



Original Research Article

REMOVAL OF LEAD (II) IONS FROM AQUEOUS SOLUTION USING SAWDUST

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ABSTRACT

The focus of this work was to assess the performance of sawdust as a potential adsorbent for removing Lead (II) ion from aqueous solution. The raw sawdust was compared with the activated Sawdust in order to see the performance of both as an adsorbent. Activation of the Sawdust was done in order to improve the adsorption capacity. The activated sawdust was characterized to determine its physiochemical properties such as moisture content, ash content, functional groups and surface structure. The effect of indirect adsorbent/adsorbate interactions was studied using adsorption isotherms of the monolayer Freundlich and Langmuir was found to match the Langmuir Adsorption isotherm with a coefficient of determination (R^2) value of 0.825. The batch experimental result revealed an increase in the percentage of Pb (II) ion removal by the raw and activated Sawdust and increased with an increase in adsorbent dose and contact time. The study of kinetics of Pb (II) ion removal onto activated Sawdust followed a second order kinetic model with an R^2 value of 0.937. In general, the result of the activated sawdust compared to the raw sawdust demonstrated that the activated sawdust is a more efficient adsorbent material capable of removing lead from waste water.

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1. INTRODUCTION

Wastewater from numerous industries such as paints and pigments, glass production, mining operations, metal plating, and battery manufacturing processes are known to contain contaminants such as heavy metal (Badmus *et al.*, 2007; Bai and Bartkiewicz, 2009). Heavy metals such as lead (Pb), Cadmium (Cd), Chromium (Cr), Nickel (Ni), Zinc (Zn), Copper (Cu) and Iron (Fe) are present in industrial wastewater, they are not biodegradable and their existence in receiving lakes and streams causes bioaccumulation in living organisms, which leads to several health problems in animals, plants and human beings such as cancer, kidney failure, metabolic acidosis, oral ulcer, renal failure and

damage in for stomach of the rodent (Mehmet *et al.*, 2007). As a result of the problems caused by heavy metals pollution, removal of heavy metals from wastewater is important (Chand *et al.*, 1994).

Generally, the techniques employed for heavy metal removal include precipitation, ion exchange, adsorption, filtration, electro deposition, reverse osmosis etc. (Rao *et al.*, 2000). However, most of these methods do not lead to a satisfactory depollution considering the operational costs (Marchetti *et al.*, 2000). Precipitation methods are particularly reliable but require large settling tanks for the precipitation of voluminous alkaline sludge and a subsequent treatment is needed (Dabrowski *et al.*, 2004). Ion exchange has the advantage of allowing the recovery of metallic ions, but it is expensive and sophisticated (Volesky, 2001). Adsorption on solid-solution interface is an important means for controlling the extent of pollution due to heavy metal ions the use of activated carbon and ion exchange resins is not suitable for developing countries due to their high capital and operational costs (Raji and Anirudhan, 1997). This has encouraged research into discovering materials that are both efficient and cheap. Interest has recently arisen in the investigation of some unconventional methods and low-cost materials for scavenging heavy metal ions from industrial waste waters (Gloaguen and Morvan, 1997). Some of the reported low-cost adsorbents include bark, tannin-rich materials, lignin, chitin, chitosan, peat moss, moss, modified wool and cotton (Yasemin and Zeki, 2007). Insoluble starch xanthates have been found to be very useful in removing heavy metal ions from solutions (Rao *et al.*, 2000).

Interest in the use of sawdust as an adsorbent has been stimulated by the good results that have been obtained. Hence, this study was focused on the use of sawdust collected from a Sawmill in Okada town, Edo State, Nigeria for the removal of lead (II) ion (Pb^{2+}) from solution. The choice was made because of its high availability and low cost.

2. MATERIALS AND METHODS

2.1. Material Collection and Preparation

Raw sawdust was collected from a Saw mill in Okada town in Ovia North-East Local Government Area, Edo state, Nigeria. It was thereafter air dried for a period of 3 days at a constant rate of 5 hours per day and oven dried at 100 °C for 2 hours. The dried Sawdust was then reduced to fine powder by grinding using a laboratory sized mortar and pestle. Thereafter, it was sifted with 425 μ m Standard Tyler Sieve.

