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Adsorption of Malachite Green from Synthetic Waste Water onto Activated Carbon from Corn Cob

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Abstract: Activated carbon which is prepared from Waste corn cob was used as the material for adsorption of the dye malachite green. This dye is widely used in the field of medicine but its release into water bodies can pose to be lethal to flora and fauna. It is also carcinogenic to human beyond certain levels. In the present investigation a trial was made to evaluate the adsorption characteristics of activated carbon from corn cob. The systematic optimization involved study of parameters like- effect of contact time, dye concentration, coke dosage, mesh size variation, pH, temperature and RPM. Langmuir and Fruendlich isotherms were applied to the data obtained at equilibrium. The adsorption capacities (Qm) obtained from the Langmuir isotherms are 52.63, 100, 90.90, 76.92 mg/g at 25, 35, 45 and 55^{o}C respectively. At low temperatures the adsorption was not favorable, but with increase in temperature there was spontaneous adsorption of the dye on the chosen substrate.

Keywords: Malachite green, Corn cob coke, Adsorption kinetics, Thermodynamic studies.

INTRODUCTION

Malachite green (MG) is a popular dye and is used extensively. This dye when released into water checks the biological activity in aquatic lives and also poses threat to human beings. It has mutagenic and carcinogenic characteristics[1]. Histopathology studies have revealed that malachite green causes detrimental effects in liver, gill, kidney, intestine, gonads and pituitary gonadotropic cells. It causes sinusoidal congestion and focal necrosis in liver, damages mitochondria and also causes nuclear alterations[2].Many conventional methods are available for removing dyes but among all adsorption on to activated carbon was found to be the most feasible one for large scale applications. Due to high porosity activated carbon adsorbs pollutants and this is less cost demanding. There are various conventional methods of removing dyes including coagulation and flocculation, oxidation or ozonation and membrane separation. However, these methods are not widely used due to their high cost and economic disadvantage. Chemical and electrochemical oxidations, coagulation are generally not feasible on large scale industries[3].

Activated carbons are widely used as industrial adsorbents for separation, purification of solid and liquid phase, and as recovery processes due to their texture being highly porous and they have large capacity to absorb pollutants but their large scale application is cost intensive[4,5].Adsorption techniques are widely used to remove certain classes of pollutants from waters, especially those that are not easily biodegradable. Dyes represent one of the problematic groups. Currently, a combination of biological treatment and adsorption on activated carbon is becoming more common for removal of dyes from wastewater. Waste materials from agriculture and industry as Activated carbons from solid wastes[6-8] and from agricultural wastes and by products of cellulosic origin like peanut hull[9], Maize bran[10] , Saw dust[11], Sugar beet pulp[12], Crab peel[13], egg shells[14], orange peel[15]. Few authors have used chicken feathers for the adsorption of dyes[16]. In the present investigation corn cobs which are left as waste are used to prepare coke for adsorption of malachite green.

MATERIALS AND METHODS

Preparation of Activated carbon: Corn cob activated carbon, is used as adsorbent for dye removal. It is a form of carbon that has been processed to make it extremely porous and thus to have a very large surface area available for adsorption or chemical reactions. Due to its high degree of microporosity, just one gram of activated carbon has a surface area of 1000 m^2. This is prepared by treating cobs pieces with concentrated sulphuric acid in 1:1 ratio for an hour. The pieces are then washed thoroughly dried and kept in furnace at 400^{o}C for 4h in a furnace for complete carbonization and activation. The dried cob pieces are then kept in oven for 100^{o}C overnight, ground to make it into a fine powder and then sieved as per the requirement.

Adsorption studies: Batch adsorption studies were carried out by varying parameters with a working volume of 100 ml of synthetic effluent. The parameters studied include effect of contact time, malachite green concentration, particle size of coke, dosage of coke, pH and temperature. The results were noted after visible equilibrium is attained. Samples were withdrawn, filtered and then optical density was taken at 624nm using a UV visible spectrophotometer[17]. This is used to determine concentration of malachite green left unadsorbed (Ce), Concentration of MG adsorbed (Qe) and percentage of adsorption[7].

