

# Sorption Potential of Ugwuoba Clay for Lead(II) Ions from Aqueous Solution.

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## ABSTRACT

The parameters affecting the adsorption of Pb(II) ions are investigated by utilizing Ugwuoba clay as a cheap adsorbent. The adsorption of Pb(II) on Ugwuoba clay was studied using batch method to determine the effect of pH, initial metal ion concentration, contact time and temperature. FTIR studies revealed the presence of some groups such as C≡N, C=C, C-O, C-Cl and C-Br on the clay surface. The effect of initial metal ion concentration showed an increase in adsorption capacity and a decrease in percentage removal with increase in concentration.

Optimum adsorption was achieved at a pH of 5.0 and equilibrium sorption was attained within 60 minutes. Equilibrium isotherm studies showed that the Freundlich isotherm gave a better description of the adsorption than the Langmuir isotherm. Kinetic studies revealed that the Lagergren first order model gave a better fit to the experimental data than the Pseudo second order model. An increase in adsorption capacity with increase in temperature was recorded. Thermodynamic studies showed negative values of  $\Delta G^0$  at all temperatures, a positive entropy and enthalpy change of 39.631J/molK and 9.757KJ/mol respectively. These results indicate a spontaneous, disordered and endothermic process and the applicability of Ugwuoba clay as an adsorbent for Pb(II) ion in solution.

(Keywords: heavy metals, lead, Pb(II), sorption, Ugwuoba clay)

## INTRODUCTION

Metals and their compounds are useful for the industrial, technological and agricultural development of any nation. As a result of increasing development in science and

technology, the number of applications of metals for commercial purposes continue to increase.

These industrial processes generate large amount of metallic waste products which are subsequently discharged into wastewaters and land sites. It has been observed that the harmful effect of heavy metals discharged annually into the environment far exceeds the combined total toxicity of all radioactive and organic wastes (Abdus-salam and Adekola, 2005). This accounts for the reason why the release of heavy metals into the environment has become a problem of great concern over the last few decades.

Some of the industrial activities that led to the release of heavy metals into the environment include leather tanning, battery manufacturing, electroplating, metal finishing, steel fabrication, paint production, ceramics, glass, dyes and paper production. Heavy metals such as Lead, Cadmium, Mercury, Nickel, Chromium and Copper are known to be toxic at certain concentrations (Deorkar and Talvarides, 1997).

The contamination of the environment with Lead is mainly due to anthropogenic activities, which makes this metal the most ubiquitous toxic metal in the environment (WHO and ILO, 1995). Lead is non-biodegradable, has the potential to bio-accumulate in the food chain causing human health hazards, when present in high concentrations can damage the brain and nervous system (Ho et al, 2001). The assimilation of relatively small amounts of Lead in humans can lead to chronic toxicity and malfunctioning of the organs (Khurshid and Qureshi, 2001).

Lead is also an enzyme inhibitor, general metabolic poison and affects the functioning of the blood, liver and kidney (Ceribasis and Yetis, 2001). Due to the magnitude of the problem resulting from Lead pollution, researches on the

removal of Lead from effluents have become a dominant topic for environmental scientist. The traditional techniques which have been used for the removal of Lead and other heavy metals from industrial wastewaters include chemical precipitation and filtration, chemical oxidation or reduction, electrochemical treatment, reverse osmosis, solvent extraction, ion-exchange and evaporation. These techniques have the disadvantages of high cost, low selectivity, incomplete metal recovery, high energy requirement, difficult to apply and the generation of toxic slurries which are difficult to dispose (Volesky, 2001).

