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**Research Article**

# Removal of Pb(II) and Ni(II) from Wastewater Using Activated and Unactivated Carbon Derived from the Bark of *Annona senegalensis*

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## Abstract

*The removal of Pb(II) and Ni(II) from wastewater using activated and unactivated Annona senegalensis bark was investigated. Chemical activation of the carbon with 85% phosphoric acid ($H_3PO_4$) was employed for the preparation of activated carbon. The effects of pH, adsorbent dosage, particle size, contact time and temperature were studied to identify the adsorption capacity of the activated and unactivated carbon. The percentage removal of Pb(II) and Ni(II) were found to be dependent on all the factors studied. For the activated carbon, the effect of adsorbent dosage indicated that the percentage removal for Pb(II) ranged from 36.67 to 73.33% and Ni(II) from 31.48 to 69.20%. For unactivated, Pb(II) ranged from 27.50 to 65.42% and Ni(II) ranged from 19.51 to 59.39%. The effects of particle size, contact time and temperature showed similar trend except for pH that showed increasing removal of Pb(II) and Ni(II) with increasing pH from 2.0 to around 7.0 before showing a decline. The equilibrium data was best described by Freundlich followed by Temkin model. Pseudo-second order and Elovich equations gave better fits of the adsorption process compared to pseudo-first order. It was concluded that activated and unactivated carbon derived from Annona senegalensis bark can be used as low-cost adsorbents for the removal of Pb(II) and Ni(II) from water.*

**Keywords:** Wastewater, adsorption, chemical activation, carbonization, adsorbent dosage.

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## Introduction

The increased and widespread heavy metal contamination of drinking water is posing significant hazards to human health as a result of their carcinogenic property, toxicity and non-biodegradability (Roghani *et al*., 2016). These elements get into the environment by natural and anthropogenic means such as natural weathering of the earth's crust, mining, soil erosion, industrial discharge, urban runoff, sewage effluents, pests or disease control agents applied to plants, air pollution fallout etc (Harvey *et al*., 2002 and Ming-Ho, 2005). Although some individuals are primarily exposed to these contaminants in the workplace, for most people the main route of exposure to these toxic elements is through food and water without any beneficial effects in humans (Draghici *et al*., 2010).

In the last few decades, a wide range of physical and chemicals methods such as chemical coagulation using alum and ferric salts followed by filtration, electrochemical precipitation, ultra filtration, ion exchange, reverse osmosis etc have been used for the treatment of water (Fatoki and Ogunfowokan, 2002; Evans and Li, 2003; Nomanbhay and Palanisamy, 2005). However, there are some limitations associated with these conventional methods of water treatment.

In recent years, there has been research focusing on appropriate technology which is considered to be simple, efficient, eco-friendly, relatively cheap, more convenient for rural application and for regeneration (Chammui et al., 2014) for the removal of contaminants from water. Several adsorbents such as feldspar (Yazdani et al., 2016), groundnut shell (Aji, 2017), cow bone (Moreno *et al*., 2010), corn cob and cocoa nut shells (Singh and Waziri, 2019) etc have been investigated for their ability to remove heavy metals from water.

In this study, *Annona senegalensis* bark has been evaluated as adsorbent for the removal of Pb(II) and Ni(II) from wastewater. The effects of pH, temperature, adsorbent dosage, initial metal ion concentration and contact time were examined to find optimum adsorption conditions in batch experiment.


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## Materials and Method

### Sample Collection

The bark of *Annona senegalensis* which were used in the production of activated and unactivated carbon were gotten from bushes around Maikunkele in Minna, Niger State, Nigeria while the stream water sample was collected from a stream in Gwagwalada Area Council of Federal Capital Territory, Abuja, in a plastic container packed with ice block in a cooler.

### Sample preparation

The bark of *Annona senegalensis* obtained were thoroughly cleaned, dried in the sun and manually reduced to small sizes of approximately 1.5 to 2.0 cm.

### Carbonization

Carbonization was carried out in the pre-heated temperature at 600˚C for 45 minutes using the method described by Gimba *et al* (2001).

### Chemical Activation

The carbon obtained was soaked in 500 ml of 10 % phosphoric acid and was left to stand for 24 hours. The carbon was then filtered and thoroughly washed using distilled water until the pH of 6.86 was achieved. It was then dried in oven set at 105˚C. The activated carbon was then charged in the muffle furnace set 600˚C for 10 minutes, cooled to room temperature and stored in a desiccator.

