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Characterization of *Eichhornia crassipes* bio-adsorbent activated by H_3PO_4 for the removal of lead ion (Pb^{2+}) from wastewater of battery industry

Reza Erlangga[1], Dessy Agustina Sari[1,2]*, Aulia Wahyuningtyas[1]
[1]Chemical Engineering Program, Faculty of Engineering, Universitas Singaperbangsa Karawang, Indonesia
[2]Department of Chemical Engineering, Faculty of Engineering, Universitas Diponegoro, Indonesia

Abstract
Lead ion (Pb^{2+}) contamination from battery industry wastewater affects significant environmental and health risks. This study explored the use of H_3PO_4-activated water hyacinth (WH) bio-adsorbent as an effective solution for removing Pb^{2+}. The WH bio-adsorbent was prepared by activating dried water hyacinth stems with 1.2 M H_3PO_4, enhancing adsorption properties. SEM-EDX analysis revealed significant morphological changes, with increased porosity and oxygen-containing functional groups (O-H, C-O-P), which improved adsorption capacity. Adsorption kinetics followed a pseudo-second-order model (R^2 = 0.99981), indicating that chemisorption dominated the Pb^{2+} removal process. Adsorption isotherms firmly fit the Langmuir model (R^2 = 0.96), confirming monolayer adsorption on a homogeneous surface. The effect of pH was also investigated, with maximum adsorption efficiency (96.928%) observed at pH 7. FTIR analysis showed changes in functional groups before and after adsorption, confirming the ion exchange mechanism between Pb^{2+} and the activated bio-adsorbent. The findings suggest that H_3PO_4 activation increases the surface area and raises the chemical activity of WH, providing new insights into the dual mechanism of physical and chemical modifications for lead removal. This study addresses a critical gap in optimizing adsorbents for heavy metal removal, demonstrating the potential of H_3PO_4-activated WH for industrial wastewater treatment.

Keywords:
H_3PO_4 activated;
Lead ion (Pb^{2+});
Metal ion adsorption;
Water hyacinth bio-adsorbent;
Wastewater of battery industry;

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Corresponding Author:
Dessy Agustina Sari
Chemical Engineering Program, Universitas Singaperbangsa Karawang, Indonesia; and Department of Chemical Engineering, Universitas Diponegoro, Indonesia
Email: dessy.agustina8@staff.unsika.ac.id

INTRODUCTION

Lead contamination in battery industry wastewater affects significant environmental and health hazards. Higher battery usage and shorter battery life due to the expansion of the battery sector, which increases battery wastewater and aggravate ecological pollution issues. Lead is a non-biodegradable carcinogen with great toxicity, posing a significant risk. Lead [1] is frequently present in wastewater from many sectors, including mineral processing, battery manufacture, textile dyeing, petroleum refining, and mining. Among them, lead is particularly alarming as it is difficult to decompose naturally and is relatively harmful to living entities. Although lead (Pb), cadmium (Cd), manganese (Mn), and lithium (Li) are usually metals included in batteries, lead shows the most toxicity to aquatic life and humans [2].

Lead exposure to water bodies can cause serious medical problems. Lead pollution can cause anemia, nervous system damage, lower immune system function, nausea, and over time, renal damage in humans [3]. Also, lead can have a lethal impact on aquatic life, as well as endangering whole ecosystems.

Consequently, before wastewater is released into the environment, heavy metals, including lead, must be eliminated from it.

Various techniques, including precipitation, ion exchange, filtration, anaerobic treatment, and adsorption, have been investigated to help reduce lead pollution. Among these, adsorption is a very efficient method because of its simplicity, cost-efficiency, and effectiveness at low concentrations, despite some constraints such as critical operating conditions and disposal costs [4][5]. Although certain advantages may come from techniques like membrane filtration, the high running costs and sustainability problems resulting from membrane fouling usually outweigh the benefits. As a result, adsorption using adsorbents has attracted people's interest as a workable wastewater treatment method, especially for removing heavy metal ions [6][7].

Adsorbents—activated carbon, nanomaterials, zeolites, silicic gel, and several natural materials—have been extensively applied because of their availability, economy, and environmental friendliness. Natural adsorbents such as water hyacinth (WH) promise high adsorption efficiency and accessibility [8][9]. WH (*Eichhornia crassipes*), due to its natural characteristics and great availability, has become a successful adsorbent for heavy metal removal.