RESULTS AND DISCUSSION

Effect of contact Time: Effect of contact time was evaluated by keeping MG at 20ppm and coke dosage at 1%. pH was not adjusted and the flask was kept shaking at 100rpm on a rotary shaker. Samples were collected at every 15minutes time interval and percentage of MG adsorbed was evaluated which is represented in **Fig.1**. It may be observed at a contact time of 90minutes would be ideal for further studies.

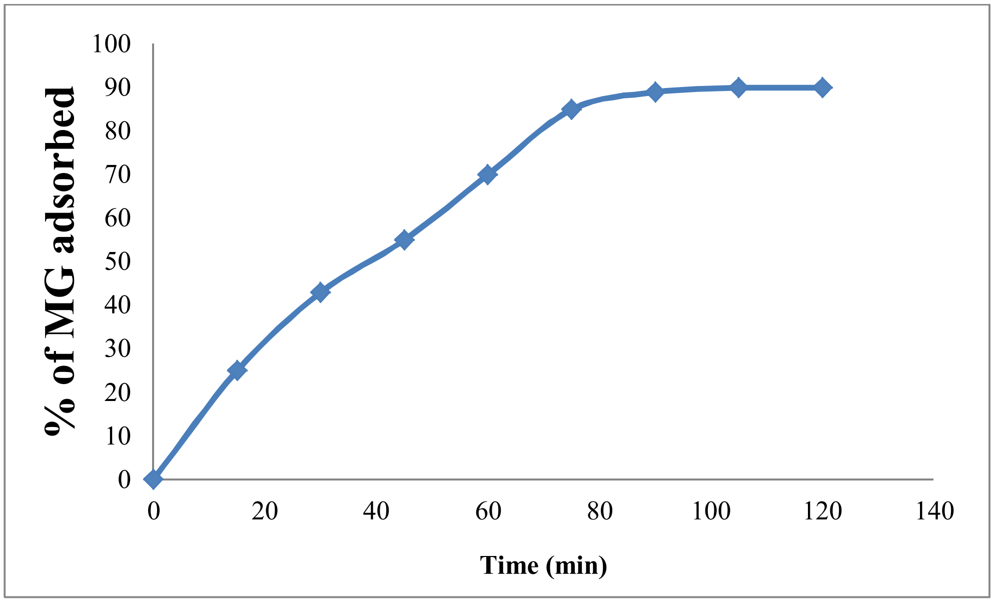

Figure 1: Effect of contact time.

Effect of Malachite green on Adsorption percentage: Effect of Malachite green concentration was studied by varying the Malachite Green Concentration between 5 to 70 ppm. A coke concentration of 1gm/100ml was added and the flask was subjected to agitation at 100 rpm at room temperature at 30^{o}C. **Fig. 1** reveals that with increase in malachite green concentration the percentage of malachite green adsorbed has decreased which is due to the saturation of surface of the coke with the dye. The coke dosage becomes the limiting factor. But there amount of dye adsorbed per gram of coke at the point of equilibrium is 56mg/g for the 70ppm dye concentration.

