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Iron (III) Contaminant Removal from Aqueous Solution of Iron Trioxonitrate (III) using Chicken Eggshell Adsorbent

OBIANYO, J. I. [1], ***IJE, I.*** [1], ***EBOH, S.*** [1], ***IKPEMO, O. C.*** [1], ***OHAZURIKE, E. E.*** [1], ***NWOBIA, L. I.*** [1], ***OBIANYO, L. I.*** [1]

[1] *Department of Civil Engineering, Michael Okpara University of Agriculture Umudike, Nigeria*

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ABSTRACT

Iron (III) contaminant removal from aqueous solution of iron trioxonitrate (III) using chicken eggshell adsorbent was investigated in this study. The objective was to assess the efficacy of chicken eggshell adsorbent in treatment of wastewater. Standard methods were in conducting laboratory experiments. Results indicated 85.58% carbon content and particle size range 0.075 – 4.75 mm, characteristics of good adsorbents. Maximum removal of 91.16% was observed at 1.0 g dosage of adsorbent. Optimum adsorption of Fe^{3+} occurred at slightly acidic pH of 6 corresponding to 91.157% removal. Contaminant removal decreases with increase in initial metal concentrations from 10 – 40 mg/l corresponding to 91.16 –76.77%, while between 20 - 60℃ optimum removal of 92.58% was observed at 40 ℃. Adsorption increased with increase in contact time between 67.84 –91.16% and equilibrium was attained at 90 minutes. The adsorption process best fits the Redlich-Peterson isotherm with highest R^2- value of 0.9996, with the least errors in SSE, SAE and ARE of 0.00988, 0.15171 and 0.74788 respectively. The pseudo second-order kinetics best described the equilibrium adsorption and FTIR spectra for loaded and unloaded cases indicate that adsorption has taken place. It is concluded that eggshell is a good adsorbent. However, more research are recommended.

1. INTRODUCTION

The deterioration of the environments is no doubt a concern to every environmental stakeholder, and this is tremendously increasing at alarming rate due to the quest for development and sustenance of the economy. In the study of economic and environmental problems, it is reported that removal of toxic metal ions and recovery of useful ions from soil, industrial wastewaters, groundwater, and marine environment is important (Chairgulprasert et al., 2013). Heavy metals in water bodies are extremely toxic in the body when consumed by man and include zinc, lead, arsenic, copper, mercury, and cadmium (Rao et al., 2013). Toxicity of heavy metals and their conservative nature of non-biodegradability make them accumulate in the food web causing serious damage to living organisms (Tirumalaraju et al., 2013). Different

*Corresponding author, e-mail:author@fupre.edu.ng
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methods are available for removal of these metals from water bodies and they could be expensive or ineffective particularly at low concentrations below 10 mg/l (Amer et al., 2010). Adsorption using biomass for treatment of wastewater is common in recent times because of their effectiveness in removal of heavy metal ions and pollutants (Osobamiro and Adewuyi, 2012). Biosorbents are cheap and are sourced from by-products associated with biomaterial production (El-Sayed et al., 2011). Studies have shown that many agricultural waste materials are effective in adsorption of heavy metals from wastewater and include, spent leaves of green and black tea (Zuorro et al., 2010), bael tree leaf (Kumar and Gayathri, 2009), orange peel (Gonen and Serin, 2012), rice husk (Khan et al., 2004), banana fibre (Bakiya et al., 2012), palm shell (Kushwaha et al., 2008), and coconut husk (Abdulrasaq et al., 2010).

2. MATERIALS AND METHOD

2.1. Preparation of Eggshell Adsorbent

Chicken eggshells were sourced locally from restaurants and were thoroughly washed to remove dirt, dust and other particulates. The samples were rinsed with distilled water, subsequently oven-dried at 105℃ for a period of 3 hours when a constant weight was obtained after two successive weighing. The samples were ground with blender prior to sieving using sieve mesh sizes 4.750 mm, 2.000 mm, 0.850 mm, 0.425 mm, 0.250 mm, 0.150 mm, 0.075 mm respectively, and experiments performed in accordance with BS 1377 (1975).