Adsorption has been discovered to be one of the most effective methods for the removal of Pb(II) and other toxic metals from aqueous solution due to its high efficiency, low cost of maintenance and very easy to apply. Activated carbon is the most widely used adsorbent. It has a high adsorption capacity and surface area but has the disadvantage of high cost, these limits its application to small scale industries and developing nations. As a result, a search for cheaper alternative adsorbents has become a major area of interest for researchers. A good number of researchers have made use of low cost adsorbent materials for Pb(II) and other heavy metals removal. Some of the adsorbents used include Clay (Akpomie et al., 2012a), Lateritic materials (Ahmad et al, 2002), Biomass (Abia et al., 2003; Huang and Morehart, 1990), Red mud (Gupta et al, 2001) and Sawdust (Yu et al., 2001).

There is an abundance of clay deposit in Enugu State Nigeria. Ugwuoba clay is a clay material found in large deposits in Oji River local Government Area of the State. This present communication was aimed at the application of Ugwuoba clay as a cheap adsorbent for the removal of Pb(II) ions from aqueous solution. The effect of initial metal ion concentration, pH, contact time, and temperature on the sorption process were investigated. Also, the equilibrium isotherm, kinetic and thermodynamic parameters were determined.

## **MATERIALS AND METHODS**

### **Adsorbent Preparation and Characterization**

The sample of Ugwuoba clay was obtained from Ugwuoba in Oji River Local Government Area of Enugu State, Nigeria on the 13th October 2011.

The clay was dispersed in excess distilled water in a plastic container, stirred to ensure uniform dissolution after which it was filtered to remove unwanted plant materials and suspended particles. The mixture was allowed to settle for 24hrs after which it was decanted to remove excess water. The clay obtained at the bottom of the container was sundried for several days, then dried in an oven at 105<sup>o</sup>C. The dried clay was crushed further and then sieved through a 100 $\mu$ m mesh sieve. The sample was ready for use as an adsorbent.

The Fourier Transform Infrared (FTIR) spectra of the clay was obtained using the FTIR spectrophotometer (Shimadzu FTIR 8400S) in the wave number region of 400-4000cm<sup>-1</sup>. The chemical characterization of the clay was performed using classical methods.

### **Preparation of Adsorbate (Pb(II) ion Solution)**

Analytical grade chemical was used in this study. Lead(II) ion stock solution of concentration 1000mg/L was prepared by dissolving appropriate quantity of Pb(NO<sub>3</sub>)<sub>2</sub> in de-ionized water. Different concentrations of Pb(II) in the range 20-100mg/L were prepared by appropriate dilution of the stock solution with de-ionized water. Using a pH meter, the pH of each solution was adjusted to the required value by the addition drop by drop of 0.1M HNO<sub>3</sub> or 0.1M NaOH. Freshly prepared solution was used for each adsorption study.

### **Batch Sorption Technique**

The adsorption potential of Ugwuoba clay for Pb(II) ions was conducted using batch method. The effect of pH, initial metal ion concentration, contact time and temperature were determined. pH study was conducted at varying pH ranges of 1-8, initial metal ion concentration was investigated in the range 20-100mg/L, contact time was varied from 10-120 minutes, while a temperature variation in the range of 27-45<sup>o</sup>C was used to determine the effect of temperature. The batch adsorption was performed by contacting 2g of the clay with 20mls of a particular solution of Pb(II) ion in a thermostat water bath (Haake Wia Model) to help regulate the temperature. At the end of the given contact time the solution was filtered and the filtrate was analyzed for the concentration of Pb(II) remaining

in solution using the Atomic Absorption Spectrophotometer (AAS) (Buck Scientific Model 210 VGP). Each study was done in duplicate and the mean value was computed to obtain quality assurance. The adsorption capacity of Ugwuoba clay for Pb(II) ion was calculated using the mass balance equation given below:

$$qe = v(C_i - C_e)/m \quad (1)$$

The percentage of Pb(II) ion adsorbed was calculated from (2)

$$\text{Removal}(\%) = C_i - C_e / C_i \times 100 \quad (2)$$

Where  $q_e$ (mg/g) is the equilibrium adsorption capacity of the clay,  $v$  (Liters) is the volume of Pb(II) solution used,  $m$  is the mass of the clay (g),  $C_i$ (mg/L) is the initial concentration of Pb(II) in solution and  $C_e$ (mg/L) is the equilibrium concentration of Pb(II) in solution remaining after adsorption.

