EQUILIBRIUM ISOTHERM STUDIES ON THE BATCH SORPTION OF COPPER (II) IONS FROM AQUEOUS SOLUTION UNTO "NSU CLAY".

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ABSTRACT

Copper (II) ions adsorption from aqueous solution unto Nsu clay was studied. The aim of the experiment was to utilize the clay as a cheap alternative adsorbent compared to more expensive and relatively less effective techniques for the removal of copper (II) ions from solution. The experiment was performed using batch adsorption technique. Optimum adsorption was achieved at a pH of 5.0 and equilibrium was established within 60 minutes of the process. The equilibrium adsorption data was analyzed by the Langmuir, Freundlich, Dubinin-Radushkevich, Redlich-Peterson and Scatchard isotherm models. The values of the regression coefficient (R^2) of the isotherms gave the best fit (0.9842) with the Redlich-Peterson isotherm. However, all the isotherms applied were appropriate in their own merit in the description of adsorption data. The Langmuir constant values R_L and Freundlich constant n showed a favourable adsorption between Nsu clay and copper (II) ions in solution. The energy of adsorption, E (15.8 KJ/mol) obtained from the Dubinin-Radushkevich isotherm suggested a chemical ion exchange mechanism. The deviation of the Scatchard plot from linearity, R^2 (0.6634) indicated the presence of more than one type of binding sites on the clay surface. The results obtained depicts the usefulness of Nsu clay as a low-cost adsorbent for copper (II) ions.

Keywords: Copper, Dubinin-Radushkevich, Freundlich, Langmuir, Nsu clay, Redlich-Peterson, Scatchard plot.

1 INTRODUCTION

The removal of heavy metals from aqueous effluents have received much attention within environmental research. This is because these metals tend to persist in the environment, are non-biodegradable and highly toxic to plants animals and humans [1]. These heavy metals include copper, lead, cadmium, zinc, mercury, nickel, chromium, iron and Aluminium [2]. Heavy metal contamination is commonly found in waste effluents of different industries such as mining operations, metal plating, ceramics, tanneries, alloy, polymer stabilizers, iron galvanizing, catalyst, semi conductors, pesticides, paints and pigment manufacturing [3]. Although most heavy metals are necessary in small amounts for the normal development of biological cycles, most of them are toxic at high concentrations [4]. Excess intake of copper (above 1.0 mg/l) accumulates in the liver and kidney of animals and humans resulting in ailments like hemochromatosis, severe mucosal irritation, capillary damages, hepatic and renal damages and gastro intestinal problems [5]. Copper acts as a toxin to aquatic and terrestrial organisms even in small concentrations [6]. Various techniques have been used for the removal of copper and other heavy metals from contaminated water which include chemical precipitation, ion exchange, solvent extraction, adsorption, membrane filtration, reverse osmosis, chemical oxidation and reduction and evaporation [7]. Most of these techniques are sometimes ineffective and involves high capital cost, which are not suitable for small scale industries. Although, adsorption unto using activated carbon is expensive. The adsorption process has been found to be more effective and cheaper for the treatment of wastewaters compared to other methods [8]. In the search for cheaper adsorbents for small scale industries many researchers have made use of low-cost adsorbents for copper and other heavy metals removal, which include the use of kaolinite [9], montmorillonite [10], bentonite [11], zeolite [12], secondary clay [13],[14], laterite [15], agricultural by-products and biomass materials [16],[17],[18].

In this present communication, Nsu clay a low cost material present in abundant amount in okigwe local government area of Imo state, Nigeria was harnessed as a cheap adsorbent for copper removal from solution. The clay was used without chemical modification in order to keep the cost of application low. Batch sorption technique was used to determine experimental parameters such as pH, initial metal ion concentration and contact time. Equilibrium isotherms such as Langmuir, Freundlich, Dubinin-Radushkevich, Redlich-Peterson and Scatchard models were used to determine essential characteristics of the adsorbent.

