



## Adsorption of Congo Red Dye onto Activated Carbon from Periwinkle Shell

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Manuscript History  
Received: 19/04/2020  
Revised: 29/05/2020  
Accepted: 02/06/2020  
Published: 10/06/2020

**Abstract:** Adsorption of Congo red dye onto activated carbon from periwinkle shell was carried out to see if the activated carbon from Periwinkle shell will provide a good alternative for the commercial activated carbon in the adsorption of Congo red dye from polluted water. Periwinkle shells carbonized at 400°C and activated with 0.1M HCl at 800°C in a furnace was used as adsorbent to remove Congo red dye from aqueous solution. The effect of the variables: contact time, temperature and initial Congo red concentration were studied through single-factor classical method. The contact time range, 30 – 180min, temperature range, 30 – 55°C and concentration range, 25 – 150mg/L were used to study the adsorption efficiency, kinetic and thermodynamic studies of the process. The results show that equilibrium is established after 120 minutes and that the percentage adsorption is highest at initial concentration of 25mg/L. Again, the adsorption kinetic followed pseudo-second order reaction, having rate constant,  $(K_2) = 0.3661 \text{ g/mg.min}$ , correlation coefficient,  $(R^2) = 0.9999$  and adsorption capacity,  $(A_{de}) = 2.3585 \text{ mg/g}$ . The thermodynamic parameters obtained are: free energy  $(\Delta G) = -6.71$  and  $-8.20 \text{ KJ/mol}$  at 303 and 328K respectively, enthalpy  $(\Delta H) = 11.52 \text{ KJ/mol}$  and entropy,  $(\Delta S) = 60.04 \text{ J/mol.K}$ . The surface area of the adsorbent was obtained as  $123.42 \text{ m}^2/\text{g}$ . These results show that activated carbon from periwinkle shell is a viable low-cost adsorbent for the removal of Congo red dye from aqueous solution.

**Key words:** Adsorption, Congo red, Kinetic, Thermodynamics, Periwinkle Shell

### INTRODUCTION

Most wastewaters from dyeing industries are released into streams or rivers without much treatment, because the conventional treatment methods are not cost effective (Belhachemi and Addoun, 2011). Dyes exhibit great structural difference, thus they are not uniformly degraded by microbial attack. They are however degraded under anaerobic conditions. Their linkage can be reduced to form aromatic amines which are colorless but may be toxic and carcinogenic (Yaneva and Georgieva, 2012). Adsorption involving the use activated carbon has long been regarded as a conventional treatment process employed for the treatment of wastewater with

multitude of pollutants. However, the high cost of commercial activated carbon has restricted its use, particularly in developing countries (Iryani *et al.*, 2017).

Many investigators have studied the feasibility of using inexpensive alternative materials like pearl millet husk, date pits, saw dust buffing dust of leather industry, coir pith, crude oil residue, tropical grass, olive stone and almond shells, pine bark, wool waste, coconut shell etc., as carbonaceous precursors for the removal of dyes from water and wastewater (Sekaran *et al.*, 1995). The discharge of highly colored effluents into natural water bodies is not only aesthetically displeasing, but it also impedes light penetration, thus upsetting biological processes within a stream (Coruh *et al.*, 2015). In addition, many dyes are toxic to some organisms causing direct destruction of aquatic communities. Some dyes can cause allergic dermatitis, skin irritation, cancer and mutation in man. Recent estimates indicate that, approximately, 12% of synthetic textile dyes used each year is lost during manufacture and processing operation and 20% of these dyes enter the environment through effluents that result from the treatment of residual industrial waters (Selvarani, 2000, Hema and Arivoli, 2008, Hakan *et al.*, 2008). In this research, the ability of periwinkle shell carbon (which currently has no known usefulness) to remove Congo red dye by adsorption was studied. The adsorption capacity of the dye was examined and the adsorption efficiency at variable contact time, concentration and temperature was determined. Adsorption isotherm, Pseudo order and Van't Hoff's models were used to fit the experimental data in order to obtain the adsorption capacity, kinetic and the thermodynamic parameters.

