

Thermodynamic and Isotherms Studies on the Application of Modified Activated Carbon Developed From Periwinkle Shells for the Adsorption of Heavy Metals from Waste Water

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ABSTRACT: The aim of this study was to investigate the thermodynamic properties of modified activated carbon developed from periwinkle shell carbon (PSC) for the adsorption of heavy metals (Fe^+ and Pb) from waste water. Metals adsorption from aqueous solutions onto periwinkle shell carbon was prepared (carbonized at 650°C and activated with phosphoric acid) and characterized to determine its physicochemical properties. Batch adsorption experiments were conducted to investigate the effects of process parameters (contact time, particle size, carbon dosage and initial concentration) on adsorption rate. Adsorption isotherms were analyzed using the Langmuir, Freundlich and Temkin isotherms while thermodynamic parameters such as enthalpy change (ΔH°), entropy change (ΔS°) and Gibbs-energy change (ΔG°) were determined. Results showed that adsorption rate increase with increase in contact time, adsorbent dosage and decreased with increase in particle size. Isotherm data showed that the Freundlich isotherm accurately described the adsorption data indicating that adsorption has a heterogeneous adsorbent surface. Thermodynamic parameters results showed that adsorption process was endothermic with enthalpy change (ΔH°) of 96.38KJ/mol ; a positive entropy change (ΔS°) of 35.11 KJ/mol , indicating an increase in the degree of freedom (or disorderliness) of the adsorbed specie and a negative Gibbs-energy change (ΔG°) at all temperature indicating that the adsorption process was spontaneous and favourable at high temperature. The study has showed PSC is a good adsorbent and it is effective enough for waste water

treatment, as such it is recommended for integration with existing technologies to explore compatibilities with current water treatment systems and scale-up studies should be conducted to evaluate performance in continuous flow system.

Keywords: Thermodynamic, Isotherm, Adsorption, Periwinkle, Activated Carbon

I. INTRODUCTION:

Industrial developments comes along with the generation of unwanted by-products, which in most cases are discharged into the ecological systems leading to different types of pollutions. Subsequent negative impact of such pollution is felt not only by human beings but also by living organisms including plants. For instance, the introduction of untreated effluents into rivers and water bodies leads to build-up of poisonous contaminants (water pollution) like heavy metals (Hg, Pb, Cu, Fe, Zn, etc.) among others (Gupta et al., 2001). Thus, water pollution remains one of the most pressing environmental challenges, with heavy metals being a significant concern due to their toxic and persistent nature. Heavy metals such as lead (Pb), cadmium (Cd), mercury (Hg), Iron (Fe) and arsenic (As) are commonly discharged into water bodies through industrial activities, including mining, manufacturing, and improper waste disposal (Fu and Wang, 2011). Unlike organic pollutants, the majority of which are susceptible to biological degradation, heavy metal ions do not degrade into harmless end products (Gupta et al., 2001). These metals pose severe risks to both human health and the environment, causing

problems such as kidney damage, neurological disorders, and even cancer upon prolonged exposure (Fu and Wang, 2011). The World Health Organization (WHO, 1984) made us to know that the most toxic metals are aluminum, chromium, cobalt, nickel, copper, zinc, cadmium, mercury and lead. The main sources of toxic metals are industrial wastes from processes such as electroplating, metal finishing, and chemical manufacturing (Abdel-Halimet al., 2003).

When heavy metals enter the food chain, they build up and cause mental retardation, reduction in hemoglobin, production and interference with normal cellular metabolism and consequently may damage the nervous system. Heavy metals are not biodegradable and can accumulate in living organisms, leading to bioaccumulation and biomagnification through the food chain. Furthermore, strong exposure to heavy metals may cause gastric pain, nausea, vomiting, severe diarrhea, hemorrhage and affect the digestive tract and lungs (Klaassen et al., 2001; Mohanty et al., 2005). This persistent nature necessitates efficient removal techniques from contaminated water to prevent adverse health effects and environmental damage (Goyer, 1997). Conventional methods for removing heavy metals from wastewater include chemical precipitation, ion exchange, reverse osmosis, and electrochemical treatment (Babel and Kurniawan, 2003). While these methods can be effective, they often suffer from high operational costs, complexity, and the generation of secondary pollution. Thus, it is desirable to remove toxic heavy metals from wastewater, probably via adsorption process, before it is discharged into the environment. Adsorption is a generally preferred method for removing heavy metal ions in terms of initial cost, flexibility and simplicity of design, ease of operation, insensitivity to toxic pollutants and the availability of different adsorbents. Furthermore, it does not result in the formation of harmful waste product, a problem with most other methods (Wang and Li, 2007; Dawodu and Akpomie, 2014). There are several methods for treatment of metal contaminated effluents such as membrane processes (Yang et al., 2001), chemical precipitation (Wingenfelder et al., 2005), coagulation-flocculation (Shammaset al., 2004), electro-dialysis (Metcalf and Eddy, 2003), reverse osmosis (Metcalf and Eddy, 2003), ultra-filtration (Eckenfelder, 2000), ion exchange (Eckenfelder, 2000), flotation (Matiset al., 2003), ion exchange (Eckenfelder, 2000) and flotation (Matiset al., 2003). They have their inherent advantages and limitations in application.

Periwinkle is a marine mollusk and generally found in rocky shores, muddy areas, swamps, oceans in Nigeria. Thus, it is mostly found in Niger Delta lagoons and mudflats between Calabar and Badagry (Gamus and Okpeku, 2015). People eat the edible portion as sea food and use the shell as coarse aggregate in concrete for control of water logged areas (Ochalo, 2018). Periwinkle shell has been found to be good material for production of activated carbon, with large surface area that is used as a powerful adsorbent, a highly porous structure, and in this way helps to reduce cost of waste disposal (Aoet al., 2018). Periwinkle shells are primarily composed of calcium carbonate, which can be transformed into activated carbon through processes such as carbonization and chemical activation. The resultant activated carbon can be further modified to enhance its properties, making it suitable for heavy metal adsorption (Oyedoh and Ekesiobi, 2023). The modification processes typically involve treatments with acids, bases, or other chemicals to increase the surface area, pore volume, and functional groups on the carbon surface, thereby improving adsorption capacity (Ioannidou and Zabaniotou, 2007).

Activated carbon is known for its high surface area, porosity, and adsorption capacity, making it a popular choice for removing various pollutants, including heavy metals, from water (Gupta and Suhas, 2009). The traditional sources of activated carbon are primarily non-renewable materials like coal and peat, contributing to its high production cost. Moreover, this economic barrier limits the widespread application of activated carbon, particularly in developing regions where the financial burden of wastewater treatment is a significant concern (Yusuf et al., 2012). Hence, activated carbons are generally prepared by carbonization involving pyrolysis of carbonaceous material (periwinkle shell) in the absence of active air (oxygen), followed by conversion of the carbonized product to a porous activated carbon with high surface area, while carbonization creates initial porosity (Odetoye et al., 2019). However, activated carbon can be used as catalyst and co-catalyst for removal of pollutants from gas, liquids and chemical recovery, in domestic, commercial and industrial settings, activated carbon helps to remove varieties of contaminants such as metallic and non-metallic pollutants, dyes, taste, odor from aqueous medium. They are also used in food industry in decolourization, deodorization and taste removal; to remove heavy metals from liquid, in gas cleaning applications and air filter and also in general air conditioning

application (Yusufuet al., 2012; Din et al., 2017; Afifet al., 2019).

