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Study of Adsorption of Lead Metal (Pb) Using Chemical Activated Nipah Frond (Nypa Fruticans) Powder Biosorbent

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Abstract

Heavy metal contamination in the environment is now quite concerning, and it is hazardous if it enters the body. Continuous human interaction with heavy metal lead (Pb) will result in various health problems. Nipa palm is a palm (Palma) thrives in mangrove forest habitats or along the seashore, with a cellulose content of 35.1%, 26.4% hemicellulose, and 17.8% lignin. This research aims to see if palm fronds (Nypa fruticans) can be used as a biosorbent to remove the heavy metal Pb in artificial solutions. The analysis was carried out in phases, beginning with the creation of biosorbents from Nipah fronds by decreasing their size to powder and drying them in the sun, followed by analyzing the efficacy of the biosorbents and the number of functional groups using the FTIR instrument. The research was carried out by varying the particle size of the nipa palm frond adsorbent, namely 40, 60, and 80 mesh, as well as contact times of 30, 60, 90, and 100 minutes, to determine the level of adsorption absorption and the adsorption mechanism using the Langmuir and Freundlich isotherm equation approach. The Pb solution used had a concentration of 20 ppm. The results showed that the maximum metal absorption level was 99.29% at a particle size of 80 mesh with a contact time of 100 minutes. The absorption mechanism is close to the *Langmuir* isotherm equation with R2 = 0.9998. It is suspected that the adsorption process occurs in one layer (*monolayer*) of the adsorbent.

***Keywords**: Adsorption, Biosorbent, Nipah Midrib, Heavy Metals, Lead*

1. Introduction

Nypa fruticans, commonly known as the Nipa palm, is a palm species that thrive along the brackish coastlines of swampy areas. It is widely found in Indonesia, covering approximately 10% of the total tidal area, around 700,000 hectares. The regions with significant Nipa palm growth include the islands of Sumatra, Kalimantan, Sulawesi, Java, Maluku, and Jayapura [1]. The Nipa palm sheath contains various components, with the highest content being cellulose at 35.1%, lignin at 17.8%, and ash at 11.7%. Due to its composition, the Nipa palm shows excellent potential as a biosorbent for removing heavy metals from liquid waste.

Additionally, it has low economic value, making it highly suitable for large-scale utilization [2]**.** The processes that are widely used to absorb or remove heavy metal content contained in water bodies and liquid waste are physical and chemical processes which include precipitation, coagulation, ion exchange, and adsorption [3]. Among the various methods for removing heavy metals from water bodies and liquid waste, adsorption is the most commonly employed, as it is easy, cost-effective, and dominant. Activated carbon derived from plants, animals, and carbonaceous minerals is widely used as an adsorbent material [4] [5]**.**

Heavy metal pollution of the environment has become increasingly severe in recent years. Direct contact with these heavy metals can harm living things' health, and in extreme cases, it can even be fatal. The increase in waste production, especially from mining, industrial processes, and transportation, where large amounts of metals enter the environment, is mostly to blame for the growth in metal pollutants. The industrial sector, in particular, has the potential to produce the most trash since it uses raw materials and supporting materials that contain a variety of metal compounds and elements, which significantly increases the quantity of waste produced throughout the production process [6] [7].

The objective of the research [8] [9]was to synthesize and characterize activated carbon utilizing HCl as an activator from the Nypa palm fruit shell (*Nypa fruticans*). The analysis evaluated the amount of water, ash, and iodine adsorption [10]. It looked at the properties of *Nypa fruticans*, a type of palm leaf sheath, as a biosorbent for lowering mercury (Hg) levels in the water. Response surface methodology (RSM) was used by [11] to optimize the adsorption of Pb2+ ions onto activated carbon obtained from rice husk in a fixed bed column.

2. Literature Review

Several studies have indicated that the Nipa palm thrives in swampy areas with organic-rich soil, making it highly suitable as a biosorbent due to its cellulose, hemicellulose, and lignin content. Additionally, the Nipa palm possesses significant levels of inorganic elements such as Na, K, Cl, Mg, Ca, Si, P, S, and Al.

Biosorption is the ability of biological materials to accumulate heavy metals. This biosorption occurs due to a natural material called a biosorbent and a solution containing heavy metals, which can easily bind to the biosorbent. The presence of cellulose and hemicellulose content has significant potential to be used as adsorbents because the bound hydroxyl (OH) groups can interact with adsorbate components. The OH groups contribute to the polar nature of the adsorbent. Thus, cellulose and hemicellulose exhibit stronger adsorption of polar substances compared to less polar substances [12]**.** The absorption mechanism between the -OH groups bound to the surface and positively charged metal ions (cations) can occur through several processes, including ionic, complex, and ligand absorption.

