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The Kinetic and Thermodynamic Study of the Removal of Selected heavy Metals from a Nigerian Brewery Wastewater Using Activated Carbon From Cheese Wood (alstonia boonei)

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Abstract

An adsorbent was developed from the bark of *Alstonia boonei* for removing Pb, Ni and Fe ion from brewery wastewater using a batch adsorption process. Various process parameters that include adsorbent dosage, contact time, solution pH and temperature were studied. For the effect of temperature, Pb had its highest percentage removal of 80.48% at 40°C, for Ni 90.26% at 60°C and Fe 98.76% at 50°C. The effect of solution pH shows highest percentage removal of 98.39% and 99.85% for Fe and Ni respectively at solution pH of 10 while Pb had its highest percentage removal of 79.9% at the solution pH of 2. The result also shows highest percentage removal of 76.01% and 99.19% at adsorbent dosage of 1.5g for Pb and Fe respectively, and 96.8% at 0.5g for Ni. Also, the highest percentage removal of Pb and Ni was at 60 minutes indicating 82.48% and 96.19% respectively while Fe showed highest percentage removal of 99.12% at contact time of 30 minutes. Negative values of entropy change ΔS° and Gibbs free energy change ΔG° indicate that this adsorption process is spontaneous. The adsorption process follows pseudo second-order kinetics. The surface functional groups and surface area of the adsorbent were examined by Fourier Transformed Infrared Technique (FT-IR) and Brunauer Emmett Teller (BET).

Keywords: Adsorption, Heavy metals, wastewater

1. Introduction

Water bodies have been contaminated due to increase in population and rapid industrialization which is a major concern to the environment (Gunatilake, *et al.*, 2015). These industrial effluents are likewise the principal source of heavy metals in wastewater (Malu *et al.*, 2014), these heavy metals are hazardous and cause different disorders which include cerebrum, brain and liver damage (Srivastava *et al.*, 2015 and Wang *et al.*, 2015). Heavy metals get into the brewery wastewater through the grains which are utilized as basic materials, since they are typically developed with herbicides, fungicides and bactericides that contain heavy metals (Cejka *et al.*, 2011).

Several techniques such as precipitation, filtration, ion exchange, advanced oxidation, reverse osmosis and so forth have been used for the removal of heavy metals from aqueous solution. However, these techniques have their very own current restrictions such as high operational cost, delicate working conditions, low efficiency (Sud, *et al.*, 2011). Adsorption using activated carbon is recognized to play a great industrial significant in removal of heavy metals from wastewater, due to its accessibility, sustainable, low cost, and high efficiency (Abdel-Fattah, *et al.*, 2015).

Due to these facts researchers all over the globe have focused on the development of alternative methods, as well as more environmentally friendly processes. Activated carbon has been recognized as an important material in purification of water, due to its unique properties such as high porosity and high surface area, has been known as an (Silgado *et al.*, 2014). However commercial activated carbons are costly which has necessitated the development of activated carbon from cheaper materials (Samson, 2015), such as *Alstonia boonei* which is an agricultural by-

products that are available at little or no cost that can be used for preparation of the activated carbon.

Activated carbon have various application which include recovery of solvents, removal of odor medical application, color removal, water purification, desalination, purifying agent in food processing and catalyst support (Yahya *et al.*, 2016). The production of activated carbon from agricultural byproducts has potential economic and environmental impacts by converting unwanted low-value agricultural waste to high-value adsorbents (Ekpete *et al.*, 2017).

2. Methodology

2.1 Sample Collection and Preparation

The Cheese wood (*Alstonia boonei*) was gathered from Kure Market in Minna Niger State, Nigeria. The Cheese wood was then thoroughly washed twice with distilled water and dried for 3 weeks in the laboratory at room temperature. The sample was crushed with mortar and pestle and sieved with 63 μ m mesh to obtain a fine sample. The industrial effluent was collected from the Nigerian Breweries, Kakuri, Kaduna State, Nigeria.