## 1.1 Theory

### A. Surface area

The surface area of the adsorbent influences its adsorption; the greater surface area, the greater number of surface sites which the adsorbates adsorption may occur. Surface area ( $S_{\text{area}}$ ) values were calculated from the experimental adsorption isotherm over a relative pressure range of 0.1 to 0.3 (Hill, 1997), using the standard BET (Brunauer, Emmett and Teller) method (Brunauer *et al.*, 1938). The BET equation is given as:

$$\frac{P}{V(P^0 - P)} = \frac{1}{V_m C} + \frac{(C-1)}{V_m C} \left(\frac{P}{P^0}\right) \quad (1)$$

$$\frac{x}{V(1-x)} = \frac{1}{V_m C} + \left(\frac{C-1}{V_m C}\right)x \quad (2)$$

$x$  = normalized pressure =  $p/p_0$

$$V_m = \frac{1}{\text{slope} + \text{Intercept}}$$

Where,  $V$  is the volume adsorbate actually adsorbed ( $\text{cm}^3/\text{g}$ ),  $V_m$  is the volume of monolayer capacity ( $\text{cm}^3/\text{g}$ ), and the term  $C$ , the BET constant, is related to the energy of adsorption in the first adsorbed layer and its value is an indication of the magnitude of the adsorbent-adsorbate interactions (Khattri and Singh, 1999, Hill, 1997).

$$S_{\text{area}} = \frac{V_m x A x N x 10^{-20}}{M} \quad (3)$$

Where  $S_{\text{area}}$  is the surface area in  $\text{m}^2/\text{g}$ ;  $A$  is the occupied specific surface area of one molecule of adsorbent (Congo red dye) =  $233\text{A}^2$  (National Centre for biotechnology information);  $N$  is Avogadro's number  $6.02 \times 10^{23} \text{ mol}^{-1}$ ; and  $M$  is the molecular weight of adsorbent (congo red dye),  $696.65\text{g}/\text{mol}^{-1}$  (Sekaran *et al.*, 1995).

### B Adsorption kinetics

The pseudo first order and second order kinetic models were tested at different concentrations in this study to determine which model is in good agreement with experiment  $q_e$  (adsorption capacity) value, thus suggesting which model the sorption system follows:

#### Pseudo First Order Equation

The Lagergren model proposed in 1898, assumes a first order adsorption kinetics and its integration can be represented by the equation (Gottipati, 2012).

$$\frac{dA_{dt}}{dt} = K_1(A_{de} - A_{dt}) \quad (4)$$

$$\text{Log}(A_{de} - A_{dt}) = \text{Log}(A_{de}) - \frac{K_1}{2.303} t \quad (5)$$

The values of  $\text{Log}(A_{de} - A_{dt})$  were linearly correlated with  $t$ . The plot of  $\text{Log}(A_{de} - A_{dt})$  versus  $t$  will give a linear relationship from which  $K_1$  and  $q_e$  can be determined from the slope and intercept of the plot.

#### The Pseudo Second-Order Equation

The pseudo-second-order adsorption kinetic rates equation is expressed as

$$\frac{dA_{dt}}{dt} = K_2(A_{de} - A_{dt})^2 \quad (6)$$

The linearized integral form is expressed as

$$\frac{t}{A_{dt}} = \frac{1}{K_2 A_{de}^2} + \frac{1}{A_{de}} t \quad (7)$$

The plot of  $(t/A_{dt})$  and  $t$  of equation (7), should give a linear relationship from which  $A_{de}$  and  $K_2$  can be determined from the slope and intercept of the plot, respectively (Gottipati, 2012).

#### Kinetic Parameters of Activation

From the Van't Hoff equation  $\frac{d(\ln k)}{dT} = \frac{\Delta H}{RT^2}$

For isobaric (constant pressure) and isochoric (constant volume) conditions,

$k = \frac{k_2}{k_1}$  = chemical equilibrium constant,  $k_1$  and  $k_2$  are rate constants of forward and reverse reactions,  $H$  = Heat of reaction,  $R$  = Universal gas constant,  $T$  = Absolute temperature

$$\frac{d \ln K}{dT} = \frac{E}{RT^2} \quad (8)$$

Integration of equation (8) assuming  $E$  to be constant yields

$$\ln K = \frac{E_a}{RT} + \ln A \quad (9)$$

Where  $k$  is the rate constant,  $A$  is a frequency factor,  $R$  is the universal gas constant (8.314 J.K<sup>-1</sup>.mol<sup>-1</sup>) and  $T$  is the absolute temperature. The value of  $E$  is calculated from the slope of plotting  $\ln K$  versus  $1/T$ , and  $A$  (min<sup>-1</sup>) is determined from the intercept (Namasivayam and Kavitha, 2012).