Studies have shown WH's high adsorption effectiveness for several heavy metals, including lead, with the ability to remove up to 90% of lead ions (Pb^{2+}) from water [10]. Active sites found in WH, including hydroxyl (-OH) groups, can draw and bind heavy metal ions [11][12], as listed in Table 1.

For Cu^{2+} and Pb^{2+} in Table 1 during a 2-hour cycle at concentrations up to 10 mg/L, for example, earlier investigations have indicated that WH bio-adsorbents might attain an adsorption efficiency of 60% [13]. Furthermore, WH adsorbents are efficient at removing ammonia from wastewater, with an average effectiveness of 87% [14].

Even with these encouraging findings, there remains a gap in the knowledge regarding the best conditions and techniques for bio-adsorbent activation to improve lead removal efficiency. Although many studies have examined the use of adsorbents for heavy metal removal, the specific use of H_3PO_4-activated water hyacinth for optimizing lead adsorption under diverse conditions has yet to be fully investigated.

Table 1. FTIR Characterization Wavelengths [1, 15, 16]

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

This work aims to assess the adsorption efficiency of WH activated using 1.2 M H_3PO_4 for the elimination of Pb^{2+} from battery industry wastewater. The activation process is supposed to expand the adsorbent's surface area and boost the quantity of oxygen-containing functional groups, thereby improving its adsorption capacity. Using SEM-EDX, FTIR, and AAS, characterizing the activated bio-adsorbent helps determine its potential use in wastewater treatment for lead ion elimination.

METHOD

Materials

The materials used were WH 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 applied to determine the optimum contact time for the adsorption of Pb^{2+} and it was taken as an artificial wastewater in the battery industry (one of the companies in Karawang). The equipment used in this study was 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 micro biuret (Pyrex) along with clamps and statives, SEM-EDX (JEOL JSM-6510LA), FTIR spectrometer (Nicolet iS50 Thermo Scientific), and atomic absorption spectrophotometry (Shimadzu AA-7000).

Methods

The stem of WH was the part that was harnessed to be an adsorbent. Pretreatment of raw materials until adsorption of the Pb^{2+} is shown in Figure 1. The adsorption rate of battery industry wastewater in determining adsorption capacity is analyzed using (1). Its effectiveness uses (2) [17][18].