2.2 Carbonization (Pyrolysis) and Activation of the Adsorbent

A known amount of Cheese wood (5.00 g) was weighed inside a crucible and then placed in a muffle furnace and heated at a temperature of 600 $^{\circ}$ C for 15 minutes. The sample was then removed and placed in a desiccator to cool. This process was repeated severally until a desired quantity of the sample was obtained. 30.00g of the carbonized sample was added to 500cm³ of 0.5mol/dm³ HNO₃ in 1000 cm³ beaker and was allowed to stay for 24hours to ensure activation of the sample. It was washed severally with deionized water until a pH value of 7 was obtained (Narin *et. al.*, 2005). It was then filtered and the residue was oven dried for 24 hours at 110 $^{\circ}$ C and then cooled and stored in a polyethene bag for further use.

2.3 Digestion of Industrial Effluent for the Heavy Metals Analysis

10cm³ of the industrial effluent was measured into a 250cm³ beaker. 2cm³ of conc. HCl and 5cm³ of conc. HNO₃ were added into the beaker and the solution was heated on a hot plate for 15 minutes to a final volume of 5cm³ in a fume cupboard. 15cm³ of deionized water was added to the solution in the beaker and filtered with whatman filter paper No. 45. The filtrate was made up to 50cm³ with deionized water. This process was repeated until the desired volume was obtained, and was taken to the Atomic Absorption Spectrophotometer for analysis.

2.4 Batch Adsorption Experiment

2.4.1 Adsorbent Dosage Effect

50cm³ of effluent was measured into a 250cm³ conical flask and 0.5g of activated carbon was added to it and shaken on a mechanical shaker for 60 minutes. This process was repeated for the dosage of 1.0g, 1.5g, and 2.0g activated carbon. The content of each conical flask was filtered and the filtrate taken for AAS analysis.

2.4.2 Contact Time Effect

The effect of contact time 30, 60, 90, and 120 minutes were experimented respectively. 0.5g activated carbon was weighed into four different conical flasks containing 50 cm³ each of the effluent and were placed on a mechanical shaker. After each time, the samples were filtered with Whatman filter paper and the filtrates were taken for AAS analysis.

2.4.3 Solution pH Effect

50cm³ of the effluent was measured inside four different conical flasks. The pH of the effluent in the 1st conical flask was made to 2, the pH of the 2nd conical flask made to 4, the pH of the 3rd made to 6, and the pH of the 4th made to 8 using either 0.1M HCl or 0.1M NaOH (Nasrabadi *et. al.*, 2010). 0.5g of activated carbon was introduced into each conical flask and macerated with a glass rod and placed on a mechanical shaker for 60 minutes. The samples were then filtered, and the filtrate taken for AAS analysis.

2.4.4 Effect of Temperature

50Cm³ of the wastewater was placed into four different 250cm³ conical flask. 0.5g of the activated carbon was added to the effluent in each flask. A series of the flask was placed in a water bath shaker at temperatures of 30°C, 40°C, 50°C, and 60°C respectively for 30 minutes each. The content was then filtered with filter paper and the filtrate was analysed using Atomic Absorption Spectrophotometer (AAS).

2.5 Data Analysis

The adsorption capacity q_e (mg/g) of the raw sample and activated carbon were calculated using the expression;

$$q_e = \frac{(C_o - C_e)V}{M} \quad (1)$$

where C_o is the initial concentration in mg/dm³, C_e is the final concentration in mg/dm³, V is the volume of effluent in dm³, and M is the mass of the adsorbent in grams. The percentage removal of heavy metals was calculated using the expression:

$$\% \text{ removal of heavy metals} = \frac{C_o - C_e}{C_o} \times 100 \quad (2)$$

2.5.1 Adsorption Kinetics

Two kinetic models were used to examine the adsorption mechanism of the heavy metals.

2.5.2 Pseudo First Order

This is given as: $\frac{dq_t}{dt} = K_1 (q_e - q_t)$ (3)

The integral form of this equation is (Santhy and Selvapathy 2006)

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (4)$$

where q_e (mg/g) and q_t (mg/g) are the amounts of adsorbed adsorbate at equilibrium and at time t , respectively, and k_1 (min⁻¹) is the rate constant of pseudo first-order adsorption.

