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Synthesis of Nano-Bioceramic Material using Chemical Precipitation Method

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ABSTRACT

In the present work, we have synthesized nano-bioceramic material by using simple cost effective chemical precipitation method. Calcium Nitrate and Di-ammonium hydrogen orthrophosphate are used as a source of calcium and phosphrous to maintain the Ca:P ratio 1.66 which is similar to our natural bone .The pH was maintained at 12 by using liquid Ammonia and synthesis is carried out at constant temperature 100^{o}C for continuously stirred for 3 hrs. Afterwords synthesized material is dried at 100degree celcius for 2 hrs. Structural investigation is carried out by using XRD and morphological evaluation is examined by Scanning Electron Microscope. It is observed that the synthesized material is found in the range of 30-60 nm as investigated by XRD and SEM. Hence it is remarkable to note that the one-pot chemical precipitation method is successfully used to synthesize nano-bioceramic.

Keywords: Calcium Nitrate, Diammonium Hydrogen Orthrophosphate, XRD, SEM.

INTRODUCTION

A biocompatible ceramic (inorganic solid held by ionic or covalent bond) consisting of calcium and phosphates. Eg. Hydroxyapetite. Nanopowder is a solid powder of artificial origin that contains nano objects, aggregates of nano objects and dimension ranging from 1-100nm. Nano powder is characterized by Mean size of particle,

Size distribution of particles and Rate of agglomeration of particle. Calcium phosphate based bioceramic are nontoxic having biological effects is widely used in biomedical, tissue engineering. It is denoted as $[M_{10}(XO_4)_6Z_2]$ where M is a metal divalent cations, such as Ca^{2+} , Sr^{2+} etc., X is a trivalent species such as phosphate and is anion such as OH^- , F^- or a halogen. If OH^- anion is present in given structure it I named as hydroxya-patite, if F^- then it is known as fluorapatite, and if Cl^- then named as chloroapetite. The chemical composi-tion of pure calcium phosphate is $Ca_5(PO_4)_3(OH)$ (pentacalcium- hydroxyl-triphosphate) , but it is usually written as $Ca_{10}(PO_4)_6(OH)_2$. The crystal unit cell consist of two molecules and its Ca:P atomic ratio is 10:6 (i.e. 1.67). Hydroxyapatite, having chemical formula $Ca_{10}(PO_4)_6(OH)_2$ shows that it contains 39.85 wt % of Calcium, 18.51 wt % of Phosphorus, 41.41 wt % of Oxygen, and 0.20 wt % of Hydrogen. If Ca/P ratio is low, the acidity and solubility of mixture is high. But for mixture having Ca/P ratio close o 1.67, it has low acidity and solubility. Hydroxyapetite exist in two phases as monoclinic and hexagonal. But chemi-cally pure Hap exist in monoclinic phase. There is phase transition of monoclinic to hexagonal above 250^0C. Hydroxyapetite crystal is basically composed of Ca ions, PO_4 tetrahedra, and OH groups. Inside the crystal, each hydroxyl cation is completely surround-ded by calcium anions, while each calcium anion is surrounded by phosphate cations. Th difference of monoclinic and hexagonal structure is due to the arrangement of hydroxyl ions. Hexagonal hydroxy-apatite has a disordered hydroxyl arrangement, while monoclinic hydroxyapatite has an ordered hydroxyl arrangement. The hexagonal structure of calcium hydroxyapatite is a more common one for biomedical applications.

Nanocrystalline hydroxyapatite is synthesized by various techniques such as sol-gel, hydrothermal, wet chemical precipitation, microwave irradiation, etc. A stoichiometric and well crystalline product is obtained with solid state method but it requires high tempera-ture- pressure and long heat treatment time. Nano-cryastal can be formed at temperature below 100^0C with the help of wet chemical precipitation method. Crystallinity and stoichiometry obtained by these method is relatively close to well crystallized stoichio-metric hydroxyapatite. So we synthesized Hap by wet chemical precipitation due to easy preparation, low cost of instrumentation, low temperature synthesis.

