

COMPARATIVE ANALYSIS OF ACTIVATED CORN COB AND BENTONITE CLAY FOR THE REMOVAL OF LEAD AND NICKEL FROM RAW WATER

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Abstract

The extensive use of commercial activated carbon as an adsorbent for the purification of industrial effluent is not economical for small and medium-sized enterprises due to its high operational cost. This study was carried out to compare the adsorptive capacity of bentonite clay and activated corn cob ("BC" and "ACC") for the removal of lead (II) and nickel (II) ions from an aqueous solution. The results obtained from the characterization of the BC and ACC are pH: 7.43 and 6.74; moisture content: 36.45kg/kg and 12.10kg/kg, and bulk density: 1.243g/ml and 1.162g/ml, respectively. Normality tests using the coefficient of skewness indicated that the set of data was not normally distributed. An analysis of variance (ANOVA) test conducted using Friedman's 2-way ANOVA test indicated *p* values of 0.0253 against an alpha value of 0.05, which indicates significance. The Friedman results indicated significance with respect to the varied dosages, initial concentrations, and contact time. The effect of the adsorbent was not significant. The adsorption isotherms were analysed using the Langmuir, Freundlich, and Temkin isotherms. Most research studies have shown that adsorption experiments performed using most low-cost materials tend to follow the Freundlich adsorption isotherm, but the results of this experimental study proved that activated corn cob and bentonite clay performed better with the Temkin adsorption isotherm with $0.879 \leq R^2 \leq 0.917$ for the bentonite clay and $0.9572 \leq R^2 \leq 0.99$ for the activated corn cob respectively. The study revealed that these materials are good adsorbents that can be used for the removal of lead (II) and nickel (II) ions from an aqueous solution.

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Key words

- Adsorbent - bentonite clay & activated corn cob,
- Adsorbates – lead and nickel,
- Langmuir,
- Freundlich and Temkin isotherms.

1 INTRODUCTION

Water is a fundamental need for human existence. Only about 0.0007% of the earth's fresh water is found in lakes, freshwater rivers, reservoirs, and groundwater resources (Anis Al-Layla et al., 1978; UN, 1997). Because of recent upsurges in the use of water, water scarcity has become a topical issue in Nigeria and the Sahelian coun-

tries in Africa. The need to recycle wastewater is obvious in the northern part of Nigeria.

Ambali et al, (2015) stated that heavy metal pollution of the environment has become a growing ecological crisis and concern and is therefore the subject of much research. Various types of health issues in human beings are reported occurring due to water pollution. As far as inorganic and organic contaminants are concerned, the most signif-

icant with regard to health are heavy metals, some of which include arsenic, lead, chromium, mercury, cadmium and nickel (WHO, 2010). Lead is one of the heavy metals of importance. Although lead is a naturally occurring substance, anthropogenic activities such as the burning of fossil fuels and mining have contributed to the discharge of high levels of lead in the environment. It is an important raw material for many products such as the production of lead-acid batteries and other metallic products. In Nigeria, most industries discharge their effluents into water bodies, particularly rivers and streams, and, as such, hazardous elements from the effluents contaminate the water (Nwaogazie and Ogelle, 1997; Nwaogazie, 1990). In humans, the kidney is most affected by lead (Abasi et al., 2011). One case of lead poisoning in recent times is the 2010 lead poisoning in the village of Bagega in Zamfara State in which 17,000 people were affected, and 500 casualties were recorded due to mining activities that led to the contamination of the domestic water source (WHO, 2010; Hassan et al., 2015).

Nickel and its compounds are used for much production in various industries. For instance, nickel is used for the production of stainless steel and is also used in the metallurgical, chemical and food processing industries. The consumption of nickel causes serious effects such as headaches, nausea, dry coughs, vomiting, chest pain, and a wide range of respiratory issues (Ambali et al., 2015).

The adsorption process is an efficient and effective method for the removal of a wide variety of toxic pollutants from raw water. Activated carbon has been globally recognized as the oldest, most widely used, and popular adsorbent in the water and wastewater treatment industries (Badmus et al., 2007). Conversely, due to the high cost of commercial activated carbon, research has been conducted on the use of more cost-effective adsorbents and lignocellulose wastes, such as corn cobs. The corn cob is the central core where kernels grow and is generally disposed of as waste. It is found in most parts of Nigeria, especially during the planting and harvesting seasons, which span from April to October. There is a vast deposit of bentonite clay in rivers in Nigeria spread across the country. Bentonite clay and corn cobs are both low-cost adsorbents found in abundance in Nigeria. Therefore, the aim of this study is to establish the comparative effectiveness of their adsorption.

2 MATERIALS AND METHODS

2.1 Collection and Preparation of the Samples

The adsorbent samples used were corn cob and bentonite clay. The raw materials were washed with water from the tap to remove dirt and rinsed with de-ionized water; they were then sun dried for twenty-four (24) hours, after which they were crushed by a local pestle and mortar and then processed in a laboratory mill. The crushed samples were oven dried at 105°C for 12 hours.

Forty grams (40g) of dried, ground, and sieved samples of corn cob were weighed into a two (2) litre beaker. Then, five hundred millilitres (500ml) of phosphoric acid (H_3PO_4) were measured and added to the samples and left for 24 hours. The activated samples were washed with distilled water from the lab and the pH was adjusted to 6. They were measured with a digital pH meter, and the samples were then dried in an oven at 105°C. The raw bentonite clay was activated by thermal activation. The preparation of the thermally activated bentonite clay was carried out by calcinating the raw bentonite clay in a muffle furnace. The thermal activation of the bentonite clay was carried out with 30 grams of bentonite clay placed in a crucible. This given amount of bentonite clay was placed in a muffle furnace. Then the temperature of the muffle furnace was set at the preferred temperature of 105°C and allowed to rise to the expected temperature of 200°C.