2.5.3 Pseudo second-order equation

The sorption kinetics can also be described by pseudo second-order model. The linear form of pseudo second-order equation is expressed as (Bulut and Ozacar 2008).

$$t/q_t = 1/k_2 q_e^2 + t/q_e \quad (5)$$

where k_2 (g/mg min) is the equilibrium rate constant of pseudo second-order adsorption.

The applicability of the two models above can be examined from the linear plot of $\ln(q_e - q_t)$ against t , and t/q_e against t , respectively.

2.5.4 Thermodynamic parameters

The value of ΔH and ΔS were calculated from the slope and intercept of the linear van Hoff's plot respectively, using the relation:

$$\ln K = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \quad (6)$$

Where:

ΔS = entropy change for the process

ΔH = enthalpy change for the process

R = gas constant

T = absolute temperature

The distribution coefficient (K_d) of the activated charcoal surface was calculated using the equation,

$$K_d = \frac{(C_i - C_e)V}{C_e M} \quad (7)$$

Where C_i = the initial concentration of the metal ion, C_e = the equilibrium concentration of the metal ion, V = the volume of the solution equilibrated in cm³ and m = mass of the adsorbent in g

The change in free energy (ΔG) for the specific adsorption has also been calculated using the equation

$$\Delta G = -RT \ln K \quad (8)$$

3. RESULTS AND DISCUSSION

Results

Table 1: Results of the physicochemical properties of the adsorbent

Parameters	Values
Bulk density (g/cm ³)	0.26
Ash content (%)	6.00
Moisture content of Activated Adsorbent (%)	0.1
Electrical conductivity (μS/cm)	2.8 x 10 ²
Iodine number (mg/g)	110.48
Volatile matter (%)	2.17
Fixed carbon (%)	91.63
Micro pore width (nm)	6.538
Pore diameter (nm)	2.427
Pore volume (cc/g)	0.2158
Surface area (m ² /g)	418.3-1344

Table 2: Adsorption kinetic constants for activated carbon

Metal ions	Pseudo-first order			Pseudo second order		
	$\ln(q_e - q_t) = \ln q_e - K_1 t$			$t/q_t = 1/K_2 q_e^2 + t/q_e$		
	R ²	q _e (mg/g)	K ₁	R ²	q _e (mg/g)	K ₂
Pb	0.0228	1.1630	-0.0210	0.954	454.55	0.00017
Ni	0.1401	0.0501	-0.0454	0.8668	125	-0.00035
Fe	0.5889	0.0021	-0.0760	1	500	-0.01000

The experimental adsorption capacity q_e (mg/g) for Pb, Ni and Fe are 445.598mg/g, 239.138mg/g and 499.878mg/g respectively.

Table 3: Thermodynamic parameters Enthalpy Change, ΔH°, and Entropy Change, ΔS°, for Pb, Ni and Fe

Item	Pb	Ni	Fe
R ²	0.1495	0.5666	0.3008
ΔH° KJ/mol	-12.8	-19.37	-31.68
ΔS° (J/mol.K)	0.0054	0.1164	0.0316

Table 4: Gibbs free energy Change (ΔG°) for Pb, Ni and Fe

ΔG° (kJ/mol)			
Temp. (k)	Pb	Ni	Fe
303	-14.44	-54.64	-41.25
313	-14.49	-55.8	-41.57
323	-14.54	-56.97	-41.89
333	-14.59	-58.13	-42.2

