REKAYASA
Journal of Science and Technology
https://journal.trunojoyo.ac.id/rekayasa

Rekayasa, 2024; 17(3): 372-379
ISSN: 0216-9495 (Print)
ISSN: 2502-5325 (Online)

Effective Lead Ions (Pb^{2+}) Mitigation in Battery Industry Wastewater via NaOH-Activated Water Hyacinth (*Eichhornia crassipes*)

Chintya Mahjati Hibatulwafi[1], Dessy Agustina Sari[2]*, Aulia Wahyuningtyas[1]
[1]Chemical Engineering Program Universitas Singaperbangsa Karawang
Jl. HS Ronggo Waluyo Puseurjaya Telukjambe Timur Karawang Jawa Barat 41361
[2]Department of Chemical Engineering Universitas Diponegoro
Jl. Prof Soedarto No 13 Tembalang Kota Semarang Jawa Tengah 50275
*E-mail Korespondensi : dessy.agustina8@staff.unsika.ac.id
DOI: https://doi.org/10.21107/rekayasa.v17i3.26083
Submitted June 19th 2024, Accepted December 8th 2024, Published December 27th 2024

Abstrak

Timbal yang merupakan salah satu logam berat yang paling penting adalah polutan beracun di seluruh dunia. Adsorpsi adalah salah satu metode yang digunakan untuk mempelajari penghilangan ion logam berat dari larutan, yang mendorong kami untuk mempelajari pengurangan konsentrasi Pb^{2+} dalam air limbah dari pabrik baterai dalam penelitian ini. Kondisi terbaik untuk adsorpsi ditemukan dengan menggunakan bioadsorben yaitu eceng gondok yang diaktivasi dengan 0,5 M NaOH. Hal ini dipelajari dengan menggunakan FTIR untuk mengidentifikasi tanaman tersebut. Penelitian ini melakukan uji adsorpsi pada larutan standar Pb^{2+} 20 ppm dengan memvariasikan waktu kontak 20, 30, 40, 50, 60, 70, 140, 210, dan 280 menit. Pada waktu kontak 140 menit, konsentrasi Pb^{2+} berkurang menjadi 0,1558 ppm dengan efisiensi adsorpsi sebesar 98,0881%. Sebelum perlakuan bioadsorben, analisis FTIR gugus fungsi O-H, C-H, C=C, dan C-O menunjukkan adanya selulosa. Aktivasi menghilangkan puncak C=C dan mengurangi intensitas puncak O-H. Hal ini menunjukkan bahwa rantai lignin dan hemiselulosa terurai. Ketika bioadsorben berikatan dengan Pb^{2+}, frekuensi vibrasi gugus C-H dan C-O berubah yang menunjukkan bahwa bioadsorben bekerja. Hasil penelitian menunjukkan bahwa bioadsorben eceng gondok teraktivasi NaOH merupakan bahan alternatif yang baik untuk menangani limbah Pb^{2+} dalam menurunkan jumlah timbal pada air limbah industri (jumlah besar).

Kata Kunci: adsorpsi, bioadsorben, limbah industri, logam berat timbal, pengolahan air

Abstract

Lead, which is one of the most important heavy metals, is a worldwide toxic pollutant. Adsorption is one of the methods used to study the removal of heavy metal ions from solutions, which encourages us to study the reduction of Pb^{2+} concentrations in wastewater from the battery factory in this study. The best conditions for adsorption were found using a bioadsorbent called 0.5 M NaOH-activated water hyacinth. This was studied using FTIR to identify the plant. This research conducted adsorption tests on a 20 ppm Pb^{2+} standard solution, varying the contact times to 20, 30, 40, 50, 60, 70, 140, 210, and 280 minutes. At the contact time of 140 minutes, the Pb^{2+} concentration reduced to 0.1558 ppm with a 98.0881% adsorption efficiency. Before the bioadsorbent treatment, the FTIR analysis of O-H, C-H, C=C, and C-O functional groups showed that cellulose was present. Activation eliminates the C=C peak and reduces the intensity of the O-H peak. This shows that the lignin and hemicellulose chains are breaking down. When the bioadsorbent binds to Pb^{2+}, the vibrational frequencies of the C-H and C-O groups change, which shows that it works. The results show that the water hyacinth bioadsorbent activated by NaOH is a good alternative material for dealing with Pb^{2+} waste, which lowers the amount of lead in industrial waste water by a large amount.