2.2 Preparation of Lead and Nickel solutions

The permissible limits set for lead (Pb) and nickel (Ni) in water by the Nigerian Standard for Drinking Water Quality (NSDWQ) are 0.01 mg/L and 0.02 mg/L respectively. For this experiment stock solutions of both lead and nickel were produced in a laboratory.

One thousand milligrams per litre (1000 mg/l) of nickel acetate tetrahydrate ($(CH_3COO)_2Ni \cdot 4H_2O$) (Source: BDH Chemicals U.K. with Mw = 248.86g. Grade: ANALAR (Analytical reagent)) were prepared for use throughout the experimental work by dissolving 4.235g of nickel acetate tetrahydrate powder in 1000 ml of distilled water. A stock solution of a single solute of Pb (II) was prepared by dissolving the requisite amount of lead nitrate in a 1000 ml volumetric flask; de-ionised water was then added up to a mark to dilute it. The concentration of the single solute in the respective stock solution prepared was 1000 mg/L. Test solutions having concentrations of 20, 40, 60, 80 and 100 mg/L were prepared for the experiment.

2.3 Data Analysis

The activated corn cob and bentonite clay produced were characterized by checking the size of the pores, pH, moisture contents, bulk density and specific gravity. The nature of the interaction between the adsorbate and adsorbent can be expressed from the shape of the isotherm. The Langmuir, Freundlich and Temkin models were used to characterise the adsorbate adsorbent interaction in the experiment. A number of statistical data analyses methods were adopted, namely, a normality test using the coefficient of skewness and the Shapiro-Wilk test. The results of the normality test determine if parametric or non-parametric statistics will be adopted for the rest of the data analyses. The Analysis of Variance was featured to facilitate the comparative analysis of the efficiencies of the adsorbents (ACC and BC) with respect to the removal of the Pb and Ni respectively. Linear regression modelling featured prominently in the data fitting.

Langmuir Isotherm

The Langmuir model can be presented by the equation:

$$\frac{C_e}{q_e} = \frac{1}{q_{max}k_l} + \frac{C_e}{q_{max}} \quad (1)$$

where: q_{max} is the monolayer adsorption capacity of the adsorbent (mg/g); k_l is the Langmuir adsorption constant (L/mg); C_e is the equilibrium metal ion concentration in the solution (mg/L); q_e is the equilibrium metal ion concentration in the adsorbent (mg/g).

The values of q_{max} and k_l are calculated respectively from the slope and intercept of the plot of C_e/q_e vs. C_e .

Freundlich Isotherm

The equations of the Freundlich adsorption models used in the study are expressed as:

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

where: K_f is a constant related to the adsorption capacity (Freundlich constant); $1/n$ is an empirical parameter related to the adsorption intensity (which varies with the heterogeneity of the material).

The values of $1/n$ and K_f are calculated respectively from the slope and the intercept of the plot of $\log q_e$ vs. $\log C_e$.

Temkin Isotherm

This isotherm is composed of a factor that clearly takes into account the adsorbent-adsorbate interactions. The Temkin model is given by the following equation:

$$q_e = B \ln A_T + B \ln C_e \quad (3)$$

where:

$$B = \frac{RT}{b_T} \quad (4)$$

A_T = Temkin isotherm equilibrium binding constant (L/g); b_T = Temkin isotherm constant; R = universal gas constant (8.314J/mol/K); T = Temperature at 298K; B = Constant related to the heat of the sorption (J/mol).

3 RESULTS AND DISCUSSION

3.1 Results

3.1.1 Characterization of the Adsorbents and Adsorption Experiments

The characterization of the selected adsorbents in terms of their moisture content, porosity, pH, bulk density and surface area was carried out (Table 1). The effect of the adsorbent dosage, contact time, and initial concentration on the removal of the Pb (II) and Ni (II) with the ACC and BC are represented in Figures 1, 2 and 3, respectively.

The surface area was calculated by the following equation:

$$s = 32V - 25 \quad (5)$$

where s = surface area of the activated sample, and V = volume of sodium hydroxide required to raise the pH of the sample from 4 to 9

for ACC, $V = 7$ ml. Substituting in Equation 5: $s = 32(7) - 25 = 199 \text{ m}^2$ for BC, $V = 5$ ml, which yields 135 m^2

The bulk density is given by the following equation:

$$\frac{\text{weight of dish + sample} - \text{weight of dish}}{\text{weight of dish + water} - \text{weight of dish}} \quad (6)$$

while the porosity is given by the following equation:

$$1 - \frac{\text{Bulk density}}{\text{particle density}} \quad (7)$$

Tab.1 PhysicoChemical Characteristics of Activated Corn Cob and Bentonite Clay

Parameter (Unit)	Activated Corn Cob (ACC)	Bentonite Clay (BC)
pH	6.74	7.43
Moisture Content, %	12.10	36.45
Bulk Density g/ml	1.162	1.243
Porosity	0.0077	0.0056
Surface Area m^2	199	135

3.1.2 Normality Test

The laboratory results on the rates of adsorption were subjected to the normality test. The coefficient of skewness (α) was used to validate if the data sets were normally distributed for $\alpha = 0$; otherwise, it is not normally distributed. The skewness approach was adopted, given that the data sets are of a small sample size. The results for the normality analysis are presented in Table 2.

