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## Research Article

### THERMODYNAMIC AND KINETIC ANALYSIS ON THE REMOVAL OF MALACHITE GREEN DYE USING ACTIVATED *CORCHORUS OLITORIUS* - L LEAVES

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#### Abstract

The research of the present work was to investigate the removal of Malachite Green from aqueous solution by using *Corchorus Olitorius*. 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. In this study, activated *Corchorus Olitorius* - L- Leaves was studied for its potential use as an adsorbent for removal of Malachite Green. The various factors affecting adsorption, such as initial dye concentration, contact time, adsorbent dose and effect of temperature, 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 ACONC may be utilized as a low cost adsorbent for Malachite Green dye removal from aqueous solution.

**Keywords:** Activated *Corchorus Olitorius* - L- Nano Carbon (ACONC), Malachite Green dye; Adsorption isotherm; Kinetics; Equilibrium models

#### Introduction

Disposal of dyeing industry wastewater pose one of the major problems, because such effluents contain a number of contaminants including acid or base, dissolved solids, toxic compounds, and color. Out of these, color is the first contaminant to be recognized because; it is visible to the human eye. Removal of many dyes by conventional waste treatment methods is difficult. Since, these are stable to light and oxidizing agents and are resistant to aerobic digestion. Possible methods of color removal from textile effluents include chemical oxidation, froth flotation, adsorption, coagulation, etc. Among these, adsorption currently appears to offer the best potential for overall treatment, and it can be expected to be useful for a wide range of compounds. Recognizing the high cost of activated Nano Carbon, many investigators have studied the feasibility of cheap and commercially available materials as its possible replacements. Such materials range from industrial waste products such as waste rubber tyres,

blast furnace slag and lignin to agricultural products such as wool, rice straw, coconut husk, saw dust and peat mass. In order to assess the ability of activated Nano Carbon for dye removal, malachite green (MG) has been selected for the present study.

#### Materials and Methods

##### Preparation of Activated Nano Carbon

*Corchorus Olitorius* - L- Leaves used in this study was collected from the local agriculture land of Thiruvarur, Tamilnadu, India. The raw biosorbent was cut into small sizes and then washed thoroughly with hot-distilled water followed by double-distilled water to remove any unwanted particles. It was then dried and activated in a muffle furnace at 1200 °C. The activated material was sieved to get uniform size particles <177µm (80mesh). The same ACONC was used throughout the experiment.



*Corchorus Olitorius* - L- L- L. (Jew's Mallow)

### Adsorbate

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

### Material Developments.

Sorption studies were performed by the batch technique to obtain rate and equilibrium data. For isotherm studies, a series of 50 mL Iodine flask were employed. Each test tube was filled with 50 mL of dye solution of varying concentrations and maintained at the desired pH and temperature. A known amount of adsorbent was added into each tube and agitated intermittently for a maximum period of 1 h. The reaction period (10-60 minutes) was found to be quite sufficient for equilibrium attainment for the dye. After this period, the supernatant solution was centrifuged and the uptake of the dye was monitored spectrophotometrically at 425 nm. These concentrations were however decided after a good deal of preliminary investigation wherein the adsorbent was found to remove the dye to different extent. Sorption studies were carried out at 30, 40, 50 and 60 °C to find out the effect of temperature. The effect of pH was observed by studying the adsorption of dye over a pH range of 2-10.

