



Synthesis and Characterization of Submicron Hydroxyapatite from Calcium Nitrate Tetrahydrate: Implications of pH Variations in the Precipitation Process

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Abstract

Hydroxyapatite (HAp) is the primary mineral component of bone and tooth tissue. Submicron-sized HAp offers enhanced osteoconductive properties, enabling it to serve effectively as a scaffold to support osteoblast activity and new bone formation. In this study, HAp was synthesized from calcium nitrate tetrahydrate using the precipitation method. The main variable investigated was the pH, adjusted by the addition of ammonium hydroxide, which influenced the yield and Ca/P ratio of the resulting HAp. The synthesized HAp was characterized using Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), Atomic Absorption Spectroscopy (AAS Flame), UV-Vis spectrophotometry, and X-Ray Diffraction (XRD). The optimal HAp product was obtained at pH 11 and sintered at 800 °C, yielding a Ca content of 2.56% and a P content of 4.28%, resulting in a Ca/P ratio of 1.6719; consistent with the theoretical stoichiometric value of 1.67. This was supported by XRD analysis, which showed the highest intensity peak at $2\theta = 31.74^\circ$, confirming the presence of hydroxyapatite. SEM analysis revealed a particle size of 460.8 nm and a porosity of 20.15%. These findings demonstrate that hydroxyapatite can be successfully synthesized via precipitation using calcium nitrate tetrahydrate under optimal conditions of pH > 11.

1. Introduction

The incidence of injuries that damage human bones and teeth remains high both globally and in Indonesia. This has prompted doctors and researchers to explore methods for repairing damaged bones and teeth without affecting other organ systems [1]. Among various musculoskeletal disorders, fractures are a common and serious condition that can lead to long-term disability. A fracture is defined as a break in the continuity of bone structure, which can manifest as cracks or complete separations resulting in displaced bone fragments. According to the Indonesian Ministry of Health, there were approximately eight million reported fracture cases in 2013, and this number is projected to rise to 11 million by 2025 [2]. One promising approach to addressing this issue is the development of biomaterials designed to

support the regeneration of bone and tooth tissue. These biomaterials must closely mimic the composition and structure of natural bone [3]. It is well established that bone is primarily composed of hydroxyapatite and collagen fibers. Hydroxyapatite is an inorganic mineral that is chemically and physically similar to the natural mineral content of human bones and teeth [4].

Hydroxyapatite is one of the most stable calcium phosphate materials and plays a vital role in bone formation, comprising approximately 60–65% of bone by weight. It possesses several important chemical properties, most notably biocompatibility, meaning it does not trigger a rejection response from the human immune system, which would otherwise recognize it as a foreign substance. Its bioactive nature enables favorable interactions between implants and body tissues,

including osteoblasts and osteoclasts, which are responsible for bone formation and resorption, respectively. Additionally, hydroxyapatite is biodegradable, meaning that, over time, it dissolves and is gradually replaced by new bone tissue forming around the implant [4].

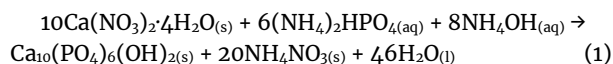
Therefore, the development of this biomaterial continues to be pursued to achieve optimal quality, particularly in terms of biocompatibility and osteoconductivity. With established quality standards, it is hoped that such implant materials can accelerate the bone tissue repair process. Osteoconductivity refers to the material's ability to support the formation of new bone tissue through the process of osteogenesis, a property typically exhibited by materials with submicron-scale structures. Osteoconductive materials can promote cell differentiation into osteoblasts, which are responsible for forming new bone tissue. Osteoclasts and osteoblasts are two types of cells that play important roles in bone remodeling: osteoclasts break down and resorb old or damaged bone, while osteoblasts synthesize and deposit new bone matrix [5].

Hydroxyapatite, commonly abbreviated as HAp, has the chemical formula $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$. The molar ratio of calcium to phosphorus (Ca/P) in hydroxyapatite is 1.67, which closely matches the Ca/P ratio found in human body tissue [6]. Currently, the demand for synthetic HAp compounds in this country is still largely met through imports. This situation is supported by research indicating that domestic production of synthetic HAp implants remains limited. In essence, the synthesis of HAp compounds involves a chemical reaction between sources rich in calcium and phosphorus. Materials such as shells, animal bones, and dromae (a term which may require clarification depending on context) are often used as calcium and phosphate precursors [7].