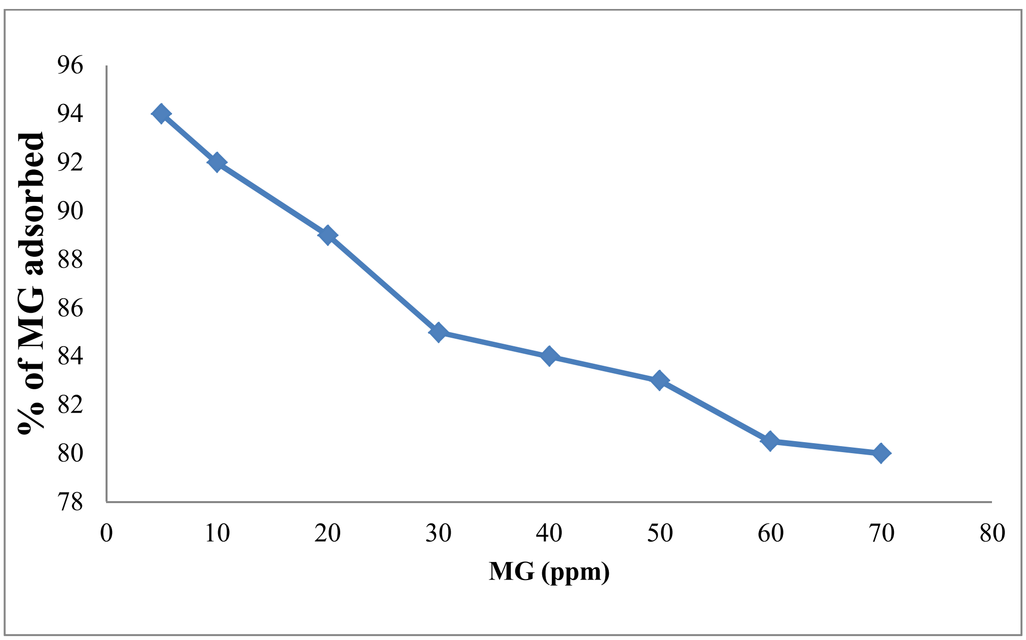

Figure 2: Effect of MG concentration on adsorption.

Table-1: Parameters studies and results obtained.

Malachite green concentration (ppm)	Ce ppm	Qe (mg/gm)	Ce/Qe	log Ce	logQe	% of Malachite Green adsorbed
5	0.3	4.7	0.06383	-0.52288	0.672098	94
10	0.8	9.2	0.086957	-0.09691	0.963788	92
20	2.2	17.8	0.123596	0.342423	1.25042	89
30	4.5	25.5	0.176471	0.653213	1.40654	85
40	6.4	33.6	0.190476	0.80618	1.526339	84
50	8.5	41.5	0.204819	0.929419	1.618048	83
60	11.7	48.3	0.242236	1.068186	1.683947	80.5
70	14	56	0.25	1.146128	1.748188	80
pH						
3	2.6	17.4	0.149425	0.414973	1.240549	87
4	2.27	17.73	0.128032	0.356026	1.248709	88.65
5	1.6	18.4	0.086957	0.20412	1.264818	92
6	2.8	17.2	0.162791	0.447158	1.235528	86
7	3.2	16.8	0.190476	0.50515	1.225309	84
8	2.2	17.8	0.123596	0.342423	1.25042	89
9	1.8	18.2	0.098901	0.255273	1.260071	91
Particle size(Micrometers)						
45	4	16	0.25	0.60206	1.20412	80
60	3	17	0.176471	0.477121	1.230449	85
85	2.47	17.53	0.140901	0.392697	1.243782	87.65
100	2.14	17.86	0.119821	0.330414	1.251881	89.3
120	2	18	0.111111	0.30103	1.255273	90
150	2	18	0.111111	0.30103	1.255273	90
200	2	18	0.111111	0.30103	1.255273	90
Coke %						
0.5	8.6	11.4	0.754386	0.934498	1.056905	57
1	1.6	18.4	0.086957	0.20412	1.264818	92
1.5	1	19	0.052632	0	1.278754	95
2	0.2	19.8	0.010101	-0.69897	1.296665	99
2.5	0	20	0		1.30103	100
3	0	20	0		1.30103	100
3.5	0	20	0		1.30103	100
4	0	20	0		1.30103	100
Temperature (^{o}C)						
25	3.6	16.4	0.219512	0.556303	1.214844	82
35	1.8	18.2	0.098901	0.255273	1.260071	91
45	1.2	18.8	0.06383	0.079181	1.274158	94
55	0.8	19.2	0.041667	-0.09691	1.283301	96
65	0.6	19.4	0.030928	-0.22185	1.287802	97
Agitation (rpm)						
0	3	17	0.176471	0.477121	1.230449	85
100	2	18	0.111111	0.30103	1.255273	90
150	0.8	19.2	0.041667	-0.09691	1.283301	96
200	1.2	18.8	0.06383	0.079181	1.274158	94
250	2	18	0.111111	0.30103	1.255273	90

