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Adsorption of Malachite Green Using Activated Carbon From Copper Pod

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Abstract: *In the present study, the adsorption capacity of waste materials of copper pod fruit was determined for the removal of malachite green dye from aqueous solutions. This batch adsorption experiment was carried out to study for the various parameters such as adsorbent dosage, contact time, initial metal ion concentration, pH value and temperature. The morphology was investigated by scanning electron microscope and X-Ray diffraction data. The equilibrium data in aqueous solution were analysed using Freundlich, Langmuir isotherm model, Tempkin and Dubinin - Radushkevich isotherm. Adsorption isotherm of malachite green dye obeyed Langmuir isotherm model, Tempkin and Dubinin - Radushkevich isotherm. The kinetic data in aqueous solution were studied using Pseudo-first and Pseudo-second order kinetic models, Intraparticle diffusion and Elovich model. It was found that the adsorption of malachite green onto copper pod fruit activated carbon (CPFAC) followed pseudo second order kinetic model and Elovich model. The high values of correlation coefficient (R^2) of intraparticle diffusion model indicated that the pore diffusion plays a significant role for the adsorption.The results indicate that the activated carbon prepared from copper pod fruit could be employed as a low-cost alternative to commercial activated carbon for the elimination of malachite green from waste water and aqueous solutions.*

Keywords : *Adsorption, Copper pod fruit, Isotherm, Kinetic and Malachite green.*

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I. Introduction

In textile industry dyes are used to colour their products. Decolourisation of wastewater has become one of the major issues in wastewater pollution [1]. Wastewater from the textile industry is directly released into the nearby water bodies or on land [2]. These wastewaters have polluted nearby water bodies and have been reported to be slightly toxic even at low concentration. The disposal of such wastewater containing dyes can affect the quality of the receiving water bodies and aquatic ecosystem due to its toxicity, high BOD and COD value [3]. Pollution degrades the environment, which in turn leads to the depletion of the earth's ozone layer and makes a hole in the ozone cover [4]. There are different views regarding the origin of pollution crisis on the planet earth. These environments affected by developed countries and yet population, municipal wastes, industrial wastes, automobile gases, agricultural wastes, solid wastes and disposable wastes [5]. These above factors are produced different type of pollutions like global warming, greenhouse effect, acid precipitations, air pollution, water pollution, noise pollution, soil pollution. These pollutions are affecting the quality of life of the living – non living organisms –present and future. Therefore treatment of these pollutions is very important. Several physical, chemical and biological methods have been used for eliminating dyes from textile waste water [7]. Some conventional methods such as membrane process, electrochemical techniques, reverse osmosis, coagulation, flocculation, ultra-filtration, biological process, chemical reaction, photo oxidation, precipitation have been used for eliminating dyes from textile waste water [8]. Most of these methods are expensive due to their high capital and operational costs. Among these methods, activated carbon is a widely used method for removal of colour from wastewater because of its well developed porous structure and tremendous surface area [9]. In the present study, copper pod fruit activated carbon (CPFAC) has been used as a new low - cost adsorbent for removal of dye from textile waste water. The effect of various operating parameters such as adsorbent dosage, contact time, initial metal ion concentration, pH and temperature were studied.

II. Materials And Methods

2.1. Adsorbent preparation

The present studies Copper Pod were used as adsorbent for the removal dye from aqueous solution. They were collected from in around PSG College of Arts and Science, Coimbatore District, Tamilnadu, India which were available in abundant.

The copper pod fruit was first peeled off to obtain the outer skin of the fruit and then removal of the inner fleshy layer. The peel was washed with ordinary tap water to remove any dirt and sands. The washed materials were dried in sun light to evaporate the moisture present in it. The dried materials were carbonized with 1:1 Sulphuric acid. The charred material was filtered and washed with excess of water to remove residual acid from pores of the carbon particles. The filtered material was kept in muffle furnace at 600 ºC for 30 minutes. The carbonized material was ground to fine powder and then sieved with a particle size of 53 μm. The sieved adsorbent was kept in an airtight container for further adsorption studies.

2.2. Preparation of Adsorbate

A stock solution of malachite green was prepared by dissolving 1 gm of the malachite green dye in a 1litre of distilled water to give concentration of 1000mg/L. The working solutions were prepared by diluting the stock solution with distilled water to get the appropriate concentration of the working solutions. The dye concentration was determined using uv/visible spectrometer at a λ_{max} value of 617nm.

2.3. Effect of adsorbent dosage

The effect adsorbent dosage was studied by preparing different dye concentration (10, 20, 30, 40 & 50 mgs/lit) and different adsorbent dosage (0.1 – 1g). 50ml of dye solution was added to different 250ml conical flask containing different dosage of the adsorbents (0.1 – 1g).The conical flasks were well corked and agitated in a shaker. After agitation, the solution was filtered and analysed using digital spectrophotometer.

2.4. Effect of contact time

The effect of contact time on the amount of dye removal was studied for a period of 100min. 50ml of the dye solution (10, 20, 30, 40 &50ppm) was added to different 250ml conical flask containing 0.1g of the adsorbents. The conical flask was well corked and agitated in a shaker. The solution was filtered and analysed after each agitated time.

2.5. Effect of pH

The effect of pH on amount adsorbed was studied by various pH of the solution from 2 to 12. 50ml of (10, 20, 30, 40 & 50ppm) dye solution of known initial pH was added to different conical flask containing 1gm of the adsorbent. The pH was controlled using 0.1M HCl or 0.1M NaOH and was measured using pH meter. The mixture of the conical flask solution was well corked and agitated in a shaker. The mixture of the conical flask solution was filtered after the agitated time. The concentration of the dye in the filtrate was analysed using digital spectrophotometer.

2.6. Effect of Temperature

The effect of temperature on the amount of dye removal was studied for different temperatures. 50ml of (10, 20, 30, 40 &50ppm) dye solution was agitated with 0.1gm of the adsorbent for different temperatures using shaker.

III. Results And Discussion

3.1. Effect of contact time and initial dye concentration

The effect of contact time on percentage of dye removal using copper pod at different initial concentration is shown in figure.1.

