

Kinetic, Equilibrium, and Thermodynamic Studies on the Adsorption of Cadmium (II) Ions using “Aloji Kaolinite” Mineral.

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ABSTRACT

The potential of Aloji kaolinite as a low-cost adsorbent for cadmium (II) ions from aqueous solution was investigated. The effect of pH, initial metal ion concentration, contact time, sorbent dose, and temperature on the adsorption process were determined using batch adsorption technique. Optimum adsorption was obtained at pH, 6.0 and equilibrium adsorption was achieved within 50 minutes. The chemical composition of the kaolinite was determined by classical method and revealed silica and alumina as the major constituents. Equilibrium adsorption isotherms such as Langmuir, BET, and Tempkin model were applied to the experimental data. The values of the linear regression coefficient (R^2) showed the best fit with the Tempkins isotherm (0.975), then the Langmuir isotherm (0.939) and the least the BET isotherm (0.812). The Elovich and intraparticle diffusion models were both applicable in the description of the kinetics of sorption. Thermodynamic analysis revealed negative values of ΔG^0 at all temperatures which indicated a spontaneous process. The entropy change obtained, (274.5J/mol/K) showed that the adsorption process is a highly disordered one. The positive value of ΔH^0 , (79.675KJ/mol) also indicated an endothermic adsorption process. The results of this study showed that Aloji kaolinite can be used as a low-cost adsorbent for cadmium (II) ions removal from aqueous industrial effluents.

(Keywords: adsorption, cadmium (II), kaolinite, equilibrium, kinetic, thermodynamic)

INTRODUCTION

Pollution by heavy metals has become a major issue in the environmental management of many nations. This is because the content of heavy

metals in most industrial effluents and wastewaters usually exceed the admissible sanitary standards. If these effluents are emitted without treatment they may result in adverse effects on the environment and consequently affect human health (Amer et al., 2010). These metals are highly toxic, non biodegradable, and tend to accumulate causing different health problems in living organisms.

Cadmium, when present in high concentrations in humans, can cause kidney problems, liver damage, a choking sensation, diarrhea, abdominal pain, vomiting, and skeletal calcification. Cadmium is very toxic because of the absence of a homeostatic control for this metal in the human body. It is an enzyme inhibitor which affects calcium metabolism and the regulation of ions in humans (Igwe and Abia, 2007).

Recently, the main goal involving treatment of heavy metals and cadmium involves reduction of these pollutants in wastewaters to very low concentrations. The techniques employed for the removal of cadmium and other heavy metals from aqueous solution include ion exchange, precipitation, filtration, membrane separation, reverse osmosis, solvent extraction, evaporation and electrochemical treatment (Gode and Pehlivan, 2006). These methods have the disadvantages of high cost, low selectivity, difficulty to apply, incomplete metal recovery, require trained personnel's, high energy requirement, time consuming and the generation of toxic slurries which are difficult to remove (Volesky, 2001). Also, ion exchange and precipitation are not useful when the metal ion concentration in solution is within the range 1-100mg/l. Therefore the development of other techniques is important.

Adsorption as a technique for wastewater treatment has been found to be effective and economically feasible. The adsorption process is usually based on the mechanism of physical adsorption, ion exchange, complexation and precipitation. Adsorption does not necessarily consist of a single mechanism and in most adsorption processes several mechanisms act in combination which makes it difficult to distinguish between the single steps. However, the adsorption technique is more effective, hence many researchers have made use of various materials for the adsorption of cadmium and other heavy metals, which include the use of agricultural waste, secondary clay, kaolinite, bentonite, montmorillonite and sepiolite (Dawodu et al., 2012a). Kaolinite has seldom been used as an adsorbent because of its small surface area which is usually less than $20\text{m}^2/\text{g}$ and its low cation exchange capacity (Suraj et al., 1998).

Aloji kaolinite obtained from Aloji in Ofu local government area, Kogi state Nigeria, is a primary clay mineral which is abundant in nature, easily accessible and so can be used as a low-cost adsorbent for the removal of heavy metals from solution. This present study investigated the use of Aloji Kaolinite as a low-cost adsorbent for cadmium (II) ions from aqueous solution. The effect of pH, initial metal ion concentration, contact time, sorbent dose, and temperature on the sorption process were studied. Equilibrium, kinetic and thermodynamic data were also determined.

