Assessing the Lead Removal Potential of Pineapple Waste Hydrochar in Simulated Wastewater Treatment

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A R T I C L E I N F O

A B S T R A C T

This study explores the viability of utilizing biochar and hydrochar derived from pineapple waste as adsorbents for removing lead (Pb) from water. Pineapple residues, including stems, leaves, and fruit, were subjected to pyrolysis and Hydrothermal Treatment to produce biochar (PWB) and hydrochar (PWH) respectively. Fourier Transform Infrared (FT-IR) analysis was employed to characterize the surface properties of PWB and PWH, validating their potential as adsorbents. A series of adsorption experiments assessed the impact of pH (ranging from 2 to 6), contact time (ranging from 15 to 90 minutes), and temperature (ranging from 30 to 90°C) on the adsorption efficiency of both materials. Results indicate PWH to be markedly more effective, with an average Pb removal efficiency of 84.07% compared to PWB's 55.68%. The optimal contact time was determined to be 60 minutes for both materials. Moreover, pH 4.0 was identified as the optimal condition for Pb adsorption, showcasing a significant increase in biosorption capacity within this pH range. Additionally, higher temperatures corresponded to enhanced Pb^{2+} removal efficiency, rising from 75.02% to 87.58% as temperature increased from 30°C to 90°C. Overall, these findings underscore the potential of pineapple waste-derived biochar and hydrochar as promising, environmentally friendly adsorbents for addressing heavy metal contamination in water, particularly in wastewater treatment applications.

INTRODUCTION

Since water is one of the most fundamental human needs—along with food, housing, and clothing—it is one of the most important resources. 71% of the Earth's surface is covered with water. Water, not food, is what most living things need to survive longer and to maintain life. These days, urbanization and population expansion have increased the demand for water use while also raising the worldwide level of water pollution (Crini et al., 2019; Vivek Kumar Gaurav et al., 2018; Vivek Kumar Gaurav & Sharma, 2020). Rapid growth has therefore resulted in a considerable volume of human waste being created, including commercial, industrial, and household trash. These wastes end up in water bodies and raise the concentration of heavy metals in rivers. Because heavy metals are poisonous and have negative effects on human health, they are a categorized chemical element that poses a serious risk to both humans and the environment (V.K. Gaurav et al., 2018; Kumar Sharma et al., 2007; Mehra et al., 1998). Lead is one of the main heavy metal components found in wastewater which is a harmful substance to human health. It can lead to abortion, miscarriage, elevated blood pressure, and brain damage. When lead reaches the fetus through the mother's placenta, it also has an impact on the developing child's brain and nervous system (Akpor, 2014).

Therefore, since the amount of lead in wastewater may rise as a result of the expansion of these companies, worries regarding lead pollution should not be disregarded. As a result, a variety of cutting-edge techniques, including membrane separation, filtration, ion exchange, aerobic and anaerobic treatment, Advanced oxidation processes, electrochemical treatment and, coagulation, are available for treating these heavy metals in wastewater. The capacity of the adsorbent to bind and sequester the metal ions from wastewater makes the adsorption process one of these techniques that is frequently used to remove heavy metals from wastewater and is seen to be a promising technique (Enaime et al., 2020; Hasham et al., 2021). Additionally, this procedure is quite effective and economical while also minimizing the various forms of heavy metals. For the removal of various heavy metals from wastewater, activated carbon has shown to be a popular and efficient adsorbent. However, because the cost of adsorbent is rather expensive, utilizing activated carbon as an adsorbent for the removal of heavy metal would raise the operating process's cost. This issue necessitates the development of readily available, inexpensive materials that may be utilized more profitably in big quantities.

Because of this, a lot of research has concentrated on low-cost substitute adsorbents made from agricultural wastes. Numerous prior investigations have demonstrated that agricultural wastes with high carbon and low ash concentrations, such as empty fruit bunches, palm shells, maize cobs, coconut shells, and date palm seeds, are appropriate precursors (Bhatnagar et al., 2015; Monisha et al., 2021; Ramesh et al., 2019). Additionally, according to Bhatnagar & Monisha, agricultural waste materials, especially those that include cellulose, can sorb a variety of contaminants. These materials also have several benefits, including high sorption capacities, good recoverability and modifiability, insensitivity to harmful compounds, and ease of use in treatment procedures.

