

Adsorption Isotherm, Kinetics and Thermodynamics Study of Cr (VI) ions onto Modified Activated Carbon from endocarp of *Canarium schweinfurthii*

**U. Bassey^{1*}, M. A. T. Suleiman², S. S. Ochigbo², M. M. Ndamitso², E. D. Daniel²,
S. E. Otoló¹ and A. Chukwudi³**

¹Department of Environmental Science, Federal University of Petroleum Resources Effurun,
Delta State, Nigeria.

²Department of Chemistry, Federal University of Technology, P.M.B, 65 Minna, Niger State,
Nigeria.

³Department of Production Planning and Quality Control, Port Harcourt Refining Company
Limited, Alesa Eleme, Rivers State, Nigeria.

Authors' contributions

This work was carried out in collaboration between all authors. Author UB designed the Study, performed the statistical analysis, wrote the protocol and wrote the draft of the Manuscript. All authors read and approved the final manuscript.

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ABSTRACT

Isothermal, kinetics and thermodynamics studies of Cr (VI) from simulated solution onto modified activated carbon from endocarp of *Canarium schweinfurthii* (African Elemi) was studied. The effect of process parameters such as, contact time, pH, adsorbent dosage, initial metal ion concentration and temperature were investigated. Process optimization was carried out at pH of 2 and contact time of 30 min. Pseudo second order kinetics gave best correlation coefficient with calculated adsorption capacity close to experimental value. The initial rate of reaction for Cr (VI) is 6.74×10^{-4} mg/g.min. Result obtained from isothermal studies showed $K_F > q_d$, and $\frac{1}{n} > 1$. Gibbs free energy

*Corresponding author: E-mail: uwembassey93@gmail.com;

investigated from thermodynamic study, showed the adsorption process is spontaneous and endothermic as enthalpy change is positive. The adsorbent is recommended for use for removal of Cr (VI) ions in industrial effluents.

Keywords: Adsorption isotherm; kinetics; thermodynamics; Cr (VI).

1. INTRODUCTION

The release of waste products into the environment is a global problem that has been highlighted by various environmentalists. Heavy metals are released spontaneously from different industrial, agricultural and domestic sources into the environment that causes a serious threat to human as well as aquatic life [1,2]. Excessive release of heavy metals into the environment through metal plating, mining operation, tanneries, chloroalkali, radiator manufacturing, smelting, alloy industries and storage batteries industries [3] and urbanization activities has posed a great problem worldwide. Unlike organic pollutants, which are susceptible to biological degradation, heavy metal ions do not degrade into harmless end pollutants [4], which is a major concern due to their toxicity to human. The presence of heavy metals above permissible levels in drinking water may cause adverse effect on human physiology. Metals such as Pb, Cu, Zn, Fe and Cr are known to be major anthropogenic pollutants in water [5]. These metals can cause several effects, including disruption of the biosynthesis of hemoglobin, anemia, rise in blood pressure, kidney damage, subtle abortion, birth defects, and disruption of nervous system, decline fertility of men through sperm damage, liver dysfunction, adrenal insufficiency, blindness, cardiovascular diseases and disruption of hormone [6,7].

However, various techniques have been applied for the removal of heavy metal ions in wastewater, such as chemical precipitation, coagulation, ion exchange, oxidation, electro dialysis, membrane separation, solvent extraction, photo-catalytic reduction and adsorption methods [8-10]. The use of conventional methods for the removal of heavy metals can be expensive, prohibitive for developing economy and most times do not effectively remove polluting metals [11], thus it becomes imperative to search for cheap and alternative means and bio-sorbents lately have become of considerable interest. The use of

indigenous biodegradable resources for treating hazardous waste would be less expensive and more effective [12]. The need for safe and economic method for the elimination of heavy metals from contaminated water has necessitated research interest towards the production of low cost alternative to commercial available activated carbon.