MATERIALS AND METHODS

Sample Preparation

Aloji kaolinite clay was collected from Aloji in Ofu local government area of Kogi state Nigeria. It was dispersed in excess distilled water in a pre-treated plastic container, stirred to ensure uniform dissolution and then filtered in order to get rid of unwanted materials. The filtrate was kept for 24hrs to settle, after which excess water was decanted off and the residue sundried to get rid of excess water molecules. The kaolinite was further oven dried at a temperature of 105°C for 2hrs, then crushed and passed through a mesh sieve of size $100\mu\text{m}$. The prepared sample was then kept until use.

Sample Characterization

The chemical composition of Aloji kaolinite was determined by classical method with the use of the Atomic Absorption Spectrophotometer (AAS) (Buck scientific model 210VGP).

The specific surface area (SSA) of Aloji kaolinite was determined by the methylene blue adsorption test method (Santamarina et al., 2002). 2g of the kaolinite was weighed and dissolved in 200ml of de-ionized water, after which 10ppm of methylene blue solution was added and agitated for 2hrs.

After the solution was kept for 24hrs to achieve equilibrium, 10ml aliquot was taken and centrifuged, then analyzed by the UV/Visible Spectrophotometer (Spectrumlab 752s) in order to determine the amount of methylene blue absorbed. This procedure was repeated for 20, 30, 40, 50, 60, and 70ppm solutions of methylene blue. The point of complete cation replacement was determined from the plot of the concentration of methylene blue against the amount absorbed. The specific surface area (SSA) was then calculated using (1).

$$\text{SSA} = [M_B \times A_v \times \text{AMB}] / 319.98M_s \quad (1)$$

Where M_B is the amount of methylene blue absorbed at the point of complete of complete cation replacement, A_v is the Avogadro's number, AMB is the area covered by one molecule of methylene (assumed to be 130\AA^2) and M_s is the mass of kaolinite.

The pH point of zero charge (pHpzc) was determined by the method described by Onyango et al. (2004). 50ml of 0.01M NaCl was placed in different flask and corked. The pH of the solution in different flask was adjusted to values ranging from 2-12. 0.5g of the kaolinite was added and agitated in a shaker for hr and allowed to settle for 48hrs at a room temperature of 27°C . The zeta potential (mV) of each solution was then determined. The pHpzc of the kaolinite was determined from the plot of the zeta potential against initial pH of solution.

Batch Adsorption Studies

De-ionized water was used for preparing and dilution of all cadmium (II) solutions. A solution of cadmium (II) ions of concentration 500mg/l was prepared by dissolving appropriate amount of analytical grade of $\text{Cd}(\text{NO}_3)_2$ in de-ionized water. Several lower concentrations ranging from 20-100mg/l were prepared from the stock solution by serial dilution. The pH of each solution was adjusted to the required value by the use of a pH meter. Freshly prepared solutions were used for each experiment. The adsorption was carried out using batch method at different pH, (1-8), initial metal in concentration, (20-100mg/l), Contact time, (10-120min), sorbent dose, (1-5g) and temperature, (27-45°C).

Each adsorption study was carried out by contacting 2.0g of Aloji kaolinite with 20ml of a given concentration of cadmium (II) ion in a thermostat water bath for temperature regulation. In each case a parameter was varied while the others were kept constant. At the end of the given contact time, the solution was filtered and the filtrate was analyzed for residual cadmium (II) ion concentration in solution using the Atomic Absorption Spectrophotometer (AAS). Each experiment was repeated and the mean value was calculated to obtain quality assurance. The adsorption uptake capacity was calculated as described previously (Dawodu et al., 2012a).

RESULTS AND DISCUSSION

Characterization of Aloji Kaolinite

The chemical composition of Aloji kaolinite as determined by classical method is represented in Table 1. The major components present on the surface are Silica and Alumina which are expected to contribute greatly in the adsorption of cadmium ions from solution (Dawodu et al., 2012b).