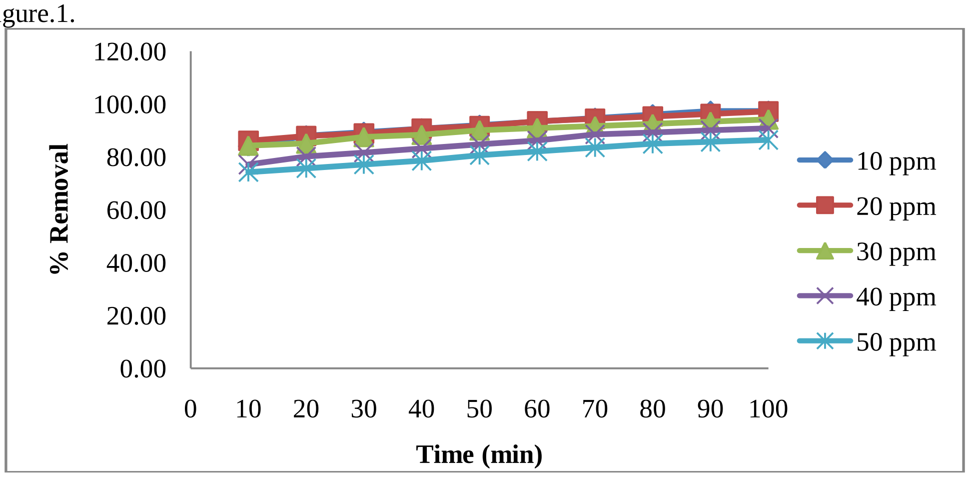

Fig.1: Effect of initial concentrations on the adsorption of Malachite green onto CPFAC.

The result (figure.1.) indicates that the percentage of dye removal increases in contact time and became constant when equilibrium was reached. The percentage of dye removal decreased with increase in initial dye concentration. It was seen that the adsorption of dye depends on its concentration [10]. The amount of dye adsorbed increases due to the increased number of active adsorption or bonding sites [11]. After attained equilibrium, there is no significant change in adsorption.

3.2. Effect adsorbent dosage

The effect of adsorbent dosage on the percentage removal of dye is shown in figure.2. Shows that increased adsorbent dosage increase the percentage of dye removal. Higher dosage of adsorbent increased the percentage of dye removal due to more surface area and functional groups or more binding sites are available on the carbon [12]. Further increase in adsorbent dose, there is no significant increase in adsorption [13].

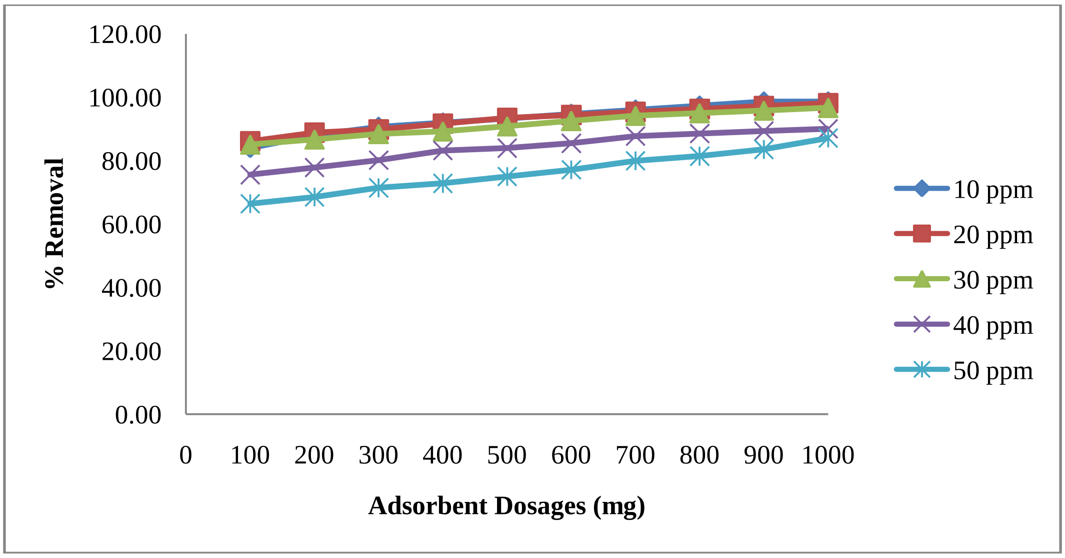

Fig.2: Effect of adsorbent dosages on the adsorption of Malachite green onto CPFAC.

3.3. Effect of pH

The pH of the adsorption has important parameters for controlling the percentage of dye removal. Figure.3. indicates that the effect of pH on the percentage removal of dye in the presence of activated carbon. The percentage of dye removal was increased with increase in initial pH of the dye solution from 2 to 7 and decreased with increase of pH of the dye solution from 8 – 12. This is due to the nature of the adsorbent [14].

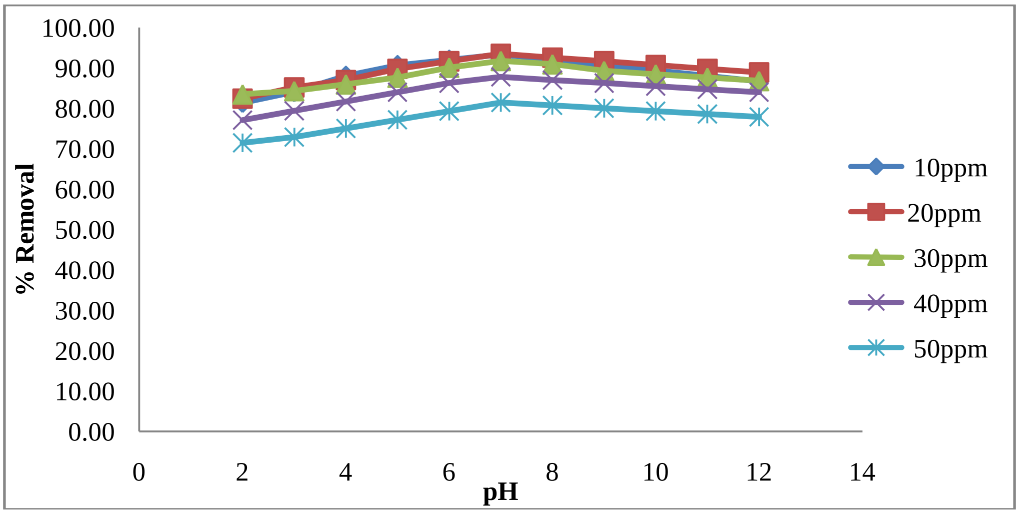

Fig.3: Effect of pH on the adsorption of Malachite green onto CPFAC.

