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# ADSORPTION STUDIES OF CADMIUM ION FROM WATER USING BIOCHAR PRODUCED FROM GOAT HORN.

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## ABSTARCT

*Biochar produced from goat horn was used to investigate the adsorption of cadmium ($Cd^{2+}$) ion. The proximate analysis of the goat horn biochar was carried out, the moisture content, ash content and bulk density were 10.62%, 6.5% and 0.57 respectively. FTIR characterization shows functional group of O-H, C=H, C-O, C=C and P-O. SEM analysis shows some distinct porosity, irregular surfaces with roughness. Adsorption batch experiment was carried out at room temperature, the effect of pH, $Cd^{2+}$ ion concentration and contact time were investigated. The optimal adsorption was achieved at pH 7, 20mg/L, 60min, and the maximum adsorption capacity was 38.84mg/g. The isotherm data fit best for both Temkins and Langmuir which indicated a uniform distribution of bonding energy between the $Cd^{2+}$ ion and biochar and also a monolayer adsorption process. The kinetic study best fits pseudo-second-order reaction, suggesting that chemisorptions dominated the adsorption process.*

***Keywords**: Toxic metal, adsorption, goat horn biochar, cadmium ($Cd^{2+}$) ion, isotherm, kinetic.*

## INTRODUCTION

Pollution of water bodies has been popular issue since the existence of man. The alarming increase in pollution of water bodies has made portable water a scares resource most especially in under-developed and developing countries [1]. Man-made activities from industrial, agricultural and commercial sectors have released a broad range of pollutants such as biopollutants, nutrients, organic matter, dyes, and toxic metals mostly [2]. Yahya et al [3], reported that about 70% of untreated effluents and 90% of raw sewage are discharged into

surface water in developing and under developed countries. Biological pollutants, nutrients, organic matter and dyes are pollutants which can be degraded through biological processes and photo-degradation. Heavy metals on the other hand are non-biodegradable and thus persist in the environment making them more dangerous in water bodies.

Toxic metals are naturally occurring elements that have a higher atomic weight and density which is five times greater than that of water. They can easily be taken up by plants, animals and humans through food chain. Toxic metals cause serious health effects, including reduced growth and development, cancer, organ damage, nervous system damage, and death in extreme cases [4]. Cadmium ($Cd^{2+}$) is one of the most harmful toxic metals. Exposure to ($Cd^{2+}$) leads to symptoms like diarrhea, cadmium blues (chills, fever and muscles ache) which may later results to respiratory damage. $Cd^{2+}$ permissible limit is 0.005mg/L [5], with concentrations above this limit resulting to serious health challenges such as bone degeneration, liver damage, renal dysfunction and reproductive failure [6]. These severe health effects have called for urgent remediation of $Cd^{2+}$ in water.

Different conventional techniques have been implored for the removal of heavy metals such as chemical precipitation, ion exchange, reverse osmosis, electro-dialysis, filtration, coagulation, flocculation and floatation. They all show a good removal for toxic metal but have cost implications, poor efficiency mostly at low metal concentration and sludge disposal problems [7]. Adsorption is a better alternative due to it's simple design with a sludge free environment, ease of operation, reasonable cost and best efficiency even at low metal concentration [8-9]. Commercially activated carbon has been widely used as adsorbent for removal of toxic metals but it's cost, unclear mode of preparation and limited regeneration ability has called for, an alternatively inexpensive, readily available, sustainable and easy to prepare substituent such as the low-cost adsorbent from agricultural wastes [10]. Biochar is a type of low-cost adsorbent composed of carbon-rich solid material generated by the thermo-chemical processing (pyrolysis and gasification) of carbonaceous waste under an oxygen-deficient condition [11]. Its abundant surface area, porosity, and high affinity of its functional group make it effective for toxic metal removal. The abundance of goat horn waste being released

daily from abattoir in Zaria metropolis with no economic importance can be converted to biochar which also could mitigate the challenges of waste disposal. The study aim to remove cadmium ion in water using biochar produced from goat horn as a low cost adsorbent.

## MATERIALS AND METHOD

### Reagent and chemicals

The list of chemicals/reagents used for this work are; Cadmium nitrate tetrahydrate salt ($Cd(NO_3)_2.4H_20$) (BDH laboratory), Phosphoric acid ($H_3PO_4$) (Sigma Aldrich), Hydrochloric acid (HCl) (Sigma Aldrich), Sodium hydroxide (NaOH) (Sigma Aldrich), Deionized water and Distilled water. All reagents/chemicals were analytical grade and used without further purification.

**Sample collection, preparation and pretreatment:** The goat horns were collected at the goat market at Gwangila, Zaria, Kaduna state. The goat horn was thoroughly washed and sun-dried for a month. The dried horns were ground into particle size ranges from 1.40mm to 2.80mm (12 to 7 mesh size) using mortar and pestle.

