



## Adsorption of Cd and Cr from Industrial Wastewater using Kaolin

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### ABSTRACT

This work was undertaken to develop a cost-effective biosorbent and to study the biosorption process involved in the adsorption of Cd and Cr ions from simulated industrial wastewater using the prepared kaolin. The prepared kaolin was characterised by its functional groups using Fourier Transform Infra-Red Spectroscopy (FTIRs). The efficiency of the adsorbent was evaluated by measuring the extent of adsorption of Cd and Cr (II) ions in the simulated industrial wastewater. Operational parameters such as contact time, stirring speed and adsorbent dosage were studied. The FT-IR spectrum of the kaolin shows peaks at 3447, 1634, 1469, 1097/462 and 576  $\text{cm}^{-1}$  attributed to the O–H stretching, H–O–H bending, Si–O–Si groups of the tetrahedral sheet, Al – O bending and stretching vibration of Si – O respectively. Adsorption data fitted well with the Freundlich and Jovanovic models. However, the Jovanovic isotherm displayed a better fitting model than Freundlich isotherm because of the higher correlation coefficient and lower standard error that the former exhibited, thus, indicating that the adsorptions of Cd and Cr metal ions in this work can be said to have taken place more on homogenous site with monolayer coverage of metal ions on the outer surfaces of the adsorbents along with some mechanical contacts between the metal ions and the adsorbent with competition for binding sites among the metal ions. Using kaolin as an adsorbent implies the availability of abundant and less expensive natural materials for treating wastewater.

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### INTRODUCTION

Minerals and mineral resources are used daily by every area of society. Kaolin is one of these minerals and is utilized in cosmetics, construction products, adsorption and filtering materials. Kaolin is a naturally occurring hydrated aluminum silicate that is processed for use in pharmaceuticals by washing with water to get rid of grit and other contaminants. The primary component of kaolin is the mineral kaolinite, which is produced by the breakdown of minerals like feldspar. Kaolinite has two layers (sheet structures made up of silica tetrahedrons and alumina octahedrons in layers, respectively) (Kloprogge, 2019).  $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$  is the molecular formula for this kaolinite group.  $\text{SiO}_2$ ,

$\text{Al}_2\text{O}_3$ , and  $\text{H}_2\text{O}$  make up the majority of the kaolinite group's chemical makeup; with trace amounts of Mg, K, Fe, and Ti also present (National Center for Biotechnology Information, 2023).

The use of kaolin as an adsorbent to remove either inorganic or organic molecules has gained popularity in recent years. The strong potential for ion-exchange, net negative charge on its surface minerals, mechanical and chemical stability, and high surface area all contribute to its high adsorption capacity and thus, it is of good prospect for pollution control (Wang and Wang, 2019).

Pollution is defined as the introduction of any substance or energy into a water body at a

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rate higher than it can accommodate (Khaleque *et al.*, 2020). When pollutants are introduced into the environment, it causes pain or discomfort to human and other living things as well as environmental degradation (Briffa, Sinagra and Blundell, 2020). Hence, environmental pollution is the contamination of the physical or biological components of the earth or atmosphere. Water is one of the habitats on Earth that is prone to contamination. Water pollution is basically any human activity that hinders the efficient use of water as a resource (Gavrilescu, 2021).

The abundance of untreated wastewater containing heavy metals and other pollutants has made heavy metal water pollution a significant environmental issue in recent years (Kumar *et al.*, 2021). Hence, techniques for cleaning heavy metal-polluted water should be developed using affordable and environmentally safe materials such as kaolin.

## MATERIALS AND METHODS

### Materials

The chemicals of high percentage purity and analytical grade were used in this study, while the kaolin clay was obtained from Lemu in Niger State, Nigeria, January, 2024, and ground to pass 90  $\mu\text{m}$  sieves.

### Methods

#### Collection and Purification of Kaolin

The purification of kaolin was carried out using the technique by Meziane *et al.* (2017). In this technique, 40 g of kaolin was suspended in one litre of distilled water and stirred. After 30 minutes of stirring, the suspension was decanted. In order to remove all the contaminants, this procedure was performed six times in quick succession. Following each washing, the solution was centrifuged at 200 rpm for 10 minutes. The resulting kaolin was then dried at 150 °C for 24 hours before being ground with a mortar and pestle.

