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REMOVAL OF Pb (II) IONS FROM AQUEOUS SOLUTION USING MAHOGANY (*Swietenia Macrophylla* King) SAWDUST AS LOWCOST ADSORBENT

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ABSTRACT

This research aimed to investigate the removal efficiency of Pb (II) ions using mahogany sawdust as an adsorbent. Several experiments were carried out to get the results. The batch adsorption method was used. The parameters studied were pH and contact time. The pH variation used was 2, 3, 4, 5, and 6. Contact time variation used was 5, 10, 15, 30, 60, 90, 120 and 150 minutes. To evaluate the isotherm model of the adsorption, the metal concentration variation used was 10, 25, 50, 100, 150, and 200 mg/L. To evaluate kinetics adsorption, pseudo-first-order and pseudo-second-order models were used. The characterization of the adsorbent was carried out by determining pH_{PZC}, FTIR analysis, and SEM analysis. The adsorbent had a pH_{PZC} of 5.63. FTIR spectra showed that the adsorbent had -OH functional group that could bind to Pb (II) ions. SEM analysis showed that the surface morphology of the adsorbent supported the removal of Pb (II) ions. In this work, pH 6 provided the highest removal of Pb (II) ions, while the contact time which provided the highest removal of Pb (II) ions was 30 minutes. The removal of Pb (II) ions followed the Freundlich adsorption isotherm, while the adsorption kinetics followed pseudo-second-order model.

Keyword: *wastewater treatment, clean water, lead, sawdust, mahogany*

INTRODUCTION

Wastes from industrial activities can be found in heavy metals that can degrade environment quality, especially liquid wastes containing toxic heavy metals that can be dissolved in the aquatic ecosystem. The discharge of heavy metals without proper environmental treatment can be toxic to aquatic life. Some industries use heavy metals as raw materials. As a consequence of heavy metals, the resulting waste also contains heavy metals. One of the toxic metals is lead (Pb). Waste containing lead comes from wastes of battery, solder, alloys, cable sheathing pigments, rust inhibitors, ammunition, glazes, and plastic stabilizers production [1]. Lead has many negative effects on humans: structural damage and failure of kidney, central nervous system problems, sperm dysfunction, pregnancy complication, preterm birth, anemia, malaise, fatigue, weight loss, neuromuscular problems, mineralizing bones, and teeth, decreased bone density [2].

There are reports of heavy metal pollution in Indonesia. In the river and coast sediments of the Jakarta Bay region, waste containing Pb, Cu, Ni, and Zn was found from metal processing industries and fertilizers [3]. The negative impact of heavy metals on organisms is possible because heavy metals can accumulate in organisms and cause growth disorders. For example, green mussels originating from Muara Kamal (Jakarta Bay) were reported to be contaminated by Hg, Pb, Cd and experienced incomplete or abnormal growth [4].

Removing heavy metals from waste-water are chemical precipitation, membrane filtration, ion exchange, adsorption, and bio-accumulation [5]. Adsorption is a purification or separation technique that is effectively used in industry because it is considered more economical for wastewater treatment than other methods of heavy metals treatment [6].

Adsorbents are used as agents to adsorb pollutants. The active carbon has good adsorption ability, but it poses, in some cases, economic problems. Face this problem; many researchers were oriented to develop alternative adsorbents. Some alternative adsorbents that have been studied include orange peels, nutshells, rice husk, sawdust, wheat straw, and sugarcane bagasse [7].

Sawdust is a waste that comes from the wood industry. This waste is relatively abundant, and the price is cheap. Based on the research done in the study of sawdust and its components (cellulose, hemicellulose, lignin), sawdust can be used as an adsorbent to sequester heavy metals [8–11]**.** Mahogany wood (*Swietenia macrophylla* King) is widely used in the furniture industry. Mahogany sawdust contains cellulose having the ability to adsorb heavy metals. This is because cellulose has a hydroxyl group that can interact with heavy metals [12,13]**.** In the lead removal study, the adsorbent used was mahogany sawdust prepared by heating at 105^{o}C [12]. The results obtained showed that the optimum conditions for removing lead ions were pH 6, a contact time of 30 minutes, and a lead metal concentration of 300 mg/L [12].