A lot of researches has been carried out on the production of activated carbon from different plants and animal originated wastes such reports from plants origin include; rice husk (Nasehir, et al., 2010; Goodhead and Dagde, 2011); shea nut shell (Itodo and Itodo, 2011); almond shell (Daud and Ali, 2004). Those from animal origin include; periwinkle shell, (Nworiet al., 2020); animal horns (Aliyor and Badmus, 2008), etc. To address these challenges, researchers have explored alternative, efficient, cost-effective, availability of raw materials and sustainable sources for activated carbon production. Agricultural and industrial waste materials, such as coconut shells, rice husks, and nutshells, have shown promising usage due to their abundance, low cost, and high carbon content (Mohan et al., 2007). Among these, periwinkle shells, a by-product of the seafood industry, present a particularly promising raw material and periwinkle shells are abundantly available in coastal regions which are often discarded as waste, posing an environmental challenge (Ademiluyi and David-West, 2012).

The use of periwinkle shell-derived as an activated carbon for heavy metal adsorption is an emerging area of research with significant potential. Studies have demonstrated that such activated carbon can effectively adsorb heavy metals from aqueous solutions, showing comparable or superior performance to conventional activated carbon (Ademiluyi and David-West, 2012; Oyedoh and Ekesiobi, 2023). However, comprehensive thermodynamic studies are limited, which hinders the full understanding and optimization of this material for practical applications. This study aims to bridge this gap by conducting a detailed thermodynamic analysis of the adsorption of heavy metals onto modified activated carbon developed from periwinkle shells, also, by characterizing the physical and chemical properties of the modified activated carbon and evaluating its adsorption performance under various conditions, this research seeks to provide valuable insights into the mechanisms and efficiency of heavy metal removal. Understanding the thermodynamics of adsorption processes is crucial for optimizing the performance of activated carbon in removing heavy metals from wastewater. Thermodynamic parameters such as enthalpy (ΔH), entropy (ΔS), and Gibbs free energy (ΔG) provide insights into the feasibility, spontaneity, and nature of the adsorption process (Hameedet al., 2009). These parameters help in understanding how

changes in temperature and other conditions affect the adsorption capacity and efficiency, guiding the design and operation of treatment systems.

However, the disposal of heavy metals in wastewater poses a significant environmental threat due to their toxic nature and persistence in the environment. Most methods for removing heavy metals from wastewater are often expensive and inefficient. Therefore there is need to explore alternative and cost-effective approaches for the removal of heavy metals from wastewater around the south-south region of Nigeria.

II. MATERIALS AND METHOD

The periwinkle shell used for this study was bought from EkambaNsukaraoffot market in Uyo, AkwaIbom State Nigeria. These shells were selected due to their high availability, low cost, and potential for sustainable usage as a precursor for activated carbon production. Their inherent properties are anticipated to contribute significantly to the adsorption capacity of the activated carbon, aligning with thermodynamic predictions of adsorption potential. Also, the synthetic wastewater solution used was prepared in the Chemical Engineering Laboratory, University of Uyo, Nigeria. The waste water being a vital raw material for this study was gotten from stock solution of the Iron(III)chloride. Most of the chemicals/ reagents used were obtained from the Chemical Engineering Laboratory but some were bought from chemicals stores in Uyo. The experiments were conducted using the basic equipment as written in literatures.

2.1 Preparation of Raw Materials and Methods

The following preparation of the raw materials and methods were adopted:

2.1.1 Adsorbent Preparation

The periwinkle shells were thoroughly washed with distilled water to remove any surface dirt and impurities. Followed by sun-dried for 24 hours and then oven-dried at 105°C for 12 hours to ensure complete moisture removal and then crushed and sieved to suitable particle or tiny size. This step is crucial as the presence of moisture can affect the activation process and the resultant adsorption efficiency (Akpa and Nduka, 2018).

2.1.2 Carbonization

Thirty-five (35) g weight of the periwinkle shell was introduced into the reactor and pyrolyzed at 650°C for two hours in an oven in the absence of air in order to produce a carbonized product. A receiver was connected to the condenser to receive

the distillates formed during the pyrolysis. The char material was then cooled at room temperature before discharging into containers. This thermal decomposition process is expected to enhance the porosity and surface area of the biochar, which are critical factors influencing adsorption capacity from a thermodynamic perspective (Akpa and Nduka, 2018).

2.1.3 Carbon Activation

The carbonated material (carbon) from the periwinkle shells was crushed into powdered form using a crusher. A measured weight of the crushed sample was soaked in phosphoric acid of known concentration in a conical flask and stirred until the mixture turned a paste. The paste was heated in a muffle furnace, the activated carbon was re-carbonized at 650°C, for 45 minutes in the absence of air to increase the surface area of the charred sample; the activated carbon was cooled at room temperature, washed with distilled water until pH is approximately 7 (no change in color when tested with red litmus paper) indicating no trace of the phosphoric acid used. The char was dried for 6 hours, sieved with sieves of various sizes (75µm, 150µm, 300µm, 500µm to obtain activated carbon of various particle sizes) and were stored in tight nylons (Akpa and Nduka, 2018; Nweze, 2024).

2.2 Batch Adsorption Experiments

The experiments to remove heavy metal from waste water were performed in batch mode. The batch experiment was conducted by varying the initial metal ion concentration and contact time at ambient temperature for isotherm and kinetic study respectively. For the isotherm studies, batch adsorption studies was conducted by adding 0.5 g of the activated carbon samples to dilute solutions of different initial concentration ranging from 10 to 50 mg/L in increments of 10mg/L in 100 mL beakers. The beakers were placed in an orbital shaker at 150 rpm and adsorption proceeded for 60 minutes. For the kinetic study, the adsorption experiment was carried out by keeping initial concentration (20 mg/L) and adsorbent mass (0.5g) constant while varying contact time in increments of 15 minutes ranging from 15 to 75 minutes. Thereafter, the effluent was filtered with a filter paper and the residual filtrate was analyzed. The heavy metal concentration was determined by Atomic Adsorption Spectroscopy (AAS) and was analyzed for the isotherm and kinetic study.

2.2.1 Preparation of Heavy Metal Solutions

Stock solutions of heavy metals were prepared by dissolving 0.3g of FeCl₃ in 1L of distilled water to obtain 100mg/L concentration. Working solutions of desired concentrations was prepared by appropriate dilution of the stock solutions (10, 20, 30, 40 and 50mg/L). These solutions were used to study the adsorption process under different thermodynamic conditions.

2.3 Data Analysis

The adsorption capacity (q_e) and percentage removal (%R) was calculated using the following equations:

$$q_e = [(C_0 - C_e)V]/m$$

Equation 1

where C_0 is the initial concentration of the metal ion (mg/L), C_e is the equilibrium concentration of the metal ion (mg/L), V is the volume of the solution (L), and m is the mass of the activated carbon (g). These calculations were critical in understanding the adsorption isotherms and the thermodynamic parameters such as Gibbs free energy, enthalpy, and entropy changes associated with the adsorption process (Augustus et al., 2020).

2.4 Thermodynamic Studies

Thermodynamic parameters were calculated to provide insights into the nature of the adsorption process. The following thermodynamic equations were utilized.

2.4.1 Gibbs Free Energy Change (ΔG^0)

The Gibbs free energy change was determined using the equation:

$$\Delta G^0 = -RT \ln K \quad \text{Equation 2}$$

where R is the universal gas constant (8.314 J/mol·K), T is the temperature in Kelvin, and K is the adsorption equilibrium constant. A negative (ΔG^0) will indicate a spontaneous adsorption process (Augustus et al., 2020).

2.4.2 Enthalpy Change (ΔH^0) and Entropy Change (ΔS^0)

The enthalpy change and entropy change was determined from the Van't Hoff equation:

$$\ln K = (\Delta S^0/R) - (\Delta H^0/RT) \quad \text{Equation 3}$$

By plotting ($\ln K$) against ($1/T$), (ΔH^0) and (ΔS^0) can be obtained from the slope and intercept, respectively. A positive (ΔH^0) will indicate an endothermic process, while a positive (ΔS^0) will suggest increased randomness at the solid-liquid interface during adsorption (Augustus et al., 2020).

2.5 Isotherm Studies

In this study, equilibrium data was analyzed using the Freundlich, Langmuir, Temkin isotherms expression.