2.2. Carbonization and Activation

Carbonization was done using the method recommended by Ekpete and Horsefall (2011) with slight modification as follows. A predetermined weight of the pulverized Sawdust was placed in a muffle furnace which allows limited supply of air at a temperature of 350 °C for 60 minutes. The carbonized samples were then activated using the method recommended by Mansfield (1998) with slight modification as follows: 25g of the carbonized sample was soaked in 250 ml of 5.5M $ZnCl_2$ solution. The mixture was thoroughly mixed until it formed a paste. The paste was then transferred to an evaporating dish which was placed in a furnace and heated at 200 °C for 30 minutes. This was allowed to cool and washed with distilled water to remove the residual salt. It was then oven dried at 105 °C for one hour, grinded using mortar and pestle and then sieved with 106 μ m Standard Tyler Sieve. The activated Sawdust was then characterized to determine its particle sizes, moisture content, ash content, functional group, elemental composition and microstructure characteristics.

2.3. Characterisation of Adsorbent

2.3.1. Functional group determination

The Fourier Transform Infra-Red (FT-IR) spectrometry was used to determine the nature of functional groups which could possibly influence the adsorption capacity of the activated and raw sawdust as suitable adsorbent for Pb (II) removal (Dawodu et al., 2012)

2.3.2. Analysis of microstructures

Scanning Electron Microscope (SEM) was employed to study the surface characteristics in terms of the presence of micro porous structure on the surface of the activated sawdust. Such presentations may provide possible explanations about the solid's behavior and its adsorption potentials (Omisanya et al., 2012).

2.3.3. Determination of pH

The standard test method for determination of activated carbon pH (ASTM D3838-80) was used in which 1.0 g of activated Sawdust was weighed into a beaker and 100 ml of distilled water was added and stirred for one hour. The samples were allowed to stabilize before the pH was measured using a digital pH meter (Hanna pH 210 model).

2.3.4. Moisture content determination

Thermal drying method was used in the determination of moisture content of the samples for which 1.0g of the dried activated sawdust was weighed and placed in washed, dried and weighed crucible. The crucibles were placed in an oven and dried at 105 °C to constant weight for 1 hour. The percentage moisture content (%MC) was computed as follows (Rengaraj, 2002).

$$\text{Moisture Content (\%)} = \frac{\text{loss in weight on drying (g)}}{\text{Initial weight of sample (g)}} \times 100 \quad (1)$$

2.3.5. Ash content determination

The standard test method for ash content-ASTM D2866-94- was used where by 1.0 g activated sawdust was taken dried in an oven at a temperature of 105 °C for 1 hr. The final weight after drying was measured and recorded as (Xg = Oven dry weight). Thereafter the dried activated sawdust was placed in a cold muffle furnace and the temperature was allowed to rise until it reached 500 °C. It was removed and allowed to cool in a desiccator to room temperature and reweighed again and its weight was again recorded as (Yg = Ash weight).

The percentage ash content was then determined as follows:

$$\text{Ash Content (\%)} = \frac{\text{Ash Weight (g)}}{\text{Oven Dry Weight (g)}} \times 100 \quad (2)$$

2.4. Preparation of Aqueous Solution

All the chemicals used in this study were analytical grade from BDH. A stock solution of lead (II) nitrate [Pb(NO₃)₂] was prepared by dissolving 5000mg of the salt in 1litre distilled water. Fresh solutions were used for each experiment and the concentration of the metal Pb(II) ions present in solution was analyzed using Atomic Absorption Spectrophotometer (AAS).

2.5. Adsorption Experiments

The adsorption study was carried out to determine the effect of adsorbent loading and contact time using batch adsorption technique. The adsorption experiment was performed at different adsorbent doses (0.5, 1.0, 1.5, 2.0, 2.5 and 3.0g), and contact times (20, 40, 60, 80, 100 and 120 minutes), at room temperature (30 – 32°C). The batch adsorption experiment was carried out with 50 ml aqueous solution of lead ion with varying load of the adsorbent was stirred for a specified contact time to ensure that equilibrium was achieved. The separation of the adsorbents from solution was carried out by filtration using 150 mm diameter Whatman filter paper. The filtrate was then stored in sample can and the residual metal ion concentration was determined using Atomic Adsorption Spectrophotometer (AAS).