#### C. Thermodynamic studies

The basic thermodynamic parameters: change in enthalpy of adsorption ( $\Delta H$ ), change in Gibb's free energy of adsorption ( $\Delta G$ ) and change in entropy of adsorption ( $\Delta S$ ), is important as it allows to estimate if the process is favorable or not, to assess the spontaneity of the system and to ascertain the exothermic or endothermic nature of the process (Zhang et al., 2011).

$$\Delta G = -RT \ln K_d \quad (10)$$

$$K_d = \frac{A_{de}}{C_e} \quad (11)$$

$$\ln K_d = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \quad (12)$$

Where  $K_d$  is the distribution coefficient for the adsorption,  $A_{de}$  is the amount of dye (mg/l) adsorbed on the adsorbent at equilibrium,  $C_e$  is the equilibrium concentration (mg/l) of the dye in solution,  $T$  is the absolute temperature,  $R$  is gas constant, respectively. The  $\Delta H$  and  $\Delta S$  values obtained from the slope and intercept of Van't Hoff plots are presented in Table 2. An adsorption process is considered physical if  $\Delta H < 84 \text{ kJ mol}^{-1}$  and chemical when  $\Delta H$  lies between 84 and 420  $\text{kJ mol}^{-1}$  (Zhang et al., 2011).

## MATERIALS AND METHODS

### A. Preparation of Adsorbents

The periwinkle shells were picked from the environment in Elele, Rivers State, Nigeria. They were washed with tap and dried in the open air. The dried periwinkle shells were carbonized in a furnace (SX-5-12) at 673K for 3 hours and the charred periwinkle shells were allowed to cool to room temperature, crushed and sieved with 150 – 600 $\mu\text{m}$  standard sieve. 100 gram of the ground carbonized periwinkle shell was weighed and added to 300 ml of 0.1M HCl solution, thoroughly mixed and heated until it formed slurry. The slurry was transferred to a crucible and heated in a furnace (SX-5-12) at 1073K for 3 hours, thereafter it was allowed to cool to room temperature, washed with de-ionized water and dried in an oven (MINO/75/F/DIG) at 383K for 2 hours (Gamus and Okpeku, 2015). Liquid nitrogen under cryogenic condition (temperature 77.4K) was introduced into the activated carbon, using an automatic adsorption unit, Autosorb – 1 (Quantachrome). The sample was degassed at 473K for 5 h before it was used so as to remove any adsorbed moisture or other impurities bound to the surface of the sample (Gottipati, 2012).

### B. Characterization of the Activated Carbon

#### i. pH

4.0 grams of the activated carbon was weighed out using an electronic weighing balance. The weighed activated carbon was washed for 10 minutes with 60 ml distilled water and filtered using a Whatmann filter paper, then, the pH of filtrate was measured using a pH meter, until 7.0 was reached.

#### ii. Pore Volume and Porosity

4.0g of the Periwinkle shell activated carbon was transferred into a measuring cylinder of (10 ml) and volume of the particles was measured. This sample was transferred to a beaker containing 20 ml of distilled water and boiled for 10 minutes (to displace air in the sample). The content was dried superficially and weighed. Increase in weight of the activated carbon was divided by the density of water to give the pore volume for the activated carbon. Porosity was obtained by dividing the pore volume of the particle with the total volume of the particle.

#### iii. Ash Content

4.0 g of the Periwinkle shell activated carbon was placed into a crucible of known weight, the weight of the crucible with the sample was also taken and then fed into a furnace set at 900°C for 3 hours. The sample was withdrawn, cooled to room temperature and reweighed. Ash content was calculated between the differences in weight.