Ionic absorption occurs when positively charged metal ions on the surface are absorbed by negatively charged -OH groups. In this process, ion exchange occurs between metal ions in solution and ions bound to a surface. Charge, size, and the intensity of the electrostatic interaction between metal ions and -OH groups all impact ionic absorption. Adsorption complexes develop when metal ions form complexes with -OH groups on the surface. Metal ions make coordinate bonds with -OH groups to generate chemical bonds. This mechanism is more common in metal ions that may form complexes with active groups, such as transition metal ions.

Ligand absorption happens when the surface's -OH group is a ligand and forms a compound with a metal ion. The -OH group can donate electrons and establish coordination bonds with metal ions, resulting in complexes [13]**.**

Lead, a Group IVA metal, is not quickly reactive. Lead has an oxidation state of +2 in compounds and is seldom oxidized to +4, as is more prevalent for the Group IVA elements above it. Organolead compounds, on the other hand, often have an oxidation state of +4. Lead is particularly toxic and detrimental to the nervous system because it inhibits the function of the body's enzymes. Information is hazardous since it can impede brain development and growth, particularly in children.

High density, malleability, softness, and corrosion resistance are among Lead's fundamental properties because of the passivation phenomena. Due to its face-centered cubic atomic configuration, which produces a high packing density and heavy nuclear weight, Lead has a high density (11.34 g/cm3). Several other metals, including iron (7.87 g/cm3), copper (8.93 g/cm3), and zinc (7.14 g/cm3), have densities that are lower than Lead's [14]**.**

Lead has an electrical resistivity (ρ) of 192 nanoohm-meters at 20 °C, which is almost ten times higher than metals commonly used in industries, such as copper (15.43 nΩ•m), gold (20.51 nΩ•m), and aluminum (24.15 nΩ•m). These figures indicate that Lead is a poorer electric current conductor than these metals. Lead has relatively low solubility, resulting in relatively low levels of Lead in water. Lead (Pb) entering water bodies results from human activities, including wastewater discharge from lead-related industries, black tin ore mining, waste from battery and fuel industries, and water transport activities. These waste discharges flow into waterways, leading to contamination [15]

3. Methods

3.1. Materials

This research utilized the following materials: Lead (Pb) solution with a concentration of 20 ppm, 5% H2SO4 (sulfuric acid), filter paper, distilled water (equates), and nipah palm fronds obtained from the waters of Kreung Geukeuh, North Aceh Regency. The equipment used in this study included glass beakers, measuring cylinders, volumetric flasks, a mixer, a grinder, a sieve with mesh sizes of 40, 60, and 80, a pH indicator, a magnetic heater, an oven, a shaker, an FTIR (Fourier Transform Infrared Spectroscopy) instrument (Shimadzu), and AAS (Atomic Absorption Spectrophotometer)

3.2. Preparation of Nypa Fronds

The Nypa fronds were cut into smaller pieces and cleaned with running water until thoroughly rinsed. Afterward, they were sun-dried until the moisture content was removed entirely. Subsequently, the drying process was continued using an oven at a temperature of 105°C until a constant weight was achieved. The dried samples were then pulverized into powder form and sieved using sieves with particle sizes of 40, 60, and 80 mesh.

3.3. Activation of Nypa Frond Powder

The obtained Nypa frond powder was subjected to a maceration process with methanol for eight days. The resulting mixture was filtered and dried in an oven at 70°C for 60 minutes. Subsequently, the activation process was carried out using a 5% H2SO4 solution for 2.5 hours, employing a magnetic stirrer at a temperature of 60°C [16]**.** The product was filtered and washed multiple times with distilled water until reaching a pH of 6. The next step involved drying the resulting powder for 4 hours at a temperature of 80°C. Furthermore, the adsorbent was characterized using FTIR (Shimadzu) analysis.

3.4. Lead Adsorption Using Nipah Palm Biosorbent

Put a sample of Lead (Pb) solution into four beaker glasses of 250 ml each. Then add 3 grams of nipa palm frond biosorbent measuring 40 mesh. Stir using a magnetic stirrer at 60 rpm with a contact time of 30, 60, 90, and 100 minutes for all solutions to be tested, then separate using filter paper. Repeat these steps with different sizes of biosorbent according to the variations, namely 60 and 80 mesh. The filtrate resulting from the absorption research treatment was then tested for the metal removal content using an AAS (Anatomic Adoption Spectrophotometer).

4. Result and Discussion

This study aimed to examine the effects of contact duration and particle size on the removal of Pb metal using nipa palm fronds as a biosorbent. Additionally, the FTIR instrument was used to examine the functional groups to determine the properties of the biosorbent.