### Batch adsorption experiments

Batch adsorption was tested by adding 25 mg of ACONC to 50 ml of the dye solution of different initial concentration (50 to 250 mg/L) at a particular pH. The experiment was carried out using a wrist action shaker for the period of 60 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 dye,  $V$  is the volume (L),  $M$  is the weight (g) of the adsorbent. The removal efficiency of the adsorbent 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 40 minutes. Hence all the remaining experiments were carried out at 40 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 ACONC doses was studied at 30°C by varying the amount of adsorbent dose 10-250 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 adsorbent 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

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

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 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 co-efficient  $K_f$  of dyes on *Corchorus Olitorius - L- Leaves* was found from 5.3577 to 5.7508 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 Nano Carbon towards the adsorbate. The values of  $n$  were in the range of 2.0839 to 2.0437 (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 Nano Carbon 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

$$C_e/q_e = C_e/q_{max} + 1/(b q_{max}) \dots\dots\dots(5)$$

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 constants,  $Q_m$  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:

$$R_L = \frac{1}{1 + b C_0} \dots\dots\dots(6)$$

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 and 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 within the range of 0.2695 to 0.0697. So, the adsorption of MG follows the Langmuir isotherm.

**Thermodynamic study**

Thermodynamic parameters, such as change in free energy (  $G^0$  ) (KJ/mol), enthalpy (  $H^0$  ) (KJ/mol) and entropy (  $S^0$  ) (JK/mol) were calculated by using the following equations (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  $H^0$  and  $S^0$  values obtained from the slope and intercept of van't Hoff plots. These values were given in Table.4. The negative  $G^0$  were indicate the adsorption is spontaneous in nature and also the magnitude of  $G^0$  indicates that the adsorption is physical adsorption (ie, less than 70 KJ/mol). The value of  $H^0$  is positive; this indicates the adsorption is endothermic process. The

positive  $S^0$  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 [22] 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 dye 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 ACONC.

**Intra-particle diffusion model**

According to Weber and Morris [25], 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 MG

The effect of sodium chloride ion the adsorption of MG on to ACONC is shown in Fig.4. In a low solution concentration of  $Cl^-$  ion 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 MG at higher ionic strength. The chloride ions can also enhances adsorption of MG ion onto ACONC by pairing

of their charges and hence reducing the repulsion between the MG molecules adsorbed on the surface. The ACONC to adsorb more of positive MG dye [16, 17, 22, and 23].

#### SEM and FTIR of ACONC

The SEM images of ACONC (Fig.4a and 4b) Shows the SEM micrographs of ACONC sample before and after dye adsorption. It is clear that ACONC has considerable numbers of heterogeneous layer of pores where there is a good possibility for dye to be adsorbed. The surface of dye-loaded adsorbent, however, clearly shows that the surface of ACONC is covered with dye molecules.

The FTIR spectrum of ACONC before and after adsorption (Fig.5a and 5b) shows that some peaks were shifted or disappeared and that new peak were also detected. These changes observed in the spectrum indicated the possible involvement of those functional groups on the surface of the ACONC in adsorption process

**Table: 1** Equilibrium parameters for the adsorption of mg dye onto ACONC

$M_0$	Ce (Mg / L)				Qe (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
50	4.582	4.258	3.487	3.755	90.836	91.484	93.024	92.490	90.836	91.484	93.024	92.490
100	12.32	11.86	9.498	8.589	175.34	176.28	181.00	182.82	87.674	88.140	90.502	91.410
150	26.58	23.54	20.88	18.21	246.82	252.90	258.23	263.57	82.274	84.302	86.078	87.857
200	48.25	44.29	20.88	37.32	303.48	311.40	358.23	325.34	75.872	77.851	89.559	81.336
250	74.25	70.25	41.26	61.32	351.48	359.48	417.46	377.34	70.297	71.896	83.492	75.468

**Table: 2.** Langmuir and freundlich isotherm parameter for the adsorption of mg dye onto ACONC

Temp. (°C)	Langmuir Parameters		Freundlich Parameters	
	$Q_m$	B	$K_f$	n
30°C	431.34	0.0542	5.3577	2.0839
40°C	444.48	0.0569	5.4215	2.0477
50°C	633.38	0.0447	5.1779	1.6082
60°C	460.75	0.0706	5.7508	2.0487