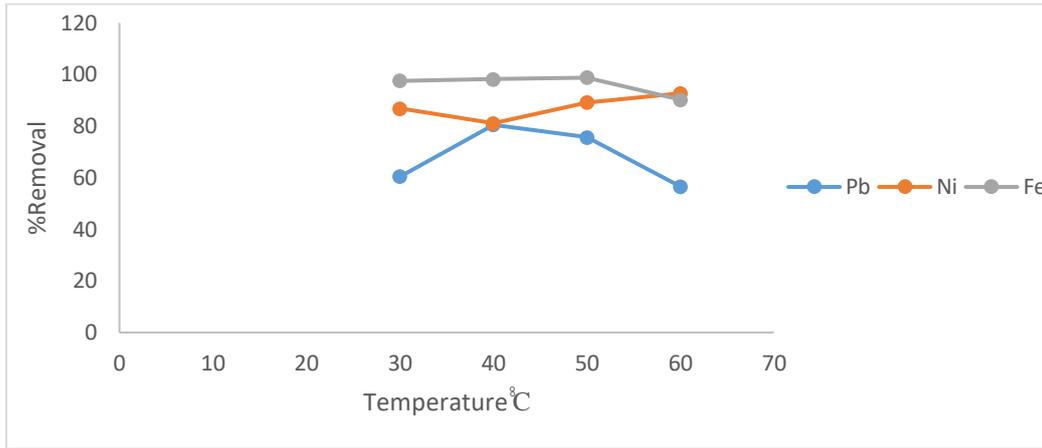


Figure 1: Effect of Temperature on Pb, Ni, and Fe

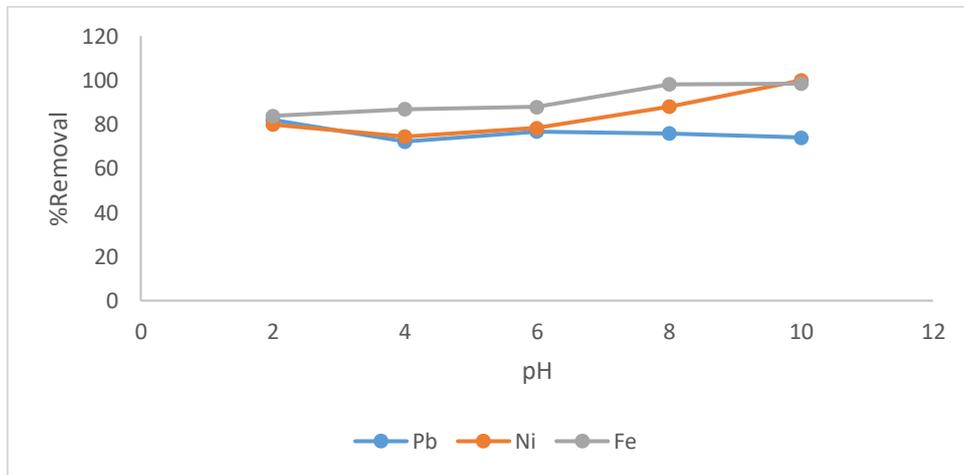


Figure 2: Effect of solution pH on Pb, Ni, and Fe adsorption

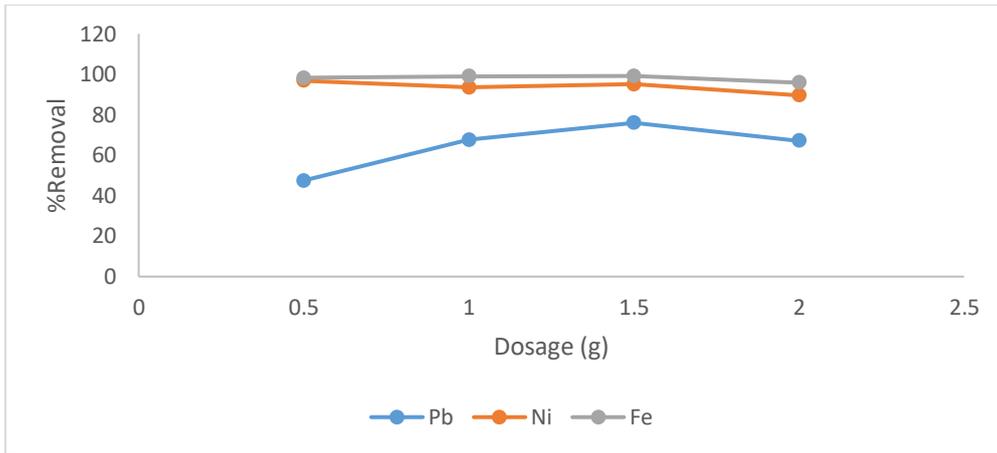


Figure 3: Effect of Adsorbent Dosage on Pb, Ni, and Fe Adsorption

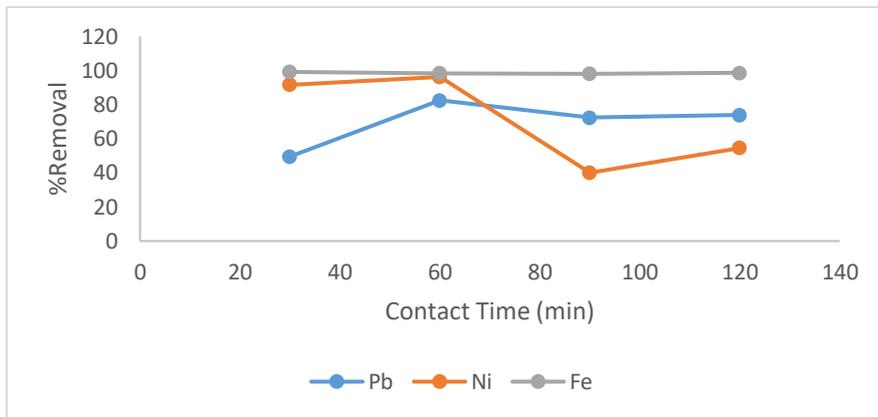


Figure 4: Effect of Contact Time on Pb, Ni, and Fe Adsorption

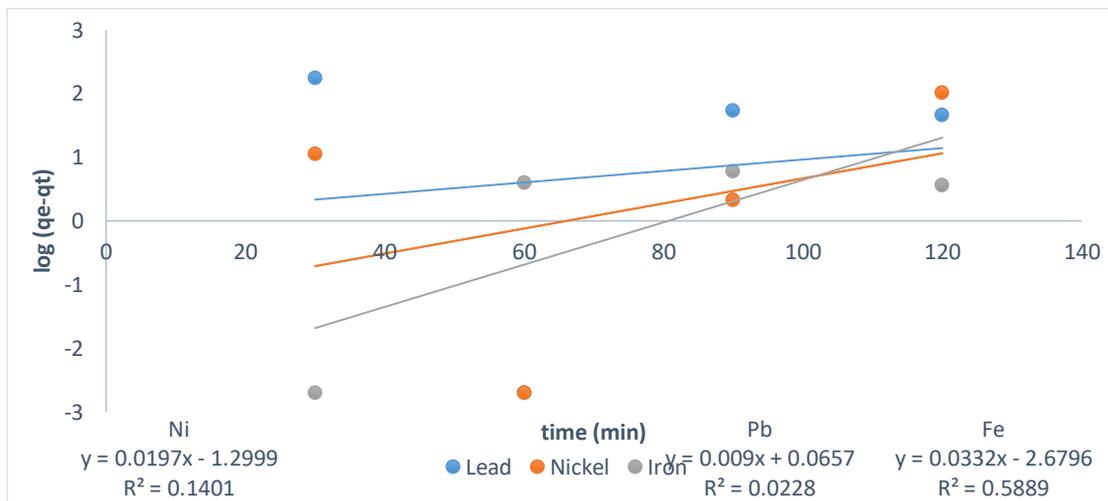