2.5.1 Freundlich Isotherm

The Freundlich (1906) equation in Equation 4 was used to calculate the model; is an empirical equation based on adsorption on a heterogeneous surface. The equation is commonly represented as:

$$q_e = K_F C_e^{1/n} \quad \text{Equation 4}$$

Equation 4 can be expressed in its linear form as:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e$$

Equation 5

Where C_e (mg/L) is the equilibrium concentration and q_e (mg/g) is the amount adsorbed metal ion per unit mass of the adsorbent. The constant n is the Freundlich equation exponent that represents the parameter characterizing quasi-Gaussian energetic heterogeneity of the adsorption surface (Bansal and Goyal, 2005). K_F (L/g) is the Freundlich constant indicative of the relative adsorption capacity of the adsorbent. The Freundlich exponent, n , should have values lying in the range of 1 to 10 for classification as favorable adsorption (Okewaleet al., 2011). From Equation 5, a plot of $\ln q_e$ versus $\ln C_e$ will give a straight line of slope $1/n$ and intercepts $\ln K_F$.

2.5.2 Langmuir Isotherm

The Langmuir Isotherm model was calculated from the expression Equation 7. The Langmuir (Langmuir, 1918) model assumes that uptake of metal ions occurs on a homogenous surface by monolayer adsorption without any interaction between adsorbed ions. The Langmuir equation may be written as:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \quad \text{Equation 6}$$

Equation 6 can be expressed in its linear form as:

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \quad \text{Equation 7}$$

where q_e is the amount adsorbed (mg/g), C_e is the equilibrium concentration of the metal ion (mg/L), q_m (mg/g) is the maximum amount of adsorbed metal ion per unit mass of sorbent

corresponding to complete coverage of the adsorptive sites, K_L (L/mg) is the Langmuir constant related to the energy of adsorption. From Equation 7, q_m and K_L was determined from the slope and intercept of the plots of C_e/q_e versus C_e .

2.5.3 Temkin Isotherm

The Temkin Isotherm model was calculated from the expression of Equation 9. The Temkin isotherm equation assumes that the heat of adsorption of all the molecules in layer decreases linearly with coverage due to adsorbent-adsorbate interactions, and that the adsorption is characterized by a uniform distribution of the bonding energies, up to some maximum binding energy (Temkin and Pyzhev, 1940). The Temkin isotherm is represented by the following equation:

$$q_e = \frac{RT}{b} \ln(K_T C_e) \quad \text{Equation 8}$$

Equation 8 can be expressed in its linear form as:

$$q_e = B_T \ln K_T + B_T \ln C_e \quad \text{Equation 9}$$

Where, T is the absolute temperature (K), R is the universal gas constant (8.314J/mol.K), K_T is the equilibrium binding constant (L/mg), and b_T is the variation of constant related to the heat of adsorption (kJ/mol). The Temkin adsorption isotherm model was chosen to evaluate the adsorption potentials of the adsorbent for adsorbates. From Equation 9, there was a plot of q_e versus $\ln C_e$.

2.6 Adsorption Capacity

Batch adsorption experiments were conducted to investigate the adsorption capacity of the modified activated carbon under the effect of contact time, particle sizes, adsorbent dosage and initial concentration variations. Fixed amount of activated carbon were added to known volumes of heavy metal solution of known concentrations in different flasks. The flasks were agitated at a certain rate (150rpm) in a temperature-controlled shaker at 30°C. These conditions allowed for the study of adsorption kinetics and equilibrium, essential for understanding the thermodynamic aspects of the process (Foo and Hameed, 2010).

The amount of heavy metals adsorbed by activated carbon was determined using a mass balance equation expressed in Equation 10:

$$q_e = \frac{V(C_o - C_e)}{M}$$

Equation 10

The removal efficiency was calculated by measuring the concentration of heavy metals before (initial concentration) and after adsorption (final concentration) using the formula below.

$$\% \text{Removal} = 100 \times \frac{C_0 - C_e}{C_0}$$

Equation 11

where q_e is metal concentration on the kaolinite (mg/g) at equilibrium, C_e is metal concentration in solution (mg/L) at equilibrium, C_0 is initial metal concentration in solution (mg/L), v is volume of initial metal solution used (L), and m is mass of activated carbon used (g) (Abbas et al., 2012).

2.7 Kinetic studies

Kinetic adsorption models describe the mechanism of adsorption of heavy metal ions by biosorbents and particularly determine the rate of biosorption during the removal of heavy metals from wastewater on an industrial scale to optimize the design parameters; including the adsorbate residence time and reactor dimension (Sadeek et al., 2015). In experimental works, the kinetic description of adsorption processes and the removal efficiency of heavy metals by various adsorbents have frequently been evaluated by several mathematical models, most of which are empirical equations (Chen et al., 2022). The common kinetics models (pseudo-first order, pseudo-second order, intra-particle diffusion kinetic, and Elovich). The choice between these equations is frequently based on the goodness-of-fit, as judged by the coefficient of determination (R^2) (Chen et al., 2022). Kinetics of the model parameters (k_1 and q_e for pseudo-first-order reaction model, and k_2 and q_e for pseudo-second-order reaction model) were estimated.

2.7.1 Pseudo-First Order Model

Lagergren's kinetics equation is widely used for the adsorption of an adsorbate from an aqueous solution (Lagergren, 1898):

$$\frac{dq_t}{dt} = k_1(q_e - q_t)$$

Equation 12

After integration and applying boundary conditions $t = 0$ to $t = t$ and $q_t = 0$ to $q_t = q_e$, the integrated form of Equation (12) becomes:

$$\ln(q_e - q_t) = \ln q_e - k_1 t$$

Equation 13

Where q_t is the amount of metal adsorbed per unit of adsorbent (mg/g) at time t , k_1 is the pseudo-first order rate constant (L/min), and t is the contact

time (min). The adsorption rate constant (k_1) were calculated from the plot of $\ln(q_e - q_t)$ against t .

The Pseudo First order model was calculated from the expression in Equation 13 The adsorption rate constant (k_1) was calculated from the plot of $\ln(q_e - q_t)$ against t

2.7.2 Pseudo- Second Order Model

Ho and McKay, (1998) presented the pseudo-second order kinetic as:

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2$$

Equation 14

For the boundary conditions $t = 0$ to $t = t$ and $q_t = 0$ to $q_t = q_e$, the integrated form of Equation 14 becomes:

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

Equation 15

Where k_2 is the pseudo-second order rate constant (g/mg.min). The initial adsorption rate, h (mg/g.min) at $t \rightarrow 0$ is defined as:

$$h = k_2 q_e^2$$

Equation 16

The h , q_e and k_2 can be obtained by linear plot of t/q_t versus t . The Pseudo Second order model was calculated from the expression in Equation 15 and 16. From Equation 16, the h , q_e and k_2 can be obtained by linear plot of t/q_t versus t .

III. RESULTS AND DISCUSSION

Here, we present the results of the adsorption experiments, focusing on the process parameters, isotherms and kinetics of the adsorption process. The data obtained from the experiments are analyzed and discussed, providing insights into the adsorption mechanisms, capacities etc.

3.1 Adsorption Characteristics

3.1.1 Effect of Initial Concentration

The amount of metal ions adsorbed is a function of the initial concentration of the metal ion, making it an important factor in effective adsorption. The sorption capacity of Periwinkle Shell Activated Carbon (PSAC) for Fe (III) ion in this study increased with increasing metal ion concentration from 10 to 50mg/l, resulting to an increase metal ion concentration gradient that overcomes the resistance to mass transfer of metal ions between the aqueous phase and the adsorbent. A higher concentration in a solution implies a higher concentration of metal ion to be fixed on the

surface of the adsorbent (Barkaet al., 2013). It is shown in Figure 1 below.

