Synthesis of Hydroxyapatite from Pig Bone Waste as Adsorbent for Heavy Metal Lead

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Abstract. The increase in industrial activities that are not accompanied by adequate waste handling has led to a decrease in the quality of water used by the community. Therefore, a solution is needed that prioritizes the concept of a green economy, where waste can be processed into useful materials. One waste-based material that has the potential to be used in improving water quality is hydroxyapatite. Hydroxyapatite is a compound with the chemical formula $Ca_{10}(PO_4)_6(OH)_2$. This compound is the main mineral component of bones and teeth in animals. Hydroxyapatite is known to have the ability to bind various types of heavy metals such as Lead (II). Hydroxyapatite was produced by calcining pig bone waste at 400, 600, and 800C for 1 hour. The size of hydroxyapatite powder was also varied to get the best size for the heavy metal adsorption process. Hydroxyapatite characterization was carried out using X-ray Diffraction (XRD) and a digital microscope. This study was conducted to determine the effect of calcination temperature and powder size on the adsorbing power of hydroxyapatite on solutions containing Lead (II) metals. The results of the study show that hydroxyapatite synthesized from pig bone waste is an effective adsorbent for removing Pb(II) from aqueous solutions. The adsorption efficiency is significantly influenced by the calcination temperature and the particle size of the hydroxyapatite, with higher temperatures and finer particles providing superior performance. These findings highlight the potential of hydroxyapatite as a sustainable solution for heavy metal treatment in wastewater management.

Keywords: Adsorbent, Calcination, Hydroxyapatite, Lead

1 Introduction

The demand for water usage is increasing in line with the rise in domestic and industrial activities. However, on the other hand, poorly managed waste handling leads to a significant increase in the amount of waste being disposed of. This problem can affect water quality and cause a decline in water quality in the community. Water quality is influenced by many factors such as rainfall, climate, soil type, vegetation, geology, water conditions, groundwater, and human activities. The greatest threat to water

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quality comes from industrial sources and urban activities (Chaudhry & Malik, 2017). Water treatment is an important process in reducing the pollutant content in water so that it can be utilized according to needs. The role of water treatment technology is vital in removing pollutants and microbes to obtain clean water. The use of treatment methods with adsorbents is considered an economical option because they are affordable, easily available in abundance, and can be obtained from various industrial, agricultural, and food processing sectors (Lim & Aris, 2014).

Adsorbents are solid materials, whether mineral, organic, or biological, used in the process of removing contaminants from solutions. There are two types of adsorbents, conventional and non-conventional. Non-conventional adsorbents attract the interest of researchers due to their capability as economical and effective adsorbent materials, such as bone charcoal and hydroxyapatite (Crini et al., 2019). Hydroxyapatite is a compound with the chemical formula $Ca_{10}(PO_4)_6(OH)_2$. This compound is the main mineral component of bones and teeth in animals (Ibrahim et al., 2020). Some advantages of hydroxyapatite as an adsorbent include specificity, fast kinetics, high binding capacity, ability to work in solutions with multiple metals, and its biocompatible nature. However, hydroxyapatite also has drawbacks, such as poor mechanical properties, a tendency to agglomerate, and a lack of research on recycling and regeneration (Nayak & Bhushan, 2021).

In this study, hydroxyapatite was synthesized from natural waste materials commonly produced by the food industry in the Bali region. The waste used is pig bone waste from various pig slaughterhouses. Several studies have successfully extracted hydroxyapatite from pig bones (Janus et al., 2008 ; Ofudje et al., 2018) Utilizing waste embodies the concept of a green economy, where waste can be processed into highly useful materials. The most influential parameter in synthesizing bones into hydroxyapatite is the temperature during the calcination process. The resulting hydroxyapatite was then tested for its adsorption capacity for heavy metals commonly found in industrial wastewater, such as Lead (II). Heavy metals are defined as elements with a density greater than 5 g/cm^3 and an atomic weight between 63.5-200.6 g/mol. Various types of heavy metals are found in the environment, including arsenic, copper, cadmium, selenium, mercury, chromium, nickel, iron, and lead (Omar et al., 2019).

