<https://doi.org/10.36293/sfj.2019.0026>

PREPARATION AND CHARACTERIZATION OF ADSORBENT DERIVED FROM CHICKEN FEATHER-KAOLIN COMPOSITE FOR HEAVY METAL REMOVAL FROM INDUSTRIAL WASTEWATER

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Abstract

Chicken feather-kaolin (CFK) adsorbent was produced and used for heavy metal removal from wastewater. FTIR and BET techniques were used to characterize the adsorbent. The FTIR spectra revealed the presence of –OH group of kaolin (K) and the –NH group of the protein keratin in the adsorbent. The BET analysis showed an increase in the surface area from 352.5m²/g in kaolin to 424.8m²/g in CFK and the pore volume increased from 21.66cc/g in kaolin to 24.27cc/g in CFK. The adsorbent was used to remove Pb²⁺, Cu²⁺ and Ni²⁺ from multi-metal component system and the order of preference of adsorption was Pb²⁺ > Cu²⁺ > Ni²⁺. The adsorption of Cu²⁺ and Ni²⁺ fitted the Langmuir isotherm while the adsorption of Pb²⁺ fitted the Freundlich isotherm.

Keywords: Kaolin, Chicken feather, Composite, Sorption, Isotherms

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Introduction

The name kaolin derives from the Chinese and means high ridge. High ridge refers to the hill in south-eastern China where the clay was originally discovered and used. The Chinese first used kaolin to make porcelain in the 7th and 8th centuries. It was until centuries later that other areas of the world could duplicate the process (Ismadji *et al.*, 2015). The mineral kaolinite is the major constituent in kaolin, with two-layer (sheet structures composed of units of one layer of silica tetrahedrons and one layer of alumina octahedrons). This kaolinite group has the molecular formula Al₂Si₂O₅(OH)₄ (Hydrated aluminosilicate) (Ismadji *et al.*, 2015).

Chicken feathers are one of the epidermal growths that form the distinctive outer

covering or plumage on birds. They are considered the one of the most complex structures found in vertebrates and indeed a premier example of a complex evolutionary novelty with about 90% Keratin protein and 10% water (Prum and Brush, 2002). Composites can be defined as natural or synthesized materials made from two or more materials with varying physical and chemical properties which remain separate and distinct at the microscopic or macroscopic scale within the material. Composites are synthesized to combine the desired properties of the materials in the composite (Noor, 2014).

This study is aimed at developing a very cheap and efficient composite material for heavy metal removal from industrial wastewater

using readily available kaolin and chicken feather and use the adsorbents to adsorb metals Pb^{2+} , Cu^{2+} and Ni^{2+} from simulated industrial wastewater.

Materials/Methods

Materials

The Kaolin clay ($Al_2Si_2O_5(OH)_4$) was obtained from Kankara in Katsina State, Nigeria, while potassium acetate was used to produce potassium acetate (KOAc)/kaolinite intermediate via intercalation reaction. The chicken feathers were collected from the poultry slaughter house in Karu Modern Market Abuja, Nigeria. Distilled water was used to prepare sample solutions and used also with detergents to wash the chicken feathers, kaolin and other apparatuses. The BET analysis was done in the main laboratory in Federal University of Technology, Minna, Niger State, Nigeria. FTIR and AAS analyses were carried out at Multi-User Research Laboratory, Ahmadu Bello University, Zaria. FTIR Spectrophotometer was used to characterize the adsorbents in terms of the functional groups present. Modern analytical balance was employed for weighing the samples.

Methods

Purification of kaolinite

A 30 to 40 g amount of kaolin was put in suspension in one liter of distilled water. The suspension was then put under agitation for 30 minute. This operation was repeated six successive times in order to eliminate all the impurities. After each washing, the suspension was centrifuged for 10 minute, then at the end, the kaolin was dried in a drying oven at $85^\circ C$ for two days. This procedure is in accordance to the method used by Meziane *et al.* (2017).

Purification of chicken feather and grinding.

The chicken feather was washed several times with tap water and detergent, and rinsed with distilled water and was left to dry in air for five days. Thereafter, the chicken feathers were dried for two days in an oven at $70^\circ C$ to ensure complete dryness. Then, a mortar and pestle was first used to pound the chicken feathers to disintegrate it and then ground using a mechanical blender to convert it to powdered form before use.