The process used to produce these implants is known as the synthesis method, a controlled chemical process designed to produce one or more compounds with specific properties. In this context, "artificial" refers to newly created materials with beneficial characteristics for various applications, even if they do not occur naturally. Chemical synthesis involves the transformation of one or more substances under defined reaction conditions to produce the desired compound [8].

The synthesis of HAp compounds can be performed using various types of calcium and phosphate precursors. One of the most commonly used phosphate precursors is diammonium hydrogen phosphate (DHP), due to its stable phosphate group and high reactivity with calcium compounds. Meanwhile, ongoing research continues to explore different calcium precursors to determine which yields the most favorable results. These calcium sources include compounds such as calcium chloride, calcium hydroxide, calcium carbonate, and calcium nitrate tetrahydrate. Among these, calcium nitrate tetrahydrate has been identified as the most effective calcium precursor when combined with diammonium hydrogen phosphate.

Studies have shown that the hydroxyapatite synthesized from this combination exhibits properties closely resembling those of natural HAp found in human body tissues, and is considered biocompatible and safe for medical applications. The synthesis is typically carried out using a precipitation method, in which calcium nitrate tetrahydrate reacts with diammonium hydrogen phosphate in the presence of ammonium hydroxide, which acts as the precipitating agent. The chemical reaction is represented by Equation (1).



Several methods can be used in the synthesis of HAp compounds, one of the most common being the precipitation method, which is widely favored for its simplicity, cost-effectiveness, and practicality. The fundamental principle of this method involves forming the desired product by inducing precipitation through the addition of a precipitating agent. This approach typically yields a high-purity product, with water being the primary byproduct, thereby minimizing the risk of contamination [1, 9]. Following the synthesis, the resulting HAp compound is subjected to sintering, a heat treatment process used to control its microstructure, enhance mechanical strength, and improve the stability of the final material [10].

The porosity of HAp material is a crucial factor to consider when selecting implants. This is because implants used in bones, teeth, and other tissues exhibit varying levels of porosity that must be matched to the natural conditions of the human body to ensure proper integration and acceptance by surrounding tissues. Porosity is defined as the ratio of empty space to the total volume of the material, expressed as a percentage ranging from 0 to 100%. Higher porosity generally enhances cell interaction and tissue growth. However, excessive porosity can accelerate degradation and compromise the mechanical strength of the HAp material [11].

When the reaction occurs at a pH below 11, there is an increase in H^+ ions in the solution, which leads to a higher concentration of HPO_4^{2-} ions. This causes a significant reduction in available calcium ions and inhibits the formation of stoichiometric hydroxyapatite, resulting instead in the formation of calcium-deficient hydroxyapatite (CdHA). Conversely, when the pH is raised to 11 or higher, the concentrations of H^+ and HPO_4^{2-} ions decrease, allowing for the formation of stoichiometric HAp, as calcium loss is minimized [12]. HAp particles with a nanoscale size (~100 nm) can interfere with osteoclast formation and function. In contrast, submicron-sized HAp particles (~500–800 nm) have surface dimensions that more effectively promote osteoblast differentiation and positively influence osteoclast activity [5].

This study aims to investigate how variations in pH affect the Ca/P molar ratio and the morphology of hydroxyapatite compounds produced via the precipitation method, to synthesize submicron-sized particles.

2. Experimental

2.1. Materials

The main raw materials used in this study were calcium nitrate tetrahydrate ($\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$) (Merck, 99%) and diammonium hydrogen phosphate ($(\text{NH}_4)_2\text{HPO}_4$) (UPT BPPTK LIPI, 99%). Other chemicals used included distilled water and ammonium hydroxide (NH_4OH) solution (UPT BPPTK LIPI, 25%).