Data, an absorption rate, and an adsorption isotherm are obtained as a result of the research that has been done. Analyzing the quantities of Lead (Pb) in waste produced by humans using a Skyray 6000 AAS (*Anatomic Adsorption Spectrophotometer*)

4.1. Effect of Contact Time on Absorption Capacity

The relationship between contact time and absorption capacity is needed because it will affect the capacity. Testing the absorption capacity of this contact time was carried out between the range of 30 minutes to 100 minutes with the use of 3 grams of nipa palm biosorbent. The results of absorption capacity research based on variations in contact time are shown in Figure 1.

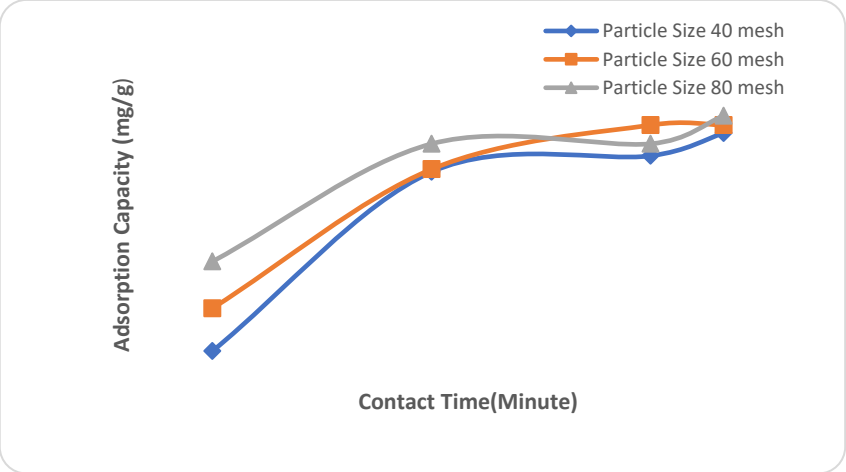

Fig 1. Graph of Relationship Between Contact Time and Absorption Capacity

The picture above illustrates the contact time of 100 minutes where there is an active absorption process and has not shown constant conditions. Like the previous absorption level, the increase in absorption power at all variations of contact time indicates that a cell surface is still active and forms bonds with metals [17]. Based on the study results, the capacity in terms of the influence of the contact time supplied to the adsorbent in lead (Pb) solution is displayed in the graph above.

The most excellent absorption capacity value was determined during a contact period of 100 minutes, with a yield of 1.6549 mg/gr (Figure 1). When the adsorbent reached the maximum duration in its removal, its efficiency and adsorption capacity dropped at a size of 80 mesh and a contact period of 90 minutes. This is due to desorption occurring when the contact period between the adsorbent and the adsorbate exceeds the optimum contact time. This symptom happens as a result of the adsorbent's surface becoming saturated, causing the adsorbed adsorbate molecules to return to the solution [18]. Desorption is removing the adsorbate from the surface of the adsorbent. This phenomenon occurs because the surface of the adsorbent has been saturated, so the adsorbate molecules that have been adsorbed back into the solution. And because there has also been an equilibrium between the adsorbate concentrations remaining in the solution [19].

4.2. Effect of Adsorbent Particle Size on Absorption Percent

The results of the absorption of lead metal with the particle size of the Nypa fruticans biosorbent powder with various variations can be seen in Figure 2.

