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Characterization of HCl-activated Water Hyacinth (*Eichhornia crassipes*) Adsorbent in Removing Lead Ion (Pb^{2+}) from Battery Industry Effluent

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Abstract

This study utilizes the adsorption process to reduce the concentration of lead ions in battery industry wastewater through the use of adsorbents from water hyacinth (*Eichhornia crassipes*). The purpose of this research is to determine the ability of a bioadsorbent activated by a 0.1 M HCl solution to adsorb Pb^{2+} through FTIR and AAS characterization. The bioadsorbent was contacted into a standardized solution of Pb^{2+} with varying contact times (viz., 20, 30, 40, 50, 60, 70, 140, 210, and 280 to determine the maximum adsorption). The highest absorption of the Pb^{2+} in battery industry wastewater took place for 210 minutes. The absorption ability of the bioadsorbent was 94.45%, with 8.1488 ppm as the initial concentration. Then, this study shows that the characterization of this bioadsorbent before the activation of 0.1 M HCl has O-H, C-H, C=O, and C-O ether functional groups. All three identified the presence of cellulose. Post-activation, lignin, and hemicellulose disappeared due to the vibration of the C=O group. However, an increase in the intensity of the vibrational peak at the C-O group indicates the presence of carbon chain linking in cellulose. Finally, in the bioadsorbent, after contact with battery industry effluent, the loss of bending vibrations occurred because the H atoms in the functional groups had been substituted with Pb^{2+}.

Keywords: Adsorption, bioadsorbent, dried water hyacinth, removal lead ions, battery industry wastewater

INTRODUCTION

Batteries are devices that can convert chemical energy into electrical energy through electrochemical processes at the electrodes and are often used in everyday electronic equipment (Fadilah, 2015). Battery waste contains heavy metals such as mercury, manganese, lead, nickel, lithium, and cadmium that have the potential to pollute water and soil and endanger human health, so it is included in hazardous and toxic material waste (Nurannisa et al., 2021). Lead (Pb) is black lead, which is classified as a hazardous pollutant because it has high toxicity. If consumed in the long term, it can cause negative effects on health such as anemia, cancer, kidney disease, mental disorders, and death (Jazuli et al., 2022).

The heavy metals that are commonly found in effluent are Pb (Permana & Andhikawati, 2022). Because this metal is more widespread than most other toxic metals (Hendrasarie & Dieta, 2019), efforts need to be made to minimize the dangers of pollution from industrial wastewater containing heavy metals(Rahmi & Sajidah, 2018). Commonly used methods for reducing heavy metal levels in water are coagulation, flocculation, evaporation, ion exchange, membrane separation, and adsorption (Ekoputri et al., 2024; Fang et al., 2018; Moelyaningrum et al., 2018). The adsorption process is the main choice. Besides being more widely used in industry, adsorption has a simpler principle, is also cost-effective, and has relatively high effectiveness and efficiency without causing toxicity as a side effect (Ganing, 2022; Hanifa et al., 2023; Moelyaningrum et al., 2018).

Some types of adsorbent media that are commonly used are activated carbon, zeolite, silica gel, bentonite, chitosan, and natural adsorbents or bioadsorbents (Ifa et al., 2020; Sekewael et al., 2015). However, bioadsorbents are most often chosen because, besides being easy to find in the environment, bioadsorbents also have good adsorption capabilities so that they can

optimize the activation process, whose purpose is to increase the pore size of the adsorbent by breaking hydrocarbon bonds or oxidizing molecules on the adsorbent surface, resulting in a significant increase in surface area, which has an impact on its adsorption ability (Ngapa & Ika, 2020; Nurohmah et al., 2019). Based on the results of research by Nurhadi *et al*., (2019) on bioadsorbent activation using HCl (hydrochloric acid) and HNO_3 activators on water hyacinth, it was found that activation using HCl adsorbs faster, while activated carbon with HNO_3 activation adsorps more.

Water hyacinth (*Eichhornia crassipes*) is a type of aquatic weed plant known for its rapid growth. It is commonly found in shallow and turbid waters with temperatures ranging from 28 to 30^{o}C and pH 4 to 12 (Dita, 2021). This plant contains 60% cellulose, 8% hemicellulose, and 17% lignin (Nurhadi et al., 2019). (Mahmudah et al., 2023) selected water hyacinth as a bioadsorbent due to its high cellulose content, which effectively removes heavy metals such as iron (Fe), zinc (Zn), copper (Cu), chromium (Cr), cadmium (Cd), manganese (Mn), and lead (Pb). This cellulose has hydroxyl groups (OH^-) that can interact with metal ions, making it effective as a bioadsorbent in the adsorption process (Abdullah & Yustinah, 2020).