**Table: 3. Dimensionless separation factor ( $r_i$ ) for the adsorption of mg dye onto ACONC**

$(C_i)$	Temperature $\hat{C}$			
	30°C	40°C	50°C	60°C
50	0.2695	0.2600	0.3088	0.2205
100	0.1557	0.1494	0.1826	0.1239
150	0.1095	0.1048	0.1296	0.0861
200	0.0844	0.0807	0.1004	0.0660
250	0.0687	0.0656	0.0820	0.0535

**Table: 4. Thermodynamic parameter for the adsorption of mg dye onto ACONC**

$(C_0)$	$G^\circ$				U $\hat{H}$	U $\hat{S}$
	30°C	40°C	50°C	60°C		
50	-5778.34	-6178.38	-6956.43	-6951.49	7.3552	43.463
100	-4942.46	-5219.54	-6053.72	-6547.04	12.178	56.191
150	-3867.06	-4374.23	-4892.41	-5479.04	12.341	53.442
200	-2886.12	-3271.08	-5771.39	-4075.41	16.092	63.187
250	-2170.27	-2444.36	-4352.97	-3111.24	12.605	49.137

**Table: 5. The kinetic parameters for the adsorption of mg dye onto ACONC**

$C_0$	Temp $\hat{C}$	Pseudo second order				Elovich model			Intraparticle diffusion		
		$q_e$	$K_2$	$x$	$h$	$r$	$s$	$x$	$K_{id}$	$x$	$C$
50	30	96.922	0.0023	0.994	22.444	1584.8	0.1011	0.998	1.6249	0.992	0.1888
	40	96.861	0.0027	0.995	25.441	3257.5	0.1089	0.991	1.7457	0.994	0.1144
	50	98.207	0.0024	0.997	24.093	4428.5	0.1120	0.993	1.7641	0.991	0.1057
	60	97.351	0.0027	0.999	26.131	7792.4	0.1191	0.992	1.7403	0.992	0.1186
100	30	187.95	0.0010	0.998	37.073	1689.3	0.0490	0.991	1.6513	0.991	0.1644
	40	188.52	0.0011	0.997	39.764	2380.5	0.0508	0.992	1.7403	0.992	0.1162
	50	193.07	0.0011	0.998	41.486	2855.6	0.0505	0.991	1.7378	0.991	0.1229
	60	194.38	0.0011	0.992	43.813	3131.5	0.0504	0.993	1.7704	0.993	0.1034
150	30	264.33	0.0007	0.994	53.351	2590.2	0.0352	0.997	1.6724	0.991	0.1360
	40	270.26	0.0007	0.991	55.825	3043.6	0.0350	0.994	1.6705	0.992	0.1427
	50	276.82	0.0007	0.992	59.619	3377.2	0.0343	0.994	1.7129	0.991	0.1193
	60	280.38	0.0006	0.991	49.138	5907.2	0.0374	0.995	1.7219	0.991	0.1172
200	30	328.63	0.0005	0.992	54.887	1223.6	0.0251	0.997	1.6105	0.991	0.1395
	40	336.67	0.0005	0.991	57.585	1542.0	0.0253	0.999	1.6033	0.992	0.1527
	50	342.17	0.0005	0.993	60.834	1848.8	0.0254	0.998	1.6390	0.993	0.1361
	60	349.32	0.0005	0.991	67.745	2706.4	0.0259	0.997	1.6469	0.992	0.1398
250	30	384.25	0.0003	0.992	58.264	872.91	0.0201	0.998	1.6176	0.993	0.1081
	40	391.70	0.0004	0.994	61.583	1072.8	0.0202	0.999	1.5877	0.992	0.1366
	50	404.26	0.0003	0.991	59.491	878.82	0.0190	0.996	1.5754	0.998	0.1546
	60	409.30	0.0003	0.992	66.713	1350.8	0.0198	0.997	1.6185	0.995	0.1383