Adsorption capacity is closely related to the surface area of the adsorbent particles, making the powder size of hydroxyapatite crucial. Therefore, this study was conducted to determine the effect of calcination temperature and powder size on the adsorption capacity of hydroxyapatite synthesized from pig bone waste. This research is expected to make an important contribution to the development of more cost-effective and environmentally friendly wastewater treatment methods.

2 Methodology

The research flow in this study is divided into three main stages: hydroxyapatite synthesis, metal adsorption process in solution, and analysis of test results. Material preparation begins with the selection and cutting of pig bones into an average length of 2 cm. The pig bones are then cleaned of any remaining fat and meat using a brush.

Further cleaning is done by soaking the bones in NaOH solution. The bones are then rinsed with distilled water to ensure no residual NaOH remains. The main process of hydroxyapatite synthesis is calcination at temperatures of 400, 600, and 800°C. The characteristics of hydroxyapatite obtained depend on the extraction method used, with the thermal method (calcination) producing highly crystalline hydroxyapatite compared to chemical precipitation (Pu'ad et al., 2019). The calcination results are then crushed using a mortar and sieved with a stainless steel sieve to obtain powders of 140 and 230 mesh sizes. The adsorption power testing process is carried out on previously prepared Lead (II) solutions. Adsorption power is analyzed through the reduction of heavy metal concentrations in the solution, obtained from AAS testing. These results, along with the characterization performed on hydroxyapatite, are then analyzed to derive research conclusions.

3 Result and Discussion

3.1 Result

The high-temperature treatment results in a significant mass reduction in the bone material. The mass of hydroxyapatite is compared to the mass of the bones before calcination. Mass reduction at 400°C was observed to be 24.3%, at 600°C 29.2%, and at 800°C 31.5%. This reduction is due to the decomposition of organic components and the removal of moisture in the bones. At 400°C, the initial decomposition of organics and evaporation of water vapor occurs, causing a 24.3% mass reduction. As the temperature increases to 600°C, more organics decompose, and volatile compounds are removed, resulting in a higher mass reduction of 29.2%. At 800°C, this process continues more extensively and completely, reaching the highest mass reduction of 31.5%, indicating that most organics and residual volatiles have been removed, leaving behind inorganic hydroxyapatite.

Characterization using XRD was performed on pig bone powder obtained from calcination at a temperature of 800°C to confirm that the calcination process produced hydroxyapatite compounds as shown in Figure 1. Analysis using standard data showed a match in peak positions on the diffraction pattern with the HA compound. According to the JCPDS standard data number 9-432, stoichiometric HA has the highest peaks at 2θ angles of 25.879°, 31.774°, 32.179°, 32.902°, and 49.469°. The sharp and narrow peaks in the diffraction pattern of the bone indicate a crystalline atomic arrangement with minimal contamination from the organic matrix (Widantha et al., 2021).

