

Adsorption of Melachite Green onto Activated Carbon Prepared from *Clerodendrum serratum*

¹Anitha M., ^{1*}Arivoli S., ²Hema M. and ³Ajithkumar M.

¹PG and Research Department of Chemistry, Rajah Serfoji Government College(AU), (Affiliated to Bharathidasan University), Thanjavur-613005, Tamilnadu, India.

^{1*}PG and Research Department of Chemistry, Poompuhar College(AU), (Affiliated to Bharathidasan University), Melaiyur-609107, Tamilnadu, India.

²Department of Chemistry, University College of Engineering, Anna University: Chennai, Thirukkuvalai Campus, Thirukkuvalai-610204, Tamilnadu, India.

³Department of Chemistry, PERI College of Arts and Science (Affiliated to Madras University), Mannivakkam-600048, Tamilnadu, India.

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ABSTRACT

The *Clerodendrum serratum* leaves obtained from nearby Nagai district, it was used as an adsorbent for the removal of malachite green dye from aqueous solution. The various factors affecting adsorption, such as initial dye concentration, contact time, adsorbent dose and initial pH of solution were evaluated on adsorption of malachite green dye on activated *Clerodendrum serratum* carbon. The equilibrium adsorption isotherms were analyzed by Langmuir and Freundlich equations. Both Langmuir and Freundlich models can describe the adsorption equilibrium but the Langmuir model shows better agreement. The kinetics of the adsorption process was tested by pseudo-first-order, pseudo-second order, Elovich and Intra-particle diffusion models. It was shown that adsorption of malachite green dye could be described by the pseudo-second order kinetic model. Thermodynamic parameters such as Gibbs free energy (ΔG°), the enthalpy (ΔH°) and the entropy change of sorption (ΔS°) have also been evaluated and it has been found that the adsorption process was spontaneous, feasible and endothermic in nature. The results indicated that activated *Clerodendrum serratum* carbon can be used as an effective and low-cost adsorbent to remove malachite green dye from aqueous solution.

Key words: Activated *Clerodendrum serratum* Carbon (ACSC); Malachite green dye (MG); Adsorption isotherm; Kinetics; Thermodynamics.

Introduction

With the revolution in the textile industry, organic dyes such as methylene blue, malachite green, rhodamine B, etc., were released into the water. These color discharges contain large amounts of suspended organic matter that are dangerous to humans and toxic to organisms. Removal of these dyes

from aqueous solution has had a significant response over the past decades to reduce their impact on the environment (Naimabadi *et al.*, 2009; Itodo and Itodo, 2010; Allen *et al.*, 1989). Many physical and chemical methods have been technologically advanced to remove organic dyes from aqueous solutions. Physical methods, primarily on multiple supports, have been recognized as a promising and

effective process for the complete removal of dyes from aqueous solutions (Alpert *et al.*, 1970; Arivoli S and Hema, 2007). The most important advantages of absorption are material recycling, low cost, simplicity of operation and short duration of operation, so it is necessary to create adsorbents that have the effect of removing dyes from the aqueous solution (Arivoli *et al.*, 2007).

Many investigators have explored the possibilities of using cheaper alternative materials such as pearl millet husk, date pits, saw dust, leather industry dust, coir pit, crude oil residue, tropical grass, olive stone, almond shells, pine bark, wool waste, coconut shell, etc., as carbonaceous precursors for the removal of dyes from water and waste water (Chien and Clayton, 1980; Bulut *et al.*, 2008).

In this work, we inform you that the use of natural biomaterials is an alternative as they are relatively abundant and their low commercial value makes *Clerodendrum serratum* the oldest plant in India. It is found throughout India, especially in Tamil Nadu, Kerala, Bengal and the entire of south region, this work explored the possibility of using the leaves of a *chlorodentrum serratum* carbon as an additive to adsorb the malachite green dye from an aqueous solution. A study of the literature shows that no work has been done to revive the *chlorodentrum serratum* leaves as an adsorbent.

