

# Characterization of Nano-Calcium Hydroxyapatite Prepared by Co-precipitation Method

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**Abstract:** Calcium hydroxyapatite is the main inorganic constituent of human bone and teeth. It has all the characteristics features of bio-materials in particularly having crystallographic similarities with natural bone materials. Ultrafine nano sized hydroxyapatite was synthesized with addition of different concentrations of Calcium nitrate tetra hydrate  $[\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}]$  and phosphoric acid  $[\text{H}_3\text{PO}_4]$  using microwave assisted chemical co-precipitation method. Fourier transform infrared spectroscopy [FTIR] analyses were used to investigate the change of crystalline phases and functional groups of hydroxyapatite at different temperatures. The energy dispersive X-ray diffraction analysis [EDAX] has confirmed the stoichiometric composition. The extent of agglomeration and morphology of the prepared and annealed samples were analyzed by Scanning electron microscope [SEM]. SEM images with EDS spectrum reveal the homogenous nature of the samples and uniform pore distribution on the surface.

**Keywords:** Nano-hydroxyapatite, chemical co-precipitation, fourier transform infrared spectroscopy, scanning electron microscopy.

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## Introduction

Bones and teeth are rigid tissues, they store substantial with calcium. Although both bones and teeth share a similar component, the teeth do not contain any living tissue, unlike bones. Living tissues allow bones to be continuously re-modeled and heal, while teeth cannot do so. Calcium hydroxyapatite is an inorganic compound in which seventy percent of bone is made up of hydroxyapatite. The basic elements of hydroxyapatite are calcium and phosphate with a stoichiometric ratio of 1.667. Various approaches were investigated the methods of creating nanoparticles. The parameters of nanoparticles that affect the shape, size and crystallinity nature needs further research [1-4]. For the repair and reconstruction of bone tissue imperfections, hydroxyapatite is widely accepted biomaterial. The process for the synthesizing of hydroxyapatite by reaction in solid state [5], co-precipitation [6], Hydrothermal method [7], microwave processing [8]

and sol-gel process [9]. Co-precipitation techniques are widely utilized due to the facile nature of the process. Chemical co-precipitation involves the use of aqueous solution, which leads to obtaining hydroxyapatite with high precise super facial area and small particle size distribution. Hydroxyapatite synthesized by this method, results in high purity and crystalline. The aim of this work is to prepare nano-hydroxyapatite by using chemical co-precipitation method for various mole concentrations.

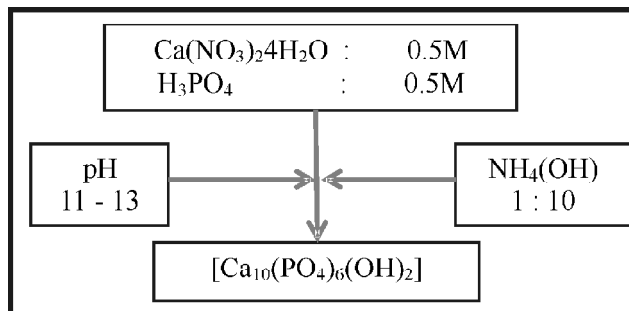
## Materials and methods

In the present study, to prepare nano sized hydroxyapatite chemical co-precipitation method was used. Calcium nitrate tetra hydrate  $[\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}]$  and phosphoric acid  $[\text{H}_3\text{PO}_4]$  are the preliminary materials used in the preparation. Calcium nitrate tetra hydrate is a white crystal, odorless and strong oxidizer. Its density is 2.36 and melting point is 45°C. Phosphoric acid is one of the most important and useful mineral acid. It is a white crystalline solid with melting point of 42.35°C. It is non-toxic and non-volatile.