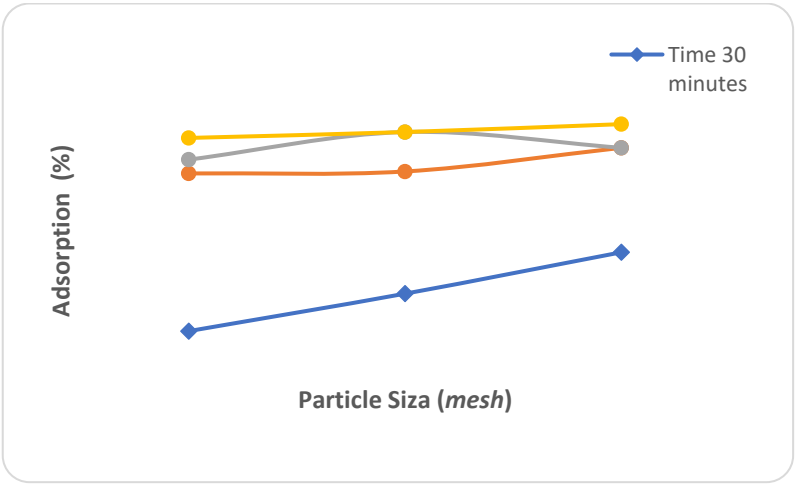

Fig 2. Graph of Particle Size and Absorption Percent

Figure 2 shows that particle size influences the proportion of absorption generated. At 100 minutes of contact time, the highest absorption of Pb metal utilizing biosorbent Nipa palm fronds occurred at a particle size of 80 mesh, with removal of 99.29%. A particle size of 40 mesh with a removal percentage of 98.24% and a contact period of 30 minutes has the lowest absorption percentage. This is consistent with the assumption that smaller particles result in a more extensive surface area. The absorption power produced will be greater if the surface area in contact with the biosorbent is big [20]. An efficient adsorbent requires a large surface area because many adsorption steps occur on the surface. The surface area can supply more active sites, increasing surface reactivity with the pollutant molecules. The surface

area is closely related to the pore size; the smaller the adsorbent's pore size, the greater the surface area, so the number of adsorbed molecules will increase [21].

4.3. Adsorption Isotherms

The adsorption process for Pb metal using Nypa fruticans petiole powder follows the Freundlich and Langmuir isotherm models. Both models are utilized to determine the appropriate adsorption process, and subsequently, the suitable model for Pb adsorption using Nypa fruticans petiole powder is determined. The models of Freundlich and Langmuir isotherms can be seen in Figures 3a and 3b

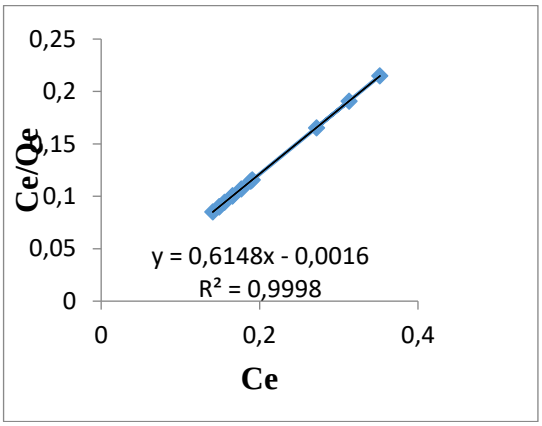

Fig 3 a. Grafik Isoterm Langmuir

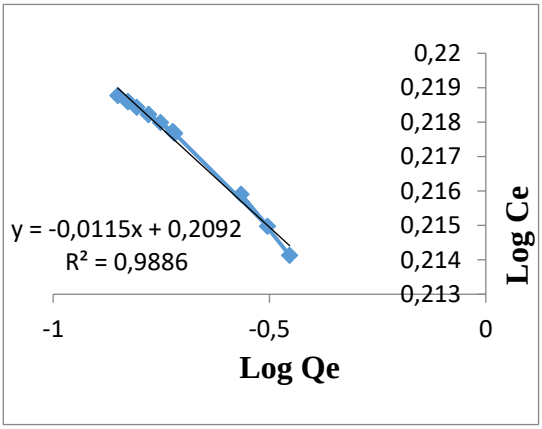

Fig 3 b. Grafik Isoterm Freumdlich

Figure 3 (a) is a Langmuir isotherm pattern curve that yields R2 = 0.9998. Meanwhile, Figure 3 (b) is a Freundlich isotherm pattern curve that produces R2 = 0.9886. It can be seen that the Langmuir and Freundlich equation curves have a different linearization. Based on these linear values, it can be concluded that the adsorption process of heavy metal Pb (Lead) using Nipah frond adsorbents follows the Langmuir Adsorption Isotherm model. Good linear results confirm this and have a coefficient of determination R closest to the value 1 [22]. The linear value for the Langmuir adsorption isotherm is higher than the Freundlich adsorption isotherm, so the Langmuir adsorption isotherm model is more appropriate to be used to characterize the adsorption mechanism of heavy metal Pb (Lead) absorption using nipah palm fiber adsorbents, which is thought to occur in the surface layer of the adsorbent (monolayer) [23]**.**

4.4. Characteristics of the Biosorbent

The characterization of the nipa palm fronds was carried out using an instrument, namely Fourier Transmittance Infrared Spectroscopy (FTIR). This analysis aims to determine the pattern of functional groups present in the adsorbent [24]. FT-IR analysis for the palm fronds' characteristics is 80 mesh size. This is because, based on research results [25], particle size does not affect the functional groups in the biosorbent. Functional group testing is performed qualitatively by interpreting the absorption peaks from the infrared spectrum. The resulting FT-IR analysis for the characteristics of the nipa palm frond biosorbent is shown in Figure 4

