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Application of Hydroxyapatite (HAp) to Remove Copper (II) from Water

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Abstract: The contamination of water sources by heavy metals such as copper (Cu^{2+}) poses a serious threat to human health and environment diversity. In this study, hydroxyapatite (HAp), a calcium phosphate-based material, was investigated for its adsorption capacity in the removal of Cu^{2+} ions from aqueous solutions. So, in this study HAp was synthesized via chemical precipitation and characterized using SEM techniques, that acts as an absorbent. This study covers, the effects of initial copper concentration, contact time, adsorbent dosage, and solution pH when exposed to HAp. The results demonstrated that HAp exhibited high affinity for Cu^{2+} ions, with maximum removal efficiency observed at pH 5.5 and contact time of 350 minutes. Based on the results of the adsorption experiments, a treatment unit was designed and developed for the removal of copper from water. Prototype units were constructed to test copper ion concentrations of 4 mg/L and 6 mg/L. These prototypes demonstrated effective performance, successfully treating over 1000 liters of copper-contaminated water.

Keywords: Hydroxyapatite (HAp); Copper Concentration; Absorbent; Water filter; Purification;

I. INTRODUCTION

Heavy metal pollution, particularly copper contamination in water bodies, is a major environmental concern and have huge impact of the health of human due to its toxicity, persistence, and bioaccumulation. Copper, though essential in trace amounts, can cause severe health issues such as liver and kidney damage at elevated concentrations that compelled the scientist to restrict its value within tolerable limit [1]. Traditional methods for heavy metal removal, such as chemical precipitation, ion exchange, and membrane filtration, are often expensive and generate secondary waste [2-3]. In addition, adsorption is always related to thermodynamic studies that suggest a correct process to remove the efficient ion content from water. Hence the thermodynamic parameters like free energy change (ΔG), enthalpy change (ΔH) and entropy change (ΔS) are significantly affect the absorption of the absorbent and selection of absorbent dependents on the said parameters. The enthalpy change (ΔH) if positive then the adsorption process will be endothermic in nature and vice versa. Likewise, entropy change is a measure for the randomness of the process [4]. The randomness decreasing and increasing can be inferred by the negative and positive value of ΔS respectively. There are wide number of researches available to choose adosrbents for the removal metal ions from water. In this regard, O.S. Amuda et al. [5] developed a composite adsorbent by modifying coconut shell carbon with chitosan and/or an oxidizing agent (phosphoric acid).

Therefore, the presence of heavy metals such as copper (Cu^{2+}) in water sources is a critical environmental concern. Although copper is an essential micronutrient, excessive intake can lead to toxic effects including liver and kidney damage, gastrointestinal distress, and even neurological disorders. Copper contamination in water arises primarily from industrial activities such as mining, electroplating, metal finishing, and fertilizer production. Over the years, numerous methods have been developed for the removal of copper from water, including precipitation, membrane filtration, ion exchange, and adsorption. Among these, adsorption is increasingly favored due to its cost-effectiveness, operational simplicity, and ability to achieve high removal efficiencies. Hence, Hydroxyapatite (HAp), with the chemical formula $Ca_{10}(PO_4)_6(OH)_2$, has gained widespread attention since the late 20th century for its use in environmental remediation, particularly in water purification showed by several researchers [6]. Furthermore, the initially investigation on HAp known for its biological application such as bone grafts and dental implants due to its similarity to human bone mineral. The application also gained HAp's potential in wastewater treatment was gradually realized. Researchers observed that the ion-exchange properties of HAp made it suitable for binding heavy metal ions, including Cu^{2+}, through various mechanisms such as surface complexation, ion exchange, and precipitation.

In the first few studies in the 1980s and early 1990s explored the use of synthetic HAp for the adsorption of toxic metal ions illustrated by Jaffar et al. and his college [7]. These initial investigations established the basic premise that HAp could remove metal ions from aqueous media by replacing calcium ions in its crystal lattice with heavy metal ions. This laid the groundwork for subsequent studies focusing on optimizing its properties for better performance in water treatment.