**Carbonization and chemical activation of the biochar:** The crushed goat horns (200g) was placed in large crucibles and the set up was kept in a muffle furnace at temperature of 400°C for 3 hrs in the absence of air, after which they were removed and kept in a desiccator to allow to cool[12]. The biochar was grinded to a size of 355 μm (44 BSS mesh size), 0.5 M HCl was used to wash and purify the carbon, then rinsed several times with distilled water and oven dried before activation. The biochar was chemically activated using 1M $H_3PO_4$. The biochar was impregnated with $H_3PO_4$ at a ratio of 1:2 with the aid of a stirrer, placed into the muffle furnace at a temperature of 500°C for 1 hr then taken into the dessicator to cool [13]. The activated biochar was rinsed several times with distilled water until a pH of 7 is maintained. The biochar is then dried in an oven at 110°C for 2 hrs, cooled and then stored.

### Preparation of metal ion concentration:

The stock solution was prepared by measuring 0.2744g of $Cd(NO_3)_2.4H_2$ into a $1dm^3$ volumetric flask and filled with deionized water to the mark, to give a 100ppm concentration. Serial dilution was used to prepare 20, 40, and 60 ppm for batch adsorption experiment.

### Proximate analysis

The moisture content (M.C), ash content (A.C) and bulk density (B.D) were determined as described by Aller et al [14].

$$MC\ (\%) = \frac{(M_{BD} - M_{AD})}{M_{BD}} \times 100 \quad (1)$$

Where; $M_{BD}$= mass of sample (g) before drying, $M_{AD}$= mass of sample (g) after drying

$$\text{A. C}\ (\%) = \frac{\text{Ash Weight (g)}}{\text{Oven Dry Wt.(g) of sample}} \times 100 \quad (2)$$

Where; Ash weight= weight after taken from furnace, Oven dry weight= weight of the sample before taken into the furnace.

$$B.D\ (\frac{g}{cm^3}) = \frac{M_{CS} - M_C}{V} \quad (3)$$

Where; $M_{CS}$ = mass of cylinder and sample, $M_C$ = mass of empty cylinder, V = volume of cylinder

**Characterization of the goat horn biochar:**

The Fourier Transform Infrared spectrophotometer (Cary 630 FTIR, Agilent Technology, USA) with wavenumber range between 400 and 4000 $cm^{-1}$ was used to give information about the characteristic functional groups on the surface of the goat horns biochar. The surface morphology of the biochar was visualized via a Scanning Electron Microscope (SEM Phenom ProX, England) coupled with an energy- dispersive X-ray spectrometer operating at 15 kV.

**Batch adsorption studies**

The equilibrium sorption was carried out at room temperature using the following factors and levels.

**Table 1**: Factors and levels

| S/N | FACTORS | LEVELS |
|---|---|---|
| 1 | Concentration ($mg/dm^3$) | 20, 40 and 60 |
| 2 | pH | 5, 6 and 7. |
| 3 | Contact Time (mins) | 5, 15 and 30 |
| 4 | Adsorbent dose ($g/dm^3$) | 2.0 |

To a series of 100ml conical flask, $50cm^3$ of cadmium ion solution was transferred. The initial pH values of each solution were adjusted by adding 0.5M HCl and 0.5M NaOH solution. Afterward, 0.1 g of goat horn biochar is then added into each conical flask. The resulting mixture in the conical flasks were corked and agitated at various time intervals with a mechanical shaker at

300 rpm as given in the experimental design matrix in Table 4.

The mixture was filtered and concentration of the residual ions in the solution was determined using Atomic absorption spectrophotometer (Varian AAS240, Agilent Technology, USA).The amount of metal ions adsorbed (Qe) and percentage removal (%R) from the solution was determined using Equations 4 and 5 respectively [15].

$$\text{Qe} = \frac{(C_o - C_e)V}{m} \quad (4)$$

$$\%\text{R} = \frac{(C_o - C_e)}{C_e} * 100 \quad (5)$$

Where; Qe (mg/g) = amount of metal ions adsorbed, Co (mg/l) = initial metal concentration in solution, Ce (mg/l) = final metal concentration in the solution, V (L) = volume of the metal solution used in litre and m (g) = mass of the biosorbent.