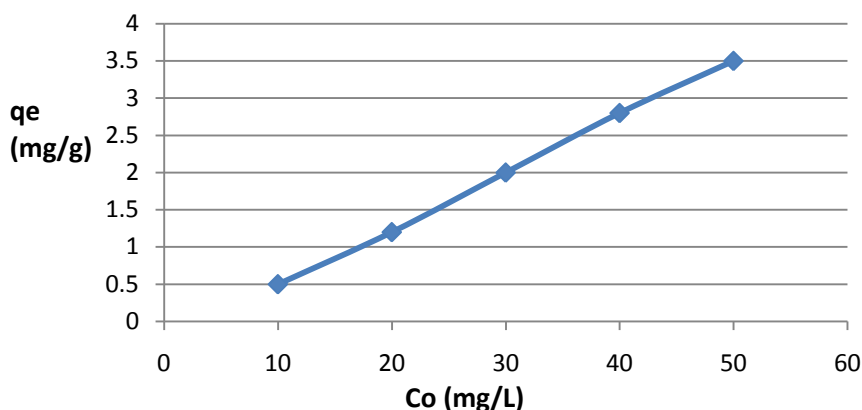


Figure 1: Effect of initial concentration on the adsorption

To investigate the effect of initial metal ion concentration, experiments were conducted with known initial concentrations. The contact time was fixed at 60 minutes for these studies. The initial concentration influenced the driving force for mass transfer, and this study provided insights into the thermodynamics of adsorption under varying concentration gradients (Langmuir, 1918). The amount of metal ions absorbed is a function of the initial concentration of the metal ion, making it an important factor in effective adsorption. The absorption capacity of PSAC for the metal ions was studied. A higher concentration in a solution implies a higher concentration of metal ion to be fixed on the surface of the adsorbent (Barkaet al., 2013). The concentration of the heavy metals in the wastewater was determined using Atomic Absorption Spectrometer.

The effect of the initial lead concentration Pb (II) on adsorption shown in Figure 2, increase in metal ion concentration lead to a corresponding increase in metal adsorption by PSAC. In this study, 5 different doses of lead ion concentrations ranging from 10 to 50mg/l were considered. It was observed that as the initial concentration increases, the adsorption of lead ion onto PSAC also increases. This was due to presence on adsorbent surface active binding sites to accommodate Pb (II) in the solution and higher concentration gradient which acts as a driving force to overcome resistances to mass transfer of metal ions between the aqueous phase and the solid phase. The availability and ability of surface functional group to bind metal ion enhances Pb (II) adsorption (Cheraghiet al., 2015)

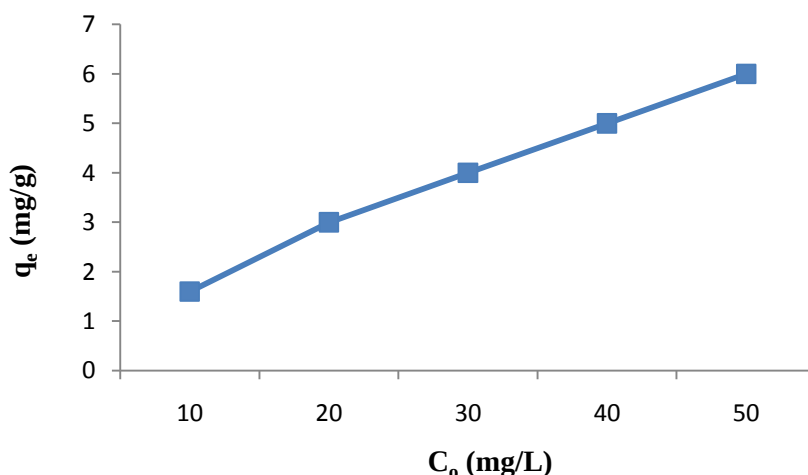


Figure 2: Effect of initial lead ion concentration of solution on PSAC at 30°C, contact time of 60mins and dosage 0.5g per 100ml solution of initial concentration.

3.1.2 Effect of Contact Time

Initially, metal ion adsorption was relatively low because most binding sites were unoccupied during the short initial period. However, adsorption increased rapidly as the

contact time increased (Borua et al., 2019). The changes in lead ion uptake over time were analyzed to fit the kinetic models. The adsorption capacities of periwinkle shell over time are illustrated in Figure 3.

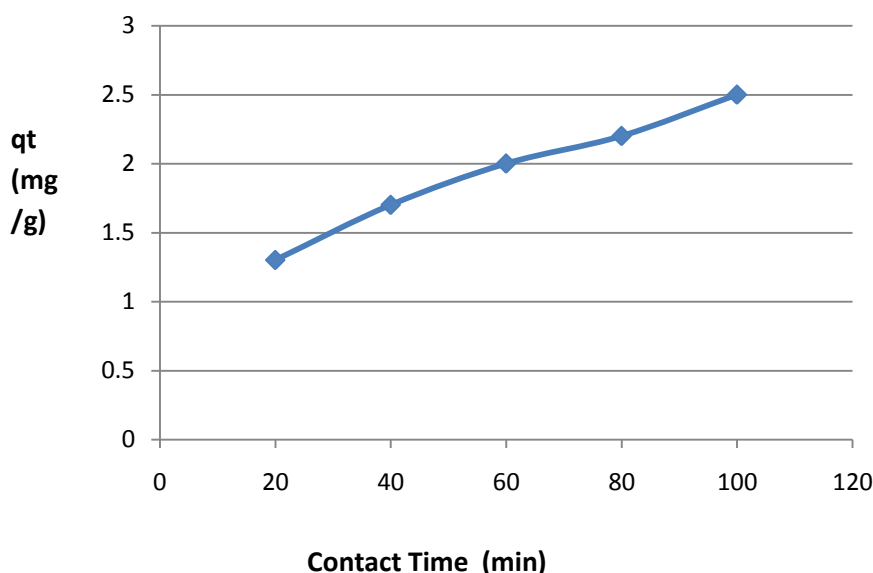


Figure 3: Effect of contact time on the adsorption

The effect of contact time on the adsorption of heavy metal ions from wastewater was determined. An initial increase in percentage removal with increased contact time within a time range in minutes was observed, and whether adsorption becomes fairly stable over time. Equilibrium removal may be achieved after which further increases in contact time may not result in significant adsorption. Initially, adsorption is controlled mainly by diffusion from the bulk to the surface of the adsorbent, whereas, later, adsorption is probably an attachment-controlled process due to the presence of fewer active sites (Dawodu and Akpomie, 2014).

The effect of contact time on the adsorption of heavy metals was studied by withdrawing samples at predetermined time intervals (in minutes). The samples were filtered,

and the residual heavy metal concentrations were determined. This time-dependent study will help in elucidating the adsorption kinetics and the thermodynamic feasibility of the process (Ho and McKay, 1998).

The adsorption potentials of the PSAC over time are presented in Figure 4. At first, metal ion adsorption was lower due to the fact that more binding sites remained free when allowed for a short period of time and increased rapidly as the time of adsorption increases. At 10 mins, it showed lowest adsorption, which increased to saturation at about 75 mins over time, and after that, the absorption remained almost the same. The variation in uptake of the lead ions with time was used in fitting the kinetic models (Aditya and Hossain, 2018).

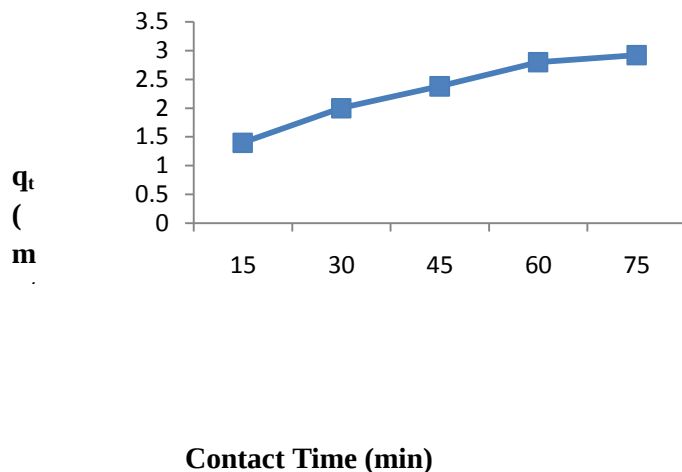


Figure 4: Effect of time on adsorption of lead on PSAC at 30°C, initial lead ion concentration of 20mg/l and adsorbent dose of 0.5g per 100ml solution.

