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Research Paper

Performance of HNO_3-Activated Water Hyacinth-*Eichhornia crassipes* Bioadsorbent in Adsorbing Lead Metal Ions (Pb^{2+}) in Battery Industry Wastewater Discharge

Wildan Aldiansyah[a], Dessy Agustina Sari[a,b*], Aulia Wahyuningtyas[a]

[a]*Chemical Engineering Program, Faculty of Engineering, Universitas Singaperbangsa Karawang, Jalan HS Ronggowaluyo Telukjambe Timur, Karawang – Jawa Barat, 41361, Indonesia*

[b]*Department of Chemical Engineering, Universitas Diponegoro, Jalan Prof. Soedarto, SH., Tembalang – Semarang, Jawa Tengah, 50275, Indonesia*

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ABSTRACT: Elemental lead is one of the pollutants generated from batteries. To overcome this problem, technologies such as the use of bioadsorbents have been developed to reduce heavy metal content in wastewater. This study aims to determine the adsorption of lead metal ions (Pb^{2+}) through the use of a water hyacinth (Eichhornia crassipe) bioadsorbent activated with nitric acid (HNO_3). Another objective is to obtain the optimum adsorption time on battery industry waste discharges. Post-treatment characterization using FT-IR. Contact time variations (20, 30, 40, 50, 60, 70, 140, 210, and 280 minutes) were used to carry out the lead metal adsorption process on a standard solution of metal ions (Pb^{2+}), 20 ppm. The results showed that the optimum contact time was 210 minutes. This achievement was used as the contact time for reducing the levels of Pb^{2+} metal ions. The quantity of the remaining Pb^{2+} metal ion content was 0.4184 ppm. Indirectly, the bioadsorbent performance was 94.8655%. Characterization through FT-IR equipment before activation of water hyacinth bioadsorbent gave results of O-H, C-H, C=O, and C-HO functional groups. These findings indicated the presence of lignin, hemicellulose, and cellulose compounds in the sample before activation. Then, the involvement of 1 N HNO_3 solution (as an activator) resulted in a decrease in the quantity of C=O and C-OH functional groups. The process of applying the solution was able to break a number of chains between lignin and hemicellulose. After the adsorption process was given, the waste left the vibrations of O-H, C-H, and ≡C-H groups at wave numbers shifted in a smaller direction. This can occur due to changes in functional groups that have bound metal ions first.

Keywords: activated nitric acid (HNO_3); battery wastewater; lead metal ion (Pb^{2+}); metal ion adsorption; water hyacinth bioadsorbent.

ABSTRAK: *Timbal merupakan salah satu pencemar yang dihasilkan dari baterai. Untuk mengatasi masalah ini, teknologi seperti penggunaan bioadsorben telah dikembangkan untuk mengurangi kandungan logam berat dalam air limbah. Penelitian ini bertujuan untuk mengetahui adsorpsi ion logam timbal (Pb^{2+}) melalui bioadsorben eceng gondok (Eichhornia crassipe) teraktivasi asam nitrat (HNO_3). Tujuan lainnya adalah mendapatkan waktu optimum adsorpsi pada buangan limbah industri baterai. Karakterisasi menggunakan FT-IR. Variasi waktu kontak (sebesar 20, 30, 40,50, 60, 70, 140, 210, dan 280 menit) digunakan untuk melangsungkan proses adsorpsi logam timbal pada larutan standar ion logam (Pb^{2+}), 20 ppm. Hasil penelitian menunjukkan bahwa waktu kontak optimum berada pada 210 menit. Capaian ini dijadikan waktu kontak penurunan kadar ion Pb^{2+}. Kuantitas sisa kadar ion logam Pb^{2+} sebeasar 0,4184 ppm. Secara tidak langsung, performa bioadsorbennya sebesar 94,8655%). Karakterisasi sebelum bioadsorben eceng gondok aktivasi memberikan hasil gugus fungsi O–H, C–H, C=O dan C–HO. Temuan ini mengindikasikan adanya senyawa lignin, hemiselulosa dan selulosa dalam sampel sebelum teraktivasi. Pelibatan larutan HNO_3 1 N (sebagai pengaktivasi) memberikan penurunan kuantitas gugus fungsi C=O dan C-OH. Pemberiaan larutan tersebut mampu memutuskan sejumlah rantai antar lignin dan hemiselulosa. Pasca adsorpsi, buangan limbah tersebut menyisakan O–H, C-H dan ≡C-H pada bilangan gelombang lebih kecil. Perihal ini terjadi karena perubahan gugus fungsi yang telah mengikat ion logam lebih dahulu.*