## RESULTS AND DISCUSSION

### Chemical Characterization

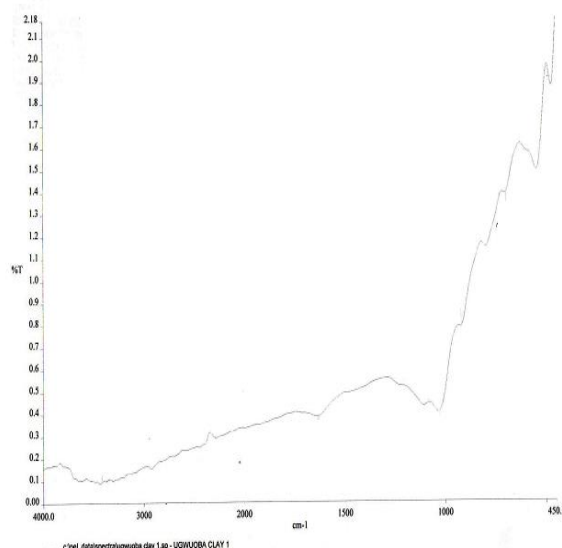
The result obtained on the chemical characterization of Ugwuoba clay is shown in Table 1. It is seen from the table that Silica and Alumina are the major constituents of this clay material, therefore Pb(II) ions in solutions are expected to be removed mainly by this group. Other elements such as Calcium, Sodium and magnesium oxide are present in considerable amounts while Iron(III), Potassium and Manganese oxide are present in minute quantities.

**Table 1:** Chemical Composition of Ugwuoba Clay.

COMPOSITION	% BY WEIGHT
SiO <sub>2</sub>	47.32
Al <sub>2</sub> O <sub>3</sub>	25.91
CaO	4.39
MgO	3.14
Na <sub>2</sub> O	2.86
Fe <sub>2</sub> O <sub>3</sub>	1.41
K <sub>2</sub> O	1.07
MnO	0.43
Loss on Ignition	13.56

### FTIR Spectra Analysis

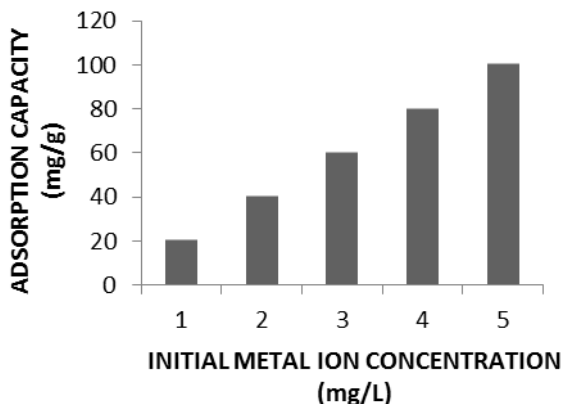
Although clay is an inorganic material, certain functional groups may be present on its surface as contaminants or impurities. These functional groups if present could influence the adsorption capacity of the clay. To investigate the presence of functional groups on the surface of the clay the FTIR spectra was conducted and is shown in Figure 1. The spectra revealed some adsorption peaks indicating the presence of functional groups. The presence of C≡N was indicated by the band at 2250cm<sup>-1</sup>. Also C=C of alkenes was observed at 1632cm<sup>-1</sup>. The peak at 1105cm<sup>-1</sup> corresponds to C-O stretching vibrations. Si-O stretching vibrations of Si-O-Si group was observed by the band adsorption at 1028cm<sup>-1</sup> (Madejova, 2003). Also, C-Cl and C-Br stretching were indicated by the bands at 696cm<sup>-1</sup> and 546cm<sup>-1</sup> respectively. Therefore the FTIR spectra analysis showed the presence of some negatively charged groups on the surface of the clay which could enhance its adsorption capacity.



