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EVALUATION OF UNWANA CLAY'S POTENTIAL FOR HEAVY METAL ADSORPTION: INSIGHTS INTO SEQUESTRATION CAPACITY AND ENVIRONMENTAL APPLICATIONS

Ikeagwuani, Chidube
Department of Science Laboratory Technology, Akanu Ibiam Federal Polytechnic Unwana, P.M.B. 1007, Unwana, Nigeria
Corresponding author: ikeagwuanichidube@gmail.com, +2348030538912

Abstract
This study investigates the potential of Unwana clay, sourced from Akanu Ibiam Federal Polytechnic in Ebonyi State, for the adsorption and sequestration of heavy metals. Utilizing a batch method at ambient temperature, we examined the sequestering process. The experimental results were found to fit the Langmuir isotherm model, indicating a high affinity for metal ion adsorption. Specifically, the maximum adsorption capacities (Q_0) were determined to be 61.3 mg Pb(II) g^{-1} and 26.8 mg Cu(II) g^{-1} at a pH of 7.0 and a temperature of 25ºC, using Unwana clay with a particle size of 200 mesh. The findings demonstrate that Unwana clay can effectively adsorb significant amounts of heavy metal cations from aqueous solutions. These results suggest that Unwana clay is a promising sorption material for heavy metals, potentially paving the way for new commercial applications and environmental remediation strategies.

Keywords: Sequestering, Adsorption, Unwana clay, Heavy metals, Isotherm

Introduction
The removal of heavy metals from aqueous solutions is essential due to their prevalence in industrial waste streams, particularly from sectors such as chemical manufacturing, electroplating, metallurgy, mining, and battery production. This issue has garnered significant attention in recent years, primarily because heavy metals can easily accumulate in marine organisms, ultimately entering the human food chain and posing serious health risks to consumers (Ahmaruzzaman, 2011).

Researchers are increasingly employing various sequestering processes to address the challenge of heavy metal removal from waste streams, with activated carbon frequently used as a conventional adsorbent. However, despite its effectiveness, activated carbon is often costly, prompting a demand for safer and more economical alternatives for treating contaminated water. This has led to a growing interest in exploring low-cost adsorbents that can replace commercially available activated carbon (Kaur & Duhan, 2017).

It is crucial to investigate a wide range of potential inexpensive materials, such as sawdust, rice husk, coconut husk, and oil palm shell, to assess their effectiveness in heavy metal removal (Rahman et al., 2014; Ayob et al., 2021; Lim & Aris,2014 and Elbehiry et al., 2021) .

The objective of this study is to contribute to the development of cost-effective adsorbents and evaluate their potential applications in the removal of heavy metals from wastewater.

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Experimental

Unwana clay was obtained from the premises of Akanu Ibiam Federal Polytechnic, Unwana, Ebonyi State, Nigeria. Physicochemical analysis of the Unwana clay was carried out and are presented in tables 1 and 2. The Unwana clays were used directly without any treatment. Chemicals used were of analytical grade and all experiments were performed using distilled water.

Table 1. Composition of Unwana clay

Composition	Content(%)
SiO_2	61.2
CaO	1.3
Fe_2O_3	2.9
MgO	8.1
Al_2O_3	6.9
NaO	0.1
K_2O	0.5

Table 2. Physical property of Unwana clay

Physical property	250 mesh clay
Bulk density	0.82
Colour	Grey
Moisture	11.0
pH	8.0
Specific gravity (g/cm^3)	2.1

Preparation of lead and copper ion solutions

Standard stock solutions of 500 mg/L Pb(ii) and Cu(ii) were prepared as follows:
Dissolve the required amount of metal salt into 1 L of distilled water and stir. The masses used are listed below:

Table 3. Metal ion salts

Metal ion	Metal salt	Mass to be dissolved
Pb(ii)	$Pb(NO_3)_2$	0.7992
Cu(ii)	$Cu(NO_3)_2.3H_2O$	1.9010

Mass calculation is as follows: 500 ppm = 500 mg metal/L. To make 1 L, you need 500 mg metal (0.500 g metal).
0.500 g metal x [MW (salt actually weighed) / AW (metal in salt)] = mass of salt to weigh out (mass of salt to be dissolved).

