Adsorption Isotherms, Kinetics and Thermodynamics of Methylene Blue Dye Onto Activated *Pergularia daemia* Carbon

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ABSTRACT

The research of the present work was to investigate the removal of Methylene Blue dye from aqueous solution by using Activated *Pergularia daemia* Carbon (APDC). Generally, dyes are used in chemical, textile, paper, printing, leather, plastics and various food industries. The need for the treatment of dye contaminated waste water passed out from the industry because it causes determent problems to human beings and animals. In this study, *Pergularia daemia* Carbon was studied for its potential use as an adsorbent for the removal of Methylene Blue dye. The various factors affecting adsorption, such as initial dye concentration, contact time, adsorbent dose, effect of temperature and pH were evaluated. The experimental data were fitted into the pseudo-second order kinetic model. The equilibrium of adsorption was modeled by using the Langmuir and Freundlich isotherm models. The objective of the present work suggests the APDC may be utilized as a low cost adsorbent for Methylene Blue dye removal from the aqueous solution.

Key words: *Activated Pergularia daemia Carbon (APDC), Methylene Blue dye (MB), Adsorption isotherm, Kinetics, Equilibrium models.*

Introduction

With the revolution in textile industries discharge if colored effluents such as organic dyes like Methylene Blue, Malachite green and Rhodamine B has increased. These colored effluents have large amounts of suspended organic solids which are harmful to human beings and toxic for organisms. The removal of these dyes from wastewater has a considerable attention over the past decades to decrease their impact on the environment (Al Duri, 1990). Several physical and chemical methods have been developed for the removal of organic dyes from industrial effluents (Arivoli, 2010). Physical methods, mainly adsorption on various supports were recognized to be a promising and effective process to remove dyes from industrial waste waters completely (Hema, 2009). The main advantages of adsorption are the reusability of material, low-cost, ease of operation and short time of operation so it is necessary to develop effective adsorbents for the removal of dyes from textile effluents (Allen, 1989 and Gupta, 1988). Activated carbon is the most widely used adsorbent and can be prepared by physical activation in which carbonation or pyrolysis of the carbonaceous material at higher temperature (500-900°C) in an inert

atmosphere followed by thermal activation at the same temperature in the presence of oxidizing agent such as air, steam and carbon dioxide or chemical activation in which pyrolysis and activation carried out only in one step simultaneously in presence of dehydrating agent such as H_3PO_4, HNO_3, KOH, NaOH, H_2O_2 and H_2SO_4. Chemically activated carbon has higher yield, more surface area and better development of porous structure than that obtained by physical activation (Vijayakumaran, 2009). Several attempts have been made to study the preparation of activated carbon from agriculture wastes.

In this work, we have reported the use of natural bio materials as an alternative due to their relative abundance and their low commercial values. *Pergularia daemiais* the most ancient plant of India. It is found throughout India, especially in Tamil Nadu, Kerala, Bengal and solve south region, the aerial parts of the tree have medicinal values and this work studied the possibility of using stem of a *Pergularia daemia* plant as an adsorbent for the adsorption of cationic dye Methylene Blue from aqueous solution. A survey of literature shows that no work has been done so for a vitalization of *Pergularia daemia* an adsorbent.

Experimental Methods

All reagents used in the experiments were of analytical (AR) grade and were obtained from scientific equipment company Trichy. Stock solutions of the test reagents were prepared by dissolving the dye in distilled water.

Preparation of Adsorbent

The natural plant material for *Pergularia Daemia* used in the present investigations was collected from a nearby Poompuhar area. The leave was washed with distilled water several times to remove the dirt and dust and was subsequently dried in a hot air oven at 110 °C. Afterward, carbonization of the stem was carried out by adding w/v ratio Con. H_2SO_4 to get the primary carbon. The primary carbon was activated at 900 °C for 6 hrs under optimized conditions to obtain the activated carbon.