Meanwhile, the temperature used to prepare the mahogany sawdust adsorbent in the nickel ions removal study was 70^{o}C [13]. The results obtained showed that at pH 9 the maximum removal of nickel ions was achieved. When using a contact time of fewer than 50 minutes, the removal of nickel ions reached 80% [13]. This fact indicates that mahogany sawdust has the potential to be adsorbent for heavy metals. The advantages of using mahogany sawdust are that it is cheap, easy to obtain, and easy to be prepared as an adsorbent.

In this work, mahogany sawdust was used to remove Pb (II) ions. Although, in this study, the novelty was in the mahogany sawdust prepared by heating at 200^{0}C, heating at this temperature was to improve the adsorption ability of heavy metals.

METHODS

1. Materials

The material used was mahogany sawdust taken from one of the wood industries in Sidoarjo, East Java, Indonesia. Pb (II) solutions were prepared from Pb $(NO_3)_2$. HCl 1 M and NaOH 1 M were used to adjust pH, and NaCl 0.01 M was used for pH_{PZC} determination.

2. Preparation and Characterisation of Adsorbent

The mahogany sawdust was obtained from the wood industry, and it was washed with water and dried under sunlight. The dried mahogany sawdust was then dried using an oven with a temperature of 200ºC. Next, the mahogany sawdust was crushed with a mortar and then sieved using a 100 mesh size. Finally, the obtained mahogany sawdust was ready as an adsorbent.

The determination of pH_{PZC} was conducted using NaCl solution (50 ml, 0,01 M) and 0.5 g adsorbent. The solution and adsorbent were shaken in a shaker with 120 rpm of agitation within 8 hours at room temperature. pH that was used was in the range of 2 to 12. The functional groups available on the adsorbents were studied using Fourier Transform Infrared Spectroscopy (FTIR Spectrometer iS10). In addition, the morphology of the adsorbents was studied using scanning electron microscopy (Inspect-S50).

3. Batch Adsorption Experiments

Variations of pH used in this work were 2, 3, 4, 5, 6, and 7. 100 ml of 50 mg/L Pb (II) solutions were used. The amount of adsorbent used was 1 g. Then, the solutions were shaken at 120 rpm at room temperature for 90 minutes. The lead concentration of sample solutions was analyzed by Atomic Absorption Spectrophotometry (Shimadzu AA-700). Variations of contact time used in this work were 5, 10, 15, 30, 60, 90, 120, and 150 minutes. The pH of the solution was fixed at 6, and the amount of the adsorbent used was 1g.

4. Adsorption Isotherm

This work used the isotherms model to study the adsorption isotherm: Langmuir and Freundlich. Langmuir model was given with equation as follows:

$$\frac{Ce}{qe} = \frac{1}{qm\,KL} + \frac{1}{qm}\,Ce \quad (1)$$

Freundlich model was given with equation as follows:

$$\log qe = logKf + \frac{1}{n}\log Ce \quad (2)$$

where:

qe : mass of adsorbate that was adsorbed in equilibrium (mg/g)

qm : capacity of maximum adsorption (mg/g)

Ce : concentration of adsorbate in bulk solution at equilibrium (mg/L)

KL : constant of Langmuir

Kf : constant of Freundlich

5. Kinetics Model

The kinetics model was used to study the adsorption rate that occurred: pseudo-first-order and pseudo-second-order models. The following equation could determine the pseudo-first-order model:

$$log(q_e - q_t) = log q_e - \frac{k_1}{2,303}t \quad (3)$$

Pseudo-second-order model was given with equation as follows:

$$\frac{t}{qt} = \frac{1}{k_2\, qe^2} + \frac{1}{qe}t \quad (4)$$

where:

qe = mass of adsorbate that was adsorbed in equilibrium (mg/g)

qt = mass of adsorbate which was adsorbed at time t (mg/g)

t = time of adsorption (min)

k_1 = pseudo first order adsorption rate (min^{-1})

k_2 = pseudo second order adsorption rate (min^{-1})

6. Data Analysis

Pb (II) ions removal efficiency was calculated based on the equation as follows:

$$\text{Efficiency (\%)} = \frac{Co-C}{Co} x\ 100\% \quad (5)$$

where Co was the initial concentration of Pb (II) ions (mg/L), and C expressed the concentration of Pb (II) ions (mg/L) after adsorption.