Where MW = molecular weight
AW = atomic weight

Sequestering Studies

All experiments were carried out at room temperature in batch mode. Batch mode was selected because of its simplicity and reliability. The batch experiments were run in different glass flasks of 100 ml capacity. Prior to each experiment, a predetermined amount of adsorbent was added to each flask. The stirring was kept constant for each run throughout the experiment to ensure equal mixing. The desired pH was maintained using dilute NaOH/HCl solutions. Each flask was filled with a known volume of sample

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having desired pH before commencing stirring. The flask containing the sample was withdrawn from the shaker at the predetermined time interval, filtered through a filter paper. The experiments were carried out under different experimental conditions.

Single Solute System

Adsorption measurements were made by a batch technique at ambient temperature. A mass of 0.1g of Unwana clay was placed in a 100 ml stopper Erlenmeyer flask containing 50 ml of metal ion solution of known concentration and pH. All experiments were carried out at pH 7.0 (except when the pH effect was studied). The pH of each solution was adjusted to the desired value by addition of dilute HCl or NaOH solutions. The solutions were shaken vigorously for a given time period to reach equilibrium. The agitation speed was kept constant for each run to ensure equal mixing. After completion of a pre-selected shaking time, the suspensions were filtered using filter paper, and then the supernatant solution in each flask was analysed by atomic absorption spectrometer (AAS) for residual metal content. Langmuir isotherm models were fitted to the adsorption data and their constants were evaluated.

The uptake of metal ions in single systems was calculated by the difference in their initial and final concentrations. A mass balance states that the amount of solute (adsorbate) adsorbed onto the solid (adsorbent) must be equal to the amount of solute removed from the solution, or in mathematical terms:

$q_e = v\ (Co - Ce)$……(1)
$q = v\ (Co - Ce) / w$…….(2)

Where
q = amount of metal ion adsorbed per unit mass of adsorbent (mg/g adsorbent).
v = volume of liquid (L).
C_o = initial concentration of metal ion in the aqueous phase (mg/L).
C_e = final concentration of metal ion in the aqueous phase (mg/L).
w = mass of adsorbent (g).

It is well known that q_e is sometimes more conveniently expressed in units of mass (or moles) of solute per unit volume of the solid phase (8); however, in this investigation, mass of solute per unit mass of the solid phase is the better choice. For this reason C_o and C_e are expressed in mg solute per liter of solution and the adsorption graphs are presented on a mg/g basis.
The percentage adsorption was calculated as follows:

(%) Adsorption = $(C_o - C_e) / C_o$ X 100…….(3)

Results and Discussion

The influence of several operational parameters such as shaking time, initial metal ion concentration, amount of adsorbent and initial pH for the effective removal of Pb(ii)and Cu(II) from solution by sequestering onto Unwana clay were studied. The results were expressed as the amount of metal ions adsorbed onto the clay at any time (q, mg/g) or at equilibrium (q_e) and the concentration of metal ions that remain in solution at equilibrium (Ce, mg/L). The % removal of
metal ions was also calculated and reported.

The Effect of Shaking Time and Initial Pb(II) Concentration

Experimental conditions: Contact time 1-35 min, Initial Pb(II) concentrations 20-100 mg/L, Mass of Unwana clay 0.1 g, pH 7