Effect of pH on Adsorption percentage: Effect of pH was studied by varying the pH of the synthetic effluent between 3 to 9. pH of the solution was adjusted using 1MNaOH or HCl. Malachite Green concentration was maintained at 20 ppm in all the flasks and after adjusting pH the flasks were subjected to agitation at 100rpm was maintained and kept at room temperature. Samples were collected and estimated as mentioned in the previous study and the results are presented in **Table 1** and **Fig.2**. It may be observed that the adsorption has increased till pH 5, then there was a drop and again a further increase in adsorption was seen. This may be due to variation in the charges at the active sites or the characteristics of MG. the dye is reported to form a zwitter ion beyond pH 7 which form aggregates and contribute to dye adsorption.

Effect of Particle size on Adsorbent percentage: Effect of adsorbent particle size was studied by varying the mesh size between 45 to 200µm. Adsorbate concentration was maintained at 20 ppm in all the flasks and after adjusting pH to 7, the flasks to be subjected to agitation at 100rpm at 37°C. Results obtained were tabulated in **Table 2**, which indicate that decrease in particle size has contributed to an increase in percentage adsorption of the dye. As particle size reduces, surface area available increases which also increase the available active sites for malachite green to bind. But during filtration, the smallest particles interfered with filtration hence a particle size of 120 µm was preferred.

Table-2: LogCo/Ct for various Malachite green concentrations at different time intervals.

Time (min)	Log Co/Ct for 5ppm	Log Co/Ct for 10ppm	Log Co/Ct for 20ppm	Log Co/Ct for 30ppm	Log Co/Ct for 40ppm	Log Co/Ct for 50ppm	Log Co/Ct for 60ppm	Log Co/Ct for 70ppm
15	0.39794	0.522879	0.59346	0.68473	0.704433	0.694649	0.657577319	0.669007
30	0.19382	0.318759	0.361511	0.464284	0.498256	0.441291	0.429846387	0.426797
45	0.075721	0.21467	0.207608	0.278464	0.325598	0.299296	0.278464168	0.280432
60	0.045757	0.124939	0.102373	0.17177	0.207608	0.207608	0.191563946	0.191886
75	0.036212	0.060481	0.065502	0.091515	0.11776	0.133122	0.13667714	0.137528
90	0.026872	0.036212	0.05061	0.070581	0.075721	0.080922	0.09420412	0.09691

Effect of maize cob coke concentration: Effect of adsorbate concentration was studied by varying the its dosage between 0.5 to 4.0. Particle size was maintained at 120 mesh size adsorbate concentration was maintained at 20 ppm in all the flasks and after adjusting pH the flasks (pH 5) were subjected to agitation at 100rpm was maintained and kept at room temperature. The results in **Table 1** indicate that increase in corn cob coke concentration contributes to increase in dye adsorption. This is due to increase in available surface area that leads to increase in parentage of dye adsorbed. But 2.5% may be considered as optimal as maximum dye has been absorbed by this concentration of coke.

Effect of RPM: Effect of RPM was studied by varying the RPM between 50 to 250. Particle size was maintained at 150 mesh size and MG concentration was maintained at 20 ppm in all the flasks and after adjusting pH to 5 the flasks were subjected to agitation at room temperature. Agitation causes uniform exposure of coke to dye there by allowing maximum saturation of active sites by malachite green.

Results indicate that (**Table 1**) when there is no agitation also there was 85% adsorption while maximum adsorption was observed at 150 rpm. Further increase in agitation has resulted in a decrease in percentage of adsorbed dye which may be due to desorption.

Effect of Temperature: Effect of Temperature was studied by varying the temperature between 25 ^{o}C to 65 ^{o}C. Particle size was maintained at 120 mesh size. Adsorbate concentration was maintained at 20 ppm in all the flasks and after adjusting pH to 5 the flasks were subjected to agitation at 120rpm was maintained and kept at room temperature. Increase in temperature has resulted in increase in adsorption which may be due to increase in diffusivity of malachite green into the adsorbate thereby contributing to increase in adsorption.

