



## Utilization of Jengkol Peel (*Pithecellobium jiringa* (Jack) Prain) as Lead (II) Ions Bio-sorbent with Column Method

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### Abstract

Problems arising from laboratory waste include the content of lead metal ions in it, which can affect health. While on the other hand, Jengkol peel is a market waste and has no economic value. Jengkol peel contains hydroxyl groups and carboxylates to bind heavy metals such as lead (II) ions. So, this study aims to determine the effect of variations in flow rate, adsorbent height, and solution acidity and determine the ability of Jengkol peel in adsorbing lead (II) ions in the laboratory liquid by the column method. Organic groups that include active sides can be removed using 1N sodium hydroxide to increase the adsorption ability. Sodium hydroxide functions to dissolve lignin in lignocellulose compounds so that carboxylic groups are formed in cellulose, which has a role in binding metal ions. The determination of functional groups was carried out by Fourier-transform Infrared Spectroscopy. The identification of the adsorbent morphology was carried out by Scanning Electron Microscope. The adsorption of lead (II) ions by Jengkol peel was determined by calculating the difference between the lead (II) ions in the solution after and before passing through the column. The amount of lead metal remaining in solution was determined using Atomic Absorption Spectroscopy. Sodium hydroxide solution with a concentration of 1N can separate lignin from cellulose contained in lignocellulose to enhance the bio-sorbent ability of the Jengkol peel in binding metal ions. The smaller the flow rate in the column, the higher the amount of lead (II) ions adsorbed in the bio-sorbent. The acidity of the solution has the best adsorption at pH 5. The metal ions from the simulation sample can reduce the number of lead (II) metal ions adsorbed by 11%.

### 1. Introduction

A laboratory is a place to carry out various activities related to education, research, and community service. The activities carried out in the laboratory are very close to the use of chemicals that are acidic, corrosive, and toxic. According to the Government Regulation of the Republic of Indonesia Number 85 of 1999 concerning the treatment of Hazardous and Toxic Waste (B3), laboratory wastewater is classified as B3 waste. Laboratory waste contains hazardous compounds, one of which is heavy metals [1].

Lead (Pb) is a heavy metal that has a high level of toxicity and is detrimental to the environment because it cannot biodegrade and settle in nature. The presence of

lead in the human body is often called “lead poisoning” and results in disruption of the central nerves and causes behavioral disorders [2].

Jengkol peel is market waste and has no economic value. Plant fibers, such as Jengkol peel can be applied as bio-sorbent material with a little cost and are available in large quantities in nature [3]. Jengkol peel can adsorb metal ions due to the presence of carboxylic groups (e.g., hemicellulose, pectin, and lignin), phenolic (e.g., lignin), and mostly in hydroxyl groups (e.g., cellulose, hemicellulose, lignin, and pectin) [4].

The compound commonly used in the bio-sorbent activation process is sodium hydroxide. The use of sodium hydroxide in the activation process can eliminate

low-grade organic compounds and produce binding sites to increase the binding affinity of metal ions [5]. In bio-sorbents activated using sodium hydroxide, there is a decrease in the intensity of the C=C aromatic group and the loss of the C-O-C ether group, which shows delignification [6]. Delignification is the process of terminating lignin from a lignocellulose complex because lignin can cover the hydroxyl group from cellulose in bio-sorbents [7]. Hydroxyl groups of cellulose are opened, it can be increasing the ability of the bio-sorbent surface to bind to metals ion. The interaction between metal ions and hydroxyl groups occurs through *Van der Waals* interactions [8]. Besides, the separation of lignin from cellulose compounds causes the formation of carboxylic groups in all cellulose compounds. This carboxylic group plays a role in the binding of metal ions because it has free-electron bonds [9, 10].

The adsorption system can be carried out in batches or columns. A batch system has a disadvantage of not being able to be carried out on wastes containing continuous heavy metal ions [11]. Jengkol peel using a batch system can adsorb lead (II) ions with an adsorbed percent ion of 60.694% [12]. Studies of metal ion adsorption by batch systems using biomass have been numerous [13, 14, 15, 16, 17]. In adsorption using a column system, to obtain optimal adsorption results, the solution is always contacted with the adsorbent so that the column size dramatically affects the contact time between the solution and the adsorbent. Therefore, this column system is more advantageous because it generally has a higher capacity than a batch system [18]. The column system is the most practical in aqueous solution [19] and wastewater treatment [20]. This study aims to determine the bio-sorbent ability of Jengkol peel in adsorbing Pb (II) metal ions using the column method. This study was carried out by varying operating parameters such as bio-sorbent height, flow rate, the acidity of metal solutions, and other metals' influence.