### Characterization of the carbon

The activated carbon was characterized according to Pore volume (Nurul'ain, 2007), Bulk density (Cigdem, 2005), Moisture content (Nurul'ain, 2007), Ash content (Bhole and Ramteke, 2011), Volatile organic matter (Bhole and Ramteke, 2011).

### Determination of Lead and Nickel Ions

The metal ions present in the sample were determined using Atomic Absorption Spectrophotometer (Model: Thermo SCIENTIFIC, iCE 3000 SERIES, manufactured in the USA).

### Batch Adsorption Experiment

The batch adsorption experiment was carried out to check the effects of pH, effects of temperature, effects of contact time, adsorbent dosage and contact time.


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**Effects of pH**

The effect of pH on the adsorption was conducted within the pH range of 2 to 8. The pH of the water sample was adjusted to the desired value by drop wise addition of 0.0M HCl and/or 0.5M NaOH (Adedirin *et al.*, 2013). This was done by contacting 0.5g each of activated and unactivated carbon with 100cm$^3$ of the water sample in separate 250cm$^3$ conical flasks and the mixture was shaken thoroughly using mechanical shaker for 90 minutes. The mixture was then filtered and the amount of lead and nickel adsorbed was calculated using equation 1.

$$q_t = \frac{(C_0 - C_t)V}{M} \quad (1)$$

In equation 1, $q_t$ is the amount metal ion adsorbed per gram of carbon, $C_o$ is the initial concentration of metal ion, $C_t$ is the residual concentration of metal ion at time t, V is the volume of solution used and M is the mass of carbon used.

**Effects of Temperature**

The batch adsorption process was studied at different temperatures of 30 - 45˚C in order to investigate the effects of temperature on the adsorption process. This was done by contacting 0.5g of the carbon with 100cm$^3$ of the water sample at optimum pH for 90 minutes. The solution was then filtered and the pH taken for AAS analysis. The amount of lead and nickel ions adsorbed was then calculated.

**Effects of Adsorbent Dosage**

0.5g each of activated and unactivated carbon was weighed into 250 ml different conical flasks containing 100 cm$^3$ of water sample and the mixture was continuously shaken for 90 minutes using a mechanical shaker. At the end of the 90 minutes, the mixture was filtered and the filtrate taken for AAS analysis. The amount of lead and nickel ion adsorbed was then calculated. This was repeated for adsorbent dosage of 1.0g, 1.5g, 2.0g and 2.5g.

**Effects of Initial Metal Ions Concentration**

0.5g each of activated and unactivated carbon were weighed and mixed with 100cm$^3$ solution of initial concentration of 10, 15, 20, 25 and 30 ml/L the mixture were shaken thoroughly for 90 minutes using mechanical shaker. At the end of the 90 minutes, the mixture was filtered and the concentration determined using Atomic Absorption Spectrophotometer (AAS). The different between the initial concentration and final


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concentration was recorded as the amount (concentration) adsorbed for each concentration.

**Effects of Contact Time on Sorption**

This was done by weighing 0.5g each of activated and unactivated carbon into 250 ml conical flask containing 100 $cm^3$ of the water sample. The mixture was shaken using mechanical shaker for 30 minutes. At the end of the 30 minutes, the solution was then filtered and the filtrate taken for AAS analysis. The amount of adsorbed lead and nickel was then calculated. This was repeated for 60, 90, 120 and 150 minutes.

**Adsorption Isotherms**

Freundlich, Langmuir and Temkin adsorption isotherms models were used to explain effect of concentration on adsorption process.

**Adsorption Kinetics**

First order, second order and Elovich equation were employed to study the kinetics of the adsorption process to shed light into the mechanism of the process.

## Results and Discussion

**Characteristic of Adsorbent**

Presented in Table 1 are the physicochemical properties of activated carbon produced in this study.