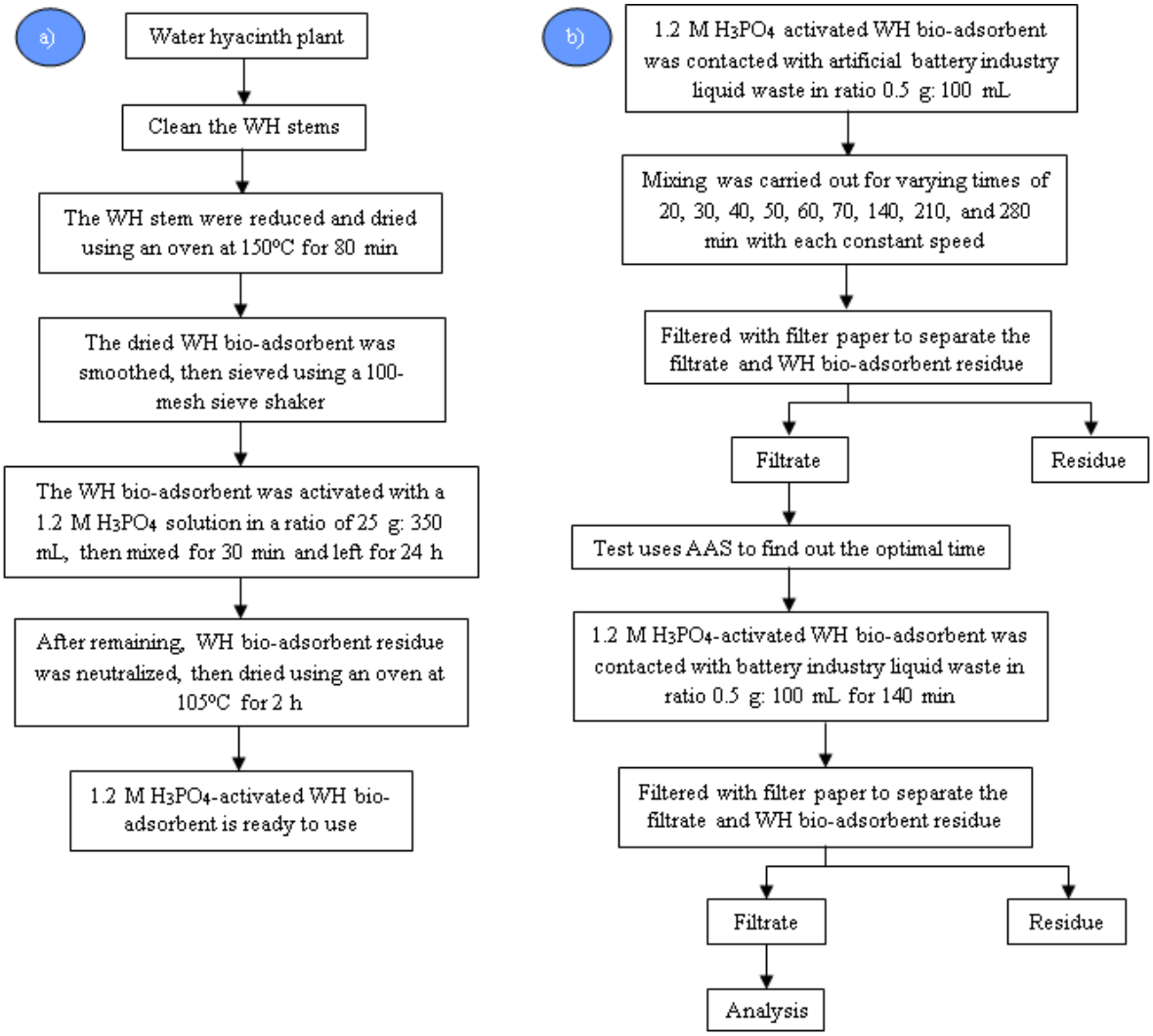

Figure 1. The Process: a) Making an Adsorbent (WH) Activated by 1.2 M H_3PO_4, and b) Bio-Adsorbent Contact with The Effluent

$$q_e = \frac{(C_o - C_e)}{m} V \quad (1)$$

$$q_t = \frac{(C_o - C_t)}{m} V \quad (2)$$

The parameters q_e and q_t represent the adsorption capacity at equilibrium and time t (in minutes) in units of mg/g. Meanwhile, R indicates the percentage of adsorbed metal, %. C_o, C_e, and C_t values represent the initial, equilibrium, and time t metal concentrations in units of mg/L. The mass of the adsorbent–m and the volume of the solution should also be considered in units of g and L.

Temperature, the mass of the adsorbent, and the ion concentration fed are the factors that influence a metal's equilibrium. The first factor contributes to solid particles' solubility and molecular interactions under endothermic or exothermic conditions. The second is to show the presence of an active site owned by an adsorbent. Excess of this factor can reduce the adsorption capacity due to the accumulation of articles and have an impact on reducing the active site surface [19]. The last factor is to increase the mass driving force on the adsorbent surface to saturated conditions.

Adsorption kinetics are used to determine the adsorption rate and provide information about the adsorption mechanism. This is essential to predict the time taken to reach adsorption equilibrium and the maximum adsorption concentration [20]. The adsorption kinetics modeling used is pseudo-first-order and pseudo-second-order. The kinetic equations are shown in (3) and (4) as [21, 22, 23]:

The pseudo-first order kinetic

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (3)$$

The pseudo-second order kinetic

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^{\,2}} + \frac{1}{q_e} \quad (4)$$

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, while, t represents the contact time (in minutes).

RESULTS AND DISCUSSION

Preparation and characterization of H_3PO_4-activated WH bio adsorbent

The WH bio-adsorbent, as depicted in Figure 2, was prepared by drying and activating the stems using a 1.2 M H_3PO_4 solution. The activation process changed the WH powder from light brown (Figure 2a) to a deeper tint (Figure 2b), indicating a reduction in contaminants. During the activation, this color change suggests the elimination of organic contaminants and an increase in porosity by generating more active surfaces. The chemical activation process can reduce the amount of impurities in natural adsorbents, supporting more effective adsorption [24].

The researchers were using SEM-EDX, and the surface shape of the adsorbent showed notable changes upon activation with H_3PO_4, as shown in Figure 3. The SEM pictures showed that the activated WH had a more porous and consistent surface than the unactivated sample, implying increased surface area and porosity—essential qualities for improving adsorption capacity.

Figure 2. Water Hyacinth Powder: (a) before, (b) after Activated

Confirming the effective activation of the WH adsorbent, the EDX analysis (Figure 3) revealed a high percentage of oxygen-containing groups.