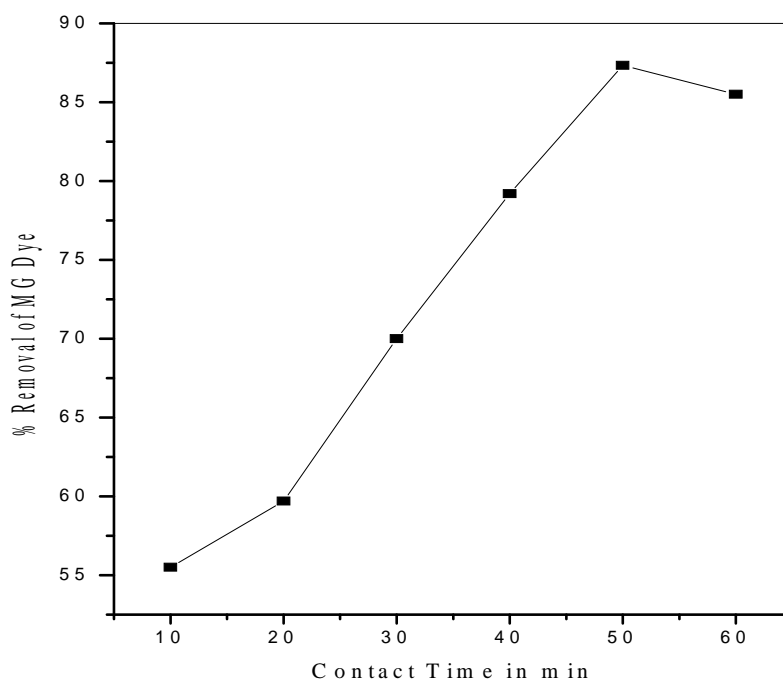


Fig:1- Effect of Contact Time on the Removal of MG dye  
[MG]=50 mg/L;Temperature 30°C;Adsorbent dose=25 mg/50 ml

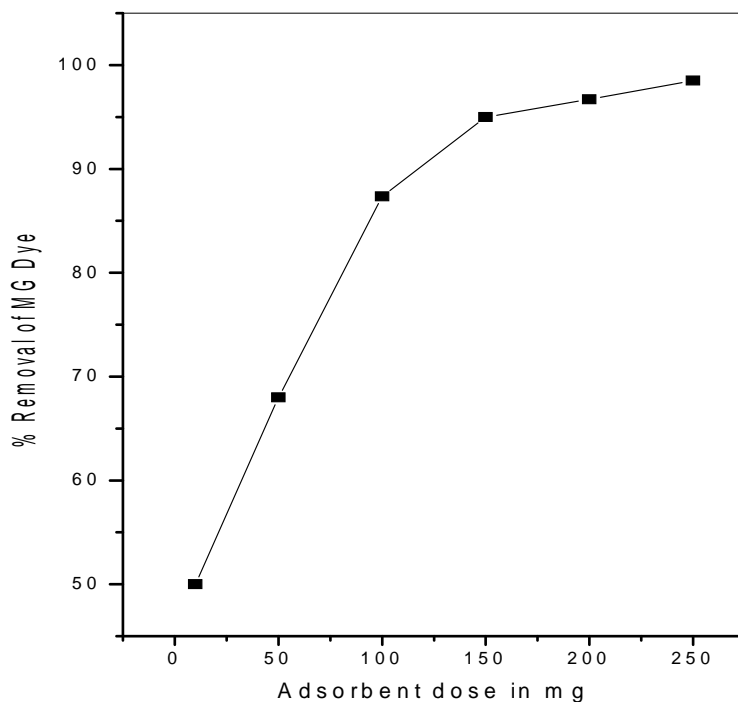
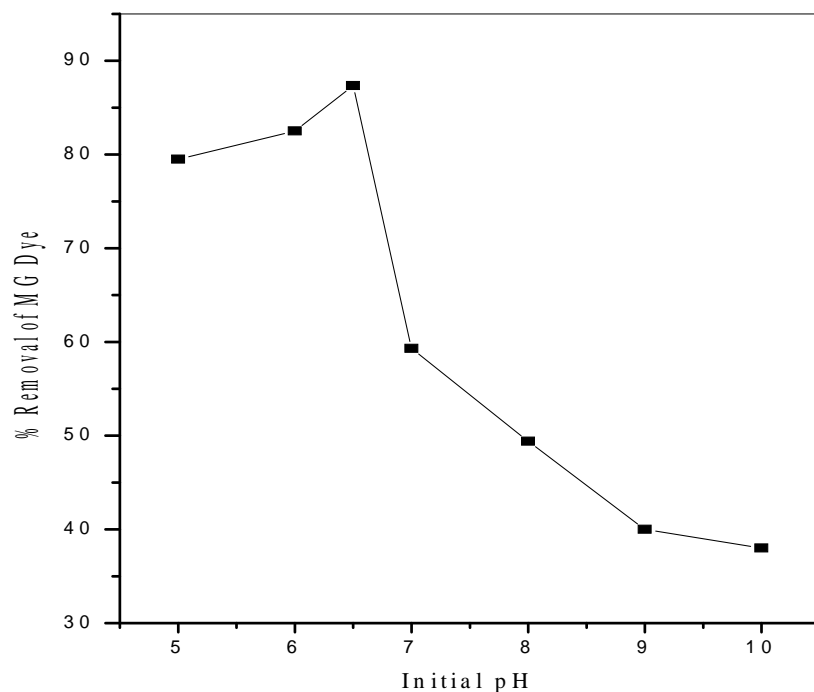