2.2. Preparation of Stock Solution

To prepare the stock solution for this study, 5.1 g of iron(III) nitrate [$Fe(NO_3)_3$] was dissolved in 1000 ml distilled water. To obtain the solutions for the experiment, various concentrations of 10 mg/l, 20 mg/l, 30 mg/l and 40 mg/l were achieved by appropriate dilutions of the stock solution. The various pH of solution for experiment was adjusted using 0.1 M solution of HCl and 0.1 M solution of NaOH respectively.

2.3. Adsorption Experiments

Adsorption experiments were carried out in order to investigate the effect of pH, initial concentration, adsorbent dosage, temperature and contact time on the adsorption of Fe^{2+} on eggshell adsorbent. Experiments were conducted at varying temperatures 28, 40, 50 and 60℃, initial concentrations were 10, 20, 30 and 40 mg/l respectively. Adsorbent dosages were 0.25, 0.5, 0.75 and 1.00 g, while pH values were 3.5, 6, 8 and 9. Citizen® electronic balance (Model MP-5000A) was used in weighing the adsorbent. Solid phase was separated from the solution after each experiment using Whitman filter paper No. 1, while the residual metal concentrations contained in the supernatant was measured using atomic absorption spectrophotometer (model: nova® 400P). The morphology of the eggshell adsorbent before and after the adsorption was analysed with the aid of scanning electron microscopy (SEM, FEI ESEM Quanta 200). Series of adsorption analysis were carried out in batches using the One-Factor-At-A-Time (OFAT) experimental approach to investigate the effects of adsorbent dosages, concentration of contaminants, contact time, pH and temperature on adsorption capacity using standard methods.

The amount of Fe^{2+} sorbed at any time t, q_t (mg/g) was computed from the relationship;

$$q_t = \frac{(C_i - C_f)V}{m} \quad (1)$$

The percentage of the adsorbed contaminant by the eggshell was calculated using the expression;

$$\text{Adsorbed metal } (\%) = \frac{C_i - C_f}{C_i} \times 100 \quad (2)$$

Where c_i and c_f (mg/mL) are the initial and final metal ion concentrations in the filtrate, V (mL) is the volume of the solution, w (g) is the mass of eggshell adsorbent used in the study.

2.4 Error Analysis

The best isotherm model that represents the experimental data was determined through analysis using three different error functions, which include the sum of squared errors (SSE), sum of absolute errors (SAE) and average relative error (ARE). The sum of squared errors is given as:

$$SSE = \sum_{i=1}^{n} (Q_{e.C} - Q_{e.M})^2 \quad (3)$$

Similarly, the sum of absolute errors is given as:

$$SAE = \sum_{I=1}^{p} [Q_{e.M} - Q_{e.C}] \quad (4)$$

The average relative error is given as:

$$ARE = \frac{100}{n} \sum_{i=1}^{n} \left[\frac{Q_{e.C} - Q_{e.M}}{Q_{e.M}}\right] \quad (5)$$

where $Q_{e.C}$ is the theoretical concentration of adsorbate on the adsorbent, which have been calculated from one of the isotherm models, while $Q_{e.M}$ is the experimentally measured adsorbed solid phase concentration of the adsorbate adsorbed on the adsorbent.

3. RESULTS AND DISCUSSION

Chemical composition of eggshell adsorbent is presented in figure 1. The eggshell adsorbent contains one important element (carbon), a very useful element possessed by good adsorbents. Based on the chemical composition of the adsorbent, carbon (C) has the highest percentage composition of 85.58%, followed by zinc oxide (ZnO) with 9.07%. Insignificant quantities of SiO_2, Al_2O_3, Fe_2O_3, MgO, Na_2O, K_2O, P_2O_5, SO_3 and ZnO in the neighbourhood of 0.52%, 0.38%, 1.56%, 1.27%, 0.13%, 0.08%, 1.16%, 0.25% and 9.07% were also evident. The chemical composition of the adsorbent recorded in this study is similar to findings from previous researchers in which carbon ranged between 70.62% and 86.74% (Ajala *et al*., 2018; El-kemary *et al*., 2018; Yusuff, 2019).

