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Characterization of H_3PO_4-activated *Eichhornia crassipes* Bioadsorbent for Removal of Lead Ion (Pb^{2+}) in Wastewater of Battery Industry

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Abstract
Lead ions (Pb^{2+}) have high toxic properties and are harmful to the bodies of living things because they cause disease and even death. Efforts that can reduce the content of Pb^{2+} ions include the adsorption of water hyacinth bioadsorbent. Therefore, this study aims to determine the ability of a 1.2 M H_3PO_4-activated bioadsorbent to reduce the amount of ion Pb^{2+} content in artificial wastewater of the battery industry with characterization using FT-IR (Fourier Transform Infra-red) and AAS (Atomic Absorption Spectrophotometers). The activated bioadsorbent was contacted with artificial wastewater from the battery industry in a 20 ppm Pb^{2+} standard solution and contact time variations of 20, 30, 40, 50, 60, 70, 140, 210, and 280 minutes to determine the optimum adsorption time. This condition lasted for 140 minutes. Then the bioadsorbent was contacted with Pb^{2+} ions from the industry wastewater. The results showed a remaining content of Pb^{2+} battery industry wastewater of 0.2503 ppm and a total adsorption percentage of 96.928%. The bioadsorbent before activation has O-H, C-H, C=C, and C-O functional groups that indicate the presence of cellulose. After the activation, there is a removal of the C=C group, indicating delignification, and there are wave numbers indicating the presence of symmetrical vibration functional groups in the C-O-P chain. Then, after adsorption of battery industry wastewater, there is an increase in the intensity of the C-O-P functional group and a decrease in the intensity of C-O, indicating a change in the functional group that has bound ions Pb^{2+}, so that the H_3PO_4-activated water hyacinth bioadsorbent becomes an alternative as a wastewater treatment for lions Pb^{2+}.

Keywords:
Activated H_3PO_4;
Lead ion (Pb^{2+});
Metal ion adsorption;
Water hyacinth bioadsorbent;
Wastewater of battery industry.

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INTRODUCTION

The development of the battery industry can cause battery wastewater to increase due to higher battery consumption and short battery life, becoming an environmental problem. Batteries contain manganese-Mn, lead-Pb, cadmium-Cd, and lithium-Li, but lead has a very high toxicity for living things [1]. Exposure to Pb in waters can cause damage or death to marine life, and if in humans, it can cause anemia, damage to the nervous system, reduced immune function, nausea, and kidney damage in the long term [2]. Therefore, there needs to be a solution for reducing heavy metals such as Pb before they are discharged into the environment.

There are several methods that can be used to reduce the concentration of lead ions (Pb^{2+}) in wastewater, including precipitation, ion exchange using resin, filtration, adsorption, and membrane. Adsorption is a highly efficient technique compared to other techniques that have limitations such as low efficiency, critical operating conditions, and high disposal costs [3]. The types of adsorbent media commonly used include activated carbon, zeolite, silica gel, bentonite, chitosan, and bioadsorbents [4]. These types of bioadsorbents are widely used because bioadsorbent manufacturing materials are easily found in the surrounding environment. One of the bioadsorbent materials that can be used is water hyacinth [5]. Water hyacinth can be used as a bioadsorbent without being made into activated charcoal first; it is simply dried and mashed [6]. This is because water hyacinth contains 60% cellulose, 17% lignin, and 8% hemicellulose, and from this content, water hyacinth has active sites such as hydroxyl (-OH), which can attract heavy metal ions [7], [8].

Hydroxyl active sites on water hyacinth can be identified from the analysis of functional groups through Fourier Transform-Infra Red (FT-IR) testing and are found in the frequency range of wave numbers 3550–3200 cm^{-1}. In addition, other functional groups contained in water hyacinth can be seen in Table 1. The activation process aims to enlarge the pores of the adsorbent by breaking hydrocarbon bonds or oxidizing adsorbent surface molecules so that it changes the surface area, which increases in size and affects the adsorption ability [9].