RESULTS AND DISCUSSION

1. Effect of Initial pH on Pb (II) Ions Removal Efficiency

There was an increase in Pb (II) ions removal efficiency from pH of 2 to 6 (Figure 1). Based on the results of the conducted experiments, the removal efficiencies of Pb (II) ions at pH of 2, 3, 4, 5, and 6 were 34.07%, 48.35%, and 55.80%, 62.45%, and 72.82%, respectively. The lowest removal efficiency of Pb (II) ions found was 34.07% at pH two, and the highest removal efficiency of Pb (II) ions obtained was 72.82% at pH 6.

pH is an important factor in the adsorption of heavy metals. The pH of the solution affects the ability to remove heavy metals because pH affects the speciation of heavy metals in solution, the chemical state of the active group of the adsorbent (protonation-deprotonation) [14]. pH affects the surface charge of the adsorbent and the speciation of heavy metals [15,16]**.** pH has an influence on the surface charge of the adsorbent and the solubility of metal ions in the solution. At low pH, there was an important number of H^+ ions on the surface of the adsorbent. This situation led the surface of the adsorbent to be covered by H^+ ions so that it caused a repulsive force against Pb (II) ions. There was a competition of H^+ ions with Pb (II) ions in the interaction with the site activities of the adsorbent. The more acidic the solution, the lower heavy metals ions removal. This can be seen from the low removal efficiency of Pb (II) ions at pH 2; the removal efficiency obtained was 34.07%.

At higher pH of Pb (II) solution, there was a decrease of H^+ ions concentration that caused the surface of the adsorbent to be less positive so that the adsorbent became easier to adsorb Pb (II) ions compared with that at the lower pH. At high pH, the hydrogen ion concentration decreases, this provides the surface charge of the adsorbent to be negative, so it increases the adsorption of positively charged heavy metals ions [17]. It can be seen that the highest removal efficiency of Pb (II) ions at pH 6, the removal efficiency was 72.82%. Hence, the optimum pH based on this research was 6, which had the highest removal efficiency.

The present work results are in line with the removal of Pb (II) ions using Dibetou sawdust [18]**.** The higher the pH, the higher the lead ions removal. Lead ions removal efficiency increased from pH 1 to pH 6. Using Dibetou sawdust without modification at pH 1 showed that lead ions removal was 48%. The highest lead ions removal was obtained at pH 6 with a removal of 78%. An increase in lead ions removal was also obtained with the use of modified Dibetou sawdust.

The use of *Eucalyptus globulus* sawdust to remove lead ions has the same trend with the results of this work [19]**.** The increase of lead ions removal increased from pH 1 to pH 4. As the pH increased, functional groups deprotonated from the adsorbent so that the active site of the adsorbent more easily bind to the adsorbate. With increasing pH, the functional group (-OH) in sawdust is deprotonated and causes the functional group to be negatively charged so that it has the ability to bind heavy metals [20].