## 2. Methodology

### 2.1. Plant Determination

The determination of Jengkol plants was carried out at the Laboratory of Plant Taxonomy, Department of Biology, Faculty of Mathematics and Natural Sciences, Padjadjaran University. The determination aims to ensure the identity of the plants used.

### 2.2. Sample Preparation

Jengkol peel waste obtained was thoroughly washed, and then the drying process was carried out using an oven to dry. Then the dried Jengkol peel was crushed by grinding it so that its size becomes smaller. The Jengkol peel was sieved using a 100-mesh sieve and stored in a tightly closed glass bottle. The characteristics of sample parameters were carried out, which included an examination of water content, drying losses, total ash content, acid insoluble ash content, water-soluble ash content, water-soluble extract content, and ethanol-soluble extract content.

### 2.3. Bio-sorbent Activation

Jengkol peel powder of 20 mg was added with 1 mL of 1 N sodium hydroxide solution, then stirred using a magnetic stirrer at room temperature for 30 minutes. Then let stand for 24 hours and filtered, then washed using distilled water until neutral pH. Jengkol peel powder, which has been neutralized, was then put into the oven at a temperature of 70°C until a dry powder was obtained. Before activation and after activation, the Jengkol peel bio-sorbent was characterized using Fourier Transform Infra-Red (FTIR) and Scanning Electron Microscope (SEM).

### 2.4. Height Variation of Bio-sorbent Test

In this study, the height of the adsorbent in the column was varied to 7, 10, and 14 cm. Then into the column, lead nitrate ( $\text{Pb}(\text{NO}_3)_2$ ) solution flowed. The filtrate was collected and analyzed using Atomic Absorption Spectroscopy (AAS).

### 2.5. The Variation of Flow Rate Test

Five grams of bio-sorbent was added to the column. The flow rate into the column was varied to 0.5, 1.0, and 2.0 L/min, and a solution of  $\text{Pb}(\text{NO}_3)_2$ . The filtrate was collected and analyzed using Atomic Absorption Spectroscopy (AAS).

### 2.6. Variation in the acidity of the test solution

Five grams of bio-sorbent were included in the column then poured  $\text{Pb}(\text{NO}_3)_2$  solution with a pH of 5, 7, and 8. The filtrate was collected and analyzed using Atomic Absorption Spectroscopy (AAS).

### 2.7. Effect of Other Metals on the Bio-sorbent Adsorption

A solution containing cadmium and silver ions with a concentration of 5 ppm was added to the lead (II) solution, then flowed into the bio-sorbent from Jengkol peel. The filtrate was taken and analyzed using an Atomic Absorption Spectrophotometer to determine the amount of lead (II) ions left in solution.

## 3. Results and Discussion

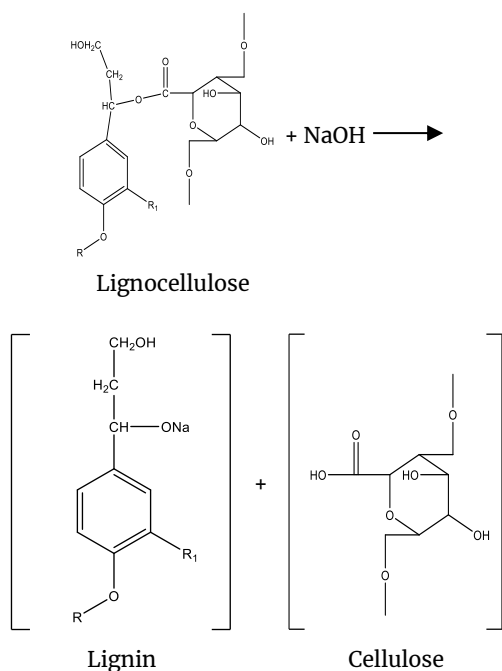
Based on the determination results, it was found that the sample used in this study was Jengkol skin, with the Latin name *Pithecellobium jiringa* (Jack) Prain. Before being used as bio-sorbents, checking the parameters of the Jengkol peel sample was performed first, to determine the quality characteristics of sample until a good quality sample was obtained. The results of the inspection data can be seen in table 1.

To increase the absorption ability of lead (II) metal ions by Jengkol peel, the Jengkol peel activation process needs to be carried out first. Jengkol peel activation as bio-sorbent aims to enlarge the pore so that its surface area increases and affects its adsorption power [21, 22]. Also, the activation process is carried out to increase the active side involved in the adsorption process [23]. The activation process of Jengkol peel bio-sorbent was carried out using sodium hydroxide solution with a concentration of 1 N. Bio-sorbent activation was carried out by soaking

for 24 hours. Soaking using a 1 N sodium hydroxide solution aims to make delignification in the adsorbent [24] to increase adsorption activity [25].