Figure 2 is the result of the particle size distribution of adsorbent. It ranged between 0.075 mm to 4.750 mm, a good prospect for adsorption as smaller particle sizes increase surface area and hence better adsorption. However, particle size below 600 μm was the optimum size for adsorption of heavy metal ions on unmodified fluted pumpkin adsorbent, while between 600 μm and 1000 μm was observed for adsorption of heavy metals on the modified adsorbent (Eze *et al*., 2013).

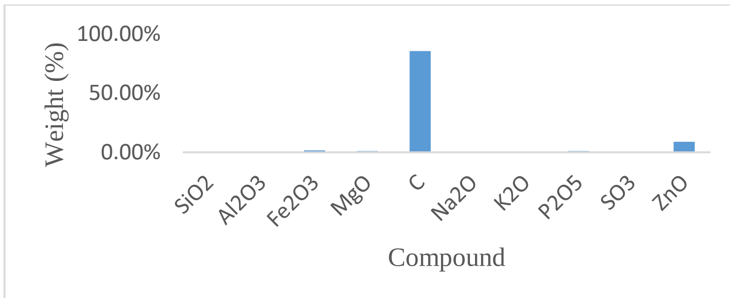


Figure 1: Chemical composition of eggshell adsorbent

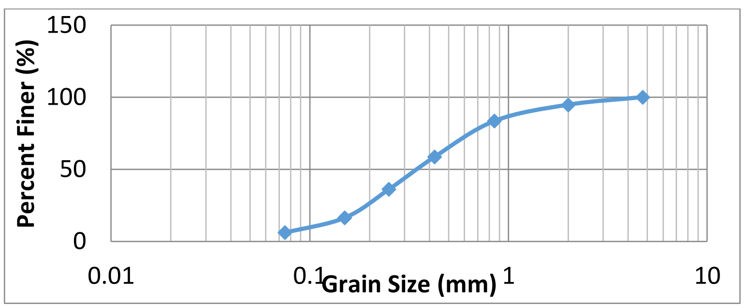
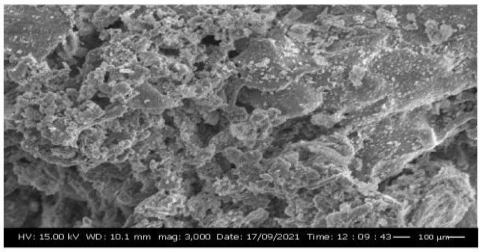


Figure 2: Particle size distribution of crushed eggshell

From the scan image in Figure 3a, microspores of different sizes are evident, which enhanced the adsorption of Fe^{2+}. However, the pores appear clogged after adsorption as shown in figure 3b, which is instrumental to the decrease in adsorption with time because of decrease in surface area of adsorption sites. However, several studies on morphology of eggshell adsorbent also revealed that the porosity of eggshell after adsorption process was reduced by the adsorbed contaminants in the solution (Abd Elhafez *et al*., 2017; Borhade and Kale, 2017).



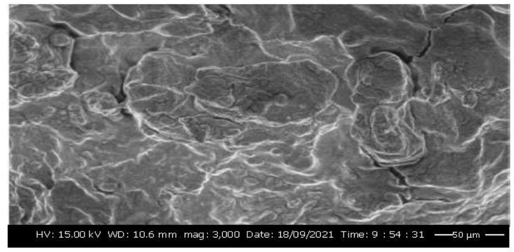


Figure 3a: Morphology of eggshell before **Figure 3b:** Morphology of eggshell

after adsorption adsorption

Effect of dosage on adsorption of Iron (III) ion

In figure 3c, increase in adsorbent dosage also increased the percentage of Fe^{3+} removed. Iron (Fe) ion removed from the aqueous solution by the adsorbent at dosages 0.25 g, 0.50 g, 0.75 g and 1.0 g ranged from 82.15 –91.16% in which optimal dosage for removal was 1.0 g. Agarwal and Gupta (2014), reported optimal removal of 92.62% of Fe ions from aqueous solution by eggshell adsorbent at dosage of 1.5 g.