**Adsorption isotherm**

Adsorption equilibrium isotherm is a mathematical model that is used to describe the dependence of the adsorbed amount ($Q_e$) as a function of the adsorbate concentration (Ce) with respect to temperature (T).

$$\text{Qe} = \text{f (Ce, T)} \quad (6)$$

3.6.1 Langmuir isotherm

It describes monolayer adsorption and assumes homogenous sorption sites and sorption energies on the surface of adsorbents [16].

$$\text{Qe} = Qmax\frac{K_L Ce}{1+K_L Ce} \quad (7)$$

Where; Qe(mg/g) = Adsorption capacity at equilibrium, Qmax(mg/g) = Theoretical maximum adsorption capacity of the adsorbent, Ce (mg/l) = Equilibrium concentration of the system, $K_L$ (l/mg) = Langmuir affinity constant.

The favourability of the adsorption process or isotherm is dependent on the dimensionless constant $R_L$, which is calculated using the mathematical form in Equation 8.

$$R_L = \frac{1}{1+K_L Co} \quad (8)$$

Where; Co = Initial metal concentration.

3.6.2 Freundlich Isotherm

In Freundlich isotherm equations, the relationship between metal uptake and equilibrium concentration is logarithmical and it is assumed that the uptake capacity of adsorbent is infinite.

$$\text{Qe} = \text{KfCe}^{1/n} \quad (9)$$

Where; Kf (mg/g) = Freundlich constant related with adsorption capacity, n = heterogeneity coefficient (dimensionless)

3.6.3 Temkins Isotherm

The assumption of Temkins isotherm was based on the linear decrease in heat of adsorption with increase surface area and that uniform distribution of binding energies up to maximum energy [17];

$$Qe = B\ln(ACe) \quad (10)$$

$$B = \frac{RT}{b_T} \quad (11)$$

Where; R (8.314J/mol/K) = Universal gas constant, T (298K) = Absolute temperature, $b_T$ (J/mol) = Temkin isotherm constant related to heat of adsorption, $A_T$ (L/mg) = Temkin isotherm eqiuilibrium binding constant

**Adsorption kinetics**

Pseudo-first-order and pseudo-second-order kinetics is to provide details about the dynamics of the adsorption process of the $Cd^{2+}$ ion on the goat horn biochar.

**Pseudo First Order Kinetics:** The Lagergren pseudo-first-order kinetic model was expressed as adopted from Gupta and Singh (28).

$$\frac{dQt}{dt} = K_1 ad(Qe - Qt) \quad (12)$$

Integrating and re-arranging Eqn 12 gives a non-linearized form in Eqn 13.

$$Qt = Qe(1 - e^{-k_1 t}) \quad (13)$$

Where; $K_1$ ($min^{-1}$) = rate constant of the pseudo-first-order adsorption, Qt (mg/g) = amount of adsorption at time t (min), Qe (mg/g) = amount of adsorption at equilibrium, t (min) = time

**Pseudo-Second-Order Kinetics:** The equation for Pseudo-second-order equation is expressed as follow;

$$\frac{dQt}{dt} = K_2 ad(Qe - Qt)^2 \quad (14)$$

Integrating and rearranging Eqn 14 gives a non-linear form in Eqn 15 [18].

$$Qt = \frac{Qe^2 k_2 t}{1 + Qe k_2 t} \quad (15)$$

Where; $k_2$ = rate constant of second-order adsorption (g $mg^{-1}$ $min^{-1}$), Qt (mg/g) = amount of adsorption at time t (min), Qe (mg/g) = amount of adsorption at equilibrium, t (min) = time

**RESULTS AND DISCUSSION**

**Proximate analysis of the biochar:** Table 2, shows the moisture content, ash content and bulk density of biochar. The moisture content of the biochar was 10.62%, indicating a good adsorbent for heavy metal removal [19]. Ash content was low at 6.5%, indicating a small amount of inorganic matter content on the biochar. Higher levels

of ash contents reduce the overall activity of biochar and reduce the efficiency of its reactivation [20]. The bulk density for goat horn biochar was 0.79 g/cm$^3$, values greater than 0.25 g/cm$^3$, indicates a suitable commercial adsorbent as reported by Denver [21].

**Characterization of the goat horn biochar:** The FTIR results shown in Figure 1 and Table 3. It can be observed that peak at 3201cm$^{-1}$ represents O-H stretching vibration of alcohol, phenol, or carboxylic acid. At 2959cm$^{-1}$ band, a peak which corresponds to C-H stretching vibration in methyl group of an alkane group, band at 2113 cm$^{-1}$ corresponds to the C≡C stretching, peak at 1640 cm$^{-1}$ represents both C=O and C=C stretching, C-O group was found at 1438 cm$^{-1}$ band, peaks ranging from 1066 cm$^{-1}$ to 931 cm$^{-1}$stretching correspond to the P-O bonding of the phosphoric acid with $PO_2$ and $PO_3$, which was similar to the report of Liu and Fan [22].