Experimental Procedure

Batch experiments were conducted to study the influence of important parameters like the pH, contact time, initial dye concentration, other ions and temperature on the removal of MB onto activated *Pergularia Daemia* Carbon. For Adsorption Iso-

therms, dye solution of different concentrations (25-125 mg/l) and at different temperatures (30 - 60 °C) with known pH and known amount of adsorbent (0.025 g/l) were agitated at 120 rpm until the equilibrium was reached then the solution was kept to settle down and the residual concentration of MB were analyzed by UV-Visible spectrophotometer at 663 nm. All experiments were carried out at normal pH for MB. Effect of pH on dye removal was studied over a pH range of 3.0-9.0 the initial pH of the solution was adjusted by addition of dil. HCl or dil. NaOH. The effect of sorbent dosage on adsorption rate was investigated using the procedures described above except that different dosages (25–250 mg/50 ml) were used. The amount of adsorption at time t, q_t(mg/g), can be determined using the following formula.

$$q_t = V \times \frac{(C_0 - C_t)}{w} \qquad .. (2)$$

Where, q(t) is the mass of adsorbed dye per unit mass of adsorbent (mg g^{-1}) (i) and (t) are the initial and actual concentration (g dm^{-3}) of dye at time, respectively V is the volume of the treated solution (ml) m is the mass of adsorbent (g)The adsorption degree. The removal percentage of MB dye can be calculated as following relationship,

$$\% \text{ Removal} = \left(\frac{C_0 - C_t}{C_0}\right) \times 100 \qquad .. (3)$$

Based on the adsorption kinetics experiments, the process time for the equilibrium adsorption experiments was chosen, long enough assuming that the considered sorbent / sorbate system is equilibrated. The following parameters of the process were changed during these experiments kind and amount of adsorbent (APDC) the particle size: The initial pH of the solutions and the initial concentration of the considered ions, all experiments were performed at ambient temperature.

Results and Discussion

Contact time

In order to establish the equilibration time for maximum uptake and to know the kinetics of the adsorption process, MB adsorption on APDC adsorbent was investigated as a function of contact time and the results were shown in Fig. 1. The figure shows that the uptake rate was initially rapid with 50% of the adsorption was complete with in 30 min, Equi-

librium was achieved with in 50 min therefore, an equilibration period of 1 h was selected for all further experiments. The time profile of MB uptake is a single Smooth and continuous curve leading to saturation suggesting the possible monolayer coverage of MB on the surface of the adsorbent (Rajagopal, 2006).

Effect of adsorbent dosage

The adsorption of the MB dye on APDC was studied by varying the adsorbent dose for 50 mg/l of dye concentration. The percentage of adsorption increased with increases in the APDC dose, which is attributed to increased carbon surface area and the availability of more adsorption site. Hence, all studies were carried out with 0.025 g of adsorbent/50 ml of the varying adsorbate solutions. 10, 25, 50, 100, 200 and 250 mg/l. The results obtained from this study are shown in Fig. 2. The amount of MB adsorbed per gram reduced with increase in the dosage of APDC. This reveals that the direct and equilibrium capacities of MB are functions of the activated APDC dosage.
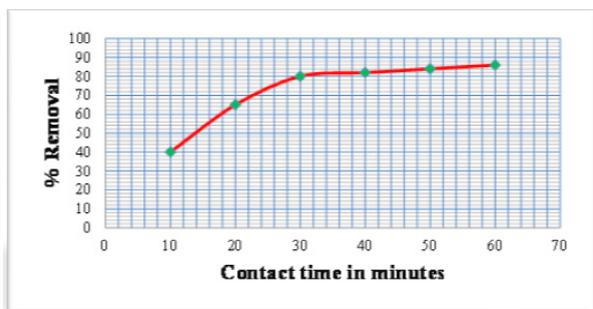