Kata Kunci*: adsorpsi ion logam; bioadsorben eceng gondok; ion logam timbal (Pb^{2+}); limbah cair baterai; teraktivasi asam nitrat (HNO_3).*

1. INTRODUCTION

Waste batteries are one of the most commonly generated types of e-waste, as almost all electronic devices use batteries. Generally, Indonesians tend to use primary batteries because they are more affordable, but they are only disposable. If not used properly, primary batteries can cause environmental problems. This is due to the content of various heavy metals in batteries, such as mercury, nickel, lead, cadmium, and lithium [1-2].

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Department of Chemical Engineering
Faculty of Industrial Technology
Universitas Muslim Indonesia, Makassar
Address
Jalan Urip Sumohardjo km. 05 (Kampus 2 UMI) Makassar- Sulawesi Selatan
e-mail : jcpe@umi.ac.id

Corresponding Author *
dessy.agustina8@staff.unsika.ac.id

Heavy metal ions are a source of water pollution that is detrimental to human and animal health, as human and animal bodies have difficulty processing them, causing serious impacts including organ shrinkage, cancer, nervous system disorders, defense system disorders, and life-threatening diseases [3]. Lead is a top choice in the battery industry due to its specific characteristics, including high conductivity and corrosion resistance [4]. This results in an increase in hazardous lead content in wastewater because this heavy metal is a substance that is toxic to humans and other living things because it cannot be degraded naturally and tends to accumulate in water [5][7].

Efforts to reduce heavy metal content in industrial waste are crucial to ensuring that the waste discharged into water bodies meets the standard quality requirements set by relevant authorities in a country. This initiative plays a significant role in mitigating the risks of pollution caused by industrial waste, particularly heavy metal contamination. Adsorption is the most effective and efficient approach for reducing heavy metal concentrations in water. Other common techniques, such as coagulation, flocculation, evaporation, ion exchange, and membrane separation, are also used [8][12]. This process utilizes affordable and easily available raw materials found in the environment [9][13][14].

Adsorption is the process of molecules or ions being absorbed from a fluid onto the surface of a solid [15]. Various types of media can be used as adsorbents, including activated carbon, zeolite, silica gel, bentonite, chitosan, and natural bioadsorbents [16]. Among these media, bioadsorbents are a popular choice due to their simple production process and easy availability of raw materials. Eceng gondok, also known as Eichhornia crassipes, is an excellent example of a bioadsorbent that does not require further processing into activated carbon. With this method, we can confidently address the issue of heavy metal pollution in industrial waste while maintaining a diplomatic approach towards the industry. The required processes are drying and size reduction [17]. This approach offers a simple, cost-effective, and easily applicable solution for managing heavy metal content in industrial waste. To modify the surface characteristics of biomass waste, a variety of chemicals can be used as activators. This method has been proven to be successful in numerous studies, demonstrating its effectiveness and reliability. For instance, phosphoric acid [18], hydrochloric acid [19], nitric acid [20], sodium hydroxide [21], and zinc chloride [22] have all been shown to be effective. The activation process aims to expand the adsorbent pores, break hydrocarbon bonds, or oxidize molecules on the adsorbent surface. It has been established by [23] that a larger surface area can significantly enhance the adsorption capacity. This finding is supported by their extensive research and analysis.

In this study, nitric acid (HNO_3) was used as an activator to widen the pores of the bioadsorbent. The H^+ ions dissolved a number of contaminant metals by replacing the metal ions bound to the adsorbent, thereby increasing the number of active sites on the adsorbent. The H^+ ions dissolved a number of contaminant metals by replacing the metal ions bound to the adsorbent, thereby increasing the number of active sites on the adsorbent. This process of adsorption was found to be effective in removing contaminants. As experts have noted, the concentration of H^+ ions in a solution increases with the concentration of acid. This leads to the formation of OH^{2+} ions when the acid binds to the hydroxyl groups in cellulose. This product is known for its ability to repel metal ions [24]. In the past, researchers have explored the use of bioadsorbents to reduce the amount of heavy metals. For instance, they activated a rambutan peel-based bioadsorbent using a concentrated HNO_3 solution. The researchers confidently reported an improvement in the adsorption ability of Pb^{2+} ions by using adsorbents. The teams [24] documented this finding. It can be concluded that the use of adsorbents is a promising method for the removal of Pb^{2+} ions from liquid waste. In a similar study, [25] utilized activated bamboo charcoal with a similar activator and successfully increased the effectiveness of Pb metal adsorption from liquid waste.