**Figure 1:** FTIR Spectrum of Ugwuoba Clay.

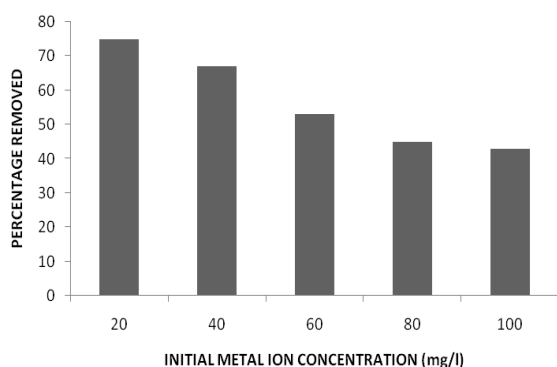
### Effect of Initial Metal Ion Concentration

The adsorption intensity of a material is a function of the initial concentration of the adsorbate (metal ion) which makes it an important parameter to be considered in adsorption study. The effect of initial Pb(II) ion concentration on its adsorption by Ugwuoba clay is presented in Figure 2. It is observed that there is an increase in the adsorption capacity (mg/g) with increase in concentration.



**Figure 2:** Effect of Initial Metal Ion Concentration in on the Sorption of Pb(II) ions unto Ugwuoba Clay ( pH 5.0, Temp 300K, Time 2hrs).

This may be accounted for as a result of increasing concentration gradient which creates an increasing driving force, hence overcoming resistances to mass transfer of Pb(II) ions between the solid and aqueous phase. However, an opposite trend was observed in Figure 3 in which a decrease in percentage removal with increase in concentration was shown, this was due to the fact that each type of adsorbent has a limited number of active sites which becomes saturated at certain concentrations (Tsai and Chen, 2010). Similar report have been obtained by other researchers (Argun et al., 2007; Souag et al., 2009).

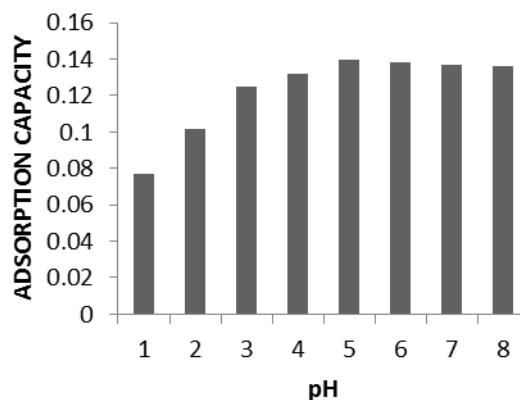


**Figure3 :**Effect of Initial Metal Ion Concentration in Terms of Percentage Removal on the Sorption of Pb(II) ions unto Ugwuoba Clay (pH 5.0, Temp 300K, Time 2hrs).

### Effect of Initial Solution pH on Sorption

The initial solution pH is one of the most important factors in the adsorption mechanism as it affects the surface charge of an adsorbent and the degree of specification and ionization of an adsorbate (Imagoglu and Tekir, 2008). The result on the effect of pH on the sorption of Pb(II) ion by Ugwuoba clay is shown in Figure 4. As observed there is an increase in the adsorption uptake and percentage removal as the pH increases, after pH 5.0 the adsorption became fairly constant up to pH 8.0 although it decreased slightly.

Optimum pH of adsorption was obtained at 5.0. pH values greater than 7.0 are usually avoided in adsorption due to the likelihood of precipitation of the hydroxide forms of the adsorbate species. The active sites on the surface of the clay are protonated at low pH values. There is also a competition between the adsorbate species (Pb(II) ions) in solution and protons for the active sites leading to a reduction in the adsorption at lower pH values. With decrease in acidity of the solution the active sites on the surface of the clay becomes deprotonated resulting in an increase in the negatively charged density on the adsorbent surface which facilitates the binding of metal cations. This increase in adsorption with increase in pH is largely due to the reduction of protons which competes with the adsorbate at lower pH values (Ofomaja et al, 2010). Similar result has been observed (Johnson, 1990).