2.2. Synthesis of Submicron Hydroxyapatite by the Precipitation Method

Hydroxyapatite (HAp) was synthesized using the precipitation method by slowly mixing a 0.167 M calcium nitrate tetrahydrate ($\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$) solution with a 0.1002 M diammonium hydrogen phosphate ($(\text{NH}_4)_2\text{HPO}_4$) solution under continuous stirring. The reaction was stirred for 1 hour while maintaining the temperature at 90°C. Ammonium hydroxide (0.1336 M, NH_4OH) was added to adjust the pH to 8, 9, 10, 11, and 12. The mixture was then aged for 15 hours at room temperature until a solid formed. The resulting precipitate was separated by filtration, and the HAp solid was dried at 110°C in an oven to remove residual solvent. Finally, the dried solid was sintered in a furnace at 800°C for 2 hours.

2.3. Characterization of Submicron Hydroxyapatite

After sintering in the furnace, the HAp powder was characterized using several techniques, including Fourier Transform Infrared Spectroscopy (FTIR; Thermo Scientific Nicolet iS10 ATR), Scanning Electron Microscopy (SEM; FEI Insect-S50), Atomic Absorption Spectrophotometry Flame (AAS Flame; Shimadzu AA-6200), UV-Vis Spectrophotometry (Shimadzu UV-1800), and X-ray Diffraction (XRD; PANalytical X'Pert Pro). The AAS Flame analysis was performed to determine the calcium (Ca) content expressed as atomic percentage (at.%). To calculate the Ca/P molar ratio of the HAp compound, phosphorus (P) content was quantified using spectrophotometric analysis.

A Ca/P ratio of 1.67 was obtained, after which FTIR analysis was conducted to identify functional groups in the HAp sample, particularly PO_4^{3-} and Ca–O groups. SEM analysis was performed to further examine the surface morphology, porosity, and particle size of the HAp. In addition, the percentage yield of HAp was calculated to determine the ratio of the final product mass to the raw material mass, which is a critical parameter for evaluating the feasibility of the precipitation method employed in this study.

3. Results and Discussion

3.1. Yield of Hydroxyapatite Products

The yield (% yield) reflects the efficiency of converting raw materials into the final product. It serves as a critical parameter for evaluating the overall synthesis process and determining its effectiveness. A high % yield indicates an efficient and effective synthesis, whereas a low % yield suggests material loss or incomplete conversion to the desired product.

Table 1. Yield data of submicron HAp at varying pH values

| Sample | pH | Sintering temperature (°C) | Product weight (g) | %Yield |
|--------|----|----------------------------|--------------------|--------|
| 1 | 8 | 800 | 1.4092 | 35.73 |
| 2 | 9 | 800 | 1.4566 | 36.93 |
| 3 | 10 | 800 | 1.6261 | 41.23 |
| 4 | 11 | 800 | 1.5379 | 38.99 |
| 5 | 12 | 800 | 1.4213 | 36.04 |

From Table 1, it can be seen that the resulting HAp exhibited variations in final mass. At pH 10 with sintering at 800°C, sample 9 produced the highest yield, with a mass of 1.6261 g and a % yield of 41.23%. However, when the synthesis was carried out at pH > 10, the final product mass decreased. This reduction is likely due to the high concentration of hydroxide ions present during the synthesis, which affected the reaction dynamics and hindered optimal hydroxyapatite crystal growth. Similar findings were reported by Andyana *et al.* [13], who noted that increasing the pH enhances the solubility of Ca^{2+} ions, leading to a higher hydroxide ion concentration in the sample and consequently reducing the yield of hydroxyapatite.

3.2. AAS and Spectrophotometry Analyses

AAS and UV-Vis spectrophotometry analyses were performed to determine the Ca/P molar ratio of the HAp samples. The AAS analysis was used to measure the Ca content expressed as atomic percentage (at.%), while the spectrophotometry analysis was used to determine the P content (at.%). The Ca/P molar ratio was then calculated for each sample by dividing the Ca (at.%) by the P (at.%) values obtained.

From Table 2, it can be observed that the Ca/P molar ratios vary significantly across the samples. Among them, sample 4, synthesized at pH 11 and sintered at 800°C, exhibited a Ca/P molar ratio of 1.6719, which is the closest to the theoretical value of stoichiometric hydroxyapatite (1.67). Similarly, sample 3 (pH 10) and sample 5 (pH 12) also produced ratios close to the theoretical value, with molar ratios of 1.6745 and 1.6692, respectively.