Table 1: Physicochemical properties of phosphoric acid activated carbon derived from the bark of *Annona senegalensis* compared with that prepared from Groundnut shell Aji (2017).

| Parameters | Bark of *Annona senegalensis* | Groundnut shell |
|---|---|---|
| pH | 6.90 | 6.80 |
| Pore volume ($cm^3$) | 0.91 | - |
| Bulk density ($g/cm^3$) | 0.78 | 0.42 ($g/cm^3$) |
| Moisture content (%) | 4.85 | 15.00 (%) |
| Ash content (%) | 6.35 | 6.00 (%) |
| Volatile organic matter (%) | 5.92 | 11.00 (%) |
| Surface Area ($m^2/g$) | 843.00 | 975.00 ($m^2/g$) |


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**Effect on experimental conditions on adsorption process**

Illustrated in Fig. 1a and Fig.1b were the variation in the adsorption processes of activated and un-activated carbon produced in this study as pH of the adsorbate (water sample) changes. Increases in the adsorption of these contaminants were observed as the pH of the water sample increases. These increment in percentage of $Pb^{+2}$ and $Ni^{+2}$ adsorbed as the pH of the water sample increased can be explain on the basis of a decrease in the competition between proton and the cation in the sample for the adsorption sites on the surfaces of the adsorbents. Also, decrease in positive surface charge, which result in lower coulombic repulsion of the sorbing cation also contributed to the observed trend (Dianaki-Tilaki et al., 2004). In this study, highest adsorption of $Pb^{+2}$ and $Ni^{+2}$ ions was observed between the pH of 6.5 and 7.0 as depicted in Fig 1.a and Fig.1b. Other investigation have reported similar trend for the sorption of contaminants onto different biomass systems (Kumar et al., 2010).

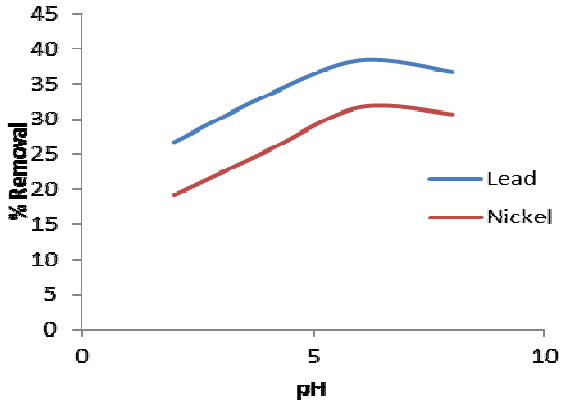


Fig.1a: Changes in percentage removal of $Pb^{+2}$ and $Ni^{+2}$onto activated carbon derived from *Annona senegalensis* bark as pH of water sample changes

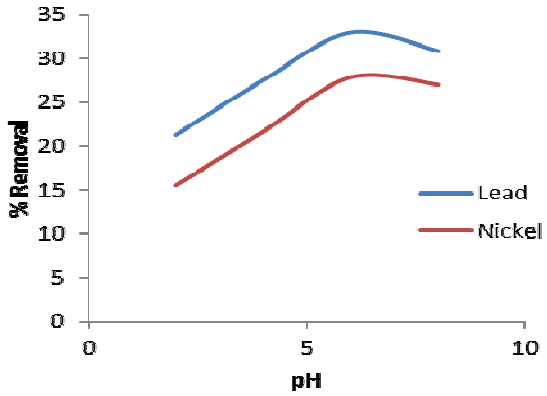


Fig.1b: Changes in percentage removal of $Pb^{+2}$ and $Ni^{+2}$onto unactivated carbon derived from *Annona senegalensis* bark as pH of water sample changes

The effects of temperature change on the adsorption of $Pb^{+2}$ and $Ni^{+2}$ ions onto the adsorbent used in this study were shown in Fig.2a and b. These figures showed increase in temperature resulted in increment in the amount of $Pb^{+2}$ and $Ni^{+2}$ ions adsorbed. With increase in temperature, the attractive forces between the adsorbents' surfaces and contaminants became stronger and this usually resulted in increase in extent of adsorption (Ho, John and Forster, 1995). This may also be attributed to increased


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penetration of contaminants inside micropores at higher temperatures and the creation of new active sites (Al-Degs *et al.*, 2007).