Figure 5: Pseudo first order kinetic plot for Pb, Ni, and Fe adsorption

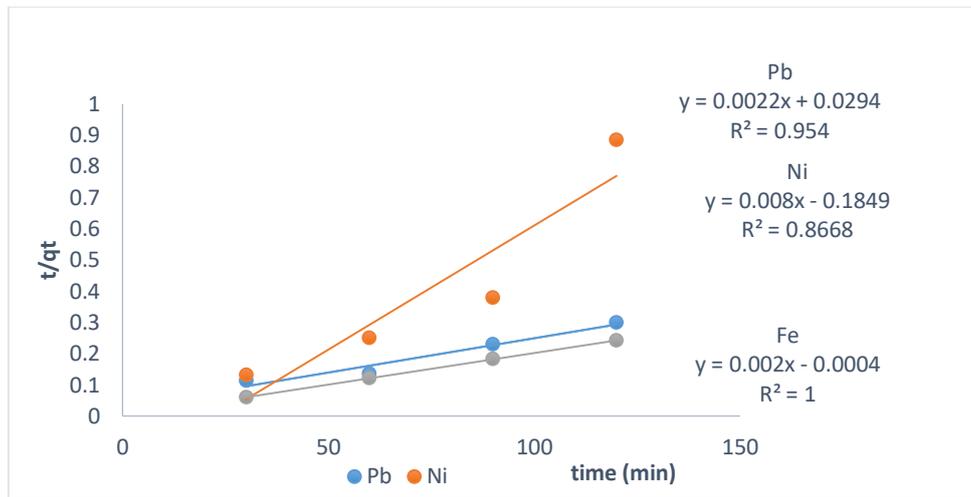


Figure 6: Pseudo second order kinetic plot for Pb, Ni, and Fe adsorption

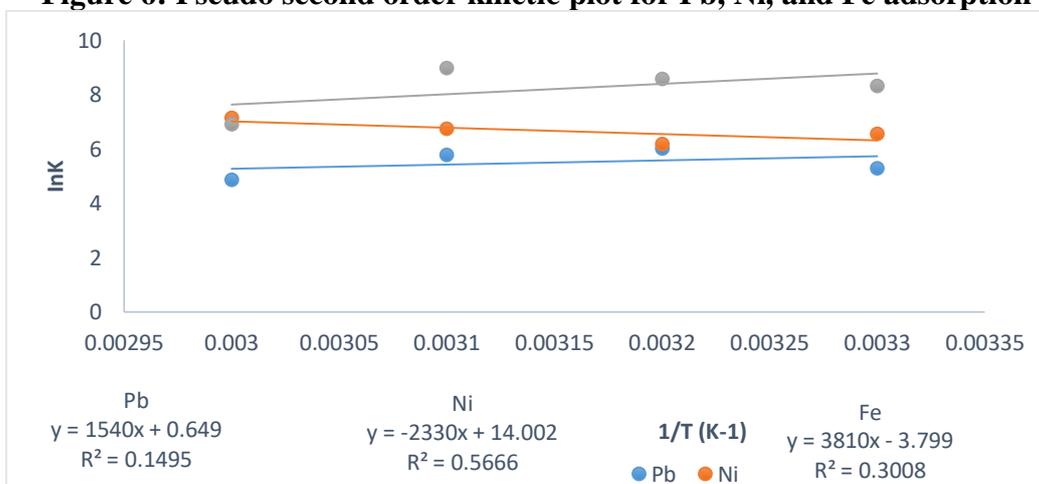


Figure 7: Van Hoff's thermodynamic plot for the adsorption of Pb, Ni and Fe

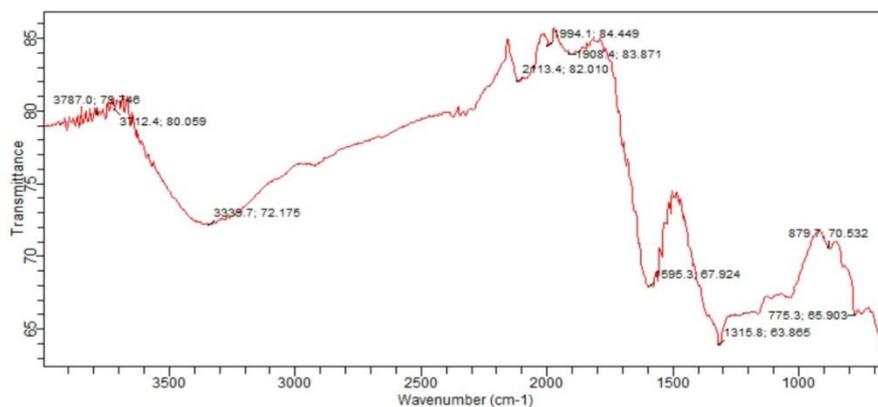


Figure 8: FTIR spectrum of Activated Carbon Prepared from *Alstonia boonei*

3.2 Discussion

From the result of the physicochemical analysis in table 1 6% ash content was obtained for this study; good activated carbon is expected to have low ash content (Abubakar *et al.*, 2012). The ash content is linked to the pore structure of the activated carbon and also reflects the purity of

the activated carbon (Nworu *et al.*, 2018), which greatly influences the adsorptive capacity of the prepared activated carbon (Ahmed *et al.*, (2008).