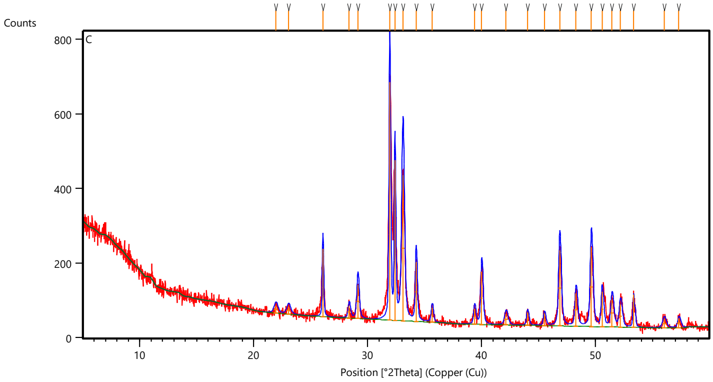

Figure 1. XRD pattern of pig bone after calcination

After calcination, the bones are ground into a fine powder using a mortar and pestle. The grinding process ensures uniform particle size, which is crucial for consistent adsorption properties. The resulting powder is then sieved through a stainless steel sieve to obtain two sizes: 140 mesh and 230 mesh. The sieving process is essential to classify the hydroxyapatite powder into uniform particle sizes, enhancing the material's effectiveness as an adsorbent. The final product, a white powder, is characterized by its homogeneous appearance, indicating the purity and success of the synthesis, as shown in Figure 1. This synthesized hydroxyapatite powder is then ready for adsorption experiments to test its effectiveness in removing heavy metals such as Lead (II).

Figure 2. Hydroxyapatite in white powder form

To assess the adsorption capability of hydroxyapatite synthesized from pig bone waste, solutions containing Pb(II) are separately prepared. The Pb(II) solution is made by dissolving $Pb(NO_3)_2$ in distilled water. Each solution is carefully prepared in 100 mL Erlenmeyer flasks. Stoichiometric calculations are meticulously done to achieve target solutions with a concentration of 20 ppm.

The adsorption test involves six different variations based on calcination temperature (400°C, 600°C, and 800°C) and hydroxyapatite particle size (140 mesh and 230 mesh). These variations are crucial for understanding how different synthesis conditions affect the adsorption efficiency of hydroxyapatite. Each Erlenmeyer flask is added with 1 gram of hydroxyapatite and stirred for 1 hour to ensure thorough mixing and interaction between the hydroxyapatite and metal ions. This shaking period provides sufficient time for effective adsorption to occur.

Figure 3. Lead solution used in adsorption tests (left) and filtration process: After shaking, the mixture is filtered using filter paper to separate solid hydroxyapatite from the solution (right)

After the shaking period, the mixture is filtered using filter paper to separate the hydroxyapatite particles from the solution. This filtration step is essential to separate the hydroxyapatite from the solution for subsequent analysis.

Figure 4. Solution comparison: Before filtration and after filtration with previously added hydroxyapatite adsorbent in vials

The filtrate, now free from hydroxyapatite particles, is collected and transferred into vials for further analysis. To ensure accuracy, the concentration of Pb(II) ions in the filtrate is determined using Atomic Absorption Spectroscopy (AAS). This analytical technique provides precise measurements, allowing the calculation of the adsorption efficiency of hydroxyapatite for Pb(II) ions. The processed data results are shown in Table 1.

Table 1. Hydroxyapatite adsorption test results for lead (II) solutions

No	Calcination temperature (˚C)	Powder size (Mesh)	Reduce in concentration of lead (II) (%)
1	400	230	96.87
2	600	230	99.31
3	800	230	98.13
4	400	140	81.01
5	600	140	92.56
6	800	140	98.54

3.2 Discussion

The data show that calcination temperature significantly affects the adsorption efficiency of hydroxyapatite. For the removal of Pb(II), the highest concentration reduction was observed with hydroxyapatite calcined at 600°C and sieved to 230 mesh (99.31%), followed by hydroxyapatite calcined at 800°C and sieved to 230 mesh (98.13%). These results indicate that hydroxyapatite calcined at moderate to high temperatures (600°C and 800°C) exhibits better adsorption properties compared to hydroxyapatite calcined at lower temperatures (400°C). Higher calcination temperatures likely enhance the crystallinity and purity of hydroxyapatite, thereby increasing adsorption sites and overall effectiveness in removing heavy metal ions.