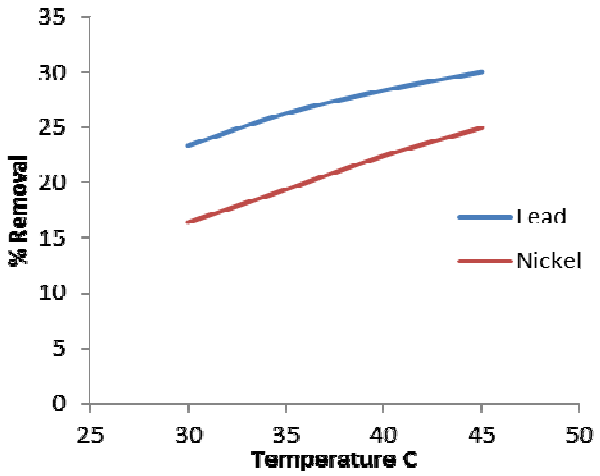


Fig. 2a: Effect of varying temperature on the adsorption of Pb(II) and Ni(II) onto activated carbon derived from *Annona senegalensis* bark

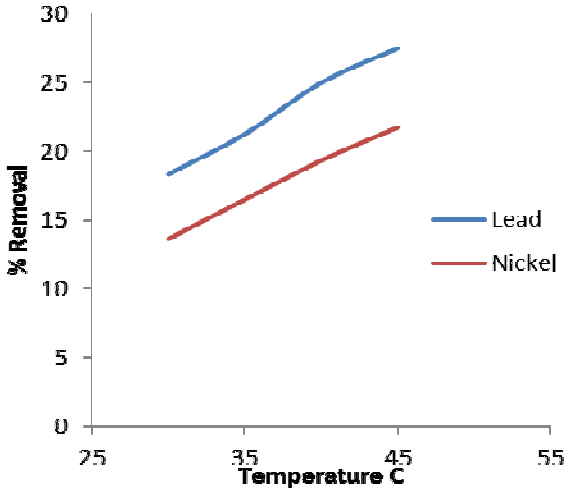


Fig. 2b: Effect of varying temperature on the adsorption of Pb(II) and Ni(II) onto unactivated carbon derived from *Annona senegalensis* bark

Changes in adsorption processes as the dosage (amount of adsorbent) of activated and un-activated carbon produced in this study were shown in Fig.3a and Fig.3b respectively. The figures showed that percentage adsorption of lead and nickel ions increased as the adsorbent dosage increased but the amount adsorbed per unit mass of the adsorbent decreased considerably as the dosage was increased further (see Table 2. The decrease in unit of adsorption as the adsorbent was increased was basically due to adsorption sites remaining unsaturated during the adsorption reaction (Bulut and Aydin, 2005). The figures revealed a definite increase in the adsorption capacity with adsorbent dosage of 0.5g to 1.5g after which the percentage increase in the adsorption of contaminants was observed to be gradual as the adsorbent dosage was increased from 1.5g to 2.0g and 2.5g. The definite increase in the adsorption capacity with adsorption dosage from 0.5g to 1.5g may be attributed to larger number of available adsorption sites favoring the enhanced uptake of contaminants (Karthikeyan *et al.*, 2005). The amount of lead and nickel ions adsorbed with activated carbon were higher than those adsorbed with unactivated carbon.


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Presented in Fig. 4a and 4b were the rate of adsorption of $Pb^{2+}$ and $Ni^{2+}$l by adsorbent used in this study as contact time increases. Percentages of metal ion adsorbed by the adsorbent produced in this study were shown to increase with increase in contact time.

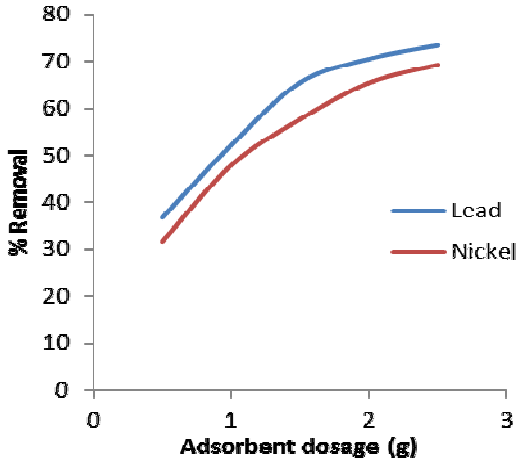


Fig 3a. Effect of varying adsorbent dosage on the adsorption of Pb(II) and Ni(II), onto activated carbon produced from the bark of *Annona senegalensis*