#### Intercalation of Kaolin using Sodium Acetate (NaAc)

Kaolinite inner surfaces are altered chemically by a process called intercalation,

which significantly alters the surface characteristics of the mineral (Maged *et al.*, 2020). A mixture of 70 g of dry Kaolin clay and 45 g of dry NaAc was placed in an oven at 110 °C overnight to undergo the NaAc treatment. Then, for 10 minutes, this was manually pounded in a mortar and pestle. 2.5cm<sup>3</sup> 8.0 moldm<sup>-3</sup> NaAc were added after half of the liquid was transferred to a suitable screw-cap centrifuge tube. The other half received the same treatment, and the two halves were combined to create a homogeneous mixture. To obtain intercalated kaolin, the sample was then covered, agitated, and let to stand for 48 hours at room temperature.

#### Characterisation of Prepared Kaolin using Fourier Transform Infrared Spectrophotometer (FTIR)

Characterization of the kaolin adsorbent was carried out by Fourier transform infrared spectrophotometer. This was employed to get insight into the kaolin and to identify the functional groups present in each sample. FTIR spectrophotometer was used to determine the functional groups in the samples responsible for metal adsorption. The FTIR studies were done in the range of 4000 – 500 nm region at a resolution of 8.0 cm<sup>-1</sup>.

#### Preparation of Simulated Industrial Wastewater

To prepare the metal solutions, a 1000 ppm stock solutions containing Cd<sup>2+</sup> and Cr<sup>2+</sup> were prepared by dissolving 1.042 g of CdSO<sub>4</sub> and 1.961 g of Cr<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> in 100 cm<sup>3</sup> distilled water contained in two different 250 cm<sup>3</sup> volumetric flasks respectively. Each flask was then thoroughly shaken for 5 minutes to dissolve the salts. The content of each flask was then emptied into a single 1000 cm<sup>3</sup> volumetric flask. The solution was thoroughly mixed and then made up to the mark to prepare a multi-elemental stock solution of the two metal ions.

#### Adsorption Experiments

After each adsorption, the residual Cd and Cr ions were determined by AAS analysis as

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a standard method. The percentage adsorption (%) of kaolin was estimated using the equations 1.

$$Q\% = \frac{(C_0 - C_e)}{C_0} \quad 1$$

Where,  $C_0$  = Initial metal concentration,  $C_e$  = final metal concentration at equilibrium.

#### Effect of contact time

The effect of contact time on the removal of Cd and Cr ions was determined by adding the 0.05 g of kaolin sample to five conical flasks containing 30 cm<sup>3</sup> of a mixture of the prepared Cd and Cr solutions and then agitated at 300 rpm. An aliquot was withdrawn from each flask at a time for Cd and Cr ions determination in every 20 minutes interval.

#### Effect of adsorbent dosage

The effect of adsorbent dosages on the equilibrium uptake of Cd and Cr ions was determined by adding 0.01 g, 0.02 g, 0.03 g, 0.04 g and 0.05 g of kaolin sample to five conical flasks respectively containing 30 cm<sup>3</sup> of a mixture of the prepared Cd and Cr ions solutions. The flasks were shaken at 300 rpm and 30 °C for 75 minutes.

#### Effect of stirring speed

To determine the effect of agitation speed, 0.05 g of kaolin sample was added to five conical flasks containing 30 cm<sup>3</sup> of the prepared kaolin sample with known metal concentrations. The stirring speed was set at 100, 200, 300, 400 and 500 rpm respectively for each flask at 30 °C orbital shakers for 75 minutes. Thereafter, an aliquot was taken for AAS analysis.

#### Adsorption isotherm and kinetic models

From the available isotherm models, this study employed two of the isotherm models, namely the Freundlich and Jovanovic isotherm models. These models were employed to fit in data obtained from adsorption studies with the aim of establishing the mechanism of the adsorption process. The obtained data were used to plot isotherms in their linear form to calculate the model constants, standard errors and correlation coefficients ( $R^2$ ). The applicability of

the isotherm model to explain the adsorption process was criticised by the correlation coefficients ( $R^2$ ) and standard error values. The formulae for these adsorption isotherm models are shown in then Equations 2 and 3.