3.4. Effect of Temperature

The effect of temperature on the removal of dye solution is shown in figure.4. It is clear that the percentage of dye removal decrease with increase in temperature for 40mgs/lit. It can be seen that the adsorption is exothermic in nature, and then high temperatures would inhibit or slow down the adsorption process [15].

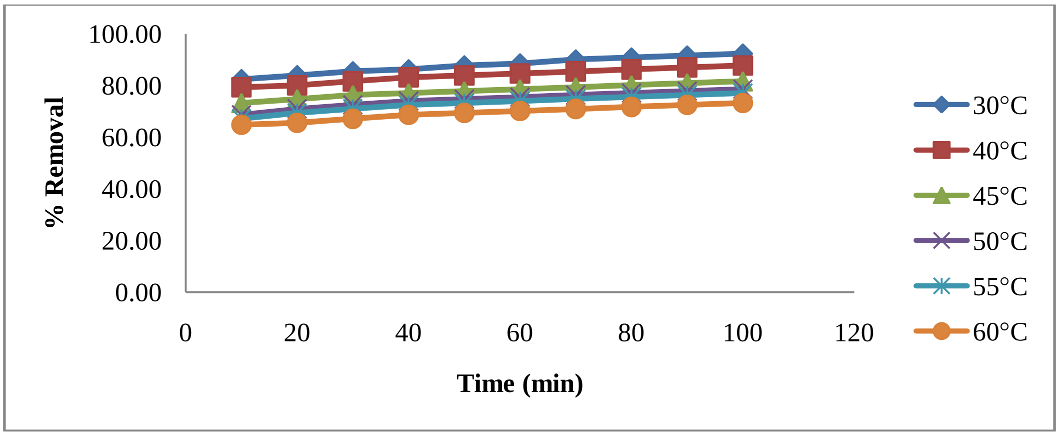

Fig.4: Effect of temperature on the adsorption of Malachite green onto CPFAC.

3.5. Scanning Electron Microscope analysis

The SEM photograph of the adsorbent is shown in figure.5. SEM characterization of adsorbent showed different structural features with non-uniform sizes and complete change in surface texture [16]. From the figure, it was also observed that wide varieties of pores and rough surface are present in the adsorbents. Pores development in an adsorbent is important since pores act as active sites or bonding sites which played the main role in adsorption [17]. The cavities and granular pores will increase the surface area of the adsorbent. The surface area of the adsorbents will be enhanced by the presence of more porosity, which can hold more solute from solution during adsorption [18]. These changes in the SEM morphologies are very good agreement with the literature for various materials [19, 20, 21].

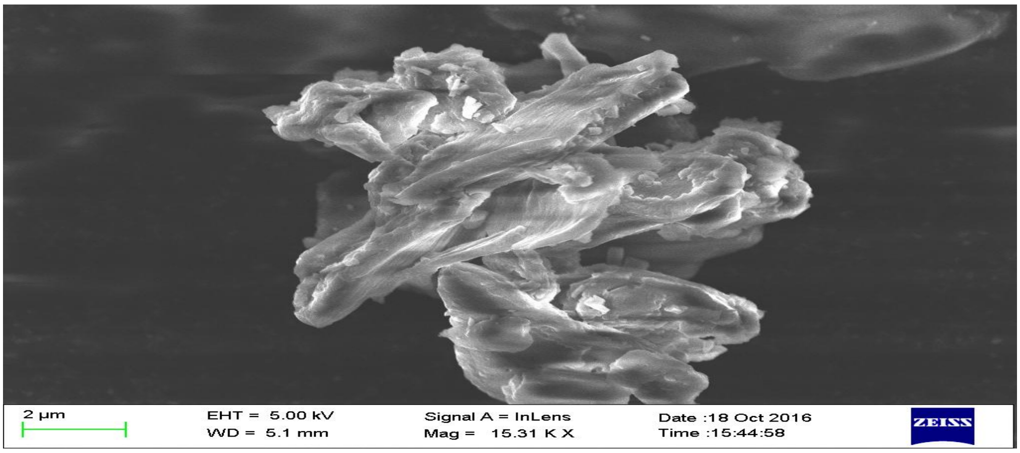

Fig.5: SEM image of activated carbon treated with Sulphuric acid

3.6. Powder X-ray diffraction study

The powder X-ray diffraction analysis of sample AC-H_2SO_4 investigated and displayed in Figure.6. In activated carbons, a broad peak due to reflections from the planes can be clearly seen. The broadness of the peak indicates the amorphous nature of the carbon sample [22, 23].

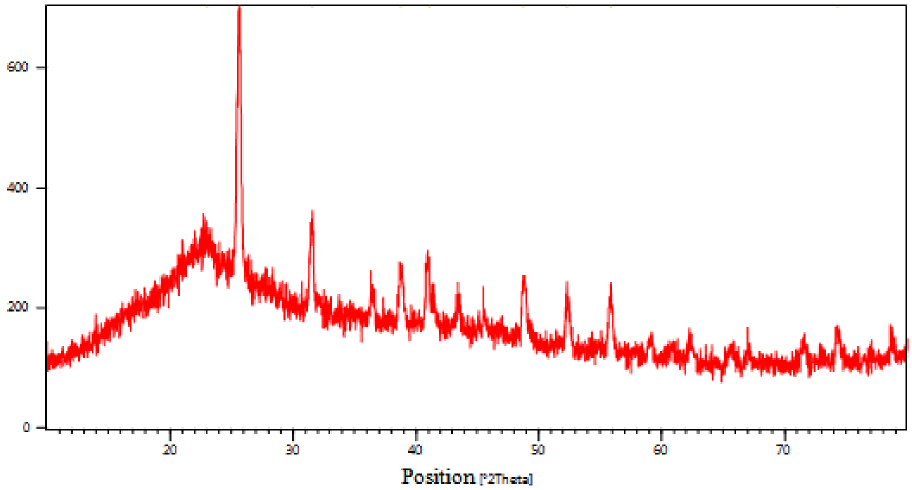

Fig.6: XRD pattern for prepared activated carbon

Figure shows XRD spectrum of the adsorbent. This spectrum clearly shows that the particle size is responsible for the broadening peaks in the XRD pattern [24, 25]. This spectrum also indicates that the presence of amorphous form of carbon which is disorderly stacked up by carbon rings.