LITERATURE REVIEW

Some researchers promoted the use of potentially less costly adsorbents to remove heavy metals from wastewater (Crini et al., 2019; Debnath et al., 2021; Hesham et al., 2021). By-products of agriculture and industry, such as fly ash and rice husk, have been utilized to remove heavy metals from wastewater to clean it for the electroplating industry. He has discovered that the removal of heavy metals with concentrations between 20 and 60 mg/L may be effectively accomplished with inexpensive adsorbents. Additionally, he discovered that employing actual wastewater demonstrated the effectiveness of fly ash in the removal of Cd and Cu and the ineffectiveness of rice husk in the simultaneous removal of Fe, Pb, and Ni. The percentage of heavy metal removal was found to be influenced by the concentration and dosage of the inexpensive adsorbent. It was discovered that two hours of contact time was required for maximal adsorption and that the ideal.

Since pineapple waste is widely available worldwide, it was selected for use as an adsorbent in this investigation. The study found that 60% of the garbage from pineapple farms is pineapple farm waste, and 40% of the fruit from the entire pineapple tree is represented. Since much of this agricultural waste is unusable, it will either be thrown away or transported to a landfill. As a result, as this garbage decomposes, methane gas is released into the atmosphere, endangering both people and the environment. Using pineapple waste as an adsorbent to extract heavy metal from wastewater may significantly cut down on waste production and directly address a portion of the environmental issue.

METHODOLOGY

Pineapple waste sourced from local juice shops served as the precursor for the synthesis of pineapple waste biochar (PWB) and pineapple waste hydrochar (PWH). The synthesis process involved distinct methodologies: PWB fabrication, illustrated in Figure 1, and PWH production via a conventional Hydrothermal Carbonization (HTC) procedure. Approximately 60 grams of naturally dried pineapple waste was individually mixed with 400 ml of deionized (DI) water and introduced into a 500 ml stainless steel autoclave. This assembly underwent heating at 300°C for 5 hours, maintaining a pressure of approximately 1000 psi as monitored by a pressure gauge. Subsequent cooling to room temperature facilitated the retrieval of solid hydrochar from the processed agricultural waste.

The obtained PWH underwent successive steps to ensure purity and consistency. Initially, rinsing with DI water and subsequent drying at 80°C removed residual impurities. Following drying, the sample underwent grinding and sieving to attain a uniform particle size fraction ranging from 0.5 to 1.0 mm. A further round of rinsing with DI water eliminated any remaining impurities, followed by another drying cycle at 80°C. The resulting hydrochar samples, now purified and devoid of contaminants, were stored for future experimental endeavors.

For the production of modified hydrochar, 3 grams of the prepared hydrochar samples underwent immersion in a solution containing 20 ml of 10% hydrogen peroxide (H_2O_2) for 2 hours at room temperature (22°C ± 0.5°C). Post-reaction, thorough rinsing with DI water and subsequent drying at 80°C yielded modified hydrochar samples, meticulously stored for subsequent experimentation and potential applications.
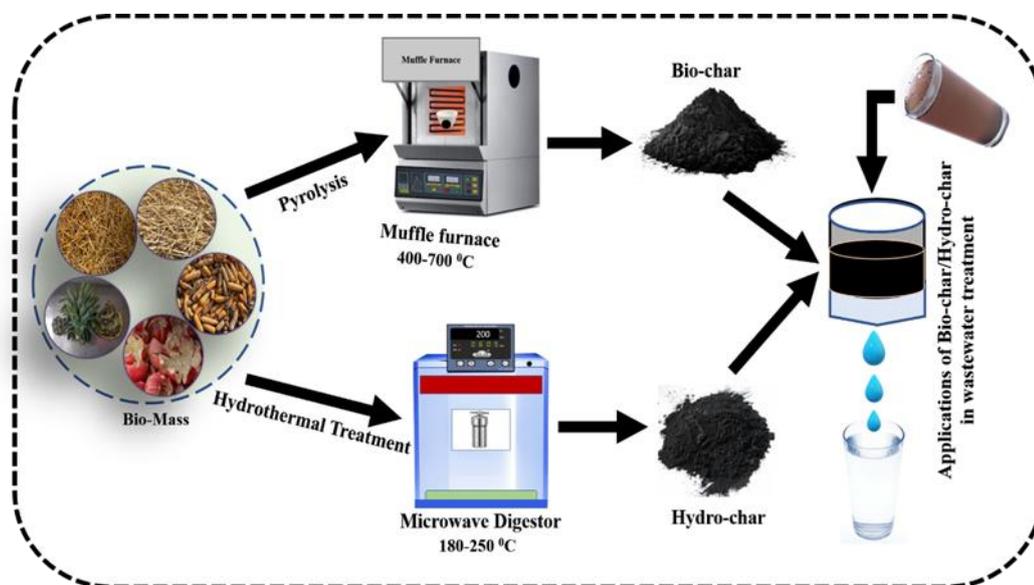

