

## EQUILIBRIUM, THERMODYNAMICS AND KINETIC STUDIES OF MALACHITE GREEN BY ACTIVATED CALCITE POWDER

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### ABSTRACT

The present work deals with adsorption of Malachite Green (MG) dyes carried out in the presence of activated calcite powder. Various parameters like the effect of initial concentration, contact time, dose of adsorbent, temperature and pH were also studied. The result shows that when the amount of adsorbent increases, the percentage removal of dye increases. The applicability of Freundlich adsorption and Langmuir adsorption isotherm had also been tested. Adsorption kinetic data have been tested using pseudo second order, intra-particles models and the Elovich model. The thermodynamics parameter such as  $\Delta G^0$ ,  $\Delta H^0$  and  $\Delta S^0$  were calculated. The adsorption capacities of Activated Calcite Powder (ACP) were calculated using batch process.

### INTRODUCTION

Dyes are commonly used in many industries, like textile, food, paper, plastic, cosmetics and coloring industries. These industries commonly use synthetic dyestuff as a colorant. Discharge of dye containing waste water into the environment contaminates surface water and ground water however  $7 \times 10^5$  tones of dye stuff are produced annually [1,2]. The dyes in the waste water even at very low concentration affect the aquatic life and human health by polluting the environment. The toxicity and carcinogenicity have lead to exploration of possible detoxicants [3, 4]. Dyes can causes allergic, dermatitis, skin irritation, cancer, mutation, etc. In general dyes are poorly biodegradable and some of the dyes produce aromatic amine which is highly carcinogenic in nature [5, 6]. Many investigators have studied the feasibility of using inexpensive alternative materials like peral millet husk, date pits, saw dust, buffing dust of leather industries, coir pith, crude oil, residue, tropical grass, olive stone almond

shell, pine bark, wool waste, coconut shell etc [7, 8]. The present study seeks to evaluate the efficiency of a activated calcite powder adsorbent in the removal of malachite green dyes from dye solution.

### EXPERIMENTAL METHODS

#### Adsorbent

Natural Calcite stone were collected from the granite quarry. The calcite stone washed with distilled water to remove the impurities and dried for four hours at  $125^\circ\text{C}$  in a hot air oven. The dried material was ground well to a fine powder and then activated around  $400^\circ\text{C}$  then sieved and stored in plastic bags and kept ready for use.

#### Adsorbate

The stock solution of malachite green concentration 1000 mg/L was prepared by dissolving 1 g of malachite green in 1000 ml of double distilled water. Different concentration of dyes solution range from (50 to 250 mg/L) were prepared from the stock solution by appropriate dilution

#### Batch adsorption experiments

Batch adsorption was tested by adding 25 mg of ACP to 50 ml of the dye solution of different initial

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concentration (50 to 250 mg/L) at a particular pH. The experiment was carried out using a wrist action shaker for the period of 180 min and 120 rpm using 250 ml stopper glass flasks at (30°C to 60°C). The residual concentrations of dyes in each sample after adsorption at different time intervals were determined by UV-Visible spectrophotometer. The equilibrium  $q_e$  (mg/g) was calculated by the following mass balance principle.

$$q_e = \frac{(C_0 - C_e)V}{M} \dots\dots\dots (1)$$

Where  $C_0$  and  $C_e$  are the initial and equilibrium concentrations (mg/L) of dyes,  $V$  is the volume (L),  $M$  is the weight (g) of the adsorbent. The removal efficiency of the adsorbents on dyes was calculated by using the following expression.

$$R\% = \frac{C_0 - C_t}{C_0} \times 100 \dots\dots\dots (2)$$

### Kinetic Experiments

The batch kinetic [9] experiments were basically similar to those used testing the adsorption equilibrium method. The dyes samples were taken at specific time intervals and the concentration of dyes was similarly measured. The all kinetic experiments were carried out at 30, 40, 50 and 60°C with initial dye concentration (50, 100, 150 200 and 250 mg/L) the amount of adsorption at time  $t$ . The  $q_t$  (mg/g) was calculated by.

$$q_t = \frac{(C_0 - C_t)V}{M} \dots\dots\dots (3)$$

Where  $C_0$  (mg/L) is the liquid phase concentration of dye at any time.