**Table 1.** The character of sample [23, 26]

Parameter	Content (%)	Terms (%) <sup>*</sup>
Water content	6.00	< 10.00
Shrinkage drying	7.45	< 10.00
Total ash content	20.42	> 15.65
Acid insoluble ash content	14.45	> 4.70
Water-soluble ash content	9.62	< 10.00
Water-soluble extract content	21.44	> 13.45
Soluble level of ethanol	30.29	> 16.50

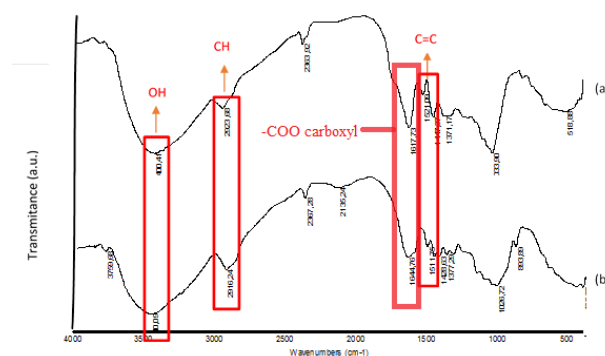


**Figure 1.** Mechanism of bonding termination between lignin and cellulose using sodium hydroxide [27]

Lignin found in Jengkol peel can be removed using a sodium hydroxide solution called delignification (Figure 1). Jengkol peel contains cellulose of (37%), hemicellulose, and lignin (14%) [24]. The presence of lignin prevents ion transfer to the active site of the adsorbent. The solution of sodium hydroxide breaks the cellulose bond with lignin. The OH<sup>-</sup> ion from sodium hydroxide breaks the bonds of the basic structure of lignin so that the lignin will dissolve easily [8] while the Na<sup>+</sup> ion binds to lignin, which forms sodium phenolate. This phenolic salt is soluble. Dissolved lignin is marked black in a solution called black liquor [28].

The Jengkol peel bio-sorbent before and after activation is characterized by the Fourier-Transform Infrared (FTIR) spectroscopy. The aim is to identify the presence of functional groups of bio-sorbents. The results of the characterization of Jengkol peel before and after activation can be seen in Figure 2. The results of Jengkol peel characterization showed the absorption at wavenumbers of 3400.41 cm<sup>-1</sup> (before activation) and 3440.09 cm<sup>-1</sup> (after activation), which are typical of the

hydroxyl group absorption. The absorption of alkyl groups (-CH) was observed at wavenumbers of 2923.68 cm<sup>-1</sup> and 2916.24 cm<sup>-1</sup>. The -C=C function group of the lignin compound is still identified in the bio-sorbent after being activated using 1N sodium hydroxide. This is indicated by the absorption of the wavenumber of 1511.76 cm<sup>-1</sup>. However, the transmittance increased from 61.251% to 67.293%, which reduced the C=C lignin group's intensity. The reduction is because the lignin compound has been partially successfully dissolved or treated with cellulose compounds using sodium hydroxide solution. Carboxylate groups were observed at wavenumbers of 1617.73 cm<sup>-1</sup> (before activation) and 1644.76 cm<sup>-1</sup> (after activation). The shift of the wavenumber towards larger wavenumbers also occurs in the carboxylic group. Shifting the wavenumbers towards more extended wavenumbers indicates an increase in the energy and bond strength [12, 29, 30].

