



Equilibrium and Kinetic Studies of the Adsorption of Pb²⁺ and Ni²⁺ Ions from Aqueous Solution by the Use of Activated Locust Bean Husk (LBH)

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Authors' contributions

This work was carried out in collaboration between all authors. Author NO designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors IBA and EIA managed the analyses of the study and literature searches. All authors read and approved the final manuscript.

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ABSTRACT

The removal of Lead and Nickel ions from aqueous solution by adsorption onto activated locust bean husk was investigated in a well stirred batch reactor. Equilibrium and kinetic experiments were performed at various pH, temperature and contact time. The metal removal approached equilibrium within 70 min for lead and nickel. It was observed that removal of Pb²⁺ and Ni²⁺ by activated LBH had highest adsorption capacity at pH 6 and that the uptake of the selected heavy metal ions by activated LBH increases with increasing temperature. Kinetic studies carried out showed that the experimental adsorption capacities of Pb²⁺ and Ni²⁺ are 4.27 mg/g and 2.9 mg/g respectively. Their calculated adsorption capacities by using pseudo-first order model are 0.05 mg/g and 0.6 mg/g respectively, while that of pseudo- second order model are 4.48 mg/g and 3.57 mg/g respectively. The result obtained showed that the calculated adsorption capacities of all the metals by pseudo-second order model are closer to their respective experimentally determined adsorption capacities than those from pseudo- first order model. Hence, pseudo- second order adsorption model is more suitable to describe the adsorption kinetics of Pb²⁺ and Ni²⁺ by activated LBH.

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1. INTRODUCTION

The removal of heavy metals from aqueous solutions is an important issue faced by industries discharging wastewater containing heavy metals. Because of rapid industrialization, an alarming amount of toxic heavy metals has been released into the environment, endangering natural ecosystems and public health. Thousands of ions of lead are discharged from electric battery manufacturing, lead smelting, internal combustion engines fuelled with leaded petroleum, and mining activities [1]. Lead affects the human central nervous system, the blood pressure and the reproduction [2]. Acute nickel poisoning in human causes headache, nausea and vomiting, dizziness, chest pain, tightness of the chest, dry cough, shortness of breath and rapid respiration [3]. Some of the techniques which have been used in the removal of heavy metals from effluents include ion exchange, chemical precipitation, electrodialysis, electrolytic extraction, reverse osmosis, and cementation [4-6]. Compared with the aforementioned techniques, adsorption has proved to be less expensive alternative for the removal of metals from aqueous solution [7]. Literature has indicated the potential of some agricultural waste products as inexpensive sorbents [8-12]. Due to their low cost, after these materials have been expended, they can be disposed of without causing environmental degradation [13]. The aim of this research is to study the adsorption of selected heavy metals (Pb^{2+} and Ni^{2+}) from aqueous solutions by activated locust bean husk [14]. This aim will be achieved by determining some physicochemical properties of the adsorbent and examining the adsorption capacity of activated locust bean husk.

2. EXPERIMENTAL

2.1 Adsorption Experiment: Procedure

The experiment was carried out using the batch method as described in the literature [15]. 50 cm³ of each of the working solution was measured using standard flask into three 100 cm³ conical flasks, which gave triplicate determination for a given concentration. 0.2 g of the adsorbent was transferred into each of these conical flasks. Each of these mixtures was then transferred into separate polythene bottles where they were agitated (at the same time) with a basic

reciprocating shaker at 150 rpm for 2 hours and left undisturbed on the desk for 24 hours to allow the system to equilibrate. After 24 hours, the mixture was filtered through a whatman filter paper into another 120 cm³ polyethylene bottle. The first 5 cm³ of the filtrate was discarded. This allows the filter paper to be saturated with the solution. The concentration of the residual metal ion remaining in the solution was determined using AAS. The amount of the metal adsorbed was calculated using the equation:

$$q_e = \frac{V(C_o - C_e)}{1000m} \quad (1)$$

where q_e is the amount of adsorbate ion adsorbed in milligram per gram of the adsorbent, c_o is the initial concentration of the metal ion before the adsorption process, c_e is the equilibrium concentration of the metal ion in the filtrate after adsorption process, m is the mass in gram of the adsorbent and v is the volume of the solution in cm³. The percentage metal ion removal (%Rem) was calculated using the equation:

$$\% \text{ Rem} = \frac{(C_o - C_e)}{C_o} \times 100 \quad (2)$$

2.1.1 The effect of initial solution pH on Pb^{2+} and Ni^{2+} adsorption

The pH of solution is important in adsorption study, in that pH of a solution determines the speciation of the metal ion in solution and also dictates the surface charge of adsorbents with variable charge characteristics. Using constant concentration of 20 mg/dm³ of each solution, 0.2 g adsorbent dose and agitation time of 2 hours at ambient temperature, the pH of the solution was varied. The pH that was tested are: 2, 4, 6, 8 and 12. The procedure followed is as described in section 2.1 above.