This research uses HCl (strong acid) as an activator. The adsorption mechanism that occurs in the characterization of HCl-activated adsorbents is reversible and allows for a regeneration process through an increase in H^+ ions as an acidifying agent of the adsorbent. The released metal ions will then dissolve in the acidic medium. The utilization of the solution is considered a very effective regeneration step (Rakhmania et al., 2017). The activation of adsorbents using HCl has been previously studied by (Fadhilah et al., 2021) with an adsorption percentage of 42.90%. Other results also show that the use of this solvent as a chemical activator can optimize the adsorption process because its ability to adsorb heavy metal ions is better than that of H_2SO_4 and HNO_3 (Huda et al., 2020).

This study aims to find out how well the functional groups in a water hyacinth adsorbent can soak up 0.1 M HCl. To do this, the sample will be characterized with Fourier transform InfraRed-FTIR and the percentage of adsorption ability will be measured with an atomic absorption spectrophotometer (AAS).

METHODOLOGY

Materials and Instrumentals

The materials utilized in this study comprised water hyacinth stems, 0.1 M HCl (Merck), deionized water, and battery industry effluent obtained from a local company in Karawang, Jawa Barat. The instrumental apparatus employed included a Fourier Transform Infrared (FTIR) spectrometer (Nicolet iS50 Thermo Scientific) for the identification of functional groups in the water hyacinth bioadsorbent and an Atomic Absorption Spectrophotometer (AAS) (Shimadzu AA-7000) to determine the residual concentration of lead ions.

Methods

Bioadsorbent production. In this research, water hyacinth stems were employed as the bioadsorbent material. The stems were initially washed, cut into small pieces, and dried in an oven at 150°C for 80 minutes. Subsequently, the dried water hyacinth stems were crushed using a crusher and sieved through a 100-mesh sieve shaker.

Activation using 0.1 M HCl. The activation process involved adding 25 g of water hyacinth powder to 350 mL of a 0.1 M HCl solution, which was then homogenized using a magnetic stirrer at a constant speed for 30 minutes and allowed to stand for 5 hours. This duration aims to ensure the activation process runs optimally in removing metal oxides that cover the pores of the bioadsorbent surface. Afterward, the water hyacinth sediment was filtered and neutralized with deionized water until the pH reached 7, followed by drying in an oven at 105°C for 2 hours. The activated water hyacinth bioadsorbent was then analyzed using FTIR to identify the functional groups present. The activation process aims to enhance adsorption capacity by physically or chemically removing impurities attached to the surface and pores of the adsorbent (Syahrir et al., 2020).

Preparation of a 20 ppm lead-ion standard solution. A 20-mL aliquot of 100 ppm Pb^{2+} standard solution was added to a 100-mL volumetric flask and diluted to the mark with deionized water. The initial concentration of lead ions was then analyzed using an AAS.

Determination of optimal contact time. Testing was conducted by adding 0.5 g of the HCl 0.1 M activated water hyacinth bioadsorbent to 100 mL of a 20 ppm Pb^{2+} standard solution in a 250 mL beaker glass, varying the contact time at intervals of 20, 30, 40, 50, and 60, 70, 140, 210, and 280 minutes to

determine the optimal contact time between the bioadsorbent and the lead solution. Stirring was performed using a simple magnetic stirrer during these intervals, followed by filtration through filter paper. The filtrate was then tested to ascertain the residual lead concentration using an AAS.

Adsorption testing for Pb^{2+} from battery industry effluent. The test involved adding 0.5 g of the HCl 0.1 M activated water hyacinth bioadsorbent to 100 mL of battery industry waste for an optimal contact time of 210 minutes. Stirring was carried out using a simple magnetic stirrer, followed by filtration through filter paper and drying in an oven at 105°C for 1 hour. The resultant filtrate was analyzed to determine the residual concentration present in the battery industry waste solution using an AAS. Meanwhile, the bioadsorbent residue was examined using FTIR to identify the functional groups present after the adsorption process.