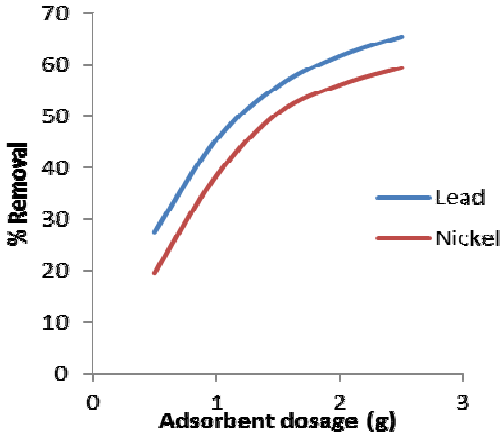


Fig 3b. Effect of varying adsorbent dosage on the adsorption of Pb(II) and Ni(II) onto unactivated carbon produced from the bark of *Annona senegalensis*

As the time was increase by 30 minutes, the adsorption of lead and nickel ions also increased until around between 120 to 150 minutes.The increase in the adsorption of these metal ions as the time was increase from 30 to 90 minutes could be attributed to available binding sites (Mashood *et al*., 2009). As shown at time 120 to 150 minutes the curves appeared somewhat flat meaning that there was no significant change in rate of adsorption with further increase in contact time. The consequence of this is that the adsorption process would not offer additional kinetic advantage when contact time longer than 120 minutes is employed.


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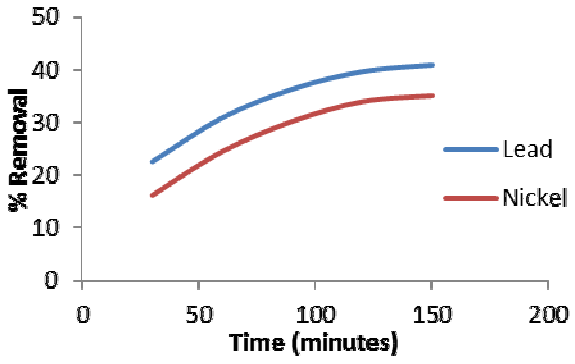


Fig.4a: Effect of varying contact time on the adsorption of Pb(II) and Ni(II) onto activated carbon derived from *Annona senegalensis* bark

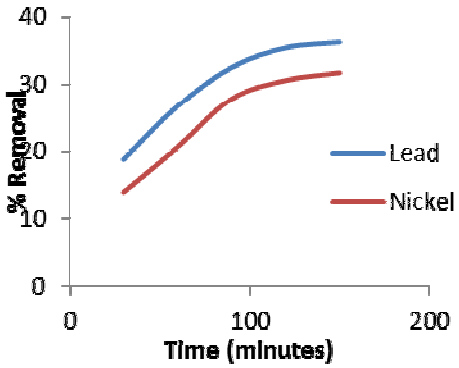


Figure.4b: Effect of varying contact time on the adsorption of Pb(II) and Ni(II) onto unactivated carbon derived from *Annona senegalensis* bark

**Adsorption isotherm**

The equilibrium adsorption data of the effect of concentration on the adsorption of Pb(II) and Ni(II) onto the adsorbent produced in this study were interpreted with Langmiur, Freundlish and Temkin adsorption isotherms represented by equation 2, 3 and 4 respectively.

$$\frac{C_e}{q_e} = \frac{1}{K_L Q} + \frac{C_e}{Q} \qquad (2)$$

In equation 2, $K_L$ (L/g) is Langmuir constant related to the adsorption/desorption energy, Q (mg/g) is the maximum sorption upon complete saturation of the adsorbent surface (Horsfall *et al.,* 2003), qe (mg/L) is amount of metal ion adsorbed per gram of the adsorbent at equilibrium and Ce is the residual concentration of metal ion at equilibrium. The plot of $\frac{C_e}{q_e}$ against $C_e$ gives a straight line. Fig. 5a represents Langmuir plot for activated carbon produced in this study. Similar result was obtained for unactivated carbon (figure not shown). $K_L$ and Q were calculated from the slope and intercept of the Langmuir curves and result presented in Table 2.

$$Log\ q_e = Log\ K_f + \frac{1}{n} Log C_e \qquad (3)$$


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In equation 3, $K_f$ and n are Freundlish constant s that indicates the affinity of the metal ions to the carbon used in the study. Fig. 5b represents Freundlish plot for activated carbon. Similar result was obtained for unactivated carbon (figure not shown) $K_f$ and n were calculated from the slope and intercept of the Freundlish curves and result presented in Table 2.