Figure1. Methodological Diagram of Biochar and Hydrochar Preparation

Preparation of Simulated Wastewater

To prepare the simulated wastewater, a 30 mg/L lead solution was formulated by diluting a 1000 mg/L stock solution of lead with distilled water. The pH of the diluted solution was regulated to 6.0 using 0.1 M NaOH (Merck). The resulting solution was then transferred into a conical flask for further use.

Batch Adsorption Analysis

250 ml conical flasks containing 100 mL of a 30 mg/L lead solution and 1 g of dry NaOH-treated adsorbents were used for batch adsorption tests. The slurry mixes were stirred for an hour at room temperature in a rotary shaker at 200 rpm. After agitation, the supernatants were separated by filtration using filter paper. The concentration of heavy metals in the supernatant was determined using inductively coupled plasma (ICP) analysis (Perkin Elmer, Optima 8000). All experiments were performed in triplicate to ensure the accuracy and reliability of the results. Additionally, batch experiments were also conducted using NaOH-untreated adsorbents following the same procedure as for the NaOH-treated ones. The adsorption efficiencies of the metals were calculated to compare the performance of the NaOH-treated adsorbents with the untreated ones.

Metal ion Removal Efficiency (%) = (C_i - C_f / C_i)*100; where C_i & C_f are the initial and final Pb^{2+} concentrations.

Experimental Setup for Adsorption Study

The preparation of the experimental solutions involved adding a 30 mg/L lead solution (100 mL) to a series of 200 mL volumetric flasks containing 1 g of adsorbents. To optimize the pH conditions, the initial pH values of the solutions were adjusted to 2, 4, or 6 using either 0.1 M HNO3 or 0.1 M NaOH. Subsequently, to investigate the effect of contact time, the slurry mixtures were agitated at 200 rpm for durations of 15, 30, 60, or 90 minutes. Temperature variations were introduced by conducting experiments at 30°C and 60°C to evaluate their influence on lead adsorption. Following agitation, the supernatants were separated from the solid phase through filtration using filter paper. The concentration of heavy metals in the supernatant was quantified using inductively coupled plasma (ICP) analysis performed with a Perkin Elmer Optima 8000 instrument. This experimental setup allowed for the comprehensive examination of various factors influencing lead adsorption, including pH, contact time, and temperature, ensuring a thorough understanding of the adsorption process.