This study confidently examines the potential of water hyacinth as a raw material for producing a bioadsorbent by activating it with a concentrated HNO_3 solution (1.2 M). It is important to acknowledge the potential of this method for addressing the issue of lead pollution in industrial wastewater while also considering the need for further research and collaboration in this area. The resulting product is expected to effectively adsorb lead (Pb^{2+}) ions in industrial battery wastewater. The condition was analyzed before, during, and after being applied as a bioadsorbent. Fourier transform infrared (FT-IR) spectroscopy was used to determine the functional groups present. Additionally, atomic absorption spectrophotometers (AAS) were used to determine the concentration of remaining metal ions adsorbed.

2. RESEARCH METHODS

The materials utilized in this study were eceng gondok stems (Telaga Munjul, Karawang), HNO_3 solvent (Merck), and aquades. The battery industry wastewater samples came from the battery industry in Karawang, Jawa Barat. The instruments used to characterize the samples were AAS to determine the concentration of Pb^{2+} metal ions and FT-IR to determine the functional groups on the water hyacinth bioadsorbent.

To produce bioadsorbents, the first step involves separating and cleaning the stem, leaves, and roots of water hyacinth plants. For this study, only the stem is utilized. The stem is cut, dried in an oven at 150°C for 80 minutes, and sieved to obtain a fineness of 100 mesh. To activate the bioadsorbent, 25 g of water hyacinth powder is mixed with 350 ml of 1 N HNO3 and agitated for 5 hours. This method has been proven effective in producing high-quality bioadsorbents. The mixture is confidently filtered and thoroughly washed with distilled water until a neutral pH of 7 is achieved. The resulting solid on the filter paper is then carefully dried at 105°C for 2 hours.

To determine the optimum contact time for lead metal adsorption, a standard Pb^{2+} solution (20 ppm) was prepared, and 0.5 g of activated bioadsorbent powder was dissolved in 100 ml of the solution. The preparation of this standard solution aims to facilitate the determination of Pb^{2+} metal ions remaining in the sample solution. The contact time was then varied between 20, 30, 40, 50, 60, 70, 140, 210, and 280 minutes. The same procedure was repeated with industrial battery wastewater to obtain the optimum contact time. The powder was dissolved in 100 ml of the wastewater with the same contact time variations.

The research data analysis requires the application of the pseudo-first and second-order kinetic in equations (1) to (3) below to determine the adsorption capacity [26].

The adsorption capacity

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

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

$$\% R = \left(\frac{C_o - C_t}{C_o}\right) \times 100\% \quad (1.3)$$

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

3. RESULT AND DISCUSSIONS

Characterization by FT-IR

This study utilizes water hyacinth stem biomass in Karawang, Jawa Barat, as a bioadsorbent raw material. For the performance of adsorbing Pb^{2+} metal ions in battery industry wastewater, the first test on the bioadsorbent was FT-IR to determine the functional groups contained in the water hyacinth bioadsorbent. The results in Figure 1 show the presence of various compounds on the surface of the bioadsorbent. The appearance is rich in hydroxyl, carbonyl, and carboxyl functional groups. A number of these groups act as active groups in carrying out the metal ion adsorption process. Their presence also indicates the presence of components such as cellulose, hemicellulose, and lignin in the water hyacinth bioadsorbent. In particular, hydroxyl groups indicate the presence of lignin, while carbonyl groups indicate hemicellulose, and aliphatic hydroxyl groups indicate the presence of cellulose, as well as hemicellulose, which acts as an active group in bioadsorbents.