Effect of pH on adsorption of Iron (III) ion

Effect of pH was investigated by varying the solution pH at 3.5, 6.0, 8.0 and 9.0 at constant of 10mg/L initial concentration of the heavy metals, 1.0g eggshell dosage and at room temperature (28 ºC) for contact time of 60 minutes. Optimum adsorption of Fe^{3+} occurred at slightly acidic pH of 6 corresponding to 91.157% removal since insignificant adsorption was observed at pH 8 and pH 9. This result agree with the work of Mashangwa *et al*. (2017). Further, in solution with low pH, the functional groups are dominated by positively charged protons on the eggshell surface, which reduces the adsorption capacity as a result of repulsion of like charges (Rao and Prabhakar, 2011)

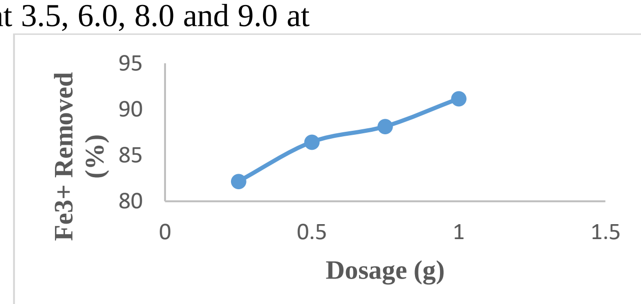


Figure 3c: Effect of dosage on removal of Fe^{3+}

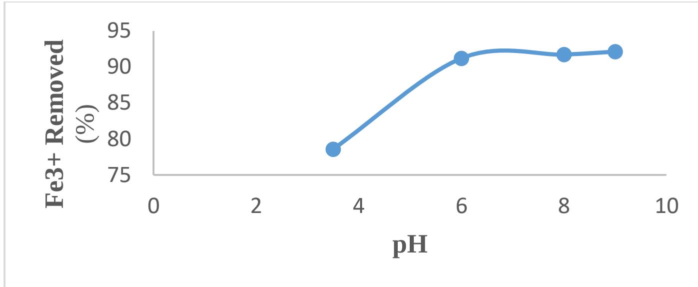


Figure 4: Variation of Fe^{3+} removed with pH

Effect of initial metal concentration on adsorption of Iron (III) ion

Figure 5 show the variation of Fe^{3+} removed with initial metal concentrations. The plot indicate that adsorption decreased with increase in metal concentrations. This is evident in figures 3a and 3b which presented the scanning electron micrograph (SEM) of eggshell adsorbent before and after adsorption. It showed that the pore spaces in the adsorbent became clogged with increase in metal concentrations, and consequently gave rise to the decreasing trend in adsorption as shown in figure 5 below. However, the percentage of Fe^{3+} adsorbed by the eggshell adsorbent at initial concentrations of 10 – 40 mg/L, ranged from 91.16 –76.77%. Various adsorption studies have also demonstrated that increase in initial concentration of metals leads to decrease in the percentage of heavy metals adsorbed (Ademiluyi and Ujile, 2013; Bamukaye and Wanasolo, 2017). Contrary to this finding, other studies have reported increase in percentage removal of chromium ion when the initial concentration was increased for neem and rice husk adsorbents (Kumar and Shrivastava, 2015) and ginger adsorbent (Sharma et al., 2016). Also, De Angelis et al. (2017) observed that increasing the initial concentration of nickel ion from 50 – 1000mg/L also led to increased amount of nickel ion adsorbed until the equilibrium was achieved.