Experimental Methods

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

Preparation of adsorbent

The natural plant material of *C. serratum* used in the present investigations was collected from a nearby Poompuhar, Nagai District, Tamilnadu, India. The *C. serratum* leaf 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. Next, carbonization of the leaf was carried out by adding w/v ratio Con. H₂SO₄ to get the primary carbon. The primary carbon was activated using Muffle furnace at >900 °C for 6 hrs under optimized conditions to obtain the activated carbon.

Experimental Procedure of Batch Method

Batch tests Errais *et al.* (2011) were conducted to

study the influence of important parameters such as pH, contact time, initial dye concentration, and other ions taking place MG dye removal on activated *C. serratum* carbon. In place of adsorption isotherms, dye solutions of different concentrations (25–125 mg / L) and known pH at different temperatures (30 - 60 °C) and known levels of adsorbent (0.025 g / L) are equilibrated at 120 rpm The solution was then stabilized and the residual concentration of MG was analyzed at 470 nm with a UV-Visible spectrophotometer. All tests were performed at normal pH to remove MG dye from the aqueous solution. The effect of pH on dye removal was studied in the pH range of 3.0-9.0, and the pH was fixed by adding dil. HCl or NaOH to the test solution. Except that different doses (25–125 mg / 50 ml) were used, the effect of the sorbent dose on the absorption rate was explored using the procedures described above. The amount of absorption at T, q_t (mg / g) can be determined using the following formula.

$$q_t = V \times \frac{(C_0 - C_t)}{w} \quad \dots (1)$$

Where, q_t is the mass of adsorbed dye per unit mass of adsorbent (mg g⁻¹) C₀ and C_t are the initial and actual concentration (g dm⁻³) of dye at time, respectively V is the volume of the treated solution (ml), 'w' is the mass of adsorbent (g). The removal percentage of MG dye can be calculated as following relationship.

$$\% \text{ Removal} = \left(\frac{C_0 - C_t}{C_0} \right) \times 100 \quad \dots (2)$$

Result and Discussion

Contact time

To establish equilibrium time Qada *et al.* (2008) for maximum improvement and to understand the dynamics of the adsorption process, MG adsorption in ACSC adsorbent was explored as a function of contact time and the results are shown in Figure 1 and the equilibrium data are presented in Table 1. The figure shows that the adsorption was initially rapid and the maximum adsorption was completed in 30 min. The equilibrium was reached in 50 min. therefore, an equilibrium period was 60 min. selected for all experiment.

Effect of adsorbent dosage

The adsorption of the MG dye on ACSC was stud-

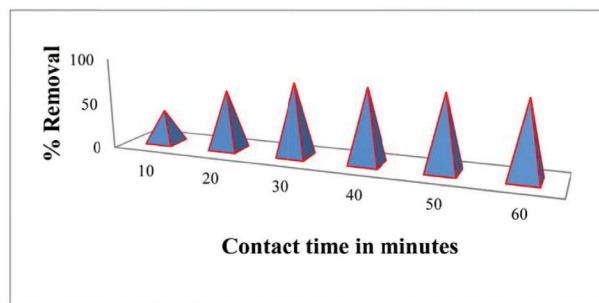


Fig. 1. Effect of Contact Time on the Removal of MG dye [MG]=25mg/L; Temperature 30°C; Adsorbent dose=0.025g/50mL.

ied by varying the adsorbent dose (Pandhare; Kumar, 2006) for 50 mg/L of dye concentration. The percentage of adsorption increased with increases in the ACSC dose, which is attributed to increased carbon surface area and the availability of more adsorption sites. Hence, all studies were carried out with 50 ml/25 mg/L solution and varying adsorbent amount such as 10, 25, 50, 100, 200, 250 mg. The results obtained from this study are shown in Figure 2. The amount of MG adsorbed per gram reduced with increase in the dosage of ACSC. This reveals that the direct and equilibrium capacities of MG are functions of the activated ACSC dosage.