Potassium acetate treatment (intercalation reaction)

The intercalation is a chemical process of modifying kaolinite inner surfaces, resulting in significant changes in the kaolinite surface properties. For example, intercalation can cause significant disordering of the kaolinites, increased surface areas and provide surfaces, which are more readily available for chemical reactions (Asmatulu, 2002; Frost *et al.*, 2001; Rintoul *et al.*, 2000). For the KOAc treatment, the method of Asmatulu (2002) with modification was employed. A mixture of 70 g of dry kaolin clay with 45 g of dry KOAc was kept in an Oven at $110^\circ C$ overnight. This was then hand-ground for 10 min in a warm ($35^\circ C$ mortar and pestle). Half of the mixture was then transferred to a suitable screw-cap centrifuge tube and 2.5 cm^3 of 8M KOAc was added. This was also done to the other half and they were then mixed up to get a homogeneous mixture. The sample was then covered, shaken and allowed to stand for 2 days at room temperature. This was thereafter used for preparing the composite via guest displacement reaction.

Preparation of CFK composite

Guest displacement reaction is one of the various techniques which were developed in order to facilitate the process of intercalation of the organic compounds between the

lamellar layers of kaolinite. Displacement involves the replacement of a directly intercalated species by a second organic molecule. The experimental method used for CFK preparation was via guest displacement reaction according to the procedure described by Hussein *et al.* (2015) and modified slightly in this work with respect to the weight of the samples. The kaolin/potassium acetate intercalate previously prepared which contained 70 g kaolin and 45 g potassium acetate was placed in a beaker (500cm³). A 70 g amount of ground chicken feather was weighed and added to the clay kaolin/potassium acetate intercalates in the beaker. The mixture was then mixed manually in a mixer in its dry form. A 250 ml of water was then added to the mixture while stirring continuously to get a kaolin-chicken feather solution. It was then washed thoroughly with more distilled water to remove any potassium acetate left. The mixture was thereafter spread in a porcelain crucible and heated in an oven at 150°C till dryness. Finally, the product was manually ground in a mortar for a period of 2.5 hour, followed by a mechanical grinding for 15 minute. It was then sieved using a 90 um sieve and this was subjected to further analyses.

Equilibrium studies experiment

100 ml of varying concentrations (50 ppm, 100 ppm, 150 ppm, 200 ppm and 250 ppm) of the Pb²⁺, Cu²⁺ and Ni²⁺ solutions were carefully measured into 120 ml tubes containing 0.5 g of the adsorbent. These were agitated at 200rpm for 1.30hour to equilibrate them at temperature (25°C). Samples were drawn out from the mixture and analyzed using AAS. The data obtained from experiment were fitted into two different isotherm models of Langmuir and Freundlich. The correlation coefficients R² were used to determine the best fit isotherm model.

Freundlich and Langmuir models were used to analyze the adsorption equilibrium data. The Langmuir (Langmuir, 1918) model assumes that uptake of metal ions occurs on a homogenous surface by monolayer adsorption without any interaction between adsorbed ions. The Langmuir isotherm in a linearized form is:

$$\frac{1}{q_e} = \frac{1}{q_{max}} + \frac{1}{q_{max}K_L C_e}$$

where q_e is the amount adsorbed (mg/g), C_e is the equilibrium concentration of the metal ion (mg/l), q_{max} (mg/g) is the maximum amount of adsorbed metal ion per unit mass of sorbent corresponding to complete coverage of the adsorptive sites, K_L (l/mg) is the Langmuir constant related to the energy of adsorption. The favourability of adsorption of the metal ions on the CFK (Preference of CFK for the adsorption of any of the three metals over the other) was tested using the essential features of the Langmuir isotherm model, expressed in terms of a dimensionless constant called separation factor (SF) given by the relationship:

$$SF = \frac{1}{1 + K_L C_o}$$

Where K_L (l/mg) is Langmuir isotherm constant and C_o (mg/l) is initial metal ion concentration. The smaller the SF value, the more favourable is the relationship between the adsorbent and the adsorbate and the higher the adsorption.

The adsorption on a heterogeneous surface is described by linearized Freundlich model (1906).

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e$$

Where C_e (mg/l) is the equilibrium concentration and q_e (mg/g) is the amount adsorbed metal ion per unit mass of the adsorbent, the constant, n is the Freundlich equation exponent that represents the

parameter characterizing quasi-Gaussian energetic heterogeneity of the adsorption surface, it should have values ranging from 1 to 10 to be classified as favorable adsorption (Chantawong *et al.*, 2003).