3.1.3 Effect of Adsorbent Dosage

Adsorbent dosage has proved to be a useful parameter for determining the capacity of an adsorbent for a given initial metal ion concentration. A higher adsorbent dose may also provide more active sorption sites, so that sorption sites remain unsaturated, leading to their full utilization (Barkaet al., 2013).

The effect of PSAC dosage on the percentage removal of Pb (II) from aqueous solutions are shown in Figure 5. An increase in the amount of PSAC in the metal solution yielded a corresponding increment in lead adsorption. Possibly the increase in the amount of PSAC increased the availability of free binding sites or exchanging functional groups facilitated lead adsorption from the solution (Boruaet al., 2019).

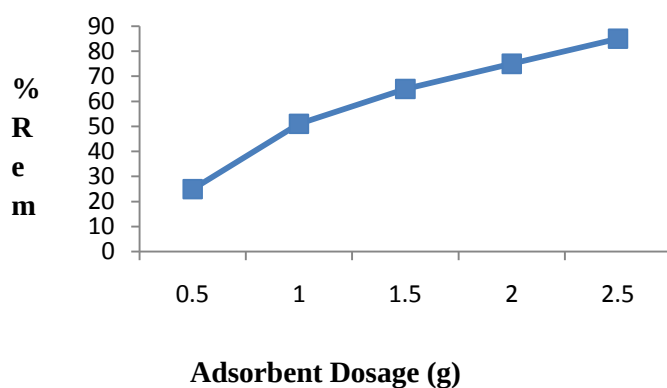


Figure 5: Influence of PSAC dose on adsorption of Pb at 30°C, initial lead ion concentration of 20mg/l, and contact time of 60mins

Adsorbent dosage has proved to be a useful parameter for determining the capacity of an adsorbent for a given initial metal ion concentration. The percentage removal of Fe (III) ion in this study, increased with increasing adsorbent dosage from 1-5g, due mainly to an increase in the number of available exchangeable binding sites for metal ion sorption. However, the

equilibrium sorption capacity per unit mass of the adsorbent decreased considerably with increase in sorbent dose for both metal ions as shown in Figure 6. This may be due to a decrease in the total sorption surface area available to the metal ions, possibly caused by the aggregation/agglomeration of sorption sites; as a result the sorption capacity of the adsorbent is not fully utilized. Conversely, a

higher adsorbent dose may also provide more active sorption sites, so that sorption sites remain

unsaturated, leading to their full utilization as reported by (Barkaet al., 2013).

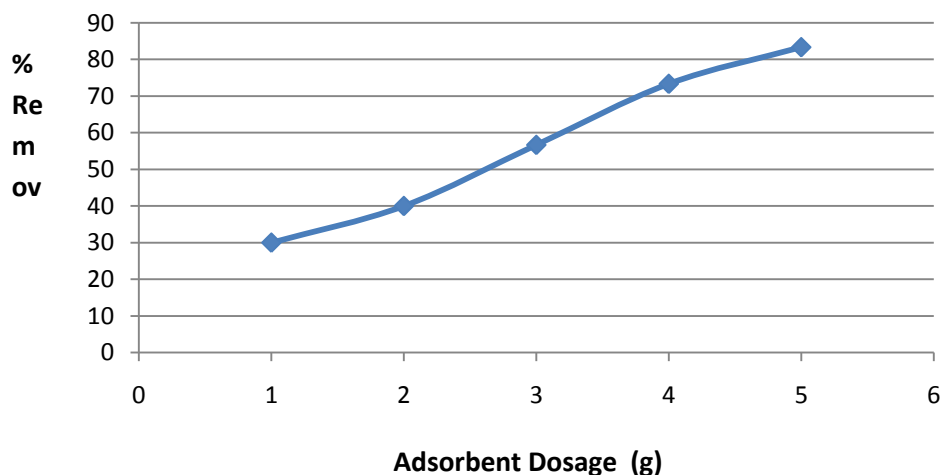


Figure 6: Effect of adsorbent dosage on the adsorption

3.1.4 Effect of Particle Size

The particle size of an adsorbent has a tremendous effect on the sorption process. The increase in the percentage removal of Fe (III) ions with decreasing sorbent particle size in this study from 500 μ m to < 75 μ m as shown in Figure 7; is attributable to an increase in the surface area of the

adsorbent, thus increasing available sites for metal ions binding. The breaking up of larger particles into smaller ones tends to open tiny cracks and channels on the adsorbent surface area, resulting in greater accessibility and better diffusion of the metal ions (Dawoduand Akpomie, 2014)

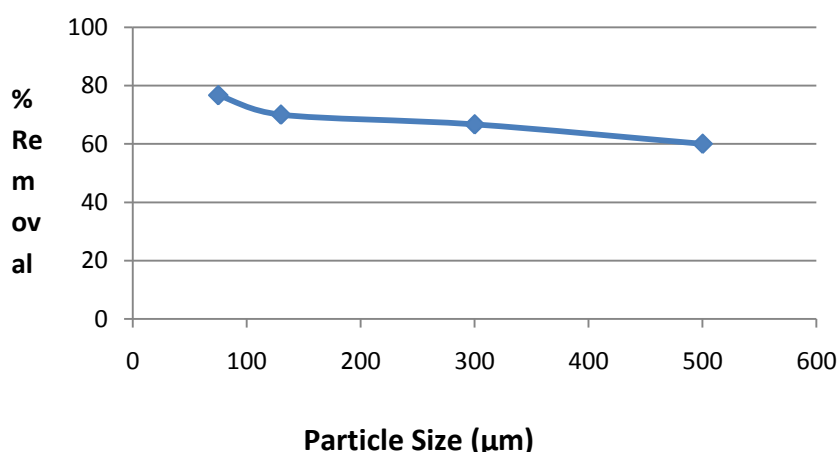


Figure 7: Effect of particle size on the adsorption

The particle size of an adsorbent has a tremendous effect on the sorption process. The breaking up of larger particles into smaller ones tends to open tiny cracks and channels on the adsorbent surface area, resulting in greater accessibility and better diffusion of the metal ions (Boruaet al., 2019).

The effect of particle size on the removal efficiency of Pb (II) from solution on particle sizes of PSAC was shown in Figure 8. It is obvious that Pb (II) removal progressed with a reduction in particle size due to the larger total mass transfer surface area available. The breaking up of larger particles into smaller ones tends to open tiny cracks

and channels on the adsorbent surface area, resulting in greater accessibility and better diffusion of the metal ions (Boruaet al., 2019). All further analyses on the adsorption of Pb (II) by the

PSAC were carried out by maintaining the particle size of 75 μ m. Similar result was shown by other researchers (Cheraghiet al., 2015).