2.6. Adsorption Capacity

The amount of Pb (II) metal ions removed during the series of batch investigation was determined using the mass balance equation of the form (Badmus et al., 2007):

$$q = \frac{V}{m} [C_0 - C_e] \quad (3)$$

Where: q, defines the metal uptake (mg/g), C₀ and C_e are the initial and equilibrium metal ion concentrations in the aqueous solution (mg/l) respectively, V is the aqueous solution volume (ml) and M is the mass of adsorbent used (g). The efficiency of Pb(II) metal ion removal (%) was computed using the mass balance equation of the form.

$$\text{Removal Efficiency (\%)} = \left(\frac{C_0 - C_e}{C_0} \times 100 \right) \quad (4)$$

3. RESULTS AND DISCUSSION

Results of characterization of activated sawdust used for this work are presented in Table 1 which is compared with other adsorbents found in literature.

Table 1: Physical properties of activated sawdust

Parameter	This work	(Ilaboya et al., 2013)			
		CAC	ACP	AYP	APP
Surface pH	6.7	6.5	6.8	6.8	6.8
Moisture Content (%)	2.3	4.33	8.7	9.8	9.2
Ash Content (%)	5.1	5.1	6.7	7.8	7.3
Apparent Density (g/ml)	0.54	0.56	---	---	---

CAC -commercial activated carbon, ACP -activated cassava peels, AYP -activated yam peels, APP -activated potato peels

Scanning Electron Micrograph was taken in order to verify the presence of micro pores in the structure of the sawdust as seen in Figures 1 and 2. The larger number of micro porous structure seen with the activated Sawdust indicates a higher surface area. This claim is based on the fact that as biosorbent materials present larger numbers of micro porous structure, it adsorbs higher amount of nitrogen, which resulted to a higher (BET) Brunauer Emmett Teller surface area and higher adsorption properties. (Abdullah et al., 2001; Arenas et al., 2004).

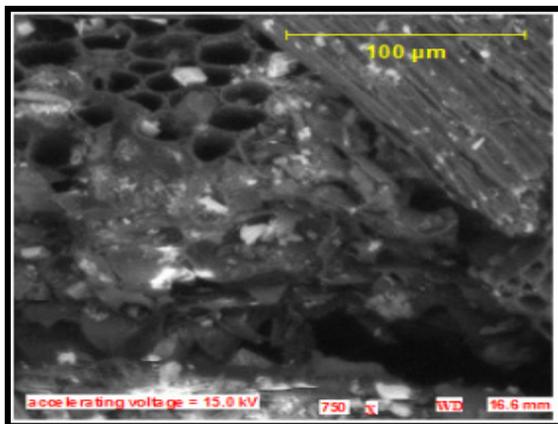


Figure 1: SEM of activated sawdust (x750)

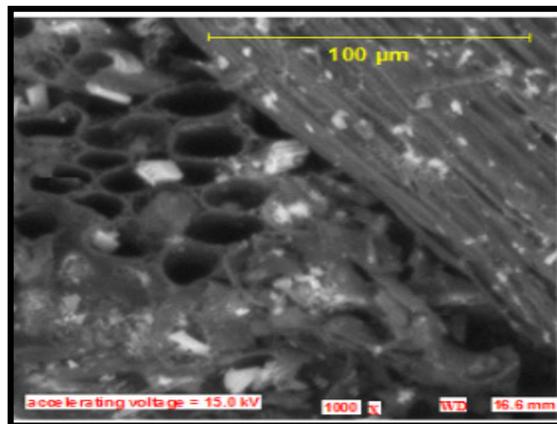


Figure 2: SEM of activated sawdust (x1000)

Insight into the nature of functional groups that make up the surface of adsorbent would create a better picture on the adsorption potentials of the material. To identify the functional groups present on the surface of sawdust, Fourier Transform Infrared (FTIR) spectroscopy was used. Figure 3 shows the Fourier Transform Infrared (FTIR) spectra of the raw sawdust used for this study.