Kinetics of Adsorption: The kinetics of sorption reveals the solute uptake rate of the reaction. It is one of the important characteristics in defining the efficiency of adsorption. In the present study, the kinetics of the dye removal were carried out to understand the behavior of the chosen carbon. The adsorption of the malachite green dye from an aqueous solution follows reversible first order kinetics, when a single species is considered on a heterogeneous surface. The heterogeneous equilibrium between the dye solutions and the activated carbon is expressed as

$$\mathrm{A} \underset{k_2}{\overset{k_1}{\rightleftharpoons}} \mathrm{B}$$

Where

*k1*is the forward rate constant and *k2* is the backward rate constant. A represents dye remaining in the aqueous solution and B represents dye adsorbed on the surface of activated carbon. The equilibrium constant (*Ko*) is the ratio of the concentration adsorbate in adsorbent and in aqueous solution (*Ko*=*k1*/*k2*).In order to study the kinetics of the adsorption process, the following kinetic equation proposed by Natarajan and Khalaf, as cited in literature, has been employed[1].

$$\log Co/Ct=(Kad/2.303)t$$

Where

Co and *Ct* is the concentration of the dye (in mg/L) at time zero and at time *t*, respectively. The rate constants (Kad) can be determined from the slopes of linear plots of log Co/Ct versus t for various concentrations and temperatures.The rate constants for the parameters effect of malachite green on adsorption was evaluated by linear method as well as by second order polynomial methods. **Fig.3** reveals the percentage of dye absorbed at various initial concentrations of malachite green. The log Co/Ct values represented in **Table 2**and R^2 values and Dissociation constant values for the parameter are presented in **Table 3**. The rate constant indicate that with increase in concentration of the dye there was an initial increase in the value of dissociation constant till 50ppm indicating the efficiency of the system. Further increase has resulted in a slight decrease in Kad indicating a saturation of the active sites rapidly by the dye and a slow process thereafter. This is also revealed by the R^2 values which are nearer to 0.9 for linear equation fit while when analyzed by second order polynomial method the values indicate a good fit.

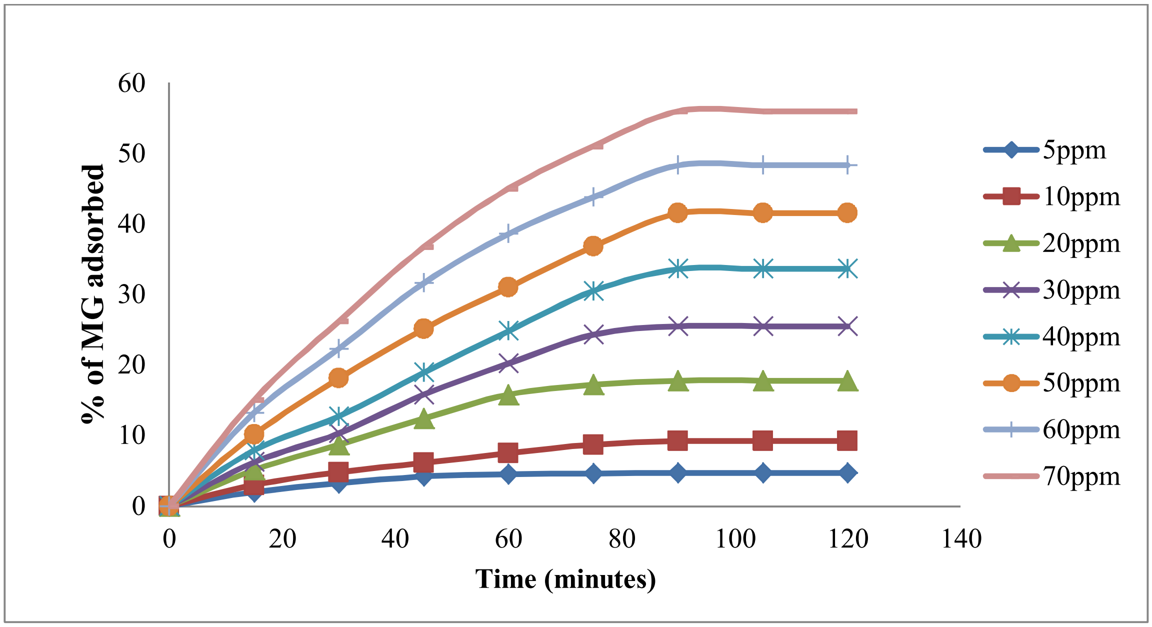

Figure 3: Effect of ppm on adsorption at different time intervals.