RESULT AND DISCUSSION

FTIR analysis was conducted to characterize the surface functional groups and chemical bonds present on the pineapple waste material. Each chemical bond exhibits oscillations at specific frequencies, which are quantified by the wavenumber. A higher frequency of oscillation corresponds to a higher wavenumber, indicating stronger bonds. Conversely, weaker bonds exhibit lower wavenumbers. In the FTIR spectrum, dips represent chemical bonds absorbing energy, with higher transmittance indicating less energy absorption and more transmission. As the wavenumber increases, the frequency and energy of the oscillating chemical bond increase. Bonds located to the left of the spectrum are stronger, while those to the right are weaker. The broad band observed around 3,200–3,600 cm^{-1} is attributed to the symmetrical stretching variations of free or hydrogen bond O–H and/or NH (amino) groups. Additionally, the peak at 2,918 cm^{-1} is assigned to aliphatic C-H groups, while a new sharp band at 2,852 cm^{-1} appears after NaOH treatment, indicating C–H stretching. The strong peak at 1,593 cm^{-1} corresponds to the C–O bond of the carboxylic group, with a new peak at 1,732 cm^{-1} appearing after NaOH treatment, attributed to the carbonyl group of ester, C=O.
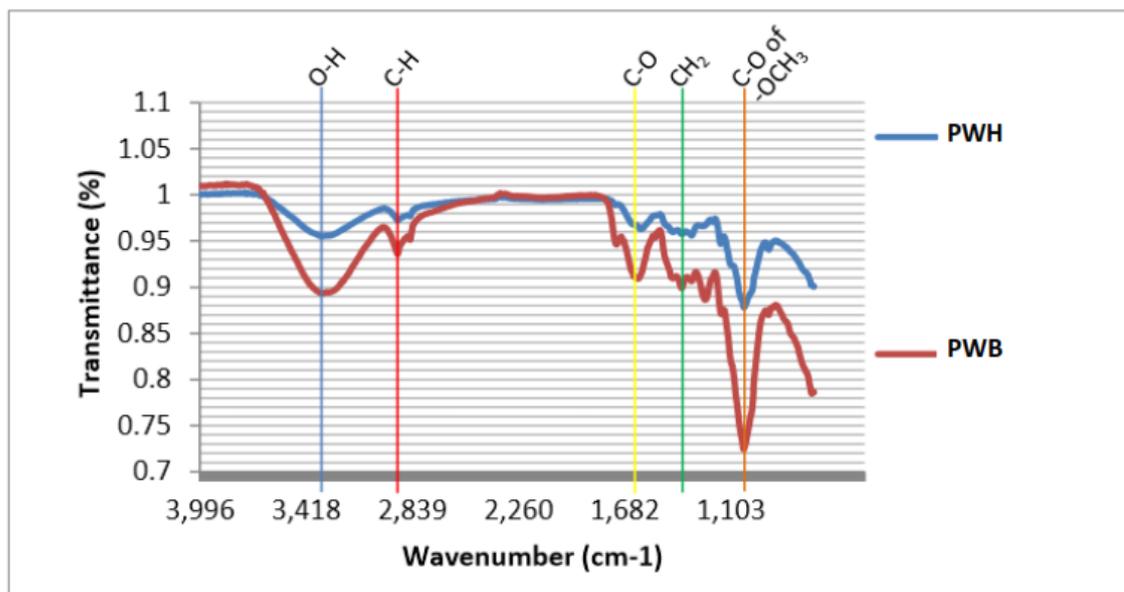
Figure 2. Percentage Transmittance of PWH and PWB in FTIR Analysis

Moreover, vibrations resulting from O-H bending, O-H stretching, or C-H bending may be responsible for peaks in the 1,369–1,245 cm-1 area. While the strong band at 1,103 cm-1 is caused by the C-O of the -OCH group belonging to the lignin structure in pineapple fruit peel, the absorption band at 1,161 cm-1 corresponds to C–O antisymmetric bridge stretching of the cellulose moiety of pineapple waste. These changes in peak appearance and absorption band shifts in the FTIR spectrum indicate successful NaOH treatment of the pineapple waste material.

Comparison of Lead Adsorption efficiency between PWH and PWB

Based on the findings presented in Table 1, the adsorption of Pb ions onto PWH exhibited a notable increase compared to PWB samples. This enhancement can be attributed to the removal of surface impurities in PWH, which consequently exposes more active sites for metal adsorption (Ahmad et al., 2021; Gumpu et al., 2015). Moreover, the deprotonation of waste materials likely occurred during treatment, facilitating the displacement of Pb ions by Microwave treatment. This exchange mechanism is facilitated by the greater electropositivity compared to Pb^{2+} ions in the simulated wastewater (Qi et al., 2023)

Table 1. Comparison of Removal Efficiency between PWH and PWB at Temperature 60°C and pH 4