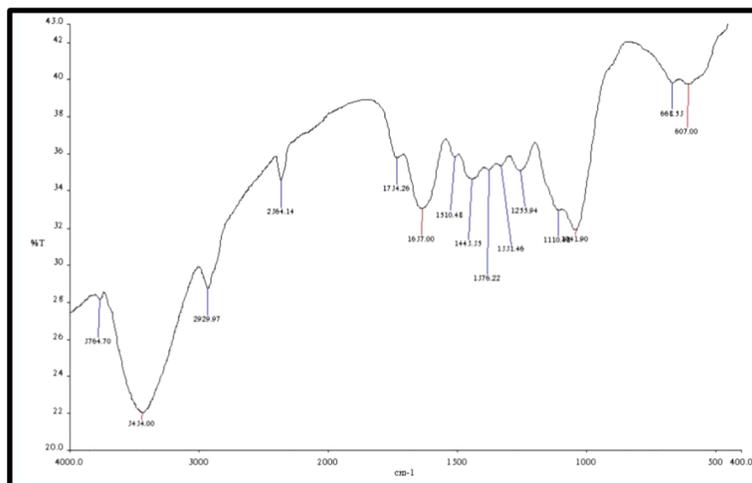


Figure 3: FTIR Spectra of raw sawdust

To identify the functional group based on the FTIR spectra, absorption assigned bands from the works of previous researchers were adopted and employed to analyze and evaluate the spectrum of the raw sawdust.

From Figure 3 it can be observed that between the wave number ranges of (3000 – 4000) cm^{-1} primary and secondary alcohol bonds are present, between the wave numbers of (1000 – 1500) cm^{-1} organic phosphate, organic siloxane and aromatic groups are present and from (400 – 500) cm^{-1} disulphides and polysulphides groups (as interpreted in Table 2).

3.1. Batch Adsorption Results

3.1.1. Effect of adsorbent dose on Pb(II) ion removal

The effect of adsorbent dose on the amount of Pb(II) ion adsorbed by raw sawdust and activated Sawdust is presented in Figures 4 and 5 respectively.

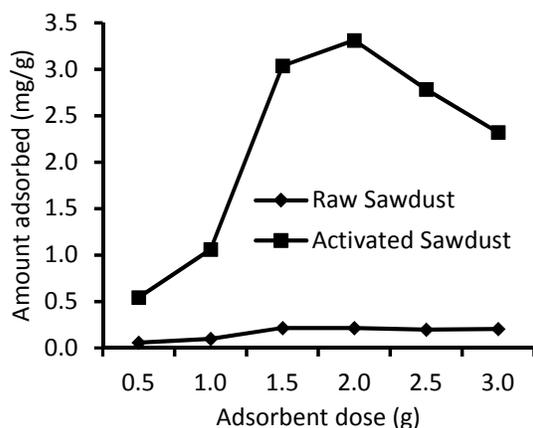


Figure 4: Effects of adsorbent dose on the amount of Pb(II) ion adsorbed

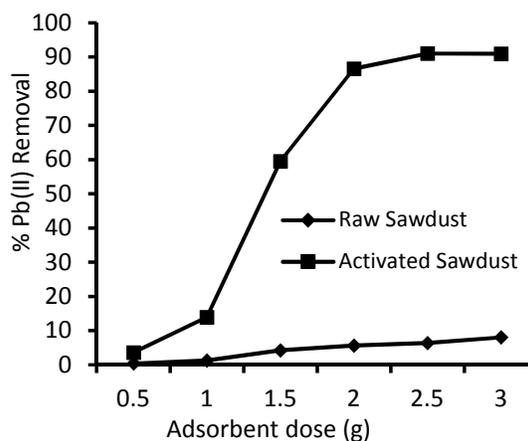


Figure 5: Effects of adsorbent dose on the efficiency of Pb(II) ion removal

From Figure 4 it can be seen that higher dosage yield a greater amount of lead ion been adsorbed until it gets to its optimal dosage where it began to decline which indicates the attainment of equilibrium for the amount of lead ion adsorbed. Also, from Figure 5 higher adsorbent dosage resulted in greater percentage removal of lead ion from solution. Higher dosage of adsorbent will increase the adsorption due to increase in availability of surface active sites of the adsorbents on which the metal could interact with (Ahmet et al., 2007; Ilaboya et al., 2013). These chemical groups were important in the formation of van der Waals bonding as the functional groups played the main role in binding metals to adsorbents during adsorption process (Srivastava and Eames, 1998). This provided more possibilities for adsorption to occur since there was less competition between metals for the binding sites.

3.1.2. Effect of contact time on Pb(II) ion removal

The effect of contact time on the amount of Pb(II) ion adsorbed by raw sawdust and activated sawdust are presented in Figures 6 and 7 respectively.