2.1.2 Effect of temperature on Pb^{2+} and Ni^{2+} adsorption

The effect of temperature was studied by varying the temperature at which the reaction took place at specific concentration (20 mg/dm³), pH, adsorbent dose (0.2 g) and agitation time of 2 hours. The temperature used is: 30°C, 40°C, 50°C, and 60°C. The experiment was carried out in a temperature regulatory water bath. The procedure followed is as described in section 2.1 above.

2.1.3 The effect of contact time on Pb²⁺ and Ni²⁺ adsorption

The effect of contact time is necessary in order to determine the time required for maximum adsorption to take place. Data generated from this study would be used to describe the kinetics of the adsorption study. Using constant concentration of 20 mg/dm³, adsorbent dose of 0.2 g, the desired pH and at room temperature, the effect of contact time was studied by varying the agitation time. The following agitation time in minutes were used: 30, 50, 70, 90, and 110. The procedure followed is as described in section 2.1 above.

2.1.4 Adsorption kinetics

Kinetic tests show the time – concentration profile for adsorption. The adsorption reaction itself is inherently an extremely fast one. It is mainly the particle mass transfer which controls the overall adsorption kinetics (adsorbent particle size, porosity, sorbate size and mixing in the adsorption system). The adsorption kinetics constitutes a criterion in the determination of adsorption processes [16]. Kinetics studies are carried out by mixing an amount of adsorbent with a given concentration of metal solution, with varying contact time [17].

2.1.4.1 Pseudo - first order

The pseudo-first order equation is generally expressed as [18,19]

$$\frac{dq}{dt} = k_1(q_e - q_t) \quad (3)$$

Where q_e and q_t are the amount of adsorbate adsorbed on adsorbent at equilibrium (mg/g) and time t , respectively, and k_1 is the rate constant of pseudo-first order adsorption (min⁻¹). Integrating equation (3) for the boundary conditions $t = 0$ to $t = t$ is the following:

$$\log (q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \quad (4)$$

The plot of $\log (q_e - q_t)$ versus t will give a straight line and the value of k_1 can be obtained from the slope of the graph while q_e (calculated) is obtained from the intercept of the graph [20].

2.1.4.2 Pseudo - second order

The pseudo-second order kinetic model is expressed as:

$$\frac{dq}{dt} = k_2(q_e - q_t)^2 \quad (5)$$

Where k_2 is the pseudo-second order rate constant of adsorption (g mg⁻¹min⁻¹). The linearised integrated form of equation (5) is given as:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (6)$$

If the pseudo-second order kinetics is applicable to the system, then the plot of $\frac{t}{q_t}$ versus t will give a linear relationship with $\frac{1}{q_e}$ and $\frac{1}{k_2 q_e^2}$ as slope and intercept, respectively. The values of q_e and k_2 can be determined from the slope and intercept and there is no need to know any parameter beforehand. The pseudo-second order kinetics model has been successfully applied to several adsorption systems [19].

3. RESULTS AND DISCUSSION

3.1 Effect of Initial Solution pH on Pb²⁺ and Ni²⁺ Adsorption

The uptake and percentage removal of metals from the aqueous solution are strongly affected by the pH of the solution [21,22]. In order to demonstrate the effect of initial solution pH on adsorption capacity, uptake of each of Pb²⁺ and Ni²⁺ onto the activated LBH as a function of pH was studied in the pH range of 2 to 12. Table 1 presents the effects of initial solution pH on Pb²⁺ and Ni²⁺ adsorption.

Fig. 1 is a graphical representation of effects of initial solution pH on uptake of Pb²⁺ and Ni²⁺ respectively. The uptake of lead and nickel increased from 2.92 mg/g to 3.9 mg/g and 1.65 to 2.64 mg/g, respectively when the solution's pH was increased from 2 to 6. For pH values above 6, the adsorption capacity decreased with increment in pH. The mechanism of metals adsorption by the adsorbent with pH variation could be explained with the same concept as put forward by literature [23-25] and that in the pH range of 2 to 4, lead and nickel ions would be present predominantly as M²⁺ ions.