Effect of temperature on adsorption of Iron (III) ion

Figure 6 is the plot of relationship between Fe^{3+} adsorption with temperature. Adsorption increased by 1.425% from temperature of 28℃ to optimum value of 40℃ and started dereasing. A decrease of 4.338% and 3.234% was observed between temperatures 40℃ and 50℃, and between temperatures 50℃ and 60℃, indicating the insignificant effect of temperature on adsorption. However, between 20 - 60℃ optimum removal of 92.58% was observed at 40 ℃. Similar results were also reported for adsorption of dye onto eggshell (Ciobanu *et al.*, 2016) and adsorption of Cu^{2+} by rice husk adsorbent (Abd Elhafez *et al.*, 2017). These studies also concluded that reduction in pollutant removal was as a result of the exothermic nature of the adsorption process

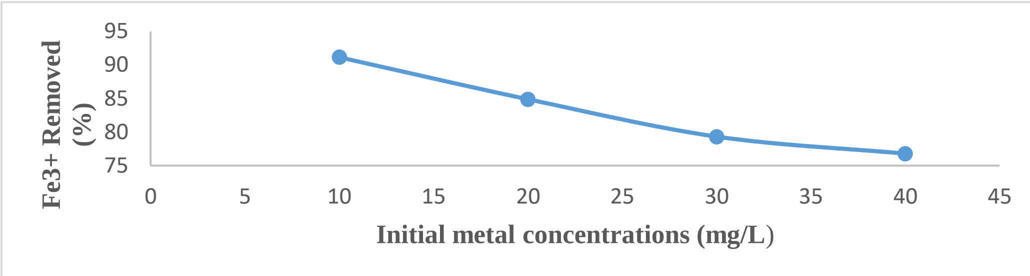


Figure 5: Variation of Fe^{3+} removed with initial metal concentrations

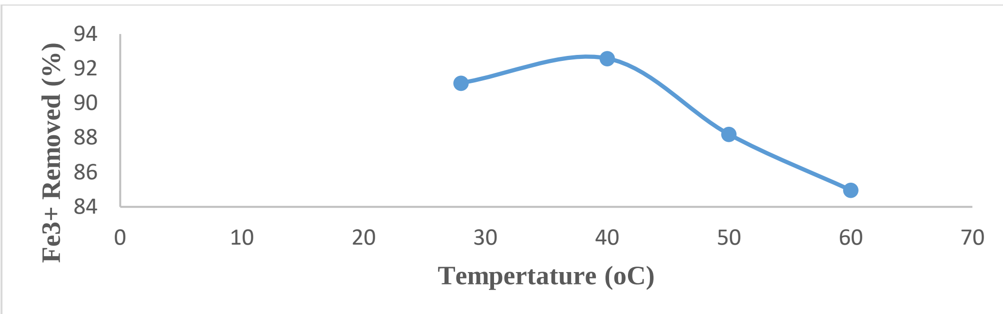


Figure 6: Relationship between Fe^{3+} adsorption with temperature

Effect of contact time on adsorption of Iron (III) ion

In figure 7, effect of contact time was studied at contact times of 30, 60, 90 and 120 minutes. Other parameters were kept constant at 1.0g adsorbent dosage, 10mg/L initial concentration of Fe^{3+} and solution of pH 6.0 at room temperature of 28 ºC. Figure 6 indicate that adsorption increased with increase in contact time between 67.84 –91.16% and equilibrium was attained at 90 minutes. Similar result was obtained by (Ipeaiyeda and Tesi, 2014)