The result of the moisture content of the activated *Alstonia boonei* was 0.1% as presented in table 1. It has been reported by Shaarani *et al.* (2010) that a good activated carbon must have a low moisture content to avoid water vapours competing in adsorption process and fill the adsorption sites within the pores, thus reducing the efficiency of the activated carbon. The moisture content obtained in this study is lower than the 4.20 % reported Maina and Anuka (2014).

The range of particle size and degree of activation affects bulk density, which have an effect on adsorption per unit volume. Bulk density of 0.26g/cm³ was obtained for this study which is lower than the 0.509g/cm³ reported by Olafadehan *et al.* (2012). TIGG Corporation, (2012) reported that higher density provides greater volume activity and indicates better-quality activated carbon.

The iodine number of 110.48 mg g⁻¹ was obtained from the analysis. Iodine number is directly linked to the adsorption capacity of an activated carbon since it is a measure of the microspore content of the activated carbon and is obtained by the adsorption of iodine from solution by the activated carbon sample (Lillo-Rodenas *et al.*, 2003). It is used to measure the porosity and adsorbent capacity of activated carbon (Ekpete and Horsfall, 2011).

Volatile matter of 2.17 was obtained. The study revealed fixed carbon of 91.63 %. Surface properties of the of the activated carbon (BET surface area, pore volume, and pore size distributions) were evaluated by nitrogen adsorption-desorption isotherm the result revealed micro pore width of 6.538 nm, pore diameter 2.427nm, pore volume of 0.2158 cc/g and surface area 418.3- 1344 m²/g for BET and Langmuir model respectively. The result is similar to the 311 to 1044 m²/g surface area and 0.18 to 0.59 cm³/g pore volume obtained for *Spent Tea Leaves* activated carbon by Singh *et al.* (2015). The result is higher than 179.7 m²/g and micropore volume 0.081 cc/g (Yakout *et al.*, 2015).

The result shows that as the temperature increases from 30°C to 40°C the percentage removal of Pb increases from 60.40% to 80.48% and Fe ion also increases from 97.59% to 98.78% as the temperature increases from 30-50°C as shown in figure 1 This phenomenon is attributed to the increased kinetic energy of the metallic ions due to the increase in temperature of the system which leads to the increased mobility of the Pb and Fe ions to the adsorption sites.

Meanwhile, there was decrease in percentage removal of Ni from 86.83% to 81.13 %. As the temperature increases before an increase in percentage removal from 81.13% to 89.13% and respectively. From the result it is observed that the percentage removal of Pb < Ni < Fe which may be attributed to their atomic size.

The effect of solution pH on adsorption of Pb, Ni and Fe is shown in figure 2. The highest percentage removal of Pb was at solution pH of 2, while Ni and Fe highest percentage removal was at pH 10. The low bio-sorption of Fe and Ni at lower pH may be attributed to the higher concentration and greater mobility of H⁺.

The highest percentage removal of Pb, Ni and Fe are 81.94%, 99.85%, and 98.39% respectively.

The result in this study shows an increase in percentage removal of Pb ions from 47.49% to 67.64% to 76.015% as adsorbent dosage increases from 0.5g to 1.0g to 1.5g respectively. This continuous increase percentage removal with increasing adsorbent dosage is due to the increased surface area of adsorbent dosage with increasing amount of adsorbent. Ranjan and Krishna (2014) gave a similar report using activated carbon from *polyathia longifolia* for the kinetic studies on bio-sorption of fluoride.

The lowest percentage removal of Pb, Ni and Fe was observed at the highest adsorbent dosage of 2g. This phenomenon can be attributed to the agglomeration of the adsorbent due to the large quantity of activated carbon, forming clusters which tends to reduce the surface area of the adsorbent and block the its pore space.

The result obtained for the effect of contact time on Pb, Ni and Fe as presented in figure 4.7 shows the rapid adsorption of Pb and Ni in the first 60 minutes and Fe in the first 90 minutes of the adsorption process. This is due to the availability of free pore space in the adsorbent in the early minutes of contact time and so these metals will readily fill up those spaces. The maximum percentage removal for Pb and Ni was 82.48% and 96.19% respectively at contact time of 60 minutes, while the maximum percentage removal for Fe was 99.96% at contact time of 90 minutes.