The isotherm parameter $\frac{1}{n}$ is a measure of the intensity of adsorption of metal ions onto the CFK surface. It takes values from 0.1-0.5. The higher the $\frac{1}{n}$ value, the more favourable is the adsorption. K_f (l/g) represents the Freundlich constant which shows the relative adsorption capacity of the adsorbent, obtained from the intercept on the plot of $\ln q_e$ against $\ln C_e$. The higher the K_f value, the higher the maximum adsorption capacity.

Results and Discussion

FTIR Analysis of Kaolin

Figure 1 shows the FTIR spectra of the kaolin sample. Kaolin showed peaks at 3,690.10 and 3,623.0 cm^{-1} due to the stretching vibrations of hydroxyl groups (Salwa *et al.*, 2015). The band at 3690 cm^{-1} was attributed to the hydroxyl group sitting at the edges of the kaolin platelets and corresponds to the hydroxyl group at the surface of the alumina octahedral layer that interacts with the oxygen atoms of the adjacent silica tetrahedral layers. The band at 3623 cm^{-1} indicates the internal hydroxyl groups. The asymmetric Si-O-Si stretching vibration was responsible for the band at 1114 cm^{-1} . The band at 998 cm^{-1} was attributed to the bending Al-OH vibration of the -OH groups on the alumina faces (Melia *et al.*, 2012). The band at 749 cm^{-1} corresponds to the Si-O-Al vibrations (Wu *et al.*, 2012).

Figure 1: FTIR Spectra of Kaolin

FTIR Analysis of Chicken Feather

Figure 2 is the FTIR spectra for the chicken feather. The peak at 3690.10 cm^{-1} indicates the O-H group of water in the feather fibre. The peak at 3265.10 cm^{-1} was due to N-H (amine) group of amino acid in the CF. The peak at 2922.20 cm^{-1} shows the C-H stretch. The peak at 1923.30 cm^{-1} (Siti *et al.*, 2014) indicates the band due to the C-C of the alkyl group of the

amino acid of the CF fibre. The characteristic peak at 2087.30 cm^{-1} corresponds to nitrile C-N stretch and the peak that appears at 1632.60 cm^{-1} is assigned to C=O group of amino acid and the ones at 1520.80 cm^{-1} and 1446.20 cm^{-1} show the N-H bending vibration and C=C stretch. The peaks which appeared around 1388.60 cm^{-1} region in the spectra shows C-H bending formation. In addition, the C-O (carboxylic acid) originating mainly

Figure 2: FTIR Spectra of CF

FTIR Analysis of the CFK Composite

Figure 3 showed that the sharp band at 3690.10 cm^{-1} in the CF spectra has shifted to 3734.80 cm^{-1} in CFK indicating an interaction between the CF and K. The band at $3,690.10\text{ cm}^{-1}$ due to stretching vibrations of hydroxyl groups of the K was still visibly present indicating the presence of kaolin in the composite. In the CFK composite, the CF provided an efficient and strong support for K in forming the composite via the interaction between the nitrogen atom of the amino group of the protein keratin in CF and the Oxygen atom of the hydroxyl group on the kaolinite surface in K with more number of hydroxyl, carboxyl, amino and alcohol groups available

for adsorption of metal ions. This was clearly evident in the appearance of new absorption bands at 3649.10 , 3619.20 and 3425.40 cm^{-1} which were assigned to O-H and N-H; inter hydrogen bonds' vibration between the K and CF. The band at 3265.10 cm^{-1} in the CF spectra which shifted to 3291.20 cm^{-1} in the CFK composite also suggested a mechanical or H-bonding interaction between CF and K. This is supported by the appearance of several C-H stretch and alkyl group bands due to the CF contribution to the composite at the region around 1950 cm^{-1} to 2950 cm^{-1} which were not originally present in the spectra of K alone. Though the C=O stretching in carboxyl or amide group at 1632.60 cm^{-1} in the spectra of CF remained unchanged in the composite, the

adsorption bands at 1520.80 cm^{-1} and 1446.20 cm^{-1} showing the N-H bending vibration and C=C stretch in CF have now shifted to 1554 cm^{-1} and 1408.90 cm^{-1} respectively in the CFK, and further indicates that, interaction has taken place between CF and K (Ratna *et al.*, 2015). The band at 1032.50 cm^{-1} assigned to PO_4^{3-} group (Egwuatu *et al.*, 2014) in the CF spectra shifted to 1092.10 cm^{-1} in the CFK indicating the bonding of CF and K in the

composite CFK. The interaction between CF and K in CFK has led to the creation of more active bonding surfaces or adsorption sites in CFK due to the presence of more number of N and O atoms occasioned by the appearance of additional bands due to new O-H and N-H groups at the region between $3000\text{ to }4000\text{ cm}^{-1}$ in CFK and this enhances the adsorption capacity of CFK.