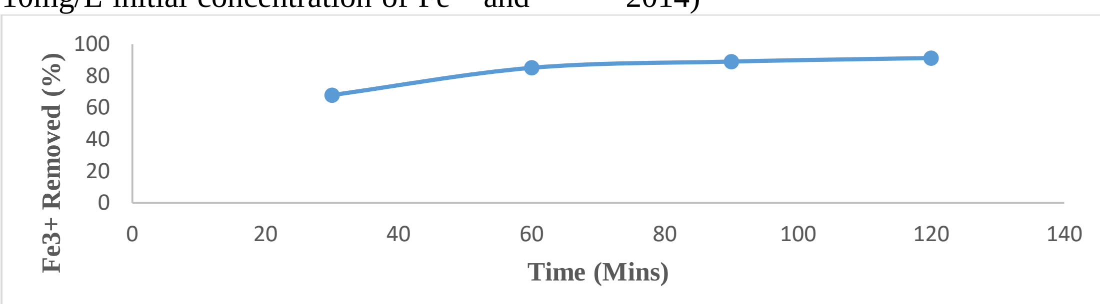


Figure 7: Percent Fe^{3+} removal with contact time

Best Fit Model for the Adsorption Isotherms

Figures 8a to 8d present the Langmuir, Redlich-Peterson, Dubinin-Radushkevich and Temkin adsorption isotherms for removal of Fe^{3+} from aqueous solution. The R^2- values for these isotherms were found to be 0.9696, 0.9996, 0.8714 and 0.9660, these results are evident that adsorption process best fits the Redlich-Peterson isotherm with highest R^2- value of 0.9996.

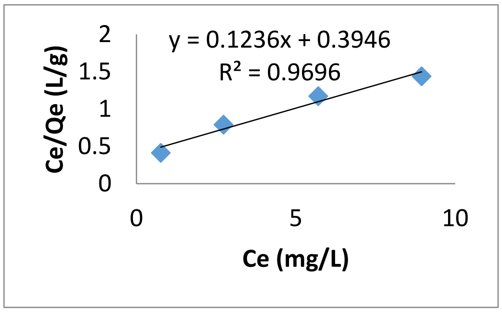


Figure 8a: Langmuir isotherm plot for

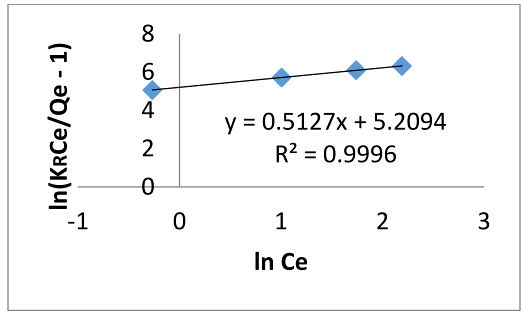


Figure 8b: Redlich–Peterson Isotherm plot for Fe^{3+} adsorption

Fe^{3+} adsorption

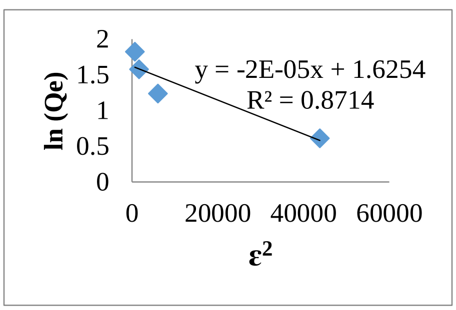


Figure 8c: Dubinin-Radushkevich Isotherm plot for Fe^{3+} adsorption

for

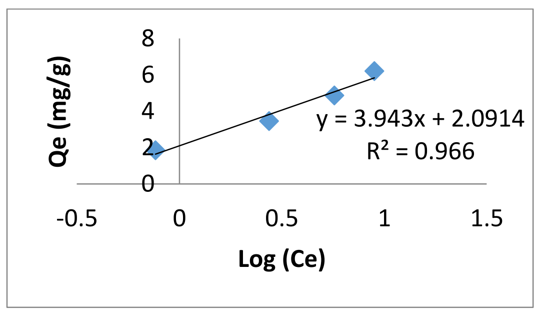
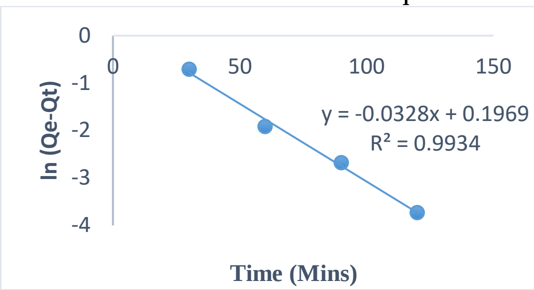