Freundlich isotherm model: This isotherm assumes adsorption occurs on heterogeneous surface

$$\text{Log}q_e = \text{Log}K_f + \frac{1}{n} \text{Log}C_e \quad 2$$

Where  $K_f$  is the Freundlich isotherm constant,  $n$  is adsorption intensity,  $C_e$  is the equilibrium concentration of adsorbate (mg/L),  $q_e$  is the amount of metal ions adsorbed at equilibrium (mg/g).

Jovanovic isotherm model: This isotherm assumes possibility of mechanical contact between the adsorbate and adsorbent.

$$\text{In}q_e = \text{In}q_{\text{max}} + K_j C_e \quad 3$$

Where  $K_j$  is Jovanovic constant ( $K_j$ ),  $q_{\text{max}}$  is maximum adsorption capacity,  $C_e$  is the equilibrium concentration of adsorbate (mg/L) and  $q_e$  is the amount of metal ions adsorbed at equilibrium (mg/g).

For the Freundlich isotherm, the plot of  $\text{In}q_e$  versus  $\text{In}C_e$  gives a straight line with slope of  $1/n$  and intercept of  $\text{In}K_f$ . For the Jovanovic isotherm, when  $\text{In}q_e$  is plotted against  $C_e$ , a straight line with slope of  $-K_j$  and intercept of  $\text{In}q_{\text{max}}$  is obtained.

The rates and mechanisms of adsorption of the Cd and Cr onto the kaolin were analyzed by applying Pseudo - first order and Pseudo - second order models. The values of the rate constants and other parameters were obtained from the slopes and intercepts of the plots. The formulae for these kinetic models are shown in Equations 4 and 5.

Pseudo –first order model: This model assumes adsorption rate decrease as temperature increase and that the rate determining step is physical adsorption.

$$\text{In}[C] = \text{In}[C_0] - kt \quad 4$$

Where  $C$  and  $C_0$  is the concentration at  $t$  and initial concentration of the heavy metals respectively and  $k$  is rate constant calculated from slope of the linear plot of  $\text{In}[C]$  against  $t$ .

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Pseudo – second order kinetic model: This model assumes adsorption rate increase as temperature increase and that the rate determining step is chemical adsorption.

$$\frac{1}{[C]} = \frac{1}{[C_0]} + kt \quad 5$$

Where  $C$  and  $C_0$  is the concentration at  $t$  and initial concentration of the heavy metals respectively and  $k$  is rate constant calculated from slope of the linear plot of  $\frac{1}{[C]}$  against  $t$ .

### Thermodynamic parameters of heavy metals adsorption

The equation 6 was employed to determine the change in Gibbs free energy ( $\Delta G$ )

of the adsorption process (Somasundaram *et al.*, 2013):

$$\Delta G = -RT \ln K \quad 6$$

The relationship between enthalpy change ( $\Delta H$ ) of adsorption, entropy change ( $\Delta S$ ) and Gibbs free energy was presented as equation (Equation 7): Equation 7 is further expressed to establish the relationship presented as Equation 8

$$\Delta G^\circ = -RT \ln K = \Delta H - T \Delta S \quad 7$$

$$\ln K = \frac{-\Delta H}{RT} + \frac{\Delta S}{R}$$

Where  $K$  is equilibrium constant

A plot of  $\ln K$  against  $1/T$  is a straight line,  $\Delta H$  and  $\Delta S$  were determined from the intercept and slope, respectively.

## RESULTS AND DISCUSSION

### Results

#### Fourier transmission infra-red (FTIR) spectrum of kaolin

Since it reveals the organic functional groups that are present on the surface of the

materials, FTIR spectroscopy is one of the crucial methods for characterizing the surface chemistry of the produced adsorbent (Lee *et al.*, 2019). The FTIR spectrum of the kaolin is displayed in Figure 1.