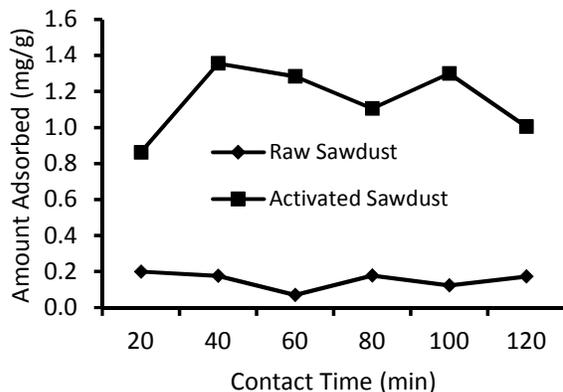


Figure 6: Effects of contact time on the amount of Pb(II) ion adsorbed

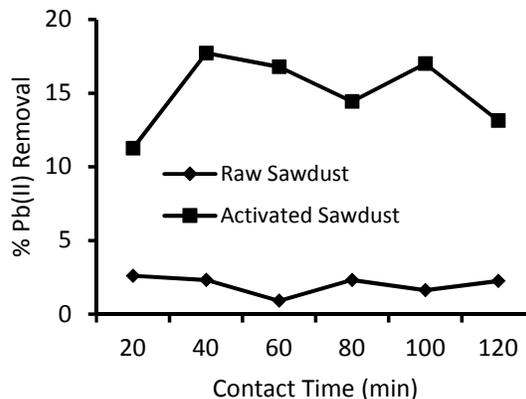


Figure 7: Effects of contact time on the efficiency of Pb(II) ion removal

The increased rate of adsorption noticed at the early stage of the process especially with activated Sawdust as seen in Figure 7 can be traced to the availability of abundant active sites and uncovered surface area of the adsorbent, since the adsorption kinetics depend on the surface area of the adsorbents (Qadeer and Akhtar, 2005). The lead adsorption takes place at the more reactive sites. As these sites are progressively filled, it becomes more difficult for sorption to take place, as the sorption process tends to be more unfavorable (Badmus *et al.*, 2007). As the surface adsorption sites become exhausted, the uptake rate is controlled by the rate at which the adsorbate is transported from the exterior to the interior sites of the adsorbent particles. This is the general characteristic of adsorption of this metal ion. These changes and differences in metal ion up take rate could be attributed to two different adsorption processes, namely a fast ion exchange followed by chemisorption (Low *et al.*, 1993).

3.2. Adsorption Isotherm Studies

Adsorption data obtained were analyzed in the light of Freundlich and Langmuir isotherm model equations. The monolayer Freundlich isotherm is an indication of the extent of heterogeneity of the adsorbent surface. The general form of the isotherm is given as:

$$q = K_f C_e^{\frac{1}{n}} \quad (5)$$

Where: q = amount adsorbed (g); K = Freundlich capacity constant; C_e = Equilibrium concentration (ppm); $1/n$ = Freundlich intensity parameter

A linear form of this expression is given as $\{\log(q) = \log(K_f) + 1/n \log(C_e)\}$ and values of K and n were calculated from the slope of the plot of $\{\log(q)$ against $\log(C_e)\}$ (Figures 8 and 9).

The Langmuir adsorption isotherm is based on the theoretical principle that only a single absorption layer exists on an adsorbent and it represents the equilibrium distribution of metal ions between the solid and liquid phases. The equation is described by Equation 6:

$$q = \frac{abC_e}{bC_e + 1} \quad (6)$$

On rearranging Equation (6) yields:

$$\frac{C_e}{q} = \left(\frac{C_e}{a}\right) + \frac{1}{ab} \quad (7)$$

Where q is the amount of metal adsorbed per unit mass of adsorbent (g), a and b are the Langmuir constants and are indicative of adsorption capacity (mg/g) and energy of adsorption (l/mg) respectively and C_e is the equilibrium concentration of adsorbate in aqueous solution sample after adsorption (mg/l). The Langmuir constants (a) and (b) were obtained from the intercept and slope of the plot of C_e/q against (C_e) (Figures 10 and 11).