Table 1. FT-IR Characterization Wavelengths [8], [10]

Functional Group	Type of Compound	Frequency Area, cm^{-1}
O-H	Alcohol, phenol	3550-3200
C-H	Alkanes	2962-2853
C=C	Alkenes	1680-1600
C-OH	Alcohol	1470-1380
C-N	Amines	1360-1250
C-O	Ether	~1240
C-O-P	Carbon, oxygen, and phosphorus	1070-1090
C-O	Ether	1250-900
C-H	m-Aromatics	800-600
≡C-H	Alkyne	700-600

The use of H_3PO_4 activator to activate water hyacinth bioadsorbent results in the formation of ionized bonds in phosphoric acid and symmetrical shifts in the C-O-P chain according to reaction equation (1) [11].

$$-C-OH + H_3PO_4 \rightarrow -C-O-PO(OH)_2 + H_2O \quad (1)$$

The use of H_3PO_4 activator can increase oxygen-containing functional groups, including acidic hydroxyl, carboxyl, and phenolic hydroxyl. Therefore, adsorption of lead ions (Pb^{2+}) can occur through ion exchange, surface complexation by oxygen bonds or weak bonds in the presence of van der Waals forces, or electrostatic attraction [11].

Based on the results of previous studies, several types of chemical activators used for bioadsorbent activation, namely, research by [12] using HCl and HNO_3 activation on water hyacinth, found that water hyacinth activated carbon activated using HCl adsorbs faster, while HNO_3 activation adsorbs more. Another type of activator is H_3PO_4. A bioadsorbent activated by H_3PO_4 is able to absorb Pb^{2+} metal ions. This is evidenced by research conducted by [13], namely water hyacinth bioadsorbent and bentonite activated by H_3PO_4 with a concentration of 1.2 M in as much as 250 mL, showing an optimum absorption efficiency of 99.990% with a composition ratio of 50:50 and an agitation of 200 rpm for 60 minutes.

H_3PO_4 activator can expand the pores of the adsorbent; this has been proven based on the research results of [14], who found that activated charcoal from Gumitir (*Tagetes erecta*) plant waste activated with H_3PO_4 has a surface area of 36.4505 m^2/g and a number of active sites of 37.1292×1020 molecules/g, while charcoal without activation has a surface area of 28.2206 m^2/g and a number of active sites of 29.9920×1020 molecules/g. Based on the above background, the purpose of this study is to determine the characterization of water hyacinth (*Eichhornia crassipes*) bioadsorbent activated with H_3PO_4 on the adsorption of lead ions (Pb^{2+}) in battery industry wastewater.

RESEARCH METHODS

Materials

The materials used were water hyacinth obtained from Kecamatan Purwakarta, Jawa Barat; aquadest; 1.2 M H_3PO_4 solution; and 20 ppm Pb^{2+} standard solution. This standard solution was used to determine the optimum contact time for adsorption of lead ions (Pb^{2+}) and was used as an artificial wastewater of battery industry (one of the companies in Karawang). The equipment used in this study includes a digital technical balance (brand SF-400C), 500 mL of beaker glass (Pyrex), crusher (Magic Blend MB21BL), oven, magnetic stirrer, 100-mesh sieve shaker, filter paper no. 42 (Whatman), 25 mL microbiuret (Pyrex) along with clamps and statives, FT-IR spectrometer (Nicolet iS50 Thermo Scientific), and atomic absorption spectrophotometry (Shimadzu AA-7000).

Methods

The part used is the stem of water hyacinth to be used as a bioadsorbent. Pretreatment of raw materials is carried out to remove adhering dirt. The raw material was chopped and dried in an oven (150°C for 80 minutes). The material was mashed and sieved using a 100-mesh sieve shaker.

The water hyacinth bioadsorbent was activated using 1.2 M H_3PO_4 solution in a ratio of 25 g to 350 mL. Both were mixed for 30 minutes and allowed to stand for 24 hours. Next, the activated water hyacinth bioadsorbent residue was neutralized through the addition of distilled water (up to pH 7). The residue was dried at 105°C for 2 hours [13].

The activated bioadsorbent was contacted with a standard solution of lead ions (Pb^{2+}) to determine the optimum contact time for adsorption and artificial wastewater of battery industry. The ratio was 0.5 g/100 mL with a contact time variation of 20, 30, 40, 50, 60, 70, 140, 210, and 280 minutes with constant agitation. Furthermore, the sample was separated between filtrate and residue through the use of filter paper.