Fig. 1. Effect of Contact Time on the Removal of MB dye [MB]=25 mg/l; Temperature 30 ^{0}C; Adsorbent dose=0.025 g/50 ml.
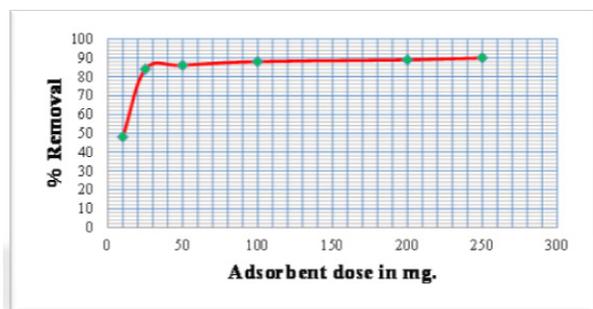

Fig. 2. Effect of Adsorbent dose on the Removal of MB dye [MB]=25mg/l; Temperature 30 ^{0}C; Contact Time 60 min.

Effect of Initial pH

Previous research has shown that the adsorption of dye molecules onto an adsorbent is highly pH dependent since, the functional groups, which are responsible for interaction between dye molecules and adsorbent, can be protonated or deprotonated to produce different surface charges in solution at different pH values. Therefore the effects of initial solution pH were studied in the pH range of 3-9 for MB. The percentage removal increased around 60 % to 84 % for MB whereas it decreased slowly after pH 6.8 for MB (Fig. 3). The pHzpc of any adsorbent is a very important characteristic that determines the pH at which the surface has net electrical neutrality. It is well-known that for basic dye adsorption, negatively charged groups on the adsorbent are necessary. At lower pH values (pH <pHzpc) the surface charge of the surface of APDC may get positively charged as a result of being surrounded by H_3O^+ ions and thus the competitive effects of H_3O^+ ions as well as the electrostatic repulsion between the dye molecules and the positively charged active adsorption sites on the surface of the APDC lead to a decrease in the uptake of dye molecules. In contrast at higher pH values (pH >pHzpc) the surface of APDC may acquire a negative charge leading to an increase in dye uptake due to the electrostatic force of attraction (Rajagopal, 2006). On the other hand no valid reason can be given for the decrease in the adsorption amount of MB after pH 6.8. Similar results were obtained for the adsorption of Methylene Blue onto APDC. As a result, the initial pH value was optimized as 6.8 for dye.

Effect of other ions

The effect of chlorine ions Cl⁻ on the adsorption process studied at different concentrations. The ions
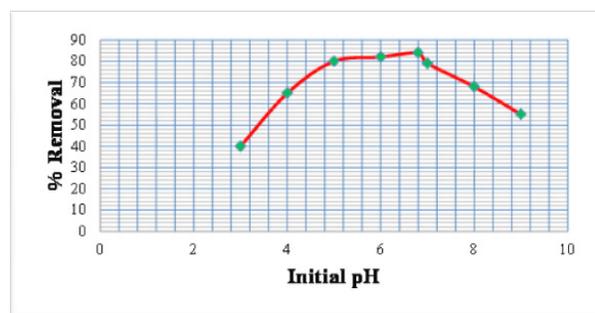

Fig. 3. Effect of Initial pH on the Removal of MB dye [MB]=25 mg/l; Temperature 30 ^{0}C; Adsorbent dose=0.025 g/50 ml.

added to 50 mg/l of MB solutions and the contents were agitated for 60 min at 30 ºC. The results had shown in the Fig. 4 reveals that low concentration of Cl - does not affect the percentage of adsorption of MB on APDC, because the interaction of Cl⁻ at available sites of adsorbent through competitive adsorption is not so effective. While the concentration of other ion increases, the interference of these ions at available surface sites of the sorbent through competitive adsorption increases that, decreases the percentage adsorption. This is so because ions with smaller hydrated radii decrease the swelling pressure within the sorbent and increase the affinity of the sorbent for such ions (Maurya, 2006 and El-Geundi, 1991).