SEM-EDX on water hyacinth bio-adsorbent

Before and after activation with 1.2 M H_3PO_4, the WH bio-adsorbent's surface morphology and elemental content were investigated using SEM-EDX analysis. The inactivated bio-adsorbent's SEM images in Figure 3a display a rough and uneven surface with low porosity. By contrast, Figure 3b shows that the adsorbent shows a more porous and ordered surface shape after using H_3PO_4 activation. Moreover, for raising the adsorption capacity, the activation process has expanded the pores and raised the surface area of the adsorbent.

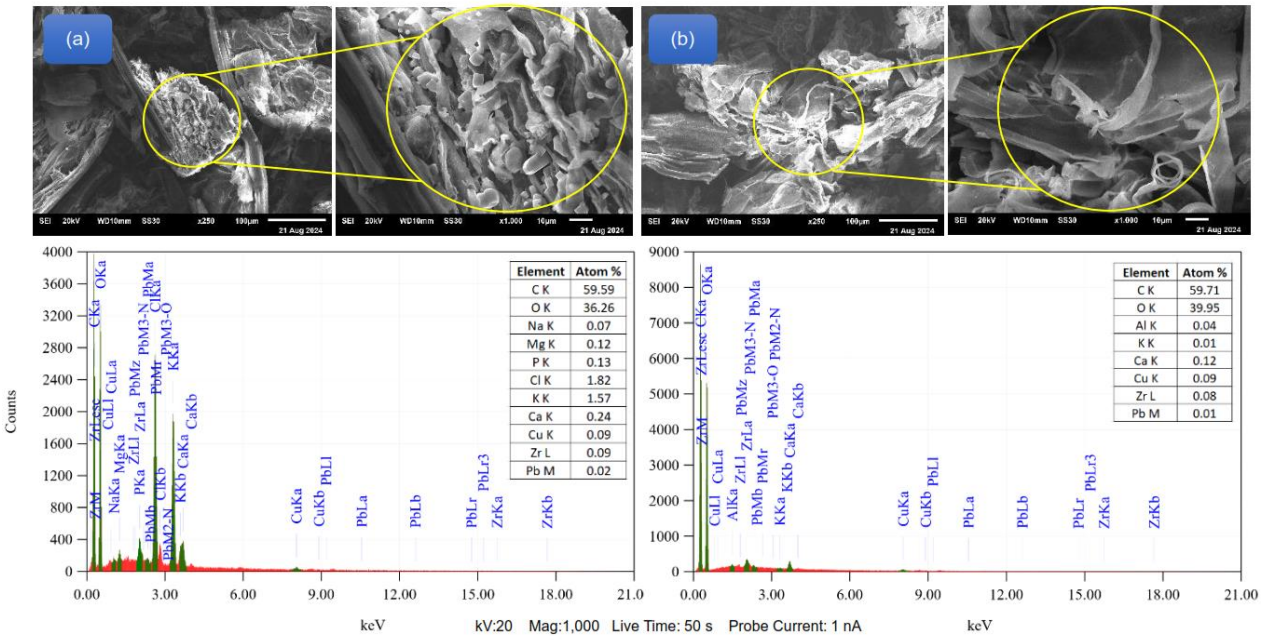

Figure 3. SEM-EDX Analysis of WH: (a) before, (b) after Activated

The estimated results at 1000x magnification revealed porosity or space ranging from 60.37 to 63.92%. Activation using 1.2 M H_3PO_4 solution indicated a slight difference between the adsorbent surface before and after activation. This is in line with those of [25], that found bio-adsorbents triggered by H_3PO_4 to have a similar surface shape. However, the spread of Pb^{2+} into the pore surface is more important than the total percentage of porosity.

The EDX analysis showed an elevated oxygen content of nearly 10% after activation, supporting our observations (Figure 3b) from 36.26 to 39.95%. According to the elemental composition study, the oxygen percentage rose from 36.26 in the inactivated bio-adsorbent to 39.95% in the activated sample. The production of extra oxygen-containing functional groups resulting from the H_3PO_4 activation—hydroxyl (O-H) and phosphate (C-O-P)—is attributed to this increase in oxygen. These groups promote the mechanism for lead removal via chemical interactions instead of only physical adsorption by adding extra active sites for binding Pb^{2+}, hence improving adsorption qualities.

Adsorption kinetics of lead ions

Analyses of the Pb^{2+} adsorption kinetics on the H_3PO_4-activated WH bio-adsorbent determined the rate and mechanism of adsorption. This study investigated the adsorption process at various contact durations (20, 30, 40, 50, 60, 70, 140, 210, and 280 minutes, as listed in Table 2), with findings based on pseudo-first-order and pseudo-second-order kinetic models (as depicted in Figure 4).