Furthermore, the specific surface area (SSA) of the clay is not large, it has been reported that the SSA for most kaolinite minerals are usually less than $20\text{m}^2/\text{g}$, however, that obtained in this study is slightly higher as seen in Table 1(Dawodu et al., 2012b).

The pH_{pzc} of the kaolinite is also shown in Table 1. A relationship between the pH_{pzc} of an adsorbent and its adsorption capacity has been

explained in that cations adsorption on any adsorbent will be favored at pH values higher than the pH_{pzc} while that of anions are favored at lower values (Dawodu et al., 2012b).

Table 1: Characterization of Aloji Kaolinite (Dawodu et al., 2012b).

Composition	Weight (%)
SiO ₂	53.9
Al ₂ O ₃	27.2
CaO	0.6
MgO	0.4
Na ₂ O	0.5
Fe ₂ O ₃	0.9
K ₂ O	1.2
TiO ₂	0.2
MnO	0.1
LOI	15.0
SSA (m ² /g) = 19.8	
pH _{pzc} = 5.5	

Effect of pH on Adsorption

The initial pH of a solution has been known to be a very important factor in adsorption studies. As a result, characterization of its effect on adsorption is necessary for an accurate evaluation of experimental data. If the metal binding sites are weakly acidic or basic, the availability of free sites is dependent on pH. The pH determines the net charge on the clay which in turn determines whether cadmium ions can bind to the surface or not. Adsorption process is reversible in the presence of mineral acid because the net charge on the adsorbent can be regulated, this accounts for the reason why desorption is possible (Anayurt et al., 2009).

At pH values greater than 7, several hydroxyl low soluble species such as, $\text{Cd}(\text{OH})_2$ and $\text{Cd}(\text{OH})_3$ can be precipitated in solution. The result for the effect of pH on the removal of cadmium (II) from solution unto Aloji kaolinite is shown in Figure 1. As observed, maximum adsorption was achieved at pH values 4-6. Subsequent studies were conducted at pH 6.0 as the optimum pH of adsorption, where metal precipitation was also avoided. The mechanism of adsorption is a reflection of the physicochemical interaction of metal ions in solution and the active sites on the clay. When the pH of the solution is low, the solution is highly acidic which makes the net charge on the surface to be positive. Subsequently, metal ions competes with protons for the active sites on the clay leading to a

decrease in adsorption. Also, the protonation of the active sites on the clay reduces the number of negatively charged sites needed for binding hence a decrease in adsorption. On the other hand, as the pH increases the binding sites becomes deprotonated and free for cadmium ions to bind, this reduces the competition between cadmium ions and the protons for the active sites, which accounts for the increase in adsorption observed. Similar results have been reported (Dawodu et al., 2012c).

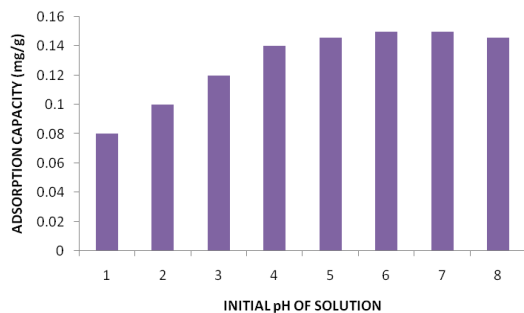


Figure 1: Effect of pH on the adsorption of Cadmium (conc,20mg/l, time,2hrs, temp, 300K).

Effect of Initial Metal Ion Concentration

The initial metal ion concentration is an important factor to be determined in adsorption studies. This is because the rate and uptake of a metal ion by an adsorbent is dependent upon its initial concentration in solution. It also makes the application of equilibrium adsorption isotherm models possible. The result on the effect of initial cadmium (II) ion concentration on its adsorption unto Aloji kaolinite is presented in Figure 2.

An increase in the adsorption uptake capacity with increase in cadmium ion concentration was recorded. This is simply due to the availability of more cadmium ions in solution at higher concentrations available for binding unto the active sites of the kaolinite. As a result of the increase in the concentration of metal ions in solution, there is a corresponding increase in the driving force generated to overcome resistance to mass transfer between the adsorbent and the liquid phases (Babarinde and Babalola, 2010).