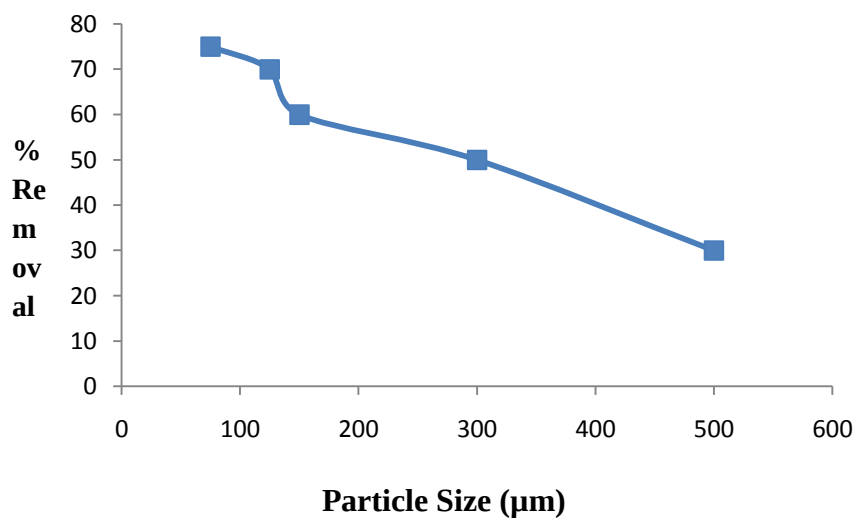


Figure 8: Effect of PSAC sizes on the removal capacity of Pb (II), 20mg/l, 0.5g.

IV. ADSORPTION ISOTHERM MODELS

Adsorption studies were done as shown in Figures 9, 10 and 11 which shows the graphical

representation of Langmuir, Freundlich and Temkin isotherm models.

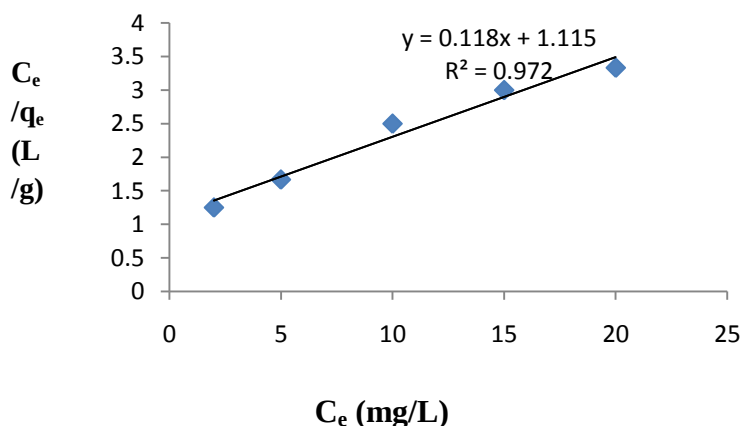


Figure 9: Langmuir Isotherm Model for the adsorption of Pb (II) ion

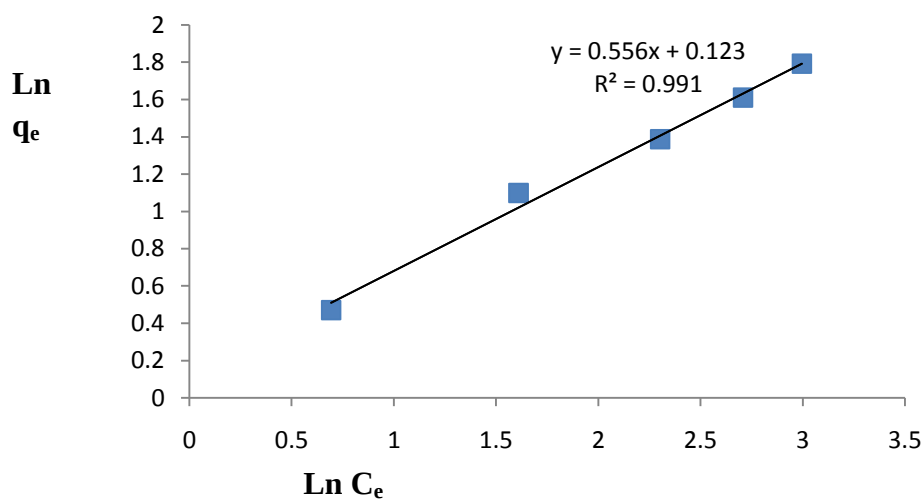


Figure 10: Freundlich Isotherm Model for the adsorption of Pb (II) ion

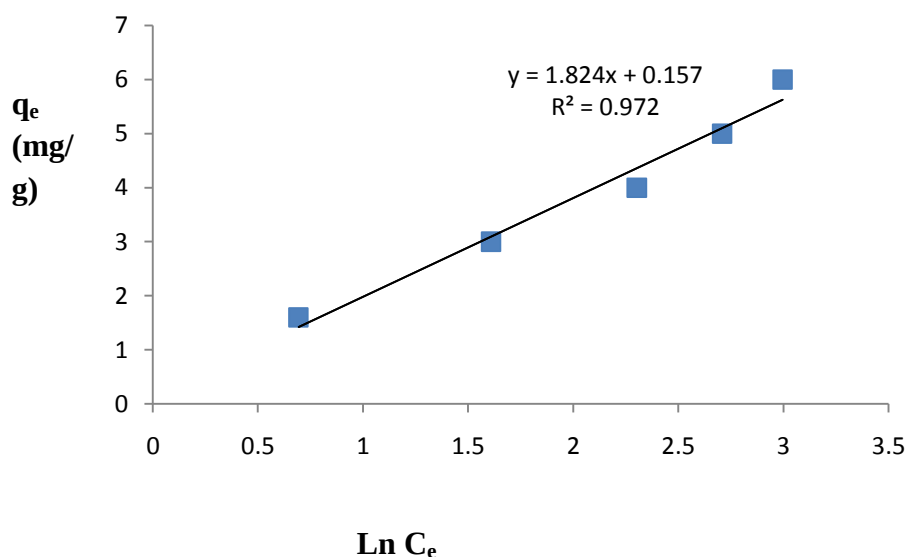


Figure 11: Temkin Isotherm Model for the adsorption of Pb (II) ion

Table 1: Isotherm parameters for lead ion adsorption unto PSAC at 30°C.

Isotherm	Parameters		
Langmuir	Q _{max} (mg/g) 8.4245	K _L (L/mg) 0.1063	R ² 0.9720
Freundlich	K _F 1.1313	N 1.7962	R ² 0.9919
Temkin	K _T (Lmg-1) 1.0902	b _T (L mol-1) 1357.72	R ² 0.9727

Three different isotherm model equations: Langmuir, Freundlich and Temkin were chosen to predict the adsorption capacity and fitness on of experimental equilibrium data. Figures 9, 10 and 11 above, showed the plots of isotherm parameters for PSAC at 30°C. Table 1 showed the values of maximum adsorption capacities (Q_{max}), correlation coefficients (R^2), and other constant parameters for three isotherm models equations for the adsorption process at 30°C.

For the Langmuir isotherm, the Q_{max} value of 8.4245 mg/g was obtained and the value of R^2 of 0.9720. It shows that this isotherm fits well with the experimental information (Table 1). A significant parameter of the Langmuir isotherm is the separation factor (R_L), for which at all the initial concentrations, it has a value greater than 0 and less than 1 ($0 < R_L < 1$) indicating favourable lead-PSAC system adsorption.

The value of n is 1.7962 ($1 < n < 10$: Adsorption is favorable) and $R^2 = 0.9919$ indicating favourable for Freundlich isotherm. Temkin isotherm ($R^2 = 0.9727$). The isotherms can be arranged according to their ability by analyzing all

the most significant parameters (and R^2) to predict the experimental behavior of the PSAC system. With respect to R^2 in this order: Freundlich > Temkin > Langmuir. In view of the highest R^2 values, the Freundlich model is therefore the most suitable for adsorption of lead to PSAC, suggesting sorption takes place on a heterogeneous adsorbent surface (Horsfall et al., 2004).

4.1 Isotherm Models For Fe(III)

The Langmuir isotherm model describes monolayer adsorption onto the surface of an adsorbent with a finite number of identical adsorption sites and no interaction between sites. An essential feature of the Langmuir isotherm can be expressed in terms of a dimensionless separation factor (R_L). Adsorption is said to be favourable if $0 < R_L < 1$ and unfavourable if $R_L > 1$. Table 3 shows high correlation coefficients (r^2) of 0.9579, indicating that the adsorption followed the Langmuir isotherm closely. A favourable adsorption process was, also indicated by the R_L values, that ranged between $0 < R_L < 1$ at all concentrations.

