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CHARACTERISATION OF HYDROXYAPATITE SYNTHESISED BY HYDROTHERMAL METHOD FOR SOCKET PRESERVATION IN DENTAL IMPLANT SITES - AN IN VITRO STUDY

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ABSTRACT:
Introduction: Hydroxyapatite (HA) is a biocompatible and bioactive material that closely resembles the mineral component of human bone. One of the key advantages of the hydrothermal synthesis method is the high degree of crystallinity that it imparts to HA. The crystalline structure of HA is crucial for its mechanical strength and its ability to interact with surrounding tissues. The aim of this study is to synthesise hydroxyapatite by hydrothermal method for regenerative application as socket preservation material for dental implants. **Materials and methods:** The hydrothermal synthesis method for hydroxyapatite involves the reaction of calcium and phosphate sources in a sealed vessel under high temperature and pressure, facilitating the crystallization of hydroxyapatite. X-ray diffraction (XRD), and Fourier-transform infrared spectroscopy (FTIR) was done to confirm its phase, morphology, and functional groups. Blood compatibility of the material was also checked. **Results:** FTIR graph highlights characteristic absorption peaks of hydroxyapatite, particularly in the 1000-1100 cm" range for phosphate (PO.3-) groups. We found that the 5-hour synthesized hydroxyapatite has better blood compatibility than the 2-hour sample. **Conclusion:** The synthesised hydroxyapatite by hydrothermal method demonstrated good biocompatibility and the characterization was done and confirmed by FTIR and XRD methods. Hence, this material can be used as a socket preservation material for dental implant sites.
Keywords: Hydroxyapatite, Hydrothermal method, Socket preservation, Regeneration, Dental implant

INTRODUCTION:

Hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$) is a biocompatible and bioactive material that closely resembles the mineral component of human bone. Its properties make it suitable for various applications, including bone grafts, coatings for implants, and scaffolds in tissue engineering. [1]The hydrothermal method for synthesizing HA offers a controlled environment that can enhance the material's crystallinity and purity, which are essential for its performance in regenerative applications. With the increasing incidence of bone-related diseases, trauma, and aging populations, there is a growing demand for effective bone repair and regeneration strategies. Traditional approaches, such as autografts and allografts, have limitations including donor site morbidity, risk of infection, and limited availability.[2] Consequently, synthetic alternatives like hydroxyapatite have garnered significant attention. The hydrothermal method for synthesizing HA offers distinct advantages over conventional techniques. It enables the formation of well-crystallized, pure HA under controlled conditions, resulting in materials with enhanced structural properties. [3]By manipulating parameters such as temperature, pressure, and precursor concentration, researchers can tailor the morphology, particle size, and crystallinity of the synthesized HA to meet specific application requirements. Moreover, the hydrothermal approach is environmentally friendly, using water as a solvent and often leading to reduced waste and energy consumption compared to other synthesis methods. [4]This makes it particularly attractive for large-scale production and applications in regenerative medicine, where safety and efficacy are paramount. As research continues to explore the interplay between hydroxyapatite's structural properties and its biological performance, the potential for innovative applications expands. This article delves deeper into the hydrothermal synthesis of hydroxyapatite, outlining the process, characterization techniques, and its promising applications in regenerative medicine, ultimately contributing to the advancement of effective treatment strategies for bone regeneration.[5] As the demand for advanced biomaterials in the field of regenerative medicine continues to rise, hydroxyapatite has become a focal point for researchers seeking to develop materials that can promote healing and enhance tissue regeneration. [6]The growing field of bone tissue engineering, which aims to repair or replace damaged bone structures, has led to significant advancements in HA-based materials. For instance, HA is often used in the creation of scaffolds that provide structural support for the growth of new bone tissue in patients with bone defects caused by injury, disease, or aging.One of the key advantages of the hydrothermal synthesis method is the high degree of crystallinity that it imparts to HA. The crystalline structure of HA is crucial for its mechanical strength and its ability to interact with surrounding tissues. In addition, the hydrothermal approach allows for tailored particle size and morphology, which can influence the material's interaction with cells and its mechanical properties.[7] By adjusting reaction conditions such as temperature, pressure, precursor concentration, and pH, researchers can fine-tune the material to optimize its performance for specific applications. Furthermore, the controlled environment of the hydrothermal process minimizes the formation of unwanted secondary phases (such as tricalcium phosphate or octacalcium phosphate), ensuring a high level of purity in the synthesized HA.[8] This makes the hydrothermal method ideal for producing HA for sensitive applications, particularly in implantable devices and drug delivery systems, where purity and biocompatibility are paramount. When implanted into the living tissues, HA forms interlocking between the bone with the surface irregularities and forming a neoformed layer that assures direct interaction between bone and biomaterials to prevent the interference of fibruos tissues. Hydroxyapatite (HA) has gained prominence as a biomaterial for socket preservation, especially due to its resemblance to the mineral component of natural bone. Following tooth extraction, alveolar bone typically undergoes significant resorption, compromising aesthetics and functionality for future prosthetic or implant placement. By introducing HA into the socket, clinicians aim to mitigate this resorption while creating an osteoconductive scaffold to promote bone regeneration. Its crystalline structure offers mechanical stability, supporting the soft tissue and surrounding alveolar ridge during the healing process. The aim of this study is to synthesize hydroxyapatite by hydrothermal method for regenerative application as socket preservation material for dental implants.