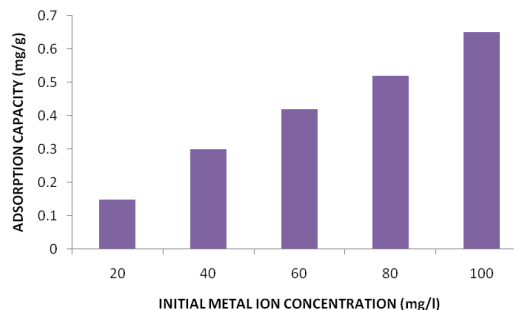


Figure 2: Effect of initial metal ion concentration on adsorption of Cadmium. (pH,6.0, time,2hrs, temp,300K).

Effect of Contact Time

The dependence of the amount of cadmium (II) ions adsorbed on contact time is presented in Figure 3. An increase in adsorption capacity with increase in contact time was recorded; equilibrium sorption was achieved within 50mins of the process after which the rate of adsorption became fairly stable. Equilibrium attainment was ensured in all other studies as experiments were performed at a contact time of 2hrs. The fast uptake of cadmium ions at the initial stage is simply due to the availability of abundant active sites on the surface of the kaolinite which became saturated as time progresses. As these sites are progressively filled the more difficult the sorption becomes, as the sorption process tends to be more unfavorable. This is the general characteristics of the effect of contact time on the adsorption of metal ions.

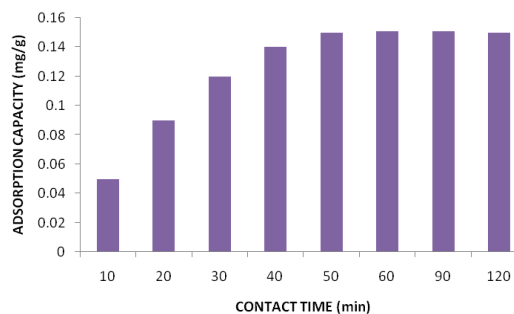


Figure 3: Effect of contact time on the adsorption of cadmium (pH,6.0, conc,20mg/l, tempo,300K).

Effect of Sorbent Dose

The result for the effect of kaolinite dose on the removal of cadmium (II) ions from aqueous solution is shown in Figure 4. As observed, there is a decrease in the adsorption capacity with increase in kaolinite concentration. This is due to the decrease in total adsorption surface area available to cadmium (II) ions resulting from the overlapping or aggregation of active sites. As the sorbent dose increased the amount of cadmium (II) ions adsorbed per unit mass of kaolinite decreased leading to the reduction in the adsorption capacity. Furthermore, other reasons have been recommended to explain the decrease in adsorption capacity with increase in sorbent dose which include electrostatic interactions, availability of solute and interferences between adsorption sites.

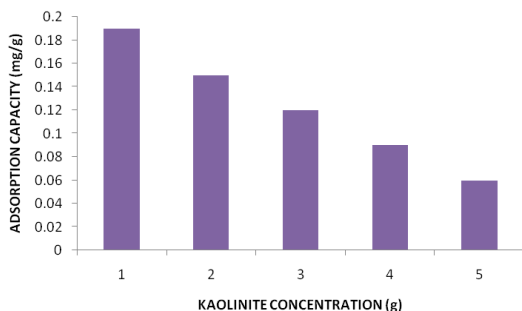


Figure 4: Effect of sorbent dose on the adsorption of Cadmium. (pH,6.0, conc,20mg/l, time,2hrs, temp, 300K).

Effect of Temperature

The adsorption of cadmium (II) ions onto Aloji kaolinite clay at different temperatures of 27-45⁰C showed an increase in adsorption capacity with increase in temperature, this is illustrated in Figure 5, indicating that the adsorption process is endothermic in nature. These increases in adsorption capacity with temperature also suggest chemical interactions between the adsorbent and adsorbate. However, most adsorption studies on the effect of temperature carried out by various researchers presented different types of results. Temperature-independent effect on adsorption has been reported (Ahuja et al., 1999). Also, a decrease in adsorption with increase in temperature has also been reported (Aksu, 2001).

