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Synthesis and Characterization of Hydroxyapatite for Biomedical Applications

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Abstract: **Hydroxyapatite (HAp) is a mineral which is the main composition of bone and teeth in human body. It is well documented that the biocompatibility and bioactivity of bone or teeth could be improved through the synthesis of nanoparticle HAp. Among several methodologies available for synthesizing HAp wet chemical technique was found to be used for well controlling varying processing parameters. This article focuses on synthesis of HAp for surface modification of orthopedic and dental implants. HAp was synthesized using DiAmmonium Hydrogen Phosphate (DHP) and Calcium Nitrate Tetrahydrate (CNT) precursors. Proper care has been taken to maintain pH around 10.8 while stirring with the help of Ammonium Hydroxide (AH). The morphological analysis by Scanning Electron Microscope (SEM) measurement shows that the particles are tightly agglomerated with an average size of 300-400 nm. X-Ray Diffractometry (XRD) analysis detects the purity of HAp. Energy Dispersive X-Ray Spectroscopy (EDX) gives the elemental composition of HAp and further characterization through Fourier Transformed Infrared Spectroscopy (FTIR) was also done.**

***Index Terms*— Hydroxyapatite (HAp), Diammonium Hydrogen Phosphate (DHP), Calcium Nitrate Tetrahydrate (CNT), Scanning Electron Microscope (SEM), X-Ray Diffractometry (XRD), Energy Dispersive X-Ray Spectroscopy (EDX), Fourier Transformed Infrared Spectroscopy (FTIR).**

I. INTRODUCTION

Hydroxyapatite (HAp) is an emerging bioceramic material this helps bone in growth and osseointegration for dental and maxillofacial applications [1], [3]. HAp is the greatest substitution for bone which gives tight bonding for bone tissue, where pores in the implants must be present for osteointegration. The minimum pore size for an implant's bone growth is said to be as 100-135 µm [2]. Synthetic HAp can be thermodynamically stable at physiological pH and osteoconductive. This HAp is widely used in reconstruction applications like implant coating. The advantages over synthetic polymers are non toxic in nature. The synthesis of HAp could be done through various techniques which include mechanochemical, sol-gel preparation, combustion preparation, hydrothermal preparation, electrophoretic deposition [4]. HAp is synthesized using wet chemical method often since it is easy to drive [5], [8]. The properties depend upon the synthesis techniques, temperature and atmosphere in which it is made [5]. HAp depends on many factors during the synthesis procedure; they also depend on the thermal treatment profile for drying and sintering [6]. Natural polymers posses great potential for industrial and medical applications [7]. Various mechanical properties like osteconductivity, osteoinductivity, improve the performance of bone regeneration which makes HAp an important material in tissue engineering. The nano level composites have been investigated and demonstrated gives a result that the nano particles create an impact on cell biomaterial interaction. The nano level HAp could cause some migration at the implant site and can cause some damages to the healthy tissues in surrounding, were some researchers came with a solution to this problem. When could be avoided by making a composite with polymers and nano HAp [8]. Even though HAp result in rapid bone formation and fast biological fixation to bony tissues and has favorable properties it has low fracture toughness and also low load bearing capacity. Thus an enhancement in their mechanical properties is very much needed. The uncoated implants needed attention over the areas such as cracking, shearing off and chipping, this could be overcome by coating the orthopedic and dental implants with the HAp which result in lesser failure rate of the implant [9]. The implants which do not go under surface modification takes relatively very long time for implant-bone fixture. The HAp could be used to coat on complex shaped substrates and complex topography which many of the orthopedic and dental implant have [10].

II. MATERIALS AND METHODS

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A.Materials

The raw materials which are used for the synthesis of HAp are DiAmmonium Hydrogen Phosphate ($(NH_4)_2 HPO_4$), Calcium Nitrate Tetrahydrate ($CA(NO_3)_2$) precursors, Ammonium Hydroxide NH_4OH, Distilled water and Ethanol.

B. Synthesis of HAp

The most popular and widely used technique for HAp synthesis is the wet chemical technique. 0.4 mol of DHP was dissolved in 200 ml of distilled water in a 200 ml beaker and a pH of 4 was maintained. In a 500 ml beaker 0.6 mol of CNT was dissolved in 200ml of distilled water and a pH of 7.4 was maintained. This whole procedure was carried out at room temperature. The phosphate solution was slowly added drop wise to the calcium solution for 4 to 5 hours. The pH has been maintained at 10.8 with AH during the precipitation reaction. The mixture was allowed to stir vigorously through the adding process. Then the mixture was allowed to stir overnight. A white precipitate was formed which was then collected and dried at 80^o Celsius. The grain size of HAp was found to be dependent on the reaction time and temperature. HAp was washed and dried simultaneously for two to three times with distilled water and Ethanol.