*iv. Determination of Moisture Content*

4.0 g of Periwinkle shell activated carbon was dried using a Moisture Analyzer at 150°C, until weight of sample was constant. The moisture content was determined using the equation:

$$X_o = \frac{W_1 - W_2}{W_1} \times 100 \quad (13)$$

Where  $X_o$  = moisture content on wet basis,  $W_1$  = initial weight of sample in grams,  $W_2$  = final weight of sample (in grams) after drying.

*v. Bulk Density*

10.0 g of the Periwinkle shell activated carbon was placed in a 25ml measuring cylinder with distilled water. The volume of the water displaced was recorded. The bulk density was obtained by dividing the mass of the Periwinkle Shell activated carbon with the volume of water displaced.

*C. Preparation of Adsorbate*

The Congo red dye used is of laboratory grade (KEM LIGHT, India), with molecular weight 696.65g/mol. The solution was prepared with de-ionized water from Ion-exchange (Indian) Ltd, Eleme, Port Harcourt, Nigeria. 1.7g of the dye was accurately weighed and placed in 50ml pycnometer, 49.9ml of de-ionized water was added to prepare 50mg/l standard solution of known volume of the solute and solvent. Further dilute solutions were prepared by successive dilutions with de-ionized water.

*D. Adsorption Experiment*

The experiment was carried out using 1000mg of the activated carbon with 50ml of 50mg/l concentration of the Congo red dye at 303K in a temperature controlled water bath (DK - 420) with shaking mechanism. The samples were withdrawn after 30, 60, 90, 120, 150 and 180minutes respectively and filtered. The concentration of the filtrate was measured with a UV spectrophotometer (2OD) at 496nm. Using the equilibrium contact time obtained, 1000mg of the activated carbon was mixed with 50ml of the solute with desired concentrations (25, 50, 75, 100, 125 and 150mg/l) at 303K in a temperature controlled water bath (DK - 420). The samples were withdrawn after 120 minutes and the dye solutions were separated from the adsorbent by filtration. The concentration of the filtrate was measured with a UV spectrophotometer (2OD) at 496nm. Again 1000mg of the activated carbon mixed with 50ml of 50mg/L concentration of congo red dye solution, first at 303 then 308, 313, 318, 323 and 328K in a temperature controlled water bath was also carried out. The samples were withdrawn after 120minutes respectively filtered and the concentration measured. The amount of Congo red dye adsorbed onto the periwinkle shell adsorbent at time t and at equilibrium were calculated with the following equation:

$$A_{de} = \frac{(C_o - C_e)V}{X} \quad (14)$$

Where  $C_o$  (mg/l) and  $C_e$  (mg/l) are the initial and equilibrium concentration of the dyes,  $v$  (l) is the volume of solution,  $X$  (g) is the weight of adsorbent in one container.

## RESULTS AND DISCUSSION

### A. Characterization of the Adsorbent

The activated carbon gives bulk density of 2.8g/ml, its pore volume was obtained as 1.6ml and its porosity as 0.29. The pH of the activated carbon was obtained as 7.0, its moisture content was obtained as 1.2%, while the ash content was obtained as 1.8g.

#### Surface Area

From Equation 2;

Volume of Congo red dye dissolved in the 50mg/ml initial concentration.

From the adsorbate preparation, 49.9ml of de-ionized water was added to the Congo red dye in a 50ml pycnometer.

Volume of Congo red dissolved in the initial concentration = (50 - 49.9) ml = 0.1ml

The amount of Congo red adsorbed at 120 min, from concentration of 50mg/ml, volume of 50ml at 30°C = initial Concentration of Congo red - final concentration of Congo red after adsorption

= (50 - 3.26) mg/ml = 46.74mg/ml

Therefore,

% amount of Congo red adsorbed =  $0.9348 \times 100 = 93.48\%$

Volume of Congo red adsorbed (V) = Volume of Congo red used X %amount of Congo red adsorbed