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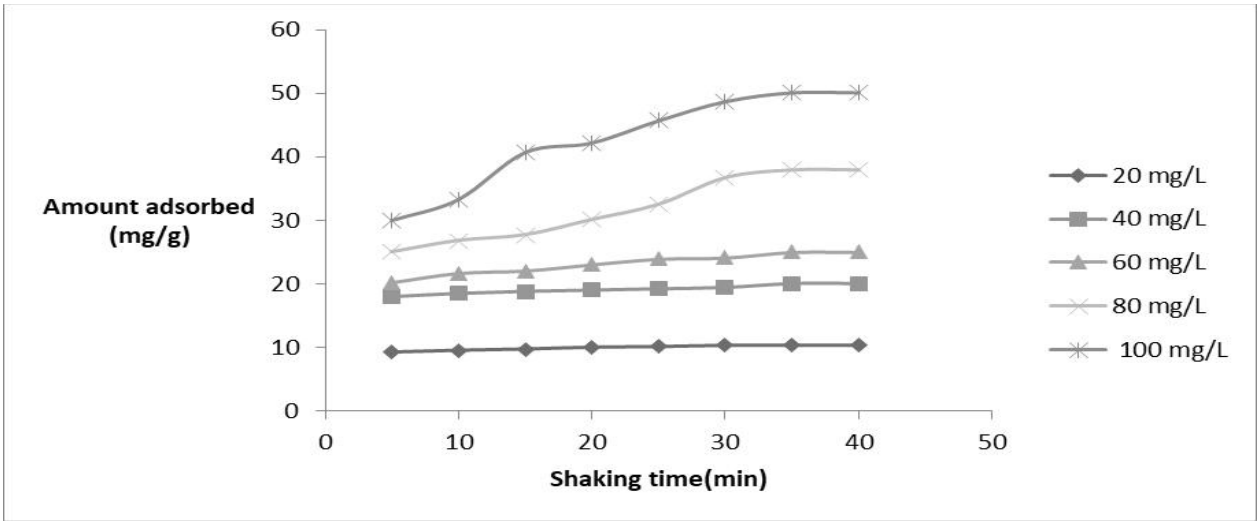

Figure 1. Effect of shaking time and initial Pb(II) concentration on the adsorption of Pb(II).

Figure 1 shows the effect of shaking time and initial Pb(II) ion concentration on adsorption from solution when using Unwana clay. Results show that the equilibrium is reached quickly (only 30 min), indicating that the adsorption sites are well exposed. Orumwense (1996) , using a kaolinitic clay for the removal of lead from water by adsorption, has found that the equilibrium time needed for Pb(II) was longer than is shown by the figure 1. However, the results from Esmaeili & Eslami (2020) were similar to those found in this investigation. These latter authors conducted experiments on adsorption of lead and zinc ions from aqueous solutions by volcanic ash soil. The difference in equilibrium time may be due to the difference in the experimental conditions, the adsorbent material, etc. As can be observed from the graph, an increasing concentration of the lead ions in solution resulted in an increase of the equilibrium time. It is clear from the results that the shaking time required for maximum uptake of metal ions by Unwana clay was dependent on the initial Pb(II) concentration. Consequently, the shaking time was fixed at 30 min for the rest of the batch experiments to ensure that adsorption equilibrium was reached in each case.

The Effect of Shaking Time and Initial Cu(II) Concentration

Experimental conditions: Contact time 1-35 min, Initial Cu(II) concentrations 20-100 mg/L, Mass of Unwana clay 0.1 g, pH 7