### Results and discussion

#### Effect of contact time and initial dye concentration

The experimental results of adsorption of various dye concentrations with contact time are shown in Fig.1. This figure shows that the % removal initially increases and reaches the limiting value. So that the equilibrium was established at 50 minutes. Hence all the remaining experiments were carried out at 50 minutes. The equilibrium data were given in Table.1 reveals that, the percentage removal was decreases with increase in initial dyes concentration. This was due to the number of available active sites was remains constant but the initial concentration of dyes increases, so that % removal decreases [10, 11].

#### Effect of adsorbent dosages

The effect of the ACP doses was studied at 60°C by varying the amount of adsorbent dose 50-250 mg for the initial concentration of 50 mg/L Fig. 2 reveals that increase in percentage removal of MG dye with increases in dose of adsorbent due to the increase in adsorbent surface area and the availability of more adsorption sites.

#### Effect of pH

The solution pH is one of the most important factors that control the adsorption of MG dye. To examine the effect of pH on the % removal of MG dye the pH of initial solution were varied from 2.0 to 10.0 by adding NaOH or HCl in to them. The % removal increases as the pH increases up to 6.5. there after the % removal decreases. At pH 6.5 the optimum % removal takes place. So the remaining experiment was carried out at pH 6.5. The experimental result was shown in Fig. 3.

### Adsorption isotherm studies

To quantify the sorption capacity of the absorbent for the removal of dyes, the most commonly used isotherms, are the Freundlich and Langmuir isotherms and hence these were used in this study.

#### Freundlich isotherm

Linear form of Freundlich isotherm model [12] is represented by the equation

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \dots\dots\dots (4)$$

Where  $q_e$  is the amount of dyes adsorbed per unit weight of the adsorbent (mg/L)  $K_f$  is (mg/g(L/mg)) the measure of adsorption capacity and  $1/n$  is the adsorption intensity. The value of  $K_f$  and  $n$  are calculated from the intercept and slope of the plot of  $\log q_e$  Vs  $\log C_e$  respectively. The constant  $K_f$  and  $n$  values are given in Table.2. In general the  $K_f$  value increases the for a given adsorbate increases. The magnitude of the exponent  $1/n$  gives an indication of the favorability of adsorption. The value of  $n > 1$  represents favorable adsorption condition [7] (or) the value of  $n$  are in the range of 1 to 10 confirms the favorable condition for adsorption. The adsorption coefficient  $K_f$  of dyes on activated calcite was found from 5.8107 to 6.1062 L/g. The  $K_f$  value indicates that the saturation time for adsorption of dyes is attained quickly due to the high affinity of activated calcite powder towards the adsorbate. The values of  $n$  were in the range of 1.8611 to 2.9265 (mg/L) for MG dyes adsorption. So Freundlich isotherm is suitable for this adsorption. The  $K_f$  values also indicate the multilayer adsorption were possible. This reveals that the activated calcite powder was more efficient for the removal of MG dyes.

#### Langmuir isotherm

The Langmuir isotherm model [13] is based on the assumption that maximum adsorption corresponds to a saturated monolayer of solute molecules on the adsorbent surface. The linear form of the Langmuir isotherm equation can be described by

$$C_e/q_e = (1/Q_m b) + (C_e/Q_m) \dots\dots (5)$$

Where  $C_e$  (mg/L) is the equilibrium concentration of the dye,  $q_e$  (mg/g) is the amount of dye per unit weight of adsorbent,  $Q_m$  and  $b$  are Langmuir constants related to adsorption capacity and rate of adsorption respectively.  $Q_m$



is the amount of dye at complete monolayer coverage (mg/g) which gives the maximum adsorption capacity of the adsorbent and  $b$  (L/mg) is the Langmuir isotherm constant that relates to the energy of adsorption or rate of adsorption. The linear plot of  $C_e/q_e$  against the equilibrium concentration  $C_e$  shows the Langmuir model. The Langmuir constant  $Q_m$  and  $b$  were determined from the slope and intercept of the Langmuir plot and these values are given in Table.2. The feasibility of the Langmuir isotherm can also be expressed in terms of the dimensionless constant separation factor  $R_L$  [14, 15] by the equation

$$R_L = \frac{1}{1+bC_0} \dots \dots \dots (6)$$

Where  $C_0$  (mg/L) is the initial concentration of adsorbent and  $b$  (L/mg) is Langmuir isotherm constant. The parameter  $R_L$  indicates the nature of the isotherm.