The FT-IR test results (Figure 1) show that there are large and wide absorption wave numbers for all water hyacinth bioadsorbent samples, indicating the presence of hydroxyl groups in all bioadsorbent samples. These groups have a role as active functional groups in the ion adsorption process, especially hydroxyl groups that specifically indicate the presence of lignin [27]. Bioadsorbents, before activation, have O-H and C-H functional groups that show stretching vibrations on O-H and C-H bonds. The peak appears due to stretching of hydrogen bonds and bending of hydroxyl (OH) groups in the cellulose structure [20]. In the 1 N HNO_3-activated bioadsorbent, some functional groups are missing, namely C=O and C-OH. This is due to the shift of the two peaks caused by the breaking of hydrogen bonds, which indicates the breaking of chains between lignin, hemicellulose, and cellulose [28]. After the adsorption process, there is a missing bending vibration, which is caused by the H atom in the functional group that has been substituted with Pb^{2+} ions. The C=O stretching bond structure, which is generally associated with carbonyl groups, lost its vibrational intensity after the bioadsorbent underwent activation and adsorption processes. Meanwhile, the characteristic ≡C-H bending vibrations also decreased in intensity after the adsorption of lead metal ions (Pb^{2+}).

Effect of optimum contact time

Contact time was given for the water hyacinth bioadsorbent in a 20 ppm Pb2+ ion standard solution. The variation of contact time aims to determine the optimum interaction time for the bioadsorbent in adsorbing Pb2+ metal ions until it reaches equilibrium conditions. The effect of contact time on the adsorption of Pb^{2+} metal ions on a nitric acid-activated bioadsorbent is presented in Table 1, which shows the difference in time for each bioadsorbent to reach equilibrium in adsorbing a 20 ppm Pb^{2+} standard solution. Bioadsorbent samples reached equilibrium within 210 minutes, indicating the time required for water hyacinth bioadsorbent to adsorb Pb^{2+} metal ions is getting faster along with the length of contact time until it reaches a saturation point, which then occurs during the during the desorption event. This occurs due to the level of saturation of the active site after adsorbing Pb^{2+} metal ions.