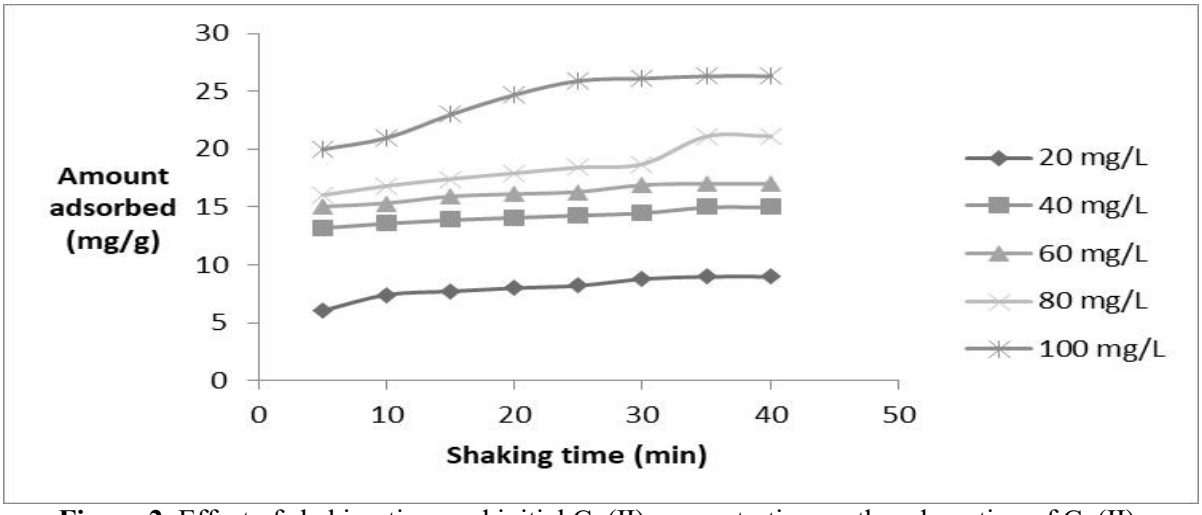

Figure 2. Effect of shaking time and initial Cu(II) concentration on the adsorption of Cu(II).

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The results of the effect of shaking time and initial Cu(II) ion concentration on adsorption from solution onto Unwana clay are shown in figure 2. The plots reveal that the amount of metal adsorbed per unit weight of adsorbent increases with time and reach equilibrium after 20 min of shaking time for the different initial metal concentrations. There does not seem to be much benefit from shaking longer than 20 min. Therefore, an equilibrium time of 20 min was selected for all further studies. The equilibrium time was less than that for the Cu(II) ions adsorbed by phenanthroline-grafted Brazilian bentonite, as found by De León et al (2003).

The Effect of the Amount of Unwana clay on Pb(ii) Adsorption

Experimental conditions: Contact time 30 min, Initial Pb(II) concentrations 20-100 mg/L, Mass of Unwana clay 0.1-1 g, pH 7

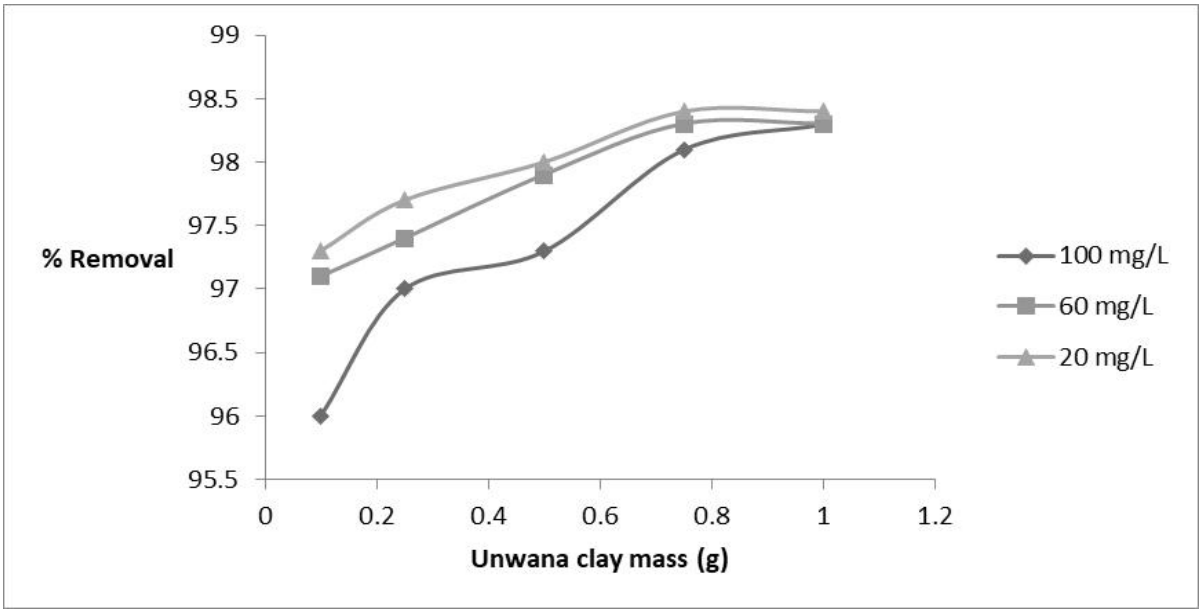

Figure 3. Effect of the mass of Unwana clay on the sequestering of Pb(II) onto Unwana clay

The results of the dependence of Pb(ii) adsorption on amount of Unwana clay used are shown in figure 3. Increasing the mass of Unwana clay slightly increased the percentage removal of Pb (II). This is an expected result because as the amount of adsorbent increases, the number of adsorbent surrounded by metal ions increases, therefore, these species can accommodate more ions onto their surfaces. In short, there are more adsorption sites available because there is more material.