$R_L > 1$  unfavorable

$R_L = 1$  Linear

$0 < R_L < 1$  Favorable

$R_L = 0$  Irreversible

The  $R_L$  values lies between 0 to 1 indicate favorable adsorption for all initial concentration study. The calculated  $R_L$  values were given in Table.3. The calculated  $R_L$  values were with in the range of 0.0365 to 0.1811. So the adsorption of MG follows the Langmuir isotherm.

### Thermodynamic study

Thermodynamic parameter such as change in free energy ( $\Delta G^0$ ) (KJ/mol), Enthalpy ( $\Delta H^0$ ) (KJ/mol) and entropy ( $\Delta S^0$ ) (JK/mol) were calculated by using the following equation (7,8) and (9)

$$K_0 = C_{Solid} / C_{Liquid} \dots \dots \dots (7)$$

$$\Delta G^0 = -RT \ln K_0 \dots \dots \dots (8)$$

$$\log K_0 = \frac{\Delta S^0}{2.303R} - \frac{\Delta H^0}{2.303RT} \dots \dots \dots (9)$$

Where  $K_0$  is the equilibrium constant,  $C_{Solid}$  is the solid phase concentration at equilibrium (mg/L).  $C_{liquid}$  is the liquid phase concentration at equilibrium (mg/L).  $T$  is temperature in Kelvin and  $R$  is the gas constant (8.314 J mol<sup>-1</sup>K<sup>-1</sup>). A graph was drawn between  $\log K_0$  vs  $1/T$ . The  $\Delta H^0$  and  $\Delta S^0$  values obtained from the slope and intercept of van't Hoff plots. These values were given in Table.4. The negative  $\Delta G^0$  were indicate the adsorption is spontaneous in nature and also the magnitude of  $\Delta G^0$  indicate the adsorption is physical adsorption (ie, less than 70 KJ/mol) The value of  $\Delta H^0$  is positive, this indicates the adsorption is endothermic process. The positive  $\Delta S^0$  indicates increased randomness during the adsorption. This also support the adsorption was physical adsorption [16, 17]

### Adsorption kinetics

The kinetics studies were done by using pseudo second order [18] Elovich [19, 20] and intra-particle diffusion [21] models.

### The pseudo-second-order kinetic model

The linear form of pseudo second order equation is expressed as

$$\frac{dq_t}{dt} = K_2(q_e - q_t)^2 \dots \dots \dots (10)$$

where  $k_2$ (g/mg min) is the pseudo second order rate constant .For the boundary conditions  $t = 0$  to  $t = t$  and  $q_t = 0$  to  $q_t = q_t$  integrated form of Eq. (10) becomes:

$$(q_e - q_t) = \frac{1}{q_e} + K_2 t \dots \dots \dots (11)$$

This is the integrated rate law for a pseudo second order reaction. Equation (11) can be rearranged to obtain Eqs (12) which has a linear form:

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} t \dots \dots \dots (12)$$

If the initial adsorption rate ( $h$ )(g/mg min) is

$$h = K_2 q_e^2 \dots \dots \dots (13)$$

Then Eqs. (12) and (13) becomes:

$$\frac{1}{q_t} = \frac{1}{h} + \frac{1}{q_e} t \dots \dots \dots (14)$$

where  $k_2$ (g/mg min) is the pseudo second order rate constant,  $q_e$  is the amount of dye adsorbed on the per unit mass of adsorbent (mg/g) at equilibrium,  $q_t$  is the amount of dye adsorbed at time "t". A Graph is drawn between  $t/q_t$  versus "t", the  $q_e$  and  $k_2$  can be calculated from the slope and intercepts of the graph. The correlation coefficient value ( $\gamma$ ) were also calculated. These values are given in Table.5. The correlation coefficient value ( $\gamma$ ) for the pseudo second order was greater than 0.9900 (ie,  $\gamma > 0.9900$ ) and also  $q_e$  value calculated from the model was almost equal to the experimental value. So the adsorption of MG follows pseudo second order model.