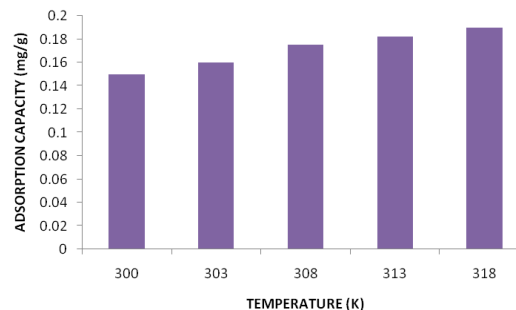


Figure 5: Effect of Temperature on the adsorption of Cadmium. (pH,6.0, conc, 20mg/l, time, 2hrs).

Table 2: Equilibrium Isotherm Parameters.

Langmuir Isotherm		
qm	b	R ²
1.247	0.029	0.939
BET Isotherm		
C _B	qs	R ²
-5.35	0.0996	0.812
Temkin Isotherm		
A	B	R ²
0.3514	0.240	0.975

Adsorption Isotherm

An adsorption isotherm expresses the functional equilibrium distribution of adsorption with different concentrations of adsorbate in solution at constant temperature. These isotherms are generally used to establish the relationship between the amount of metal ion adsorbed and its equilibrium concentration in solution. They also help provide vital information to understand the mechanism of the adsorption process. A number of isotherms have been developed to describe equilibrium relationships. In this study, the Langmuir, Brunauer-Emmett-Teller (BET) and Temkin isotherms were applied to the experimental data (Foo and Hammed, 2010).

The Langmuir isotherm is applied to monolayer adsorption onto a surface containing a finite number of identical binding sites, with no interaction between the sites on the adsorbent. The linear form of the Langmuir isotherm equation is given in (2).

$$C_e/q_e = C_e/q_m + 1/qmb \quad (2)$$

Where C_e (mg/L) is the equilibrium concentration of cadmium (II) ions in solution, and q_e (mg/g) is the adsorption capacity, q_m (mg/g) is the maximum adsorption capacity corresponding to a complete monolayer coverage and b (L/mg) is the Langmuir isotherm constant related to the energy of adsorption which quantitatively reflects the affinity between the adsorbent and adsorbate. The applicability of the isotherm was confirmed by a linear plot of C_e/q_e against C_e shown in Figure 6, the constants q_m and b were calculated from the slope and intercept, respectively. Table 2 showed the Langmuir parameters obtained.

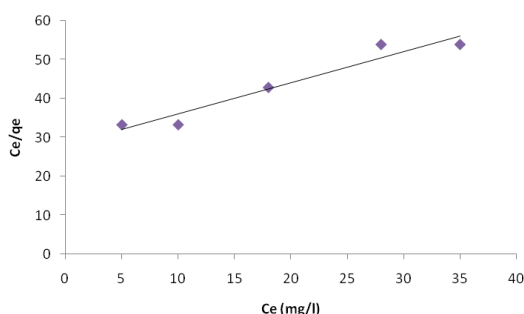


Figure 6: Langmuir Isotherm Plot. (pH,6.0, time,2hrs, temp, 300K).

The BET isotherm is a theoretical equation applied widely in gas-solid equilibrium systems. It was developed to derive multilayer adsorption systems with relative pressure ranges from 0.05 to 1.50 corresponding to a monolayer coverage lying between 0.50 to 1.50. This isotherm has been applied in liquid solid interface and is given in its linear form in (3).

$$C_e/q_e[C_s-C_e]=1/q_s C_B+[C_B-1/q_s C_B]C_e/C_s \quad (3)$$

Where C_B (L/mg) is the BET adsorption isotherm, C_s (mg/L) is the adsorbate monolayer saturation concentration and q_s (mg/g) is the theoretical isotherm saturation capacity. This isotherm was applied by a linear plot of $C_e/q_e[C_s-C_e]$ versus C_e/C_s shown in Figure 7 and the BET isotherm parameters are given in Table 2.