Agricultural waste material being economic and ecological friendly due to their unique chemical composition, availability, abundance, renewability, low cost and more efficient seem to be a viable option for heavy metal ions remediation. Such wastes like rice bran, rice husk, wheat bran, wheat husk, saw dust of various plants, bark of trees, groundnut shells, coconut shells, black gram husk, hazelnut shells, walnut shells, cotton seed hulls, waste tea leaves, cassia fistula leaves, maize corn cob, jatropha de-oiled cakes, sugarcane bagasse, apple, banana, orange peels, soya bean hulls, grapes stalk, water hyacinth, sugar beet pulp, *Dalbergia sisso*, *Arachis hypogeal*, sunflower stalks and cotton stalks [10,13-18], have been reported as capable of adsorbing and removing large amount of metal ions from aqueous solution.

This work is aimed at performance assessment of *Canarium schweinfurthii* (African Elemi) in removing Cr (VI) ions from simulated aqueous solution.

2. MATERIALS AND METHODS

2.1 Sample Collection

Seeds of *Canarium schweinfurthii* were sampled in the month of July, 2013 from Kutigi in Lavun Local Government area of Niger state, Nigeria and stored in polyethylene bag prior to the commencement of pretreatment. Sample pretreatment was carried out in the laboratory of Chemistry Department, Federal University of Technology, Minna.

2.2 Sample Pretreatment

The samples were washed with distilled water and air dried at room temperature for three weeks, and cracked to remove the kernels. The endocarps were ground using mortar and pestle, sieved using 250 micron mesh screen. The sieves were kept for carbonization and chemical activation process.

2.3 Carbonization of Adsorbent

The sieved biomass was placed in an electric furnace (Gallenkamp muffle furnace) and heated for one hour at 300°C, after which it was allowed to cool and stored in an air tight polyethylene bag [20].

2.4 Chemical Activation of Carbonized Adsorbent (Modification)

10 g of carbonized endocarp of *Canarium schweinfurthii* was carefully weighed and transferred into a beaker containing 50 ml of 0.5 M trioxonitrate (V) acid. The content of the beaker were thoroughly mixed and the paste formed was transferred to crucible, placed in a muffle furnace and heated at 400°C for 30 minutes. The activated samples were cooled to room temperature, washed with distilled water and dried in an oven at 105°C for 20 minutes. The final product screened through a 250 µm mesh size sieve and kept in an air tight polyethylene bags ready for use [21].

2.5 Preparation of Simulated Wastewater

The aqueous solution containing Cr (VI) ions were prepared from analytical grade $K_2Cr_2O_7$ respectively. 1000 ppm stock solutions of the salts were prepared. Other concentrations of 5 ppm, 10 ppm, 25 ppm, 50 ppm, 75 ppm, 100 ppm and 150 ppm were prepared by diluting the stock solution.

3. SORPTION EXPERIMENT

3.1 Effect of Contact Time

The effect of contact time was studied using an initial concentration of 5 ppm. The time interval chosen for the experiment was 30, 60, 90, 120, 150 minutes. 0.5 g of the pretreated powdered samples was mixed with 40 ml of the prepared aqueous solution in a conical flask, well corked and the mixture shaken on a rotator shaker at 250 revolutions per minutes at 30°C. At the end

of each contact time the mixture was filtered. Metal ions concentrations in the filtrate were analyzed using AA-6300 Shimadzu atomic absorption spectrophotometer.

3.2 Effect of pH

The effect of pH was studied using microprocessor pH meter. This was done by varying pH in the range of 2, 4,6,8,9, and 10 while keeping all other parameters such as, stirring speed 250 revolutions per minutes, at room temperature, adsorbent dose, 0.5 g and metal ion concentration, 5 ppm constant. The pH of each solution was adjusted using 0.1 M HCl or 0.1 M NaOH. After which the solution was filtered using whatmann filter paper No. 1 and the filtrate analyzed for metal ion concentration using atomic absorption spectrophotometer.