### The Elovich equation

The Elovich model equation is generally expressed as

$$\frac{dq_t}{dt} = \alpha \exp(-\beta q_t) \dots \dots \dots (15)$$

where  $\alpha$  is the initial adsorption rate (mg g<sup>-1</sup> min<sup>-1</sup>) and  $\beta$  is the desorption constant (g mg<sup>-1</sup>) during any one experiment. To simplify the Elovich equation. Chien and Clayton assumed  $\alpha\beta t \gg 1$  and by applying boundary conditions  $q_t = 0$  at  $t = 0$  and  $q_t = q_t$  at  $t = t$  Eq.(15) becomes:

$$q_t = \frac{1}{\beta} \ln(\alpha\beta) + \frac{1}{\beta} \ln t \dots \dots \dots (16)$$

If dyes adsorption fits 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 parameters  $\alpha$ ,  $\beta$  and correlation coefficient ( $\gamma$ ) were given in Table.5. The initial adsorption rate ( $\alpha$ ) and desorption constant ( $\beta$ ) increases with increase in initial concentration of MG dyes and also the correlation coefficient ( $\gamma$ ) were greater than 0.9900 (ie,  $\gamma > 0.9900$ ). The Elovich model can also suitable for the MG adsorption onto ACP.

### Intra-particle diffusion model

According to Weber and Morris suggested the intra-particle diffusion model is



$$q_t = K_{id} t^{1/2} + C \dots\dots\dots (17)$$

Where  $k_{id}$  is the intra-particle diffusion constant (mg/g min), and  $q_t$  is the amount of the dye adsorbed at time "t". According to Weber and Morris model, a graph is drawn between  $q_t$  and  $t^{1/2}$ , the line was passing through the origin. But here the intercept value indicates the lines were not passing through origin. This was due to that the intra-particle diffusion takes place along with some other process. This may be boundary layer adsorption or instantaneous adsorption.

### Effect of the ionic strength on the adsorption of Malachite Green

The effect of sodium chloride on the adsorption of malachite green on to ACP is shown in Fig.4. In a low solution concentration of NaCl had less influence on the adsorption capacity. The partial neutralization of the positive charge on the adsorbent surface and a consequent compression of the electrical double layer by the  $Cl^-$  anion cause the increase in the adsorption of the malachite green at higher ionic strength. The chloride ions can also enhances adsorption of malachite green ion onto activated calcite by pairing of their charges and hence reducing the repulsion between the malachite green molecules adsorbed on the surface. The ACP to adsorb more of positive malachite green dye [16, 17, 22, 23].

**Table 1. Equilibrium parameter for the adsorption of MG dye onto ACP**

MG (mg / L)	Ce (mg / L)				Qe (mg / g)				% Removal of MG dye			
	30°C	40°C	50°C	60°C	30°C	40°C	50°C	60°C	30°C	40°C	50°C	60°C
50	4.9720	4.5824	4.2496	4.0432	90.056	90.83	91.5008	91.9136	90.056	90.83	91.500	91.913
100	17.269	15.620	13.397	11.804	165.46	168.7	173.204	176.390	82.730	84.38	86.602	88.195
150	37.479	33.803	30.272	27.155	225.04	232.3	239.454	245.689	75.013	77.46	79.818	81.896
200	73.236	68.576	63.679	58.819	253.52	262.8	339.454	282.361	63.381	65.71	84.863	70.590
250	119.95	114.05	63.679	102.74	260.08	271.8	372.641	294.504	52.017	54.37	74.528	58.900

**Table 2. Langmuir and Freundlich isotherm parameter for adsorption of MG dye onto ACP**

Temperature (°C)	Langmuir parameter		Freundlich parameter	
	$Q_m$	b	$K_f$	n
30	286.1309	0.0909	5.8107	2.9265
40	299.1008	0.0941	5.8979	2.8731
50	494.764	0.0452	5.1320	1.8611
60	323.577	0.1055	6.1062	2.7871