During the early 2000s, more systematic studies began to emerge, focusing on the removal of specific heavy metals, including copper, using HAp [8]. Researchers experimented with different synthesis techniques that exception showed value added result such as wet chemical precipitation, sol-gel methods, and hydrothermal synthesis due to tailoring the surface area and porosity of HAp, both of which are crucial for adsorption efficiency [9]. However, in the case of Pb^{2+} adsorption, the dominant mechanism is dissolution followed by precipitation, rather than simple ion exchange. In parallel, investigations into the factors affecting the adsorption of copper ions that crucially have impact are pH, contact time, initial metal concentration, and adsorbent dosage. It was observed that HAp had a higher affinity for Cu^{2+} at slightly acidic to neutral pH (around 5.5–6.5), where copper ions remain soluble and were not precipitated as hydroxides showed by Šljivić, et al. [10]. The studies also demonstrated that HAp could achieve copper removal efficiencies of over 90% under optimized conditions, making it a highly promising candidate for practical applications. A major turning point in the development of HAp as an adsorbent came with the realization that it could be synthesized from biological waste materials such as fish bones, animal bones, egg shells, and teeth. Materials contain naturally occurring calcium phosphate and could be easily processed to produce HAp through calcination or chemical treatment. Several studies from 2010 onward showed that fish-derived HAp exhibited comparable or even superior adsorption performance for Cu^{2+} ions due to the presence of naturally incorporated trace elements like magnesium and sodium, which enhanced ion-exchange activity. This approach aligned with the global movement towards "waste-to-wealth" technologies, providing a dual benefit of heavy metal remediation and solid waste minimization.

Hence, despite the wide availability of various adsorbents, there remains a need for the development of new materials that are cost-effective, easy to synthesize, and efficient in removing copper from water. In this study, hydroxyapatite (HAp) has been employed as an adsorbent for the removal of copper (II) ions from aqueous solutions. HAp was chosen due to its low-cost synthesis from readily available biowaste materials such as fish scales, eggshells, animal bones, and teeth. This approach not only addresses the issue of heavy metal contamination but also promotes waste minimization, aligning with the "waste to wealth" concept. Therefore, this work aims to evaluate the adsorption capacity of HAp for the effective removal of copper ions from water.

II. METHODS OPTED TO PREPARE ABSORBENT AND EXPERIMENTAL PROCEDURE

A. Hydroxyapatite preparation through chemical process

This investigation starts with preparation of hydroxyapatite (HAp) obeying chemical precipitation method i.e. chemical solution of di-ammonium hydrogen phosphate, calcium nitrate tetrahydrate, and ammonium hydroxide solution. The required quantities of the chemicals were measured and mixed in distilled water within an open beaker to prepare an aqueous solution. This solution was then combined with a 0.1 M ammonium hydroxide solution to initiate the precipitation of HAp. Once the precipitate formed, the mixture was filtered using a filter unit equipped with Whatman filter paper to separate the solid from the liquid. The collected filter cake was then dried in a heated environment for 24 hours. Furthermore, the HAp powder, which contained lumps after dryning or larger granules, was manually crushed using a mortar and pestle. The crushed powder was subsequently subjected to calcination at 1000 °C in a high-temperature furnace to enhance its crystallinity. To achieve a finer particle size, the calcined powder was mechanically milled in an alcoholic medium using a planetary ball mill for 8 hours, with alumina balls serving as the grinding media. The resulting fine powder was once again dried in a heated environment for 24 hours. Finally, the dried powder underwent a second calcination at 1000 °C to ensure the development of a well-crystallized hydroxyapatite structure.

B. Experimental Characterization

This To carry out the experiments, a custom filter unit was developed using a plastic container. The schematic of the filter setup is illustrated in Figure 1. The unit consisted of two PVC containers, each with a capacity of 10 liters. The filtration cartridge was filled with a combination of hydroxyapatite (HAp) powder and activated charcoal, the latter incorporated to enhance porosity and improve water flow. Angular-shaped activated charcoal was selected, and due to its larger particle size compared to the HAp powder, it was expected to facilitate a better flow rate. The HAp powder and activated charcoal were alternately layered within the candle housing of the filter unit. To further improve the flow rate, it was necessary to increase the pressure within the system. As the setup operated on gravity-driven flow, this was achieved by elevating the height of the upper container. For testing purposes, copper (II)-contaminated water samples were prepared at varying concentrations of 3, 5, 6, 7, and 8 mg/L to evaluate the performance of the filter unit. The morphological study of developed powder was analysed through SEM images. From SEM images the porous morphology with irregularly shaped particles, enhancing surface adsorption potential was analysed. Furthermore, the equilibrium data were analysed using Langmuir isotherm models, which is observed to be most prominent method. Pseudo-first-order and pseudo-second-order kinetic models were applied to determine the adsorption mechanism.