3.7. Analysis of adsorption Kinetics

Kinetic models are used to study the rate of the adsorption process and factors affecting adsorption rate [26]. The following three kinetic models were used to explain the experimental data.1.Pseudo first order equation 2. Pseudo second order equation 3. Intra particular diffusion equation.

3.7.1. Pseudo-first order equation

The pseudo- first order equation of lagergen is expressed as follows.

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \quad (1)$$

Where, q_e is the amount of dye removed at equilibrium (mg/g)

q_t is the amount of dye removed at time t (mg/g)

k_1 is the pseudo- first order rate constant (min^{-1})

A plot of $\log(q_e - q_t)$ versus time (t) gives a straight line and is shown in figure.7. The values of k_1 and q_e can be calculated from the slope and intercept of the linear plot. The pseudo first order rate constant (k_1), correlation coefficients (R^2), experimental q_e values and calculated q_e values are illustrated in table.1. There is no close difference between the experimental q_e values and calculated q_e values [27, 28]. It can be concluded that the adsorption of malachite green dye on CPFAC did not follow the pseudo first order reaction.

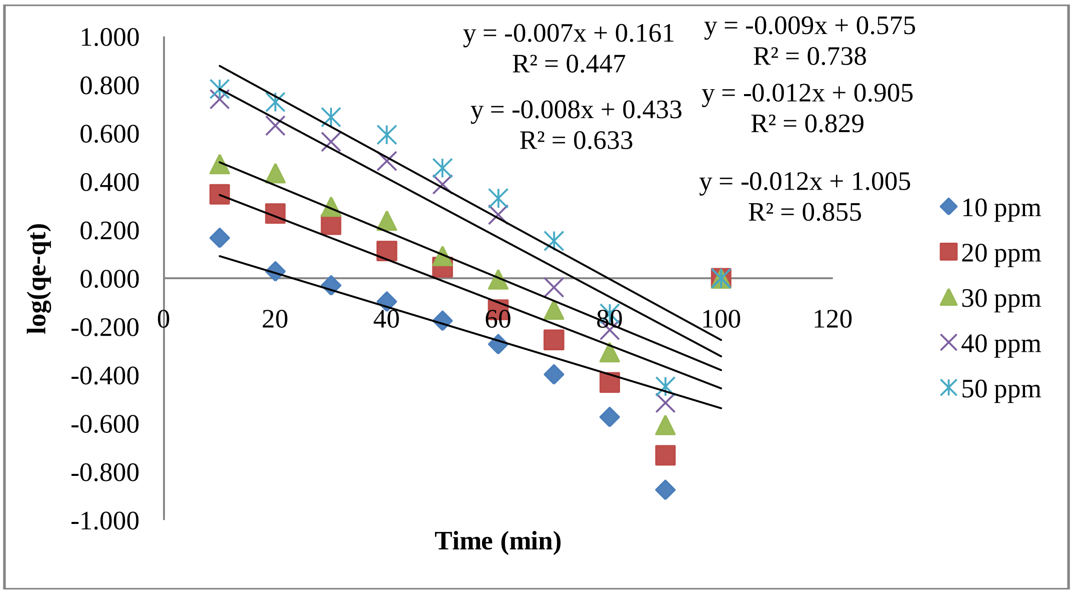

Fig.7: Pseudo-first order kinetics for adsorption of Malachite green onto prepared activated carbon at 30°C

3.7.2. Pseudo-second order equation

The pseudo second order equation is expressed as follows.

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

Where, q_e is the amount of dye removed at equilibrium (mg/g)
q_t is the amount of dye removed at time t (mg/g)
k_2 is the pseudo- second order rate constant (min^{-1})

If the pseudo second order kinetic model is follow, the plot of $\frac{t}{q_t}$versus t should give a linear relationship. k_2 and q_e values can be determined from the slope and intercept of the plot are presented in table.1.The figure.8 shows various initial dye concentration and temperature of pseudo second order. The experimental data (q_e), rate constants(k_2) and correlation coefficients (R^2) are shown in table.1. It can be concluded that R^2 values for the pseudo second order kinetic model is higher than that of R^2 values for the pseudo first order kinetic model for all initial malachite green concentrations [29, 30]. There is close difference between the experimental q_e values and calculated q_e values [28, 29]. It can be said that the adsorption of malachite green on CPFAC is well suitable for the pseudo second order kinetics model compared to the pseudo first order model.

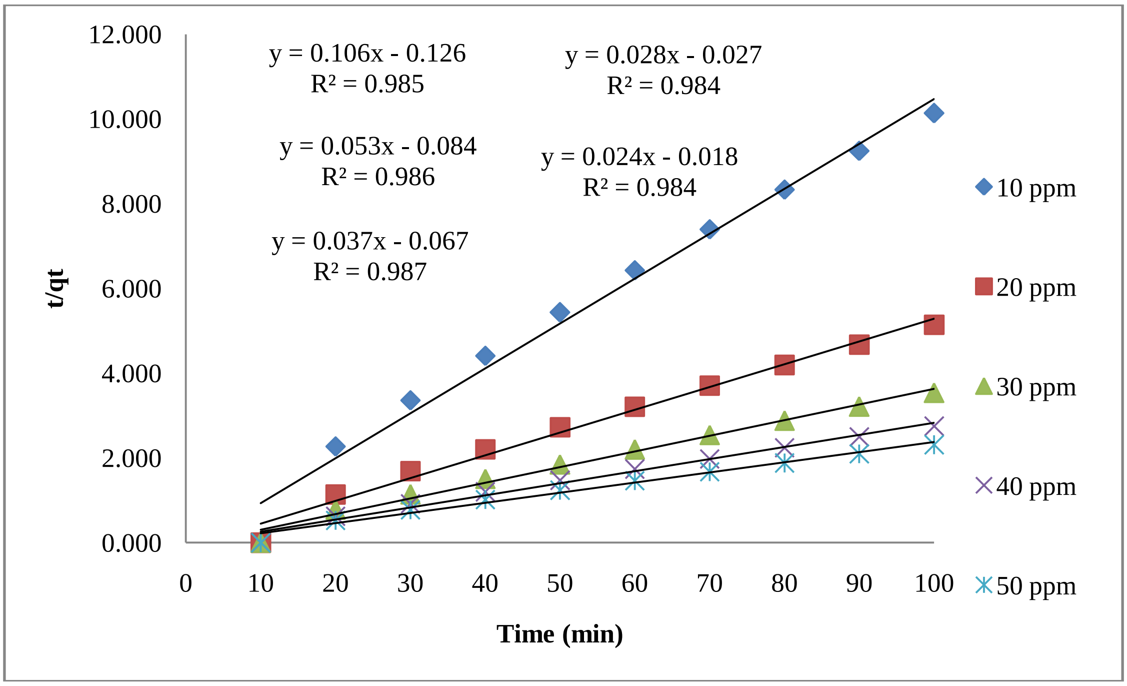

Fig.8: Pseudo- second order Kinetics for the adsorption of Malachite green onto prepared activated carbon at 30°C