Table-3: R square values and Dissociation constant (Kd) values for linear and polynomial equations.

MG conc (ppm)	Linear derivation		Polynomial second order	
	R^2	Kd	R^2	Kd
5	0.754	0.365	0.978	0.599
10	0.916	0.542	0.995	0.719
20	0.817	0.600	0.998	0.859
30	0.916	0.723	0.999	0.959
40	0.945	0.761	0.999	0.953
50	0.918	0.718	0.993	0.930
60	0.905	0.670	0.995	0.895
70	0.897	0.682	0.994	0.911

Adsorption Isotherm: The experimental data obtained for the parameter effect of concentration of malachite green on adsorption at various temperatures was analysed by linear form Langmuir and Freundlich isotherms[18,19]. The equation for Langmuir isotherm is:

$$C e/Q e = 1/Qmb + Ce /Qm$$

Where

*Ce*is the equilibrium concentration (mg/L), *Qe*is the amount adsorbed at equilibrium (mg/g), and *Qm*and *b* areLangmuir constants related to adsorption efficiency and energy of adsorption, respectively.

The values are represented in **Table 4**. The linear plots of *Ce/Qe*versus *Ce*suggest the applicability of the Langmuir isotherms (**Fig.4**). The values of *Qm*and *b* were determinedfrom slope and intercepts of the plots and the R^2 value were noted in **Table 5**. Langmuir isotherms indicate that the process is reasonably efficient indicating total utilization of surface area has been sufficiently done. The favourability of the adsorption process was calculated from the separation factor (R_L) which is represented in **Table 6**. The values range between 0 to 1 and indicate the ongoing process of adsorption[20].