Fig:2- Effect of Adsorbent dose on the removal of MG dye  
[MG]=50 mg/L;Contact Time 60 min;Temperature 30°C



Fig;3- Effect of Initial pH on the removal of MG dye  
 [M G ]=50 m g/L;Temprature 30°C;A dsorbent dose=25 m g/50 m l

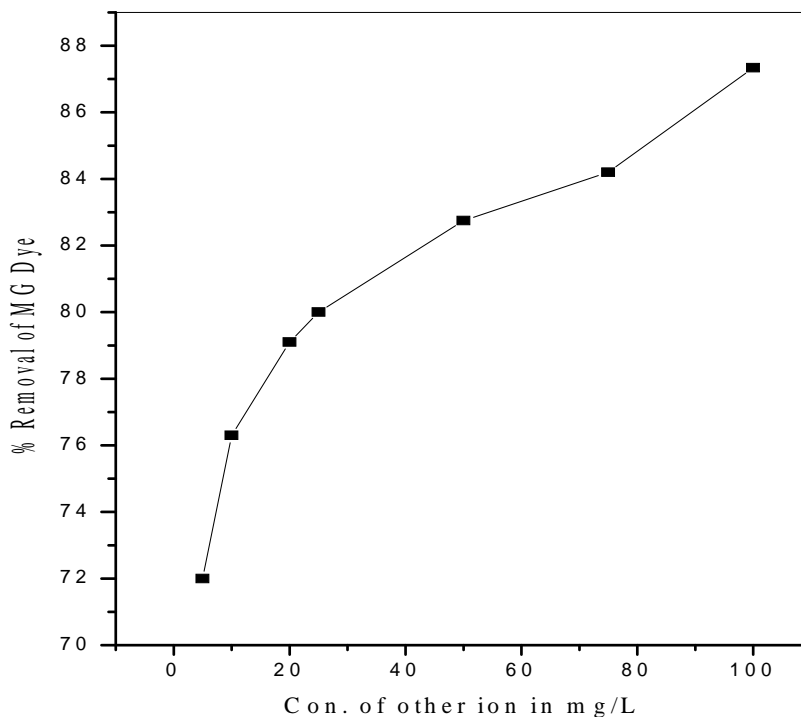


Fig.4-Effect ionic strength on the adsorption of MG dye  
 [M G ]=50 m g/L;C ontact time=60 m in;D ose=25 m g/50 m l