2 MATERIALS AND METHODS

2.1 Preparation of Adsorbent

Nsu clay was collected from Okigwe zone, Imo state, Nigeria. The clay was dispersed in excess distilled water in a pre-treated plastic container, stirred vigorously to ensure uniform dissolution and then filtered in order to get rid of unwanted particles. The filtrate was allowed to settle for 24hrs, excess water was decanted off and the residue was sundried for several days. The clay was dried further in an oven at 105^0C to get rid of remaining water molecules. The dried clay was pulverised and then passed through a 100µm mesh sieve and used as an adsorbent.

2.2 Adsorbent Characterization

The adsorbent was characterized to determine its chemical composition by classical method using the Atomic Absorption Spectrophotometer (AAS) (Buck scientific model 210VGP). The specific surface area (SSA) of the clay was determined using the methylene blue absorption test (MBT) method described by Santamarina et al [19]. The pH point of zero charge (pHpzc) was determined by the method described by Onyango et al [20].

2.3 Preparation of Copper (II) Ion Solution

All the chemicals used in this study were of analytical grade. A stock solution of Copper (II) ion of concentration 1000mg/l was prepared by dissolving appropriate amounts of $CuSO_4.5H_2O$ in doubly distilled water. Lower concentration were prepared from the stock solution by accurate dilutions. The pH of each of the solution was adjusted to the required value by the drop wise addition of 0.1M NaOH or 0.1M H_2SO_4 and checked using a pH meter. Freshly prepared solution was used for each study.

2.4 Adsorption Procedure

The adsorption experiment was carried out using batch technique to determine the effect of pH, (1-8), contact time (10-120 minutes) and initial metal ion concentration, (20-100mg/l). 2g of the adsorbent was added to 20ml of a given solution of copper (II) ions at a room temperature of 27^0C. To determine the effect of a particular parameter on sorption, the parameter was varied while other parameters were kept constant. After a given time the contacted mixture was filtered and the filtrate was analyzed for residual copper (II) ion concentration by the use of the AAS. Each experiment was repeated and the mean value was calculated to ensure quality assurance. The amount of copper (II) ion adsorbed by the clay was calculated from the mass balance equation given in (1).

$$qe = v[Co-Ce]/m \qquad (1)$$

where qe (mg/g) is the equilibrium adsorption capacity of the clay for copper (II) ions, Co (mg/l) is the initial copper ion concentration in solution, Ce (mg/l) is the equilibrium concentration of copper ions in solution, v (litres) is the volume of solution used and m (g) is the adsorbent mass.

3 RESULTS AND DISCUSSION

3.1 Characterization of Clay Mineral

The classical method was used to determine the chemical composition of Nsu clay and the result obtained is presented in Table 1. It is seen from the Table that the major constituents are Silica and Alumina, other elements are present in minute amounts. Therefore, copper(II) ions in solution should be removed mainly by the silica and Alumina groups on the clay surface.

The specific surface area (SSA) obtained using the methylene blue absorption test method is given in Table 1. The SSA of an adsorbent material is the ratio of its surface area to its mass. The SSA of an adsorbent has been reported to have a great effect on its adsorption capacity [21]. The SSA of the clay obtained is not very high, usually SSA of clay minerals are usually lower than that of biomass materials and activated carbon. The pH point of zero charge (pHpzc) obtained is given in Table 1. It has been reported that a relationship exist between the pHpzc and adsorption capacity of a material, and suggested that cations adsorption on an adsorbent will be favoured at pH values higher than the pHpzc while anions adsorption will be favoured at lower pH values [22].

TABLE 1:Chemical Characterization of Nsu Clay.

Composition	% by Weight
SiO_2	46.25
Al_2O_3	38.41
CaO	0.87
Fe_2O_3	0.71
MgO	0.39
LOI	13.37
Specific Surface Area (m^2/g) = 21.53	
pH point of zero Charge (pHpzc) = 4.2	

3.2 Effect of pH and Contact Time

The effect of initial pH of solution on adsorption was determined because it is known to be one of the most important factors affecting sorption. The result on the effect of pH on the adsorption of copper (II) ions is shown in fig.1. Optimum pH of adsorption was recorded at pH 5.0, hence all the adsorption studies were carried out at this optimum pH. Also, the optimum pH value obtained is higher than the pHpzc which agrees with the report that cations adsorption is favoured at higher pH values greater than the pHpzc.