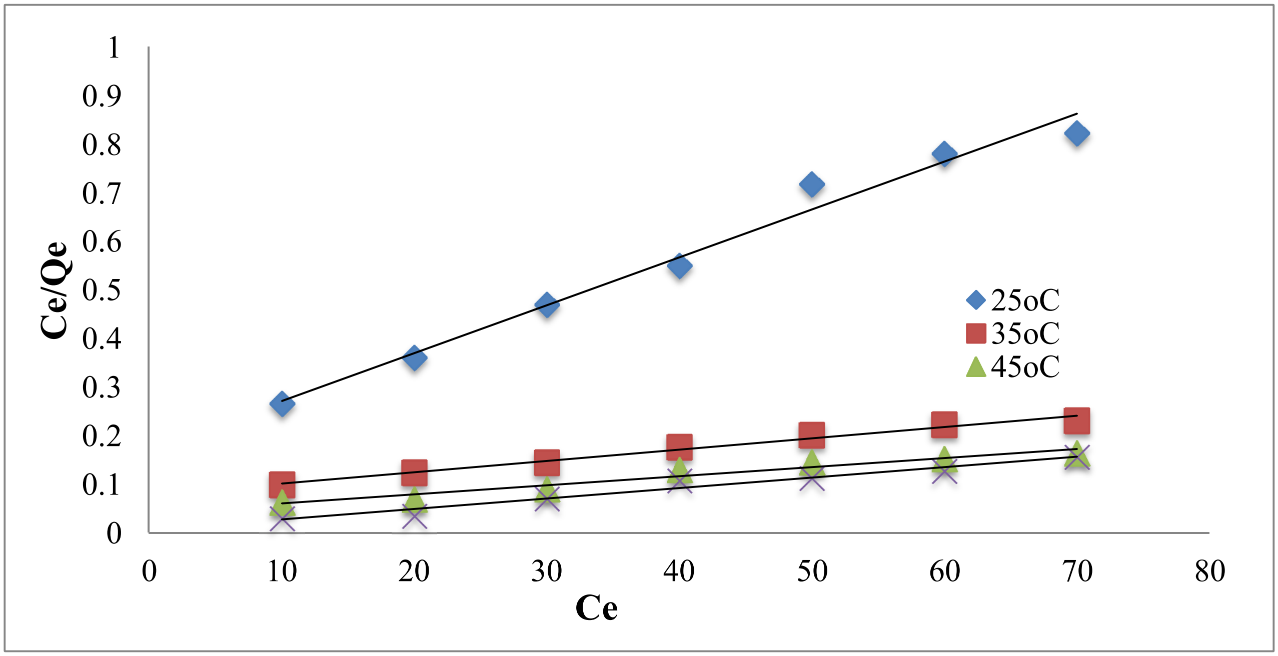

Figure 4: Linear Langmuir isotherms for the adsorption of malachite green on Corn Cob Coke.

Table-4: Equilibrium parameters for the adsorption of malachite green onto corn cob coke.

[MG]	**Qemg/g**				**Ce**				**% adosrbed**			
	Temperature (°C)											
	25°C	35°C	45°C	55°C	25°C	35°C	45°C	55°C	25°C	35°C	45°C	55°C
10	7.9	9.1	9.4	9.7	2.1	0.9	0.6	0.3	79	91	97	97
20	14.7	17.8	18.7	19.3	5.3	2.2	1.3	0.7	73.5	89	93.5	96.5
30	20.4	26.2	27.5	28	9.6	3.8	2.5	2	68	87.33	91.66	93.33
40	25.8	34	35.4	36.08	14.2	6	4.6	3.92	64.5	85	88.5	90.2
50	29.1	41.6	43.6	44.9	20.9	8.4	6.4	5.1	58.2	83.2	87.2	89.8
60	33.7	49.06	52.04	53.2	26.3	10.94	7.96	6.8	56.16	81.76	86.73	88.67
70	38.4	56.9	60.1	60.5	31.6	13.1	9.9	9.5	54.85	81.286	85.86	86.43

The Freundlich equation was employed for the adsorption of malachite green dye on the adsorbent. TheFreundlich isotherm was represented by

$$\log Qe = \log Kf + 1/n \log Ce \quad \ldots(1)$$

Where

*Qe*is the amount of malachite green dye adsorbed (mg/g), *Ce*is the equilibrium concentration of dye insolution (mg/L), and *Kf*and *n* are constants incorporating the factors affecting the adsorption capacity and intensityof adsorption, respectively. Linear plots of log *Qe*versus log *Ce*were drawn and values of *Kf*and n are obtained which are represented in table 6. R^2 value indicates best fit of work. High values of n indicate high affinity between adsorbate and the adsorbent and this is also an indication of chemisorptions[2].The thermodynamic studies help in understanding the nature of adsorption. The capacity of carbon is known to increase with increase in temperature.The thermodynamic parameters such as changes in free energy (ΔG^o)(KJ/mol), enthalpy (ΔH^o)(KJ/mol) and entropy (ΔS^o)(J/K/mol) were determined for the adsorption processes. This is done according to Van't Hoff equation 2 and free energy calculation using equation 3 and by plotting logarithmic values of the equilibrium constant (keq) as lnQe/Ce against temperature as 1/T.

$$\ln Keq = \frac{-\Delta H}{RT} + \frac{\Delta S}{R} \quad \ldots(2)$$

$$\Delta G = -RT \ln Keq \quad \ldots(3)$$

Where

R is gas constant,

Keq is adsorption equilibrium constant.