Data Analysis

Determination of adsorption kinetics using pseudo-order. The adsorption rate indicates the adsorbent's ability to absorb the adsorbate. The adsorption rate can be known from the adsorption rate constant (*k*) and the reaction order resulting from the adsorption kinetics model (Anggriani et al., 2021). Time affects the rate of adsorption, which is the speed of adsorption from the adsorbent to the adsorbate, following adsorption kinetics. According to (Haryanto et al., 2019), the contact time required measures the adsorption rate in reaching equilibrium conditions.

In this study, testing the adsorption rate uses a pseudo-first-order model where the reaction speed depends on one of the reacting substances or is proportional to one power of the reactants. While the pseudo-second-order reaction rate is directly proportional to the product of the concentrations of the two reactants or directly proportional to the square of the concentration of one of the reactants (Aldiansyah et al., 2023), the pseudo-first-order kinetic model estimates the rate of metal sorption (Gupta et al., 2021) through the use of equations (1) to (3).

the adsorption capacity
$$q_e = \frac{(C_o - C_e)}{m} V \quad (1.1)$$

$$q_t = \frac{(C_o - C_t)}{m} V \quad (1.2)$$

the pseudo-first-order kinetic
$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (2)$$

the pseudo-second-order kinetic
$$\frac{t}{q_t} = \frac{1}{k_2 {q_e}^2} + \frac{1}{q_e} \quad (3)$$

The parameters q_e and q_t in units of mg/g represent the adsorption capacity at equilibrium and at time *t* (in minutes), while *R* represents the percentage of adsorbed metal. The values of C_o, C_e, and C_t, respectively, in units of mg/L, represent the concentrations of metal at initial, equilibrium, and time *t*. Consider the mass of the adsorbent *m* and the volume of the solution *V* in units of g and L. The values of k_1 and k_2 represent the rate constants for pseudo-first-order reactions (min^{-1}) and pseudo-second-order reactions (in $g.mg^{-1}.min^{-1}$), respectively, with *t* denoting the contact time (in minutes).

RESULTS AND DISCUSSION

HCl-Activated Water Hyacinth Bioadsorbent

In this study, water hyacinth stems, mashed to a weight of up to 25 g, serve as the bioadsorbent. The material is activated using a 0.1 M HCl solution in a volume of up to 350 mL. Using HCl as an activator is done to get rid of metal oxides that cover the pores for 5 hours. Figure 1 illustrates the bioadsorbent's appearance. This is due to the acidic nature of HCl, which can damage plant tissue and increase the pores during adsorption between the adsorbate and adsorbent (Arung et al., 2014).

Figure 1. Water hyacinth powder: a) before, and b) after activation

Characterization of Water Hyacinth Bioadsorbent

This study used a test known as Fourier Transform Infrared (FTIR) with a wavelength of 500–4000 cm^{-1} to identify the functional groups in the water hyacinth bioadsorbent. Figure 2 shows the results. Before activation, the study used the water hyacinth bioadsorbent to identify functional groups critical to

the adsorption process, as well as functional groups and structural changes that occur after 0.1 M HCl activation. Finally, the study uses samples after Pb^{2+} adsorption to identify functional groups and structural changes that occur once the metal adsorbed. The process exposes the sample to infrared radiation, influencing the molecular atoms' vibrations and causing a specific energy transfer. Therefore, FTIR helps determine the specific molecular vibrations contained in the sample (Nandiyanto et al., 2019).