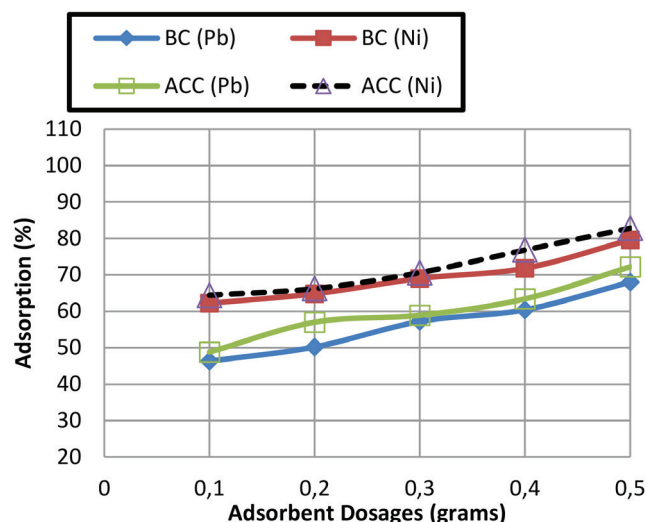


Fig. 1 Effect of adsorbent dosage on removal of adsorbate

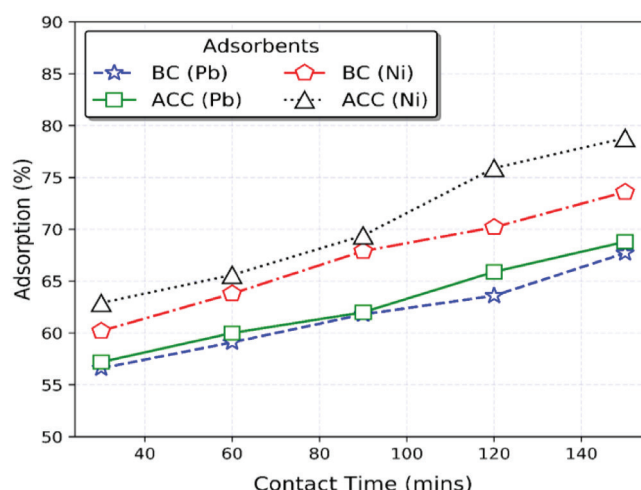


Fig. 2 Effect of contact time on removal of adsorbate

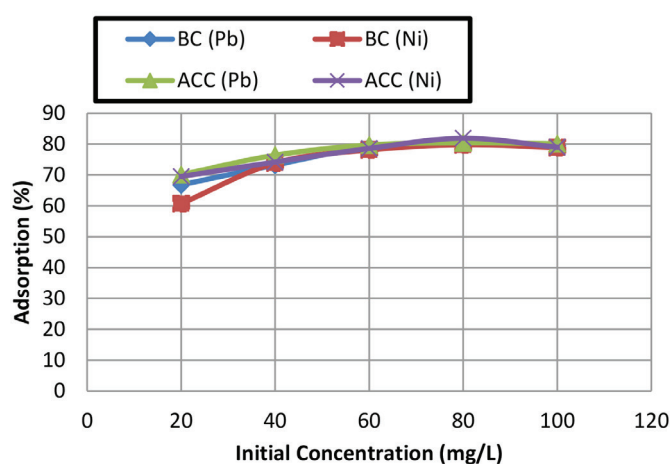


Fig. 3 Effect of initial concentration on removal of adsorbate

Tab. 2 Normality Test for adsorption rates of Pb and Ni using ACC and BC

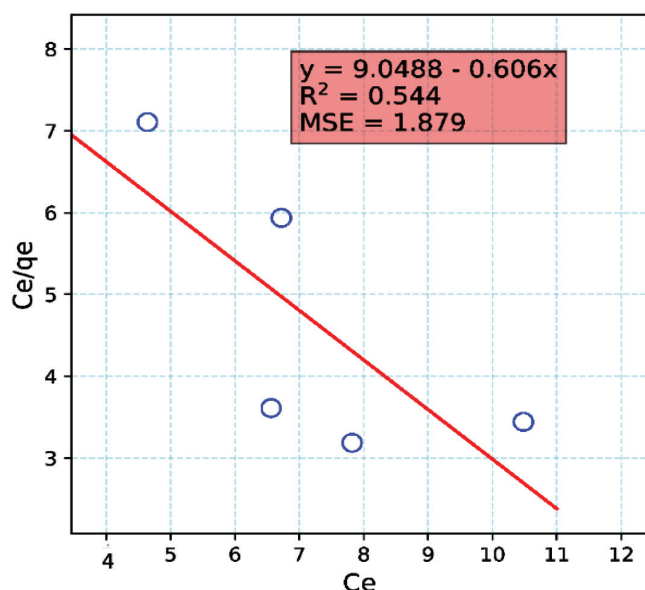
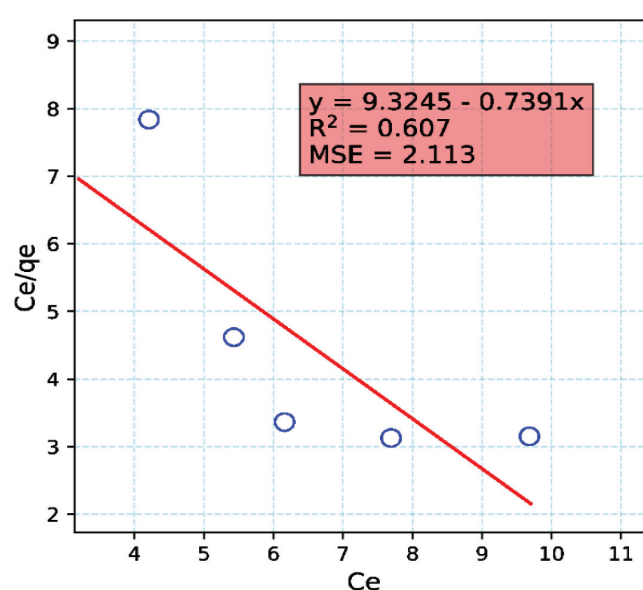
Adsorption Test	Adsorbent	Coefficient of Skewness	Remark
Adsorbent dosage (Pb)	BC	0.2271	NND ⁺
	ACC	0.2725	NND
Adsorbent dosage (Ni)	BC	0.7551	NND
	ACC	0.5829	NND
Contact Time (Pb)	BC	0.325	NND
	ACC	0.223	NND
Contact Time (Ni)	BC	-0.208	NND
	ACC	0.220	NND
Initial Conc (Pb)	BC	0.678	NND
	ACC	0.585	NND
Initial Conc (Ni)	BC	1.351	NND
	ACC	1.088	NND