METHODOLOGY

Synthesis for Hydroxyapatite

1) For 0.6 M of di-ammonium hydrogen orthrophosphate-

   Molecular weight of $(NH_4)_2HPO_4$= 132.06gm

   For 0.66 M of $(NH_4)_2HPO_4$ x=8.7159 gm in 100ml

   For 6.6 M of $(NH_4)_2HPO_4$ x=87.159 gm in 1000ml

   For 50ml x= 4.357gm

   Dissolve 4.357 gm of di-ammonium hydrogen orthrophosphate in 50ml of distilled water and stir to form clear solution.

2) For 1M of calcium nitrate tetra hydrate-

   Molecular weight of $Ca(NO_3)_4H_2O$ = 236.15gm

   For 1M of solution in 100ml y= 23.615gm

   For 1M of solution in 50ml y= 11.08 gm

   Dissolve 11.08 gm of calcium nitrate tetra hydrate in 50ml of distilled water and stir to form clear solution.

3) Now dropwise 0.6 M of di-ammonium hydrogen orthrophosphate is added into 1M of calcium nitrate tetra hydrate solution.

4) Liquid ammonia is added to maintain pH of solution upto 12.

5) Process is carried out by stirring continuously at 100^0 C for 3 hrs.

6) Solution is kept in steady state for a night to settle ppt at bottom.

7) Formed white ppt is washed 3-4 times with double distilled water and dried in air oven for a two nights.

8) Now it is grinded with the help of mortar and hydroxyapetite is formed.

9) Further it is used for characterization.

The reaction is as follows

$$6(NH_4)2HPO_4 + 10Ca(NO_3)_2 + 8NH_4OH$$

$$\downarrow$$

$$6H_2O + 20NH_4NO_3 + Ca_{10}(PO_4)_6(OH)_2$$

[Hydroxyapetite]

Table 1: Table showing properties of Hap

Paremeters	Values
Chemical composition	$Ca_{10}(PO_4)_6(OH)_2$
Ca/P ratio	10:6 (1.67)
Crystal	Hexagonal
Biocompatibility	High
Bioactivity	High
Biodegradation	Low
Osteoconduction	High

Characterization Techniques-

X-ray Powder Diffraction (XRD):

X-ray diffraction (XRD) is used for the characterization of crystalline solids and determination of their structure. When X-ray is incident on given structure, it interact with atoms and constructive interference is obtained which gives detailed information.

By Braggs law, $n\lambda=2d\sin\theta$

Where, n= an integer, λ= the wavelength of the X-rays, d= the spacing between atomic layers, θ=the angle between the incoming X-ray and the atom layer.

Mean dimension D is given as

$$D = \frac{K\lambda}{\beta cos\theta}$$

Where, K is constant approximately equal to unity and related to thecrystalline shape, β is FWHM of the diffraction peak.

The wavelength of the X-ray source empoloyed is 1.54056 A^0 with photon energy of 8 KeV. The XRD peaks are recorded in the 2θ range of 20^0-60^0. We saw peaks at 2θ value 25.6^0, 25.4^0 , 28.5^0 , 30.1^0 , 31.7^0 ,32.7^0, 35.1^0, 36.1^0, 40.1^0, 43.3^0, 49.3^0, 52.5^0, 57.5^0, which confirm the crystalline hexagonal phase. The average crystalline size calculated is 27 nm.

FTIR Analysis:

Fourier Transform Infrared Spectroscpy(FTIR) is a technique used to obtain an infrared spectrum of absorption or emission of solid, liquid, gas. FTIR spectrometer simultaneously collects high spectrum resolution data over a wide spectral range. FTIR spectra of Hap film is recorded at room temperature in the wave number range 4000 and 400 cm^- . The spectra confirm the presence of water, hydroxyl and phosphate species. The FTIR spectrum of the pure Hap shows the presence of hydroxyl stretching modes in the apatite structure at 3572 cm^- .The absorption peaks for hydroxyl liberation mode are found at 727 cm^- and 1603 cm^-. The bands appearing at 963 cm^- and 901 cm^- correspond to anti symmetric bending motion to v_3 stretching mode in $PO4^{3-}$ group. The study of FTIR spectra showed all bands corresponding to pure Hap structure.