The Tempkin isotherm model was applied and is based on the assumption that the free energy of adsorption is a function of the surface coverage. The linear form of the Tempkin isotherm equation is given in (4).

$$q_e = B \ln A + B \ln C_e \quad (4)$$

where $B = [RT/b_T]$ in (J/mol) is a constant related to the heat of adsorption, A (L/g) is the equilibrium binding constant which corresponds to the maximum binding energy, R is the ideal gas constant and T (K) is the absolute temperature. The Tempkin isotherm plot is shown in Figure 8. The isotherm parameters are given in Table 2.

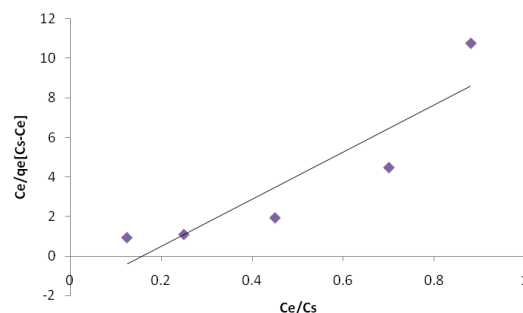


Figure 7: BET Isotherm Plot. (pH,6.0, time,2hrs, temp,300K).

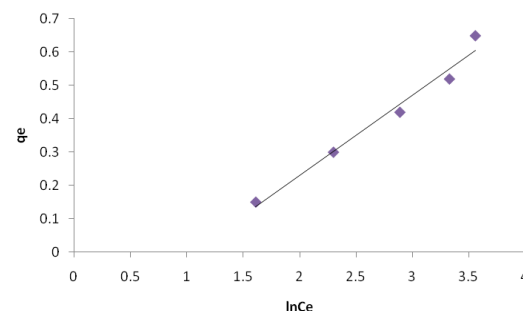


Figure 8: Tempkin Isotherm plot. (pH,6.0, time,2hrs, temp,300K).

A comparison of the isotherms based on their coefficient of determination (R^2) showed the best fit with the Tempkin, followed by the Langmuir and then the BET isotherm. However, the high value of the regression indicated that the adsorption of cadmium (II) ions by Aloji kaolinite could be well described by the three isotherms.

Adsorption Kinetics

The study of the adsorption kinetics is the main factor for designing an appropriate adsorption system and quantifying the changes in adsorption with time requires that an appropriate kinetic model is used. The kinetics of metal sorption governs the rate, which determines the residence time and is one of the important characteristics defining the efficiency of an adsorbent. The

sorption kinetics can be controlled by several independent processes which could act in series or in parallel, such as external mass transfer, bulk diffusion, chemisorptions, and intraparticle diffusion (Ho and Mackay, 2000). This Elovich equation and Weber and Morris intraparticle diffusion model were applied in the description of the kinetics of sorption.

Table 3: Kinetic Model Parameters.

Elovich Kinetic Model		
β	α	R^2
16.95	0.014	0.986
Weber and Morris Model		
K_d	I	R^2
0.022	-0.014	0.951

The Elovich equation was developed to describe the kinetics of chemisorptions of gas onto solids. The simplified form of the Elovich equation is given in (5).

$$qt = [1/\beta]\ln(\alpha\beta) + [1/\beta]\ln t \quad (5)$$

where β (g/mg) is a constant related to the extent of surface coverage and the activation energy of chemisorptions, α (mg/g/min) represent the rate of chemisorptions at zero coverage. The linear plot of qt against $\ln t$ is shown in Figure 9 and the constants α and β were obtained from the intercept and slope respectively. The Elovich isotherm parameters are given in Table 3.

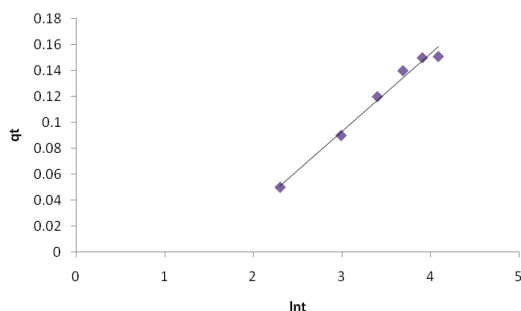


Figure 9: Elovich Model Plot (pH,6.0, conc,20mg/l, temp,300K).