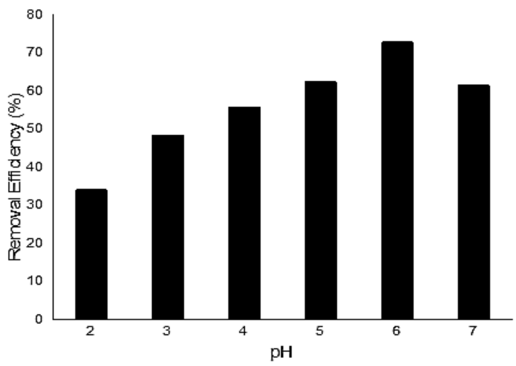

Figure 1. Effect of Initial pH on Pb (II) Ions Removal Efficiency

2. Effect of Contact Time on Pb (II) Ions Removal Efficiency

The removal efficiencies of Pb (II) ions with contact time of 5, 10, 15, 20, 30, 60, 90, 120 and 150 minutes were 68.33%, 69.80%, 75.80%, 84.87%, 82.00%, 79.48%, 78.48% and 70.89% respectively (Figure 2). The lowest removal efficiency of Pb (II) ions obtained was 68.33% at contact time of 5 minutes and the highest efficiency removal of Pb (II) ions obtained was 84.87% at contact time of 20 minutes.

The results of the experiments showed that the removal efficiency of Pb (II) ions with mahogany sawdust increased sharply in the beginning of the process (contact times to 30 minutes), then it decreased slightly after 30 minutes of contact time.

This study showed that at the initial contact time (until 30 minutes), the adsorbent experienced maximum adsorption and then the Pb (II) ions removal decreased after 30 minutes of contact time. This was due to the reduced adsorption capacity at the active site of the adsorbent so that the Pb (II) ions removal efficiency decreased. Pb (II) ions adsorption decreased due to the saturation of the adsorbent used, Pb (II) ions were released back into the solution. At the beginning of adsorption, there is high interaction between the adsorbate and the adsorbent; after a certain time, heavy metals accumulate on the surface of the adsorbent. The adsorption rate becomes slow until it reaches equilibrium (saturation on the surface of the adsorbent) [8,16]. The decrease in adsorption can occur in batch mode adsorption due to the release of the adsorbate from the adsorbent because of agitation using during the process.

The use of *Picea smithiana* sawdust to remove Pb (II) ions yielded similar results. Pb (II) ions removal took place rapidly at the beginning of the process (10 minutes), then there was a slight increase in Pb (II) ions removal up to 60 minutes of contact time. After passing 60 minutes of contact time, the Pb (II) ions removal was relatively constant and decreased after 180 minutes of contact time [21].

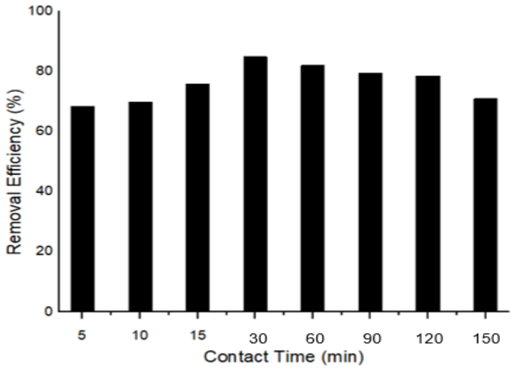

Figure 2. Effect of Contact Time on Pb (II) Ions Removal Efficiency

3. Adsorption Isotherms

The Langmuir isotherm adsorption model was carried out by plotting C_e versus C_e/q_e. The line equation obtained from the Langmuir adsorption model was y = 0.0672x + 1.2053. Therefore, the value of R^2 from the Langmuir adsorption model was equal to 0.9222. Linearization of the Langmuir model can be seen in Figure 3.

The Freundlich isotherm adsorption model was carried out by plotting log C_e versus log q_e. The line equation obtained was y = 0.5714x + 0.0732 with R^2 = 0.9941. Based on the results of these studies, the graph of the log Ce relationship with the log qe has R^2 value that was close to 1. Linearization of the Freundlich model is presented in Figure 4.

The experimental data from this work could fit the Freundlich isotherm better than the Langmuir isotherm. It means that the adsorption of lead ions into mahogany sawdust implies multilayer adsorption. Another study that reported the adsorption of lead into poplar sawdust revealed that the adsorption process followed the Langmuir model [22]. These two works showed that the adsorption isotherm of lead ions into sawdust can be varied from one adsorbent to another. The adsorption of heavy metals into sawdust is not unique [8]. The model parameters of this work are tabulated in Table 1.