Naseem & Tahir (2001) reported similar findings for Pb(II) removal from aqueous/acidic solutions by using bentonite as an adsorbent. They found that the percentage adsorption of Pb(II) increases with an increase of the amount of bentonite and remains constant above 0.5 g. In the case of lead for our investigation, a minimum of 0.75 g of sorbent is required for complete adsorption of all the solution concentrations up to 100 mg/L tested.

The Effect of the Amount of Unwana Clay on Cu(II) Adsorption

Experimental conditions: Contact time 20 min, Initial Cu(II) concentrations 20-100 mg/L, Mass of Unwana clay 0.1-1 g, pH 7

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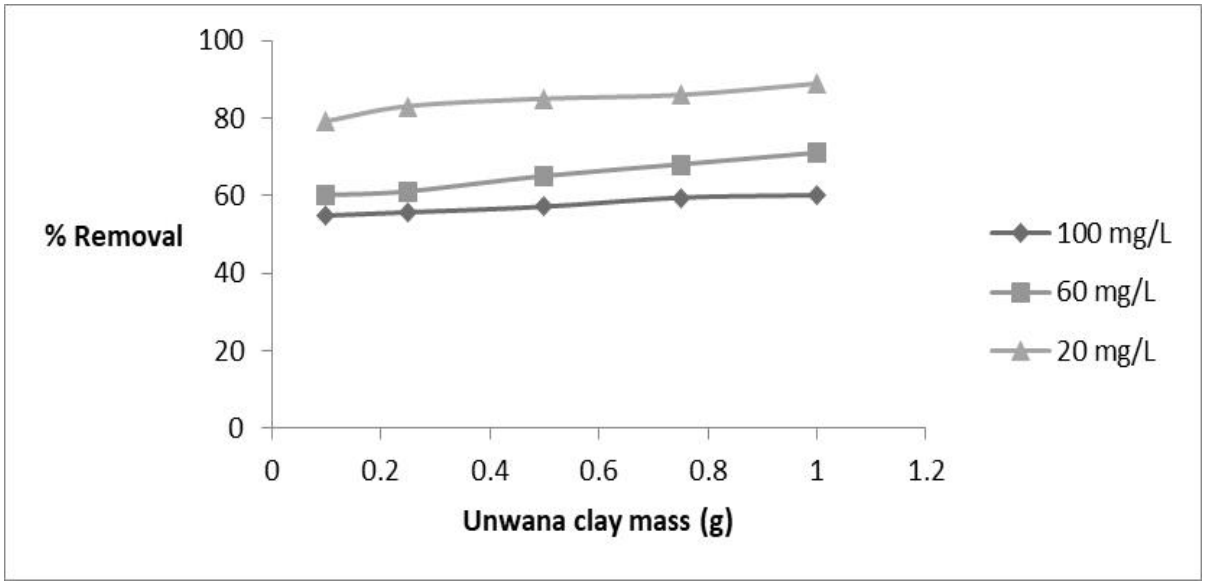

Figure 4. Effect of the mass of Unwana clay on the adsorption of Cu(II) onto Unwana clay

The results of the dependence of Cu(II) adsorption on the amount of Unwana clay are shown in figure 4. It is apparent that the percentage removal of copper increases slightly with increases in the mass of Unwana clay. Similar results have been found with the adsorption of copper(II) ions on other adsorbents (Aydın et al., 2008). Their results revealed that the percentage removal of copper increases with increases in the mass of adsorbent. The copper adsorption onto Unwana clay displays a completely different trend with increasing mass of adsorbent than the rest of the metal ions evaluated. There is either not enough adsorbent, even at 1 g, to completely remove all the Cu(II) ions from the higher concentration solutions ($\geq$ 60 mg L^{-1}), or the adsorption capacity is poorer for Cu(II) ions than the rest of other metal ions. Alternatively there is a different equilibrium for Cu(II) compared to the rest, or the time to reach equilibrium was too short (i.e. the kinetics of the process is too slow).