The analysis of the adsorption rate of battery industry wastewater in determining adsorption capacity utilizes equations (2) and (3). Its effectiveness uses equation (4) [15], [16].

the adsorption capacity

$$q_e = \frac{(C_o - C_e)}{m} V \tag{2.1}$$

$$q_t = \frac{(C_o - C_t)}{m} V \tag{2.2}$$

Effectiveness

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

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.

Adsorption kinetics are used to determine the rate of adsorption and also provide information about the adsorption mechanism. This fulfills the need to predict the time taken to reach adsorption equilibrium and the maximum adsorption concentration [17]. The adsorption kinetics modeling used is pseudo-first-order and pseudo-second-order. The kinetic equations are shown in equations (4) and (5) [18], [19].

the pseudo-first order kinetic

$$\ln(q_e - q_t) = \ln q_e - k_1 t \tag{4}$$

the pseudo-second order kinetic

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} \tag{5}$$

The values for k_1 and k_2 represent the rate constants for pseudo first-order (min^{-1}) and pseudo second-order (in $g.mg^{-1}.min^{-1}$) reactions, respectively, with t denoting the contact time (in minutes).

RESULTS AND DISCUSSION

The use of water hyacinth bioadsorbent is obtained from the drying process, followed by activation with 1.2 M H_3PO4 to expand the pores of the water hyacinth bioadsorbent [13]. Figure 1 shows the water hyacinth bioadsorbent before activation, and after activation there is a change in color due to the removal of hydroxyl groups on the surface of the water hyacinth stem and an increase in carbon content in the water hyacinth bioadsorbent after activation.

Figure 1. Water Hyacinth Powder: (a) Before, (b) After Activation

FT-IR on Water Hyacinth (*WH*) Bioadsorbent

Characterization of bioadsorbent research samples using FT-IR analysis to determine the functional groups that adsorb lead ions (Pb^{2+}). The results of qualitative analysis through this method both before and after activation of 1.2 M H_3PO_4 and after adsorption of battery industry wastewater are able to provide different spectra patterns. The presentation of the results in Figure 2 shows the difference in spectra between the three processes. The results indicate a shift, reduction, or disappearance of the spectrum when the activation and adsorption processes take place [20].