**Table 2:** Proximate analysis of goat horn biochar.

| Moisture Content (%) | Ash Content (%) | Bulk Density (g/cm$^3$) |
|---|---|---|
| 10.62 ± 0.12 | 6.5 ± 0.20 | 0.57 ± 0.00 |

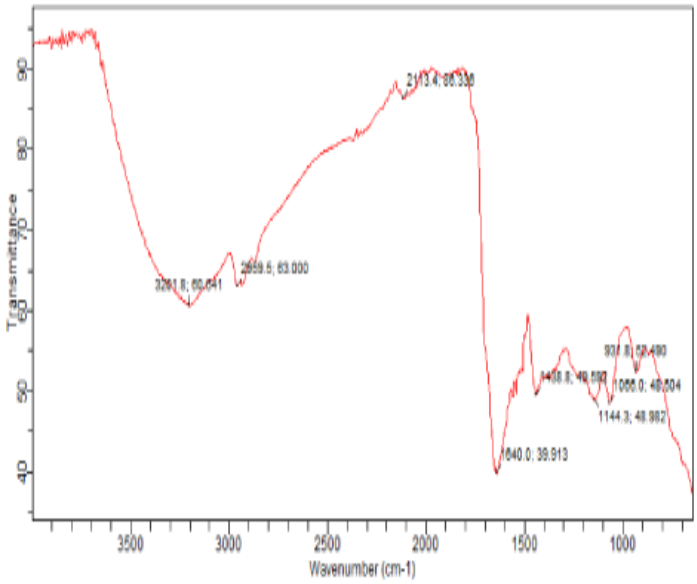


**Figure 1:** FTIR of goat horn biochar.

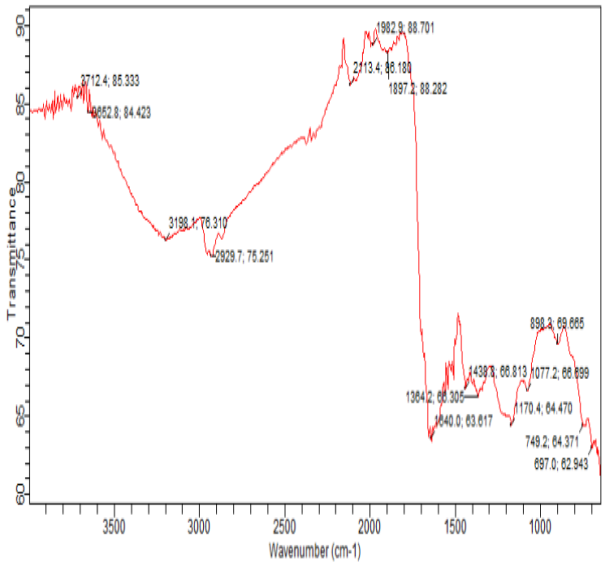


**Figure 2:** FTIR of adsorbed Cd ion on biocha

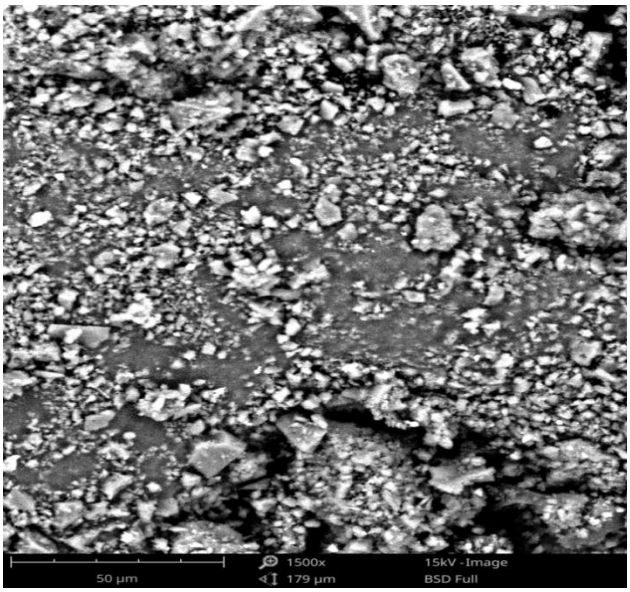


**Figure 3:** SEM image of the goat horn goat horn biochar

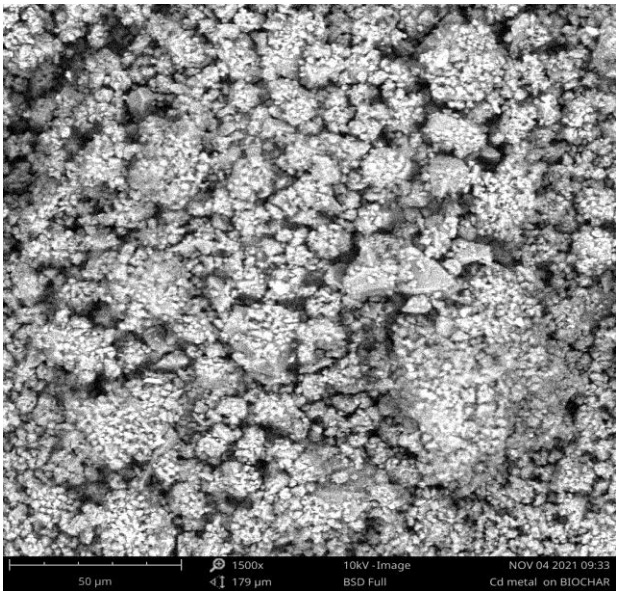


**Figure 4:** SEM image of Cd ion adsorbed on biochar.

The FTIR spectrum for Cd ion adsorbed on biochar as shown in Figure 2 and Table 3, shows changes in peaks with respect to the goat horn biochar in Figure 1 in O-H, C-H, C-O and P-O bonds from 3201cm$^{-1}$ to 3198cm$^{-1}$, 2959cm$^{-1}$ to 2929 cm$^{-1}$, 1438 cm$^{-1}$ to 1170cm$^{-1}$ and 931 & 1066 cm$^{-1}$ to 898 – 1077 cm$^{-1}$ respectively. Some peaks have shrunk while some have broadened, which was a results of the potential interaction between the functional group on biochar with cadmium ion.

**TABLE 3:** FTIR results of biochar and Cd adsorbed on biochar

| | O-H | C-H | C-O | P-O |
|---|---|---|---|---|
| Biochar (cm$^{-1}$) | 3291 | 2959 | 1144 | 931 & 1055 |
| Cd-Biochar (cm$^{-1}$) | 3198 | 2929 | 1170 | 898 & 1077 |

The SEM image of the goat horn biochar was shown in Figure 3. It was observed that biochar possesses some distinct porosity, irregular surfaces with roughness. The SEM image of the $Cd^{2+}$ion on biochar shows a impregnation of Cd ion on the surface of the biochar as shown in Figure 4. Comparing the biochar before and after adsorption showed that the size and surface of the biochar gradually changed due to particle

aggregation resulting from Cd adsorption on the biochar.