$V = \frac{0.1 \times 93.48}{100} = 0.09348\text{ml}$

Plotting  $\frac{x}{V(1-x)}$  against x gives Fig.1

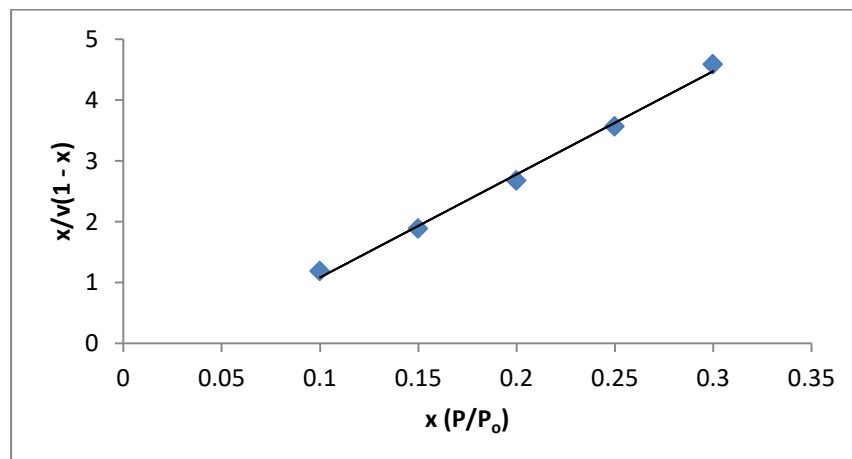


Fig.1 Brunauer Emmett Teller Graph

Slope = 16.93, Intercept = -0.607

Surface area ( $S_T$ ) = 123.42m<sup>2</sup>/g

The surface area for this periwinkle shell activated carbon was obtained as 123.42m<sup>2</sup>/g. Iryani et al, (2017), obtained a surface area of 333m<sup>2</sup>/g for ZSM-5 synthesized from Bangka Kaolin.

Belhachemi and Addoun (2011), obtained  $1069\text{m}^2/\text{g}$  for Date pith,  $950\text{m}^2/\text{g}$  for Date pith oxidized with  $\text{HNO}_3$  and  $1160\text{m}^2/\text{g}$  for Date pith thermally treated with nitrogen, but the surface area for commercial activated carbon is  $492\text{m}^2/\text{g}$  (Yaneva and Geogieva, 2012). The larger the surface area value, the better the adsorption.

#### B. Effect of contact time on the adsorption

The experimental results of adsorption of Congo red onto periwinkle shell activated carbon at  $50\text{mg}/\text{l}$  with variable contact time and constant temperature is presented in Fig. 2. It shows that percentage adsorption increases with time and reached equilibrium at 120 minutes. The kinetic data for the second-order adsorption is more satisfactory than that of the pseudo first order, so the pseudo second order model was adopted with correlation coefficient ( $R^2$ ) = 0.9999, Fig.3. Therefore, the adsorption can be approximated more appropriately by pseudo-second order kinetic model.

In Fig3, there is no significant increase in the adsorption of Congo red with contact time at temperatures of 303, 313 and 323K. Therefore, adsorption of Congo red onto Periwinkle shell activated carbon with variable contact time is independent of temperature.