Table 1: Pseudo first and pseudo second order kinetic parameters for different initial dye concentration

Intial Conc.(ppm)	Pseudo-first order kinetic model				Pseudo-second order kinetic model			
	q_{eexp} (ppm)	q_{ecal} (ppm)	k_1 (ppm)	R^2	q_{eexp} (ppm)	q_{ecal} (ppm)	k_2 (ppm)	R^2
10	9.87	1.448	0.0161	0.447	9.87	9.43	0.0891	0.985
20	19.44	2.710	0.0184	0.633	19.44	18.86	0.0334	0.986
30	28.26	3.758	0.0207	0.738	28.26	27.02	0.0204	0.987
40	36.34	8.035	0.0276	0.829	36.34	35.71	0.0290	0.984
50	43.21	10.115	0.0276	0.855	43.21	41.66	0.0320	0.984

3.7.3. Intra particle diffusion studies

Intra particle diffusion model is used for identifying the mechanism involved in the adsorption process [30]. Intraparticle diffusion (k_d) is given by weber morris and is expressed as follows

$$q_t = k_d t^{1/2} \qquad (3)$$

Where, q_t is the amount adsorbed (mgg^{-1}) at time t(min).
k_d is the rate constant of intraparticle diffusion($mgg^{-1}min^{1/2}$).

If the mechanism of adsorption process obey the intraparticle diffusion, the plot of amount adsorbed (q_t) versus time would be a linear relationship and is shown in figure.9.The rate constant of intraparticle diffusion (k_d) can be calculated from slope of the plot and the values are illustrated in table.2. The initial portion of the intraparticle diffusion plot indicates that a boundary layer effect while the second portion of the linear curve is due to intraparticle diffusion [29]. The linear portion of the plot did not pass through the origin indicates that intraparticle diffusion is not the only rate controlling step, and also other mechanism may control the adsorption rate [31]. The high values of correlation coefficient (R^2) indicated that the pore diffusion plays a significant role for the adsorption of Malachite green dye onto the activated carbon prepared from copper pod fruit. This result also revealed that the intra particle diffusion process is supposed to be rate-limiting step.

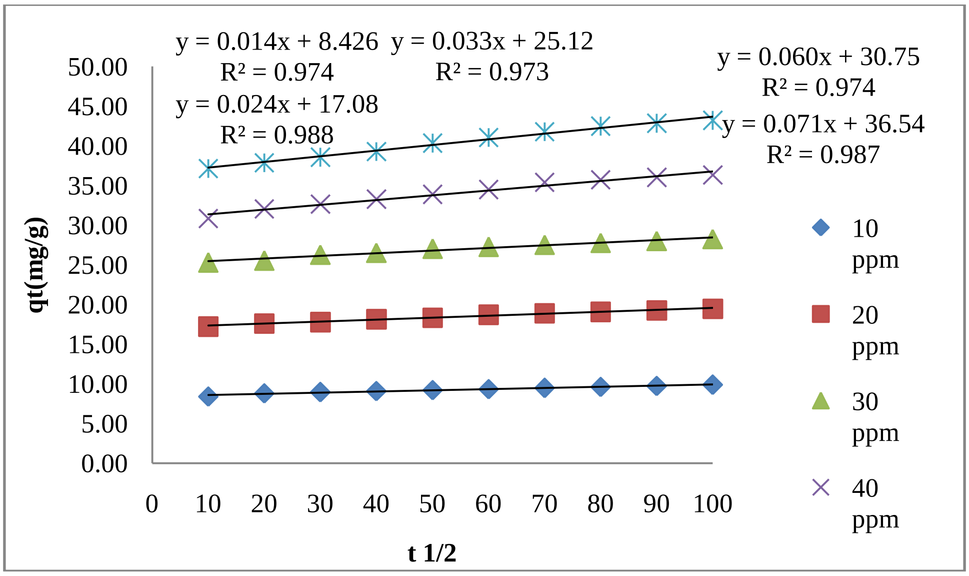

Fig.9: Intra particle diffusion for the adsorption of Malachite green onto prepared activated carbon at 30°C

Table 2: Intra Particle Diffusion model

Initial Concentration (ppm)	Intra Particle Diffusion model		
	C	K_d (mg/g.min)	R^2
10	8.42	0.014	0.974
20	17.08	0.024	0.988
30	25.12	0.033	0.973
40	30.75	0.060	0.974
50	36.54	0.071	0.987

3.7.4. Elovich Model

Elovich model can be expressed as follows

$$\frac{dq_t}{dt} = \alpha e^{-\beta q_t} \quad (4)$$

Where, α is the initial adsorption rate constant (mg/g min) and β is related to the extent of surface coverage and activation energy for chemisorption (g/mg).

Integrating this equation for the boundary conditions, expressed as follows

$$q_t = 1/\beta[\ln(\alpha\beta)] + 1/\beta \ln t \quad (5)$$

α and β values can be determined from the slope and intercept of plot qt Vs lnt. Figure 10 illustrates the Elovich isotherm model for the dye adsorption onto the adsorbents from which the relevant isotherm parameters are listed in table.3. It has been suggested that decrease in β value would increase the rate of the adsorption process [32]. There is linear plot with good correlation coefficient (R^2) values ranges from 0.973 to 0.988, which gave a close fit to the malachite green adsorption on CPFAC.