**Batch Adsorption Experiment.**

The adsorption experimental runs showing responses with factors and levels is shown in Table 4.

**Table 4:** Adsorption experimental design matrix and responses

| Std | Run | Factor 1 A:pH | Factor 2 B:Cocentration mg/L | Factor 3 C:Contact time mins | Response 1 $Cd^{2+}$ Removal % |
|---|---|---|---|---|---|
| 17 | 1 | 6 | 60 | 15 | 44.21 |
| 4 | 2 | 5 | 40 | 5 | 44.69 |
| 25 | 3 | 5 | 60 | 30 | 46.02 |
| 19 | 4 | 5 | 20 | 30 | 54.58 |
| 12 | 5 | 7 | 20 | 15 | 96.07 |
| 23 | 6 | 6 | 40 | 30 | 57.65 |
| 13 | 7 | 5 | 40 | 15 | 55.18 |
| 9 | 8 | 7 | 60 | 5 | 79.92 |
| 3 | 9 | 7 | 20 | 5 | 94.36 |
| 18 | 10 | 7 | 60 | 15 | 89.64 |
| 5 | 11 | 6 | 40 | 5 | 61.60 |
| 14 | 12 | 6 | 40 | 15 | 62.30 |
| 7 | 13 | 5 | 60 | 5 | 52.85 |
| 21 | 14 | 7 | 20 | 30 | 96.64 |
| 6 | 15 | 7 | 40 | 5 | 90.84 |
| 10 | 16 | 5 | 20 | 15 | 61.19 |
| 8 | 17 | 6 | 60 | 5 | 61.78 |
| 16 | 18 | 5 | 60 | 15 | 51.84 |
| 22 | 19 | 5 | 40 | 30 | 47.36 |
| 20 | 20 | 6 | 20 | 30 | 69.50 |
| 2 | 21 | 6 | 20 | 5 | 73.82 |
| 15 | 22 | 7 | 40 | 15 | 93.98 |
| 11 | 23 | 6 | 20 | 15 | 71.35 |
| 26 | 24 | 6 | 60 | 30 | 48.92 |
| 27 | 25 | 7 | 60 | 30 | 88.94 |
| 1 | 26 | 5 | 20 | 5 | 59.01 |
| 24 | 27 | 7 | 40 | 30 | 92.42 |

**Effects of pH on percentage removal:** Figure 5 shows the effect of pH on Cd removal. It was observed that at 20mg/L and 30mins, increase in % removal was observed as pH increases from 5 to 7. The %removal of $Cd^{2+}$ on the biochar at pH 5 was 55% while at pH 6 was 70%. This was as a result of the strong competition between $Cd^{2+}$ ion and hydrogen ion ($H^{+}$) for binding sites on the biochar, and also the protonation of the goat horn biochar. Further increase in pH to 7 gave the maximum % removal of 97%, which was a results of the negatively charge sites on the biochar and the de-protonation of $H^{+}$ leading to the removal of $Cd^{2+}$on the biochar [23].