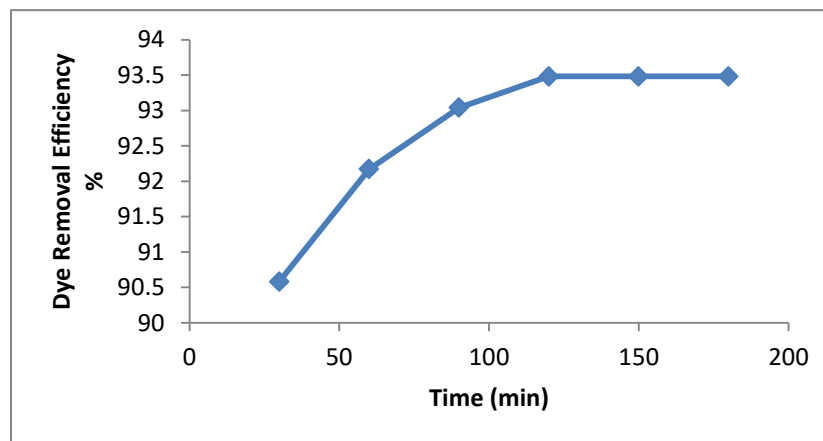


Fig. 2. Effect of contact time on dye removal

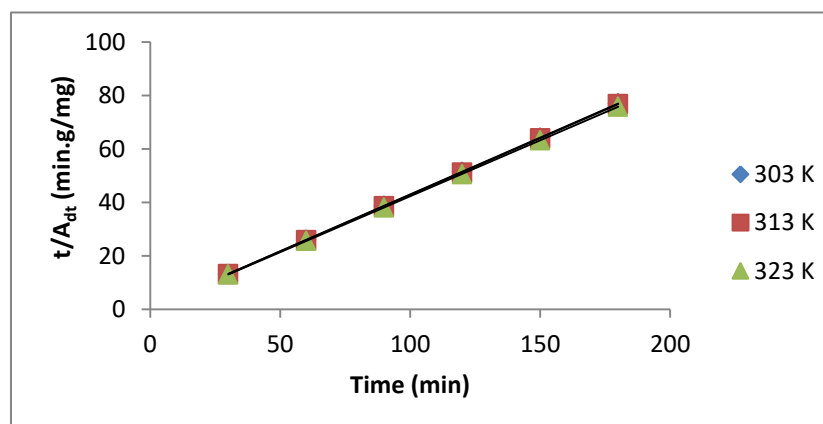


Fig. 3. Pseudo-second order reaction

Also, Fig.4 shows the dependency of the rate constant on temperature at 303, 313 and 323K, while values of the activation energy and frequency factor were obtained from the plot using eq (9). The parameters are presented in Table 1.

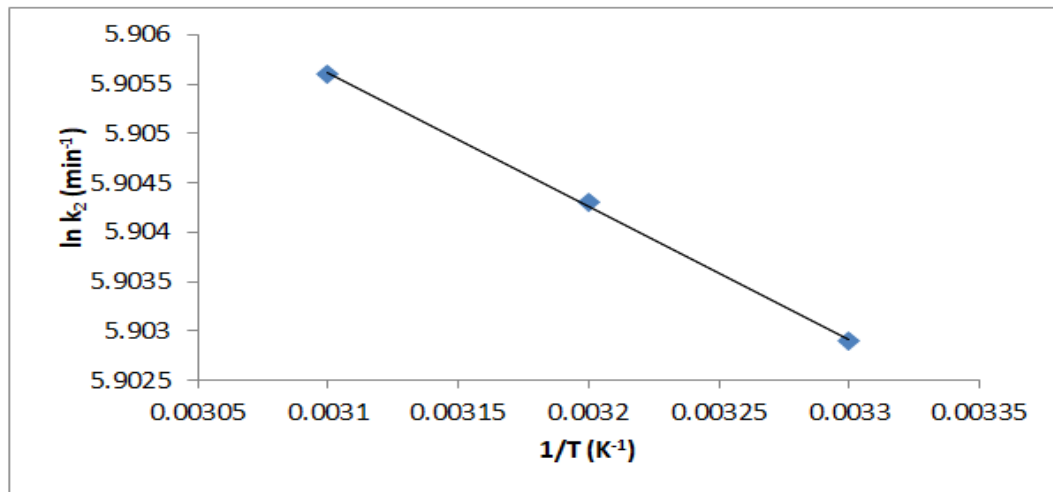


Fig. 4. Temperature Dependency of Rate Constant

Table 1. Kinetic and Activation energy parameters

$K_2$ (g/mg $R^2$ .min)	$A_{de}$ (mg/g)	$K_2$ (g/mg.min) $A_{de}$ (mg/g)	$R^2$ E	$K_2$ (g/mg/min) C	$A_{de}$ (mg/g)	$R^2$
303K		313K		323		
0.3661	2.359	0.3666	2.3641	0.3641	2.3923	0.999
0.999		0.999	0.112	1.783		

### C. Effect of Initial Concentration on Dye Removal

The effect of Congo red concentration in the range of 25- 150 mg/l on the adsorption was investigated under the specified conditions. Increasing the dye concentration led to percentage decrease in the Congo red adsorption. Adsorption isotherm indicates how adsorbed molecules are distributed between the liquid phase and the solid phase when the adsorption process occur at a fixed temperature. The Langmuir and Freundlich isotherms give correlation coefficients of 0.942 and 0.995 respectively, which are both good fit for the adsorption and this agrees with the trend which reported that Freundlich isotherm may be the best fit for that particular adsorption (Hema and Arivoli, 2008).