Table 2. AAS Results for Contact Time and Modeling of Adsorption Kinetics

CT, minutes	R, %	Parameters	1st-Order Pseudo	2nd 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 higher R^2 value (0.99981) compared to the pseudo-first-order model (0.58412) suggested that the adsorption followed a pseudo-second-order kinetic model, implying that chemisorption is the primary process. This result is aligned with the studies by [26], which showed that chemisorption controls the adsorption of heavy metal ions onto adsorbents.

Adsorption isotherms

The adsorption isotherms were evaluated using the Freundlich and Langmuir models to understand the adsorption mechanism further. The Langmuir isotherm model, which assumes monolayer adsorption on a homogeneous surface, provided a better fit (R^2 = 0.96) than the Freundlich model. This indicates that the adsorption of Pb^{2+} onto the H_3PO_4-activated WH bio-adsorbent is primarily monolayer, with adsorption sites being uniformly distributed. Similar findings were reported by [27], where H_3PO_4 activation improved adsorption uniformity for heavy metal removal.

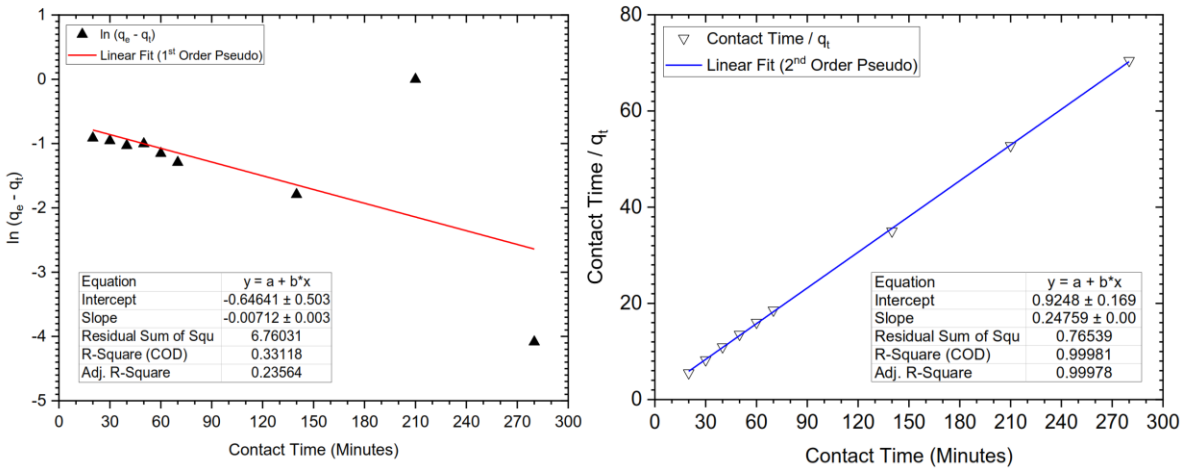

Figure 4. Modeling of Adsorption Kinetics: First, and Second-Order Pseudo

Effect of pH on adsorption efficiency

The pH of the solution strongly influences the adsorption effectiveness of lead ions. The update data showed that the adsorption effectiveness performed the highest at a pH of 7 and attained a maximum adsorption percentage of 96.928%. The adsorbent surface becomes more positively charged at low pH values, lowering the electrostatic attraction between the negatively charged Pb^{2+} and the adsorbent. On the other hand, the surface charge becomes more damaging when pH values increase, improving the adsorption capacity. This tendency is compatible with the results of [28], who noted poorer adsorption effectiveness at lower pH levels due to growing competition between hydrogen and metal ions.

FTIR on water hyacinth bio-adsorbent

The adsorbent's FTIR spectra, as shown in Figure 5, revealed significant functional group modifications both before and after activation and following Pb^{2+} adsorption. Adsorption peaks at 3340.51, 2914.45, 1608.78, and 1419.82 cm^{-1}, in that order, confirmed the presence of O-H, C-H, C=C, and C-O functional groups. When H_3PO_4 was added, the C=C peak's intensity clearly dropped, which means it became less lignified. At 1093.59 cm^{-1}, a new peak for the C-O-P group appeared.