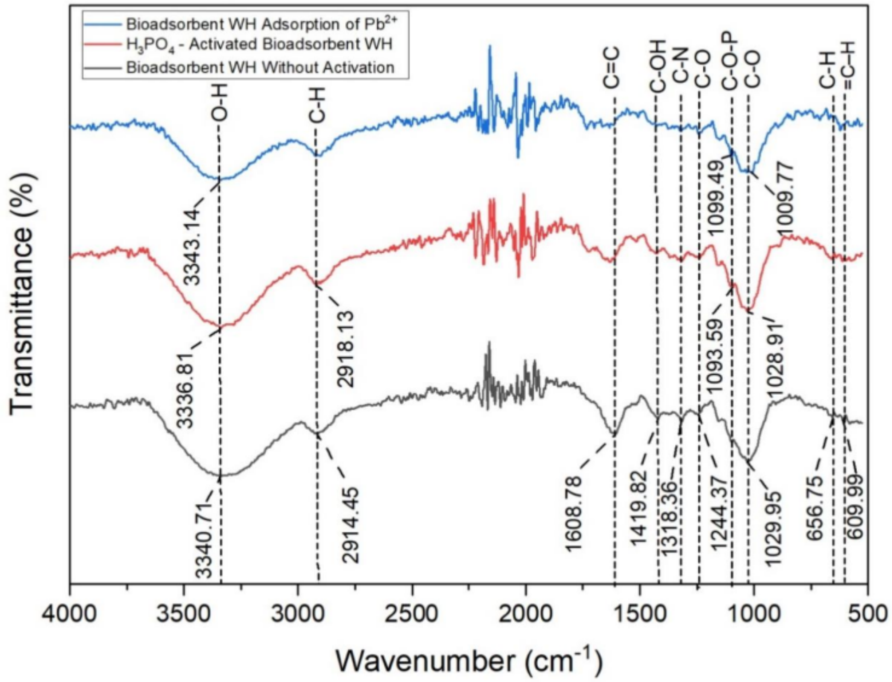

Figure 2. Comparison of Spectra of Water Hyacinth Bioadsorbents

The results of FT-IR analysis in Figure 1 show the presence of widened wave numbers in the three bioadsorbent samples in the spectrum range of 3550–3200 cm^{-1}. These results indicate the presence of active sites, namely the hydroxyl group (O-H), which functions as an ion exchange with lead ions (Pb^{2+}). This functional group also indicates the presence of cellulose [8]. The wavelengths of 2914.45 and 2918.13 cm^{-1} in the water hyacinth bioadsorbent sample without activation and water hyacinth bioadsorbent after H_3PO_4 activation, respectively, are able to show the stretching vibrations in the O-H and C-H bonds. This indicates the stretching of hydrogen bonds and the bending of hydroxyl groups in the cellulose structure [21]. Then, the adsorption peak appears at 1608.78 cm^{-1} and this indicates the presence of the C=C functional group as an increase in carbon content [22]. However, the removal of the C=C functional group also took place due to the delignization process, so that the intensity of C=C after H_3PO_4 activation decreased. The sharp peak was 1029.95 cm^{-1}. This figure displays the adsorption of C-O group stretching vibrations similar to the results of research [23], which is a carbon chain link in cellulose (glycosidic bond) [24].

The wavelength number after H_3PO_4 activation displays a small peak of 1093.59 cm^{-1}. The achievement is due to the presence of ionized P^+-O^- bonds in phosphoric acid and symmetrical vibrations in the C-O-P chain in accordance with the equation (1) [11]. Then, the sample after adsorption of battery industry wastewater showed an increase in the intensity of the C-O-P functional group and a decrease in C-O intensity. This can occur due to changes in functional groups that have bound lead ions (Pb^{2+}).

Contact Time (*CT*) for Artificial Wastewater of Battery Industry

In conducting adsorption, it is necessary to vary the contact time between H_3PO_4-activated water hyacinth bioadsorbent and artificial wastewater from the battery industry. This stage aims to determine the interaction time needed by H_3PO_4-activated water hyacinth bioadsorbent in adsorbing the artificial wastewater of the battery industry until it reaches equilibrium. Table 2 shows the adsorption efficiency of H_3PO_4-activated water hyacinth bioadsorbent on artificial wastewater of battery industry.

Table 2. AAS results for contact time and modeling of adsorption kinetics

CT, minutes	R, %	Parameters	1st- Order Pseudo	2nd- Order Pseudo
20	88.118	k_1	-0.011100	-
30	88.904	k_2	-	0.066291
40	89.189	q_e calc	-0.63526	4.0388
50	90.109	q_e exp	3.9975	3.9975
60	91.281			
70	91.950			
140	97.551			
210	97.108			
280	96.939			

Initial concentration = 20.489 ppm

The optimum adsorption of lead ions (Pb^{2+}) took place at a contact time of 140 minutes by leaving the wastewater concentration of 0.50170 ppm. Extension beyond the optimum time does not provide a significant effective value. This event is thought to be due to the pores of the bioadsorbent being in a saturated condition when adsorbing lead ions (Pb^{2+}). This finding is also in accordance with research [25] showing that adsorption of artificial wastewater using water hyacinth activated charcoal with contact time above 150 minutes experienced a reduction in lead (Pb) levels with a percentage of 99.99%.

Kinetics of Reaction

Adsorption kinetics are used to determine the rate of adsorption that occurs on the adsorbent to absorb adsorbate and is influenced by contact time. Contact time is used to achieve adsorption equilibrium, so contact time is used as a measure of adsorption rate. The adsorption rate can be known from the adsorption rate constant (*k*) and the reaction order resulting from an adsorption kinetics model [26]. The adsorption rate test was conducted to estimate the reaction order model (pseudo first-order and pseudo second-order) presented in Figure 3 and Table 2.