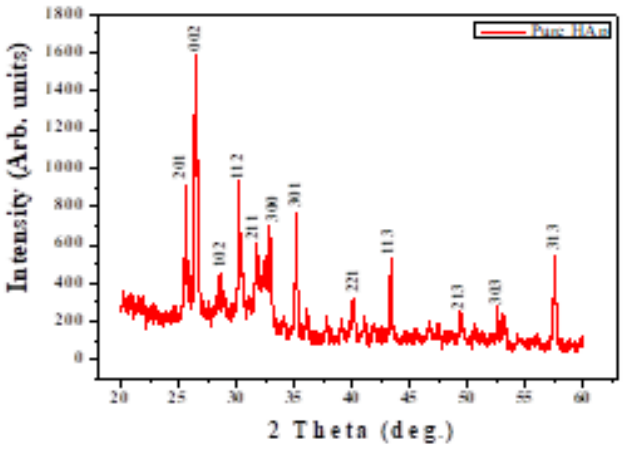

Fig.1: XRD pattern of pure HAp thick films

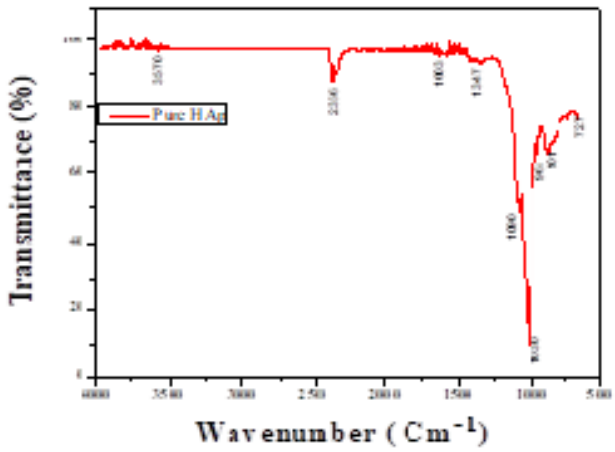

Fig.2: FTIR spectrum of HAp thick films

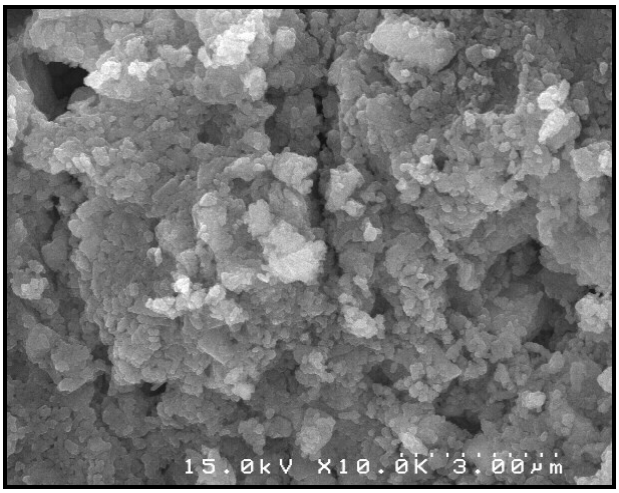

Fig.3: SEM pattern of pure HAp thick films

Scanning Electron Microscopy (SEM):

SEM is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. Electrons interact with atoms in the sample, producing various signals that can be detected and contain information about sample surface composition. The principle is based on the interaction of an incident electron beam and the solid specimen. The study of SEM shows presence of smaller grains with a uniform size and shape covering whose surface along with microporous structure.

CONCLUSION

Nano-crystalline hydroxyapatite is synthesized by using wet chemical process. The synthesized Hap powder shows hexagonal phase with nano-sized grains. The surface morphology of Hap film showed separated grains with of micro porous structure. The elemental composition of Hap film is confirm by EDS.

Conflicts of interest: The authors stated that no conflicts of interest.

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