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To form 0.5 mole solution, calcium nitrate tetra hydrate  $[\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}]$  was dissolved in double distilled water and phosphoric acid  $[\text{H}_3\text{PO}_4]$  was added with a Ca/P ratio of 1.67 and the mix was stirred constantly for 30 minutes using a magnetic stirrer. To form precipitate, ammonium hydroxide was poured in to the mixed solution and it also stirred constantly for another 30 minutes. The obtained product was filtered through filter paper. To remove the unwanted ions  $[\text{NH}_4^+$  and  $\text{NO}_3^-]$  from the obtained hydroxyapatite powder it was washed repeatedly and then set for drying at  $75^\circ\text{C}$  for 3 hours using an electric oven. The dried powders were then irradiated using a microwave oven at  $300^\circ\text{C}$  and  $600^\circ\text{C}$  to check the effect of temperature on the grain coarsening of the microwave heating. The figure 1 shows the experimental procedure for 0.5M:0.5M concentration. The final product was characterized by instrumentation techniques to study its phase behavior and crystalline nature.

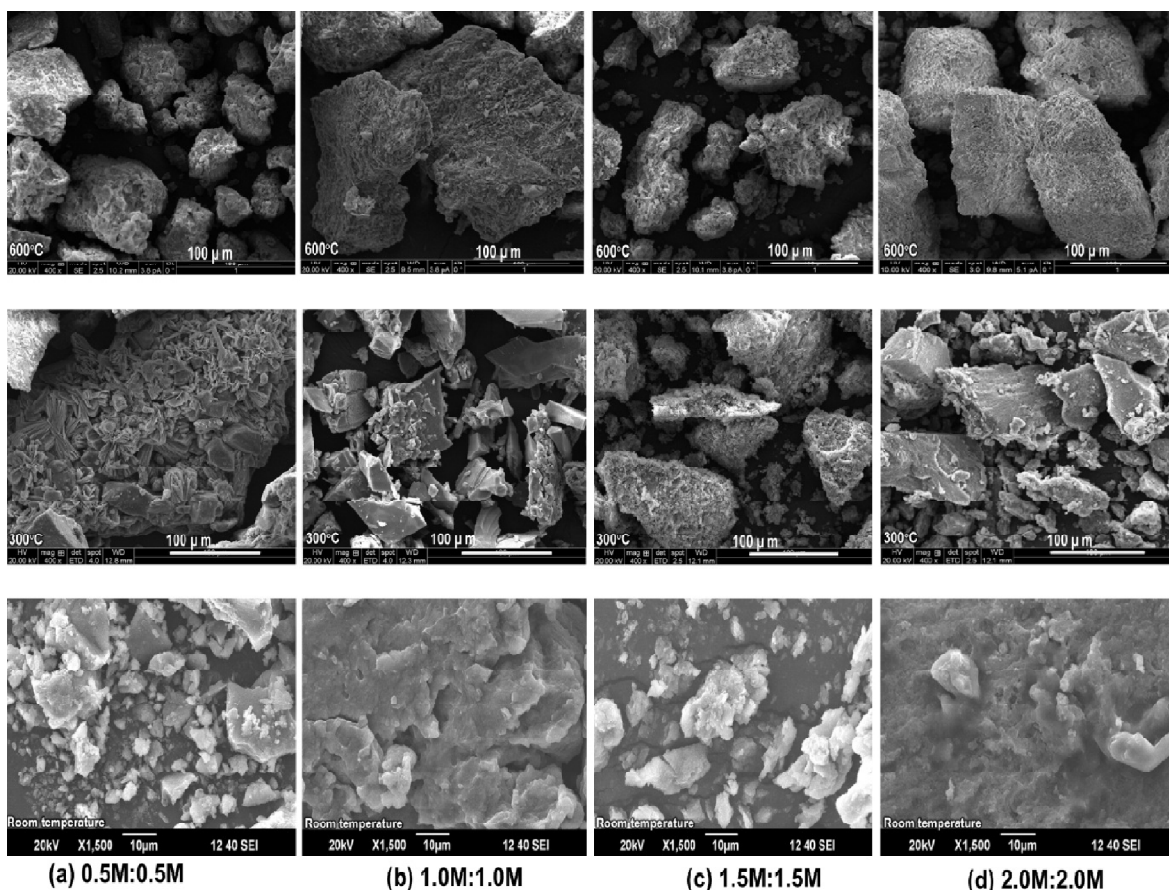


**Figure 1:** Experimental Procedure for 0.5M:0.5M concentration

The obtained nano-hydroxyapatite was characterized by SEM, FTIR and EDAX analysis. SEM is used to identify the phase composition, crystallinity, functionality and size of the crystal. Spectroscopic data's are analyzed with the help of spectroscopic instruments like spectral analyzers, spectrophotometers, spectrometers and spectrographs.