**Table 3. Dimensionless separation factor ( $R_L$ ) for adsorption of MG dye onto ACP**

$C_0$ (mg/L)	$\Delta G^0$				$\Delta H^0$	$\Delta S^0$
	30°C	40°C	50°C	60°C		
50	- 5550.835	- 5968.794	- 6381.572	- 6729.448	6.4266	39.5732
100	- 3946.587	- 4389.473	- 5011.674	- 5567.706	12.6685	54.7089
150	- 2769.424	- 3213.115	- 3692.364	- 4178.738	11.4882	47.0177
200	- 1382.059	- 1692.702	- 4629.514	- 2424.078	17.7098	63.6556
250	- 203.415	- 456.772	- 2883.101	- 996.3114	14.9639	50.6270

**Table 4. Thermodynamic parameter for the adsorption of MG onto ACP**

$C_0$	Temp °C	pseudo second order				Elovich model			Intra-Particle diffusion		
		$q_e$	$K_2$	$\gamma$	h	$\alpha$	$\beta$	$\gamma$	$K_{id}$	$\gamma$	Intercept
50	30	100.080	0.00128	0.9919	12.8452	110.5234	0.0691	0.9918	1.6225	0.9942	0.1832
	40	100.2021	0.00135	0.9921	13.5907	146.8037	0.0724	0.9920	1.6443	0.9958	0.1724
	50	100.2511	0.001443	0.9932	14.5075	198.9961	0.0758	0.992	1.6650	0.9960	0.1625
	60	100.8469	0.00142	0.9936	14.4698	187.4683	0.0745	0.9927	1.6635	0.9975	0.1646
100	30	184.2176	0.000672	0.9941	22.7999	177.5959	0.0367	0.9926	1.5748	0.9976	0.1887
	40	187.3143	0.00676	0.9926	23.7098	209.8361	0.0371	0.9929	1.5943	0.9979	0.1821
	50	190.9157	0.000718	0.9929	26.1605	280.9139	0.0379	0.9935	1.6238	0.9940	0.1725
	60	194.6650	0.00070	0.9911	26.8841	279.7291	0.0370	0.9939	1.6310	0.9950	0.1737
150	30	254.8363	0.000421	0.9909	27.3184	155.4677	0.0246	0.9941	1.4945	0.9960	0.2095
	40	260.6738	0.00044	0.9910	30.2472	201.5094	0.0250	0.9948	1.5291	0.9961	0.1983
	50	267.7725	0.000462	0.9913	33.1244	249.6425	0.0251	0.9950	1.5585	0.9920	0.1903
	60	272.2697	0.00038	0.9922	28.1846	307.9750	0.0268	0.9991	1.5751	0.9913	0.1750

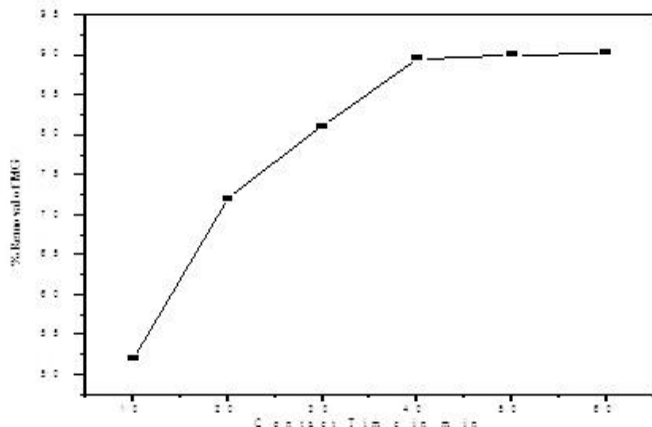


200	30	297.9219	0.00027	0.9927	24.5051	85.6862	0.0183	0.9994	1.3339	0.9931	0.2583
	40	305.3655	0.00028	0.9937	26.8841	105.9773	0.0186	0.9960	1.3751	0.9915	0.2436
	50	314.0891	0.00029	0.9950	29.4047	127.8459	0.0186	0.9985	1.4105	0.9920	0.2329
	60	321.6071	0.00032	0.9959	33.6923	171.4670	0.0190	0.9986	1.4557	0.9936	0.2176
250	30	322.7917	0.00019	0.9957	19.5742	49.6622	0.0150	0.9980	1.1362	0.9938	0.3205
	40	333.8572	0.00019	0.9980	21.1575	55.8777	0.0148	0.9983	1.1731	0.9958	0.3101
	50	348.8991	0.000179	0.9982	21.8066	57.7381	0.0148	0.9927	1.1899	0.9988	0.3105
	60	353.7305	0.00020	0.9985	25.2662	73.5208	0.0145	0.9929	1.2486	0.9986	0.2878