Figure 3: FTIR Spectra of CFK

BET Surface Area for Kaolin (K) and the Composite (CFK)

Table 1 shows the BET results for K and CFK. The results indicate that there was an increase in the surface area of $352.5\text{ m}^2/\text{g}$ in K to $424.8\text{ m}^2/\text{g}$ in CFK and a pore volume increase from $21.66\text{ cm}^3/\text{g}$ in K to $24.27\text{ cm}^3/\text{g}$ in CFK, but there were no appreciable changes in their

pore sizes. The presence of CF that served as a support upon which K particles were attached in the composite CFK contributed more actively adsorbing groups (as already established by the results from FTIR) and this is believed to be responsible for the increase in the surface area and pore volume of the composite.

Table 1: Sample Surface Area by BET Method

Samples	BET surface Area (m^2/g)	Pore Volume(cc/g)	Pore Size (nm)
K	352.5	21.66	2.132
CFK	424.8	24.27	2.131

Adsorption Isotherms

Langmuir isotherm

The Langmuir isotherm model was chosen for the estimation of maximum adsorption capacity corresponding to complete monolayer coverage on the CFK surface. The plots of $\left(\frac{1}{q_e}\right)$ against $\left(\frac{1}{C_e}\right)$ for Pb^{2+} , Cu^{2+} and Ni^{2+} are shown in Figures 4a-c and the isotherm parameters, $q_{\max}$, K_L and R^2 values are presented in Table 2. The higher the K_L value, the more favorable is the monolayer adsorption. The adsorption coefficient, K_L that is related to the apparent energy of sorption for Ni^{2+} (2.54 l/g) was greater than that of Cu^{2+} (0.56 l/g) and Pb^{2+} (-1.14 l/g). The effectiveness of CFK in the monolayer adsorption of the three metals from aqueous system was $\text{Ni}^{2+} > \text{Cu}^{2+} > \text{Pb}^{2+}$. This preferential sorption may be explained in terms of the ionic radii of the metal ions ($\text{Pb}^{2+} = 1.19\text{\AA}$, $\text{Cu}^{2+} = 0.73\text{\AA}$; $\text{Ni}^{2+} = 0.69\text{\AA}$). The element with smaller ionic radius competes faster for exchange sites during physical adsorption than those of larger ionic radius. As ionic radius increases, charge density decreases and the adsorption of Pb^{2+} on CFK therefore decreases.

The sorption capacity, $q_{\max}$ measures the maximum sorption capacity corresponding to complete monolayer coverage. The $q_{\max}$ for Cu^{2+} is (78.13 mg/g) which is greater than Ni^{2+} (44.25 mg/g) and Pb^{2+} (-9.35mg/g). This implies that, in the multi-component system comprising Pb^{2+} , Cu^{2+} and Ni^{2+} , Pb^{2+} adsorption cannot be explained by Langmuir model. Its $q_{\max}$ value was the smallest and negative with R^2 value of 0.6544 indicating that Pb^{2+} did not follow the Langmuir model and monolayer adsorption by CFK with respect to Pb^{2+} is impossible but is possible with respect to Cu^{2+} and Ni^{2+} . The highest $q_{\max}$ and R^2 values for Cu^{2+} followed that of Ni^{2+} and

implies that Langmuir isotherm is best fitted for Cu^{2+} adsorption followed by Ni^{2+} with a slight difference. So, the ideal order is $\text{Cu}^{2+} > \text{Ni}^{2+} > \text{Pb}^{2+}$.

The fitting of Cu^{2+} and Ni^{2+} to Langmuir model also implies their adsorption mechanism is via chemisorption. The SF value for the adsorption of Pb^{2+} , Cu^{2+} and Ni^{2+} on CFK at initial concentration of 50 ppm (lowest concentration studied) and 250 ppm (highest concentration studied) are listed in Table 2. The SF parameters for the three metals are less than unity signifying that CFK is an ideal adsorbent capable of adsorbing the metal ions from the solution.