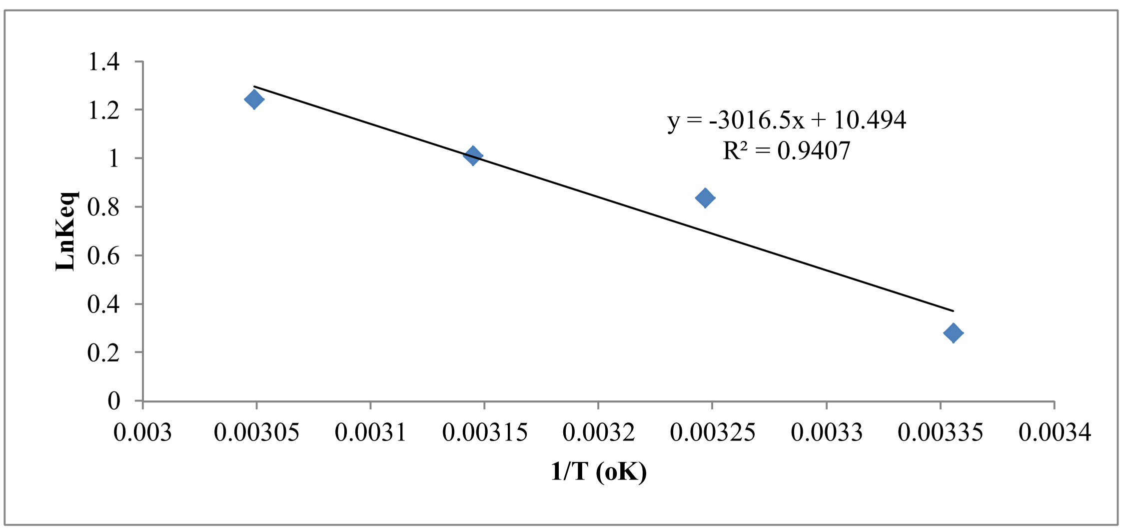

Figure 5: Van't Hoff plot of MG adsorption.

Table-5: Langmuir isotherm results.

Dye	Temp	Statistical parameters/constants		
MG	^oC	r^2	Q_m	b
	25	0.973	52.63	13.89
	35	0.965	100	10.00
	45	0.946	90.90	5.63
	55	0.945	76.92	2.77

Table-6: Dimensionless factor Separation factor (R_L).

MG (mg/L)	Temperature (^oC)			
	25	35	45	55
10	0.007	0.091	0.0175	0.035
20	0.0036	0.05	0.009	0.0178
30	0.0085	0.0033	0.006	0.012
40	0.0018	0.0025	0.0044	0.0089
50	0.0014	0.0020	0.0035	0.0071
60	0.0012	0.0016	0.0030	0.0060
70	0.0010	0.0014	0.0051	0.0051

Table-7: Fruendlich isotherms results.

Dye	Temp	Statistical Parameters/Constants		
	^oC	r^2	K_f	n
	25	0.993	5.47	1.769
MG	35	0.997	10.162	1.494
	45	0.987	14.256	1.538
	55	0.982	19.678	1.99

Table-8: Equilibrium constants and thermodynamic parameters for the adsorption of malachite green onto Corn cob coke.

[MG]	K_{eq} Temperature ^oC				ΔG^o (KJ/mol)				ΔH^o (KJ/mol)	ΔS^o (J/K/mol)
	25°	35°	45°	55°	25°	35°	45°	55°		
10	1.325	2.314	2.752	3.476	-0.7	-2.113	-2.676	-3.397	56.10	199.95
20	1.020	2.091	2.666	3.317	-0.049	-1.888	-2.536	-3.269	60.85	213.50
30	0.755	1.931	2.398	2.639	0.7	-1.648	-2.262	-2.646	50.216	176.67
40	0.597	1.735	2.041	2.219	1.277	-1.410	-1.886	-2.174	42.518	149.65
50	0.331	1.596	1.919	2.176	2.739	-1.203	-1.723	-2.119	48.047	166.20
60	0.248	1.501	1.878	2.057	3.455	-1.039	-1.665	-1.967	47.69	164.29
70	0.195	1.469	1.803	1.851	4.051	-0.984	-1.559	-1.679	43.72	150.90

Plots of Van'thoff were drawn (**Fig. 5**) and resulting values are represented in **Table 8**. Positive ΔH values obtained between the ranges 50 to 61KJ/Mol were indicate that malachite green adsorption is more favorable. The negative value of ΔG^o indicate the highly favorable and spontaneous nature of adsorption but positive value at 25°C indicate that the temperature is nor favorable for adsorption of malachite green. The positive values of ΔS^oindicate the increased disorder and randomness at solid surface interface between malachite green and Corn cob coke[7].

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