Table 2. Calculated Ca/P molar ratios of submicron HAp (sintering temperature: 800°C)

| Sample | Yield (g) | pH | Ca (%) | P (%) | Ca/P molar ratio |
|--------|-----------|----|--------|-------|------------------|
| 1 | 1.4092 | 8 | 2.43 | 4.23 | 1.7407 |
| 2 | 1.4566 | 9 | 2.48 | 4.25 | 1.7137 |
| 3 | 1.6261 | 10 | 2.55 | 4.27 | 1.6745 |
| 4 | 1.5379 | 11 | 2.56 | 4.28 | 1.6719 |
| 5 | 1.4213 | 12 | 2.57 | 4.29 | 1.6692 |

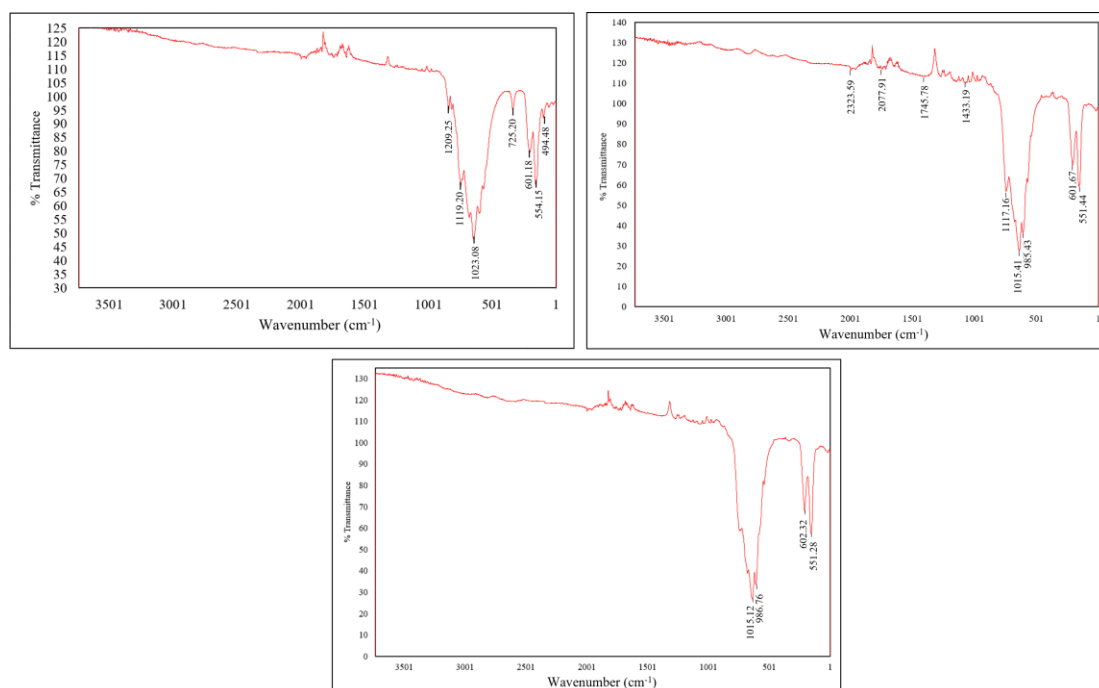


Figure 1. FTIR spectra of HAP samples

These findings demonstrate that varying the pH during synthesis affects the resulting Ca/P molar ratio. In general, increasing the pH tends to lower the molar ratio; however, at pH 12, a more noticeable decrease occurs. This may be attributed to the higher concentration of hydroxide ions (OH^-) in the system, which can alter the incorporation of calcium and phosphate into the HAP structure. This is supported by Andiyana *et al.* [13], which states that higher pH levels increase the solubility of calcium ions, leading to an elevated OH^- content in the sample. As a result, greater Ca ion solubility at high pH can reduce the overall Ca/P molar ratio.

Additionally, research by Yanti and Nia [14] suggests that pH 11 provides a stable condition for hydroxyapatite formation. Achieving a Ca/P molar ratio close to the stoichiometric value of 1.67 is desirable, as it is associated with superior material properties. According to Fihri *et al.* [15], non-stoichiometric hydroxyapatite exhibits several disadvantages, such as defects caused by vacancies in cationic or hydroxyl functional groups, poor crystallinity, and excessively high solubility. These characteristics are detrimental, particularly in biomedical applications, as excessive solubility can lead to rapid degradation of the implant material before sufficient new bone formation occurs, potentially compromising structural integrity.