3.3 Effect of Temperature

The effect of temperature on adsorption studies using an initial concentration of 5 ppm was studied. The temperature range of (30, 40, 50, 60, 70, 80)°C was used, using a water bath. 0.5 g of the pre-treated powdered sample was added to 40 ml of aqueous solution in a 250 ml beaker, keeping other experimental conditions constant (stirring speed and optimum contact time) for the metal ions under investigation. The solution was stirred, filtered and analysed for metal ion concentration.

3.4 Effect of Adsorbent Dose

The percentage adsorption of metal ion on different forms of adsorbent was studied using different adsorbent doses (0.5, 1.0, 1.5, 2.0, 2.5 and 3.0) g in 40 ml of the stock solution keeping other experimental conditions constant, after which the mixture was filtered using whatmann filter paper No. 1 and the filtrate analyzed using atomic absorption spectrophotometer.

3.5 Effect of Initial Metal ion Concentration

The percentage removal of metal ions was investigated by varying the concentration of the ions (10, 25, 50, 75,100 and 150) ppm. 0.5 g of prepared activated carbon was be added to 40 ml of aqueous solution in a 250 ml beaker, keeping other experimental conditions constant (stirring speed and optimum pH and optimum contact time at temperature of 30°C). After which it was filtered using a whatman filter paper

No. 1 and the residual concentration of the metal ions in the filtrate was determined using an atomic absorption spectrophotometer.

3.6 Measurement of Physical Properties of Activated Carbon

- Moisture content of adsorbent was determined by weighing 5 g of both adsorbent into a crucible. This was placed in the oven and heated for 5 hours at constant temperature of 105°C. The sample was then removed and transferred into desiccator. This procedure was repeated several times until a constant weight was obtained. The difference in the mass constitutes the amount of moisture content of the adsorbent.

$$\text{Percentage moisture content} = \frac{W_2 - W_3}{W_2 - W_1} \times 100 \quad (1)$$

W_1 = Weight of crucible

W_2 = Initial weight of crucible with sample

W_3 = Final weight of crucible with sample

- Bulk density was determined by placing 2 g of granular activated carbon of 10-30 mesh size carbon in a 25 ml cylinder and tapping the cylinder for at least 1-2 minutes after which the volume of carbon was measured. The bulk density was measured as:

$$\text{Bulk density (g/m}^3\text{)} = \frac{\text{weight of dry sample (g)}}{\text{volume of packed dry sample (g)}} \dots\dots \quad (2)$$

- Ash content was determined by weighing 2 g of activated carbon (< 325 mesh size) in a porcelain crucible and heating the carbon to 115°C for 6 hours. The carbon was then heated in a muffle furnace at 950°C for 1.5 hours. The crucible was then removed and placed in a dessicator and weighed as

$$\text{Ash (\%)} = \frac{\text{Final solid weight}}{\text{Initial carbon weight (g)}} \times 100 \quad (3)$$

- Hardness of the sample was measured using wet attrition method. One gram of sample was added to 100 ml of acetate buffer (0.07 M sodium acetate and 0.03 M acetic acid of pH 4.8) in a 150 ml beaker. The solution was stirred for 24 hours for proper agitation. The solution was filtered using mesh screen and the retained sample was thoroughly washed and dried at 90°C under vacuum for 4 hours and

weight. Percentage hardness was measured as

Hardness (%)

$$= \frac{\text{Initial weight (g)} - \text{final weight (g)}}{\text{Initial weight}} \times 100 \quad (4)$$

3.7 Data Analysis

The adsorptive capacity of metal ions per unit adsorbent (mg/g) was determined using the expression:

$$q_e = \frac{(C_i - C_e)}{M} \times V \quad (5)$$

Where q_e represent the amount of metal removed per unit of the adsorbent (mg/g), V is the volume of metal solution (litres), C_i is the initial concentration of metal ions in the solution (mg/L), C_e is the final concentration of metal ions in the solution and M is the dry weight of adsorbent. Percentage removal of heavy metal ions from initial solution concentration can be calculated from the following Equation.

$$\text{Percentage adsorption} = \frac{(C_i - C_e)}{C_i} \times 100 \quad (6)$$

3.8 Kinetics Modeling

3.8.1 The expression for the Lagergren pseudo-first order model [22]

$$\log (q_e - q_t) = \log q_e - \frac{K_1 t}{2.303} \quad (7)$$

Where, K_1 is the Lagergren rate constant for adsorption (min^{-1})

q_t is equilibrium concentration of metal ion in solution at a particular time in (mg/g)

3.8.2 The Lagergren pseudo-second order kinetic model [23]

$$\frac{t}{q_t} = \frac{1}{h} + \frac{t}{q_t} \quad (8)$$

K_2 is equilibrium rate constant of second order kinetics model (g/mg/min).