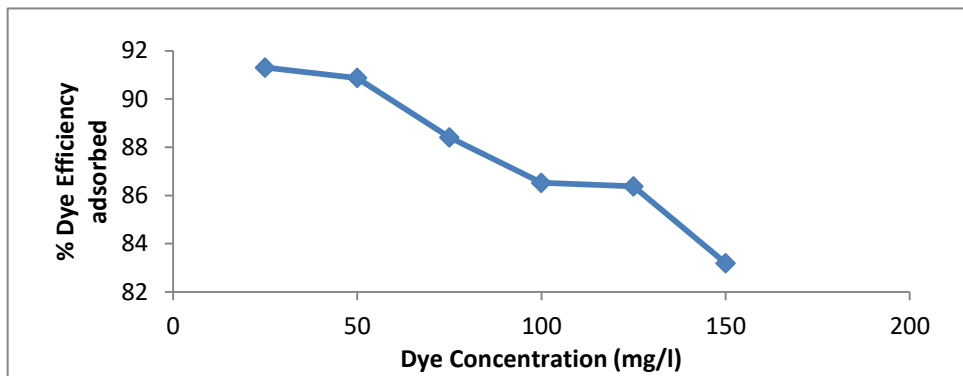


Fig.5. Percentage Adsorption with Concentration

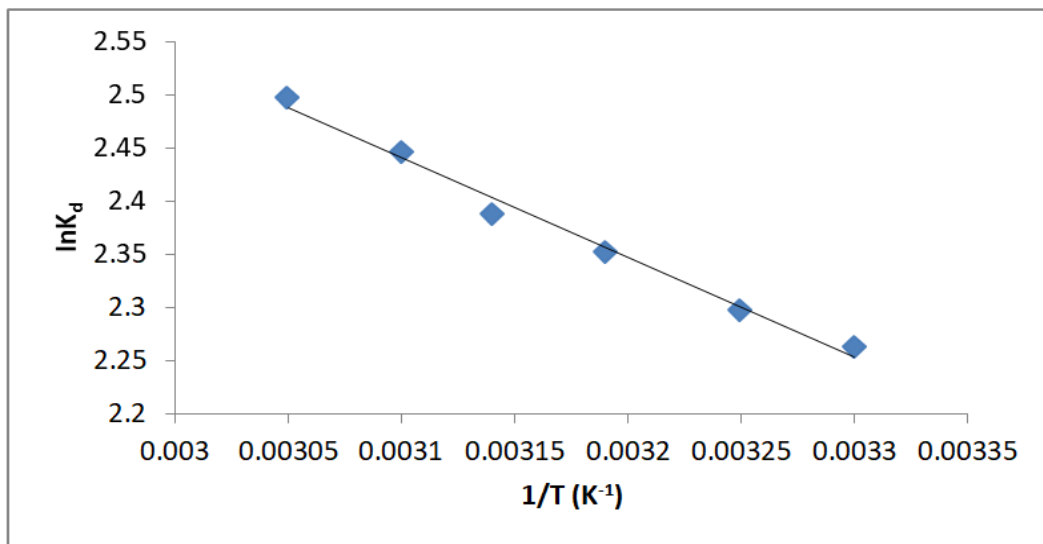


Fig. 6. Plot of Van't Hoff model

#### D. Effect of Temperature

The adsorption capacity of the activated carbon significant increase as the temperature of the system increases from 303-328K. The obtained thermodynamic parameters are given in Table 2. Using the Van't Hoff's equation, the plot of  $\ln K_d$  versus  $1/T$  is shown in Fig 6. The values of  $\Delta H$  and  $\Delta S$  of Congo red adsorption were calculated by fitting the experimental data to Eq. (12). The values of  $\Delta G$  were also obtained by using Eq. (10). The enthalpy change ( $\Delta H$ ) for the adsorption is positive, indicating that the process is endothermic in nature.