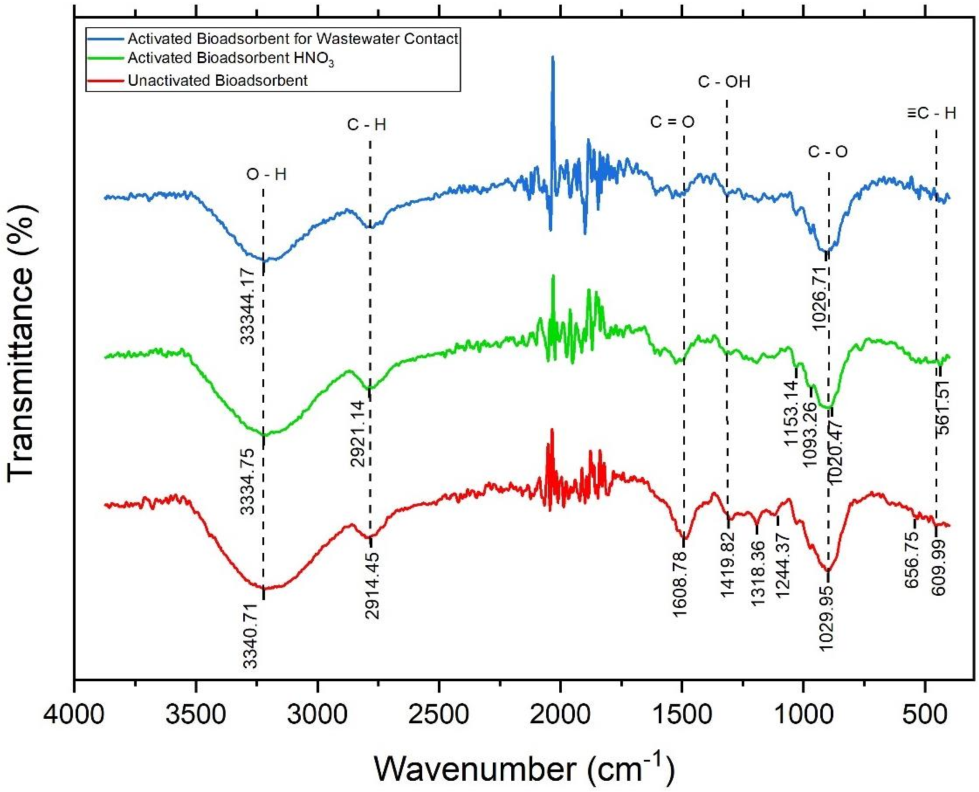

Figure 1. Results of the FT-IR characterization test

Table 1 shows that the adsorption activity of nitric acid-activated water hyacinth bioadsorbent is able to adsorb Pb^{2+} metal ions. This is due to the activation treatment that has released impurities and metal ions such as Ca^{2+}, Mg^{2+}, and K^{+} that cover some of the pores of water hyacinth bioadsorbent, so that the pores of water hyacinth bioadsorbent become larger to adsorb Pb^{2+} [24].

Table 1. AAS test results for optimum contact time and modeling of adsorption kinetics

The Contact Time, minutes	R, %	Parameters	First-	Second-
			Order Pseudo	
20	86.1600	k_1	0.0079	-
30	86.7037	k_2	-	0.0534
40	87.4733	q_e calc	0.4152	4.0339
50	88.5110	q_e exp	3.9759	3.9759
60	89.5530			
70	92.1671			
140	94.7884			
210	97.0243			
280	96.6558			

Initial concentration = 20.4891 ppm

The duration of the interaction between the absorbed substance and the sorbent material will have an impact on the sorption ability. Increasing the duration can increase the amount of lead metal ions that are successfully absorbed until it reaches an equilibrium point. The length of contact time between the absorbed substance and the absorbent material will affect the efficiency and capacity of absorption, with an increase in duration causing an increase in the amount of Pb^{2+} metal adsorbed [29]. The adsorption process will stop if an equilibrium has occurred between the levels of adsorbate in the solution and the adsorbent material. This is because the active hydroxyl group (-OH) of cellulose has experienced equilibrium, so the solution is saturated and no longer able to absorb metals optimally [30].

3.1. Kinetics of reaction

Adsorption kinetics is used to determine the rate of adsorption that occurs from the adsorbent to the adsorbate and is influenced by time. The contact time required to reach adsorption equilibrium is used as a measure of the adsorption rate. In this study, adsorption rate testing was carried out by estimating the reaction order (Table 1). Then, the kinetics of adsorption were obtained empirically using first-order pseudo and second-order pseudo models (Figure 2.a and b).