Level of significance = 0.05; ⁺NND = Not Normally Distributed

Given that the experimental data indicates “not normally distributed”, further statistical analyses on the data sets followed non-parametric methods.

Tab. 3. Friedman two-way test for the adsorption rates of Pb and Ni using ACC and BC

Adsorption Test	Q (Observed value)	Q (Critical value)	DF	p-value	Alpha value
Adsorbent dosage (Pb)	5	3.8415	1	0.0253	0.05
Adsorbent dosage (Ni)	5	3.8415	1	0.0253	0.05
Contact Time (Pb)	5	3.8415	1	0.0253	0.05
Contact Time (Ni)	5	3.8415	1	0.0253	0.05
Initial Conc (Pb)	5	3.8415	1	0.0253	0.05
Initial Conc (Ni)	5	3.8415	1	0.0253	0.05

**Fig. 4** C_e versus C_e/q_e for BC adsorbent Pb adsorption (Langmuir model)**Fig. 5** C_e versus C_e/q_e for ACC adsorbent Pb adsorption (Langmuir model)

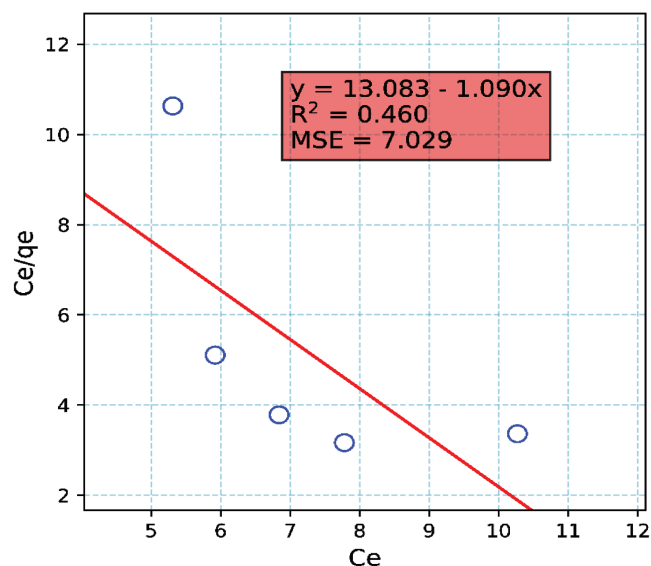
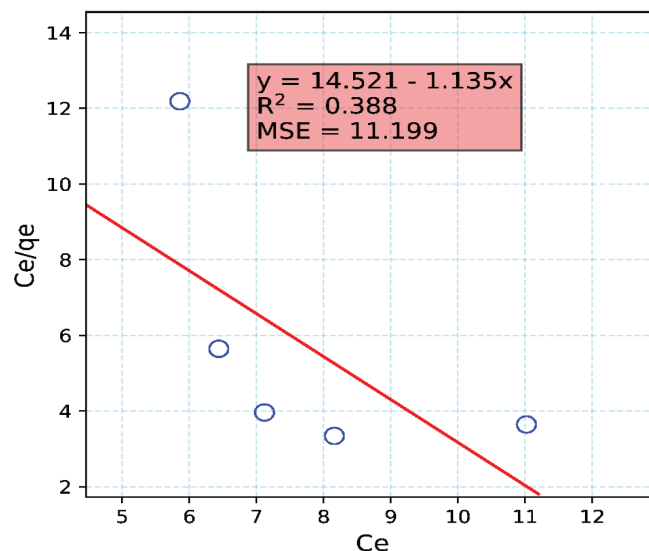
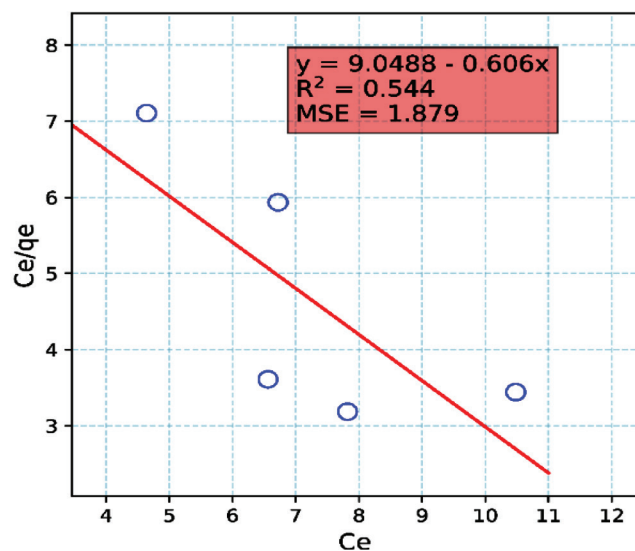
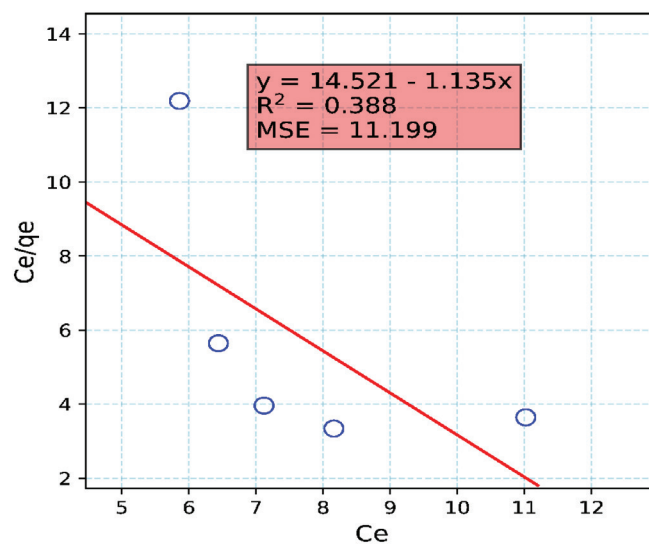
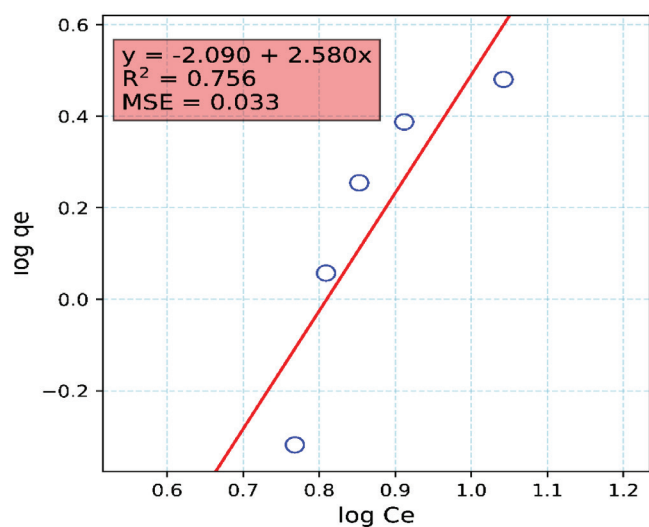
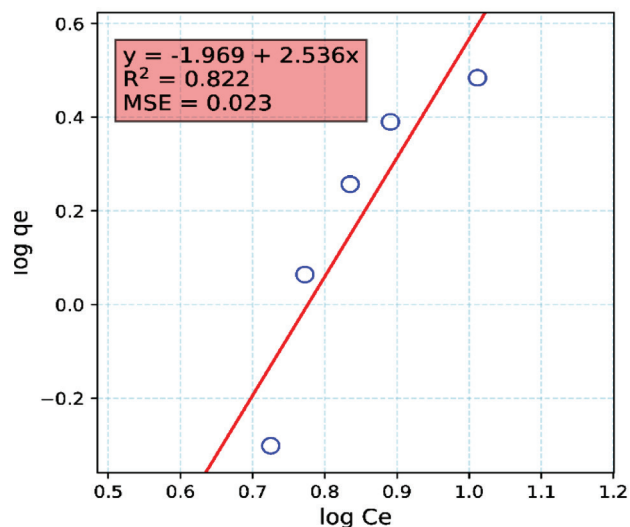
3.1.3 Analysis of Variance (ANOVA)

The Mann-Whitney and Friedman two-way ANOVA tests, which are non-parametric, were carried out on the data sets. First, the Mann-Whitney test was performed for the two adsorbents (ACC and BC) with respect to the adsorption rates, dosage, contact time and varied initial concentration, which indicate “not-significant” in six (6) cases.

Second, the Friedman two-way ANOVA test was conducted for the six (6) mentioned cases, and the summary results are as shown in Table 3.

3.1.4 Adsorbate- Adsorbent Modelling

The adsorbate-adsorbent concentrations were represented using the Langmuir, Freundlich, and Temkin isotherm models. For the Langmuir isotherm, Figures 4 to 7 show the extent of the adsorption of the lead and nickel and the concentration of adsorbate. The models developed relate the extent of adsorption of the lead and nickel, and the concentration of the adsorbate using the Freundlich adsorption Isotherm are presented in Figures 8 to 11. For the Temkin Adsorption isotherm the models developed relating to the extent of the adsorption of the lead and nickel and the concentration of adsorbate are presented in Figures 12 to 15.