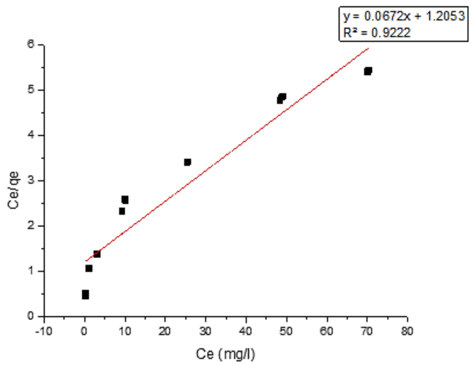

Figure 3. Linearization of Langmuir Model for Mahogany Sawdust

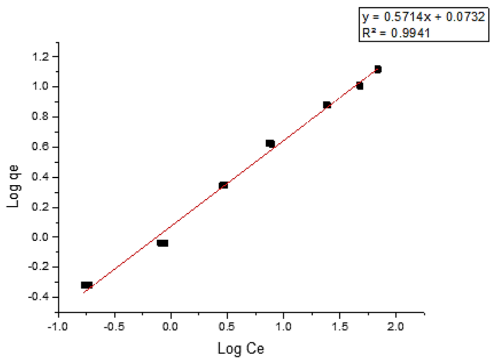

Figure 4. Linearization of Freundlich Model for Mahogany Sawdust

Table 1. Langmuir and Freundlich Isotherm Constants for Pb (II) Ions Removal onto Mahogany Sawdust

Langmuir Model			Freundlich Model		
q_{max} (mg/g)	K_L (L/g)	R^2	n	K_F (mg/L)	R^2
14.66	0.07	0.9222	1.75	1.18	0.9941

4. Adsorption Kinetics

Adsorption kinetics are used to determine the rate of determining the step of adsorption of any material or pollutants into the specified adsorbent. Pseudo first order and pseudo-second-order kinetic models were used to test the data. The pseudo-first-order model describes the adsorption based on solid adsorption capacity [23]. At the same time, the pseudo-second-order model assumes that the adsorption capacity is proportionally dependent on the number of active sites of the adsorbent [23]. This work showed that the pseudo-second-order obtained a better fit.

Furthermore, the pseudo-second-order model was better suited because its R^2 value was higher (0.9931) than the pseudo-first-order model (0.0969). This suggests that the rate-limiting step can be chemical sorption involving valence forces through sharing or exchanging electrons between heavy metals ions and the adsorbent [24]. Similar results have been reported for the adsorption of lead using other adsorbents [25–27].

The pseudo-first-order adsorption kinetics model gave a non-linear result. The rate of adsorption that occurred was not proportional to or greater than the amount of adsorbed material. The first-order kinetic model plot can be seen in Figure 5. The pseudo second-order adsorption kinetics model was done by plotting between t/qt versus t. The line equation obtained was y = 0.2773x + 0.5939 with R^2 value of 0.9931. The second-order kinetic model plot can be seen in Figure 6. Based on the results of this work, it indicated that the data was fitted best to the pseudo-second-order kinetic model. Kinetics parameters for lead ions adsorption are tabulated in Table 2.