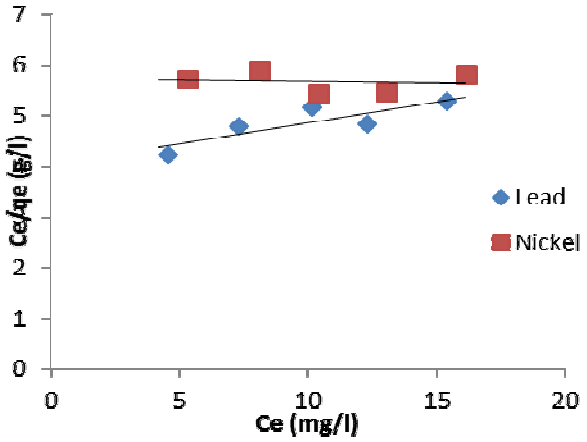


Figure 5a: Langmuir isotherm for the adsorption of Pb(II) and Ni(II) onto activated carbon derived from *Annona senegalensis* bark

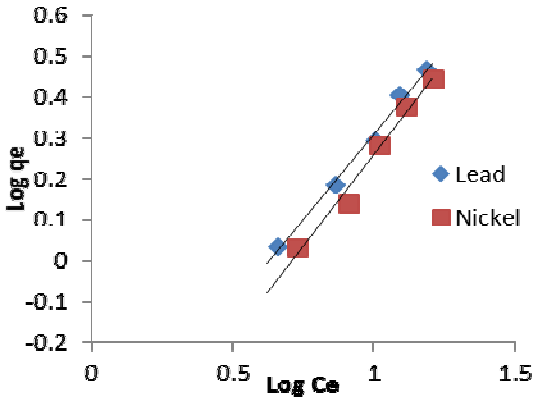


Figure 5b: Freundlich isotherm for the adsorption of Pb(II) and Ni(II) onto activated carbon derived from *Annona senegalensis* bark

$$q_e = \frac{RT}{b_T} InA_T + \frac{RT}{b_T} InC_e \qquad (4)$$

In equation 4, R is gas constant, T is temperature in Kelvin and $A_T$ and $b_T$ are constant determined from the slope and intercept of Temkin curve. The Temkin plot for the adsorption of Pb(II) and Ni(II) ion adsorption onto the activated and unactivated carbon produced in this study are presented in Fig. 6a and 6b respectively. $A_T$ and $b_T$ were calculated from the slope and intercept of Temkin curves and results were presented in Table 2.


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**Table 2: Isotherm parameter for metal ion and carbon derived from *Annona senegalensis* bark.**

| | ACTIVATED CARBON | | UNACTIVATED CARBON | |
|---|---|---|---|---|
| | Pb(II) | Ni(II) | Pb(II) | Ni(II) |
| | | Langmuir | | |
| $Q_0$(mg/g) | 12.09 | 178.6 | 12.18 | 6.035 |
| b | 0.021 | 0.001 | 0.015 | 0.027 |
| $R^2$ | 0.730 | 0.014 | 0.622 | 0.626 |
| | | Freundlish | | |
| $k_f$ | 0.297 | 0.229 | 0.280 | 0.387 |
| 1/n | 0.835 | 0.898 | 0.765 | 0.551 |
| $^*R^2$ | 0.991 | 0.982 | 0.970 | 0.819 |
| | | Temkin | | |
| $A_T$ | 2.467 | 3.314 | 2.892 | 3.323 |
| $b_T$ | 1.619 | 1.447 | 1.820 | 2.115 |
| $^{**}R^2$ | 0.957 | 0.980 | 0.949 | 0.952 |

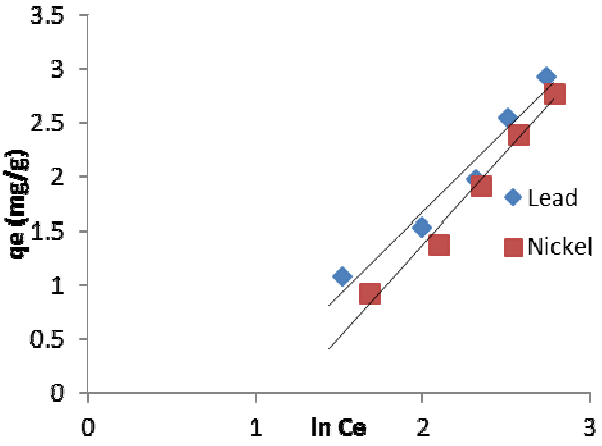


Figure 6a: Temkin isotherm for the adsorption of Pb(II) and Ni(II) from aqueous system onto activated carbon derived from *Annona senegalensis* bark.