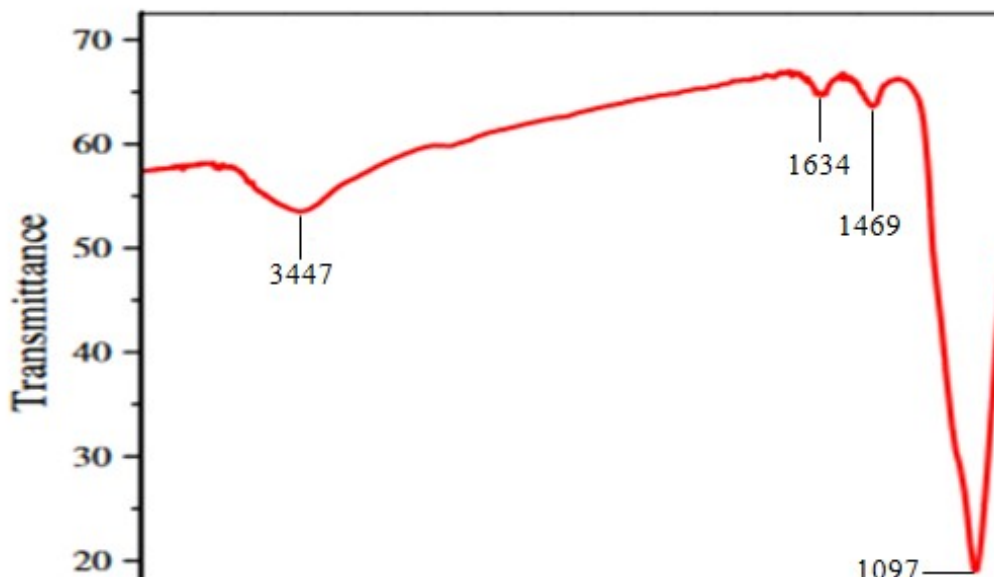


Figure 1: The FTIR spectrum of the prepared kaolin adsorbent

Results of removal efficiency of kaolin based adsorbent on Cd and Cr in simulated industrial wastewater

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### Effect of contact time on adsorption

Figure 2 shows the effect of contact time on the percentage removal of Cd and Cr

respectively from the simulated industrial wastewater using kaolin.

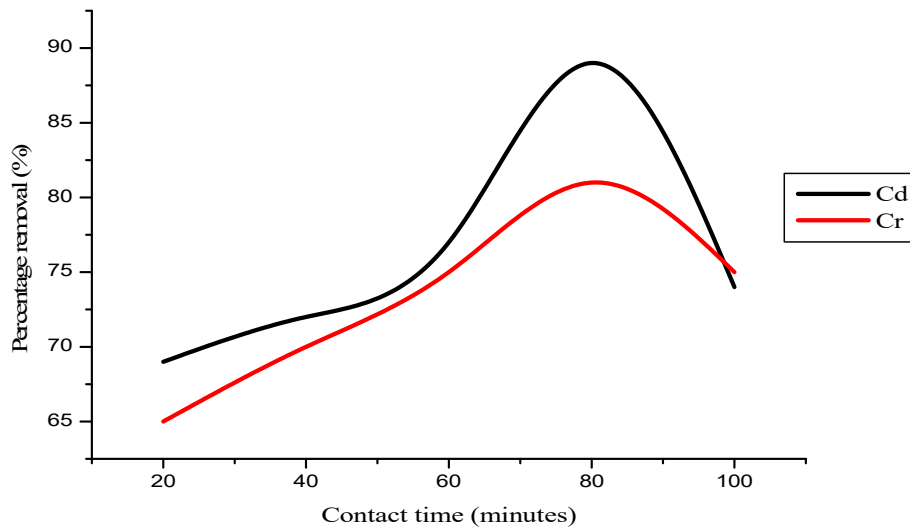


Figure 2: Effect of contact time on Cd and Cr removal

### Effect of adsorbent dosage

The dosage of adsorbent has a significant consequence on the adsorption process and it describes the capacity of the adsorbent, through the number of active binding

sites accessible to remove heavy metal ions (Vinu *et al.*, 2022). The effect of adsorbent dose on the percentage removal of Cd and Cr are shown on Figures 3.