Fig.4.a- SEM image of ACONC before adsorption

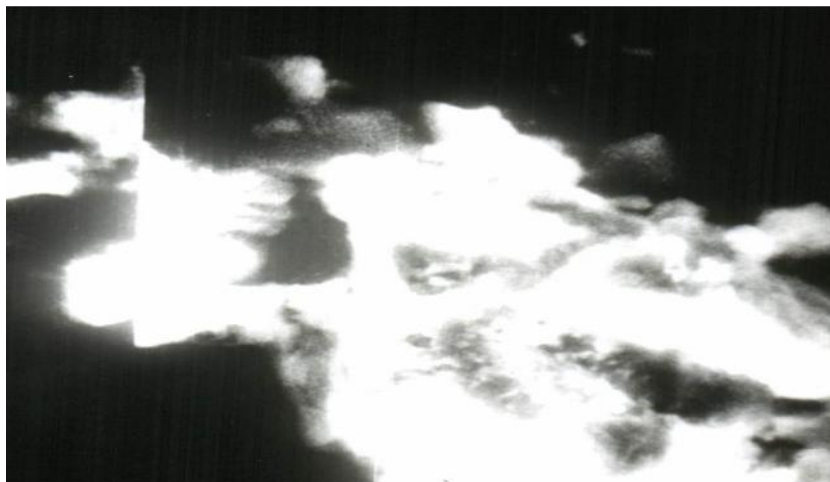


Fig.4.b-SEM image of ACONC after the adsorption of malachite green

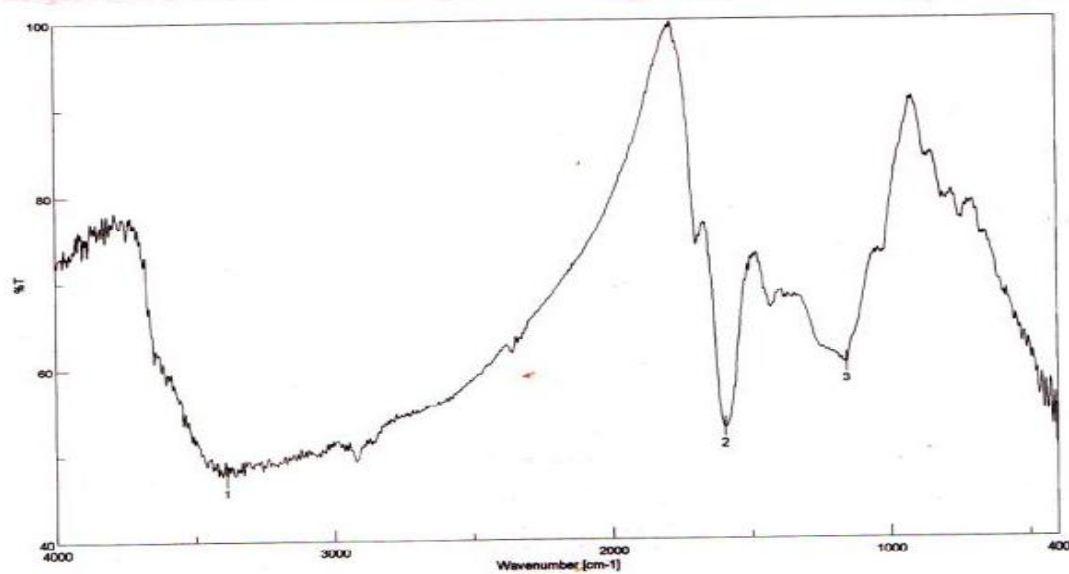
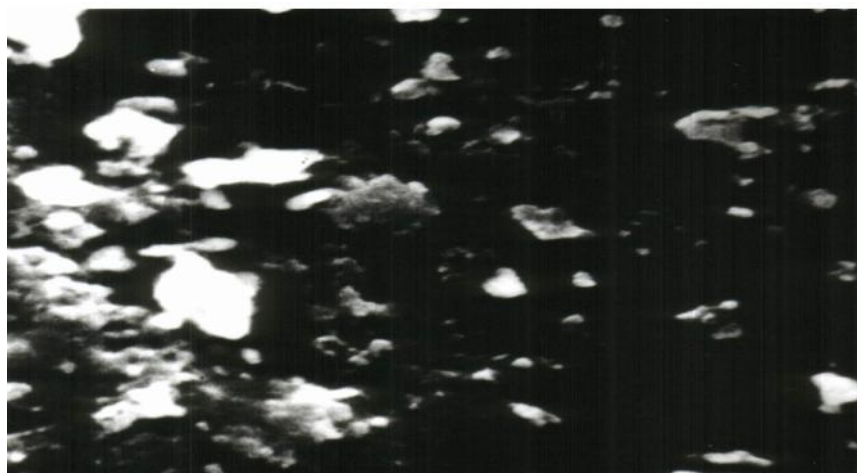