***Key words**: adsorption, industrial effluent, bioadsorbent, heavy lead metal, water treatment*

INTRODUCTION

The existence of the battery industry plays a significant role in reducing emissions, but it also has the potential to have an adverse impact on the environment. The development of the battery industry can cause battery waste (effluents) to increase due to higher battery consumption and short battery life, which are environmental problems. Batteries contain manganese (Mn), lead (Pb), cadmium (Cd), and lithium (Li), but lead (Pb) has a very high toxicity for living things (Andarista *et al.*, 2023). Because it cannot degrade naturally and tends to accumulate in water, heavy lead toxic to humans and other living things (Moelyaningrum *et al.*, 2018; Putra & Widada, 2022). The resulting impacts include organ damage, nervous system disorders, anaemia, impaired immune systems, and low intelligent quotient (IQ) levels in children (Saravanan *et al.,* 2021). The presence of heavy metals in a body of water can reduce water quality and endanger the environment and aquatic organisms (Ganing, 2022; Naulina *et al.*, 2023). Therefore, it is necessary to reduce heavy metals in waste before discharging into waters.

Precipitation, ion exchange using resin, filtration, flocculation, coagulation, and adsorption are some techniques that can lower the concentration of metal ions in liquid waste (Ekoputri *et al.*, 2024; Nurohmah *et al.,* 2019). Adsorption is the most commonly used method because it has a simpler concept and is also economical (Ganing, 2022). Activated carbon, zeolite, silica gel, bentonite, chitosan, and natural adsorbents or bioadsorbents are some of the adsorbent media that are often used (Ifa *et al.*, 2020). The most commonly used type of adsorbent is bioadsorbent, due to the easy availability of raw materials in the surrounding environment. Water hyacinth (WH) is one of the bioadsorbent materials available for use (Nurhadi *et al.*, 2019; Sari *et al.,* 2021). There are active sites in water hyacinth, such as hydroxyl (O-H), which can attract heavy metal ions (Andarista *et al.,* 2023; Rakhmania *et al.,* 2017). WH is made up of 60% cellulose, 17% lignin, and 8% hemicellulose. Fourier transform infrared (FTIR) testing can identify the hydroxyl active group sites on WH, and Table 1 displays the other functional groups present.

Table 1. Wavenumbers for FTIR characterization (DL Pavia, 2009; Rakhmania et al., 2017)

The bond	Type of compound	Frequency region (cm^{-1})
O-H	Alcohols, phenol	3200-3550
C-H	Alkanes	2853-2962
C=C	Alkenes	1600-1680
C-OH	Alcohols	1380-1470
C-N	Amines	1250-1360
C-O	Ether	~1240
C-O	Cyclic ether	900-1250
C-H	m-aromatics	600-800
≡C-H	Alkyne	600-700

The researchers (Mahmudah *et al.,* 2023) recommend drying and mashing WH to use it as a bioadsorbent. This approach is quicker, less costly, and easier to implement. Activation can also optimize bioadsorbents. The activation process aims to enlarge the adsorbent pore, either by rupturing hydrocarbon bonds or oxidizing adsorbent surface particles. This alters the surface area, leading to an increase in adsorption activity (Nurohmah *et al.,* 2019).

Bioadsorbents have been activated in the past by chemicals like HCl, HNO_3, NaOH, and H_2SO_4 (Aldiansyah *et al.,* 2023; Nurhadi *et al.*, 2019; Sa'diyah *et al.*, 2020; Utami *et al.*, 2024). This research uses an activator based on NaOH. The researchers will soak the surface of WH in NaOH solvent to degrade lignin and solubilize part of the hemicellulose (Jasman & Ahmad, 2021). The study's goal is to find out how lead ions stick to a 0.5 M NaOH-activated WH bioadsorbent. This study will use FTIR to figure out the best contact time for adsorption on a Pb^{2+} standard solution, which will be used to absorb waste from the battery industry. Similar studies rarely discuss topics as thoroughly as this research does. This research introduces a novel method for reducing the concentration of metal ions in battery industry wastewater, which involves activating a water hyacinth bioadsorbent using a 0.5 M NaOH base solution to optimize the adsorption process.

