of MLP and MLB adsorbents. Figures 5 (a), 5 (b), and 5 (c) for pseudo-first-order[24], pseudo-second-order[25], and intra-particle diffusion model[26] obtained from three linear equations namely equations 8, 9, and 10 and their physical parameters are listed in Tables 2 (a), 2 (b), and 2 (c), respectively. It was observed that pseudo-second-order kinetic model was the best fit model for both the adsorbents (MLP and

Table 1 — Isotherms data (a) Langmuir (b) Freundlich (c) Temkin for MG- MLP & MLB adsorbate adsorbent systems

		(a): Langmuir isotherm data for MLP & MLB		
MLP	Q_o (mg/gm.)	b (l/mg)	R_L	R^2
	4.9751	0.41702	0.07189	0.9838
MLB	Q_o (mg/gm.)	b (l/mg)	R_L	R^2
	6.1349	0.2289	0.12365	0.986
		(b): Freundlich isotherm data for MLP & MLB		
MLP	1/n	n	K_f	R^2
	0.75	1.333	1.39096	0.9965
MLB	1/n	n	K_f	R^2
	0.81	1.2345	1.09417	0.99
		(c): Temkin isotherm data for MLP & MLB		
MLP	A_T	B	b_T	R^2
	4.2016	1.116	2109.416	0.9328
MLB	A_T	B	b_T	R^2
	2.8942	1.180	1995.007	0.933

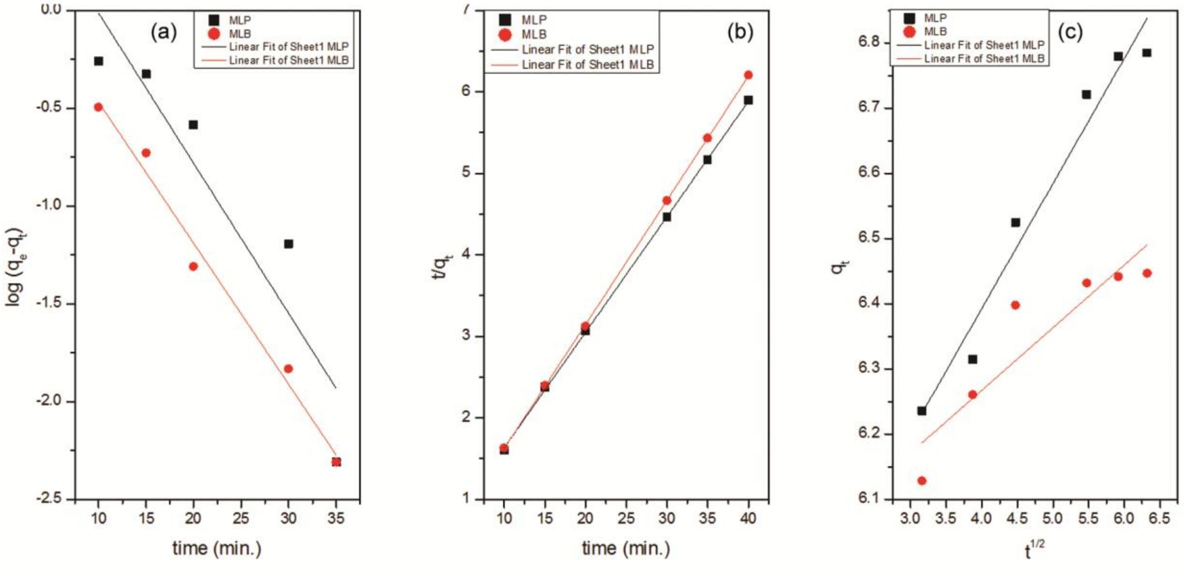

Fig. 5 — (a) Pseudo first order kinetic model of MG dye on MLP & MLB (b) Pseudo second order kinetic model of MG on MLP & MLB (c) Intraparticle diffusion model of MG on MLP & MLB.

