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Preparation and Characterization of Nano Size Hydroxyapatite Powder (n- HAp): Study of Swelling Behavior for its perspective use in various Biological Applications

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Abstract**—***Hydroxyapatite (n-HAp) is effectively used as a bioimplant material because it closely resembles bone apatite and exhibits good biocompatibility. The FT-IR studies of the powder showed that the powder contains carbonate phase similar to the natural HAp. The nano size hydroxyapatite powder (n-HAp) was prepared by wet chemical process. XRD measurements showed that n-HAp crystals contains with β-TCP (tricalcium phosphate) and calcium oxide as secondary phase. The morphological evaluation of the SEM images showed n-HAp nano powders were synthesized with 20–60 nm size.*

Keywards:— *Nano hydroxy apatite, FT-IR, XRD, TEM.*

1. INTRODUCTION

Hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$, nHAp) is an important inorganic biomaterial which n-HAp attracted the attention of researchers related to biomaterials field in recent years. It is also observed that dense sintered n-HAp is used in many bone replacement applications and is used for repairing bone defects in dental and orthopedic sites, immediate tooth replacement, augmentation of alveolar ridges, pulp capping material and maxillo facial reconstruction, etc [1].

The mineral component of bone is a form of calcium phosphate known as hydroxyapatite (HAp), with the chemical formula $Ca_{10}(PO_4)_6(OH)_2$ and the hexagonal crystalline structure. n-HAp accounts for about 65 wt % of bone and provides most of its strength and stiffness. n-HAp crystals in bone are generally in the form of needle-like crystals in the nanometer-sized range of 5-20 nm width by 60 nm length [2]. The synthesis of nano-structured n-HAp is of considerable interest due to its broad applications in orthopedic, dental, and drug delivery application [3-4]. Studies of n-HAp showed that a high surface energy, which may improve its mechanical properties and allow for a faster implant surface turnover [5].

In the biomedical field, the synthesis of Hydroxyapatite (n-HAp)/polymer composite materials is of great interest for the development of biomaterials especially suitable to repair the skeletal system [6,7]. It is considered as an appropriate reinforcement for organic polymers for the possible use as bone cements [8].

2. MATERIALS

Method:

Wet chemical precipitation method reported elsewhere [9] is generally performed in water by mixing solutions of calcium hydroxide and phosphoric acid as follows equation 1.

$$10Ca(OH)_2 + H_3PO_4 \rightarrow Ca10(PO_4)_6(OH)_2 + 18H_2O \quad \text{..... (1)}$$

3. CHARACTERIZATION

FTIR spectroscopy:

The presence of functional groups was confirmed by using Fourier transform infrared spectroscopy **(Thermo NICOLET 5700, FTIR).** The FT-IR spectra were obtained over the region 500-4000 cm^{-1} using KBr pellet technique.

X- ray diffraction:

The crystallographic phase of n-HAp was determined by X-ray diffractometer (XRD) using a **(Bruker D8 Advance, Germany)** diffractometer in reflection mode with Cu Kα (λ=1.5405 Å) radiation. The data were collected in the 2ºθ range from 15º to 80º with a scanning speed of 1.5º / min.

TGA (Thermogravimetric Analysis):

The inorganic contents of the HAp powder were determined by using thermogravimetry analysis, about 10mg of samples were heated at the heating rate of 20ºC/min and the measurements were recorded from 50ºCto 800ºC.

TEM (Transmission Electron Microscopy) :

Tem studies of the prepared HAp helps to elucidate the nano size of material.

Swelling Studies:

The dried n-HAp was allowed to swell in excess distilled water at 30 ° C. The weight of swollen n-HAp was measured for selected time intervals. Triplicate data were obtained for each measurement. Eq. 2 was used to calculate the mass swelling ratio based on dry weight.

$$Q = \frac{(Ws - Wd)}{Wd} \quad \text{.....(2)}$$

Where Ws is the weight of swollen HAp and Wd is the weight of dried powder. Results have been shown in flowing graph

4. RESULTS AND DISCUSSION

FT-IR Analysis:

The FT-IR spectra of n-HAp sample has been shown in Figure 1 From the graph it is indicated that there is a broad envelop between 3500 $cm^{-1}$and 2550 cm^{-1}. The O-H stretching bond has been shown at 3520 cm^{-1} in sample. The peak at 731 cm^{-1} also confirms the presence of hydroxyapatite powder [10, 11]. A weak band of CO_3^{2-} was detected in the region around 1567 cm^{-1} which confirms the synthesis of carbonated HAp. The peak at 976 cm^{-1} corresponds to symmetric stretching mode of $PO4^{3-}$ and the peak at 700°C indicates the bending mode of $PO4^{3-}$. The large separation of these bands indicates the presence of crystalline phase of n-HAp [12, 13].