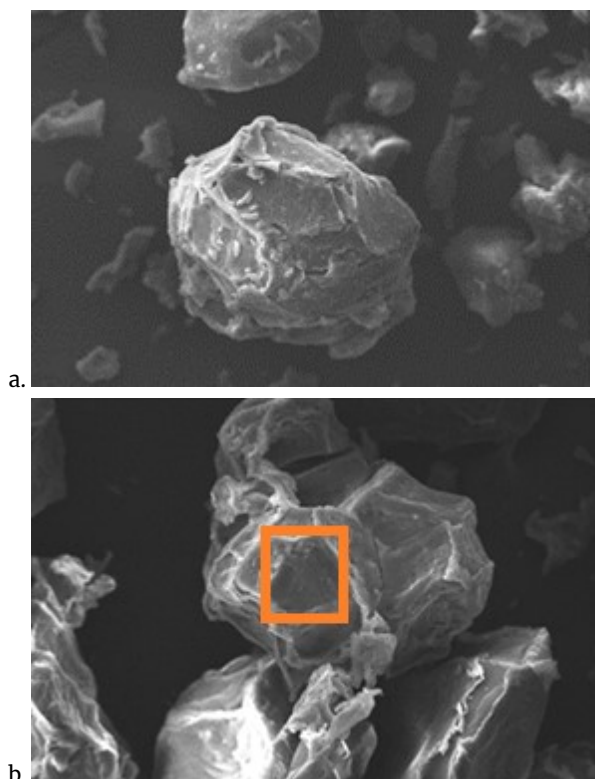


**Figure 2.** Spectra of FTIR Jengkol peel before (a) and after (b) activation with NaOH 1 N

The Jengkol peel bio-sorbent surface can be determined using a Scanning Electron Microscope (SEM). It aims to know the morphology, including the shape and size of the Jengkol peel bio-sorbent pore. The results of the characterization of Jengkol peel before and after activation are displayed in Figure 3.

SEM observation of Jengkol peel before activation is shown in Figure 3 (a) having a non-uniform particle size, and the pores have not been formed yet. This is because the activation process has not been carried out so that the surface shape is closed, which causes the morphology of the adsorbent not to form pores. SEM observation of bio-sorbent of Jengkol peel after activation using sodium hydroxide is shown in Figure 3 (b). In bio-sorbent (b), the pores are increasingly exposed, spread on the surface, and bio-sorbent cavity walls of Jengkol peel. If the adsorbent is clean of impurities, the pores will increase, and the surface area will increase. Open the active site of the adsorbent because it has been activated by 1N sodium hydroxide. Pores are formed because lignin is released from the bio-sorbent material, as previously described. Pores that are formed are not very visible, and this is because activation with sodium hydroxide solution is only able to release lignin compounds from lignocellulose to cellulose. Some mineral salts absorbed by plants through the roots may still not be separated from the Jengkol peel. One can overcome by adding acid in the activation process

so that it can release mineral salts that are still bound to bio-sorbent [8].

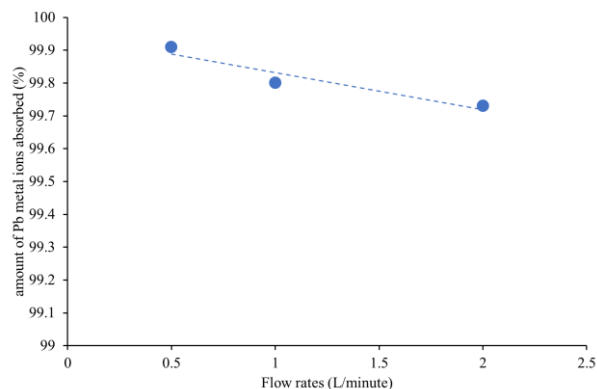


**Figure 3.** Results of SEM characteristics Jengkol peel before (a) and after (b) being activated with NaOH 1 N with 500x magnification.

The adsorbent height variation aims to determine the effect of the adsorbent height on the absorption of Pb (II) ions, which is applied with various modifications of the adsorbent height, 7; 10; and 14 cm. At each adsorbent height, the amount of Pb (II) ions absorbed is 99.06; 98.55; and 99.91%, respectively. From these results, it is known that the adsorbent with a height of 14 cm has the highest adsorption power compared to the other adsorbent height. This result is due to the adsorbent having a higher volume of adsorbent so that it has a greater number of particles. Thus, the bio-sorbent surface, which interacts with lead (II) ions, is more excellent. The rule of lead (II) ion adsorption by bio-sorbents from Jengkol peel follows the Freundlich equation model [31], which means that the adsorption process that occurs is physisorption [32]. Freundlich Isothermal adsorption occurs on very different and multilayer surfaces [33]. Interactions that occur in physical adsorption are intermolecular interactions such as Van der Waals and hydrogen bonds. The height of adsorbent is one of the factors that will affect the adsorption process [34]. The adsorption process will increase with increasing height from the adsorbent because this determines the need for the amount of adsorbent.

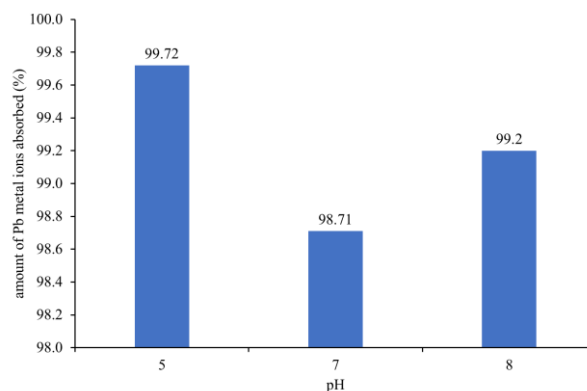
The variation in flow rate aims to determine the effect of the solution flow rate on the absorption of Pb (II) ions which are applied with a variety of flow rates, which is 0.5; 1; and 2 L/minute by flowing a solution of Pb (II) ions in the column. At each of these flow rates, the

number of Pb (II) ions absorbed is 99.91; 99.80; and 99.73%, respectively (Figure 4). From these results, it is known that the adsorbent with a flow rate of 0.5 L/minute has the highest adsorption power compared to other flow rates. This is because the lower the flow rate, the more Pb (II) ions are absorbed. This condition is caused by a lower flow rate, the longer the contact time between the ions in the water, the more ions are adsorbed.