SAMPLE BATCH	REMOVAL EFFICIENCY (%)	
	PWH	PWB

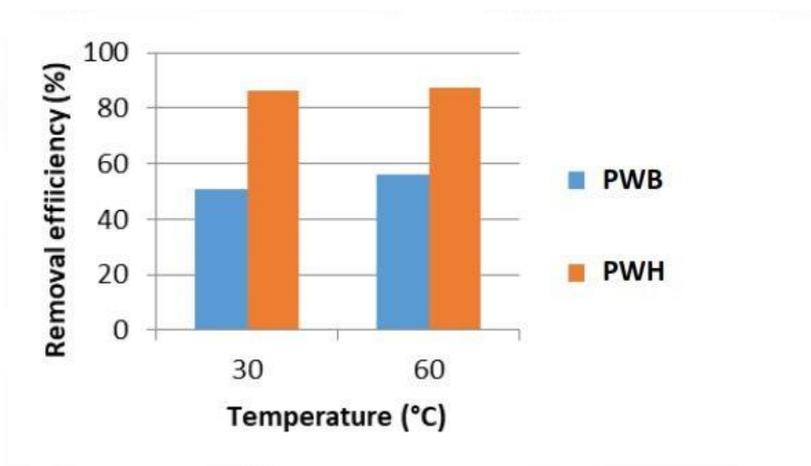|		
1.	83.15	56.24
2.	84.22	55.86
3.	84.85	54.96
Average	**84.07**	**55.68**

Figure 3. Comparison of Removal Efficiency between PWH and PWB at Temperature 60°C and pH 4

Additionally, ion exchange processes involving carboxylate and hydroxylate anions as acidic groups may contribute to the observed increase in metal adsorption capacity (Xu et al., 2014; Zhou et al., 2017). The -OH groups introduced by PWH serve as functional groups responsible for metal ion adsorption (Gómez-Navarro et al., 2023; Xu et al., 2014), thereby improving the metal binding capability and resulting in higher adsorption of metal ions onto PWH compared to PWB counterparts.

Furthermore, the temperature dependency of Pb^{2+} removal efficiency was investigated, revealing a temperature-induced increase in adsorption efficiency. At higher temperatures (60°C), the adsorption efficiency increased significantly, consistent with the endothermic nature of the Pb^{2+} adsorption process (Leng et al., 2015). These results underscore the superior performance of PWH in adsorbing lead from wastewater compared to PWB, highlighting the efficacy of PWH treatment in enhancing adsorption efficiency.

Impact of Contact Time and pH on Pb Adsorption by Pineapple Waste Hydrochar (PWH)

The effect of contact time on the adsorption of Pb using pineapple waste hydrochar (PWH) at 30°C and 60°C is shown in Table 2 and Figure 3. Notably, adsorption efficiencies exceeding 80% were observed, indicating a relatively rapid adsorption process. The experimental results reveal that the adsorption

efficiency of Pb^{2+} ions was higher at 60°C compared to 30°C when utilizing PWH. Analysis of the graph indicates that equilibrium adsorption was achieved in approximately 30 minutes at 60°C, whereas it took about 60 minutes at 30°C. This phenomenon can be attributed to the spongy structure of the waste material at elevated temperatures, facilitating the diffusion of metal ions from the solution onto the adsorbent surface (Enaime et al., 2020; Guo et al., 2019; Li et al., 2021). Furthermore, at lower pH levels, competition between H_3O^+ ions and metal ions occurs for exchange sites in the adsorbents. Conversely, at higher pH levels, the abundance of metal ions relative to H_3O^+ ions results in less competition and more available exchange sites for metal ions (Zhang et al., 2020). The adsorption efficiency of Pb^{2+} using PWH is shown to be more rapid at pH 4 than at pH 2, as the former facilitates deprotonation and the formation of negatively charged surfaces, enhancing electrostatic interactions with metal ions (Li et al., 2021; Petrović et al., 2023; Tran et al., 2017). However, at pH levels higher than 6, a slight decrease in Pb adsorption is observed due to the formation of soluble hydroxyl complexes, consistent with previous findings. This underscores the importance of pH regulation in optimizing the efficiency of heavy metal adsorption processes.
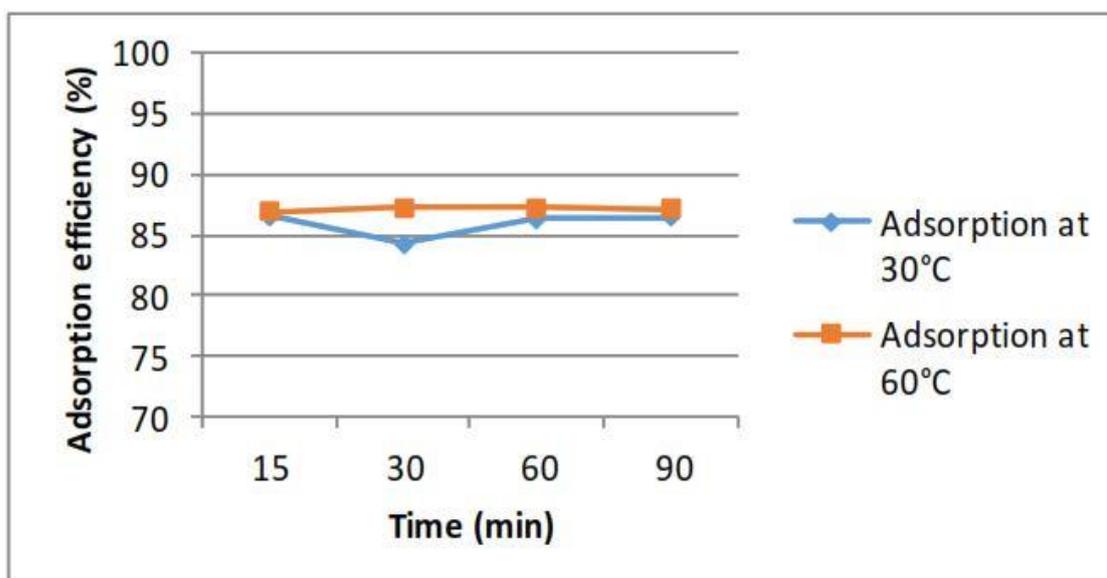