Adsorption Models

The adsorption equilibrium data were further analyzed into two well-known isotherm models via Freundlich and Langmuir models (Vasantha Kumar, 2006 and Aravindhan, 2007).
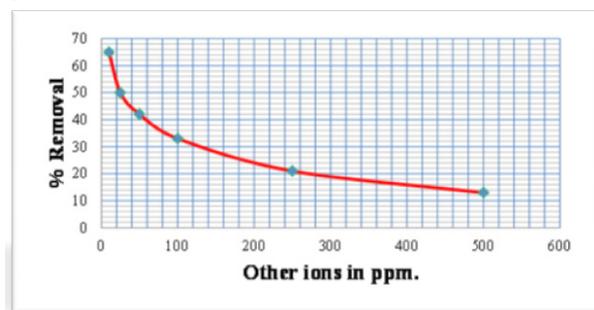
Fig. 4. Effect of other ionic strength on the removal of MB dye
[MB]=25 mg/l; Contact time=60 min.; Adsorbent dose=0.025g/50 ml.

Langmuir model

The adsorption isotherm was also fitted to Langmuir model (Langmuir, 1918). The Langmuir equation which is valid for monolayer adsorption on to a surface is given below,

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \quad .. (4)$$

Where, qe (mgg⁻¹) is the amount adsorbed at the equilibrium concentration Ce (mol L⁻¹), qm (mgg⁻¹) is the Langmuir constant representing the maximum monolayer adsorption capacity and b (L mol⁻¹) is the Langmuir constant related to energy of adsorption. The plots 1/Qe as a function of 1/Ce for the adsorption of MB was found linear. Suggesting the applicability if Langmuir model in the present adsorption system. The correction coefficient (R^2 = 0.99) confirm good agreement between both theoretical models and our experimental results the values of the monolayer capacity (Qm) and equilibrium constant (b) have been evaluated from the intercept and slope of these plots and given in Table 2 (Fig. 5). These facts suggest that MB is adsorbed in the form of monolayer coverage on the surface of the prepared adsorbent. Satisfactory fitting of the Langmuir model to the adsorption of MB on APDC. The Langmuir constant Qm and b were determined from the slope and intercept of the plot and are presented in Table 2. In order to find out the feasibility of the isotherm, the essential characteristics of the

Table 1. Equilibrium Parameters for the Adsorption of MB onto APDC

M₀	Ce (Mg/l)				Qe (Mg /l)				Removal %			
	30ºC	40ºC	50ºC	60ºC	30ºC	40ºC	50ºC	60ºC	30ºC	40ºC	50ºC	60ºC
25	4.74375	4.459125	3.8064	3.7948	40.513	41.082	42.387	42.410	81.025	82.164	84.774	84.821
50	12.33375	11.3894	10.4761	8.5410	75.333	77.221	79.048	82.918	75.333	77.221	79.048	82.918
75	22.8482	20.6868	18.0264	16.7020	104.304	108.626	113.947	116.596	69.536	72.418	75.965	77.731
100	33.2859	30.4403	28.8744	27.0845	133.428	139.119	142.251	145.831	66.714	69.560	71.126	72.915
125	49.335	47.623	46.573	43.807	151.330	154.753	156.855	162.386	60.532	61.901	62.742	64.954

Table 2. Langmuir and Freundlich Isotherm Parameter for the Adsorption of MB Onto APDC

Temp. (ºC)	Langmuir Parameters			Freundlich Parameters		
	Qm	B	R^2	Kf	N	R^2
30ºC	220.10	0.0438	0.9903	17.169	1.7449	0.9929
40ºC	223.82	0.0485	0.9930	18.207	1.7323	0.9848
50ºC	213.27	0.0625	0.9940	21.666	1.8408	0.9772
60ºC	219.89	0.0675	0.9968	22.801	1.8164	0.9597

Langmuir isotherm can be expressed in terms of dimensionless constant separation factor R_L by the equation.

$$R_L = \frac{1}{1 + K_L C_0}$$ .. (5)

Where, Co (mg/l) is the highest initial concentration of adsorbent and b (L/mg) is Langmuir isotherm constant. The parameter RL indicates the nature of shape of the isotherm accordingly.