Fig 5.a-FT-IR Spectrum of ACONC before adsorption

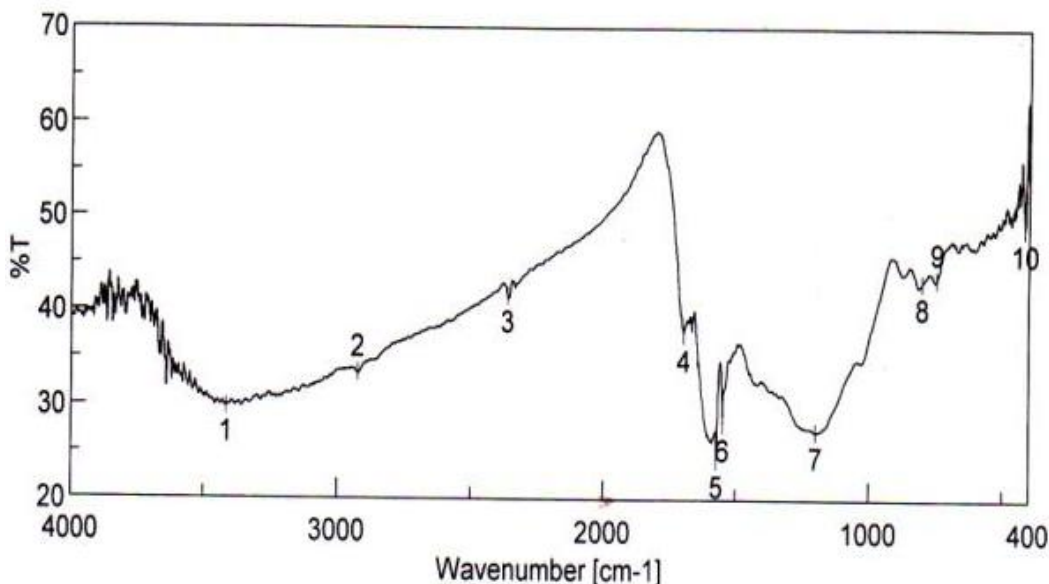


Fig 5b FT-IR Spectrum of ACONC after adsorption of malachite green

## Conclusion

The adsorption characteristics of MG dye onto Activated Nano Carbon (ACONC) 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 ACONC. The  $R_L$  values and other adsorption parameters indicate both Langmuir and Freundlich isotherms favorable for ACONC adsorption. The pseudo second order, Elovich and intra-particle

kinetic model were found to be applicable for the adsorption of MG onto ACONC reaction model. The thermodynamic parameters  $G^0$ ,  $H^0$  and  $S^0$  values indicate the adsorption is endothermic and physical adsorption.

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