The absorption graph was plotted for every concentration and plotted on same abscess for better comparison. The graph clearly indicates the absorption capacity of HAp with the time.

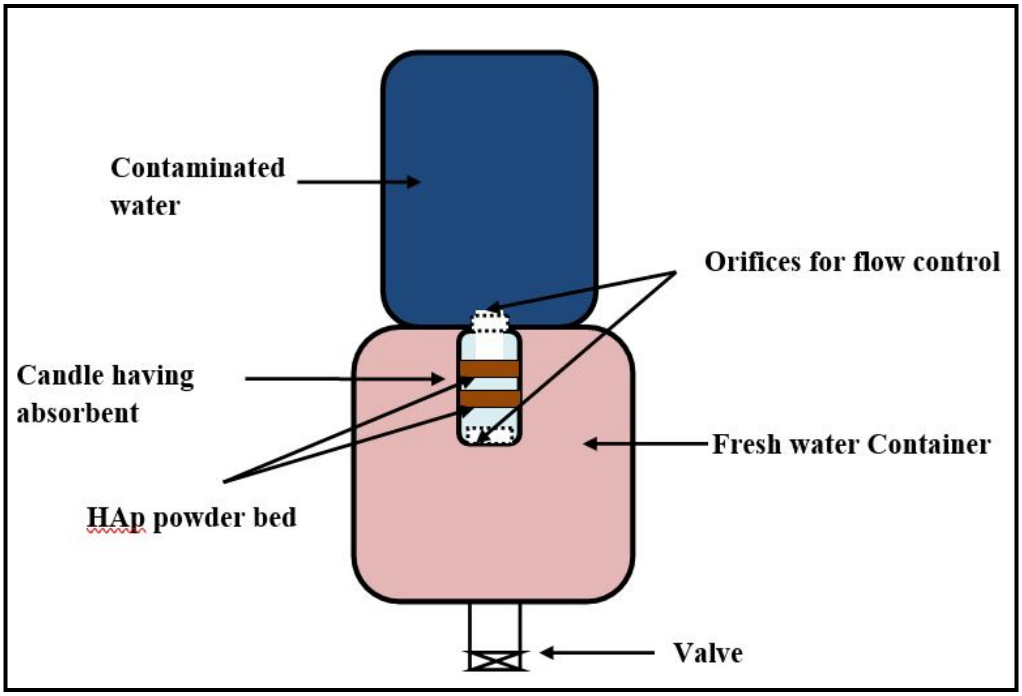

Fig. 1 Line diagram of experimental set up

III.RESULT AND DISCUSSION

The morphological microstructure in terms of SEM images of developed HAp is illustrated through Figure 2. From the image a highly porous structure could directly observed which is encourage its application as an absorbent. Uniform morphology which similar structure of grains are also observed that resembles a good morphology suitable for acting as absorbent.