METHODS

The materials used in this study are: water hyacinth stems, NaOH (Merck, 0.5 M), distilled water, artificial waste from a standard solution of Pb^{2+} with a concentration of 20 ppm and battery industry waste (PT "WHY" in Karawang-Jawa Barat). The tools used in this research are: digital technical balance (SF-400C), crusher (magic blend MB21BL), oven (Memmert), simple magnetic stirrer, simple 100 mesh sieve shaker, filter paper (Whatman, No 42), FTIR spectrometer (Nicolet iS50 Thermo Scientific), and Atomic Absorption Spectrophotometer-AAS (Shimadzu AA-7000).

Bioadsorbent preparation

Water hyacinth stems were cleaned to remove any dirt that was still attached. Next, WH stems were reduced and dried to remove moisture content using an oven at 150°C for 80 minutes. After drying, it was crushed and sieved using a 100 mesh sieve shaker.

Activation using 0.5 M NaOH

25 g of WH powder was activated with 350 ml; of 0.5 M NaOH solution mixed for 30 minutes and then allowed to stand for 24 hours. The mixture of WH powder and NaOH solution was filtered and the sediment was neutralised using distilled water until pH 7, then the activated and neutralised WH sediment was dried using an oven at 105°C for 2 hours (Fitriani *et al.*, 2019; Nurhidayanti *et al.*, 2021; Wibawa *et al.*, 2014).

Determination of optimum contact time

A total of 0.5 gram of 0.5 M NaOH-activated WH bioadsorbent was contacted with 100 ml of artificial wastewater from a standard solution of Pb^{2+} with a concentration of 20 ppm and the contact time was varied for 20, 30, 40, 50, 60, 70, 140, 210, and 280 minutes to determine the optimum contact time between NaOH-activated WH bioadsorbent and artificial effluents. After that, the same procedure was contacted with 100 ml of PT "WHY" battery industry waste in Karawang-Jawa Barat using the optimum contact time obtained from the artificial waste. The mixture was then filtered to separate the filtrate and residue.

Determining adsorption kinetics

Adsorption kinetics are generally used to predict the ability of the sorption process of various adsorbents, also providing information about the adsorption mechanism and possible rate control measures such as mass transfer or chemical reaction processes (Gupta *et al.,* 2021). This analysis is done to calculate the adsorption capacity using equations (1) and (2) while the adsorption efficiency can be done using equation (3) (Alghamdi et al., 2019; Haryanto *et al.*, 2019).

$$q_e = \frac{(C_0 - C_e)}{m} \times V \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots..(1)$$

$$q_t = \frac{(C_0 - C_t)}{m} \times V \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots..(2)$$

$$(\%)R = \left(\frac{C_0 - C_t}{C_0}\right) \times 100\% \dots\dots\dots\dots\dots\dots\dots(3)$$

Description:

q_e, q_t = adsorption capacity at equilibrium and at t (mg/g)

C_0, C_e, C_t = initial metal concentration, equilibrium concentration, and concentration at t (mg/L)

R = percentage of metal adsorbed (%)

V = volume of solution (L)

m = mass of adsorbent (g)

First-order pseudo kinetics can be calculated using equation (4) and second-order pseudo kinetics using equation (5) (Gupta *et al.*, 2021; Wulandari *et al.,* 2023).

$$\text{In } (q_e - q_t) = ln\, q_e - k_1 t \dots\dots\dots\dots\dots\dots(4)$$

$$\frac{t}{q_t} = \frac{1}{k_2 {q_e}^2} + \frac{1}{q_e} t \dots\dots\dots\dots\dots\dots\dots\dots\dots.\dots.(5)$$