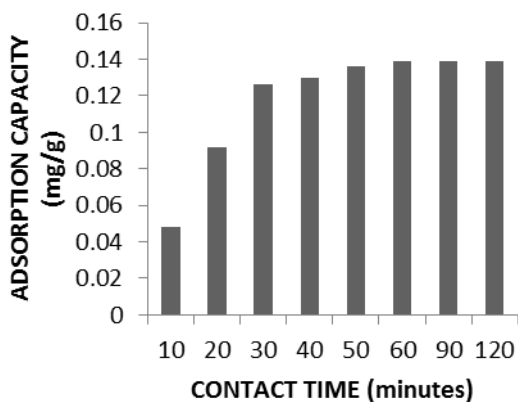


**Figure 4:** Effect of pH on the Sorption of Pb(II) ions unto Ugwuoba Clay ( Temp 300K, Conc 20mg/L, Time 2hrs).

### Effect of Contact Time

In order to investigate the kinetics of sorption of Pb(II) onto Uguwuoba clay, the effect of contact time on adsorption was determined. As observed in Figure 5, there was a rapid initial uptake of Pb(II) ion on the clay within the first 30 minutes. After 30 minutes there was a slight increase in adsorption up to 60minutes after which the adsorption became constant up to 120minutes. This indicates the establishment of equilibrium within 60minutes. Sorption experiments in this study were performed at a contact time of 2hrs hence equilibrium achievement was ensured. Similar result have been reported by other workers (Abia and Asuquo, 2006; Pandey et al, 2009).

The fast adsorption at the initial stage is due to the availability of abundant active sites which became saturated or used up with time. In the later stage the sorption is likely an attachment-controlled process due to less availability of active sites. The differences in the metal ion uptake rate is due to two different adsorption processes, namely, a fast ion exchange followed by chemisorptions (Low et al, 1993).

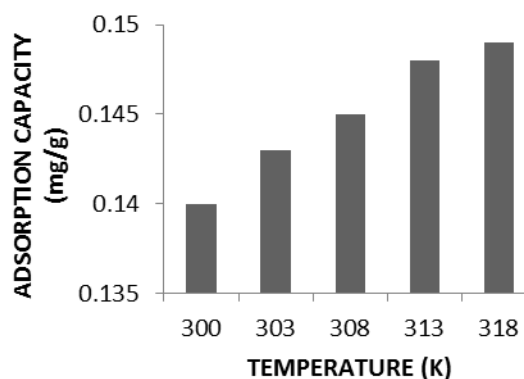


**Figure 5:** Effect of Contact Time on the Sorption of Pb(II) ions onto Uguwuoba Clay (pH 5.0, Temp 300K, Conc 20mg/L).

### Effect of Temperature

The effect of temperature on adsorption have been reported to have different types of behavior, some revealed an increase in adsorption capacity with temperature increase, also, a decrease and temperature-independent effects were also observed (Lodeiro et al., 2006). The result on the

influence of solution temperature on the adsorption of Pb(II) ions by Uguwuoba clay is shown in Figure 6. A slight increase in the adsorption with temperature increase was observed. This suggest that the process is endothermic in nature, it also indicates a chemical interaction between Pb(II) ions and the adsorbent. This adsorption is limited to just a single layer of molecules on the surface but may be followed by additional layers of physically adsorbed molecules (Trybal, 1968). Similar result has been reported on the adsorption of Cd(II) ions by Nsu clay (Akpomie et al., 2012b).