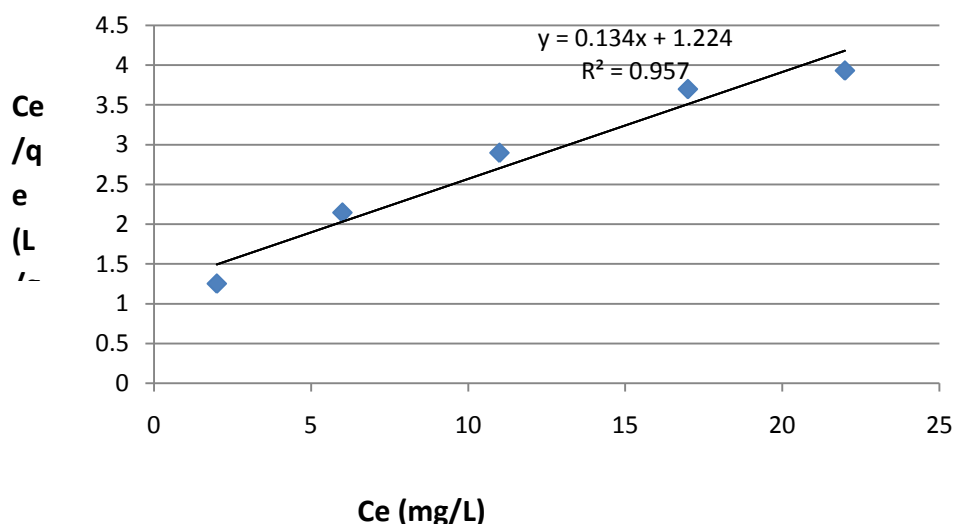


Figure 12: Langmuir isotherm model for the adsorption of Fe (III) ions onto PSC

The Freundlich model is based on the assumption that sorption takes place on a heterogeneous adsorbent surface, where the sorption energy distribution decreases exponentially. As can be seen in Table 3, the r^2 value Fe (III) ions, 0.9978 was higher than that of

the Langmuir model, indicating a good fit of the Freundlich model to the adsorption process. The value of n obtained lie between 1 and 10, suggesting favourable adsorption (Slejko, 1985).

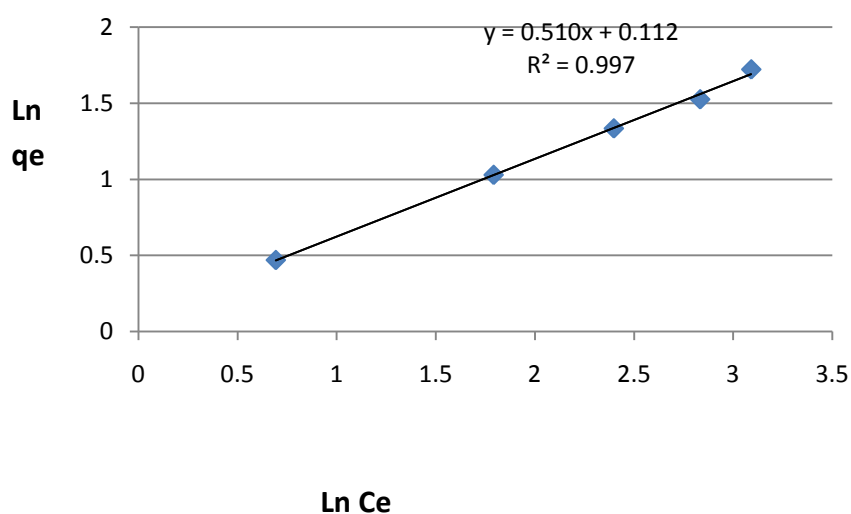


Figure 13: Freundlich isotherm model for the adsorption of Fe (III) ions onto PSC

The Temkin model is based on the assumption that the free energy of adsorption is a function of the surface coverage; The constants A and B as shown in Table 3 were determined from

the plot of q_e versus $\ln C_e$. The r^2 values obtained (Table 3) showed a bad fit for the data compared to Langmuir and Freundlich models.

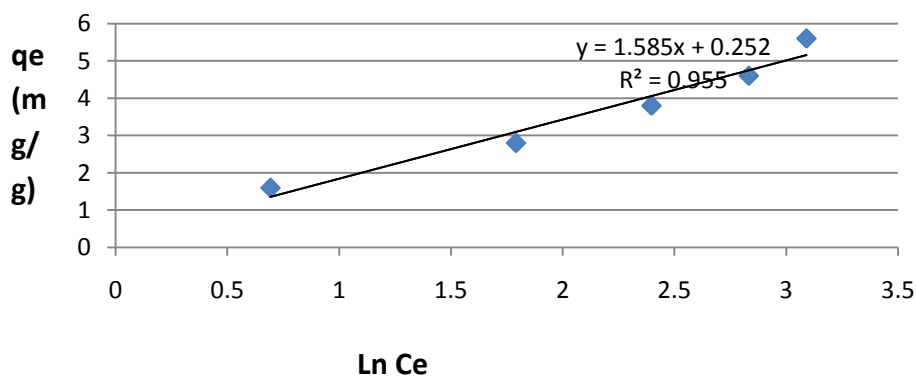


Figure 14: Temkin isotherm model for the adsorption of Fe (III) ions onto PSC

Table 3: Isotherm parameters for Fe (III) ion adsorption

Isotherm	Parameters		
Langmuir	$Q_{\max}(\text{mg/g})$ 7.4460	$K_L(\text{L/mg})$ 0.1096	r^2 0.9579
Freundlich	K_F 1.1190	N 1.9573	r^2 0.9978
Temkin	$K_T(\text{Lmg}^{-1})$ 1.1723	$b_T(\text{L mol}^{-1})$ 1358.50	r^2 0.9551

V. ADSORPTION KINETIC MODELS

The kinetics of the mechanism of adsorption were explored with the following kinetic

models: - pseudo-first order kinetics model and pseudo second order kinetic model are represented in Figure 15 and 16 respectively.

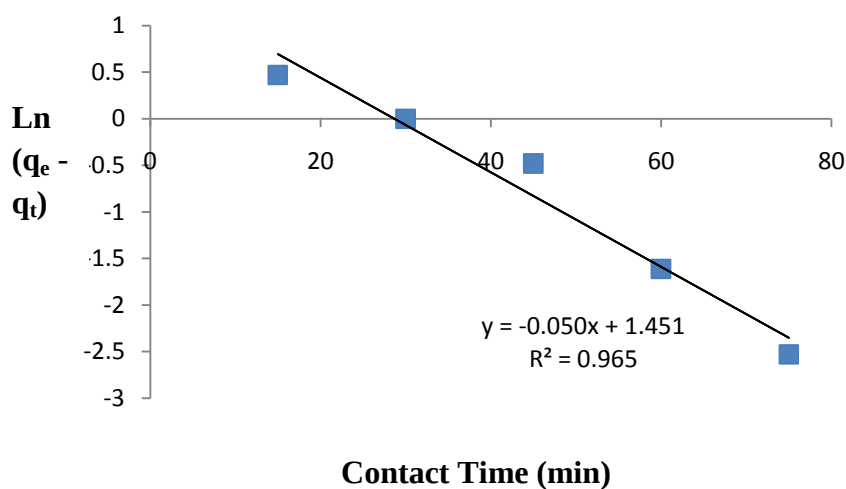


Figure 15: Pseudo first order kinetic model

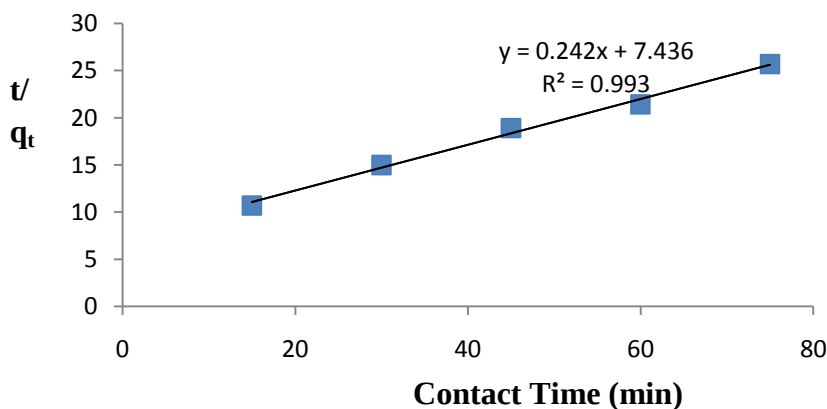


Figure 16: Pseudo second order kinetic model

Table 4: Kinetic models constant values for lead ion adsorption unto PSAC at 30°C.