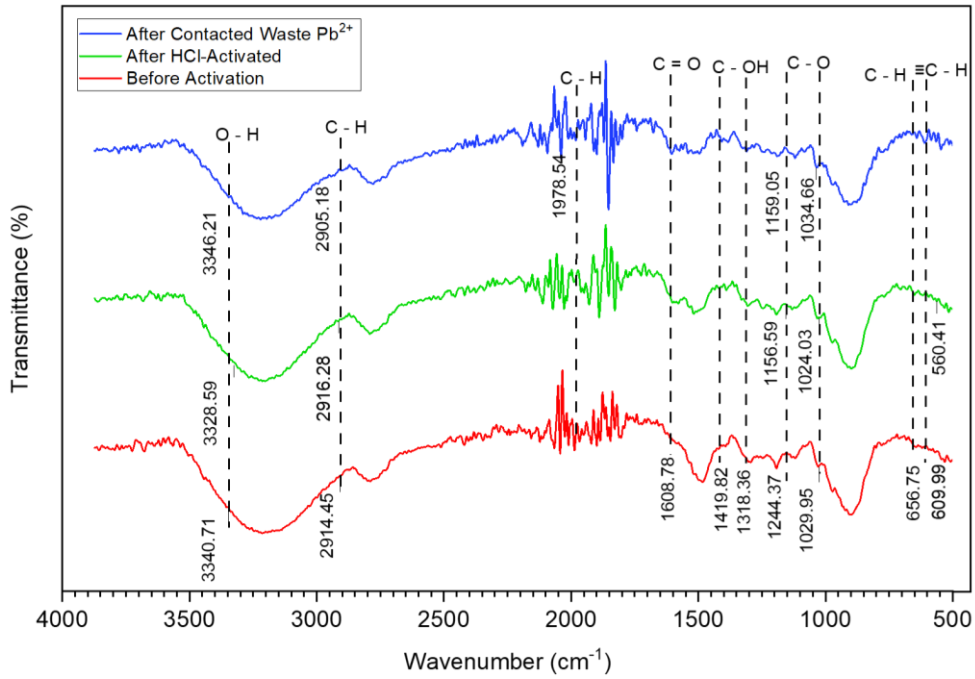

Figure 2. Comparison of spectra of functional groups in the water hyacinth bioadsorbent

Figure 2 illustrates that the adsorbent surface has large and wide absorption wave numbers. These numbers show that hydroxyl functional groups are present and working in the ion adsorption process. Hydroxyl groups specifically indicate the presence of lignin (Rakhmania et al., 2017). The analysis results reveal that covalent coordination bonds, at wavelengths 3340.71, 3328.59, and 3346.21 cm^{-1}, form the bond between metal ions and OH on cellulose. Metal ions hold empty orbitals that the O atom's free electrons in the OH group fill up to form a coordinated complex (Azhari et al., 2017).

Before activation, the water hyacinth bioadsorbent exhibited peaks in the O-H, C-H, C=O, and C-O ether functional groups. These three groups identify the presence of cellulose. According to (Kusumawardani et al., 2018), these vibrations occur when hydrogen bonds stretch and hydroxyl groups bend in the cellulose function. The addition of 0.1 M HCl causes the water hyacinth to lose its C=O functional group vibrations. This shows that lignin and hemicellulose are also breaking down. The change of hydroxyl and carboxyl groups into ester groups takes place and causes a decrease in the ability to adsorb ions (Ahdiyati, 2020). The appearance of a sharp peak at the C-O functional group indicates the presence of carbon chain links in cellulose. This states the presence of cellulose functional groups (Astari & Utami, 2018). Meanwhile, water hyacinth bioadsorbent, after contact with waste containing (Pb^{2+}), displays missing bending vibrations. This is because the vibration peaks at the O-H and C-H wave numbers are weaker rather than wider. This means that there is a break in the chain between lignin, hemicellulose, and cellulose (Pratama et al., 2019). Lead ions can substitute the H atom in the functional group, causing this phenomenon.

Determination of the Pseudo Order of Adsorption Kinetics

Adsorption rate is defined as the ability of an adsorbent to absorb adsorbate. The adsorption rate constant (k) and the reaction order of an adsorption kinetics model determine this parameter. As stated by (Wulandari et al., 2023) a high R^2 value helps to validate the model by showing the best adsorption kinetics.

Figure 3 displays the adsorption kinetics model of a water hyacinth bioadsorbent activated by 0.1 M HCl.

It has a residual square that is very close to one. The second-order pseudo-kinetic model has a point of 0.9961 compared to the first-order, 0.33118, with an experimental q_e value of 3.9538 mg/g (see equations (1) and (3)). This best model assumes that the adsorption capacity is proportional to the number of active sites (Aldiansyah et al., 2023; Kurniasari, et al., 2020). The adsorption process occurs when metal ions attach to the adsorbent surface through chemical bonds, tending to determine the sites that maximize the number of ion bonds with the adsorbent surface (Wulandari et al., 2023).