MATERIAL AND METHODS :

Hydrothermal synthesis method - Principle.

The hydrothermal synthesis method involves the reaction of calcium and phosphate sources in a sealed vessel under high temperature and pressure, facilitating the crystallization of hydroxyapatite. This method allows for better control over particle size, morphology, and structural properties compared to conventional synthesis techniques.

Key reagent for HA synthesis includes:

1) **Calcium sources**: Calcium nitrate tetrahydrate ($Ca(NO_3)_2 \cdot 4H_2O$) or calcium hydroxide ($Ca(OH)_2$).
2) **Phosphate sources:** Diammonium hydrogen phosphate ($(NH_4)_2HPO_4$) or sodium phosphate (Na_3PO_4).
3) **Solvent**: Deionized water.
4) **pH adjusting agents**: Sodium hydroxide (NaOH) or hydrochloric acid (HCl).
5) **Hydrothermal reactor**: Teflon-lined autoclave.

Synthesis Procedure

1. **Preparation of Solutions**:
   - Dissolve the calcium source in deionized water to achieve the desired concentration.
   - Prepare a separate phosphate solution by dissolving the phosphate source in deionized water.

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2. Mixing:Slowly add the phosphate solution to the calcium solution under constant stirring to form a homogeneous mixture. Adjust the pH to between 8 and 10 using NaOH or HCl.

3. Hydrothermal Treatment:Transfer the mixture into the Teflon-lined autoclave and seal it. Heat the autoclave to temperature between 120°C and 200°C and maintain it for 4 to 24 hours, depending on the desired characteristics of HA.

4. Cooling and Filtration:After the reaction, allow the autoclave to cool to room temperature. Collect the precipitate by filtration and wash it with deionized water and ethanol to remove any unreacted materials.

5. Drying and Characterization:Dry the obtained HA powder at 60°C for 12 hours. Characterize the synthesized HA using techniques such as X-ray diffraction (XRD), and Fourier-transform infrared spectroscopy (FTIR) to confirm its phase, morphology, and functional groups. Blood compatibility of the material was also checked.

The Figure 1 appears to depict a chemical synthesis procedure involving the mixing of calcium nitrate (CaNO3) and diammonium hydrogen phosphate(DAHP) in ammonium solution followed by heating at 200 degree celsius for different durations (2 hours and 5 hours)

1) Ca(NO3)2 (Calcium nitrate) solution: 0.3 mol in 50 ml.
2) DAHP (Diammonium hydrogen phosphate) solution: 0.2 mol in 50 ml.
3) The two solutions are mixed with ammonium solution.
4) The resulting mixture is subjected to heat treatment at 200 degree celsius for 2 hours and 5 hours.

RESULTS:

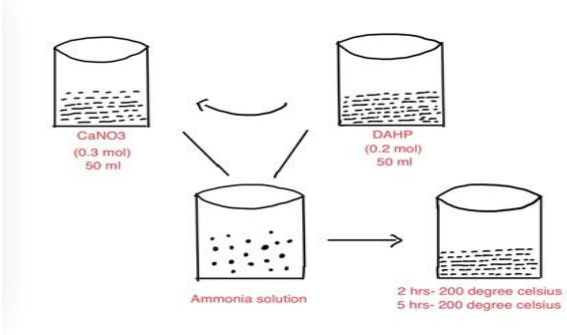

Figure 1: Material Synthesis

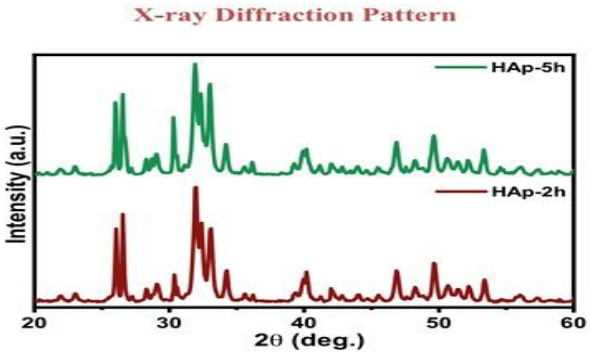

Figure 2: Xray Diffraction Pattern

In our study, Figure 2 represents an x- ray diffraction (XRD) pattern, a common technique used to identify the crystallographic structure of materials. This particular graph compares two samples labelled “HAp- 2h” and “HAp- 5h”, which likely refers to hydroxyapatite (HAp) synthesized under different heating conditions- 2 hours and 5 hours at 200 degree celsius respectively. HAp- 2h(red curve) refers to the peaks that are present but relatively broad and less intense. Broader peaks suggest that the sample has low crystallinity or smaller crystallite size. This could mean that the material has less well- ordered crystals after just 2 hours of heat treatment. HAp- 5h(green curve) refers to the peaks being sharper and more intense, indicating a more crystalline material. Longer heat treatment at 5 hours likely allowed the hydroxyapatite crystals to grow larger.

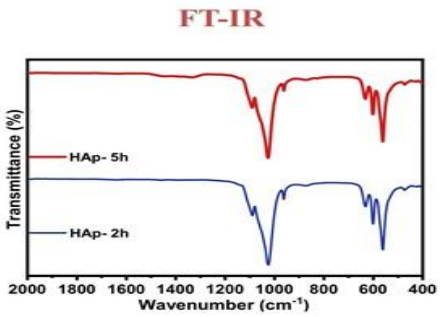

Figure 3: FTIR analysis

FT-IR (Fourier-transform infrared spectroscopy) image (Figure 3) displays the transmittance spectra for hydroxyapatite (HAp) samples synthesized at two different reaction times: 5 hours (red line) and 2 hours (blue line). The x-axis represents the wavenumber in cm", indicating specific bond frequencies, while the y-axis shows transmittance in percentage. The graph highlights characteristic absorption peaks of hydroxyapatite, particularly in the 1000-1100 cm" range for phosphate (PO.3-) groups. Differences in peak intensity between the two spectra suggest variations in crystallinity or structural properties due to synthesis time.The differences in the spectra suggest that synthesis time affects

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the crystallinity and stability of hydroxyapatite. Longer synthesis (5h) leads to higher crystallinity, while shorter times (2h) may result in a more amorphous structure. These structural variations can influence the material's biological properties, such as solubility and mechanical strength, which are critical for applications in bone tissue engineering and dental implant prosthetics.

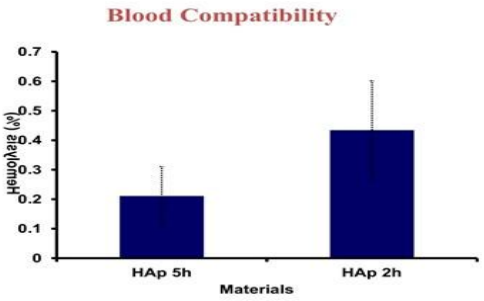

Figure 4: Blood compatibility

Figure 4 bar graph illustrates the blood compatibility of hydroxyapatite (HAp) samples synthesized at different times—5 hours (HAp 5h) and 2 hours (HAp 2h)—measured by hemolysis percentage (%). The y-axis represents hemolysis, an indicator of blood compatibility, with lower values indicating higher compatibility. The HAp 5h sample shows a lower hemolysis rate, around 0.2%, compared to the HAp 2h sample, which has a higher hemolysis rate, approximately 0.4%. This suggests that the 5-hour synthesized hydroxyapatite has better blood compatibility than the 2-hour sample. Thus, our novel material can be used along with bone grafts as a socket preservation material in dental implant sites.