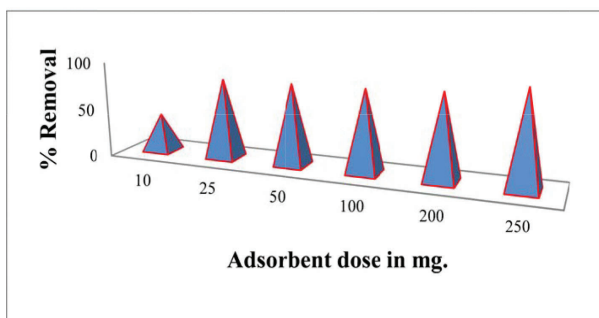


Fig. 2. Effect of Adsorbent dose on the Removal of MG dye [MG]=25mg/L; Temperature 30°C; Contact Time 60 min.

Effect of Initial pH

Adsorption of dyes highly pH dependent, therefore the effects of initial solution pH were studied in the pH range of 3-9 under the precise conditions (at optimum contact time of 60 min, 120 rpm shaking speed, with 25mg of the adsorbents used, and at a room temperature of 30 °C) for removal of MG dye from aqueous solution. The percentage removal maximum at pH 6.9 that is pH_{zpc} (Zero point charge) shown in Figure 3. The pH_{zpc} of adsorbent is a very important characteristic that determines the pH at which the surface has net electrical neutrality (Langmuir, 1918; Hema and Arivoli, 2009; Hossain and Alam, 2012).

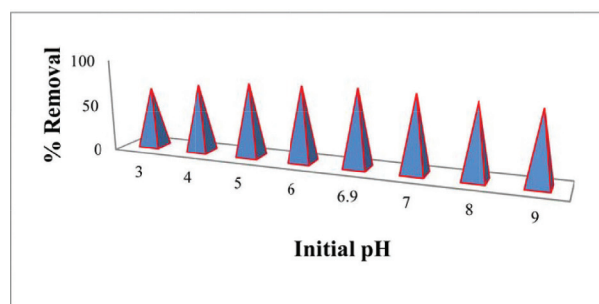


Fig. 3. Effect of Initial pH on the Removal of MG dye [MG]=25mg/L; Temperature 30°C; Adsorbent dose=0.025g/50mL.

Effect of other ions

The effect of other ions Maurya *et al.* (2006) on the adsorption process studied at different concentrations of chlorine ions. The ions added to 50 mg/L of MG solutions and the contents were agitated for 60 min at 30 °C. The results had shown in the figure 4 reveals that low concentration of chlorine ions does not affect the percentage of adsorption of MG on ACSC, because the interaction of chlorine ions at available sites of adsorbent through competitive adsorption is not so effective. While the concentration of other ion increases, the interference of these

Table 1. Equilibrium Parameters for the Adsorption of MG dye Onto ACSC

C_0	C_e (Mg / L)				q_e (Mg / L)				Removal %			
	30°C	40°C	50°C	60°C	30°C	40°C	50°C	60°C	30°C	40°C	50°C	60°C
25	4.94	4.64	3.96	3.95	40.13	40.72	42.08	42.10	80.25	81.44	84.15	84.20
50	12.84	11.85	10.90	8.89	74.33	76.29	78.19	82.22	74.33	76.29	78.19	82.22
75	23.78	21.53	18.76	17.38	102.44	106.94	112.47	115.23	68.29	71.29	74.98	76.82
100	34.65	31.68	30.05	28.19	130.71	136.63	139.89	143.62	65.35	68.32	69.95	71.81
125	51.35	49.57	48.47	45.60	147.30	150.86	153.05	158.81	58.92	60.35	61.22	63.52

ions at available surface sites of the sorbent through competitive adsorption increases that, decreases the percentage adsorption.

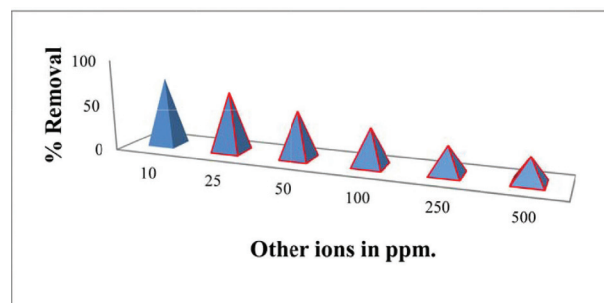


Fig. 4. Effect of other ionic strength on the removal of MG dye
[MG]=25mg/L; Contact time=60 min.; Adsorbent dose=0.025g/50mL.