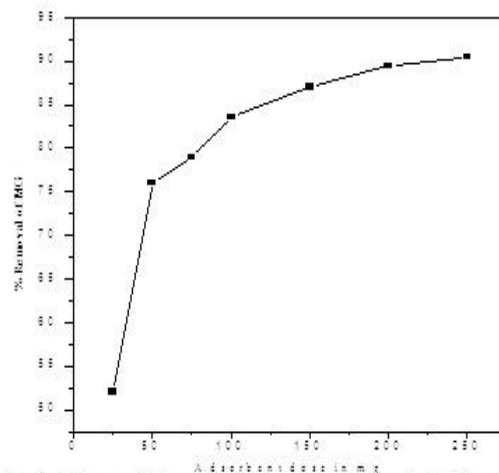
**Table 5. The kinetic Parameter for the adsorption of MG on to ACP**

C <sub>0</sub> (mg/L)	Temperature (°C)			
	30	40	50	60
50	0.1803	0.1753	0.3067	0.1593
100	0.0991	0.0960	0.1811	0.0866
150	0.0683	0.0661	0.1285	0.0594
200	0.0521	0.0504	0.0995	0.0452
250	0.0421	0.0407	0.0812	0.0365

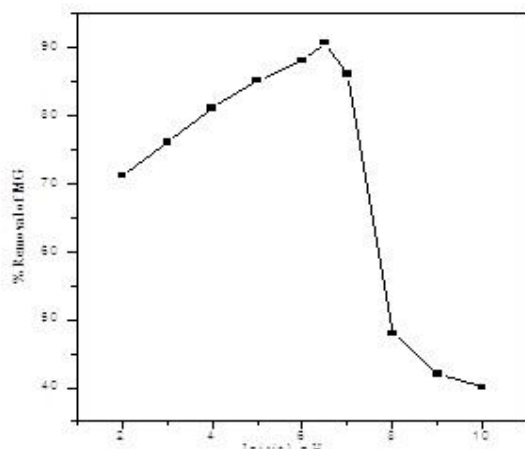
**Fig.1. Effect of Contact time on the removal of MG dye onto ACP**



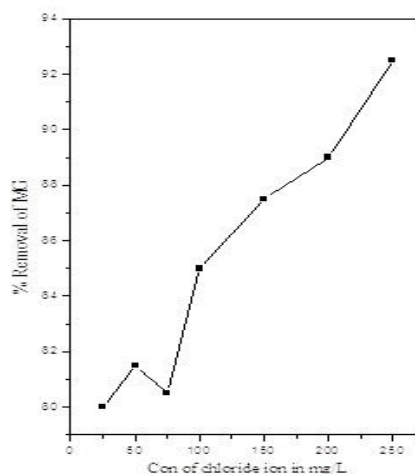
[MG] = 50mg/L; Temp 30°C; Adsorbent dose = 25mg/50ml



**Fig. 2. Effect of Adsorbent dose on the removal of MG onto ACP**  
[MG] = 50mg/L; Contact time = 30 min; Temp 30°C.



**Fig. 3. Effect of Initial pH on the removal of MG onto ACP**  
[MG] = 50mg/L; Contact time = 30 min; Temp 30°C; Adsorbent dose = 25mg



**Fig. 4. Effect of Other ions on the removal of MG onto ACP**  
[MG] = 50mg/L; Contact time = 30 min; Temp 30°C; Adsorbent dose = 25mg/50ml



## CONCLUSION

The adsorption characteristics of MG dye onto Activated Calcite Powder are strongly affected by the initial dye concentration, initial pH and the adsorbent dose. The pH 6.5 was favorable for the optimum adsorption of MG dye by ACP. The  $R_L$  values and other adsorption parameters indicate both Langmuir and Freundlich isotherms favorable for ACP adsorption. The pseudo second order, Elovich and intra-particle kinetic model were

found to applicable for the adsorption of MG onto ACP reaction model. The thermodynamics parameters  $\Delta G^0$ ,  $\Delta H^0$  and  $\Delta S^0$  values indicate the adsorption is endothermic and physical adsorption.

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