The effect of contact time was determined in order to establish the minimum necessary time for equilibrium sorption to be achieved. The result of contact time effect on adsorption is shown in fig.2. It is seen that equilibrium adsorption was attained within 60 minutes. Sorption experiments in this study

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were performed at a contact time of 2hrs hence we ensured equilibrium attainment.

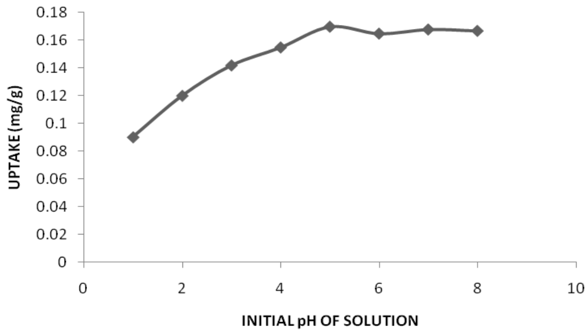

Fig.1: Effect of pH on Copper adsorption unto Nsu clay.

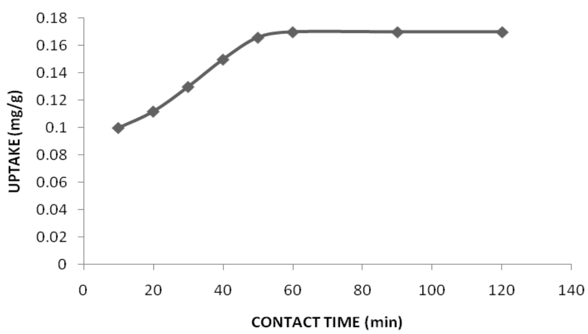

Fig.2: Effect of Contact Time on Copper adsorption unto Nsu clay.

3.3 Effect of Initial Metal Ion Concentration

The result on the effect of initial metal ion concentration on the adsorption of copper(II) ions by Nsu clay is presented in fig.3. An increase in adsorption capacity with increase in metal ion concentration from 20-100mg/l was recorded. This increase is due to the presence of more metal ions in solution available for binding unto the active sites of the clay. However, the percentage of copper adsorbed decreased with increase in initial metal ion concentration as shown in fig.4. This can be explained that all adsorbents have a limited number of active sites and at higher concentrations the active sites become saturated [23].

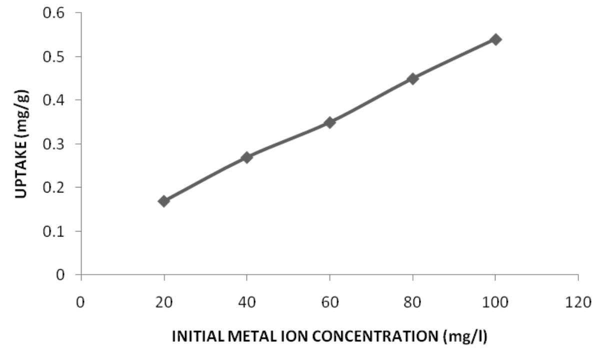

Fig.3: Effect of initial metal ion Concentration on the uptake of Copper adsorption by Nsu clay.

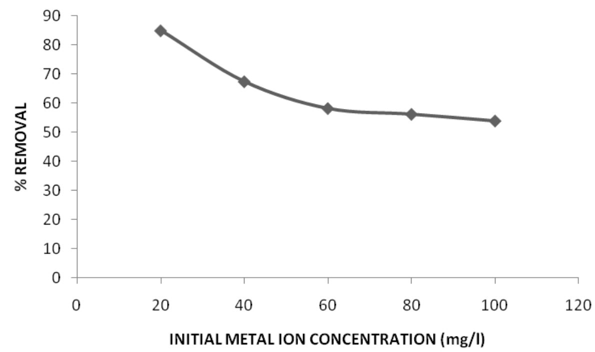

Fig.4: Effect of Initial metal ion concentration on the percentage removal of Copper by Nsu clay.