DISCUSSION:

Hydrothermal synthesis, often performed at moderate to high temperatures and pressures, typically results in highly crystalline hydroxyapatite. Studies like those by [9] demonstrate that this method produces fewer impurities compared to sol-gel or precipitation methods, where uncontrolled conditions may lead to carbonate or hydroxyl substitutions, altering bioactivity. Hydrothermal synthesis offers control over particle size and morphology, which are crucial for regenerative applications. Smaller, nano-sized HAp particles have been shown to enhance bioactivity and integration in bone. A comparison study by [10] found that hydrothermal methods yielded more uniform nano-sized particles compared to mechanical milling techniques, which often produce larger or irregular particles with lower surface area. The high purity and controlled structure of hydrothermal HAp improve its biocompatibility and stability in physiological conditions. Studies like [11] emphasize that HAp synthesized through hydrothermal processes shows less inflammatory response and higher cell viability compared to HAp from other methods. Hydrothermal HAp is favorable for scaffolds and bone substitutes. Comparing with methods like microwave-assisted synthesis, hydrothermal HAp generally demonstrates better osteoconductivity, as seen in studies by [12]making it highly suitable for load-bearing applications.

Hydrothermal synthesis is renowned for producing high-purity, well-crystallized hydroxyapatite. This high crystallinity is important for applications in bone tissue engineering, as it provides mechanical strength and stability. A study by [13]reported that hydrothermal HAp closely mimics the crystalline structure of natural bone, making it more effective in osteointegration compared to HAp produced via sol-gel or precipitation, which may have phase impurities like calcium phosphate or carbonate substitutions. These impurities can affect bioactivity and degrade faster in physiological conditions. Particle size and morphology significantly impact HAp's interaction with cells.

Hydrothermal synthesis allows control over particle characteristics by adjusting temperature, pressure, and time. Smaller, nano-sized particles exhibit a higher surface area, promoting cellular adhesion, proliferation, and faster bone regeneration. For example,[14]found that hydrothermal HAp nanoparticles had better cell compatibility and osteoinductive properties than particles synthesized by high-temperature calcination, which tends to produce larger, irregular particles. The biocompatibility of HAp synthesized via hydrothermal methods is often superior due to reduced contamination from other phases. In a comparative study, [15] found that cells cultured on hydrothermal HAp scaffolds had higher proliferation rates than those on conventionally synthesized HAp. The high biocompatibility can be attributed to the controlled, pure environment of hydrothermal synthesis, which reduces inflammatory responses and ensures a stable, osteoconductive scaffold structure. Hydrothermal HAp is particularly useful in load-bearing applications due to its higher mechanical strength. In contrast, microwave-assisted or wet chemical HAp, while quicker to produce, tends to have lower density and crystallinity, which can compromise structural integrity. A study by [16] showed that implants coated with hydrothermal HAp withstood greater compressive forces compared to sol-gel HAp coatings, making them preferable for dental implant applications where mechanical stability is crucial. Hydrothermal synthesis often produces HAp that more closely resembles the chemical composition of natural bone mineral, especially when carried out at moderate temperatures and pressures. This feature is crucial for regenerative applications, as it improves material-cell interactions and facilitates natural bone growth. In contrast, rapid methods like flame spray synthesis may yield HAp with non-stoichiometric ratios of calcium to phosphate, which affects dissolution rates and bioactivity. Future result should also focus on development of 3D-printed HA scaffolds using hydrothermal synthesis could revolutionise bone tissue engineering. By controlling the morphology and microstructure, HA scaffolds can be designed to closely mimic natural bone, promoting better integration and faster healing..[17]

The limitations of hydrothermal synthesis of hydroxyapatite include difficulty in achieving precise control over particle size and morphology, high energy consumption, and scalability challenges. Additionally, variations in synthesis conditions may lead to inconsistencies in material properties, limiting reproducibility and practical applications in large-scale regenerative medicine. In terms of patient outcomes, the use of HA for socket preservation minimizes post-extraction complications, such as soft tissue collapse, while maintaining a natural contour of the ridge. However, clinicians must carefully evaluate each case to determine whether HA is the optimal material, as other options, such as xenografts, allografts, or autologous bone, may provide faster remodeling in certain scenarios. Cost and availability may also influence the choice, as HA tends to be more expensive than other grafting materials. Overall, HA remains a reliable option for socket preservation, particularly in patients where long-term stability and biocompatibility are paramount. For decades, hydroxyapatites (HA) are one of the biomaterials used in socket preservation procedure and was thought to be biocompatible, long-term resorbable or non-resorbable and osteoconductive. Several improvements have been made to enhance the properties of hydroxyapatites that acted in providing a framework during the healing process to provide better outcomes.