Adsorption Models

The adsorption equilibrium data were further analyzed into two well-known isotherm models via Freundlich and Langmuir models.

Freundlich model

The Freundlich model (Arivoli *et al.*, 2010; Ibrahim *et al.* 2006; Mazloumi *et al.*, 2009) revealing the surface

heterogeneity of the adsorbent is described by the following equation.

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

Where, K_f and $1/n_f$ are Freundlich constants related with adsorption capacity and adsorption intensity respectively. The Freundlich plots drawn between $\log q_e$ and $\log C_e$ this one shown in Figure 5 and these values listed in Table 2.

Langmuir model

The Langmuir adsorption isotherm equation which is valid for monolayer adsorption on to a surface is given below.

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

Where, q_e (mgg^{-1}) is the amount of dyes adsorbed at the equilibrium concentration C_e (mol L^{-1}), q_m (mgg^{-1}) is the Langmuir constant representing the maximum monolayer adsorption capacity and K_L (L mol^{-1}) is the Langmuir constant related to energy of adsorption. The plot drawn between C_e/q_e and C_e as well as it's shown in Figure 6. The correction coefficient (R^2) values confirm good agreement our experimental results the values of the monolayer ca-

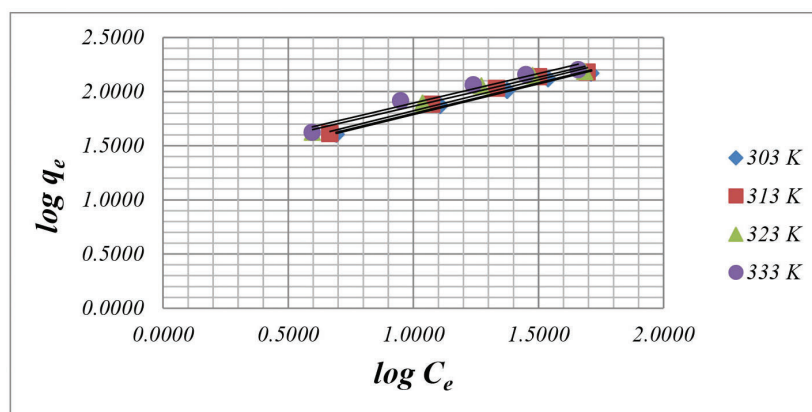


Fig. 5. Freundlich adsorption isotherm for the removal of MG dye

Table 2. Langmuir and freundlich Isotherm Parameter for the Adsorption of MG Onto ACSC

Model	Constant	Temperature ($^{\circ}\text{C}$)			
		30	40	50	60
Freundlich	K_f (mg/g) (L/mg) $^{1/n}$	16.83	17.84	21.27	22.37
	n_f	1.77	1.75	1.86	1.84
	R^2	0.99	0.98	0.98	0.96
Langmuir	q_m (mg/g)	212.13	216.20	206.46	213.51
	K_L (L/mg)	0.04	0.05	0.06	0.07
	R^2	0.99	0.99	0.99	1.00

capacity (q_m) and equilibrium constant K_L have been evaluated from the intercept and slope of these plots and given in Table 2. These facts suggest that MG is adsorbed in the form of monolayer coverage on the surface of the prepared adsorbent. High K_L values indicate high adsorption affinity the monolayer saturation capacity q_m were around 216 mg/L for ACSC. In order to find out the feasibility of the isotherm, the essential characteristics of the Langmuir isotherm can be expressed in terms of dimensionless constant separation factor R_L by the equation.

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

Where, C_0 (mg/L) is the highest initial concentration of adsorbent and K_L (L/mg) is Langmuir isotherm constant. The parameter R_L 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 (C_0) and temperatures studied, these values are scheduled in Table 3.