3.4 Adsorption Isotherms

An adsorption isotherm defines the functional equilibrium distribution of adsorption with different concentrations of adsorbate in solution at constant temperature. They are useful in the evaluation of adsorption capacity and to determine the characteristics of an adsorbent if suitable for application. The Langmuir, Freundlich, Dubinin-Radushkevich, Redlich-Peterson and Scatchard isotherms were applied in this study.

TABLE 2: Equilibrium Isotherm Model Parameters.

Langmuir Isotherm Model					
qmax	**K_L**	**R^2**			
0.655	0.067	0.9014			
R_L	0.43	0.27	0.19	0.15	0.13
Co(mg/l)	20	40	60	80	100
Freundlich Isotherm Model					
1/n	**n**	**Kf**	**R^2**		
0.41	2.45	0.103	0.9664		
Dubinin-Radushkevich Isotherm					
q_D	**B_D (mol2/KJ2)**	**E**	**R^2**		
0.4	0.002	15.8	0.7131		
Redlich- Peterson Isotherm					

K_R	α_R	β	R^2
50	482.4	0.593	0.9842
Scatchard Isotherm			
Q	**b**	**R^2**	
0.57	0.107	0.6634	

3.4.1 Langmuir Isotherm

The Langmuir isotherm is based on the theoretical principle that only a single adsorption layer (monolayer) exist on an adsorbent. It assumes that all the active sites on the adsorbent are homogenous and there is no interaction between active sites [24]. The linear form of the Langmuir isotherm equation is given in (2).

$$Ce/qe = Ce/q_{max} + 1/q_{max}K_L \qquad (2)$$

q_{max} (mg/g) is the maximum uptake capacity for a monolayer coverage, K_L (L/mg) is the Langmuir isotherm constant related to the free energy of sorption. q_{max} and K_L were obtained from the slope and intercept of the plot of *Ce/qe* versus *Ce* shown in fig.5. The Langmuir isotherm parameters are given in Table 2. The value of the regression R^2 obtained (0.9014) showed the applicability of the Langmuir isotherm. This isotherm can be explained with the help of a dimensionless constant separation parameter R_L, given in (3)

$$R_L = 1/[1+K_LCo] \qquad (3)$$

If the value of R_L lies between 0 and 1, the adsorption process is favourable, if R_L is greater than 1, the process is unfavourable [25]. The R_L values obtained in this study lies between 0 and 1, which indicates a high affinity of Nsu clay for copper (II) ions.

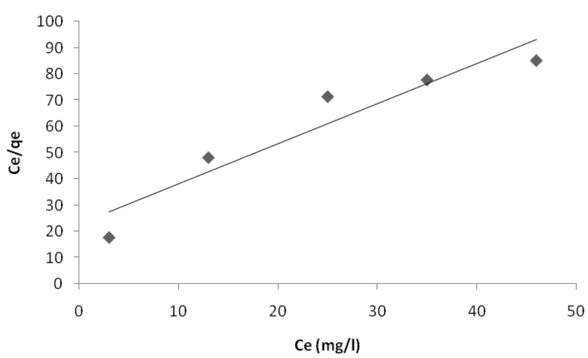

Fig.5: Langmuir Isotherm plot for the adsorption of Copper unto Nsu clay.

3.4.2 Freundlich Isotherm

The Freundlich isotherm describes the extent of heterogeneity of the adsorbent surface involving a multilayer adsorption [24]. The linear form of the Freundlich isotherm equation is given in (4).

$$lnqe = [1/n]lnCe + lnKf \qquad (4)$$

Kf (L/g) and *n* (dimensionless) are Freundlich isotherm constants related to the adsorption capacity and intensity respectively. The linear plot of *lnqe* against *lnCe* is shown in fig.6 and the constant *n* and *Kf* are calculated from the slope and intercept. The Freundlich isotherm constants are presented in Table 2. This isotherm gave a good fit to the experimental data as indicated by the R^2 value (0.9664). Furthermore, if the value of *n* is below or equal to unity, the process is a chemisorptions, but if greater than unity the process is a favourable physical adsorption [26]. Again, the *n* value obtained is greater than unity which indicates a favourable physical adsorption mechanism between copper ions and Nsu clay.