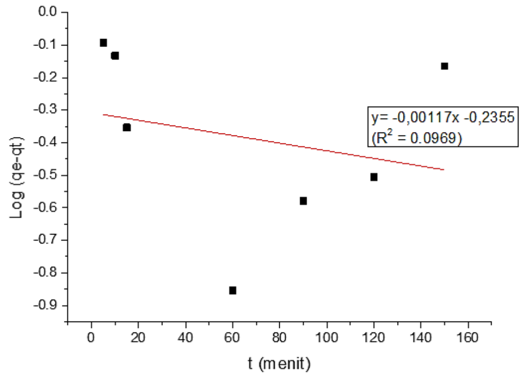

Figure 5. Pseudo First Order Model Plot

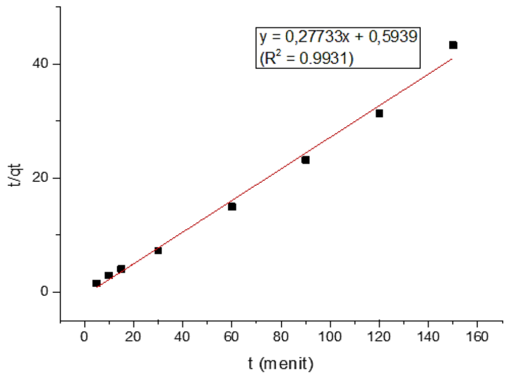

Figure 6. Pseudo Second Order Model Plot

Table 2. Kinetics Parameters for Lead Ions Adsorption onto Mahogany Sawdust

Langmuir Model			Freundlich Model		
q_e (mg/g)	K (L/min)	R^2	q_e (mg/g)	K (g/mg.min)	R^2
0.58	0.004	0.0969	3.6	0.13	0.9931

5. pH_{PZC} (pH Point of Zero Charge) of Adsorbent

Based on the experiment, pH_{PZC} of the adsorbent was 5.63. At pH under 5.63, the adsorbent had a positive charge, while pH was above 5.63, the adsorbent had a negative charge. Therefore, theoretically, adsorption Pb (II) ions would be better implemented at pH above pH_{PZC} of adsorbent. However, there was a limit to precipitation of Pb (II) ions when adsorption carried out at pH above pH_{PZC} of adsorbent (hydroxide of solution of the Pb (II) ions was to be formed and Pb (II) ions ion would be precipitated as Pb $(OH)_2$). This indicates that adsorption is better carried out at below pH_{PZC} of the adsorbent. The pH_{PZC} of mahogany sawdust can be seen in Figure 7.

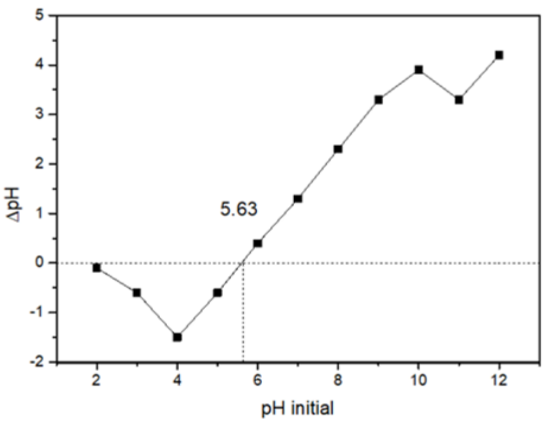

Figure 7. pHpzc of Mahogany Sawdust

6. FTIR Analysis

FTIR analysis was performed on adsorbent samples before and after contact with Pb (II) ions to study functional groups in the adsorbent and their role in removing Pb (II) ions.

From Figure 8, it can be seen that there is no fat in mahogany sawdust, but there are carbohydrates derived from cellulose. The transmittance of FTIR spectra of mahogany sawdust showed a different pattern and had distinctive characteristics. Mahogany sawdust had a specific functional group: OH, -CH, and C-O. The stretching vibration of the -OH hydroxyl group of alcohol from cellulose structure occurred at 3,337 cm^{-1} [28–30], and the absorption at 2,897 cm^{-1} indicated the presence of $-CH_2-$. [29]. The absorption at 1,508 cm^{-1} and 1,458 cm^{-1} indicated the presence of a C=C aromatic type of lignin which was the characteristic of wood having hard properties [30]. The wave peaks at wavenumber 1,029 cm^{-1} indicated vibration of C-O group [28]–[30].

The FTIR spectra of the adsorbent after contact with the Pb (II) solution showed several changes in the peaks. Shifts of wavenumbers and intensity decreases occurred especially in the wavenumber under 600 cm^{-1}. Vibration in the region of 300-600 cm^{-1}, especially of 595, 518 cm^{-1}, showed the bond of Pb (II) with the alcoholic group of cellulose structures (Pb-O) [30].