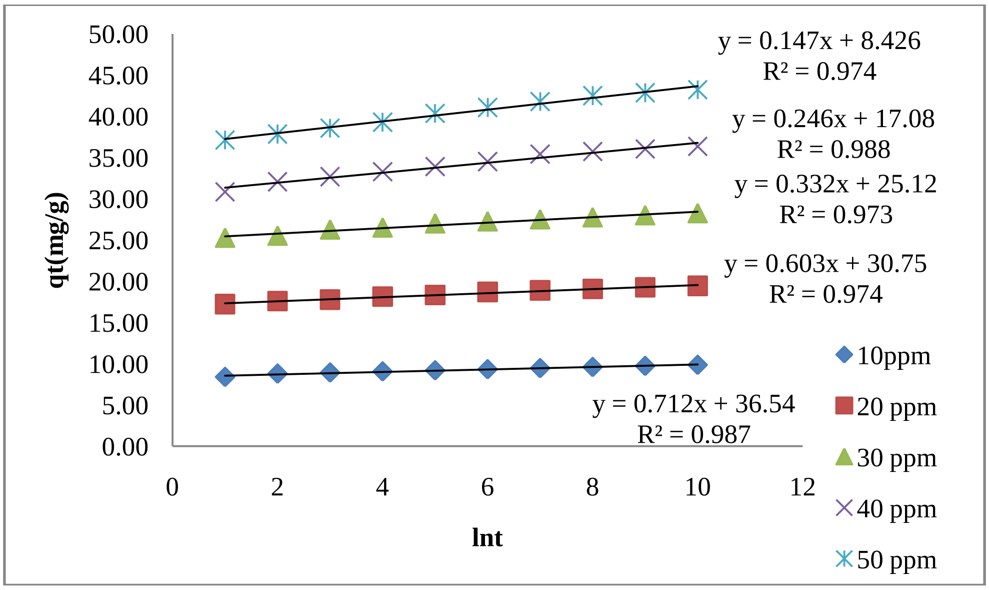

Fig.10: Elovich model for the adsorption of Malachite green onto prepared activated carbon at 30°C.

Table 3: Elovich model

Initial Concentration (ppm)	Elovich model	
	β	R^2
10	6.802	0.974
20	4.065	0.988
30	3.012	0.973
40	1.658	0.974
50	1.404	0.987

3.8. Adsorption isotherm

The adsorption isotherm describes the mechanism of the adsorption process between the adsorbate and the adsorbent [18, 20]. The Langmuir and Freundlich isotherm were analysed to study the adsorption isotherm.

3.8.1. Langmuir isotherm

The linear form of langmuir isotherm equation is expressed as follows.

$$\frac{C_e}{q_e} = \frac{1}{Q_0 b} + \frac{C_e}{Q_0} \qquad (6)$$

Where, q_e is the amount of dye adsorbed at equilibrium (mgg^{-1})

C_e is the concentration of dye solution at equilibrium (mgL^{-1})

Q_0 is Langmuir constant related to adsorption capacity (mgg-1)

b is Langmuir constant related to rate of adsorption(Lmg-1)

A plot of $\frac{C_e}{q_e}$ versus C_e gives a straight line with slope of $\frac{1}{Q_0}$ and intercept of $\frac{1}{Q_0}$ b and is shown in figure.11.

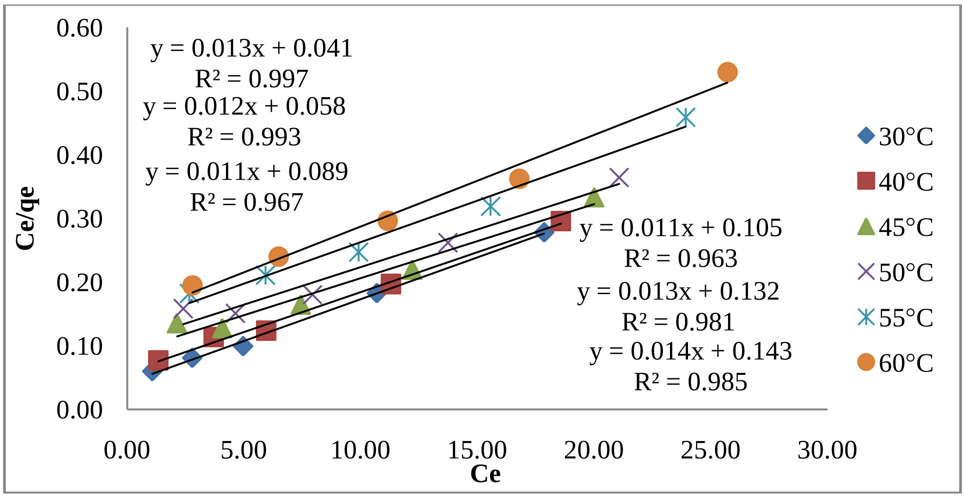

Fig.11: Langmuir isotherm for the adsorption of Malachite green onto prepared activated carbon.

Table 4: Langmuir isotherm parameters

Temp.(D_c)	Langmuir Constants		
	R^2	Q_0(mg/g)	b. (L/mg)
30	0.997	76.92	0.3170
40	0.993	83.33	0.2068
45	0.967	90.90	0.1235
50	0.963	90.90	0.1047
55	0.981	76.92	0.0984
60	0.985	71.42	0.0979

Q_0 and b values are decreases with increase in temperature. The experimental data indicates that the amount of malachite green dye adsorbed on adsorbents decreased with increasing temperature from 30°C to 60°C.The linearity of the plots and high correlation co-efficient (R^2) values indicated that the adsorption process obeys the Langmuir isotherm model with monolayer adsorption[33, 34]. This result clearly indicates that the adsorption of malachite green dye on activated carbon prepared from copper pod fruit takes place as monolayer adsorption on the surface of the adsorbent, homogenous in adsorption affinity and with constant adsorption energy.

The constant (Q_0) is a measure of maximum adsorption capacity of the adsorbent under the experimental conditions. The constant (b) decreased from 0.3170 to 0.0979 with the increased temperature of 30°C to 60°C. The result indicates that the maximum adsorption occurs at temperature 30°C.

The essential characteristics of Langmuir isotherm equation can be expressed in terms of dimensionless separation factor, R_L, which is defined by the following equation.

$$R_L = \frac{1}{1+bc_0} \qquad (7)$$

Where C_0 is the initial concentration of dye solution (mgL^{-1}) and b is the Langmuir constant. The value of R_L indicates that the shape of the adsorption isotherm to be either linear (R_L =1), favourable ($0<R_L<1$), unfavourable ($R_L>1$), or irreversible (R_L=0) [26, 28]. The values of R_L are shown in table .5. A low value of R_L favours adsorption [23]. The R_L values for the present experiment data fall between 0 and 1, which is an indication of the favourable adsorption process.