3.8.3 Isotherms modeling

3.7.3.1 The Dubinin-Radushkevich model

$$\ln (q_e) = \ln (q_d) - k_{ad} \epsilon^2 \quad (9)$$

Where

q_d = theoretical isotherm saturation capacity (mg/g)
 q_e = adsorption capacity of adsorbent in mg/g
 k_{ad} = Dubinin-Radushkevich isotherm constant ($\text{mol}^2\text{kJ}^{-2}$) related to free energy of sorption.
 ϵ = $RT \ln \left[1 + \frac{1}{C_e} \right]$ which represent potential energy

R, T and C_e signifies gas constant (8.314 J/mol/K), temperature in Kelvin and concentration of metal ions (mg/L) in solution at equilibrium.

$$E = \frac{1}{\sqrt{2}K_{ad}} \quad (10)$$

3.7.3.2 The Freundlich model:

$$\log \frac{x}{m} = \frac{1}{n} \log C_e + \log K_F \quad (11)$$

Where

n and K_F represent the Freundlich constant, related to the adsorption intensity and adsorption capacity.

3.9 Thermodynamics Analysis

The temperature effect on the biosorption of Cr (VI) ions onto the endocarp of *Canarium schweinfurthii* helps in evaluating the change in free energy (ΔG°), enthalpy (ΔH°) and entropy (ΔS°). The free energy change was estimated using the relation below as proposed by other researchers [24,25]. T (K) denotes the absolute temperature; R is the gas constant (KJ/mol). The equilibrium constant (K_c) was evaluated using the following equation:

$$K_c = \frac{C_{ad}}{C_e} \quad (12)$$

Where C_{ad} and C_e are the equilibrium concentrations of metal ions (mg/g) in biosorbent and on solution respectively. Having estimated the value of K_c , a graph of $\ln K_c$ versus $1/T$ was plotted based on the equation below.

$$\ln K_c = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \quad (13)$$

Values of ΔH° and ΔS° which represent the slope and intercept were obtained from the plots

4. RESULT AND DISCUSSION

4.1 Physical Properties of Modified Activated Carbon

Table 1 shows physical properties of modified activated carbon from *Canarium schweinfurthii*, for the adsorption of Cr (VI). Ash content and bulk density compares favorably with reference activated carbon reported in literatures by [26,27]. Measurement of hardness of activated carbon aids in predicting its resistance to degradation and other factors such as crushing and abrasion.

Table 1. Physicochemical properties of MAC

Parameter	Values
Bulk density (g/m^3)	0.58
Ash content (%)	4.00
Moisture content (%)	0.10
Hardness (%)	6.25

4.2 Contact Time

Effect of contact on adsorption of Cr (VI) is shown in Fig. 1. An optimum time of 30 minutes was obtained, after which there was percentage decrease in adsorption. High uptake of Cr (VI) ions at the start of the reaction occurred as a result of availability of adsorption sites. Variation of percentage of metal ions adsorbed at different contact time is due to adsorption and desorption of Cr (VI) ions from adsorption sites.

4.3 pH

Fig. 2. Shows the effect of pH on adsorption of Cr (VI) ion. An optimum pH of 2 was obtained. Higher pH values resulted to decrease in concentration of Cr (VI) ions adsorbed. This is due to repulsion between OH^- and HCrO_4^- , $\text{Cr}_2\text{O}_7^{2-}$, $\text{Cr}_4\text{O}_{13}^{2-}$ [28].