$R_L > 1$	Unfavorable adsorption
$0 < R_L < 1$	Favorable adsorption
$R_L = 0$	Irreversible adsorption
$R_L = 1$	Linear adsorption

The R_L values between 0 and 1 indicate favorable adsorption for all initial concentration (Co) and temperatures studied. The calculated R_L values are given in Table 3. The values of b were increased with increasing the dose of adsorbent for APDC High b values indicate high adsorption affinity the monolayer saturation capacity Qm were around 219.89 mg/l for APDC.

Table 3. Dimensionless Seperation Factor (R_l) for the Adsorption of MB onto APDC

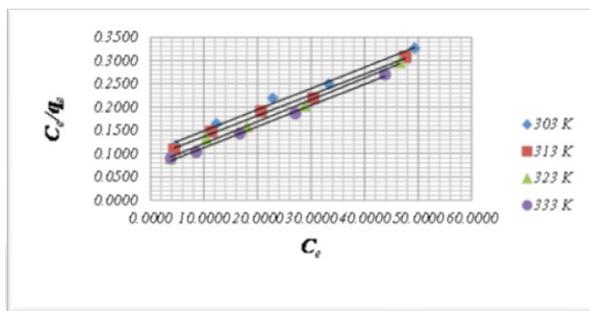| (C$_i$)	Temperature °C			
	30°C	40°C	50°C	60°C
25	0.4773	0.4522	0.3903	0.3720
50	0.3135	0.2922	0.2425	0.2285
75	0.2333	0.2158	0.1758	0.1649
100	0.1859	0.1711	0.1379	0.1290
125	0.1544	0.1417	0.1135	0.1059

Freundlich model

The Freundlich model (Frendlich, 1906) which is an indicative of surface heterogeneity of the adsorbent is described by the following equation.

$$log q_e = log K_f + \frac{1}{n_f} log C_e$$ .. (3)

Where, K_f and $1/n$ are Freundlich constants associated with adsorption capacity and adsorption in-

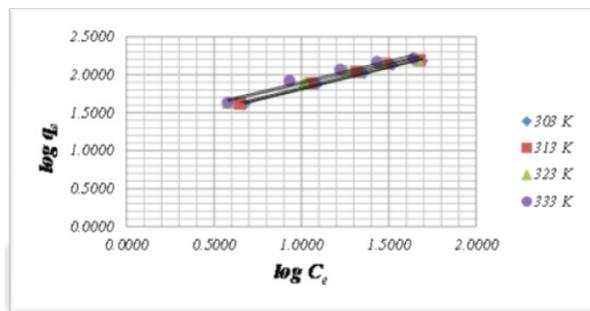**Fig. 5.** Langmuir adsorption isotherm for the removal of MB dye

tensity respectively, The Freundlich plots between log qe and logC$_e$ for the adsorption of MB were drawn. It was found that correlation efficient values were close to 0.99 at both the temperature studied indicating that Freundlich model was applicable to the present study (Table 2 and Fig 6).

Fig. 6. Freundlich adsorption isotherm for the removal of MB dye

Thermodynamic treatment of the adsorption process

Thermodynamic parameters (Gupta, 2010 and Fornwalt, 1966) associated with the adsorption, via standard free energy change (ΔG°), standard enthalpy change (ΔH°), and standard entropy change (ΔS°) were calculated as follows. The free energy of adsorption process considering the adsorption equilibrium constant K$_o$ is given by the equation:

Table 4. Thermodynamic Parameter for the Adsorption of MB onto APDC

(C$_0$)	ΔG°				ΔH°	ΔS°
	30 °C	40 °C	50 °C	60 °C		
25	-3656.9	-3974.9	-4610.9	-4763.6	8.391	39.756
50	-2812.4	-3177.0	-3565.7	-4373.8	12.4746	50.178
75	-2079.0	-2511.9	-3090.2	-3460.8	12.25878	47.309
100	-1751.5	-2150.6	-2420.9	-2741.8	8.07310	32.514
125	-1077.4	-1263.0	-1399.5	-1708.3	5.04287	20.141

$$\Delta G^0 = - RT\ln K_0 \qquad\qquad .. (6)$$

Where, ΔG is the free energy of adsorption (kJ/mol), T is the temperature in Kelvin and R is the universal gas constant (8.314 J mol/K). The adsorption distribution coefficient K_o for the sorption reaction was determined from the slope of the plot of ln(Qe/Ce) against Ce at different temperature and extrapolating to zero Ce according to the method. The adsorption distribution coefficient may be expressed in terms of enthalpy change (ΔH) and entropy change (ΔS) as a function of temperature,

$$\ln K_o = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \qquad\qquad .. (7)$$

Where, $\Delta H°$ is the standard heat change of sorption (kJ/mol) and $\Delta S°$ is standard entropy change (kJ/mol). The value of $\Delta H°$ and $\Delta S°$ can be obtained from the slope and intercept of plot of $\ln K_o$ against 1/T. The value of thermodynamic parameter calculated from equation 6 and 7 are shown in Table 4. The thermodynamic treatment of the sorption data indicates that $\Delta G°$ values were negative at all temperature. The results point out that physisorption is much more favorable for the adsorption of MB dye. The positive values of $\Delta H°$ show the endothermic nature of adsorption and it governs the possibility of physical adsorption. Because in the case of physical adsorption, while increasing the temperature of the system, the extent of MB adsorption increases, this rules out the possibility ofchemisorptions. The low $\Delta H°$ value depicts MB dye is physisorbed onto adsorbent APDC.

The negative $\Delta G°$ values (Table 4) was confirm the spontaneous nature of adsorption MB dye onto APDC. The lesser values of $\Delta G°$ suggest that adsorption is physical adsorption process. The positive value of $\Delta H°$ further confirms the endothermic nature of adsorption process. The positive values of $\Delta S°$ in Table 4, showed increased randomness of the solid solution interface during the adsorption of MB onto APDC.

Kinetic modelling in a batch system

Pseudo-second-order

In order to investigate the mechanism of adsorption kinetic models are generally used to test experimental data. Pseudo-second-order equations can be used assuming that the measured concentrations are equal to surface concentrations (Weber, 1964 and McKay, 1982). The pseudo-second-order model is:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \left(\frac{1}{q_e}\right) t \qquad\qquad .. (9)$$

Where, qt (mgg 1) is the amount of adsorbed MB dye on the adsorbent at time t, qe the equilibrium sorption uptake and k_2, (min^{-1}) is the rate constant of pseudo-second-order adsorption. The plot t/qt versus t gives a straight line says second order kinetic model is applicable then qe and k_2 are determined from the slope and intercept of the plot, respectively. The high regression value indicate the adsorption reaction exist a pseudo-second-order and these values shown Table 5.

The Elovich equation

The Elovich model (Chien, 1980) equation is generally expressed as

$$q_t = \frac{1}{\beta}\ln(\alpha\beta) + \frac{1}{\beta}\ln t \qquad\qquad .. (8)$$

Where α is the initial adsorption rate (mg g^{-1} min-1) and β is desorption constant (g/mg) during any one experiment. If MB dye adsorption fits with the Elovich model, a plot of qtVsln(t) yields a linear relationship with a slope of (1/β) and an intercept of (1/β)ln($\alpha\beta$). The Elovich model parameters α, β, and correlation coefficient (R2) are summarized in Table 5. This model indicates that the initial adsorption (α) increases with temperature similar to that of initial adsorption rate (h) in pseudo-second–order kinetics models. This may be due to increase the pore or active site on the APDC adsorbent.

Intra-particle diffusion model

Kinetic data was further analyzed using the intra-particle diffusion model (Weber, 1964) based on the following equation.

$$log\ R = log\ K_{id} + \alpha\ log\ t \qquad\qquad .. (9)$$

Where, kid is the intra-particle diffusion rate constant and it is related to the thickness of the boundary layer. According to above equation a plot of log R versus log t gives a straight line that's says the adsorption mechanism follows the intra-particle diffusion process and the evidence of correlation coefficient values (Table 5) are close to unity.