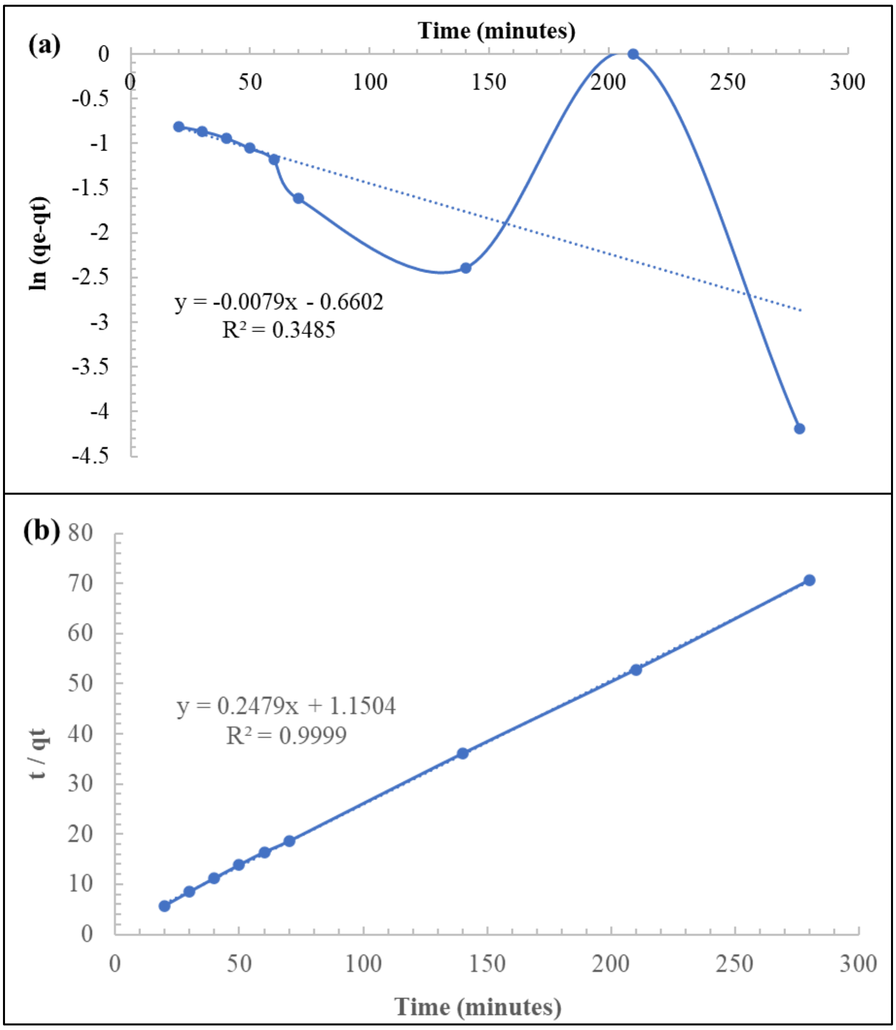

Figure 2. Modeling of adsorption kinetics: (a) first-,and (b) second-order pseudo

The results of theoretical calculations show that the kinetics of the adsorption reaction of a nitric acid-activated water hyacinth bioadsorbent follow a second-order pseudo-kinetics model. This is due to the comparison of the linear regression correlation coefficient value (R^2), which can be used to determine the appropriate modeling of the adsorption process. The first-order pseudo-equation has a linear regression coefficient value (R^2) of 0.3485, and the second-order equation has a value (R^2) of 0.9999. From the data obtained, it can be concluded that the correlation coefficient (R^2) value close to 1 in the linear equation indicates that, when compared with the first-order and second-order pseudo adsorption kinetics, the second-order pseudo adsorption kinetics model is more suitable. One of the other fit parameters is the comparison of qe values. The calculation of qe, which is close to the extrapolated qe value, also shows that the second-order pseudo-kinetics modeling provides insignificant results [31].

Effect of industrial wastewater (Pb^{2+}) adsorption results

The initial concentration of Pb^{2+} metal ions in battery industry wastewater was 8.1488 ppm. The results showed that the concentration of lead metal ions in battery industry wastewater decreased the level of Pb^{2+} metal ions in industrial waste by 0.4184 ppm, with an adsorption percentage of 94.8655%. Similar research was conducted by [32] on Fe(III) and Mn(II) ions using water hyacinth stems for the manufacture of activated carbon, resulting in efficiency values of 23.37 and 79.25%, respectively. Other research conducted by [21] on Cu(II) ions and [33] on Cr(IV) ions using water hyacinth as a bioadsorbent produced efficiencies of 42.89 and 43%.

There is a difference in adsorption in this study between the Pb^{2+} standard solution and battery industry wastewater. This is because the Pb^{2+} standard solution only contains Pb^{2+} metal ions, but for battery industry wastewater, there are several other metal ion contents that affect the adsorption results of the water hyacinth bioadsorbent, so the percentage of adsorption is not as large as using the Pb^{2+} standard solution. Adsorption of different metal ions on heterogeneous surfaces can occur depending on the type of adsorbent; in this case, there is competition between metal ions for the adsorption process, which is dominant to one of the adsorbates, causing selective adsorption [34]. So it can be concluded that contact-time treatment with nitric acid-activated water hyacinth bioadsorbent can reduce Pb^{2+} metal ion levels in battery industry waste.

4. CONCLUSIONS

The results of the characteristics of the water hyacinth bioadsorbent using FT-IR testing showed that the bioadsorbent before activation had O-H, C-H, C=O, and C-O-H functional groups each at a wavelength of 3340.7, 2914.45, 1608.78, and 656.75 cm^{-1}, respectively. After activation using HNO_3 1 N, there is a missing carbonyl functional group, namely C=O, and some carboxylic acid functional groups on C-OH bending vibrations. When the bioadsorbent was contacted with battery industry waste, only the functional groups of alcohol O-H, alkane C-H, and alkyne ≡C-H remained. The results of the optimum contact time test on the absorption of the best metal ion levels occurred at 210 minutes with an adsorption percentage of 97.0243%. Then the adsorption results on battery industry waste after adsorption for 210 minutes have an adsorption percentage of 94.8655%.

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