3.3. FTIR Analysis

The characterization test using FTIR was used to identify the functional groups present in the HAP samples, primarily the PO_4^{3-} and Ca-O groups. The FTIR test results obtained on several samples are shown in Figure 1. Figure 1 shows that all HAP samples exhibit similar spectral patterns. The FTIR spectrum of sample 4 shows a peak with very strong intensity at 1015.41 cm^{-1} . The phosphate group (PO_4^{3-}) exhibits the highest intensity, indicated by stretching vibrations in the range of $1000\text{--}1150\text{ cm}^{-1}$ and bending vibrations in the range of $560\text{--}610\text{ cm}^{-1}$ [9]. In the FTIR spectra, the phosphate

group (PO_4^{3-}) was identified at 551.44 cm^{-1} , 601.67 cm^{-1} , 985.43 cm^{-1} , 1015.41 cm^{-1} , and 1117.16 cm^{-1} .

The Ca-O bond of the calcium group typically appears in the range of $1400\text{--}1700\text{ cm}^{-1}$ [16]. In the sample, a Ca-O bond with relatively low intensity was observed at 1433.19 cm^{-1} . These FTIR results confirm the presence of phosphate (PO) and calcium (Ca-O) bonds, which are the main structural components of hydroxyapatite. This indicates that a bond was successfully formed between calcium and phosphorus, derived from calcium nitrate tetrahydrate and diammonium hydrogen phosphate precursors. Furthermore, the presence of these characteristic functional groups confirms that the sample contains the fundamental constituents of hydroxyapatite.

3.4. XRD Analysis

The phases formed during hydroxyapatite synthesis were identified by XRD analysis, and the diffraction patterns were compared with JCPDS data no. 009-0432 (Joint Committee on Powder Diffraction Standards). The patterns of samples synthesized at pH 10, 11, and 12 (Figure 2) were analyzed within the 2θ range of $20^\circ\text{--}90^\circ$. All samples exhibited nearly identical patterns, with peaks predominantly corresponding to hydroxyapatite. In the sample synthesized at pH 10 (Figure 2a), the most intense peak was observed at $2\theta = 31.70^\circ$ (211), along with peaks at 25.81° (002), 32.92° (112), and 34.10° (300). At pH 11 (Figure 2b), the strongest peak appeared at $2\theta = 31.74^\circ$ (211), with additional peaks at 25.87° (002), 32.99° (112), and 34.15° (300). The sample at pH 12 (Figure 2c) showed its main peak at $2\theta = 31.80^\circ$ (211), with peaks at 25.93° (002), 33.04° (112), and 34.20° (300). Match! software confirmed that the samples possess a hexagonal crystal structure with space group $\text{P6}_3/\text{m}$, indicating a highly ordered atomic arrangement characteristic of hydroxyapatite.