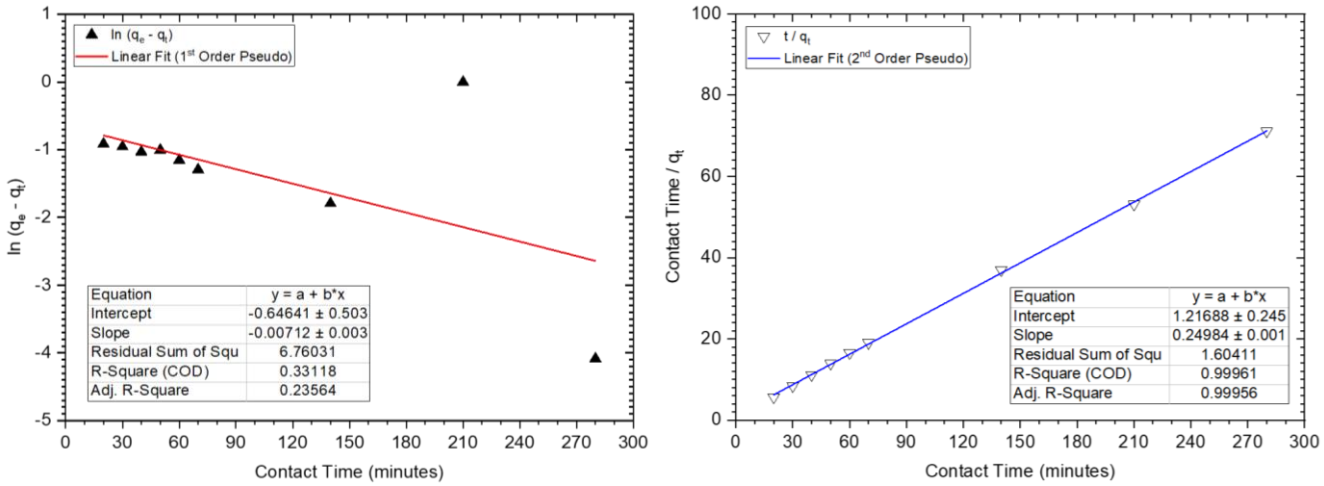

Figure 3. Modeling of adsorption kinetics: 1st and 2nd order pseudo

The Adsorption of Bioadsorbent on Battery Industry Effluent

The bioadsorbent that had been activated with 0.1 M HCl was able to take in the most lead ions from the standard Pb^{2+} solution (96.48% at 210 minutes with 20.4891 ppm as C_0). Then, the bioadsorbent was contacted for battery industry wastewater and AAS testing. During the contact time, 94.45% of the Pb^{2+} was taken up by the bioadsorbent, which started with 8.1488 ppm of waste (5 g of bioadsorbent in 100 mL of waste). There were other researchers, like (Amanah et al., 2022; Fadhilah et al., 2021), who also worked on the adsorption of Pb^{2+} and Cu^{2+} ions using 3.6 and 0.1 M HCl as activators. The results showed adsorption efficiency values of 42.90% and 65.27%. The difference in achievements between the results of the study and those of other researchers is due to the use of a standard solution of lead ions containing only Pb^{2+} alone. Meanwhile, the battery industry's effluent contains several other metal ions. This affects the adsorption capacity, which is not as large as contact with the Pb^{2+} standard solution.

CONCLUSION

Characterization of water hyacinth bioadsorbent using FTIR. The results showed that the bioadsorbent before activation had O-H, C-H, C=O, and C-O ether functional groups at wavelengths of 3340.71, 2914.45, 1608.78, and 1029.95 cm^{-1}, respectively, which identified the presence of cellulose. When 0.1 M HCl was used to activate the material, the C=O functional group vibrations stopped, which showed that the amount of lignin and hemicellulose had decreased. However, an increase in the vibrational peak intensity in the C-O functional group can indicate the presence of carbon chain linking in cellulose. The bioadsorbent experienced a decrease in the intensity of the O-H and C-H vibrational peaks upon contact with waste (Pb^{2+}). Lead ions have substituted the H atom in the functional group. Then, the optimum contact time between the bioadsorbent and the standard Pb^{2+} solution was 210 minutes, with an adsorption efficiency of 96.485% (an initial concentration of 20.49 ppm). With this contact time, the adsorption efficiency of the bioadsorbent on battery industry effluent was able to reach 94.446% with an initial concentration of 8.19 ppm. The utilization of the adsorption kinetics model in this study is best at pseudo-second-order with an adsorption rate (k_2) of 0.051278 $g.mg^{-1}.min^{-1}$. Linear regression results that are close to number one show that adsorption capacity is proportional to the number of active sites.

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