Figure 4. Pb Removal Efficiency of PWH at Temperatures 30°C and 60°C, with pH 4

According to the data presented in Table 2, the impact of temperature on Pb^{2+} adsorption efficiency using PWH is evident. As the temperature rises from 30°C to 60°C, there is a corresponding increase in the percentage of adsorption efficiency. This phenomenon aligns with the understanding that the adsorption process for Pb^{2+} is inherently endothermic (Shi et al., 2022). Higher temperatures are necessary to optimize the adsorbent's performance, as it enhances the availability of active sites or reduces the thickness of the boundary layer surrounding the adsorbents (Ighalo et al., 2022). The data in Table 2 further illustrates that with increasing temperature, the removal efficiencies of

Pb^{2+} also exhibit an upward trend. These findings suggest that the adsorption capacity of pineapple waste improves significantly at elevated temperatures.

CONCLUSIONS AND RECOMMENDATIONS

The findings of this study demonstrate that Microwave treatment significantly enhances the adsorption efficiency of Pb^{2+} ions in pineapple waste compared to PWB. This highlights the ability of Microwave treatment to convert pineapple waste into a highly efficient Pb^{2+} ion adsorbent. On average, a notable increase in adsorption efficiency, averaging 85.88%, was observed following three treatment cycles. The experimental results reveal that adsorption equilibrium for Pb^{2+} ions on PWH is reached as early as 30 minutes. Moreover, the adsorption efficiency steadily increases from 30 to 90 minutes of contact time. Temperature variations also influence adsorption efficiency, with Pb^{2+} adsorption at 60°C outperforming that at 30°C.

Additionally, the pH of the solution impacts the adsorption process. PWH at pH 2 requires 30, 60, and 90 minutes to reach equilibrium for Pb^{2+} adsorption. Conversely, using waste with pH 4 enhances adsorption efficiency, while a pH of 6 results in a slight decrease in efficiency, suggesting that pH 2 and pH 4-treated waste are more effective in adsorbing heavy metals from wastewater.

Overall, pineapple waste emerges as a promising adsorbent due to its structural properties, making it suitable for widespread use. Its abundance and affordability further support its potential for large-scale application in heavy metal adsorption from wastewater. Moreover, the successful conversion of pineapple waste to Pb^{2+} ion adsorbents through Microwave treatment underscores its high potential for practical implementation.

FURTHER STUDY

Hydrochar offers a promising solution to address challenges in water treatment, including regeneration, environmental impact, commercial viability, and disposal management. To ensure widespread industrial acceptance and utilization of HC, research efforts should prioritize its reusability. Embracing HC can lead to a significant reduction in solid waste and sludge discharge, thereby mitigating land pollution and promoting the principles of the circular economy.

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