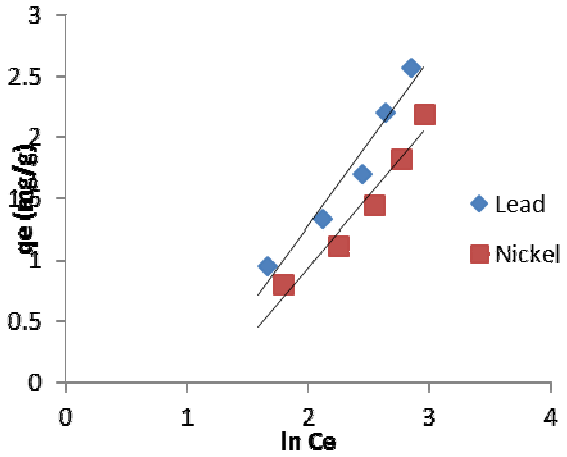


Figure 6b: Temkin isotherm for the adsorption of Pb(II) and Ni(II) from aqueous system onto unactivated carbon derived from *Annona senegalensis* bark


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The results presented in Table 2 showed that Freundlich and Temkin had the highest $R^2$. This implies that a multi-layer adsorption occurred over the surface of the adsorbents and this reflects chemical nature of the adsorption process (El-chaghaby *et al*., 2007).

**Kinetics of the adsorption study**

The rate of adsorption of study metal ions unto adsorbent produced in this study was accessed with pseudo-first order, pseudo-second order and elovich kinetic model represented by equations 5, 6 and 7 respectively. gave correlation coefficients greater than 0.9. This indicated that pseudo-second order kinetic and elovich equation may be used to fit the adsorption data.

Table 3: **Kinetic parameters for metal ion and carbon derived from *Annona senegalensis* bark.**

| | ACTIVATED CARBON | | UNACTIVATED CARBON | |
|---|---|---|---|---|
| | Pb(II) | Ni(II) | Pb(II) | Ni(II) |
| | Pseudo-first order kinectic | | | |
| $Q_e$(mg $g^{-1}$) | 0.0368 | 0.2549 | 0.0312 | 0.1873 |
| $K_1$ ($min^{-1}$) | 0.0071 | 0.0076 | 0.0078 | 0.0108 |
| $R^2$ | 0.7761 | 0.7564 | 0.8902 | 0.7612 |
| | Pseudo-second order kinectics | | | |
| $Q_e$(mg $g^{-1}$) | 0.1387 | 2.6932 | 0.1398 | 2.6089 |
| $K_2$ ($gmin^{-1}mg^{-1}$) | 0.6913 | 0.0758 | 0.5748 | 0.0593 |
| $^{*}R^2$ | 0.9989 | 0.9996 | 0.9974 | 0.9992 |
| | Elovich kinetics | | | |
| α(mg $g^{-1}$) | 13.810 | 10.713 | 15.863 | 20.382 |
| β($mgg^{-1}min^{-1}$) | 28.409 | 3.7272 | 28.090 | 3.1898 |
| $^{**}R^2$ | 0.9554 | 0.9295 | 0.9919 | 0.9264 |

## Conclusion

Removal of Pb(II) and Ni(II) from water using activated and unactivated carbon derived from *Annona senegalensis* bark has been investigated in this work. The result of the study revealed that carbon (activated and unactivated) derived from *Annona senegalensis* bark could be used as effective adsorbent for the removal of Pb(II) and Ni(II) from water. The amount of Pb(II) and Ni(II) adsorbed was found to vary with pH, temperature, contact time and adsorbent dosage. Activated carbon derived from *Annona senegalensis* bark showed better adsorptive capacity than the unactivated carbon


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derived from the same source, therefore it can be deduced that modification had overall enhancement on its physicochemical and adsorptive properties. The adsorptive equilibrium data were fitted to Langmuir, Freundlich and Temkin isotherm models but the Langmuir isotherm gave better fit than Freundlich and Temkin isotherm models. The adsorption data were also fitted to pseudo-first order, pseudo-second order and elovich kinetic equations. However, pseudo-second order best described the adsorption process.

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