The minimum adsorption observed at low pH of 2 could be, on one hand due to the fact that the presence of higher concentration and higher mobility of H⁺ ions favoured H⁺ adsorption compared to M²⁺ ions and on the other hand due to the high solubility and ionization of metals salt

in the acidic medium. It would be plausible to suggest that at lower pH value, the surface of the adsorbent is surrounded by hydronium ions (H^+), thereby preventing metal ions (M^{2+} ions) from approaching the binding sites of the adsorbents. At higher H^+ concentration, the adsorbent surface becomes more positively charged such that the attraction between adsorbents and metal cations is reduced. In contrast, as the pH increases, more negatively charged surface becomes available thus facilitating greater metal removal. It could be deduced that the adsorption of the metal cations increased with increasing pH as the metal ionic species become less stable in the solution. The maximum adsorption observed in the pH range of 6 to 8 might be due to partial hydrolysis of metal ions, resulting in the formation of $M(OH)^{2+}$ which would be adsorbed to a greater extent on a less-polar carbon surface of the adsorbents compared to M^{2+} ions. Furthermore, the low solubility of hydrolyzed metal species may be another reason for maximum adsorption in this pH range.

3.2 Effect of Temperature on Pb^{2+} and Ni^{2+} Adsorption

Olayinka et al. [26] noted that metal uptake and removal efficiency is dependent on temperature. Table 2 presents equilibrium data on effect of temperature on adsorption of Pb^{2+} and Ni^{2+} . Fig. 2 represents effect of temperature on Pb^{2+} and Ni^{2+} adsorption respectively. It was observed that all the metals showed increase in adsorption capacity with increase in temperature. This could be due to increase in average kinetic energy of the metal ions in solutions containing the adsorbent which increases the number of metal ions interacting with the adsorbent surface by increasing the rate at which the metal ions hit the binding sites at the surface of the adsorbent thus increasing the adsorption capacities.

The uptake of lead and nickel increased from 3.71 mg/g (74.2%) to 3.9 mg/g (78%) and 2.79 mg/g (55.8%) to 3.1 mg/g (62%), respectively when the solution's temperature was increased from 30°C to 60°C.

Table 1. Effect of initial solution pH on Pb^{2+} and Ni^{2+} adsorption

Ph	Lead		Nickel	
	q_e (mg/g)	%Rem	q_e (mg/g)	%Rem
2	2.92	58.4	1.65	33.0
4	3.44	68.7	1.92	38.1
6	3.90	77.9	2.64	52.9
8	3.62	72.4	2.64	52.8
12	3.00	60.1	2.10	42.0

(Adsorbent Dose = 0.2 g, Initial Concentration = 20 mg/dm³, Temperature = 27°C)

(Key: q_e is the metal uptake in mg/g, i.e. the amount of metal ion adsorbed in milligram per gram of the activated LBH while % Rem is the removal efficiency i.e. percentage metal ion removed)

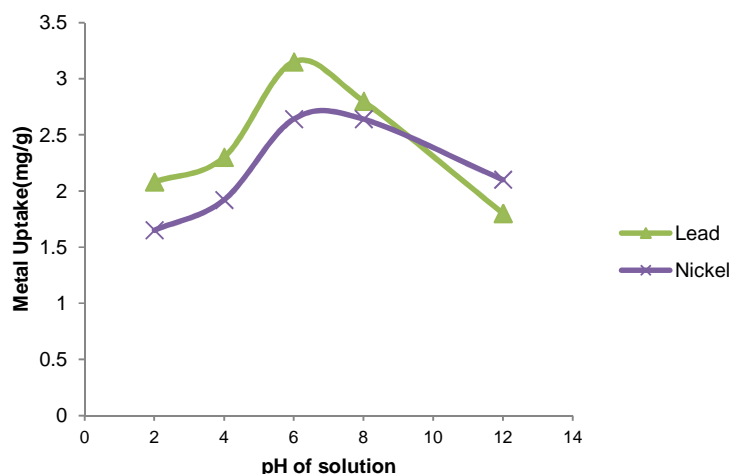


Fig. 1. Effect of initial solution pH on Pb^{2+} and Ni^{2+} adsorption

Table 2. Effect of temperature on Pb²⁺ and Ni²⁺ adsorption

Temp.(K)	Lead		Nickel	
	q _e (mg/g)	%Rem	q _e (mg/g)	%Rem
303	3.71	74.2	2.79	55.8
313	3.71	74.2	2.79	55.8
323	3.89	77.8	2.99	59.8
333	3.90	78.0	3.10	62.0