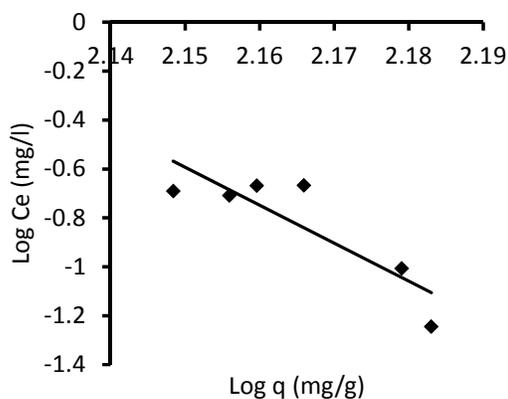


Figure 8: Freundlich Isotherm plot for adsorption of Pb(II) ion onto raw Sawdust

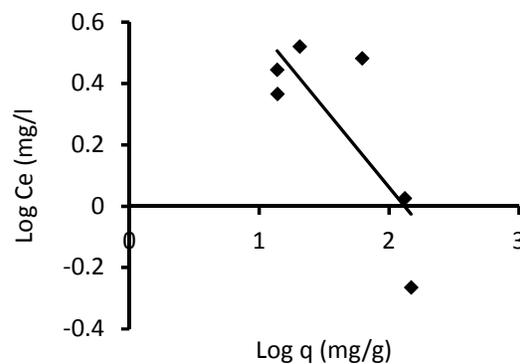


Figure 9: Freundlich Isotherm plot for adsorption of Pb(II) ion onto activated Sawdust

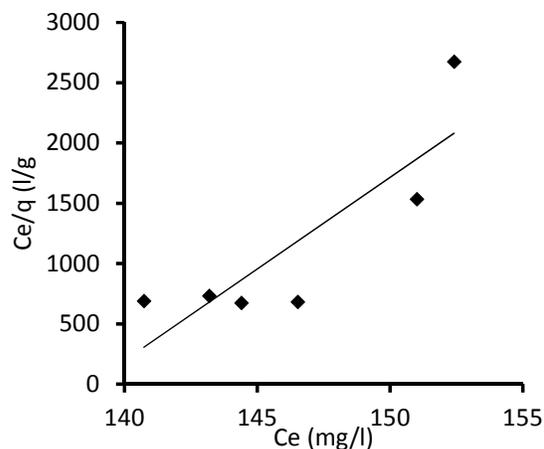


Figure 10: Langmuir Isotherm plot for adsorption of Pb(II) ion onto raw Sawdust

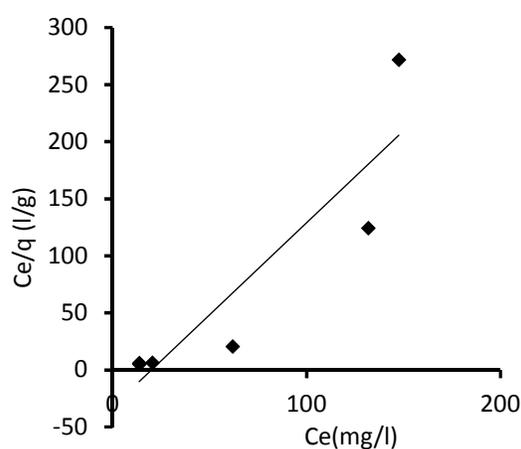


Figure 11: Langmuir Isotherm plot for adsorption of Pb(II) ion onto activated Sawdust

Based on the result of Table 2, it is observed that the Langmuir isotherm model best explain the adsorption data which indicates that the adsorption of the activated Sawdust is a monolayer type (Pimentel et al., 2008). Also, activated Sawdust performed better than the raw Sawdust.

Table 2: Computed coefficient of linear determination

Model	Adsorbent	R ²
Freundlich	Raw sawdust	0.754
Freundlich	Activated sawdust	0.614
Langmuir	Raw sawdust	0.726
Langmuir	Activated sawdust	0.825

3.3. Adsorption Kinetic Studies

To study the kinetics of Pb(II) ion removal onto raw and activated Sawdust, experimental data obtained were fitted into the first and second order kinetic model. The kinetic model was employed to study the time dependent effects of the adsorption process. The pseudo first-order equation (Lagergren, 1898) is generally expressed as follows:

$$\frac{dq_t}{dt} = K_1(q_e - q_t) \quad (8)$$

Where: q_e and q_t are the adsorption capacities at equilibrium and at time t , respectively (mg/g^{-1}), K_1 is the rate constant of pseudo first-order adsorption. After integration and applying boundary conditions $t = 0$ to $t = t$ and $q_t = 0$ to $q_t = q_t$, the integrated form of Equation (8) becomes,

$$\log[q_e - q] = \log q_e - \frac{K_{ad}t}{2.303} \quad (9)$$

The linear plots of $\log[q_e - q]$ against time (t) shows the appropriateness of the above equation and subsequently the first order nature of the adsorption process involved.