Description:

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

k_2 = rate constant of pseudo second order ($g.mg^{-1}.min^{-1}$)

t = contact time (min)

RESULT AND DISCUSSION

Using NaOH to activate the process breaks the bonds in lignin's basic structure, making it easy to dissolve. Na^+ ions then bind to lignin to make sodium phenolate, which is also easy to dissolve (Jasman & Ahmad, 2021). As shown in Picture 1, the WH bioadsorbent's color changes before and after activation. The WH bioadsorbent before activation is light brown in colour, but after activation with 0.5 M NaOH immersion, it turns into a solid brown colour. This colour change signifies the release and dissolution of lignin content during the activation process (Ayuni & Hastini, 2020; Safaria *et al.,* 2013). Using NaOH to soak something is meant to damage the crystalline and amorphous parts of lignin and separate some of the amorphous hemicellulose. This way, NaOH can get rid of the lignin while extracting the hemicellulose (Jasman & Ahmad, 2021).

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

The FTIR test results for the function groups of the WH bioadsorbent are available. We carried out characterization in this study using the FTIR test to identify the presence of functional groups in WH bioadsorbent before activation, after activation using NaOH, and after adsorption of battery industry wastewater. The adsorbance value and intensity of the wavelength reading of the FTIR spectrum show a difference, Picture 2 below. This indicates that there is a shift, reduction, or loss of spectrum during the activation and adsorption processes (Munene et al., 2020).

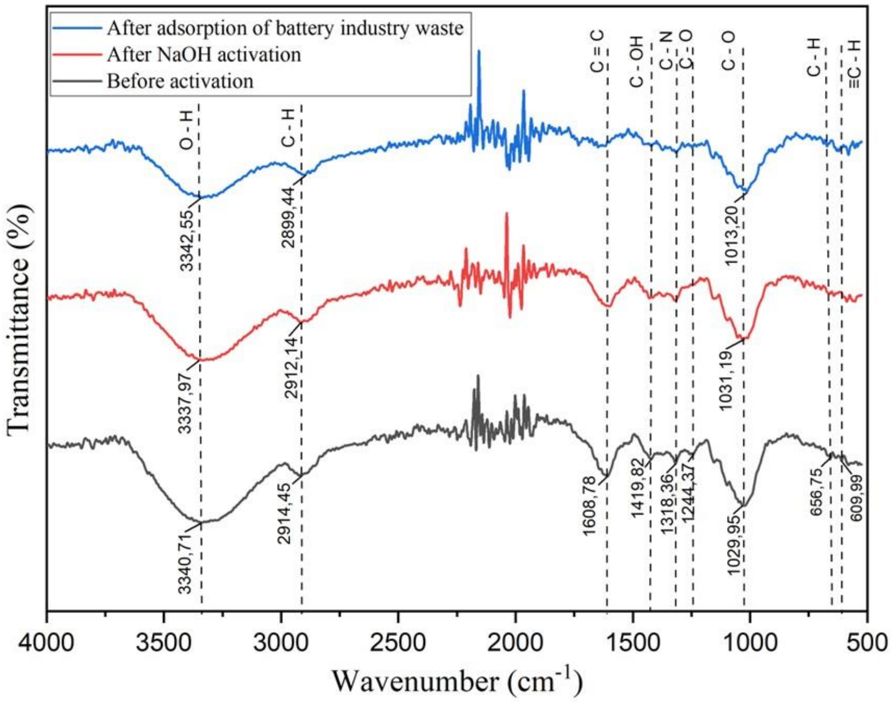

Figure 2. The spectra of the water hyacinth bioadsorbent

Figure 2's FTIR spectra results reveal the presence of hydroxyl functional groups on the adsorbent surface. It is possible for water hyacinth bioadsorbent to have adsorption peaks at 3340.71 and 2914.45 cm^{-1}, which show the stretching of O-H and C-H bonds. These peaks happen because hydrogen bonds are stretching and hydroxyl groups (O-H) are bending in the cellulose structure (Kusumawati & Hariyadi, 2021). The size of the hydroxyl group indicates the presence of strong hydrogen bonds (from carboxyl, phenol, or alcohol). The adsorption that appears in the 1608.78 cm^{-1} region indicates the presence of a C=C group, which suggests an increase in carbon content (Mentari *et al.,* 2018). Also, there is a clear peak at wave number 1029.95 cm^{-1}, which shows that the C-O group is being adsorbed. This is a carbon chain link in cellulose called a glycosidic bond (Astari & Utami, 2018).