(Adsorbent Dose = 0.2 g, Initial concentration = 20 mg/dm³, Temperature = 27°C)(Key: q_e is the metal uptake in mg/g, i. e. the amount of metal ion adsorbed in milligram per gram of the activated LBH while % Rem is the removal efficiency. i. e percentage metal ion removed)

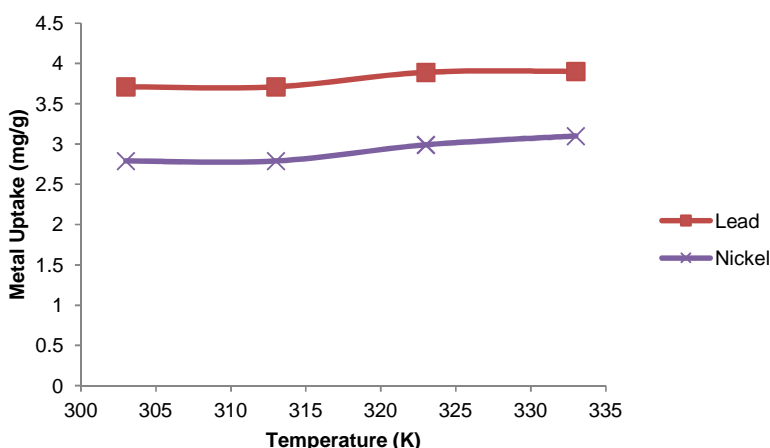


Fig. 2. Effect of temperature on Pb²⁺ and Ni²⁺ adsorption

3.3 Effect of Contact Time on Pb²⁺ and Ni²⁺ Adsorption

The effect of varying contact time (30, 50, 70, 90 and 110 min) on adsorption efficiency of an adsorbent for Pb²⁺ and Ni²⁺ was investigated by using a constant initial metal ion concentration of 20 mg/dm³ for the two metals. Table 3 shows the effect of contact time on the adsorption of Pb²⁺ and Ni²⁺. The relationship of adsorption capacity by adsorbent with contact time was plotted and presented in Fig. 3. The amount of the adsorbed

metal ions increased as the time lapsed, similar case was recorded by Onundi et al. [27]. The metal removal approached equilibrium within 70 min for lead and nickel. The adsorbent showed a progressive increase in the uptake of lead and nickel.

The fast adsorption at the initial stage was probably due to the initial concentration gradients between the adsorbate in solution and the number of vacant sites available on the activated LBH surface at the beginning. The progressive

Table 3. Effect of contact time on Pb²⁺ and Ni²⁺ adsorption

Time (min.)	Lead		Nickel	
	q _e (mg/g)	%Rem	q _e (mg/g)	%Rem
30	4.09	81.8	1.99	39.9
50	4.20	84.0	2.42	48.3
70	4.27	85.3	2.90	57.9
90	4.27	85.3	2.90	57.9
110	4.27	85.3	2.90	57.9

(Adsorbent Dose = 0.2 g, Initial concentration = 20 mg/dm³, Temperature = 27°C)(Key: q_e is the metal uptake in mg/g, i. e. the amount of metal ion adsorbed in milligram per gram of the activated LBH while % Rem is the removal efficiency i.e percentage metal ion removed)