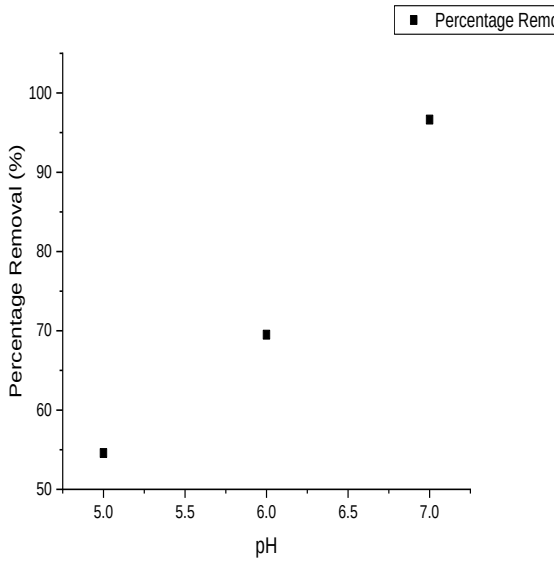


**Figure 5:** Effect of pH on percentage removal of cadmium ion on biochar

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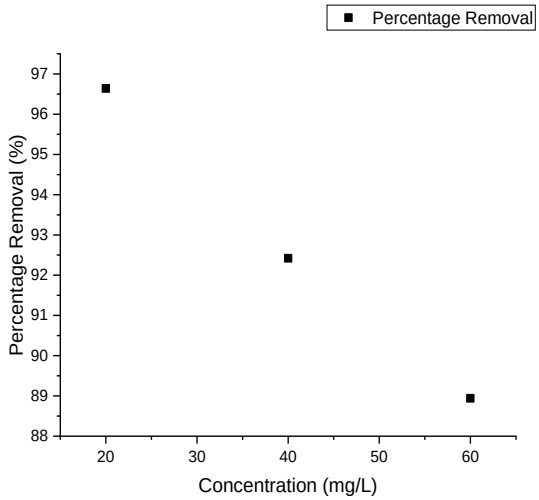


**Figure 6:** Effect of concentration on percentage removal of cadmium ion on biochar

**Effect of initial concentration of percentage removal**: The effect of metal concentration on Cd removal was shown in Figure 6. It was observed at pH 7 and at 30mins, an increase in concentration from 20 to 40mg/L lead to a decrease in % removal from 97 to 92% and further increase in concentration from 40 to 60mg/L resulted to a continuous decrease from 92 to 89% for Cd on biochar. A plausible reason was that at lower metal concentration, the ratio of available binding sites was larger while at higher metal concentration sites are limited leading to a faster saturation of the vacant sites. This was in agreement with the work of Dawood *et al* [24].

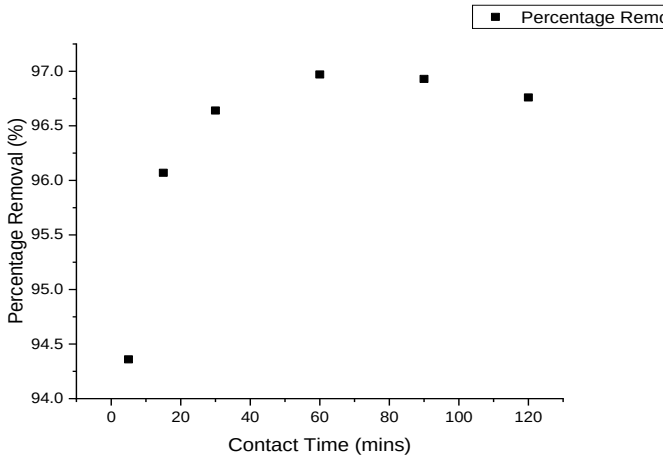


**Figure 7:** Effect of contact time on percentage removal of cadmium ion on biochar

**Effect of contact time on percentage removal:** The effect of contact time was carried out at pH 7 and at 20mg/L because of their optimal % removal as shown in

Figure 7. It was observed that increase in contact time from 5 to 60mins increased the % removal of cadmium ion on the biochar from 94.36 to 96.97%, This was as a result of the abundance of accessible vacant pore sites available on the biochar surface at the beginning of the adsorption process. Further increase in contact time from 90 to 120 mins resulted in a gradual decrease in % removal from 96.97 to 96.76 %. This might be as a result of saturation of the vacant poresites of the biochar making them been occupied. This effect of contact time on percentage removal was in agreement with the report of Kumar *et al* [25].