Table 3. Dimensionless Separation Factor (R_L) for the Adsorption of MG onto ACSC

(C_0)	Temperature °C			
	30°C	40°C	50°C	60°C
25	0.48	0.45	0.39	0.37
50	0.31	0.29	0.24	0.23
75	0.23	0.22	0.18	0.17
100	0.19	0.17	0.14	0.13
125	0.15	0.14	0.11	0.11

Thermodynamic treatment of the adsorption process

Thermodynamic (Mazloumi *et al.* 2009; Singh *et al.* 1994; Sciences and Emerging Technologies, 2013)

Table 4. Thermodynamic parameter for the Adsorption of MG dye Onto ACSC

(C_0)	ΔG°				ΔH°	ΔS°
	30°C	40°C	50°C	60°C		
25	-3531.82	-3847.54	-4483.65	-4632.48	8.46	39.58
50	-2677.67	-3041.28	-3429.00	-4239.61	12.61	50.18
75	-1932.69	-2366.94	-2947.81	-3317.39	12.44	47.42
100	-1598.80	-1999.47	-2268.48	-2588.66	8.22	32.49
125	-908.55	-1092.61	-1226.09	-1535.78	5.17	20.00

parameters associated with the adsorption, the free energy of adsorption process considering the adsorption equilibrium constant K_0 is given by the equation:

$$\Delta G^\circ = -RT \ln K_0 \quad .. (6)$$

Where, ΔG° is the free energy of adsorption (kJ/mol), T stands temperature (K), R stands gas constant (8.314 J mol/K) and K_0 stands equilibrium constant (C_{solid} / C_{liquid}). The equilibrium constant ($\ln K_0$) may be expressed by following mathematical forms.

$$\ln K_0 = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \quad .. (7)$$

Where, ΔH° is the standard heat change of sorption (kJ/mol) and ΔS° is standard entropy change (kJ/mol). The value of ΔH° and ΔS° can be obtained from the slope and intercept of plot of $\ln K_0$ against $1/T$ and These values are listed in Table 4. The thermodynamic treatment of the sorption data indicates that ΔG° values were negative at all temperature. The positive values of ΔH° show the endothermic nature of adsorption and the low ΔH° value depicts MG dye is physisorbed onto adsorbent ACSC.

Kinetic models

Pseudo-second-order

In order to investigate the mechanism of adsorption kinetic models Vijayakumaran *et al.* (2009) are generally used to test experimental data. Pseudo-second-order equations can be used assuming that the measured concentrations are equal to surface concentrations. The linearized form of pseudo second order equation becomes.

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

Where, q_t (mgg⁻¹) is the amount of adsorbed dyes on the adsorbent at time t, q_e is the amount of adsorbed dyes on the adsorbent at equilibrium and k_2 is the rate constant of pseudo-second-order ad-

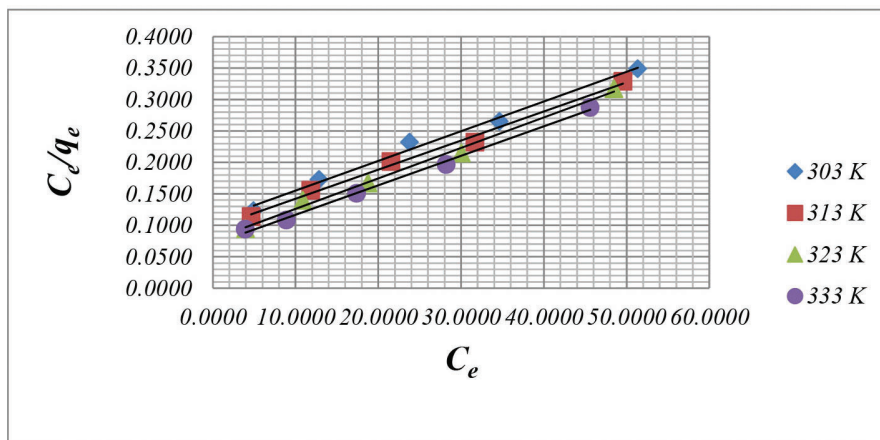


Fig. 6. Langmuir adsorption isotherm for the removal of MG dye

sorption (min⁻¹). The plot t/q_t versus t give a straight line and (R^2) values states second order kinetic model is applicable then q_e 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 equation is generally expressed as