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CONCLUSION: The synthesised hydroxyapatite by hydrothermal method demonstrated good biocompatibility and the characterization was done and confirmed by FTIR and XRD methods. Hence, this material can be used as a socket preservation material for dental implant sites. Further clinical trials are required to know the ability of this material as a novel socket preservation material.

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CONFLICT OF INTEREST:
The authors have none to declare.

REFERENCES:

[1] Sampath V, Krishnasamy V. Synthesis and characterization of hydroxyapatite self-assembled nanocomposites on graphene oxide sheets from seashell waste: A green process for regenerative medicine. J Mech Behav Biomed Mater 2024;151:106383.
[2] Santos C, Gomes P, Duarte JA, Almeida MM, Costa MEV, Fernandes MH. Development of hydroxyapatite nanoparticles loaded with folic acid to induce osteoblastic differentiation. Int J Pharm 2017;516:185–95.
[3] Gshalaev VS. Hydroxyapatite: Synthesis, Properties, and Applications. Nova Biomedical Books; 2012.
[4] Raymond S, Maazouz Y, Montufar EB, Perez RA, González B, Konka J, et al. Accelerated hardening of nanotextured 3D-plotted self-setting calcium phosphate inks. Acta Biomater 2018;75:451–62.
[5] Elliott JC. Structure and Chemistry of the Apatites and Other Calcium Orthophosphates. Elsevier; 2013.
[6] Barbeck M, Jung O, Smeets R, Koržinskas T. Biomaterial-supported Tissue Reconstruction or Regeneration. BoD – Books on Demand; 2019.
[7] Palmero P, De Barra E, Cambier F. Advances in Ceramic Biomaterials: Materials, Devices and Challenges. Woodhead Publishing; 2017.
[8] Wu S-C, Hsu H-C, Wu W-H, Ho W-F. Enhancing Bioactivity and Mechanical Properties of Nano-Hydroxyapatite Derived from Oyster Shells through Hydrothermal Synthesis. Nanomaterials (Basel) 2024;14. https://doi.org/10.3390/nano14151281.
[9] Kumar S, Gautam C, Mishra VK, Chauhan BS, Srikrishna S, Yadav RS, et al. Retraction of "Fabrication of Graphene Nanoplatelet-Incorporated Porous Hydroxyapatite Composites: Improved Mechanical and in Vivo Imaging Performances for Emerging Biomedical Applications." ACS Omega 2020;5:26956.
[10] Peters J, Singh G, Hakobyan H. Surgical Treatment of Clavicular Fractures, Refractures, Delayed and Non-Unions Using a Resorbable, Gentamicin-Eluting Calcium Sulphate/Hydroxyapatite Biocomposite. Ther Clin Risk Manag 2022;18:551–60.
[11] Ma Q, Hou X, Zhao C, Yan Y, Cheng X, Li J, et al. Diagnostic power of vertebral hydroxyapatite concentration measurements in spectral CT for osteoporosis-associated fractures and impact of intravenous contrast administration. Eur Radiol 2023;33:4016–23.
[12] Guo X, Xue M, Chen F, Guo Q, Zhou X, Lin H, et al. Local delivery and controlled release of miR-34a loaded in hydroxyapatite/mesoporous organosilica nanoparticles composite-coated implant wire to accelerate bone fracture healing. Biomaterials 2022;280:121300.
[13] Dorozhkin SV. Hydroxyapatite and Other Calcium Orthophosphates: Nanodimensional, Multiphasic and Amorphous Formulations. 2017.
[14] Hui Y, Dong Z, Wenkun P, Yao D, Huichang G, Tongxiang L. Facile synthesis of copper doping hierarchical hollow porous hydroxyapatite beads by rapid gelling strategy. Mater Sci Eng C Mater Biol Appl 2020;109:110531.
[15] Rech I, Kamogawa MY, Jones DL, Pavinato PS. Synthesis and characterization of struvite derived from poultry manure as a mineral fertilizer. J Environ Manage 2020;272:111072.
[16] Li X, Xiao J, Gai X, Du Z, Salam MMA, Chen G. Facilitated remediation of heavy metals contaminated land using Quercus spp. with different strategies: Variations in amendments and experiment periods. Sci Total Environ 2023;876:163245.
[17] Aoki H. Science and Medical Applications of Hydroxyapatite. Medico Dental Media International; 1991.