Fig. 6 C_e versus C_e/q_e for BC adsorbent for Ni adsorption (Langmuir)Fig. 7 C_e versus C_e/q_e for ACC adsorbent for Ni adsorption (Langmuir)Fig. 8 $\log q_e$ versus $\log C_e$ for BC adsorbent for Pb adsorption (Freundlich)Fig. 9 $\log q_e$ versus $\log C_e$ for ACC adsorbent for Pb adsorption (Freundlich)Fig. 10 $\log q_e$ versus $\log C_e$ for BC adsorbent for Ni adsorption (Freundlich)Fig. 11 $\log q_e$ versus $\log C_e$ for ACC adsorbent for Ni adsorption (Freundlich)

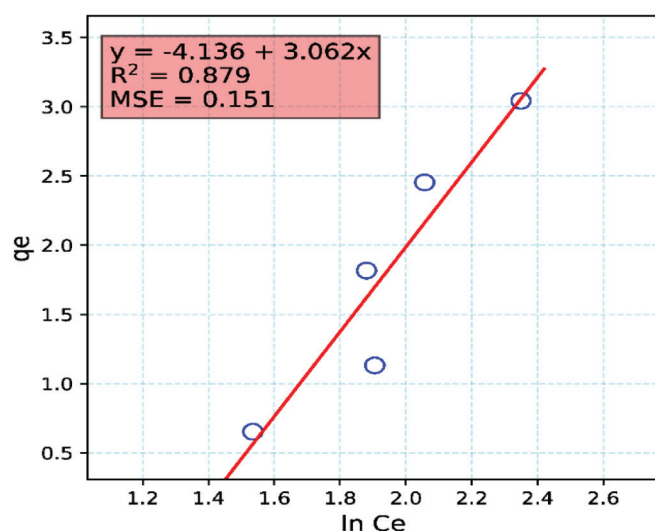


Fig. 12 q_e versus $\ln C_e$ for BC adsorbent for Pb adsorption (Temkin's model)

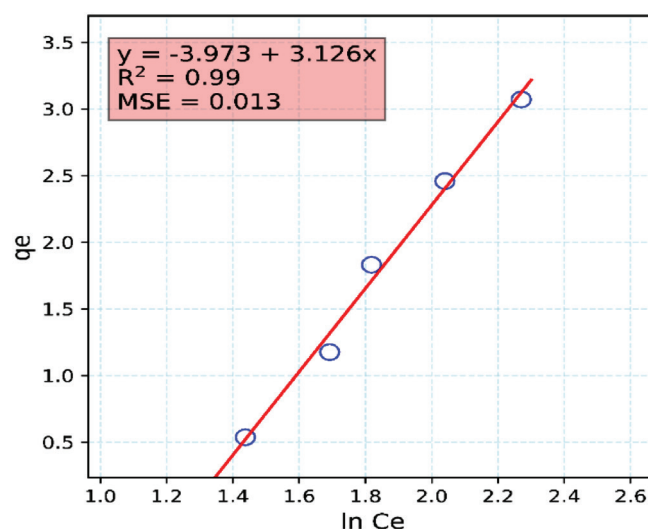


Fig. 13 q_e versus $\ln C_e$ for ACC adsorbent for Pb adsorption (Temkin)

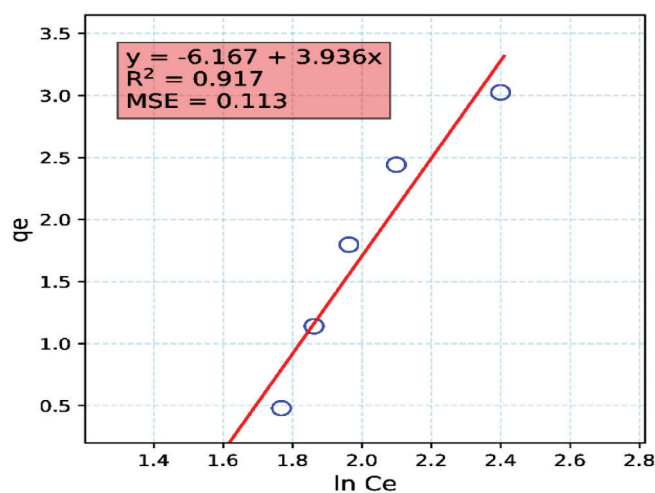


Fig. 14 q_e versus $\ln C_e$ for BC adsorbent for Ni adsorption (Temkin)

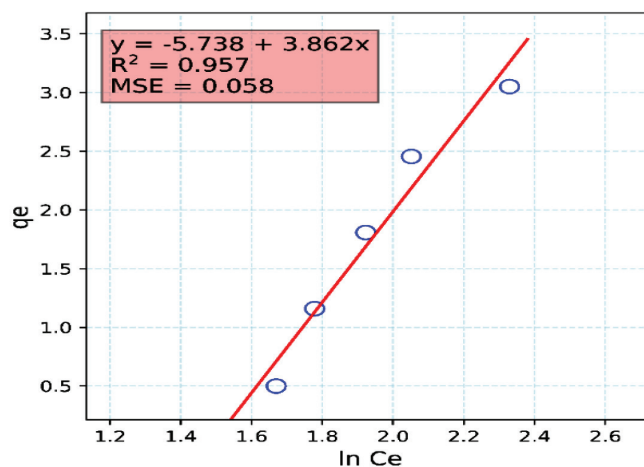


Fig. 15 q_e versus $\ln C_e$ for ACC adsorbent for Ni adsorption (Temkin)