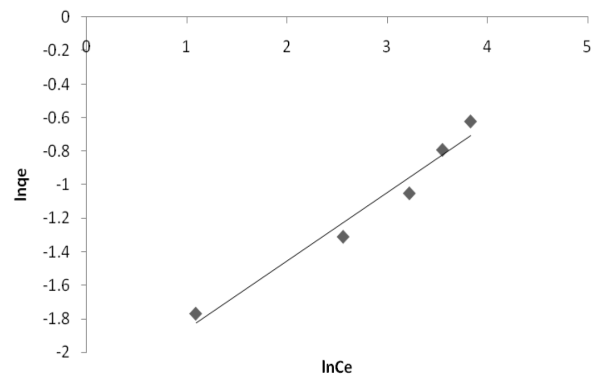

Fig.6: Freundlich Isotherm plot for the adsorption of Copper unto Nsu clay.

3.4.3 Dubinin-Radushkevich (D-R) Isotherm

The Dubinin-Radushkevich (D-R) isotherm was applied in order to deduce the heterogeneity of the apparent energy of adsorption on the adsorbent surface [24]. The linear form of the D-R isotherm equation is given in (5).

$$lnqe = lnq_D – B_D\mathcal{E}^2 \qquad (5)$$

$$\mathcal{E} = RTln[1+1/Ce] \qquad (6)$$

Where q_D (mg/g) is the adsorption capacity of the adsorbent, B_D (mol^2/KJ^2) is the D-R isotherm constant related to the adsorption energy, $\mathcal{E}$ is the Polanyi potential, *R* is the gas constant (KJ/mol/K) and *T* (K) is the absolute temperature.

The D-R isotherm was applied by a linear plot of *lnqe* against $\mathcal{E}^2$ shown in fig.7. The D-R isotherm parameters are given in Table 2. The mean adsorption energy *E* (KJ/mol) can be obtained from the value of B_D [27] by using (7).

$$E = 1/[2B_D]^{1/2} \qquad (7)$$

When the value of *E* is less than 8 KJ/mol the sorption process is said to be dominated by physisorption, if *E* is between 8 and 16 KJ/mol, the process is dominated by a chemical ion exchange mechanism and, the value of *E* when greater than 16 KJ/mol is dominated by a chemical particle diffusion [27]. The value of *E* obtained in our study is 15.8 KJ/mol which indicated a chemical ion exchange process between Nsu clay and copper (II) ions in solution.

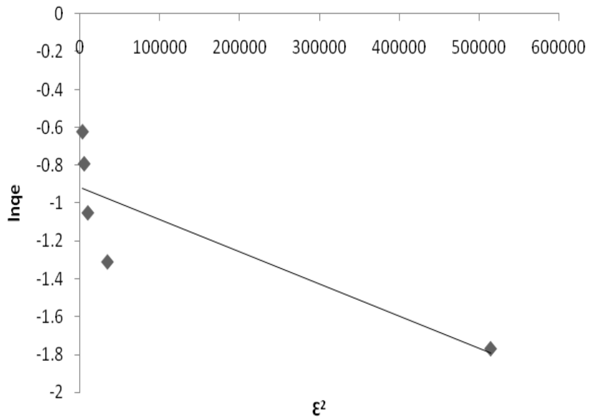

Fig.7: Dubinin-Radushkevich Isotherm plot for the adsorption of Copper unto Nsu clay.