**Figure 6:** Effect of Temperature on the Sorption of Pb(II) ions onto Uguwuoba Clay (pH 5.0, Time 2hrs, conc 20mg/L).

### Adsorption Isotherms

Adsorption isotherms are mathematical equations used to explain the adsorption behavior of a particular adsorbent-adsorbate combination. These isotherms model adsorption behavior and help in calculating the adsorption capacity of materials. The Langmuir and Freundlich isotherm were applied to the experimental data and there calculated parameters are given in Table 2. They Freundlich isotherm gave a better fit than the Langmuir isotherm as indicated by their  $R^2$  values and is thus described.

**Table 2:** Equilibrium Isotherm Parameters on the Sorption of Pb(II) ions Unto Uguwuoba Clay.

LANGMUIR ISOTHERM			
qm	b	$R^2$	
0.58	0.038	0.789	
FREUNDLICH ISOTHERM			
1/n	n	Kf	$R^2$
0.49	2.04	0.05	0.933

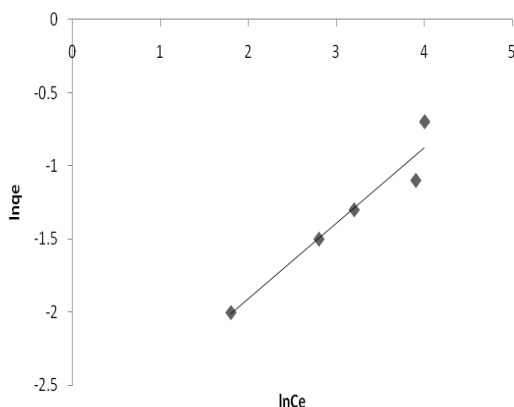


### Freundlich Isotherm

The Freundlich isotherm is widely applied to the description of adsorption processes. This isotherm is applicable to non-ideal adsorption on heterogeneous surfaces and involves multilayer adsorption (Freundlich, 1906). The linear form of the isotherm equation is expressed in Equation (3).

$$\ln q_e = \ln K_f + 1/n \ln C_e \quad (3)$$

where  $K_f$  is the Freundlich isotherm constant (L/g) related to the adsorption capacity of the adsorbent,  $n$  is a dimensionless constant which indicates the degree of non-linearity between solution concentration and adsorption. This isotherm was applied by the linear plot of  $\ln q_e$  against  $\ln C_e$  as shown in Figure 7 and the isotherm parameters  $K_f$  and  $n$  are obtained from the intercept and slope respectively. The value of the linear regression ( $R^2$ ) in Table 2 indicated that this isotherm provided a good and better fit to the experimental data than that obtained in the Langmuir isotherm. If the value of  $n$  is greater than unity it indicates a favorable or beneficial adsorption process (Kadirvelu and Namasivayam, 2000). The value of  $n$  obtained is greater than unity which shows a favorable adsorption between Pb(II) ions and Ugwuoba clay.



**Figure 7:** Freundlich Isotherm Model on the Sorption of Pb(II) ions unto Ugwuoba Clay (pH 5.0, Temp 300K, Time 2hrs).

### Kinetic Studies

Adsorption kinetics is an important factor which governs the rate of metal ion sorption, determines the residence time and helps define the efficiency of an adsorbent material (Krishnan and

Anirudhan, 2003). The kinetics of adsorption can be controlled by several independent processes which could act in series or parallel, these processes include chemical reaction, bulk diffusion, external mass transfer and intraparticle diffusion (Ho et al., 2000). In this study both the Lagergren first-order and Pseudo-second order kinetic models were applied to the experimental data to investigate the kinetics of sorption.