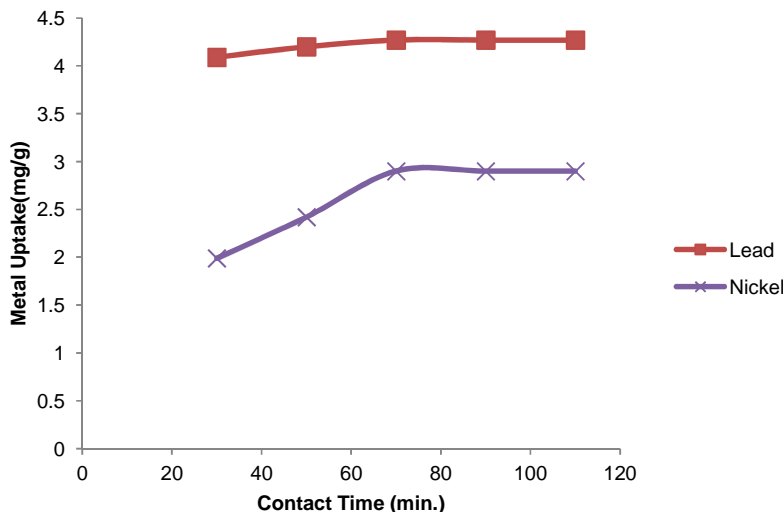


Fig. 3. Effect of contact time on Pb²⁺ and Ni²⁺ adsorption

increase in adsorption and consequently the attainment of equilibrium adsorption may be due to limited mass transfer of the adsorbates molecules from the bulk liquid to the external surface of activated LBH. It was noted that the adsorption capacities corresponding to equilibrium varied for each metal, with lead 4.27 mg/g (85.4%) and nickel 2.9 mg/g (58%), giving a trend of Pb²⁺>Ni²⁺ on the adsorbent. Contact times were used to generate the rate of the reaction [20]. To generate the rate of adsorption, pseudo-first order and pseudo-second order were used on the experimental data in Table 3.

3.3.1 Adsorption kinetics

In order to investigate the mechanism of adsorption and potential rate controlling step, pseudo-first order and pseudo-second order kinetic models described in section 2.1.4 were used to test the equilibrium data in Table 3. Moreover, information on the kinetics of metal

uptake is required for selecting optimum conditions for full scale batch metal removal processes. A kinetic study with different time intervals, fixed metal and adsorbent concentration was performed and the obtained results are presented in Table 4.

Table 4 shows comparison between adsorption rate constants, q_e estimated and correlation coefficient associated to the Lagergren pseudo – first and pseudo– second order adsorption of all the metals. Figs. 4 and 5 represent pseudo–second order kinetics modeling of Pb²⁺ and Ni²⁺ adsorption onto activated LBH respectively, while Figs. 6 and 7 represent pseudo– first order kinetics modeling of Pb²⁺ and Ni²⁺ adsorption onto activated LBH respectively. The experimental adsorption capacities of Pb²⁺ and Ni²⁺ are 4.27 mg/g and 2.9 mg/g respectively. Their calculated adsorption capacities by using pseudo-first order model are 0.05 mg/g and 0.6 mg/g respectively.

Table 4. Comparison between adsorption rate constants, q_e estimated and correlation coefficient associated to the lagergren pseudo- first and pseudo- second order adsorption

	Pseudo- first order model			q _e exp. (mg/g)	Pseudo- second order model		
	q _e (calc)	k ₁ (min ⁻¹)	R ²		q _e (calc)	k ₂ (gmg ⁻¹ min ⁻¹)	R ²
Lead	0.050	-0.030	0.598	4.27	4.48	0.041	0.999
Nickel	0.614	-0.005	0.204	2.90	3.57	0.013	0.986

(key: k₁ is the rate constant for pseudo- first order adsorption, k₂ is the pseudo-second order rate constant of adsorption, q_e(calc) is metal uptake (mg/g) calculated using pseudo- first and pseudo-second order equation, q_e(exp) is metal uptake obtained experimentally during adsorption of the metal ions)