**Figure 4.** The relationship between the amount of lead (II) ions adsorbed on the flow rates

The variation of acidity of the solution aims to determine the effect of the pH of the solution on the absorption of Pb (II) ions, which are applied various pHs of the solution, which is 5; 7; 8 by flowing Pb (II) ion solution in the column. The result of the concentration of Pb (II) ions absorbed is 99.79; 98.71; and 99.22%, respectively (Figure 5). Adsorption is better at higher acidity levels because, at this pH, a more significant ionization occurs, and adsorption can occur if metals form ions and are bound by an active group on the Jengkol peel adsorbent [13].



**Figure 5.** The relationship between the amount of lead (II) ions adsorbed on the pH

The results obtained that at pH 5, the concentration of the adsorbed metal lead (II) ions increased. The lead nitrate solution used as a sample is very much affected by acidity. The pH value influences the shape of the lead ion. At pH 3–5, the lead solution will be in the main species of a solution of  $Pb^{2+}$ ,  $PbNO_3^+$ , and  $Pb(NO_3)_2^-$ . These species have the potential to be attached to active groups in bio-sorbent because they are in the form of aqueous solutions. At pH 6.3 and above, solid  $Pb(OH)_2$  deposits begin to form so that they cannot interact with the bio-sorbent active



site (Figure 6) [35]. Thus, the lead (II) metal ions will be more absorbed under acidic conditions. The amount of lead (II) ions absorbed in the bio-sorbent occurs at pH 8. The formation of solid  $Pb(OH)_2$  causes deposition under these conditions, which can be trapped in the bio-sorbent. Hence it does not flow through the column and the formation of  $Pb(OH)_2$  deposits, which cause the  $Pb^{2+}$  content in the filtrate to decrease [36]. Thus, the increase does not occur due to the absorption of metal ions by the bio-sorbent active side but occurs due to precipitation of lead (II) ions.

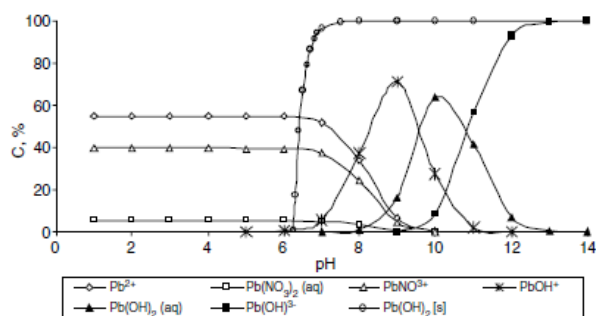
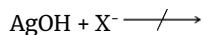


Figure 6. Speciation of single lead [35]

Simulation waste is made using the same concentration of 5 ppm so that the mixture becomes more homogeneous. Metal ions used are Cd (II), Ag (I), and Pb (II). This waste is made by mixing a solution of metal ions with the treatment process carried out at room temperature and adjusted to what is done on the adsorption of Jengkol peel to pure lead metal ions that had previously been carried out. The number of Pb (II) ions absorbed is 88.11%. There is a decrease in the amount of Pb (II) ion concentration absorbed due to competition between Cd and Pb [13].



Whereas, silver is not possible to compete with lead because when it reacts, it will turn to silver hydroxide, which is neutral.



Jengkol peel has functional groups such as cellulose, hemicellulose, and lignin, which contain the hydroxyl group, bound and can interact with the adsorbate component. The presence of hydroxyl groups on cellulose causes the adsorption power to be stronger in polar compounds.

#### 4. Conclusion

Based on the research that has been done, it can be concluded that the flow rate in the column that has the best absorption is 0.5 L/minute, and the best adsorbent height is 14 cm with percent adsorbed of 99.91%. On the contrary, the acidity of the solution has the best absorption at pH 5 with the percentage of adsorbed ions of 99.79% and the addition of other metal ions, i.e., cadmium (II) and silver metal ions from the simulation

sample has a percentage of absorption of Pb (II) ions of 88.11%.

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