Conclusion

The composite adsorbent exhibited effectiveness in the removal of MB dye from aqueous solution. The

Table 5. The Kinetic Parameters for Adsorption of MB Onto APDC

C_0	Temp °C	Pseudo second order				Elovich model			Intraparticle diffusion		
		q_e	k_2	R^2	H	α	β	R^2	α	K_{id}	R^2
25	30	57.4624	6.16E-04	0.9648	2.0330	4.1E+00	7.57E-02	0.9592	0.4473	12.5901	0.9884
	40	55.2744	7.67E-04	0.9711	2.3429	5.0E+00	8.05E-02	0.9592	0.4026	15.3425	0.9864
	50	52.1480	1.19E-03	0.9820	3.2432	8.5E+00	9.43E-02	0.9592	0.3137	22.8451	0.9817
	60	52.1089	1.20E-03	0.9821	3.2622	8.6E+00	9.46E-02	0.9592	0.3123	22.9933	0.9816
50	30	93.3073	5.99E-04	0.9592	5.2161	1.4E+01	5.28E-02	0.9058	0.3196	19.3483	0.9429
	40	93.0915	7.09E-04	0.9743	6.1455	1.8E+01	5.53E-02	0.9288	0.2910	22.5501	0.9586
	50	95.4107	8.19E-04	0.9888	7.4555	2.3E+01	5.46E-02	0.9510	0.2775	25.4451	0.9626
	60	93.0504	1.05E-03	0.9763	9.1280	5.1E+01	6.72E-02	0.8810	0.2128	33.1890	0.9112
75	30	118.6468	7.57E-04	0.9731	10.6529	5.9E+01	5.26E-02	0.8541	0.2155	27.3716	0.8840
	40	125.1780	8.37E-04	0.9920	13.1100	6.5E+01	4.84E-02	0.9402	0.2196	29.4356	0.9502
	50	133.8237	6.87E-04	0.9937	12.2977	4.5E+01	4.15E-02	0.9677	0.2491	27.4530	0.9765
	60	130.5434	1.08E-03	0.9999	18.3947	1.1E+02	4.89E-02	0.9889	0.2004	34.8154	0.9819
100	30	154.2824	7.39E-04	0.9987	17.5824	6.2E+01	3.59E-02	0.9734	0.2439	25.3452	0.9657
	40	159.9668	7.00E-04	1.0000	17.9077	6.7E+01	3.52E-02	0.9905	0.2398	26.5788	0.9815
	50	161.8962	7.65E-04	0.9999	20.0540	8.5E+01	3.61E-02	0.9820	0.2265	28.7863	0.9717
	60	165.3502	7.75E-04	0.9998	21.1842	9.7E+01	3.61E-02	0.9835	0.2196	30.3341	0.9746
125	30	176.9456	4.00E-04	0.9553	12.5272	4.3E+01	3.06E-02	0.8589	0.2666	19.1346	0.8999
	40	180.9300	3.96E-04	0.9581	12.9483	4.5E+01	3.02E-02	0.8635	0.2639	19.8167	0.9036
	50	180.3165	4.42E-04	0.9632	14.3668	5.9E+01	3.19E-02	0.8563	0.2420	22.0153	0.8940
	60	187.5705	4.01E-04	0.9549	14.1066	5.3E+01	2.99E-02	0.8425	0.2526	21.7014	0.8833

removal efficiency was controlled by solution pH, adsorbent concentration, contact time and initial ion concentration. Adsorption data fitted well with the Langmuir and Freundlich models However, Langmuir isotherm displayed a better fitting model than Freundlich isotherm because of the higher correlation coefficient that the former exhibited, thus, indicating to the applicability of monolayer coverage of the dye on the surface of adsorbent. Using *Pergularia daemia* to produce activated carbons potentially provide a less expensive raw material and a highly effective adsorbent.

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