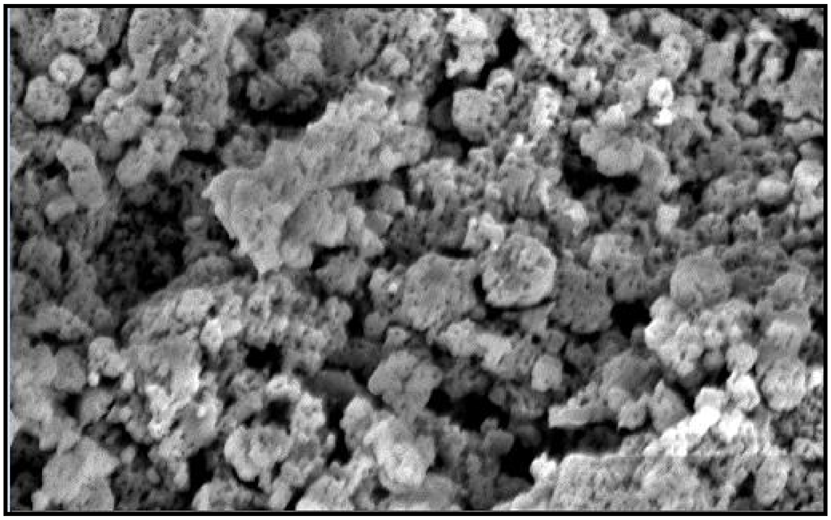

Fig. 2. SEM and EDAX picture of prepared Hap

A. Analysis on HAp as an absorbent

The adsorption study was conducted to evaluate the effect of varying copper(II) ion concentrations on a constant dose of hydroxyapatite (HAp) over different contact times. The tested concentrations included 4 and 6 mg/L. In each experiment, 0.05 mg of HAp was used with 100 mL of copper-contaminated water. The results indicated that as the concentration of copper ions increased, the percentage removal efficiency decreased.

This trend is attributed to the higher number of copper ions being rapidly exposed to the HAp surface, which requires more time for effective interaction and adsorption. Interestingly, at higher copper (II) concentrations, a greater driving force was required to maintain flow through the filter system. The increased mass of copper ions generated resistance within the flow, which in turn created more favorable conditions for copper removal [11-12]. This resistance effectively extended the contact time between the ions and the HAp surface, thereby enhancing the adsorption process. As a result, despite lower percentage removal, the overall purification was more effective at higher concentrations due to improved ion trapping. The adsorption performance across different concentrations is illustrated in Figure 3, and the equilibrium adsorption capacities for each concentration are summarized in Table 1. The results confirm that the system demonstrates efficient copper ion removal, particularly under higher contaminant loads.

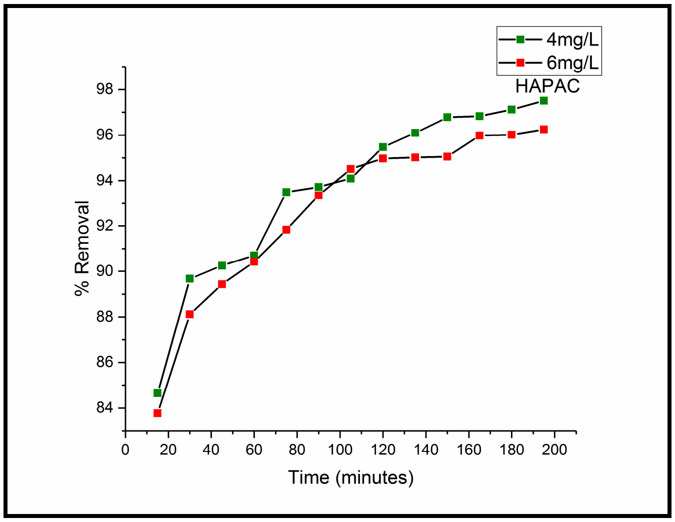

Fig. 3 Effect of exposure time with concentration

TABLE 1. REMOVAL OF COPPER CONTENT FROM WATER

Copper Content (mg/L)	Removal of Copper in (%)	Adsorption (mg/g)
3	95.37	6.77
5	95.02	9.42
6	94.17	11.16
7	93.65	12.97
8	93.54	14.82

IV. CONCLUSION

This study confirms that hydroxyapatite (HAp) serves as an effective and environmentally friendly adsorbent for the removal of copper (II) ions from aqueous solutions. The adsorption efficiency was notably influenced by factors such as pH, contact time, and the dosage of the adsorbent. The research successfully demonstrated the synthesis of HAp powder using a chemical precipitation method. Additionally, a functional filter unit with a specially designed cartridge was developed for capturing copper (II) ions from polluted or industrial wastewater. Experimental results clearly showed that the presence of HAp significantly enhanced the removal of copper (II) ions from water. The quantity of HAp used played a crucial role in determining the removal efficiency. Therefore, it can be concluded that HAp is a highly effective adsorbent for the treatment of water contaminated with heavy metals.

V. ACKNOWLEDGMENT

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