NaOH activation led to a decrease in the intensity of vibration peaks at the O-H, C-H, and C-O wavenumbers, which did not widen, suggesting a break in the chains between lignin, hemicellulose, and cellulose (Pratama *et al.*, 2019). A NaOH activator can increase the hydroxyl group on cellulose to bind metal ions; this will have electronegative oxygen atoms that easily release H as H^+ (protons) and quickly capture heavy metal cations to replace the released proton position (Mahmudah *et al.*, 2023). Following the adsorption of battery industry waste, the vibrations of the C-H and C-O groups at wave numbers shifted in a more directional manner. This can occur due to changes in functional groups that have bound Pb^{2+}.

Through the equation (3), percentage of lead ions adsorbed (*R*) shows in Table 2. After that, the optimal contact time used for the artificial wastewater. Table 2 shows that the best adsorption of metal ion levels occurs at 140 minutes of contact. At that time, the percent adsorbed was 99.0014%, with the remaining adsorbed concentration of 0.2046 ppm, which was originally 20 ppm. This is because the soaking process with the NaOH activator can reduce tar or impurity levels, causing the pores of the adsorbent to grow larger (Ganing, 2022). Other researchers can adsorb 34.29 until 48.58% the lead via coffe ground residues (Moelyaningrum *et al.*, 2018).

Table 2. Adsorption efficiency of water hyacinth bioadsorbent

Contact time (minutes)	**Concentration at *t* (ppm)**	**R (%)**
20	1.9232	90.6135
30	1.7957	91.2358
40	1.4790	92.7815
50	1.3008	93.6513
60	1.2110	94.0895
70	1.0938	94.6616
140	0.2046	99.0014
210	0.2815	98.6261
280	0.3142	98.4665

The larger the pores, the greater the surface area of the adsorbent; the increase in surface area results in increased adsorption ability, so the better the quality of the adsorbent (Sa'diyah *et al.,* 2020). The electrostatic attraction between positively charged metal ions and negatively charged bioadsorbent functional groups effectively increases adsorption capacity. The process of metal adsorption on the bioadsorbent surface can also be affected by forces that pull things together, like hydrophobic interactions, Van der Waals forces, and hydrogen bonds (Ifa *et al.,* 2020). After that, this research will use an optimum contact time of 140 minutes to adsorb Pb^{2+} in battery industry wastewater.

When the contact time exceeds 140 minutes, the adsorption efficiency of the WH bioadsorbent in adsorbing artificial effluents from a standard solution of lead ions with a concentration of 20 ppm is reduced. This is because the pores of the bioadsorbent reach the saturation point of Pb^{2+} (Andarista *et al.*, 2023). They conducted research on the adsorption of artificial liquid waste using WH activated charcoal, stating that a contact time of 150 minutes resulted in a 99.99% reduction in Pb levels. Time influences the rate of adsorption, or the speed of adsorption, from the adsorbent to the adsorbate, as determined by adsorption kinetics through the equation (4-5). The researchers (Aldiansyah *et al.*, 2023; Haryanto *et al.*, 2019) use the contact time required to reach adsorption equilibrium as a measure of the adsorption rate.

Figure 3 shows that the NaOH-activated WH bioadsorbent's adsorption kinetics model has a linear regression coefficient (R^2) value close to 1, which the first-order pseudo with a value of 0.5329 and a second-order pseudo-kinetic model with a value of 0.9999. This second-order pseudo-kinetic model assumes that the adsorption capacity is proportional to the number of active sites (Kurniasari *et al.,* 2020). This adsorption occurs when Pb^{2+} attach to the adsorbent surface by forming chemical bonds, and it tends to determine the sites that maximize the number of ion bonds with the adsorbent surface (Riyanto *et al.,* 2021).