## Adsorption Isotherm

The Langmuir isotherm model for $Cd^{2+}$ ion gave $R^2$-values of 0.9432 as showed in Figure 8 and Table 5. The high $R^2$-value shows that the Langmuir isotherm fits with the adsorption isotherm, suggesting a monolayer adsorption process [29]. The estimated maximum adsorption capacity Qmax for adsorption of $Cd^{2+}$ was 38.844 mg/g. Langmuir constant ($K_L$) from the graph was 0.4670. The Langmuir dimensionless constant ($R_L$) for the Langmuir model was 0.0967. The $R_L$ value indicates the adsorption process was favorable been greater than 0 but less than 1.

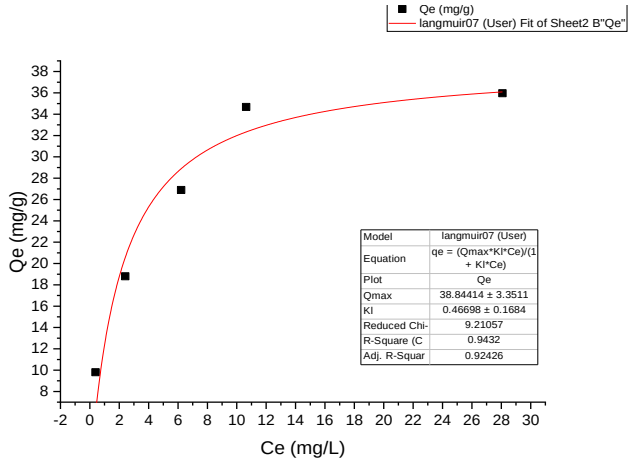


**Figure 8**: Langmuir adsorption isotherm for cadmium onto goat horn biochar

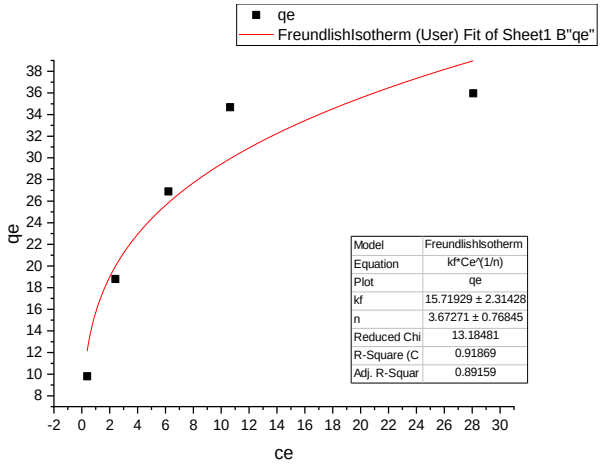


**Figure 9**: Freundlich adsorption isotherm for cadmium onto goat horn biochar

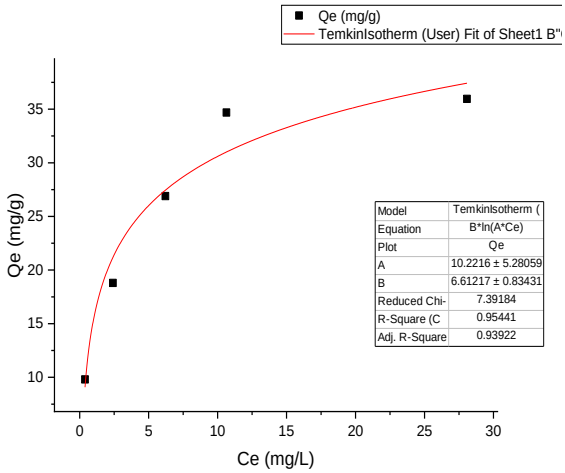


**Figure 10**: Temkin's adsorption isotherm for cadmium onto goat horn biochar

The Freundlich isotherm for $Cd^{2+}$ ion on goat horn biochar as shown in Figure 9, gave $R^2$-value of 0.9189. The Freundlich constant ($K_F$) is 15.7193, indicating a higher adsorption capacity or adsorption affinity of the goat horn biochar on $Cd^{2+}$. The *n* value is 3.6727, indicating a favourable and feasible adsorption process as *n* value ranges between 1-10.

The Temkins isotherm for $Cd^{2+}$ion on goat horn biochar gave $R^2$-values of 0.9544 as shown in Figure 10 and Table 5. The $A_T$ values was 10.222, indicates a high binding energy between Cd ion the goat horn biochar. The $b_T$ values for $Cd^{2+}$ion is 373.887, suggesting a very low heat of adsorption. This was similar with report by Abia *et al* [26]. The best fitted isotherm for the adsorption of Cd ion on goat horn biochar was Temkins isotherm having the highest $R^2$ value of 0.9544.