## Results and discussion

### SEM Analysis



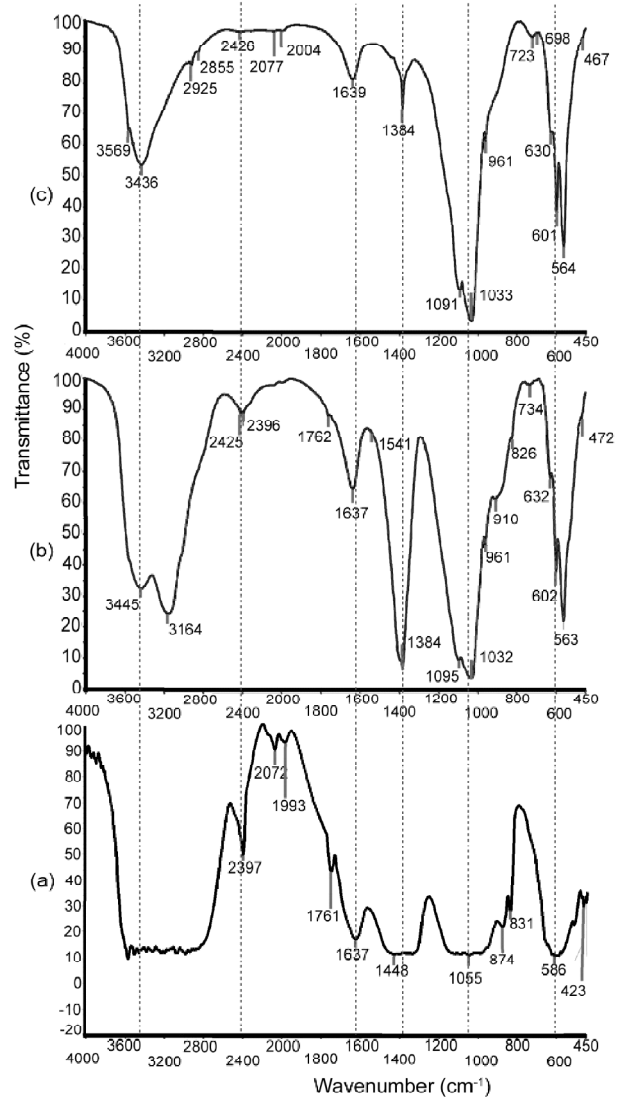
**Figure 2:** SEM images of prepared samples a,b,c,d with various mole Concentration

Figure 2 represents the SEM images of nano-hydroxyapatite prepared by chemical co-precipitation method. The SEM images of hydroxyapatite with various molar concentrations are shown in figure 2(a-d). On comparing the SEM images it is found that with molar concentration 0.5 M-0.5 M are non-homogenous with highly aggregated state at lower annealing temperature. In the case of HA with molar concentration 1 M-1 M the uniformity in the size of the particle improves but slight agglomeration is still observed. However as the molar concentration was further increased to 1.5 M- 1.5 M and 2 M-2 M, the nanopowders are found to be more homogenous and uniformly distributed. The SEM results indicate that the different annealing temperature playing an important role into produce different morphologies and aggregation status of the HA nanoparticles[10].

From the SEM images it can be seen that the synthesized hydroxyapatite is porous in nature. The closed pores are formed when the mole concentration increases. Nano-hydroxyapatite is homogeneously dispersed in the pore wall as well pore surface. The pores are regular, uniformly distributed and are interconnected which are used in the application of bone tissue engineering. This porous nature is desirable property and can have a positive impact when used in the implant as it facilitates interaction between the implant and the biological environment[11].