Kinetics	Parameters		
Pseudo first order	k ₁ 0.0507	q _e _{cal.} 4.2699	R ² 0.9652
Pseudo second order	k ₂ 0.0079	q _e _{cal.} 4.1237	R ² 0.9937

In describing the kinetic adsorption of Pb (II) onto the PSAC, the two kinetic models in a

nonlinear form: pseudo first order model and pseudo second order models was used to analyze

the experimental data. The non-linear regression analysis (correlation coefficient, R^2) was used to analyze fitness of the kinetic models. Table 4 above presented pseudo-first order and pseudo-second rate constants and calculated q_e .

The correlation coefficient obtained when the pseudo-first order model was applied to metal ions adsorption have very low values of (k_1 of 0.0507, R^2 0.9652) i.e. the calculated adsorption capacities value shows great difference by comparison with expected value. The Pseudo-second order correlation coefficient (k_2 0.0079, R^2 0.9937). A comparison between the correlation

coefficient (R^2) showed that pseudo second order kinetic model has the best fitting. This kinetic model suggests that the Pb (II) metal ion adsorption is controlled by chemisorption mechanism (Adewoye et al., 2017).

5.0 Thermodynamic Studies

The thermodynamic parameters for the adsorption of Fe^{3+} ions calculated from Figure 17 and appropriate equations are given in Table 5. This could be seen in the relationship between $\ln K_e$ and $1/T$ as showed in Figure 17.

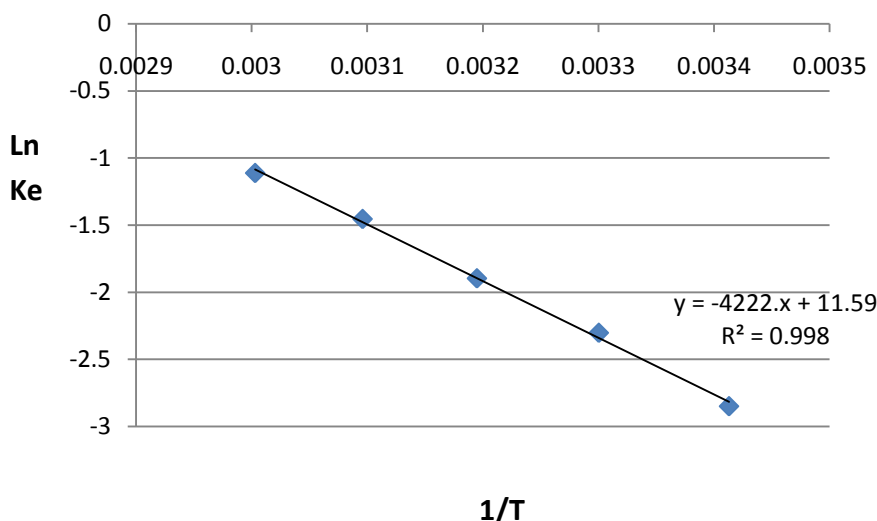


Figure 17: Thermodynamic study for the adsorption of Fe (III) ions onto PSC

Table 5: Thermodynamic parameters for the adsorption of Fe (III) ion onto periwinkle shell carbon

Temperature (°C)	ΔG° (kJ / mol)	ΔH° (kJ / mol)	ΔS° (J / mol· K)
20	-3081.41		
30	-3908.06	96.38	35.11
40	-4936.84		
50	-5800.54		
60	-6940.48		

The Gibb's free energy change of the process (ΔG°) were all negative and decreased with increase in temperature at all temperatures the experiments were conducted. This indicates that the adsorption mechanism of periwinkle shell activated carbon toward iron (III) is spontaneous in nature and thermodynamically favorable and more favorable at higher temperatures (Zakiet al., 2000). This is in agreement with the favorable adsorption process predicted by the langmuir isotherm model which fitted most the adsorption isotherm data. The enthalpy change of the process (ΔH°) is positive,

hence the process is endothermic. Therefore, adsorption capacity increase with temperature increase as predicted by the values of the maximum adsorption capacities (Q_0) from the langmuir isotherm model and in agreement with the Gibb's free energies obtained. The entropy change of the process (ΔS) is positive. Entropy is defined as the degree of freedom or chaos of a system. The positive value of the entropy change indicates that the degree of freedom or randomness at the solid/liquid interface of the activated carbon increases during the adsorption of Fe^{3+} ions onto

the active sites of the periwinkle shell activated carbon. This is in agreement with the work of Saha and Chowdhury(2011) and explained to be because the mobility of adsorbate ions/molecules in the solution increase with increase in temperature and that the affinity of adsorbate on the adsorbent is higher at high temperatures.

VI. CONCLUSION

The adsorption performance of periwinkle shell activated carbon for the removal of heavy metals (Pb(II) and iron (III) oxide) from aqueous solution was investigated. The periwinkle shell activated carbon samples were characterized and examined for its potential to adsorb metal ions (Pb (II) from wastewater in a batch mode. The adsorption rate increased with increase in contact time and adsorbent dosage aqueous solution and decrease with increase in particle size. The study revealed that the adsorption process is influenced by initial concentration of metal ions, contact time, adsorbent dosage and particle size. The increase in the initial metal ions concentration reduced the metal uptake of the metal ions while the increase in contact time increased the uptake of metal ions.

The kinetics of Fe^{3+} adsorption onto periwinkle shell activated carbon obeys the pseudo-second-order model, suggesting that chemisorption is the rate-limiting step in the adsorption process. The adsorption equilibrium data fitted the Freundlich isotherm model in comparison to the Langmuir and Temkin isotherm models, therefore adsorption took place on a heterogeneous adsorbent surface of periwinkle shell activated carbon. The kinetic modelling performed showed that the adsorption process was better described using pseudo-second order kinetics as it best fits the. The dimensionless separation factor or equilibrium parameter (R_L) values calculated were greater than zero but less than 1, which showed that the adsorption process was favorable (Periwinkle shell activated carbon was an efficient adsorbent for heavy metals removal). The thermodynamic parameters obtained indicated that the adsorption process of Fe^{3+} ions was spontaneous (negative values of ΔG°) and endothermic in nature (positive ΔH°). The positive value of the entropy change (ΔS°) showed increased randomness with adsorption. Hence the adsorption process is thermodynamically favorable. The results show that periwinkle shell activated carbon is a promising adsorbent for the removal heavy metal ions from waste water. The use of periwinkle shell activated carbon provides a sustainable and cost-effective alternative to conventional adsorbents,

contributing to waste utilization and environmental protection. The results indicate that periwinkle shell activated carbon could be effectively employed in wastewater treatment systems to mitigate heavy metal pollution. The following are recommendations are made: Optimize Contact Time and Adsorbent Dosage: Fine-tuning these parameters for improved efficiency, a comprehensive chemical analysis should be carried out on the adsorbent (periwinkle shell) to know the chemical composition of the adsorbent, explore compatibility with current water treatment systems.

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