4.4 Adsorbent Dosage

From Fig. 3. an increase in adsorbent dosage led to increase in removal of Cr (VI) ion this is not surprising as increase adsorbent dosage enhances availability of adsorption sites and creates more surface area. From adsorbent dosage of 1.5 g, 100% removal of metal ion was obtained.

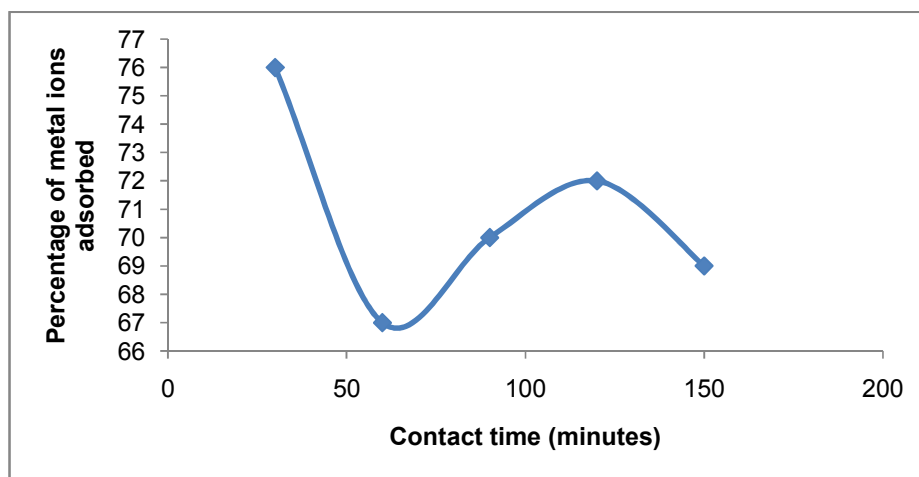


Fig. 1. Effect of contact time variation on adsorption of Cr (VI) at initial concentration of 5 ppm

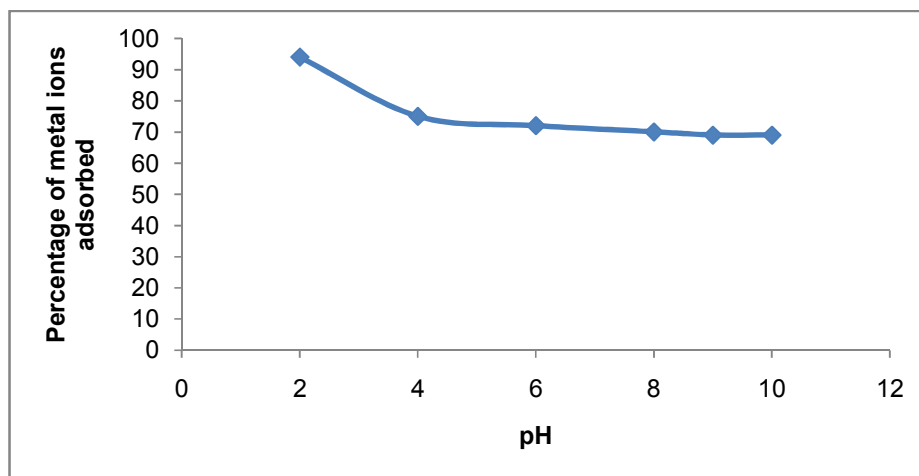


Fig. 2. Effect of pH variation on adsorption of Cr (VI) at initial concentration of 5 ppm