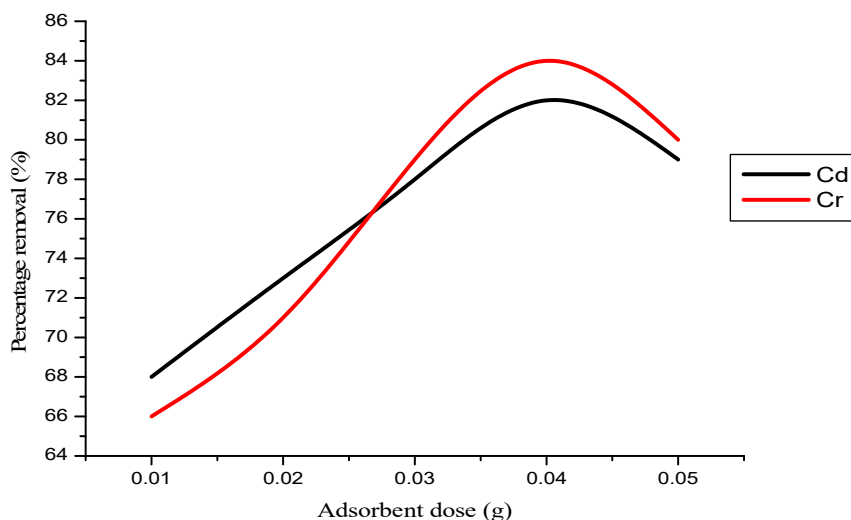


Figure 3: Effect of adsorbent dosage on Cd and Cr removal

#### Effect of stirring speed on adsorption

The effect of stirring speed on the removal of Cd and Cr by the prepared adsorbents was studied. The stirring speed of the wastewater was varied in the range of 100 – 500 rpm while other parameters (adsorbent dosage

and contact time) were kept constant at 0.04 g and 80 minutes respectively at room temperature. The effects of stirring speed on the percentage removal of Cd and Cr metals are shown in Figures 4.



Figure 4: Effect of stirring speed on Cd and Cr removal

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### Isotherm Models Parameters for the Adsorption of Cd and Cr

The data obtained from the adsorption studies were used to plot isotherms in their linear forms to calculate the model constants, standard error and correlation coefficients ( $R^2$ ). The applicability of the isotherm models to explain the adsorption processes involved were checked by the correlation coefficients ( $R^2$ ) and standard error values. Table 1 shows the isotherm models used, their constants, standard errors and  $R^2$  values for the prepared kaolin.

**Table 1:** Isotherm Models Parameters for the Adsorption of Cd and Cr from Simulated Industrial Wastewater using Kaolin

| Heavy metal | Freundlich |        |        |       |       | Jovanovic |           |       |       |
|-------------|------------|--------|--------|-------|-------|-----------|-----------|-------|-------|
|             | $K_f$      | 1/n    | n      | $R^2$ | SE    | $K_j$     | $q_{max}$ | $R^2$ | SE    |
| Cd          | 2.302      | -0.611 | -1.761 | 0.966 | 0.005 | 22.146    | 35.422    | 0.995 | 0.003 |
| Cr          | 7.125      | -0.433 | -2.153 | 0.943 | 0.013 | 11.645    | 65.204    | 0.996 | 0.010 |

\*SE means standard error

### Kinetic model of heavy metal ions adsorption

The rate of adsorption and possible adsorption mechanism of the Cd and Cr metal ions onto kaolin were analyzed by applying adsorption kinetic models to the experimental data. In this, study Pseudo-first order and

Pseudo-second order adsorption kinetic models were used to explain the kinetic of the adsorption of the selected heavy metals. The values of the rate constants and other parameters obtained are shown in Tables 2.

**Table 2:** Pseudo–first Order and Pseudo–Second Order Models Parameters for the Adsorption of the Cd and Cr Metals from Simulated Industrial Wastewater using Kaolin

| Pseudo – First Order  |                       |       |                |
|-----------------------|-----------------------|-------|----------------|
| Heavy metal           | $K_1$                 | $R^2$ | Standard error |
| Cd                    | $1.57 \times 10^{-4}$ | 0.951 | 0.001          |
| Cr                    | $1.41 \times 10^{-4}$ | 0.885 | 0.001          |
| Pseudo – Second Order |                       |       |                |
| Heavy metal           | $K_2$                 | $R^2$ | Standard error |
| Cd                    | $6.43 \times 10^{-5}$ | 0.977 | 0.0005         |
| Cr                    | $2.54 \times 10^{-4}$ | 0.968 | 0.0004         |