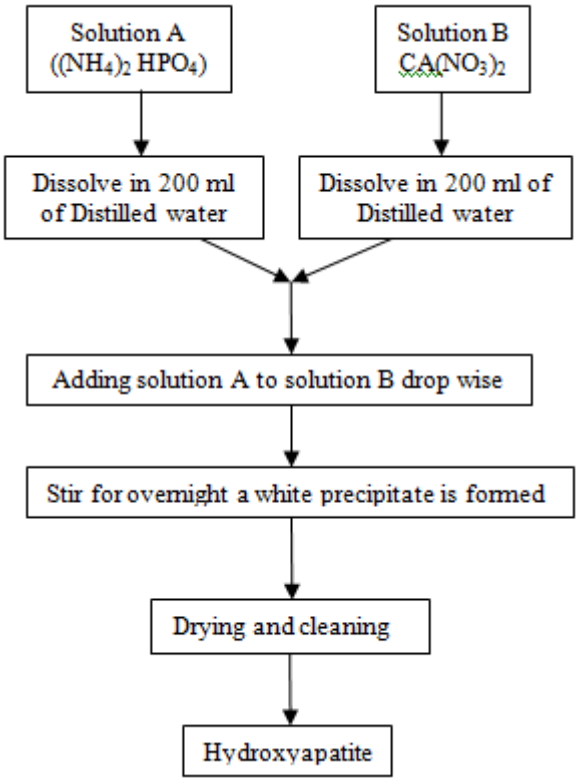

Fig 1: Flowchart for Synthesis of HAp

III. CHARACTERIZATION

A.FTIR

FTIR spectroscopy was done to determine the functional groups associated with HAp. The spectrum range for investigation was 400 cm^{-1} to 4000 cm^{-1}. Fig 2 shows the peak values of synthesized HAp the strong peak of ion stretching vibration at 3418.495 cm^{-1} OH stretch, hydrogen boned hydroxyl group. The peak 1634.991 cm^{-1} gives the carbonyl double bonded stretching group. The bending mode phosphate group was confirmed with the 563.422 cm^{-1} from the FTIR spectrum.

B.XRD

The x-ray analysis was done using powder x-ray diffraction. Scan range was given as 2θ and wavelength of x-ray was given as 1.5405 A^o. The XRD analysis gives the nano crystalline nature of the compound. Better crystallinity has been observed from the sharper peaks. The high intensity peak values are referred with the standard JCPDS(2300273) values for HAp. Table 1 shows the miller indices values of the reflection panel are assigned with the d-spacing values which confirm the presence of HAp. The particle size can also be determined with the Scherrer equation. Fig 3 shows the Miller indices values of (h,k,l) for corresponding reflection panel.

(h,kl) values	High Intensity Peak values (2θ)
(1,0,2)	29.5
(3,2,0)	31
(3,0,0)	33
(3,1,3)	49.8

Table1: Miller indices values for High Intensity Peaks

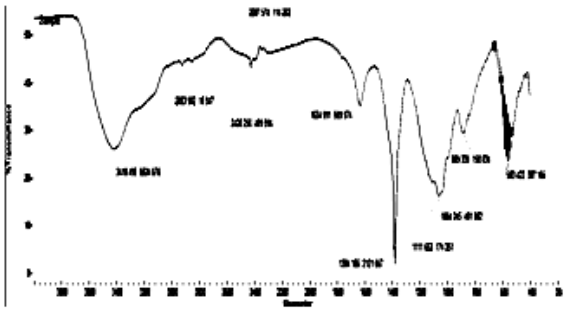

Fig 2: FTIR Spectra of HAp

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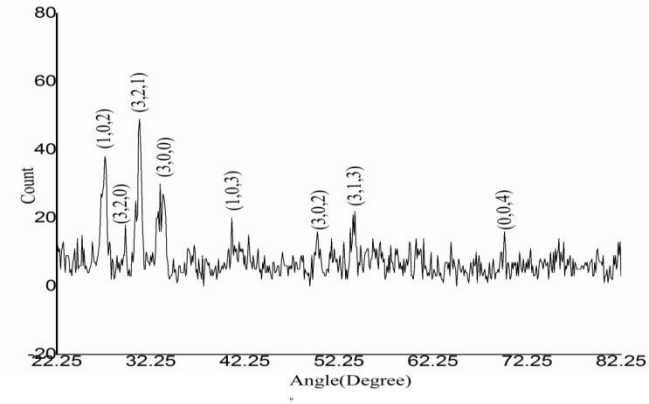

Fig 3: XRD Spectrum of HAp

C.SEM

Scanning Electron Microscope was used to know the morphology of HAp. The morphology study using SEM shows a homogeneous particle shape. Fig. 4 and 5 are the SEM image of HAp at a magnification range of 30,000x. The particles are agglomerated and dense. The particles are rough granular which shows the typical apatite appearance. The grain size ranges from 300 nm to 400 nm which can give a good impact on its application. These images were further processed for finding the exact size of HAp.