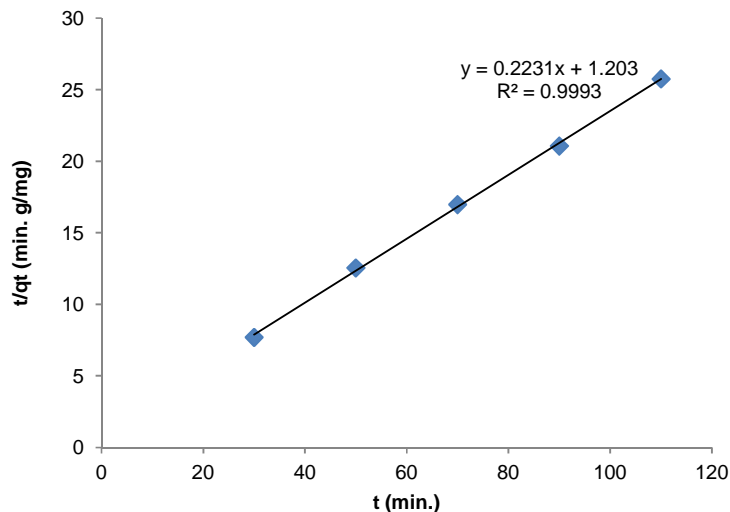


Fig. 4. Pseudo- second order model for pb²⁺ adsorption

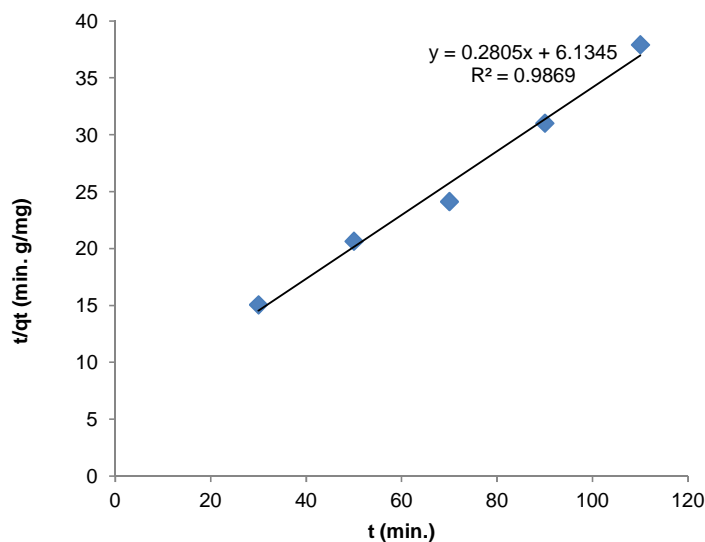


Fig. 5. Pseudo- second order model for ni²⁺ adsorption

while that of pseudo- second order model are 4.48 mg/g and 3.57 mg/g. The calculated adsorption capacities of all the metals by pseudo-second order model are very close to their respective experimentally determined adsorption capacities unlike pseudo- first order, where there is large difference between the experimental and calculated adsorption capacities of all the metals.

The correlation coefficients, R^2 values for the pseudo- second order adsorption model of all the

metals are higher (>0.986) than that of pseudo- first order adsorption model (<0.780). Thus, the correlation coefficient values and calculated adsorption capacities of all the metals by pseudo- first order model are not satisfactory, which suggests that they are dependent on initial concentration. Hence, pseudo- second order adsorption model is more suitable to describe the adsorption kinetics of Pb^{2+} and Ni^{2+} onto the activated LBH and this relies on the assumption that adsorption may be the rate-limiting step [20].