The thermodynamic parameters, namely free energy (ΔG°), enthalpy (ΔH°) and entropy (ΔS°) have an important role in determining spontaneity and heat change for the adsorption process. The values of entropy change (ΔS°) and enthalpy change (ΔH°) were obtained from intercept and slope of the plot of $\ln K$ versus $1/T$ (K^{-1}).

The estimated values of the thermodynamic parameters for the four operation temperatures were calculated and presented in table 4 and 5. The negative values of the change in free in energy (ΔG°) confirms the adsorption process is spontaneous and does not require energy input outside the system while the positive value of the entropy change (ΔS°) confirms the spontaneity of the adsorption process (Zhou *et al.*, 2014). The negative value of ΔH° -12.6, -19.37 and -31.69 kJ/mol for Pd, Ni and Fe respectively indicates that the adsorption is exothermic in nature, and in such instance, increase in temperature leads to increase in adsorption rate since the adsorbed molecules gain more kinetic energy and are readily desorbed. In other words, in exothermic adsorption process, the rate of adsorption-desorption increases.

In adsorbed state, the adsorbate is held on the surface of the adsorbent by attractive forces (bond). Depending on the nature of attractive forces, adsorption can be classified as physical adsorption (physisorption) or chemical adsorption (chemisorption) as reported by Alzaydien in 2016. The ΔH° values for physical adsorption is in the range of -10kJ/mol to 40kJ/mol (Alkan *et al.*, 2004), this shows that the adsorption in this study is physical adsorption since the ΔH° values obtained is less than 40kJ/mol, while it has been reported that chemical adsorption are rarely less than 80kj/mol and sometimes exceed 400kj/mol (. Chemisorption has higher enthalpy of adsorption values because in chemisorption, the chemical bonds are stronger, while the physical adsorption has lower enthalpy of adsorption because weak forces such as vanderwaal's forces exist.

Fourier transform Infrared spectroscopy was used to study the functional group and obtained information on the chemical structure of the prepared activated carbon. The surface chemistry of the activated carbon was determined by the type, quality and bonding of the functional groups. Figure 8 from the result shows the spectra of activated carbon from *Alstonia boonei*. The broad band around 3100-3600 cm^{-1} is attributed to the presence of hydroxyl group, either aliphatic alcohol/phenol or adsorbed water molecule. The 2200-2000 cm^{-1} is possibly caused by $C\equiv C$ stretch. The band around 1650-1500 cm^{-1} is possibly caused by the stretching vibration of $C=O$ in carboxyl, ketones, aldehydes or lactone group. The band around 1400 cm^{-1} is attributed to the presence of NO_2 . 1400-1000 cm^{-1} indicates C-F bond and 800-600 cm^{-1} indicates C-Cl bond.

The mechanism of the adsorption of Pb, Ni and Fe ions by activated carbon from *Alstonia boonei* were examined by using the pseudo-first and pseudo second models. The pseudo-second order kinetics has higher correlation coefficient (R^2) values in comparison to the correlation coefficient values of the pseudo-first order. Also, the calculated adsorption capacity (q_e) values for the pseudo-second order kinetics for Pb, Ni and Fe were found to be 454.55mg/g, 125mg/g, and 500mg/g respectively, which are very much closer to the

experimental adsorption capacity (q_e) values of Pb, Ni and Fe, which are 445.598mg/g, 239.138mg/g and 499.878mg/g respectively, while the calculated q_e values for the pseudo-first kinetics for Pb, Ni and Fe which are 1.1630mg/g, 0.0501mg/g, and 0.0021 mg/g respectively, are nowhere close to the experimental q_e values. Therefore, the pseudo second order model best fits the experimental data than the first order and is more relevant to this study.

4.0 Conclusion

Activated carbon prepared from *Alstonia boonei* bark which is abundant in Nigeria can be used as effective sorbents for purification of drinking and waste waters. From the results obtained, it is evident that activated carbon produced from *Alstonia boonei* bark can remove 80.48%, Pb, 99.19% of Fe and 99.85% of Ni ions which also dependent on parameters such as time, adsorbent dosage, pH and temperature.

The thermodynamic parameters indicated that the adsorption process is spontaneous. .

5.0 REFERENCE

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