### Thermodynamics of the heavy metal ions adsorption

The nature of adsorption process can be known from thermodynamic parameters which are changes in the Gibb's energy ( $\Delta G^\circ$ ), enthalpy ( $\Delta H^\circ$ ), and entropy ( $\Delta S^\circ$ ). The change in Gibb's

energy  $\Delta G^\circ$  indicates the spontaneity of the adsorption process. Adsorption only occurs spontaneously if  $\Delta G^\circ$  at a given temperature is negative. In this study, Tables 3 show the thermodynamic parameters of the adsorption of the selected heavy metal ions on the kaolin.

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**Table 3:** Gibbs free energy (kJ/mol), Enthalpy change ( $\Delta H$ ) (kJ/mol) and Entropy change ( $\Delta S$ ) kJ/molK for the Adsorption of the Cd and Cr Metals on the Kaolin

| Temperature (K) | Gibbs free energy (KJ/mol) |        |
|-----------------|----------------------------|--------|
|                 | Cd                         | Cr     |
| 303             | -1.705                     | -1.397 |
| 318             | -1.801                     | -1.599 |
| 333             | -1.933                     | -1.704 |
| 348             | -1.951                     | -1.903 |
| 363             | -2.122                     | -1.796 |

## DISCUSSION

From the Figure 1, the FT-IR spectrum (4000 – 400  $\text{cm}^{-1}$ ) of the kaolin adsorbent, the peak at 3447  $\text{cm}^{-1}$  is related to the O–H stretching and the peaks at 1634 and 1469  $\text{cm}^{-1}$  refer to the H–O–H bending. The strong bands at 1097 and 462  $\text{cm}^{-1}$  represent the Si–O–Si groups of the tetrahedral sheet. The peak at 576  $\text{cm}^{-1}$  is attributed to the Al – O bending and the spectral band at 857  $\text{cm}^{-1}$  reflects the stretching vibration of Si – O. The results show that the adsorbent is a pure kaolin with chemical formula of  $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$  (Karl-Heinz *et al.*, 2016).

The result in Figure 2 shows that for the first 20 minutes of contact time, the percentage removals of Cd and Cr ions onto kaolin was more than 50 %. This implies that most of the metal ions in the wastewater were removed. On the other hand, the maximum percentage removals of Cd and Cr ions on the kaolin were at optimum contact time of 80 minutes. The high removal efficiency of the adsorbent ions may be attributed to the exceptionally electrostatic interaction between the metal ions and adsorbents surfaces containing large number of negatively charged binding sites (Gupta *et al.*, 2021).

Figure 3 shows that the percentage removal of Cd ions increases with increase in adsorbents doses in the range of 0.01 – 0.04 g. This increase in percentage removal of the metal ions may be attributed to increase in the availability of more adsorption binding sites (Darweesh *et al.*, 2022). It can be seen that the adsorbent showed appreciable optimum percentage removal for Cd. From this outcome, it can be seen that the removal efficiency was dependent on the number of available binding

sites. It can be seen from Figure 3 that for the adsorbent the initial percentage removals of Cr ions were high and increased significantly as adsorbent dosages increased. The initial high adsorption of Cr by the adsorbent may be attributed to the presence of large number of accessible binding sites available at the adsorbents surface (Darweesh *et al.*, 2013).

The effects of stirring speed upon adsorption of Cd and Cr ions were studied by varying speed from 100 to 500 rpm (Figure 4). It was observed that the percentage removal of the metal ions continued to increase significantly until at stirring speed of 400 rpm for the maximum percentage removals. At this optimum stirring speed, adsorption equilibria between bulk phase and the adsorbing surface of the adsorbent were reached. After these, the amounts of metal ions in the bulk phase were very low compared to the adsorbent surfaces, further increase in stirring speed led to releases of some of the adsorbed species back to the bulky phase, this is known as desorption.