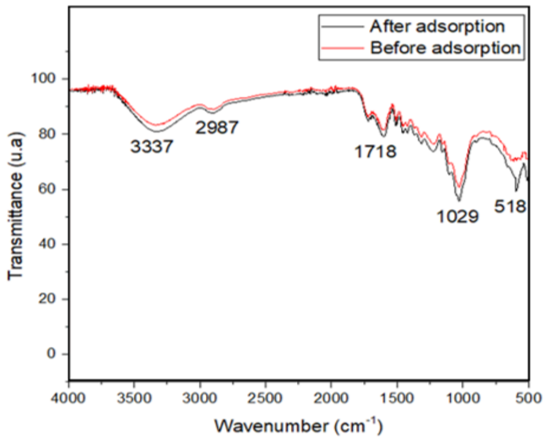

Figure 8. FTIR Spectra of Mahogany Sawdust

7. SEM Analysis

To study the surface morphology of mahogany sawdust, SEM analysis was used. The results of the analysis can be seen in Figure 9. Mahogany sawdust had an irregular shape, a mixture of sticks and wood chips. The existing forms of mahogany sawdust had a sheet-like surface shape with varying sizes. In the sheets, it was possible to interact with Pb (II) ions so that removal of Pb (II) ions occurred. Based on the results of SEM analysis, there is a heterogeneous structure because there are many wood constituents, such as cellulose, hemicellulose, and lignin which can adsorb heavy metals [18,31].

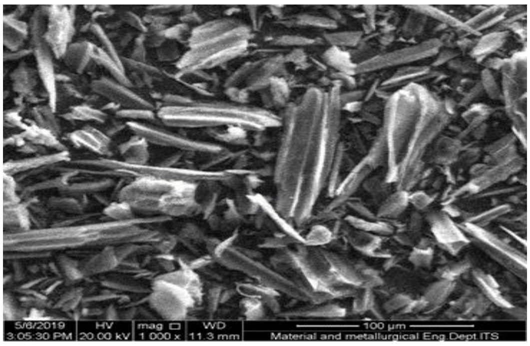

Figure 9. Morphology of Adsorbent Surface

8. Adsorption Mechanism

From the studies that have been conducted on the removal of Pb (II) ions with various sawdust-based adsorbent, it was reported that the removal of Pb (II) ions could occur because sawdust was able to form bonds with Pb (II) ions [18,19,21,22]. The functional group found in mahogany sawdust that can be involved in the removal of lead ions was -OH (hydroxyl group) of cellulose. In the -OH group there are free electrons on the O atom which act as electron donors to bond with metal ions through ion-dipole interactions [32]. The scheme of bond formation between Pb metal and functional groups contained in mahogany sawdust can be seen in Figure 10. As it was known that mahogany sawdust contains a large amount of cellulose, so mahogany sawdust could remove Pb (II) ions.

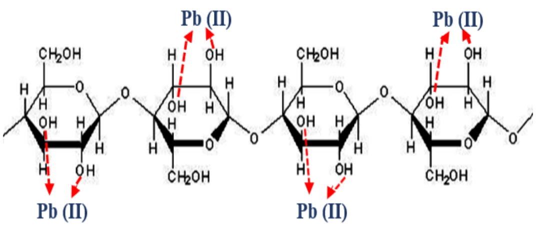

Figure 10. Possible Adsorption Mechanism of Pb (II) on to Mahogany Sawdust

CONCLUSION

This study shows that mahogany sawdust, as one of the common wastes, is a good adsorbent to remove Pb (II) ions from liquid waste. Adsorbent had pH_{PZC} of 5.63. Characterization of adsorbent using FTIR indicated a hydroxyl (-OH) group. It showed the presence of lignin, which was one characteristic of wood having properties hard and vibration of Pb-O interactions (under 600 cm^{-1}). Based on SEM analysis, it was found that the surface morphology of the adsorbent supported the interaction with Pb (II) ions. The removal of Pb (II) ions was affected by pH and contact time. In this study, pH 6 provided the highest removal while the contact time which provided the highest removal was 30 minutes. The removal of Pb (II) ions followed the Freundlich adsorption isotherm, while the adsorption kinetics followed the pseudo second-order model.

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