**Table 3:** Kinetic Parameters on the Sorption of Pb(II) ions Unto Ugwuoba Clay.

LAGERGREN FIRST ORDER		
$q_{e,cal}$	$K_i$	$R^2$
0.24	0.087	0.978
PEUDO SECOND ORDER		
$q_{e,cal}$	$K_2$	$R^2$
0.21	0.18	0.933

### Lagergren First Order Model

The Lagergren first order kinetic rate equation was applied (Lagergren, 1898) and is given in Equation (4).

$$\ln(q_e - q_t) = \ln q_{e,cal} - K_i t \quad (4)$$

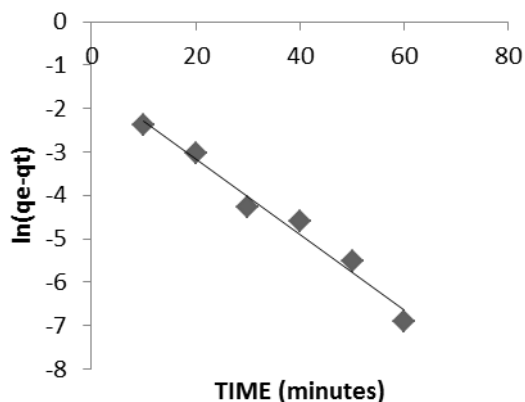
where  $q_e$  and  $q_t$  represents the amount of Pb(II) ions adsorbed at equilibrium and at time  $t$  in mg/g respectively,  $q_{e,cal}$  is the calculated value obtained in mg/g.  $K_i$  is the Lagergren first order rate constant ( $\text{min}^{-1}$ ). The plot of  $\ln q_e$  against  $t$  is shown in Figure 8 and the constants  $K_i$  and  $q_{e,cal}$  are obtained from the slope and intercept respectively. The Lagergren first order kinetic parameters evaluated are presented in Table 3. This model can be applied to the sorption process as indicated by the value of the regression coefficient ( $R^2$ ) being close to 1. This indicates a good fit to the experimental data.

### Pseudo Second Order Model

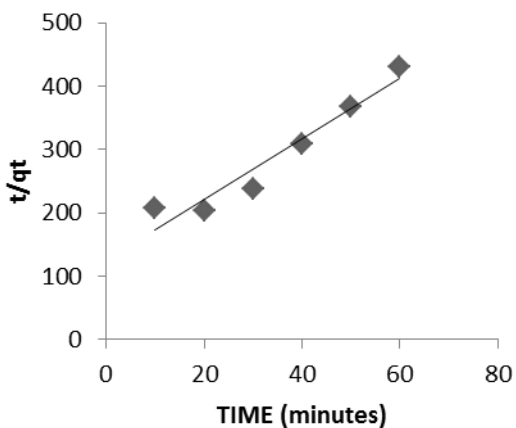
The Pseudo-second order model is based on the assumption that chemisorptions is the rate determining step (Ho and Mckay, 1998). It also assumes that most assumption follows a second order mechanism. The linear form of the Pseudo-second order equation is expressed in Equation (5).

$$t/q_t = 1/K_2 q_{e,cal}^2 + t/q_{e,cal} \quad (5)$$

$K_2$  is the equilibrium rate constant of Pseudo-second order adsorption (g/mgmin). Values of  $K_2$  and  $q_{e_{cal}}$  were obtained from the intercept and slope of the plot of  $t/qt$  against  $t$  and is shown in Figure 9. The evaluated parameters of this model are given in Table 3. The value of  $R^2$  obtained suggested a good fit to the experimental data. However, the Lagergren first order model is more likely applicable to the sorption process as the  $R^2$  value obtained is better than that of the Pseudo-second order model. Similar result has been reported (Akpomie et al., 2012b).



**Figure 8:** Lagergren First Order Plot on the Sorption of Pb(II) ions onto Uguwoaba Clay (pH 5.0, Temp 300K, Conc 20mg/L).



**Figure 9:** Pseudo Second Order Plot on the Sorption of Pb(II) ions onto Uguwoaba Clay (pH 5.0, Temp 300K, Conc 20mg/L).