Adsorption is usually studied using a graph which is known as adsorption isotherm. These models are commonly employed to fit in data obtained from adsorption studies with the aim of establishing the best correlation for the isotherms (Batool *et al.*, 2018). From the available isotherm models, this study employed two commonly used isotherm models, namely the Freundlich and Jovanovic isotherm models. For the Freundlich isotherm, the plot of  $\text{Log}q_e$  versus  $\text{Log}C_e$  gives a straight line with slope of  $1/n$  and intercept of  $\text{Log}K_F$ . For the Jovanovic isotherm, when  $\text{In}q_e$  is plotted against  $C_e$ , a straight line with slope of  $-K_j$  and intercept of  $\text{In}q_{\text{max}}$  is obtained.

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From Tables 1, the regression coefficients ( $R^2$ ) and standard errors of both the Freundlich and Javonovic isotherm models tested show that the adsorption isotherms data for Cd and Cr using kaolin best fitted the Jovanovic isotherm model. Meanwhile, Jovanovic isotherm model best explained the adsorption behaviour of Cd metal ions more than Cr its higher  $R^2$  and lower standard error values. Thus, the adsorptions of Cd and Cr metal ions in this study can be said to have taken place more on homogenous site with monolayer coverage of metal ions on the outer surfaces of the adsorbents along with some mechanical contacts between the metal ions and the adsorbent with competition for binding sites among the metal ions (Ajala *et al.*, 2022).

From Tables 1, The higher adsorption capacities of kaolin for Cr ions compared to the Cd ions may be attributed to the higher initial amount of Cr ions in the simulated industrial wastewater (Abdus-Salam and Adekola, 2018).

The Freundlich isotherm ( $1/n$ ) which stands for the adsorption efficiency is a parameter related to the surface heterogeneity and the energies linked in adsorption processes. It also indicates a favourable adsorption of the adsorbate on the given adsorbent (Togue, 2019). The values of  $1/n$  for the adsorption of the metal ions on the adsorbent using Freundlich adsorption isotherm are shown on Table 1. According to Mustapha *et al.*, (2019) for an adsorption,  $1/n = 0$  indicates an irreversible adsorption,  $1/n < 1$  shows a favourable adsorption and  $1/n > 1$  indicates not a favourable isotherm. This implies that the adsorption of both metal ions on the adsorbent in this study is favourable and beneficial. Also, the value of  $1/n$  less than 1 implies significant adsorption at low concentration of the metal ions takes place.

From Tables 2, Regression coefficients ( $R^2$ ) and standard errors for all the two kinetic models showed that the adsorption kinetic data for the metal ions best fit pseudo-second order kinetic models. This is due to the higher  $R^2$  and lower standard errors obtained for pseudo-second order kinetic models in the study. Therefore, the entire rate-determining steps of

adsorptions of the metal ions in this study are controlled by Chemisorption which involves the contribution or sharing of electrons between the metal ions and the functional groups (-OH and -NH<sub>2</sub>) in the kaolin (Dina *et al.*, 2022). Also, the pseudo-second-order rate constants  $K_2$  (g/mg min), obtained were more significant, which means that the heavy metal ions uptake onto the prepared adsorbents from the electroplating wastewater was fast and favourable.

The positive values of  $\Delta H^\circ$  for the adsorption of Cd and Cr by kaolin showed that the adsorption of these metals is endothermic (Surchi, 2011). On the other hand, randomness during adsorption process can be described using the value of  $\Delta S$ . The small and positive values of  $\Delta S$  obtained in this study, as showed on Table 3 suggest insignificant increase in randomness at the solid/solution interface (Bello *et al.*, 2019). Since, from the values of  $\Delta G^\circ$  for the adsorption of all the heavy metals in this study were negatives the adsorption processes were spontaneous in nature (Gusain *et al.*, 2019).

## CONCLUSION

The aim of this research was to prepare, characterise and use kaolin for the removal of Cd and Cr from simulated industrial wastewater. The research included adsorption isotherm experiments. The FTIR results showed that the prepared adsorbent had surface functional groups (-OH) which could act as binding sites for the heavy metal ions in the wastewater. The adsorption studies with the wastewater revealed that the heavy metals removal potentials of adsorbent was influenced by contact time, adsorbent dose and stirring speed and that the heavy metals were adequately removed. The adsorption isotherm data for the metal ions removal from the wastewater showed that Jovanovic isotherm model gave the best fit for the studied heavy metals. The findings established that the prepared adsorbent can be used definitely as the promising adsorbent for heavy metals removal from wastewater.

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