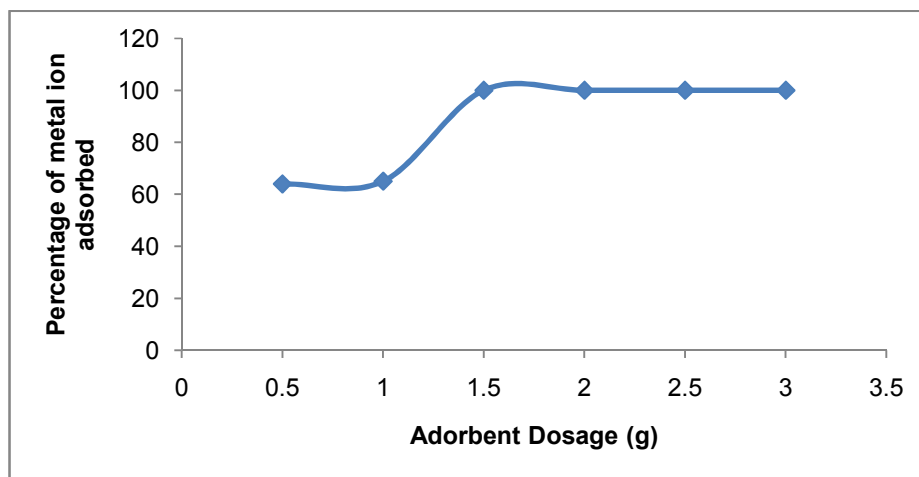


Fig. 3. Effect of adsorbent dosage variation on adsorption of Cr (VI) at initial concentration of 5 ppm

4.5 Initial Concentration

Effect of varying metal ion concentration is shown in Fig. 4. This was done in order to determine the maximum concentration of Cr (VI) ions adsorbed. Adsorption increased up to 50 ppm and gradually reduced due to coverage of adsorption sites.

4.6 Temperature

Effect of temperature on adsorption of Cr (VI) is shown in Fig. 5. An increase in temperature led to increase in percentage adsorption of the metal ion. This is not surprising as the kinetic energy of the metal ion was increased, which enhanced its fast movement onto the surface of the adsorbent.

Table 1 gives parameters for first and second order kinetics. Theoretical adsorption capacity (Q_c) for second order kinetic is close to experimental value (Q_e) with high correlation coefficient compared to first order kinetics, hence suitable for kinetic modeling of adsorption process.

From Table 2., value of n obtained is less than unity, and $1/n > 1$, indicating that the amount of Cr (VI) ions in the adsorbent is more compared to that in the solution implying existence of physical bonding between Cr (VI) ions and the adsorbate [29,30]. Adsorption capacity K_F and correlation coefficient obtained for Freundlich isotherm is greater than that of Dubinin-Radushkevich isotherm implying suitability of Freundlich isotherm for isothermal study of the adsorption process.

Table 2. gives value for the mean free energy of Cr (VI) ions, which is less than 8 kJ/mol implying physisorption played significant role in the adsorption process [31].

From Table 3, value of ΔG° obtained for the metal ion was negative, indicating spontaneity of the reaction process. Positive values obtained for ΔH° and ΔS° implies endothermic adsorption process, and increase in temperature causes rapid movement of metal ions onto the surface of the adsorbent.

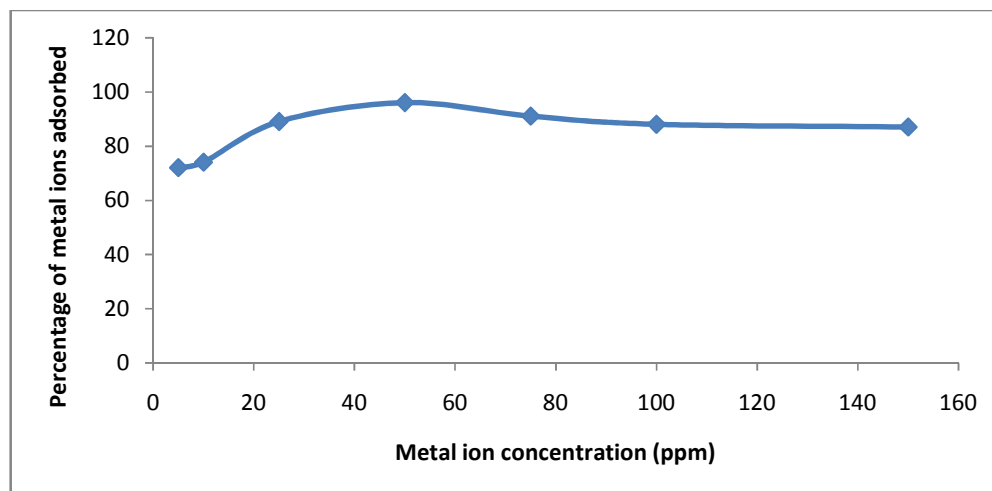


Fig. 4. Effect of metal ion concentration variation on adsorption of Cr (VI)