**Table 4:** Thermodynamic Parameters on the Sorption of Pb(II) ions Unto Uguwoaba Clay.

$\Delta S^0$ (J/molK)	$\Delta H^0$ (KJ/mol)	$R^2$
39.631	9.757	0.976

### Thermodynamics of Sorption

In order to investigate the feasibility of the adsorption process, thermodynamic parameters such as free energy change,  $\Delta G^0$ , Enthalpy change,  $\Delta H^0$  and Entropy change,  $\Delta S^0$  were determined (Sujana et al., 2009). These parameters were determined from Equations (6)-(8).

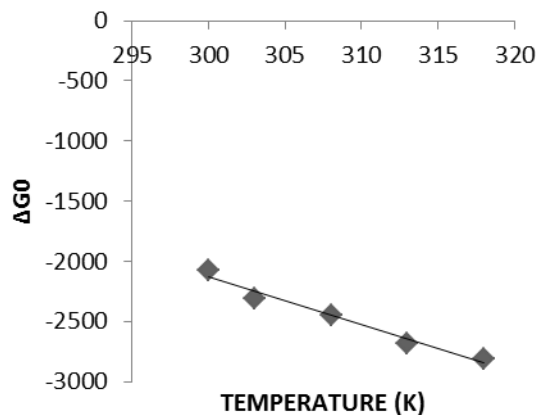
$$Kc = Cad/Ce \quad (6)$$

$$\Delta G^0 = -RT \ln Kc \quad (7)$$

$$\Delta G^0 = -\Delta S^0(T) + \Delta H^0 \quad (8)$$

Where  $C_e$  is the equilibrium Pb(II) ion concentration in solution (mg/L),  $C_{ad}$  is the concentration of Pb(II) ions adsorbed by the adsorbent at equilibrium (mg/L).  $Kc$  is the equilibrium constant,  $R$  is the universal gas constant (8.314J/molK) and  $T$  is the temperature (K). The plot of  $\Delta G^0$  against  $T$  is shown in Figure 10 and the values of  $\Delta S^0$  and  $\Delta H^0$  were deduced from the slope and intercept respectively. The thermodynamic parameters obtained are given in Table 4.

The value of the regression coefficient  $R^2$  gave a good fit to the data. It is clear that the reaction is spontaneous in nature as  $\Delta G^0$  values are negative at all temperatures. Again, the positive values of  $\Delta H^0$  obtained indicates that the adsorption process is endothermic in nature. The heat evolved or absorbed for a physisorption process lies generally in the range of 2.1-20.9KJ/mol while that of chemisorptions is in the range 80-200KJ/mol (Lui and Lui, 2008). Therefore the sorption of Pb(II) ions unto Uguwoaba clay may be attributed to a physical adsorption process. Also the positive value of  $\Delta S^0$  obtained indicated a highly disordered process, which corresponds to an increase randomness at the solid/solute interface during the adsorption of Pb(II) ions unto Uguwoaba clay.



**Figure 10:** Thermodynamic Plot on the Sorption of Pb(II) ions onto Ugwuoba Clay (pH 5.0, Time 2hrs, conc 20mg/L).

## CONCLUSION

The research result obtained in this study clearly showed that Ugwuoba clay can be used as a cheap and effective adsorbent for the removal of Pb(II) ions from aqueous solution. FTIR spectra analysis revealed the presence of some functional groups on the clay surface. The Freundlich isotherm gave a better fit to the experimental data than the Langmuir isotherm. Similarly kinetic studies showed the Lagergren model with a better fit than the Pseudo second order model. Thermodynamic studies revealed the process to be highly disordered, endothermic and spontaneous.

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#### **SUGGESTED CITATION**

Dawodu, F.A. and G.K. Akpomie. 2013. "Sorption Potential of Ugwuoba Clay for Lead(II) Ions from Aqueous Solution". *Pacific Journal of Science and Technology*. 14(2):290-299.

 [Pacific Journal of Science and Technology](http://www.akamaiuniversity.us/PJST.htm)