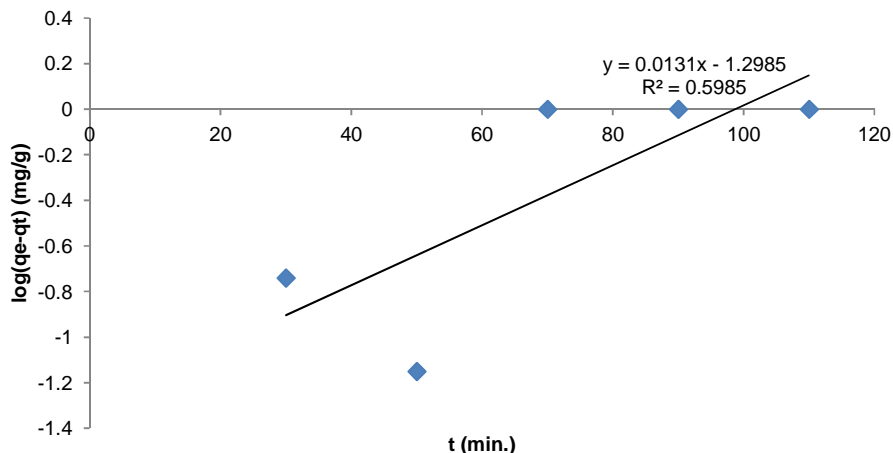


Fig. 6. Pseudo- first order model for pb²⁺ adsorption

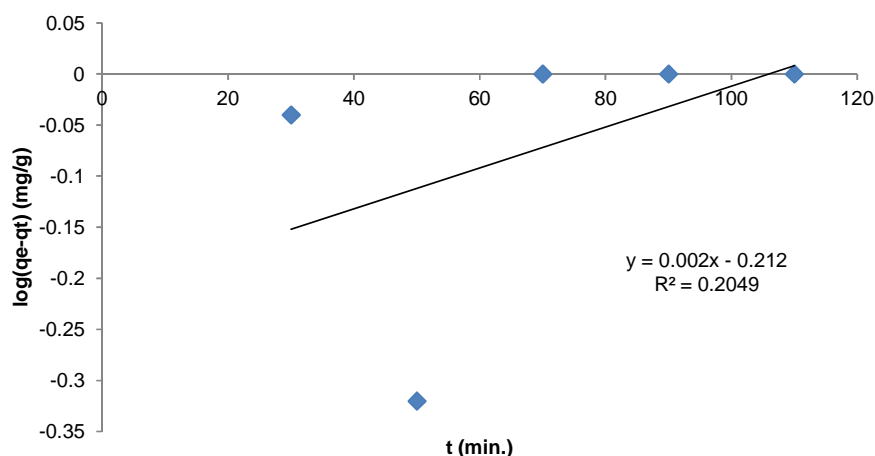


Fig. 7. Pseudo- first order model for ni²⁺ adsorption

3.4 FTIR Analysis

The spectra of activated and heavy metal treated adsorbent in a range of 350–4700 cm⁻¹ were analyzed in order to detect the functional groups that were responsible for the adsorption process. The result showed the difference between the activated and heavy metal (Pb²⁺ and Ni²⁺) treated adsorbent.

The spectrum of activated LBH (Fig. 8) shows peaks at 3550-3350 cm⁻¹ indicating the presence of OH or -NH groups. Figs. 9 and 10 represent the spectra of heavy metal treated adsorbent. The absorption peaks at 1654-1540 cm⁻¹ corresponds to the 1° amide and 2° amide

bands, respectively [28]. The bands between 1740 and 1650 cm⁻¹ corresponds to C=O of carboxyl group present in the adsorbent. Similarly, band between 1540 and 1500 cm⁻¹ are due to 2° amide stretching frequency. The above three bands indicate the presence of amide group with hydrogen bond linkage in the adsorbent. The region between 3200 and 3500 cm⁻¹ represents the overlapping peaks of stretching vibration of O-H and N-H [29]. The region between 3000 and 2800 cm⁻¹ exhibits the C-H stretching vibrations of -CH₃ and >CH₂ functional groups and 1300–1470 cm⁻¹ is the deformation stretching of C-H, -CH₃ and >CH₂ functional groups [28].