Tab. 4 Summary of the Model Constants of the Adsorption Isotherms

Adsorbents	Adsorbate	Adsorption Isotherm Models		
		Langmuir	Freundlich	Temkin
BC	Pb (II) ion	$q_o = -1.650$ $k = -0.067$ $R^2 = 0.544$ $MSE = 1.879$	$k_f = 0.036$ $n = 0.509$ $R^2 = 0.864$ $MSE = 0.013$	$B_1 = 3.126$ $k_T = 0.281$ $R^2 = 0.99$ $MSE = 0.013$
	Ni (II) ion	$q_o = -1.353$ $k = -0.079$ $R^2 = 0.607$ $MSE = 2.113$	$k_f = 0.033$ $n = 0.481$ $R^2 = 0.928$ $MSE = 0.0087$	$B_1 = 3.062$ $k_T = 0.259$ $R^2 = 0.879$ $MSE = 0.151$
ACC	Pb (II) ion	$q_o = -0.881$ $k = -0.078$ $R^2 = 0.388$ $MSE = 11.199$	$k_f = 0.008$ $n = 0.288$ $R^2 = 0.756$ $MSE = 0.033$	$B_1 = 3.862$ $k_T = 0.226$ $R^2 = 0.957$ $MSE = 0.058$
	Ni (II) ion	$q_o = -0.917$ $k = -0.083$ $R^2 = 0.460$ $MSE = 7.029$	$k_f = 0.011$ $n = 0.394$ $R^2 = 0.822$ $MSE = 0.023$	$B_1 = 3.936$ $k_T = 0.209$ $R^2 = 0.917$ $MSE = 0.113$

A summary tableau of the isotherm models used for the data fitting with their respective calibrated constants is as shown in Table 4. The graphic abstract is presented in Figure 16.

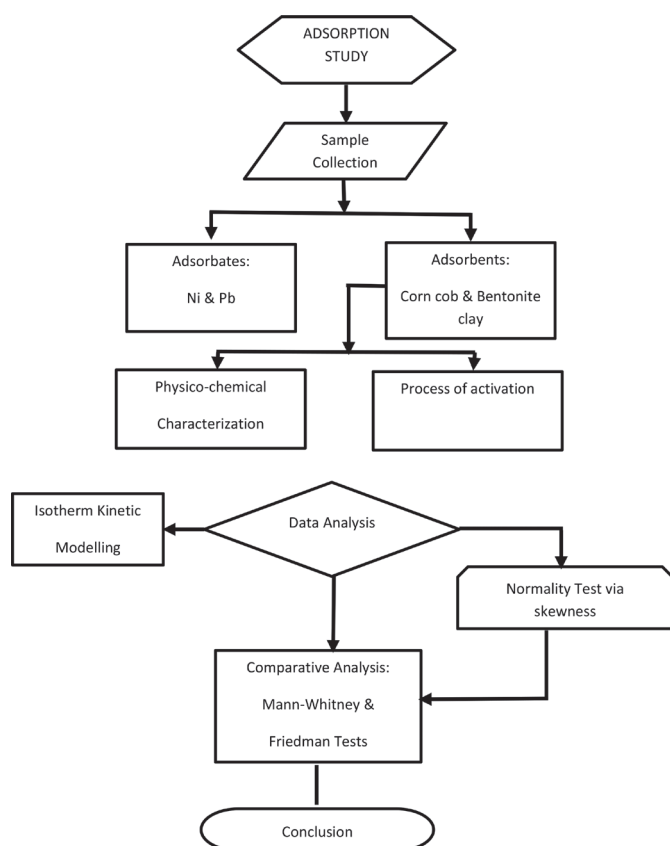


Fig. 16 Graphic abstract for the process

3.2 Discussion

3.1.1 Characterization of the Adsorbents and Adsorption experiments

From Table 1 it can be seen that while the BC had a higher moisture content of 36.45% versus 12.10% for the ACC (Nwosu et al., 2018), the ACC presented a higher surface area of 199 m² versus the 135 m² presented by BC. Figure 1 showed that as the adsorbent dosage increased, the percentage of Pb(II) and Ni(II) ions removed also increased for both the BC and ACC adsorbents. This observation is in agreement with that of Bartczak et al. (2015). Figure 2 shows that as the contact time increased, so did the percentage of Pb (II) and Ni (II) removal. It also indicated a strong linear relationship between the contact time and percentage of the adsorption of the Pb (II) and Ni (II) ions. Figure 3 shows that as the initial concentration increased, so did the percentage removal of the Pb (II) and Ni (II). The same effect was observed by Marshall and Champagne (1995).

2.1.2 Normality Test

Normality tests using the coefficient of skewness were carried out on the data sets to determine if the data sets were normally distributed. Most normality tests have little power to reject the null hypothesis, which states that the samples are from a normally distributed population when the sample size is relatively small (Oztuna et al., 2006). Table 2 shows that the coefficients of skewness of the ACC and BC were not zero, thereby indicating that the data sets are not symmetrical. They are therefore not normally distributed.