Table 2 — Kinetic study data (a) pseudo-first order (b) pseudo-second order (c) intra-particle diffusion models for MG - MLP & MLB adsorbate adsorbent systems

		(a): Pseudo-first order kinetic model		
MLP	R^2	K_1		q_e
	0.829	-0.1612		5.649
MLB	R^2	K_1		q_e
	0.980	-0.1612		1.774
		(b): Pseudo-second order kinetic model		
MLP	R^2	q_e	K_2	V_0
	0.9997	7.0531	0.0927	4.6163
MLB	R^2	q_e	K_2	V_0
	0.9999	6.5629	0.2318	9.9860
		(c): Intraparticle diffusion model		
MLP	R^2	Intercept (I)		Slope (K_{id})
	0.9571	5.625		0.191
MLB	R^2	Intercept (I)		Slope (K_{id})
	0.802	5.882		0.096

Table 3 — Thermodynamic parameters for MG- MLP & MLB adsorbate adsorbent systems

Temperature(k)	MLP	MLB	MLP	R^2	ΔS	ΔH
	ΔG=-RTlnKc	ΔG=-RTlnKc		0.986	-0.0290	-9.5671
283.15	-1.29034	0.294121	MLB	R^2	ΔS	ΔH
293.15	-1.11911	0.077724		0.852	0.0853	24.839
303.15	-0.75546	-0.80956				
313.15	-0.41553	-1.39283				
323.15	-0.18479	-3.3432				

MLB) with correlation coefficients (R^2) equal to 0.99. Also, the experimental amount of MG dye adsorbed at equilibrium for MLP and MLB adsorbents were almost similar to that of calculated amount of MG dye adsorbed onto the said adsorbents. For intra-particle diffusion model the intercept is not zero indicating that more than one mechanism is involved in the present adsorption process. Such results were also found in the literature[27]. From equations 8, 9, and 10; K_1, K_2, and K_{id} represent pseudo-first-order rate constant (min.$^{-1}$), pseudo-second-order rate constant (g mg^{-1} min^{-1}), and intra-particle diffusion rate constant (mg/g min$^{0.5}$). q_e (mg/gm) and q_t (mg/gm) are the amounts of MG dye adsorbed at equilibrium and at time t respectively. From constant K_2 another parameter i.e initial sorption rate (ν_o) can be calculated with the help of equation 11.

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

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

$$q_t = K_{id} t^{0.5} + I \quad \ldots (10)$$

$$v_o = K_2 \times q_e^2 \quad \ldots (11)$$

Thermodynamic studies

Free energy change (ΔG^o) thermodynamic parameter is the driving force for adsorption process. The thermodynamic parameters (ΔG^o, ΔH^o and ΔS^o) are responsible for predicting the spontaneity, endo- or exo-thermicity of the adsorption process[28]. The change in free energy (ΔG^o), change in enthalpy (ΔH^o), and change in entropy (ΔS^o) were computed with the help of following equations.

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

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

Applying Van't Hoff equation (14) $\Delta H°$ and $\Delta S°$ were calculated from slope ($\Delta H^o/R$) and intercepts ($\Delta S^o/R$) of plot $\ln K_c$ vs. 1/T (Fig. 3c).

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

Where, K_c is equilibrium constant and its value was obtained by using following relation (15).

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

From Table 3, it can be observed that the adsorption process for the present investigation was spontaneous as ΔG^o values were negative and these negative values decrease and increase with rise in temperature for MLP and MLB adsorbents. The enthalpy change (Δ*H*°) calculated from the experimental data shows endothermic (MLB) and exothermic (MLP) adsorption process of MG dye. The disorder or randomness at solid-liquid interface was determined by calculation of standard entropy change (Δ*S*°).

Conclusion

The low cost adsorbents (MLP and MLB) prepared from mulberry leaves proved to be efficient and feasible for the removal of malachite green (MG) dye from aqueous solution thus themselves from wastewater containing MG dye. Among three well known isotherm models namely Langmuir, Freundlich, and Temkin, Freundlich isotherm model is best fit model with R^2 = 0.99 for both the adsorbents. The maximum adsorption capacity (q_o) of MG dye at the given conditions for MLP and MLB is 4.97 and 6.13 respectively. Kinetic studies reveal that pseudo-second-order kinetic model is best one followed by the present MG-adsorbent systems with R^2 = 0.99 for both the adsorbents (MLP and MLB). From intra-particle diffusion model the intercepts (I) are not zero indicating intra-particle diffusion was not only the rate controlling step but some other mechanisms like boundary layer control are also involved. Thermodynamic parameters indicate spontaneous, endothermic (MLB) and exothermic (MLP) adsorption process of MG dye onto MLP and MLB adsorbents. The salt KCl has positive effect for MG dye percentage removal onto MLB adsorbent up to a particular amount but negative effect on MLP adsorbent. However, $CaCl_2$ salt has negative effect for the present adsorption process. Lastly, towards basic *p*H the percentage removal is better except MLB adsorbent for MG dye removal from its aqueous solution.

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