Figure 8d: Temkin Isotherm plot for Fe^{3+} adsorption

Kinetics of Iron (III) Adsorption on Eggshell adsorbent

The kinetics of iron (III) ions adsorption was studied to know the best fit kinetic model that describes the adsorption process. Figure 8a shows the linear plot of pseudo first order kinetics, while figure 8b is the linear plot of pseudo second order kinetics for Fe^{3+}. The first order rate constant, k_1 was obtained as 0.0396 min^{-1}, while the equilibrium adsorption capacity, Q_e was predicted to be 1.2176 mg/g. The second order rate constant, k_2 was 0.4881 min^{-1}, while the equilibrium adsorption capacity, Q_e was predicted to be 2.0488 mg/g and R^2-values for both are 0.9934 and 0.9988 respectively. From these results, the pseudo second order kinetics best described the equilibrium adsorption. However, previous studies by Dagde and Daka (2019) and Chie-Amadi *et al*. (2020) reported that adsorption process follow the second order kinetic.



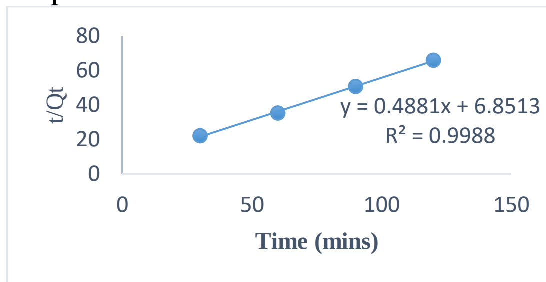


Figure 8a: Pseudo first order kinetic for adsorption of Iron (III) ion

Figure 8b: Pseudo second order kinetic for adsorption of Iron (III) ion

Table 1 presents results from the error analysis. Error values for SSE, SAE and ARE are least in Redlich-Peterson isotherm for adsorption of iron (III) ion and highest in Temkin isotherm. The order is such as Temkin > Dubinin-Radushkevich > Langmuir > Redlich-Peterson. This results contradicts the study conducted by Baybars (2023) in which such order as Sips > Langmuir > Khan > Toth > Temkin > Freundlich was obtained, so that Langmuir isotherm has a higher error than Temkin isotherm. The studies affirm that outcomes differ according to study samples because eggshell adsorbent was used in this study for adsorption of iron (III) ion from its aqueous solution, while Baybars (2023) used montmorillonite adsorbent and basic orange 2 dye as adsorbate.

Table 1: Result from error analyses for adsorption of iron (III) ion from aqueous solution

Error Type	Langmuir	R-P	D-R	Temkin
SSE	2.32501	0.00988	2.65377	2.79908
SAE	2.6516	0.15171	2.62535	2.97224
ARE	15.2258	0.74788	16.9563	26.3894

Figure 9a is the FTIR spectrum of transmittance as a function of wave number for unloaded eggshell adsorbent. Wave bands 3857.76 cm^{-1}, 3726.60 cm^{-1}, 3686.43 cm^{-1}, 3417.92 cm^{-1}, 3232.20 cm^{-1} show the presence of single bond amine N-H, and carboxylic acid O-H, stretches respectively. Band 3109.36 cm^{-1} belong to single bond C-H stretching vibration of both alkyl and aromatic groups, while band 2847 cm^{-1} identify with the alkanes C-H stretching vibration. Wave bands 2786 cm^{-1} and 2685 cm^{-1} are indications of O=C-H stretch of the aldehydes, band 2337 cm^{-1} is of the amino-related component NH and band 2114 cm^{-1} is the terminal alkyne, C≡C stretch. Wave band 1820 cm^{-1} is in the category of transition metal carbonyl stretch and wave band 1712 cm^{-1} is for carbonyl stretching vibration C=O, of either α or β-unsaturated aldehydes and ketones in the region of 1730-1735 cm^{-1}, with characteristic high intensity particularly useful for diagnostic purposes. Band 1512 cm^{-1} is for N-O asymmetric stretch and 1373 cm^{-1} belong to the O-H stretch, while bands 1213 cm^{-1} and 1103 cm^{-1} identify with the C-O stretches in the region 1300-1000 cm^{-1}. Bands 896 cm^{-1} and 794 cm^{-1} respectively, belong to strong broad band due to N-H wag usually observed only for primary and secondary amines. These are the functional groups present in the unloaded eggshell adsorbent before adsorption of Iron (II) ion started taking place.