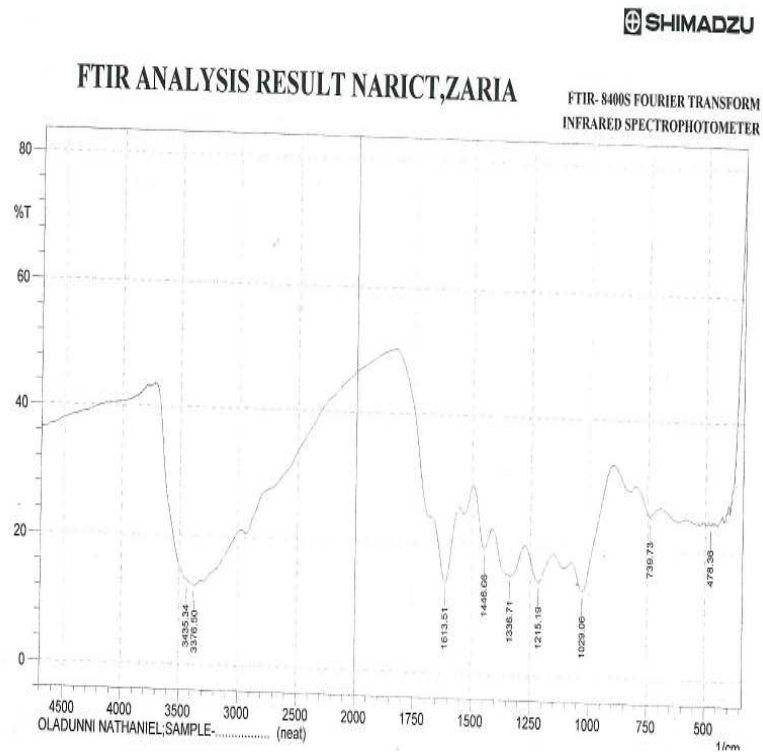


Fig. 8. FTIR spectra of activated locust bean husk

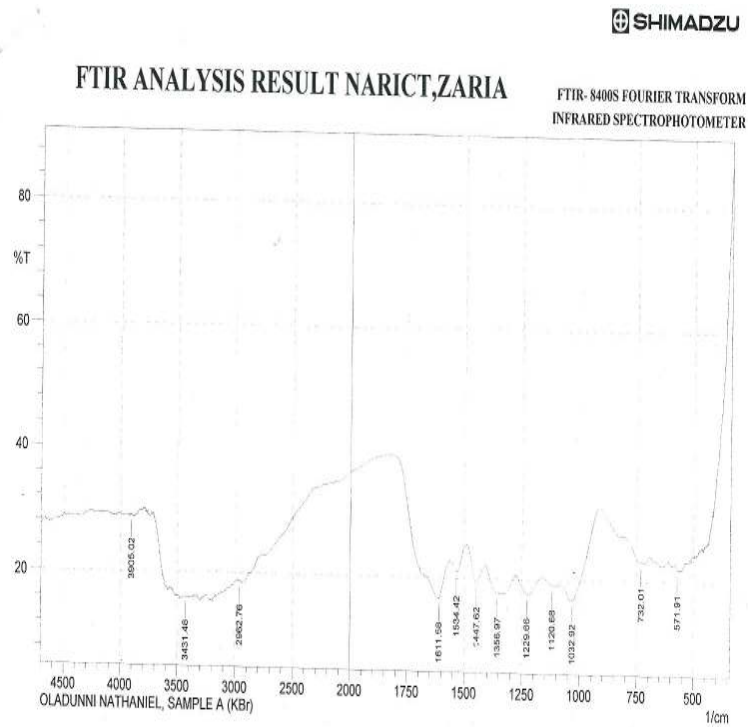


Fig. 9. FTIR spectra of lead treated activated locust bean husk

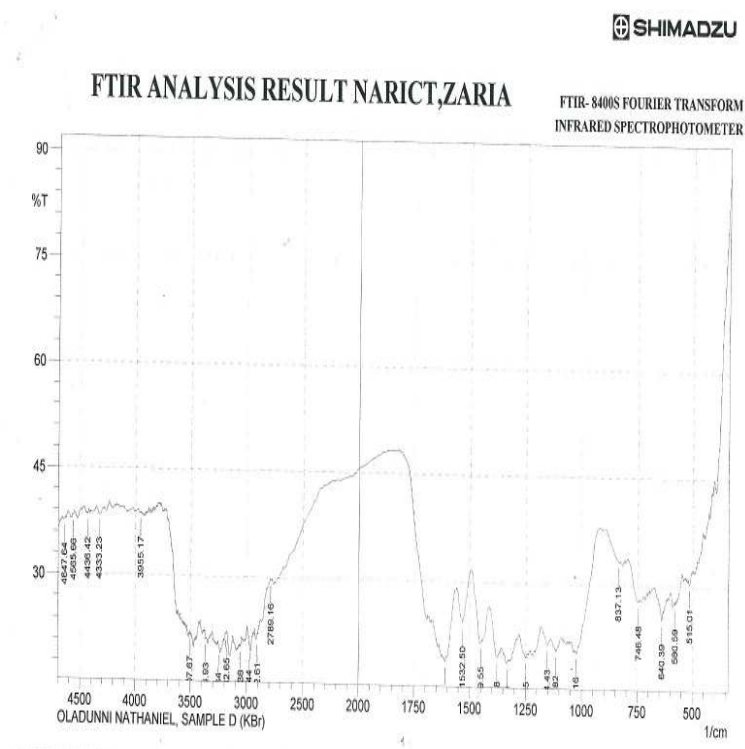


Fig. 10. FTIR spectra of nickel treated activated locust bean husk

4. CONCLUSION

This research showed that activated locust bean husk is an effective adsorbent for Pb^{2+} and Ni^{2+} removal from aqueous solution. It was observed that removal of Pb^{2+} and Ni^{2+} by activated LBH had highest adsorption capacity at pH6 and that the uptake of the selected heavy metal ions by activated LBH increases with increasing temperature. It was also established that the amount of adsorbed metal ions increased as the time lapsed. The metal removal approached equilibrium within 70 min for lead and nickel. Hence, the batch studies conducted in this present work provides vital information, that adsorption of Pb^{2+} and Ni^{2+} by activated LBH is dependent of initial solution pH, contact time, and temperature.

Kinetic studies carried out showed that the calculated adsorption capacities of all the metals by pseudo- second order model are closer to their respective experimentally determined adsorption capacities than those from pseudo-first order model. Hence, pseudo- second order adsorption model is more suitable to describe the adsorption kinetics of Pb^{2+} and Ni^{2+} by activated LBH.

FTIR spectra analysis confirmed the occurrence of a successful adsorption of these metals by activated LBH. FTIR spectra display characteristic absorbance peaks of carboxyl, hydroxyl, amines, amides, alkenes, alkynes, methyl and methylene functional groups. Some of these peaks were found to disappear in the spectra of heavy metal-treated adsorbent. Hence, these functional groups corresponding to the disappeared peaks are responsible for the adsorption of the selected heavy metals (Pb^{2+} and Ni^{2+}).

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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