Table 2. Thermodynamic and Kinetic Parameters of the Adsorption

Temp(K) (KJ/mol)	$\Delta G$	$\Delta H$ (KJ/mol)	$\Delta S$ (J/mol.K)
303	-6.71	11.52	60.04
308	-6.94		
313	-7.26		
318	-7.52		
323	-7.88		
328	-8.20		

Table 3. Adsorption Capacities of some Adsorbents for Congo Red Removal

Adsorbent	Adsorption capacity (mg/g)	Temp(K)	Conc. (mg/l)	Reference
Biogas waste slurry	9.50			Grabowska
Waste red mud	4.05	&Gryglwixz, 2007		
Date pith	36.0			Grabowska
Date pith oxidized with HNO <sub>3</sub>	33.2	&Gryglwixz, 2007		Belhachami &
Date pith thermally with nitrogen	45.2	Addoun, 2011		
Coir pith	4.546	298		Belhachami &
Kaolin	6.53	Addoun, 2011		
Commercial activated carbon	48.7-183.1	298		Belhachami &
Cetyltrimethylammonium bormide modified kaolin	19.82 0.451	Addoun, 2011 308		Namasivayam
AgNPs-coated AC	0.552	&Kavitha, 2012		
AuNPs-coated	35.85			Mohamed et al.,
Eichlomia charcoal	2.36	2014		
Periwinkle shell		303-323	50-545	Yeneva &
				Georgieva, 2012
				Mohammed et al.,
		2014		
		300		Jolly & Mana, 2013
		300		Jolly & Mana, 2013
		308		Sumanjit et al, 2013
		303	50	Present

Table 4. Enthalpy and Entropy Change of some Adsorbent with Congo Red

Adsorbent	$\Delta S^\circ$	Reference
$\Delta H^\circ$	23.549	Namasivayam & Kavitha, 2012
Coir	pith 141.0	Sumanjit et al., 2013
7.705	194.0	Sumanjit et al., 2013
Groundnut	shell 44.43	Present
16.06		
Eichlomia	Charcoal	
32.53		
Periwinkle	shell	
8.2		

#### D. Efficiency of the Adsorbent

Congo red attained maximum or optimum adsorption at 120 minutes with the activated carbon. This trend agrees with Sekaran et al. (1995), which reported 120 minutes as the optimum contact time of Congo red (Coruh et al., 2015). Adsorption of the dye increases with increase in temperature, but after 308K, the increase in percentage adsorption of Congo red was no longer economical. Therefore, the optimum temperature for Congo red dye adsorption onto periwinkle shell activated carbon by graphical method is 308K, which agrees with (Namasivayam and Kavitha, 2012). Also, the concentration at which adsorption efficiency occur is 25mg/l with 91.30% (Fig 4). Therefore, the optimum concentration for the adsorption of Congo red is 25mg/l.

## CONCLUSION

The adsorption of Congo red onto periwinkle shell based activated carbon was investigated in this research work. The adsorption followed a pseudo-second order kinetic model, the amount of Congo red dye adsorbed was found to vary with concentration and contact time with the adsorbent and reached equilibrium at 120min. The effect of temperature increase with contact time on this adsorption with Periwinkle shell activated carbon was negligible. The surface area was obtained as 123.42m<sup>2</sup>/g. This study indicates that periwinkle shell has the potential to be an excellent low-cost adsorbent for the removal of Congo red dye from wastewater.

## ACKNOWLEDGEMENTS

The authors sincerely appreciate Mr. Ikhazuangbe Benson T, of NNPC-PPMC, Operations Department, Aviele Pump Station, for his financial assistance. Mr. Adeleke Kolapo and Adegbemi Jacob of Pharmacognosy and Pharmaceutical Technology Department respectively, Madonna University, Elele, Rivers State, for their technical support. Also, our thanks go to Mr. Daramola Abayomi, Water Analysis Department, Ion-exchange (Indian) Ltd, Eleme, Port Harcourt, Rivers State, for his material assistance.

## CONFLICT OF INTEREST

There is no conflict of interested associated with this article.

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