The Weber and Morris intraparticle diffusion was used to elucidate the mechanism of diffusion (Weber and Morris,1963) . The model equation is given in (6).

$$qt = K_d t^{1/2} + I \quad (6)$$

where I is the intercept and K_d is the intra-particle diffusion rate constant. The larger the intercept the greater the contribution of surface sorption in the rate determining step. This model was applied by a linear plot of qt versus $t^{1/2}$ illustrated in Figure 10. The constants I , K_d and the regression, R^2 are presented in Table 3. Intraparticle diffusion is the sole rate determining step if the plot passes through the origin. The deviation from linearity indicated the existence of some boundary layer effect and showed that intraparticle is not the only rate determining step.

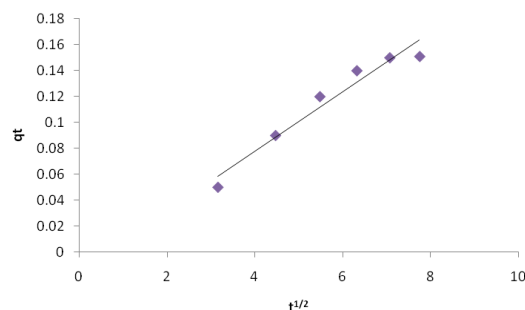


Figure 10: Weber and Morris Intraparticle Diffusion Model Plot. (pH,6.0, conc,20mg/l, temp, 300K).

Thermodynamic of Adsorption

The thermodynamics was applied from the data obtained on the effect of temperature on sorption. The thermodynamic parameters such as changes in Gibbs free energy, ΔG^0 (KJ/mol), Enthalpy, ΔH^0 (KJ/mol) and entropy, ΔS^0 (J/mol/K) were calculated from Equations (7) - (9).

$$K_{ad} = C_{ad}/C_e \quad (7)$$

$$\Delta G^0 = -RT\ln K_{ad} \quad (8)$$

$$\Delta G^0 = \Delta S^0(T) + \Delta H^0 \quad (9)$$

The plot of the change in free energy against temperature is shown in Figure 11 and the thermodynamic parameters are given in Table 4. The value of the Regression coefficient (R^2) showed that this plot gave a good fit to the experimental data. ΔS^0 and ΔH^0 were obtained from the slope and intercept. respectively. Negative values of ΔG^0 obtained at all temperatures indicated a spontaneous process. The positive value of the entropy change

obtained showed that the adsorption process is a highly disordered one. This positive value has been reported to be as a result of increase in the dehydration steps of the metal ions which are known to possess relatively high energies of solvation thereby resulting in the stabilization of water sheaths in the absence of the adsorbent [39]. Also, the positive value of the enthalpy change obtained indicates an endothermic process, which accounts for the increase in adsorption capacity with increase in temperature.

Table 4: Thermodynamic Parameters.

T(K)	ΔG^0	ΔH^0	ΔS^0	R^2
300	-2.74	79.675	274.5	0.994
303	-3.49			
308	-4.98			
313	-6.02			
318	-7.78			

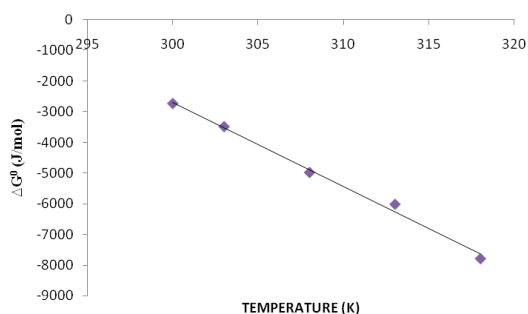


Figure 11: Thermodynamic Plot (pH,6.0, conc, 20mg/l, time,2hrs.).

CONCLUSION

The results of this study showed that Aloji kaolinite can be used as a low cost adsorbent for the removal of cadmium (II) ions from aqueous solution.

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