The Effect of Ph on the Adsorption of Pb(II*)*

Experimental conditions: Contact time 30 min, Initial Pb(II) concentrations 20-100 mg/L, Mass of Unwana clay 0.1 g, pH 3-10.

Table 3. Influence of pH on the adsorption of Pb(II) onto Unwana clay

pH	20 mg/L % Removal	40 mg/L % Removal	60 mg/L % Removal	80 mg/L % Removal	100 mg/L % Removal
3	79	59.4	36	25.7	27
5	82	71.1	53	43	52
8	86	88	87	86	74
10	81	86	81	79	61

The pH of the aqueous solution is an important variable which controls the adsorption of the metal at the clay-water interfaces. Hence, the influence of pH on the adsorption of Pb(II) onto Unwana clay was investigated in the pH range of 3-10. The results are presented in Table 3

It can be observed from the results that the adsorption of Pb(II) increases with an increase in pH of the solution to a maximum around a neutral pH or slightly basic pH, and then decreases as the pH becomes more basic. This is true regardless of the concentration of the solution investigated.

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Clays are known to possess a negative surface charge in solution. As pH changes, surface charge also changes, and the sorption of charged species are affected (attraction between the positively charged metal ion and the negatively charged clay surface). It is conceivable that at low pH values, where there is an excess of H_3O^+ ions in solution, a competition exists between the positively charged hydrogen ions and metal ions for the available adsorption sites on the negatively charged clay surface. As the pH increases and the balance between H_3O^+ and OH^- are more equal, more of the positively charged metal ions in solution are adsorbed onto the negative clay surface and thus the percentage removal of the metal ions increases. This is in agreement with work by other authors (Ijagbemi et al., 2009) on clay studies

The Effect of pH on the Adsorption of Cu(II)

Experimental conditions: Contact time 20 min, Initial Cu(II) concentrations 20-100 mg/L, Mass of Unwana clay 0.1 g, pH 3-10.

Table 4. Influence of pH on the adsorption of Cu(II) onto Unwana clay

pH	20 mg/L % Removal	40 mg/L % Removal	60 mg/L % Removal	80 mg/L % Removal	100 mg/L % Removal
3	74.0	73.0	52.0	54	49.0
5	83.0	79.1	63.0	57	52.0
8	87.0	88.0	57.0	61	57.0
10	85.0	86.0	53.0	59	54.0

The effect of pH on copper sorption on unwana clay was studied at room temperature (25°C) by varying the pH of copper solution-Unwana clay suspension from 3 to 10. The results are shown in Table 4.

It can be observed that the removal of copper(II) by Unwana clay increases with increasing pH, from its minimum value at a pH 3.0 to its maximum value at a pH of about 8. After that the percentage adsorption decreases slightly in pH range of 8-10. This is similar to the trend that
was observed in the case of Pb(II). Yu et al (2001) found similar results for the removal of copper from aqueous solutions using sawdust. Their results indicated that the removal of copper (II) by sawdust adsorption increases with increasing pH from its minimum value at a pH 2.0 to its maximum value at a pH of about 7.0. After that the percentage adsorption decreases slightly in pH range of 8.0-10.0

Adherence to Adsorption Isotherms

To examine the relationship between sorbed (q_e) and aqueous concentrations (C_e) at equilibrium, sorption isotherm models are widely employed for fitting the data. The Langmuir equations are the most widely used. Langmuir have been tested to fit the equilibrium data.

	Q_o (mg/g)	b (l/mg)	R^2
Pb	61.3	0.71	0.962
Cu	26.8	0.14	0.9151

The Langmuir isotherm provided a good correlation for Cu(II) adsorption onto the Unwana clay. This is in accordance with work by Veit et al (2005) on Cu(II) adsorption on dead fungal biomasses (*Pleurotus pulmonarius* and *Schizophyllum commune*). These authors found that the best adjusted model to the experimental equilibrium data for both biomasses was the Langmuir model.

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