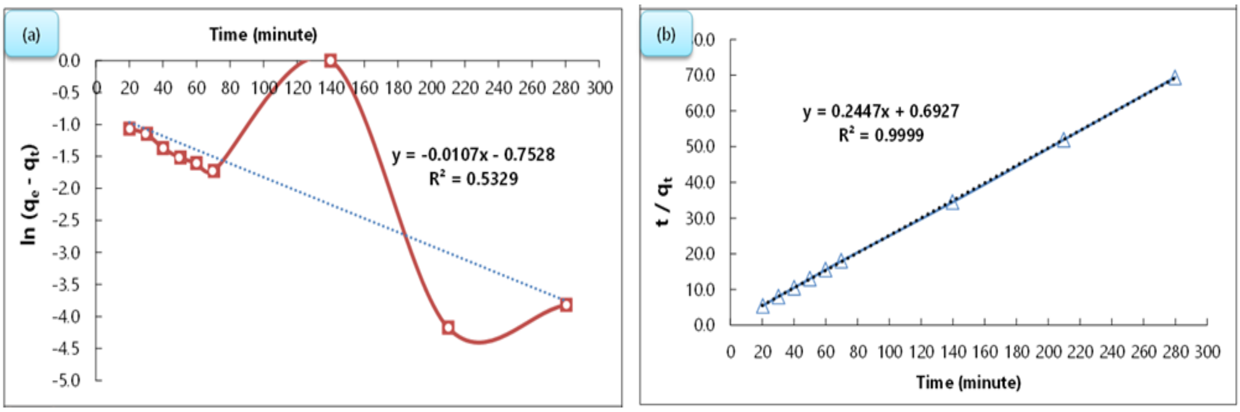

Figure 3. Model kinetics of WH bioadsorbent: (a) the first, and (b) second-order pseudo

Adsorption testing of Pb^{2+} from effluent of battery industry used a NaOH-activated WH bioadsorbent with a 0.5 M contact time for 140 minutes to reduce the level of lead ions in PT "WHY"'s battery industry wastewater in Karawang, based on the ideal contact time with artificial waste. The results shows a decreasing significantly from 8.1488 to 0.1558 ppm with an adsorption percentage of 98.0881%. These achievement align with the lead metal threshold value set by Regulation Kementerian Lingkungan Hidup Republik Indonesia No. 5 of 2024 about surpass the quality standard threshold of SNI No. 7387 of 2009, which is 0.5 ppm (Saraswati & Rachmadiarti, 2021).

CONCLUSIONS

This study successfully demonstrates the effectiveness of NaOH-activated water hyacinth bioadsorbent in reducing lead ions (Pb^{2+}) concentrations in battery industry wastewater. The activation process enhances the bioadsorbent's capacity by increasing the number of active sites for Pb^{2+} binding. FTIR analysis confirmed significant changes in functional groups, indicating the breakdown of lignin and hemicellulose post-activation. The optimal adsorption was achieved at a contact time of 140 minutes, reducing Pb^{2+} concentration in the standard solution from 20 to 0.2046 ppm, corresponding to a 99.0014% adsorption efficiency. The adsorption kinetics followed a second-order pseudo-kinetic model, suggesting that the process is dependent on the availability of active sites. When applied to actual battery industry wastewater, the bioadsorbent reduced Pb^{2+} levels from 8.1488 ppm to 0.1558 ppm, and achieving a 98.0881% adsorption efficiency. These results confirm that NaOH-activated water hyacinth bioadsorbent is an effective solution for treating lead-contaminated wastewater, meeting environmental regulations for heavy metal discharge. Future research should explore the long-term stability and reusability of the NaOH-activated water hyacinth bioadsorbent. Additionally, scaling up the process and evaluating its economic feasibility in industrial applications would be beneficial. Investigating the bioadsorbent's effectiveness against other heavy metals and in different wastewater matrices could further broaden its applicability.

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