Table 1. Kinetic model for adsorption of Cr (VI) onto modified activated carbon from endocarp of *Canarium schweinfurthii*

Pseudo- first order parameters				
K ₁ (Min ⁻¹)	Q _e (mg/g)	Q _c (mg/g)	R ²	
1.407	0.285	1.000	0.275	
Pseudo- second order parameters				
K ₂ (g/mg.min)	Q _e (mg/g)	h x 10 ⁴ (mg/g.min)	Q _c (mg/g)	R ²
0.008	0.285	6.74	0.340	0.978

Q_e and Q_c are experimental and theoretical adsorption capacity at studied concentration of 5ppm

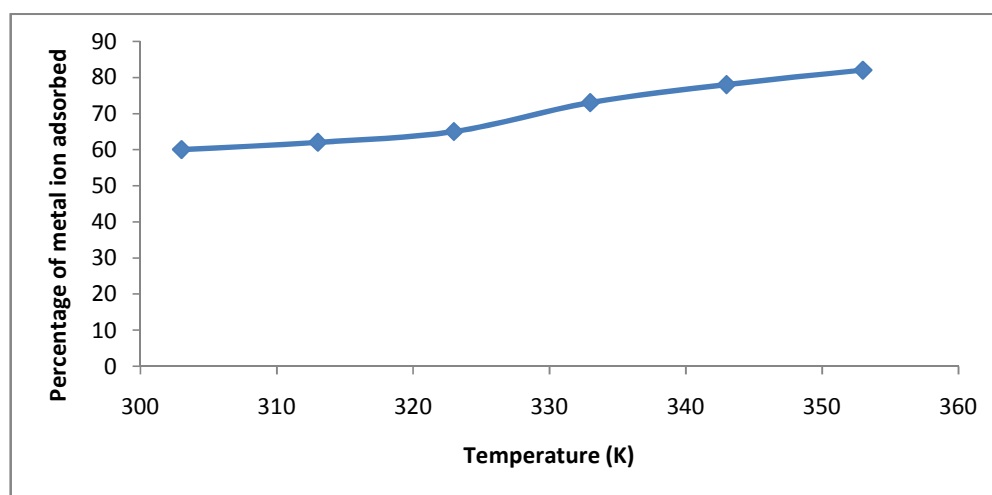


Fig. 5. Effect Temperature variation on adsorption of Cr (VI) at initial concentration of 5 ppm

Table 2. Isotherm model for adsorption of Cr (VI) onto modified activated carbon from endocarp of *Canarium schweinfurthii*

Freundlich parameters			
K_F (mg/g)	n	$1/n$	R^2
10.960	0.860	1.160	0.898
Dubinin- Radushkevich parameters			
$K_{ad} \times 10^7$ (mol ² /KJ ²)	E (KJ/mol)	q_d (mg/g)	R^2
10.000	0.745	7.614	0.148

Table 3. Thermodynamic parameter for adsorption of Cr (VI)

ΔH° (kJ/mol)	ΔS° (J/mol/K)	ΔG° (kJ/mol)	R^2
17.760	0.070	-2.740	0.949

5. CONCLUSION

The results obtained, showed that pH influences the uptake of Cr (VI) ions onto modified activated carbon from endocarp of *Canarium schweinfurthii*. An increase in adsorbent dosage resulted to increase in percentage removal of Cr (VI) ions from the simulated solution. Higher concentration of the metal ions investigated showed that the adsorbent has the potential to completely adsorb 50 ppm of Cr (VI) ions. Hence it would be sustainable for use as substitute for the remediation of wastewater from industrial effluent in order to curb the menace of heavy metals pollution.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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