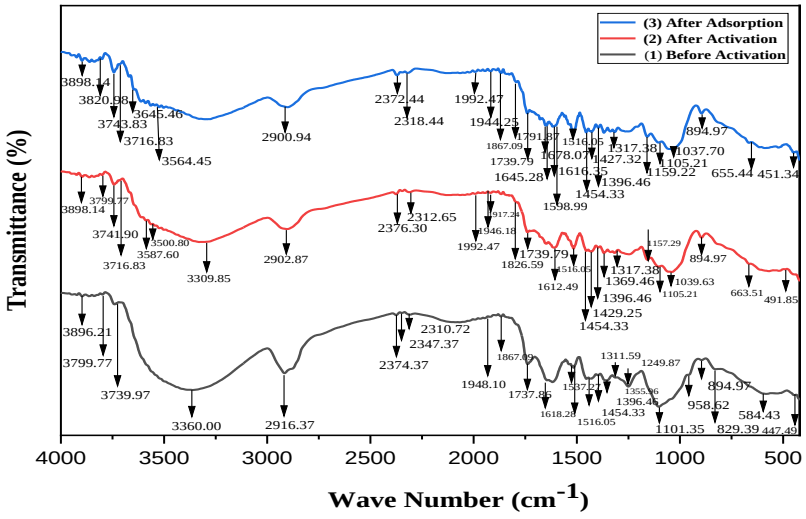

Fig 4. Graph of FTIR Analysis Before Activation, After Activation and After Adsorption

Figure 4 shows the functionality available for the Nipa palm frond biosorbent. Wavelength 3360.00; 3500.80; 3564.45 cm^{-1} describes the content of hydroxyl groups (O-H), following the explanation [25]**.** This explains that the O-H group is in the 3000 cm-1 – 3700-1 absorption range. In the absorption band, 2916.37; 2902.87; 2900.94 cm^{-1} illustrate the presence of the C-H functional group, which is the building block for the lignocellulosic structure according to research [26], which states that the absorption area is between 2853 cm^{-1} – 2962 cm^{-1}.

At a wavelength of 1039.63; 1037.70 cm^{-1} (after activation and after adsorption), it shows the presence of C-O functional, which ranges from 1000 cm^{-1} – 1100 cm^{-1}. In the Nipah frond powder adsorbent, the active group C=O has an absorption band of 1618.28, 1612.49, and 1616.35 cm^{-1}. This aligns with research [21], which reports that the C=O group is present in the absorption wave area of 1600 cm^{-1} – 1820 cm^{-1}. The O-H, C-H, and C=O groups in the palm frond biosorbent also have a N=O group at a wavelength of 1516.05 cm^{-1} and the CH3 functional group at 1396.46 cm^{-1}. The N=O and CH3 groups are following research [27] which informs that the N=O groups are found at 1500 cm^{-1}–1600 cm^{-1}, and the CH3 groups are found at 1375 cm^{-1} – 1450 cm^{-1}.
The picture also shows that there are wave changes. There is a shift in wave number and % T in the –OH functional group after activation. This indicates that the O-H bond has increased the bond and the wave area has increased due to a decrease in vibration. Therefore, there will be a shift in wave number and cause an increase in % T after activation occurs. The absorption rate decreases, and the formed radical compounds are unstable, which then react to form new compounds. Before the palm fronds are activated, absorption bands at 1000 cm^{-1} – 1100 cm^{-1} are not created. Still, absorption bands appear in the wave number region after the activation process occurs, which describes the C-O stretching vibrations. This happened because the activation process dissolved the impurities attached to the C-O group. The expected results illustrate that the biosorbent groups contain O-H, C-H, and C-O functional groups as the main constituents of cellulose [21]. The –OH group is a constituent component that can interact and adsorb heavy metals. The C=O group indicates the presence of lignin in nipa palm biosorbents contained therein [14]. And there are also impurities, namely N=O and CH3. The N=O content that appears is suspected to be an impurity originating from the nipa palm fronds, which cannot be removed so it can be read by absorption. The CH3 group is still present in it because residual reactions in the methanol maceration process still occur with the nipah fronds in the cellulose extraction.

5. Conclusions

Based on the results of research and discussion, the following conclusions can be obtained:
The maximum absorption rate in the biosorption of nipa palm fronds for Pb metal was obtained at 99.29% with a maximum contact time of 100 minutes and a full adsorbent particle size of 80 mesh.
The absorption that occurs using the nipa palm frond biosorbent for the heavy metal Pb refers to the Langmuir isotherm equation with R2 0.999, which suggests that absorption occurs in the surface layer of the adsorbent (monolayer), which occurs due to an ion exchange process.

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