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

Where α is the initial adsorption rate (mg g⁻¹ min⁻¹) and β is desorption constant (g/mg) during any one experiment. If MG adsorption fits with the Elovich model, a plot of q_t Vs $\ln(t)$ yields a linear relationship with a slope of $(1/\beta)$ and an intercept of $(1/\beta)\ln(\alpha\beta)$. The Elovich model parameters α , β , and correlation coefficient (R^2) 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

Table 5. The Kinetic Parameters for Adsorption of MG dye Onto ACSC

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	59.41	5.23E-04	0.96	1.84	3.72	7.27E-02	0.96	4.80E-01	10.89	0.99
	40	56.59	6.68E-04	0.97	2.14	4.41	7.74E-02	0.96	4.31E-01	13.55	0.99
	50	52.72	1.08E-03	0.98	3.00	7.31	9.06E-02	0.96	3.33E-01	20.91	0.98
	60	52.67	1.09E-03	0.98	3.02	7.39	9.09E-02	0.96	3.32E-01	21.05	0.98
50	30	94.14	5.35E-04	0.95	4.74	11.54	5.07E-02	0.91	3.43E-01	17.32	0.95
	40	93.61	6.42E-04	0.97	5.63	15.05	5.31E-02	0.93	3.11E-01	20.50	0.96
	50	95.88	7.47E-04	0.99	6.87	18.95	5.25E-02	0.95	2.95E-01	23.42	0.96
	60	93.08	9.79E-04	0.97	8.48	40.58	6.45E-02	0.88	2.26E-01	31.17	0.91
75	30	117.93	6.96E-04	0.97	9.67	44.75	5.05E-02	0.85	2.31E-01	25.15	0.89
	40	124.70	7.72E-04	0.99	12.00	50.60	4.65E-02	0.94	2.34E-01	27.28	0.95
	50	133.88	6.31E-04	0.99	11.30	36.64	3.99E-02	0.97	2.65E-01	25.36	0.98
	60	130.10	1.01E-03	1.00	17.02	85.47	4.70E-02	0.99	2.13E-01	32.75	0.98
100	30	153.20	6.77E-04	1.00	15.89	48.99	3.45E-02	0.97	2.62E-01	23.11	0.97
	40	159.10	6.43E-04	1.00	16.28	53.21	3.38E-02	0.99	2.57E-01	24.37	0.98
	50	160.98	7.06E-04	1.00	18.31	66.66	3.47E-02	0.98	2.42E-01	26.60	0.97
	60	164.53	7.17E-04	1.00	19.42	76.09	3.47E-02	0.98	2.34E-01	28.18	0.97
125	30	175.54	3.59E-04	0.95	11.07	32.82	2.94E-02	0.86	2.90E-01	16.84	0.90
	40	179.66	3.56E-04	0.95	11.49	34.75	2.90E-02	0.86	2.87E-01	17.54	0.91
	50	178.68	4.01E-04	0.96	12.81	45.04	3.07E-02	0.86	2.62E-01	19.71	0.90
	60	186.36	3.63E-04	0.95	12.62	41.24	2.87E-02	0.84	2.73E-01	19.44	0.89

models. This may be due to increase the pore or active site on the ACSC adsorbent.

Intra-particle diffusion model

Kinetic data was further analyzed using the Intraparticle diffusion model based on the following equation.

$$\log R = \log K_{id} + \alpha \log t \quad \dots (9)$$

Where, k_{id} is the Intraparticle diffusion rate constant and its 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 co-efficient values are close to unity.

Conclusion

This study reveals that adsorbent exhibited effectiveness in the removal of MG dye from aqueous solution. The 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 *Clerodendrum serratum* to produce activated carbons potentially provide a less expensive raw material and a highly effective adsorbent.

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