3.4.4 Redlich-Peterson (R-P) Isotherm

The Redlich-Peterson (R-P) isotherm can be applied to a homogenous or heterogeneous system due to its high versatility. Unlike the Langmuir, Freundlich and D-R isotherm, the R-P isotherm incorporates three parameters in a single equation [28]. The linear form of the R-P isotherm is given in (8).

$$ln[K_RCe/qe\text{-}1] = ln\alpha_R + \beta lnCe \qquad (8)$$

where K_R (L/g) and α_R $(L/mg)^{1/\beta}$ are the R-P isotherm constant, β is the exponent which lies between 0 and 1. At high concentrations the R-P isotherm approaches the Freundlich isotherm while at low concentration it approximates to Henry's law. Since this isotherm incorporates three parameters, the application is more complex in term of linear regression analysis. This isotherm was applied by varying the isotherm parameter K_R to obtain the maximum regression R^2 possible for the linear plot of $ln[K_RCe/qe\text{-}1]$ against *lnCe*. This plot is shown in fig.8 and the constants β and α_R were obtained from the slope and intercept respectively. The values of K_R, α_R, β and the regression R^2 obtained are recorded in Table 2. From the values of the regression coefficient obtained in Table 2, it is seen that the R-P isotherm gave the best fit, followed by the Freundlich, then the Langmuir and the D-R isotherm.

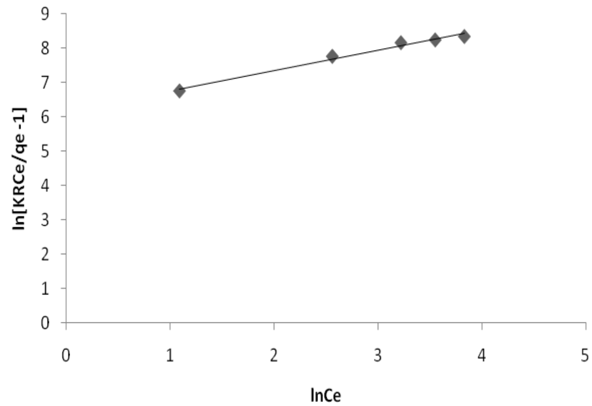

Fig.8: Redlich-Peterson Isotherm plot for the adsorption of Copper unto Nsu clay.

3.4.5 Scatchard Plot Analysis

The Scatchard plot analysis also called independent site oriented model was applied in order to obtain a comprehensive study on the affinity of binding sites and to analyse the result of the adsorption isotherm [29]. The Scatchard plot equation is given in (9).

$$qe/Ce = Qb - qeb \qquad (9)$$

where *Q* (mg/g) and *b* (L/mg) are the Scatchard adsorption isotherm constants. If the plot of *qe/Ce* versus *qe* (fig.9) gives a straight line, then the adsorbent consist of only one type of binding site (homogenous surface). However, if the plot deviates from linearity, then the adsorbent consist of more than one type of binding site [29]. The value of the regression R^2 obtained (0.6634) showed a deviation from linearity which implies that Nsu clay is made up of more than one type of binding site. The presence of different types of active sites on a clay surface is useful for effective adsorption of metal ions from solution.

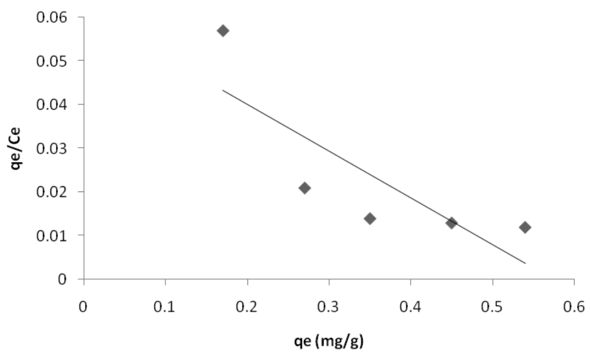

Fig.9: Scatchard plot for the adsorption of copper unto Nsu clay.

4 CONCLUSION

The equilibrium isotherm models applied in the analysis of experimental data showed the potential of Nsu clay as a low cost adsorbent for copper (II) ions removal from solution.

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