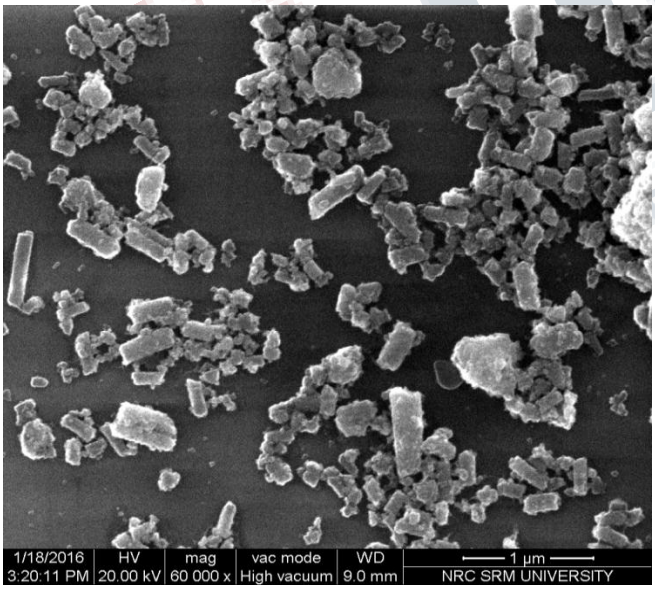

Fig 4: SEM micrograph of HAp of 60,000x

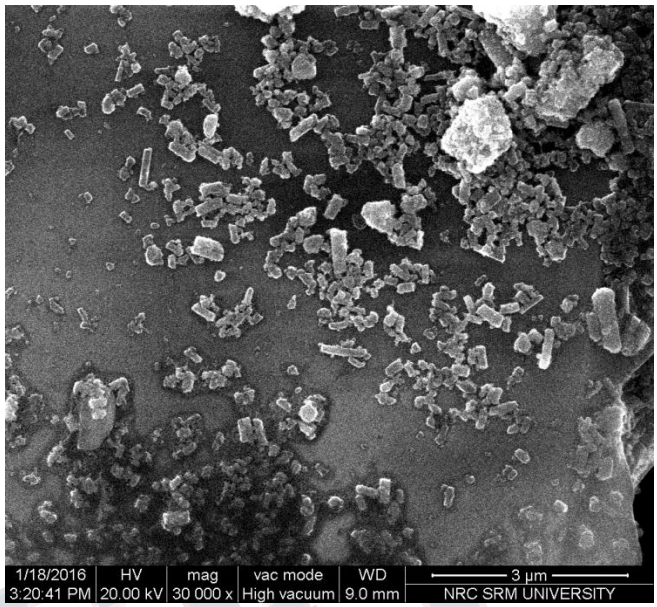

Fig 5: SEM Micrograph of HAp of 30,000 x

D.EDS

The elemental analysis or chemical characteristics of HAp could be obtained. The elemental composition of HAp can be obtained from the EDX analysis as Calcium, Phosphate and Oxygen. Calcium and Phosphate are the two basic elements present in HAp. Fig 6 confirms the presence of Calcium and Phosphate element. The amount of the element present is given as table in table 1.

Elements	Series	Weight
Oxygen	K-Series	14.57
Calcium	K-Series	1.43
Phosphate	K-Series	0.83

Table 1: HAp sample elemental presence

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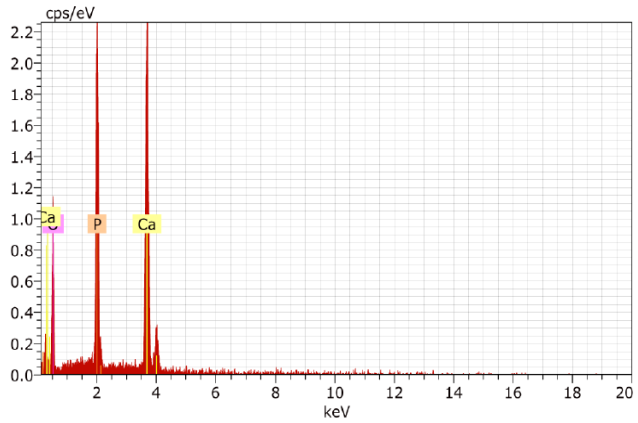

Fig 7: EDS Spectrum of Hap

IV. APPLICATION

Surface coating of HAp has very high research attention since they are attractive biomaterials which have a very good biocompatibility and nontoxicity. They also include applications such as periodontal defects, augmentation of alveolar bone, repair of large bone defects and tooth replacements. HAp is that which mimics the bone properties and hence can be used in various biomedical applications and the enhancement of mechanical properties extends its scope of application.

V. CONCLUSION

Synthesis of HAp was successful by using wet chemical method which is used due to its economical benefits and simplicity. Characterization was done using FTIR, XRD, SEM and EDS. The FTIR test results confirm the presence of functional group like Hydroxyl, carbonate and phosphate groups. The SEM image gives the particle size which varies from 300-400 nm range. This HAp can be use for surface coating of orthopedic and dental implants.

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