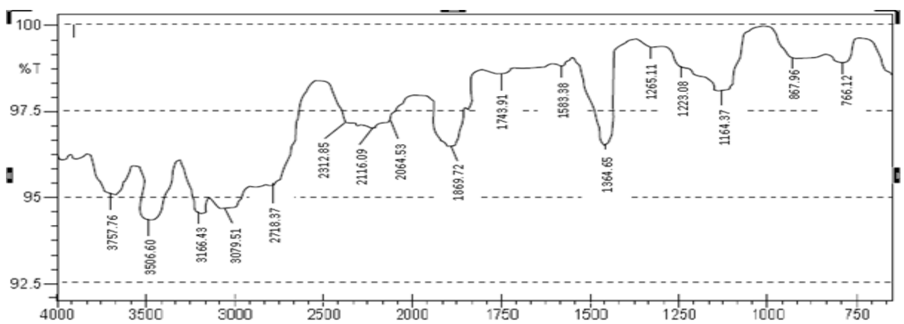


Figure 9a: FTIR spectra of eggshell adsorbent before adsorption

Figure 9b is the FTIR spectra after adsorption. Some old bands disappeared while new bands appeared which is a strong indication that adsorption has taken place. The disappearance of these bands could be as a result of some unknown reactions taking place in the system. However, the wave bands indicated after adsorption are 3471 cm^{-1} and 3394 cm^{-1}, the aliphatic N-H stretching, primary amine of medium intensity. The alkane family, C-H stretching vibration with medium intensity of bands 2876 cm^{-1} and 2501 cm^{-1} respectively, and the carbon dioxide stretching, O=C=O of band 2324 cm^{-1} of strong intensity.

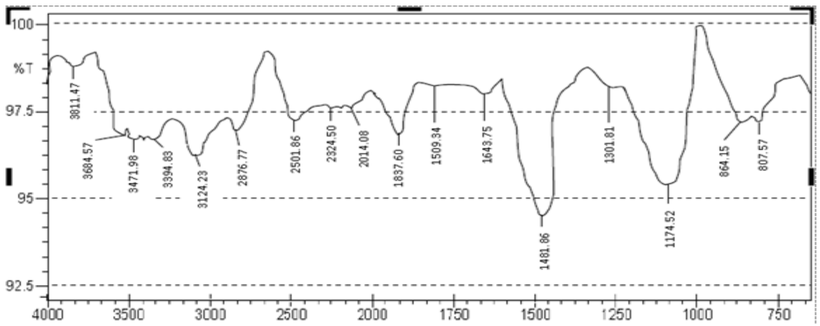


Figure 9b: FTIR spectra of eggshell adsorbent after adsorption

4. CONCLUSION

Adsorption of iron (III) iron from aqueous solution of iron trioxonitrate (III) was studied,
research findings allow for the following deductions;

- That chicken eggshell is very effective as an adsorbent in removal heavy metal contaminants from wastewater
- All the reactions obey the pseudo second order kinetic
- Results from adsorption studies vary according to samples used. This implies that the isotherms may have the same behavior or closely related behavior if the same adsorbent and adsorbate are used for study. On the other hand, usually isotherms behave differently when the adsorption studies are conducted with different adsorbents and adsorbates.

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