The particle size of hydroxyapatite also plays a crucial role in its adsorption performance. Generally, hydroxyapatite with a finer particle size (230 mesh) shows better adsorption efficiency compared to hydroxyapatite with a coarser particle size (140 mesh). For instance, hydroxyapatite calcined at 400°C and sieved to 230 mesh reduced Pb(II) concentration by 96.87%, while the same hydroxyapatite with 140 mesh particle size achieved a reduction of only 81.01%. The higher surface area-to-volume ratio of finer particles likely explains the superior adsorption capacity. Smaller particles provide more surface area to interact with heavy metal ions, thereby enhancing adsorption.

4 Conclusion

The research findings indicate that hydroxyapatite synthesized from pig bone waste is an effective adsorbent for removing Pb(II) from aqueous solutions. The adsorption efficiency is significantly influenced by the calcination temperature and the particle size of the hydroxyapatite, with higher temperatures and finer particles providing superior performance. These findings add to the understanding of hydroxyapatite's potential as a sustainable solution in the field of heavy metal remediation in wastewater treatment.

References

Crini, G., Lichtfouse, E., Wilson, L. D., & Morin-Crini, N. (2019). Conventional and non-conventional adsorbents for wastewater treatment. *Environmental Chemistry Letters* 171, pp. 195–213. Springer Verlag. https://doi.org/10.1007/s10311-018-0786-8

FN, C., & MF, M. (2017). Factors affecting water pollution: A Review. *Journal of Ecosystem & Ecography*, *07*(01). https://doi.org/10.4172/2157-7625.1000225

Ibrahim, M., Labaki, M., Giraudon, J. M., & Lamonier, J. F. (2020). Hydroxyapatite, a multifunctional material for air, water and soil pollution control: *Journal of Hazardous Materials,* 383, Elsevier B.V. https://doi.org/10.1016/j.jhazmat.2019.121139

Janus, A. M., Faryna, M., Haberko, K., Rakowska, A., & Panz, T. (2008). Chemical and microstructural characterization of natural hydroxyapatite derived from pig bones. *Microchimica Acta*, *161*(3–4), 349–353. https://doi.org/10.1007/s00604-007-0864-2

Lim, A. P., & Aris, A. Z. (2014). A review on economically adsorbents on heavy metals removal in water and wastewater. *Reviews in Environmental Science and Biotechnology*, *13*(2), pp. 163–181). Kluwer Academic Publishers. https://doi.org/10.1007/s11157-013-9330-2

Pu'ad, N. A. S. M., Koshy, P., Abdullah, H. Z., Idris, M. I., & Lee, T. C. (2019). Syntheses of hydroxyapatite from natural sources. *Heliyon,* *5*(5). Elsevier Ltd. https://doi.org/10.1016/j.heliyon.2019.e01588

Nayak, A., & Bhushan, B. (2021). Hydroxyapatite as an advanced adsorbent for removal of heavy metal ions from water: Focus on its applications and limitations. *Materials Today: Proceedings*, *46*, 11029–11034. https://doi.org/10.1016/j.matpr.2021.02.149

Ofudje, E. A., Rajendran, A., Adeogun, A. I., Idowu, M. A., Kareem, S. O., & Pattanayak, D. K. (2018). Synthesis of organic derived hydroxyapatite scaffold from pig bone waste for tissue engineering applications. *Advanced Powder Technology*, *29*(1), 1–8. https://doi.org/10.1016/j.apt.2017.09.008

Omar, S., Muhamad, M. S., Te Chuan, L., Hadibarata, T., & Teh, Z. C. (2019). A Review on lead sources, occurrences, health effects, and treatment using hydroxyapatite (HAp) adsorbent made from fish waste. *Water, Air, and Soil Pollution*, *230*(12). Springer. https://doi.org/10.1007/s11270-019-4312-9

Widantha, K. W., Basuki, E. A., Martides, E., & Prawara, B. (2021). Effect of hydroxyapatite/alumina composite coatings using HVOF on immersion behavior of NiTi alloys. *Journal of Biomaterials Applications*, *36*(3), 375–384. https://doi.org/10.1177/08853282211022531