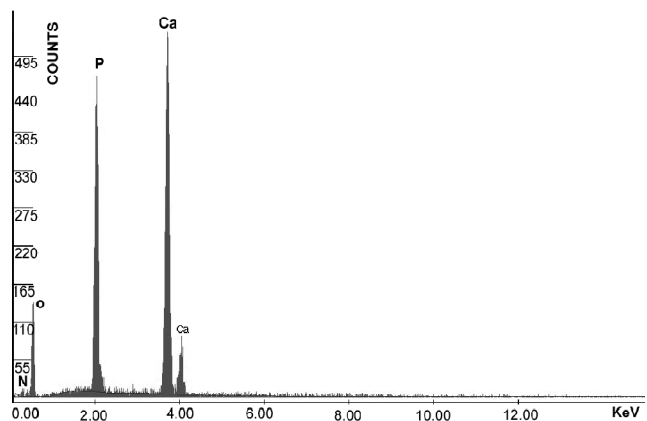
*FTIR Spectral analysis*

FTIR pattern curve of hydroxyapatite at room temperature, 300°C and 600°C are depicted in figure 3. The sharp band centered at 3456cm<sup>-1</sup>, 3445cm<sup>-1</sup> and 3436cm<sup>-1</sup> represents the stretching vibration of combined water in the HA lattice[12]. As the temperature increases the liberation and stretching band of OH<sup>-</sup> gradually decreases in their intensity. The peak at 2397cm<sup>-1</sup>, 2396cm<sup>-1</sup> and 2426cm<sup>-1</sup> are obtained due to the presents of HPO<sub>4</sub><sup>2-</sup> ions. The sharp bands at 1637cm<sup>-1</sup>, 1637cm<sup>-1</sup> and 1639cm<sup>-1</sup> were attributed to H-OH bending vibration. Very intense peak centered at 1448cm<sup>-1</sup>, 1384cm<sup>-1</sup> and 1384cm<sup>-1</sup> represents hydrogen bonded O-H symmetric stretching vibration. The point with wavenumber 1091 cm<sup>-1</sup>, 1095cm<sup>-1</sup> and 1055cm<sup>-1</sup> shows the presents of P-O asymmetric stretching of PO<sub>4</sub><sup>3-</sup>[13] functional group. Bands 586 cm<sup>-1</sup>, 563cm<sup>-1</sup> and 564cm<sup>-1</sup> assigned the presence of calcium phosphate.



**Figure 3:** FTIR Spectrum for sample (B1)0.5M : 0.5M

*EDAX Spectral analysis*



**Figure 4:** Edax spectrum of hydroxyapatite for 300°C 1.5M:1.5M concentration

**Table 1**  
Stoichiometric ratio of Ca, P, O and C (atomic %)

| Element | (keV) | Mass% | Atom% | K      |
|---------|-------|-------|-------|--------|
| C K     | 0.277 | 10.28 | 22.42 | 2.9183 |
| O K     | 0.525 | 12.62 | 20.67 | 1.1103 |
| P K     | 2.013 | 33.98 | 28.73 | 1      |
| Ca K    | 3.69  | 43.12 | 28.18 | 1.3304 |
| Total   |       | 100   | 100   |        |

The chemical composition of the prepared sample was confirmed by EDAX analysis. The EDAX spectra of nano-hydroxyapatite for 300 °C with 1.5 M: 1.5 M concentration was shown in figure 4. The stoichiometric ratio of Ca, P, O and C (atomic %) is given in table 1. The presents of Ca, P, O and C as the elementary species in the sample indicate the high purity of the product.

### Conclusion

In the present study, Calcium hydroxyapatite have been successfully prepared by chemical co-precipitation method, From the SEM analysis, it is found that when the mole concentration increases, the nano-hydroxyapatite are homogeneously dispersed in the pore wall as well as pore surface. These pores enhance the osteoblasts to interact and grow over its surface[14]. EDAX confirms the high purity of the product. FTIR spectra revealed the presence of the hydroxyl functional group. With increase in temperature, the intensity decreases.

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