Figure 4a: Langmuir Equilibrium Isotherm Plot for the Sorption of Pb^{2+} onto CFK Composite

Figure 4b: Langmuir equilibrium isotherm plot for the sorption of Cu^{2+} onto CFK composite

Figure 4c: Langmuir equilibrium isotherm plot for the sorption of Ni^{2+} onto CFK composite

However, the SF values in the order $\text{Cu}^{2+} > \text{Ni}^{2+} > \text{Pb}^{2+}$, show that in a mixed metal ion system or multi-component system, Pb^{2+} will compete for the binding sites better and faster than Ni^{2+} and Cu^{2+} .

Freundlich isotherm

The Freundlich isotherms for the metal ions are as shown in Figures 5a-c, and the isotherm constants for Pb^{2+} , Ni^{2+} and Cu^{2+}

on CFK are presented in Table 2. The high $\frac{1}{n}$ value of Pb^{2+} (0.47) in comparison to Cu^{2+} (0.1) and Ni^{2+} (0.04), first indicate the preferential sorption of Pb^{2+} than Cu^{2+} and Ni^{2+} . It also indicates the ability of the CFK to remove the metal ions from solution even at high concentrations. The K_f value of Pb^{2+} (115.58 l/g) is greater than that of Cu^{2+} (42.52 l/g) and Ni^{2+} (32.79 l/g) suggesting that Pb^{2+} has greater adsorption tendency towards the CFK than the other two metals. This remarkable difference is due to the considerable difference in their hydration radii and hydration enthalpies.

Table 2: Langmuir and Freundlich isotherm parameters

Metal Ions	Q_m (mg/g)	K_L (l/g)	Langmuir R^2	Freundlich R^2	$1/n$	K_f (l/g)	SF at (50ppm)	SF at (250ppm)
Pb^{2+}	9.35	-1.14	0.6544	0.8002	0.47	115.58	-0.02	-0.004
Cu^{2+}	78.13	0.56	0.9205	0.9212	0.1	42.52	0.034	0.007
Ni^{2+}	44.25	2.52	0.9089	0.8779	0.04	32.79	0.008	0.002

The hydration radii of the metal ions are 4.01Å for Pb^{2+} , 4.19Å for Cu^{2+} and 4.50Å for Ni^{2+} .

Ideally, the smallest cation will be adsorbed faster and in larger quantities than the larger cations because the smaller the cations, the easier it is for it to pass through the pores and channels of the adsorbent (CFK). Therefore, the smaller the hydrated radius, the higher the affinity for adsorption onto the CFK surface. Cations with low hydration

energies are preferably adsorbed than the ones with high hydration energies. This implies that the more a cation is hydrated, the stronger is its hydration enthalpy and the less it can interact with the adsorbent. Consequently, Pb^{2+} with hydration energy of 1481 kJ/mol was adsorbed preferably compared to Cu^{2+} and Ni^{2+} with 2010 kJ/mol and 2105 kJ/mol respectively. The order of sorption as described by the Freundlich isotherm is $\text{Pb}^{2+} > \text{Cu}^{2+} > \text{Ni}^{2+}$.

Figure 5a: Freundlich Equilibrium Isotherm Plot for the Sorption of Pb^{2+} onto CFK Composite

Figure 5b: Freundlich Equilibrium isotherm plot for the sorption of Cu^{2+} onto CFK composite

Figure 5c: Freundlich Equilibrium isotherm plot for the sorption of Ni^{2+} onto CFK composite

Conclusion

The adsorbent derived from chicken feather-kaolin composite has been shown to be very effective for heavy metal removal from solutions and suitable for demineralization

of industrial wastewater. The BET results revealed an increase in the surface area and pore volume of the chicken feather-kaolin composite compared to either of the constituents of the composite. The order of

the preference of sorption in multi-metal solution was $\text{Pb}^{2+} > \text{Cu}^{2+} > \text{Ni}^{2+}$. The adsorption data fitted the Langmuir and Freundlich isotherms for Cu^{2+} and Ni^{2+} , while adsorption of Pb^{2+} fitted the Freundlich Isotherm.

Funding

No funding by any agency

Conflict of Interest

The authors declare no conflict of interest.

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