The pseudo second-order equation is of the form:

$$\frac{dq_t}{dt} = K_2(q_e - q_t)^2 \quad (10)$$

Where K_2 is the rate constant of pseudo-second order adsorption ($\text{gmg}^{-1}\text{min}^{-1}$). For the boundary conditions, $t = 0$ to $t = t$ and $q_t = 0$ to $q_t = q_t$, the integrated form of Equation (10) becomes:

$$\frac{1}{(q_e - q_t)} = \frac{i}{q_e} + Kt \quad (11)$$

Equation 11 can be rearranged to obtain Equation (12) which has a linear form:

$$\frac{t}{q_t} = \frac{i}{K_2 q_e^2} + \frac{1}{q_e} (t) \quad (12)$$

The plot of $\left(\frac{t}{q_e}\right)$ against (t) should give a linear relationship from which q_e and K_2 can be determined from the slope and intercept of the plot. The graphical variations for the pseudo first and second order kinetics are presented in Figures 12 and 13.

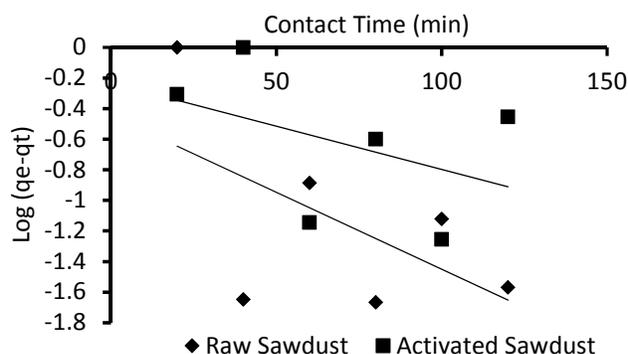


Figure 12: First order kinetic model plot for Pb(II) ion adsorption

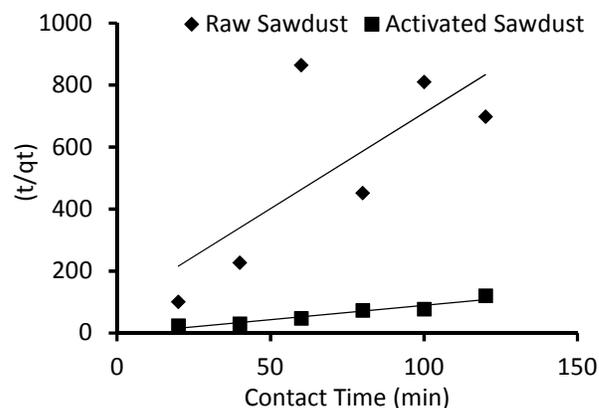


Figure 13: Second order kinetic model plot for Pb(II) ion adsorption

Table 4: Kinetic data of the adsorption process

Adsorbent	R ²	Order of Reaction
Raw sawdust	0.1882	1 st Order
Activated sawdust	0.3405	1 st Order
Raw sawdust	0.5338	2 nd Order
Activated sawdust	0.937	2 nd Order

From the value of co-efficient of correlated (R^2) calculated from Table 4, it is seen that the second order kinetic model best fit the adsorption data. Hence, the adsorption of Pb(II) onto activated sawdust is purely a second order reaction.

4. CONCLUSION

The following conclusions can be drawn from this study:

- Activated sawdust produced from freshly cut sawdust showed very high potential in the removal of lead ion under optimized condition of dosage 1.0g/l for an equilibrium adsorption time of 120 minutes
- Based on the equilibrium data obtained, the Langmuir was found to be in good agreement from the value of co-efficient of correlated (R^2) calculated which indicates that the adsorption of Pb(II) ion by activated Sawdust is a monolayer type.
- The method employed was the batch operation mode and the adsorption process followed a second (2nd) order kinetics.
- Activated sawdust performed better than the raw Sawdust and has a higher potential to remove Pb(II) ion from aqueous solution.

5. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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