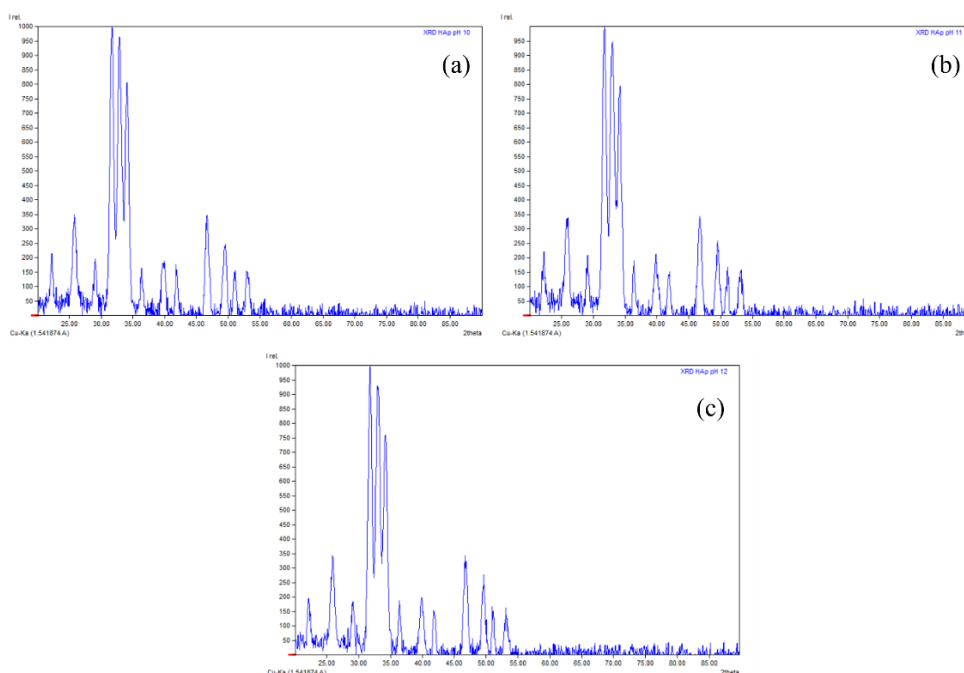


Figure 2. XRD pattern of HAp: (a) sample 3, (b) sample 4, and (c) sample 5

3.5. SEM Analysis

Particle morphology, porosity, and diameter were examined using SEM on sample 4 with a Ca/P molar ratio of 1.6719. The SEM results revealed the elemental distribution and surface texture of the sample. The sample appeared pure white without visible contamination. As shown in Figure 3, the sample exhibited good morphology with uniform crystal shapes. SEM observations indicated that HAp particles underwent agglomeration, forming irregular spherical clusters. Hydroxyapatite crystallites tended to aggregate with an average size of 15–50 nm [17]. However, hydroxyapatite with a submicron size (~500–800 nm) offers advantages in osteoclast formation and bone regeneration, compared with nanosized hydroxyapatite (~100 nm), which tends to inhibit osteoclast function [5]. In this study, the average particle size was submicron, measured at 460.8 nm. This characteristic is beneficial for biomedical applications, as the interaction of integrin proteins with lignans in bone enhances protein absorption, thereby accelerating the bone regeneration process.

Porosity analysis of the sample was carried out using ImageJ software based on the SEM images. The sample exhibited a porosity of 20.149%. This relatively low porosity can be attributed to the high-pH synthesis conditions, which accelerated the hydroxyapatite formation process and promoted the growth of larger, more ordered crystals that filled the interstitial spaces, reducing pore volume. Porosity plays a crucial role in the selection of implant biomaterials, as the required porosity varies with tissue type. For example, cancellous bone implants typically require 70–95% porosity, dental implants 40–60%, and compact bone implants 5–30%. Therefore, the synthesized submicron hydroxyapatite with 20.149% porosity is well suited for applications in compact bone implants.

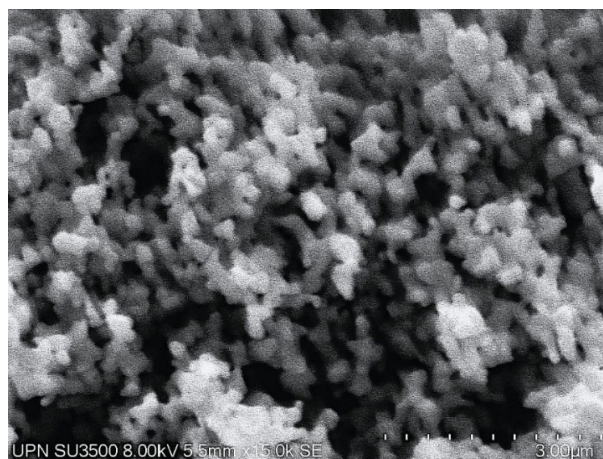


Figure 3. SEM image of sample 4 at 15,000× magnification

4. Conclusion

The results of this study demonstrate that hydroxyapatite (HAp) compounds were successfully synthesized, as confirmed by XRD analysis. FTIR spectra further indicated the presence of the characteristic functional groups of HAp. A pH of 11 was found to be optimal for obtaining stoichiometric hydroxyapatite (Ca/P ratio of 1.67), likely due to reduced calcium loss during synthesis. SEM analysis revealed that HAp synthesized at pH 11 had a submicron particle size of 460.8 nm. These findings indicate that the synthesized HAp possesses favorable properties for potential applications as a biomaterial in bone implants.

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