Table 5: R_L values at various initial dye concentration

Initial Dye	R_L Value					
Concentration (ppm)	30°C	40°C	45°C	50°C	55°C	60°C
10	0.2397	0.3258	0.4472	0.4883	0.5038	0.5053
20	0.1362	0.1946	0.2880	0.3230	0.3367	0.3380
30	0.0951	0.1387	0.2124	0.2413	0.2528	0.2539
40	0.0730	0.1078	0.1682	0.1926	0.2024	0.2034
50	0.0593	0.0881	0.1392	0.1603	0.1687	0.1696

3.8.2. Freundlich Isotherm

The Freundlich isotherm equation is expressed as follows

$$log\ q_e = log k_f + \left(\frac{1}{n}\right)\log C_e \qquad (8)$$

Where, q_e is the amount of dye adsorbed ($mg\ g^{-1}$)

C_e is the concentration of dye solution at equilibrium (mgL^{-1})

k_f is Freundlich constant related to the adsorption capacity of adsorbent

n is Freundlich constant related to the adsorption intensity

k_f and n values can be calculated from the slope of the straight line and is shown in figure.12.

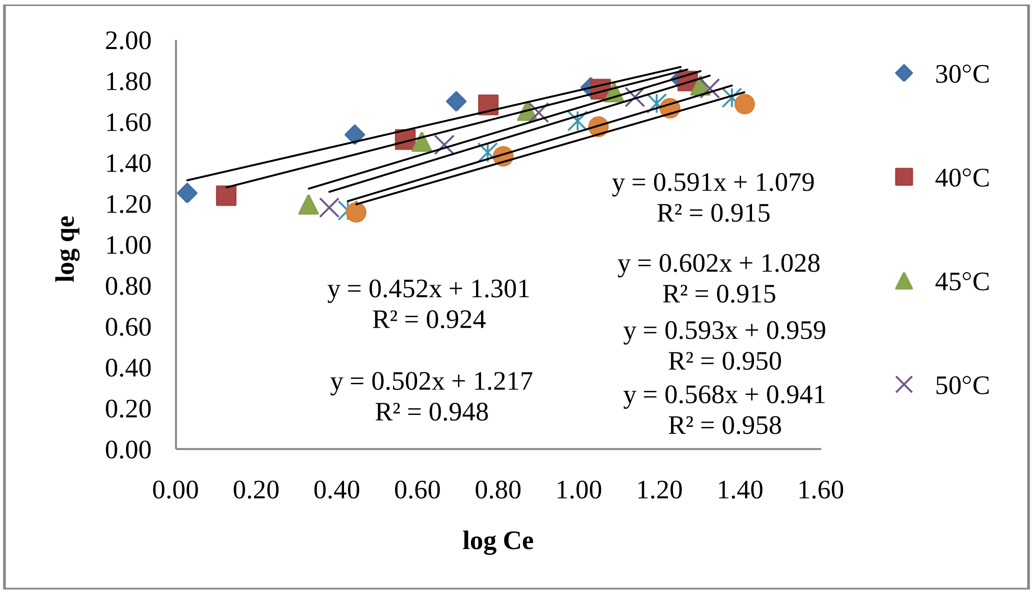

Fig.12: Freundlich isotherm for the adsorption of Malachite green onto prepared activated carbon.

Table 6: Freundlich isotherm parameters

Temp.(0 ^{0}C)	Statistical Parameters / Constants		
	R^2	n	K_f (mg/L)
30^{0}C	0.924	2.2123	19.998
40^{0}C	0.948	1.9920	16.481
45^{0}C	0.915	1.6920	11.994
50^{0}C	0.915	1.6611	10.665
55^{0}C	0.950	1.6863	9.099
60^{0}C	0.958	1.7605	8.729

The value of R^2 is used to measure goodness of fit of experimental results on the adsorption isotherm models [35, 30]. According to the adsorption isotherm results, the Langmuir isotherm (R^2= 0.963 to 0.997) is more favourable than Freundlich isotherm (R^2= 0.924 to 0.958). The adsorption process is said to be favourable [33, 21] when the value of n satisfies the condition n<1, otherwise it is unfavourable. While the n values in Table.6 for adsorption of malachite green dye on CPFAC are situated outside the range of 0 to 1 indicating unfavourable adsorption process.

3.8.3 Tempkin isotherm

The linear form of Tempkin isotherm equation is given as follows

$$q_e = B\,(lnA + lnC_e) \qquad (8)$$

Where, B is the Tempkin constant related to heat of adsorption

A is the equilibrium binding constant (mg/l)

The values of the Tempkin constants A and B can be determined from the intercept and slope of the linear plot of lnC_e versus q_e and is shown in Figure.13. The correlation coefficients (R^2) values are listed in Table.7.The result shows that the values of R^2 are located in the range of 0.976 to 0.985, which indicate a better fits to the malachite green adsorption onto CPFAC.