**Table 5:** Adsorption isotherm parameters.

| Langmuir isotherm | Qmax (mg/L) | $K_L$ (L/mg) | $R^2$ | $R_L$ |
|---|---|---|---|---|
| | 38.844 | 0.4670 | 0.9432 | 0.0967 |
| **Freundlich isotherm** | $K_F$ | *n* | $R^2$ | |
| | 15.7193 | 3.6727 | 0.9189 | |
| **Temkins isotherm** | $A_T$ (L/g) | b (J/mol) | $R^2$ | |
| | 10.222 | 373.887 | 0.9544 | |

**Adsorption Kinetic**

Kinetic studies were determined using the non-linear regression for Pseudo-first-order and second-order kinetic. Pseudo-first-order kinetic gave $R^2$-values of 0.8948, the qe of 9.6675 and $K_1$ is 0.7463 as shown in Figure 11 while Pseudo-second-order kinetics gave $R^2$-values of 0.9878, qe of 9.7050 and $K_2$ is 0.7211 as shown in Figure 12 and Table 6. The higher $R^2$-values and the closeness of the qe with the $qe_{cal}$ (9.697) suggest that pseudo-second-order kinetic fit best for the

adsorption kinetic. This was in agreement with report of Mustapha *et al* [27]. The kinetic is favoured by chemisorptions where sharing through valence forces or the exchange of electrons take place between the goat horn biochar and the cadmium ion.

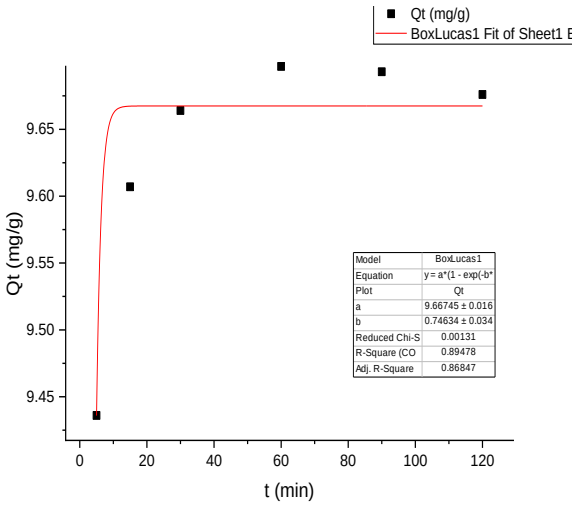


**Figure 11:** Pseudo First –Order adsorption kinetics of Cd ion on biochar.

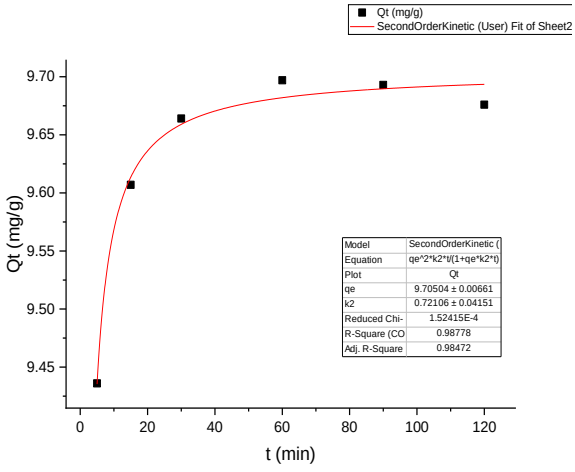


**Figure 12:** Pseudo second –Order adsorption kinetic for Cd ion on biochar.

**Table 6: Pseudo first order and pseudo second order kinetics.**

| Parameter | Pseudo-first-order | Pseudo-second-order |
|---|---|---|
| $qe_{cal}$(mg/g) | 9.697 | 9.697 |
| qe(mg/g) | 9.6675 | 9.7050 |
| $K_{ad}$ | 0.7463 | 0.7211 |
| $R^2$ | 0.8948 | 0.9878 |

## CONCLUSION

In this study, the goat horn biochar produced was used for the adsorption of cadmium ion in water. It was concluded that pH7, 20mg/L gave the optimal removal capacity as deduced from Table 4. Adsorption equilibrium time was at 90 mins and adsorption capacity for cadmium was 33.84mg/L. The isotherm studies data was best fitted for Temkins isotherm, which indicates a uniform distribution of bonding energy between the $Cd^{2+}$ ion and biochar. The kinetics data reveal that pseudo-second-order fit best for the study, showing a chemical interaction between the $Cd^{2+}$ and biochar. Goat horn biochar as a low-cost adsorbent was effective for the removal of cadmium ion in water.

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