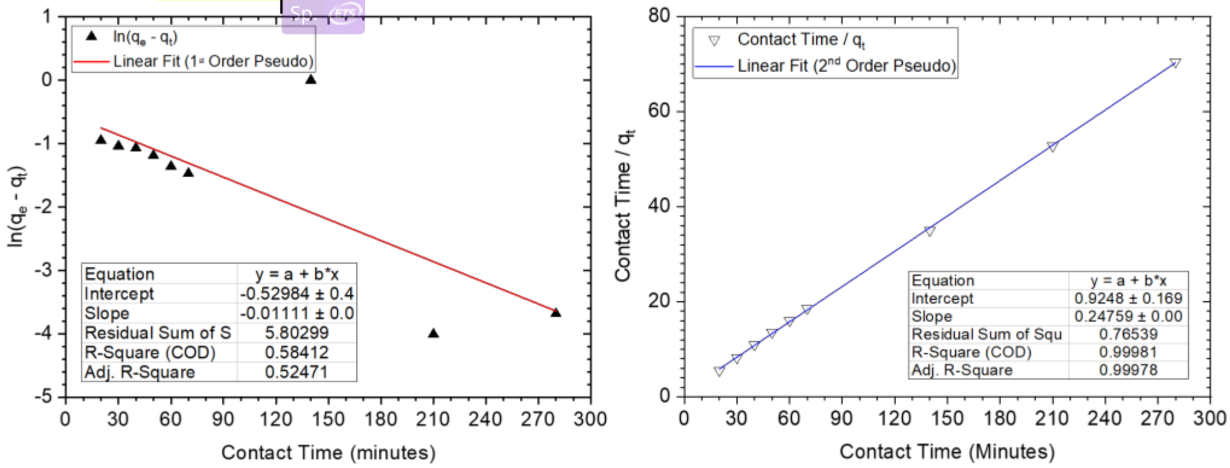

Figure 3. Modeling of adsorption kinetics: first, and second-order pseudo

Both appearances in Figure 3 show that the adsorption kinetics of H_3PO_4-activated water hyacinth bioadsorbent on lead ions (Pb^{2+}) follow second-order pseudo-modeling. The second-order pseudokinetics in this study indicate the presence of a chemical adsorption process or chemisorption of the adsorbate on the adsorbent. Chemisorption takes place when metal ions attach to the surface of the adsorbent by forming chemical bonds and tend to find sites that maximize the number of ion bonds with the bioadsorbent surface. This is in accordance with the statement of research conducted by [27]. The second-order pseudo-kinetics model has a rate-limiting step, which is adsorption on the surface of bioadsorbents involving chemisorption, where there is removal from a solution caused by physicochemical interactions between the two phases.

Lead Ion Adsorption (Pb^{2+}) from Wastewater Battery Industry

Preliminary analysis through the AAS showed that the initial concentration of lead ions (Pb^{2+}) in the wastewater of the battery industry was 8.1488 ppm. The Regulation of Kementerian Lingkungan Hidup Republik Indonesia No. 5 Tahun 2014 on the standard quality of waste water indicates that the threshold value of metal Pb is 0.1 mg/L [28]. After activation and contact with the artificial, the results of this study showed a questionable decrease in concentration to the remaining 0.2503 mg/L. This case proves that its effectiveness in absorbing lead ions (Pb^{2+}) from the wastewater of the battery industry was 96.928% with the previous optimal time contact. On the other hand, the findings do not fully meet the criteria of the regulation. The wastewater also has the possibility of containing metals or other complex compounds that have not been further analysis (other treatments) [29], [30].

CONCLUSION

The results of the characteristic bioadsorbents of goiter through FT-IR analysis showed that the pre-activated water hyacinth bioadsorbent had functional groups of O-H, C-H, C=C, and O-OH with wavelengths of 3340.71, 2914.45, 11608.78, and 1419.82 cm^{-1}, respectively. All four groups of functions indicate the presence of cellulose. Bioadsorben after 1.2 M H_3PO_4-activated showed the elimination of functional groups C=C and the decrease in the intensity of the number of wavelengths in O-H and C-H bonds at 3336.81 and 2918.13 cm^{-1}. Then, the presence at 1093.59 cm^{-1} indicated the functional group existence of a symmetrical vibration in the chain C-O-P. The results of this study also showed that the optimal contact time of bioadsorbents in 1.2 M H_3PO_4-activated solutions to the standard solution of lead ions (Pb^{2+}) is 140 minutes, with a value of effectiveness in the adsorption of these lead ions of 96.928%.

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