It's possible that the lead ions formed complexes with these functional groups because the C-O-P group intensity went up while the C-O group intensity went down after the Pb^{2+} was absorbed. The ion exchange mechanism is shown in more detail in (5) and (6).

$$-C-OH + H_3PO_4 \rightarrow -C-O-PO(OH)_2 + H_2O \quad (5)$$

$$-C-O-PO(OH)_2 + Pb^{2+} \quad (6)$$

This is when hydroxyl groups (-OH) on the adsorbent react with Pb^{2+} to form covalent bonds. These changes to the functional groups add to what was found in [29], who also found that activated adsorbents and heavy metal ions interact similarly. The findings of [12] similarly suggest that the change in functional groups after Pb^{2+} adsorption indicates an ion exchange mechanism between Pb^{2+} and the bioadsorbent.

Comparison with similar studies

The study's results show that activating H_3PO_4 has a big effect on the ability of water hyacinth (WH) bio-adsorbents to take in Pb^{2+}. It was most effective at adsorption (96.928%) when the pH was 7. This is in line with other studies of activated bio-adsorbents for heavy metal removal (R^2 = 0.96), as well as pseudo-second-order kinetics (R^2 = 0.99981). For instance: a study [10] from 2022 found that magnetic bio-adsorbents from water hyacinth activated by citric acid were 91% effective at getting rid of Pb^{2+}. This study demonstrated functionalized WH's potential, but it couldn't match the adsorption capacity of H_3PO_4 activation under optimal conditions.

The researchers [8] got 87% efficiency using activated carbon from *Rumex abyssinicus*. They emphasized the significance of porosity and oxygen-containing functional groups, which we also observed in our study. Then, the study [27] [30] looked at how to get rid of chromium and lead using water hyacinth bio-adsorbents activated by phosphoric acid and nitric acid.

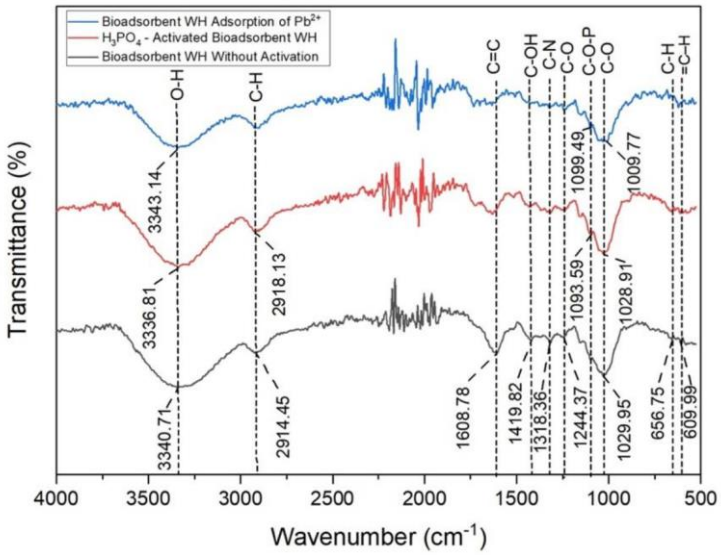

Figure 5. Comparison of Spectra of Water Hyacinth Bio-Adsorbents

It found that bio-adsorbents activated by H_3PO_4 had similar surface modifications and adsorption mechanisms, which showed that phosphoric acid is an effective way to activate bio-adsorbents.

These vanity comparisons reflect the unique merits of H_3PO_4 activation, particularly the development of substantial surface area as well as active functional groups (O-H and C-O-P), yielding higher Pb^{2+} adsorption performance. Also, while NaOH and citric acid have been studied as activation agents before, we show that H_3PO_4 is even better at getting rid of heavy metals in the conditions we tested. Future work could build on this by varying the H_3PO_4 concentrations used or determining their efficacy against other pollutants and combination wastewater level detection monitoring system [31] with the fabrication of the lead sensor [32].

CONCLUSION

With an optimum contact period of 140 minutes and maximum efficiency at pH 7, the paper validates that the H_3PO_4-activated water hyacinth (WH) bio-adsorbent shows a high adsorption capacity for Pb^{2+}. Langmuir isotherm model and pseudo-second-order kinetics point to monolayer and chemisorption as adsorption mechanisms. This research anwers a significant knowledge gap on how bio-adsorbent activation improves adsorption capacity under practical conditions by optimizing the conditions for H_3PO_4-activated WH bio-adsorbent and proves its efficiency in removing Pb^{2+} from battery industry wastewater. These results highlight the physical (more surface area) and chemical (improved functional groups) changes obtained by H_3PO_4 activation, implying possible uses for treating industrial wastewater.

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