2.1.3 Analysis of Variance

The six (6) repeated tests (Table 3) according to Mann-Whitney indicated “non-significant” as computed p values ranged from 0.5309

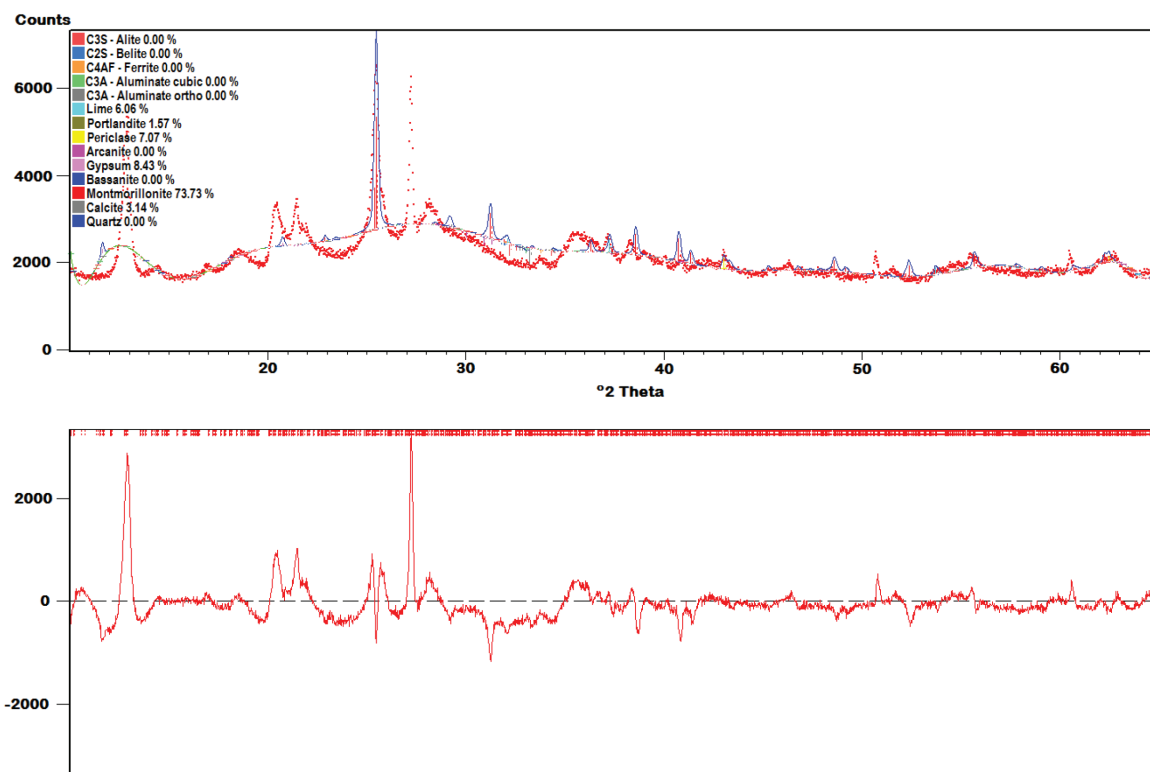


Fig. 17 X – ray diffraction spectrum of the bentonite (Opafola, et al. 2021)

to 0.6761 against an alpha value of 0.05. However, the Friedman two - way Anova test for the six (6) cases (Table 3) recorded p values of 0.0253 against the alpha value of 0.05, which indicates “significance”. Comparatively, as per the Mann-Whitney test for which the input data sets do not include the dosage, the Friedman results indicate “significance” with respect to the varied dosages, initial concentrations, and contact time. The effect of the adsorbent is not significant.

2.1.4 Modelling the Adsorbate Adsorbent

The linear models developed were compared with the Langmuir adsorption model, and the constant terms of the Langmuir adsorption model were obtained by directly comparing the two equations for each of the graphs. Figures 4 and 5 indicate that the coefficients of determination of the model developed were 0.544 and 0.607, while the mean squared errors were 1.879 and 2.113, respectively, for the lead adsorption, while Figures 6 and 7 show the coefficients of determination as 0.388 and 0.46 with the mean squared errors as 11.199 and 7.029, respectively, for the nickel adsorption.

Figures 8 and 9 show that the coefficients of determination of the model developed (the Freundlich isotherm model) were 0.864 and 0.928, while the mean squared errors were 0.013 and 0.0087, respectively, for the lead adsorption. Figures 10 and 11 indicate the coefficients of determination as 0.756 and 0.822 with the mean squared errors as 0.033 and 0.023, respectively, for the nickel adsorption.

Figures 12 and 13 show the coefficients of determination of the Temkin model developed were 0.99 and 0.879, while the mean squared errors were 0.013 and 0.151, respectively, for the lead adsorption. Figures 14 and 15 indicate the coefficients of determination were 0.957 and 0.917 with mean squared errors as 0.058 and 0.113, respectively, for the nickel adsorption.

The results in Table 5 detailing the coefficient of determination of the isotherms used show that the Temkin isotherm model best described the adsorption of both adsorbates (Demir et al., 2019).

2.1.5 X-ray microanalysis

The X-ray microanalysis for the bentonite clay is shown in Figure 17. The results revealed that the dominant mineral in bentonite is montmorillonite with a 72.73 wt %.

4 CONCLUSION

The present study shows that chemically-treated corn cob and thermally-activated bentonite clay are effective adsorbents for the removal of lead and nickel ions from aqueous solutions. The adsorption process is a function of the adsorbent and adsorbent concentrations, dosages, and time. The equilibrium adsorption data are satisfactorily fitted in the empirical models of Langmuir <Freundlich < Temkin, in the case of both the lead and nickel ions. Both adsorbents were found to be very effective in the removal of heavy metals from raw water with about 82 % removal achieved. The economic feasibility of these low - cost adsorbents over commercially- activated carbon (CAC) is another important aspect of the present study. It was found that both corn cobs and bentonite clay are abundant locally and are much cheaper than CAC. The results are not only important for the local industries, but also for developing nations due to the resultant social and environmental benefits (they keep the environment clean from corn cob litter, etc.).

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