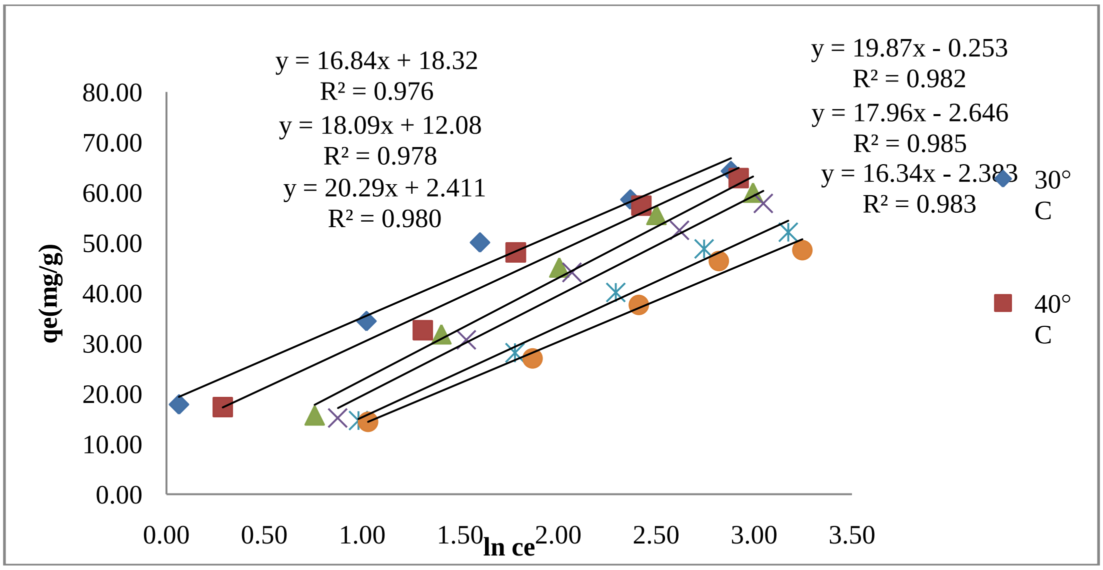

Fig.13: Tempkin isotherm for the adsorption of Malachite green onto prepared activated carbon.

Table 7: Tempkin isotherm parameters

Temp.(0 ^{0}C)	Statistical Parameters / Constants		
	R^2	B	A
30^{0}C	0.976	16.84	2.5069
40^{0}C	0.978	18.09	4.4694
45^{0}C	0.980	18.29	0.0577
50^{0}C	0.982	19.87	0.0417
55^{0}C	0.985	17.96	0.0011
60^{0}C	0.983	16.34	0.0011

3.8.4. Dubinin – Radushkevich isotherm

The linear form of the Dubinin-Radushkevich isotherm equation can be expressed as follows

$$\ln qe = \ln Xm - \beta E^2 \qquad (9)$$

Where, Xm is the theoretical saturation capacity (mg/g), β is a constant related to mean free energy of adsorption per mole of the adsorbate (mol^2/J^2) and E^2 is the Polanyi potential. The values of E, Xm and β can be calculated from the slope and intercept of the plot of E^2 versus ln qe gives a straight line and is shown in figure.14.The correlation coefficients (R^2), E, Xm and β values are listed in Table.8.

The mean free energy of adsorption E is determined from β using the following equation

$$E = 1/(2\beta)^{1/2} \qquad (10)$$

The activation energy of adsorption decreases with increase of temperature from 30 to 60 ^{0}C. The values correlation coefficients (R^2) are in the range of 0.911 to 0.969, revealing that the experimental data fitted well with the Dubinin-Radushkevich isotherm model. Based on this energy of activation one can predict whether an adsorption is physisorption or chemisorptions [36]. If the energy of activation is, <8 kJ mol^{-1}, the adsorption is physisorption. If the energy of activation is >8 kJ mol^{-1}, the adsorption is chemisorptions [37, 38]. From table.8, it can be observed that the obtained the obtained values of mean free energy, E, are limited within the range of 34.921 to 48.795 kJ mol^{-1}. Based on these data, it can be concluded that the effect of chemical adsorption will play a dominating role in the adsorption process of malachite green dye onto the adsorbents.

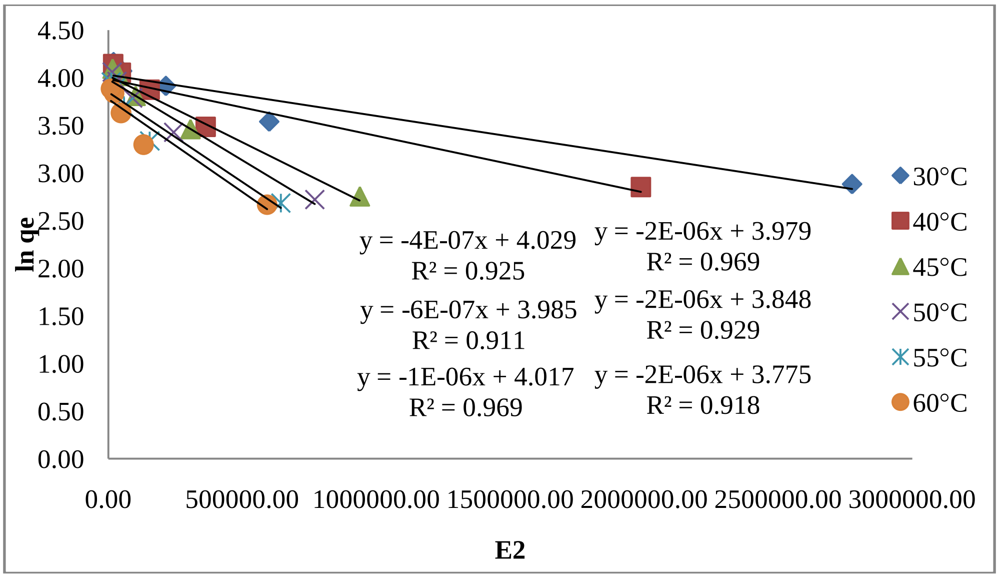

Fig.14: Dubinin -Radushkevich isotherm model for the adsorption of Malachite green onto prepared activated carbon.

Table 8: Dubinin -Radushkevich isotherm parameters

Temp.(0 ^{0}C)	Statistical Parameters / Constants		
	R^2	Xm (mg/g)	E (KJ/mol)
30^{0}C	0.925	56.163	34.921
40^{0}C	0.891	53.746	28.629
45^{0}C	0.969	55.494	67.419
50^{0}C	0.969	53.425	48.795
55^{0}C	0.929	46.866	48.795
60^{0}C	0.918	43.567	48.795

IV. Conclusion

The adsorption data for the uptake of malachite green dye increased with increase in initial concentration, contact time and adsorbent dosages. The adsorption process is an exothermic. The optimum temperature was found to be 30^{0}C. The effect of pH on the dye solution is most important parameter in adsorption process. The maximum dye adsorption occurred at a pH 7.0. The adsorption isotherm data was well described with Langmuir isotherm model, Tempkin and Dubinin - Radushkevich isotherm better than Freundlich isotherm model. R_L values indicate favorable adsorption process. The adsorption processes for malachite green dyes were found to follow the pseudo-second order kinetic model, intraparticle diffusion model and Elovich model. The SEM study observed that difference in surface morphology of adsorbent. Finally, the results clearly indicated that copper pod fruit activated carbon(CPFAC) could be used as an alternative to highly efficient low cost and abundant materials for removal of malachite green dye from contaminated aqueous solutions.

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