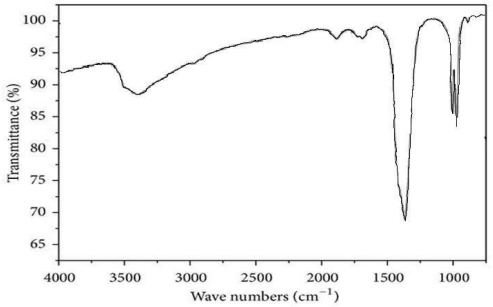

Figure 1: FT-IR results

X- ray Diffraction Analysis:

The X-ray diffraction (XRD) pattern of the final n-HAp was obtained with CuKα radiation (λ = 1·5418 Å) in the step scanning mode, with tube voltage of 40 kV and tube current of 50 mA. The XRD patterns were recorded in the 2θ range of 20–60°, with a step size of 0·02° and step duration of 0·5 s. The mean crystallite size (D) of the particles was calculated from the XRD line-broadening measurement from the Scherrer equation(3):

$$D = \frac{0.89\,\lambda}{\beta\,\cos\theta} \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(3)$$

Where λ is the wavelength (CuKα), β the full width at half-maximum of the n-HAp (211) line and θ the diffraction angle [14]. The mean crystallite size (D) was found to be 9.47 nm

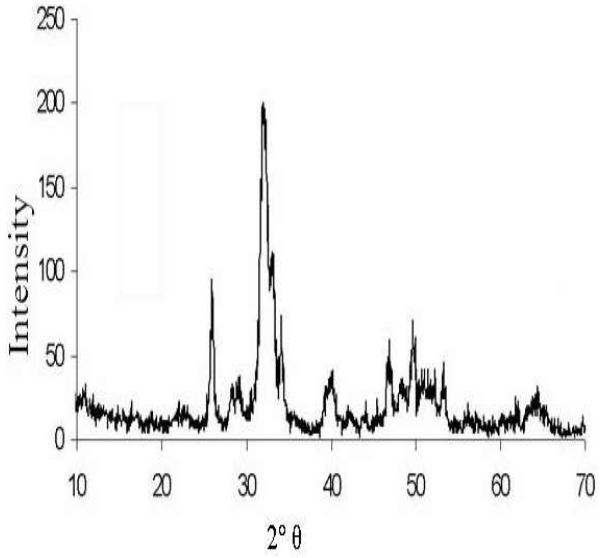

Figure 2: XRD results

TGA Analysis TG (Fig.3.) :

Analysis shows the n-HAp there is weight loss of around 12% up to temperature 220 °C and approximately 40% in the range 220°C to 350 °C. This major loss confirmed the formation of n-HAp, similarly about 5% wt. loss was observed up to 600 °C. Beyond 600°C to 1200 °C no significant wt. loss was observed. Almost stable curve was noticed within this temperature range, which indicates thermal stability of n-HAp powder.

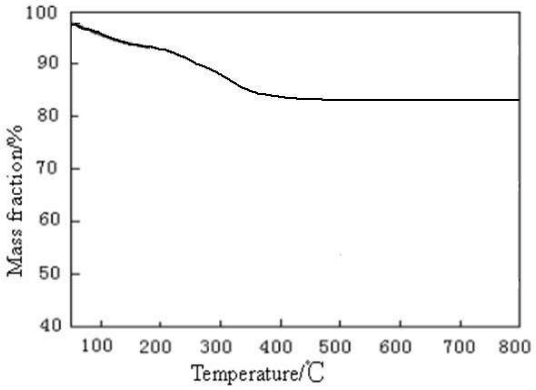

Figure 3: TGA results

TEM - Analysis:

In order to confirm the XRD results concerning the distribution of HA crystallites in the composite, TEM measurements were performed. The size of HAp was fund to be minimum 10 nm

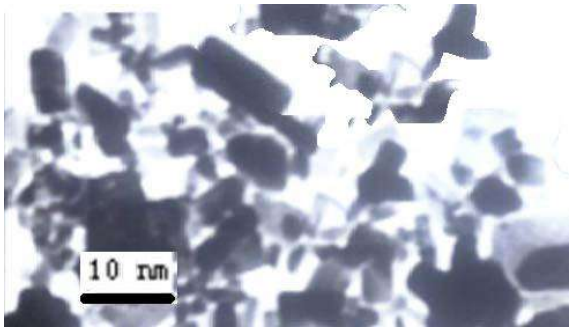

Figure 4: TEM results

Swelling Studies:

Swelling results of the prepared samples have been shown in the following graph that showed the fact that equilibrium swelling has been achieved for 60 minutes and then decreases. (Figure 5)

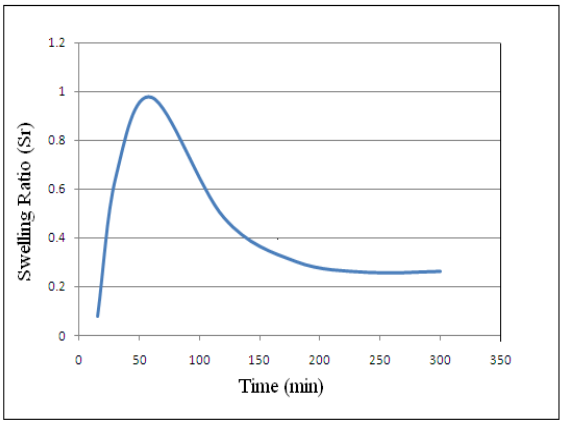

Figure 5: Swelling ratio

5. CONCLUSION

Although HAP is not yet commercially available as a competitive material with respect to other forms of n-HAp, nanosized n-HAp is currently used for several applications, which are either in advanced research states or undergoing development with considerable commercial opportunities. From their results, it was seen n-HAp both materials were suitable for bone replacement and for drug release such as antibiotics, growth factors or other substances. In addition, the organic component can be used to control physical properties in the bone implantation bed. Among other applications, the following can be considered.

- n-HAp coating based on sol-gel technology or electrodeposition allowing for the formation of thin adherent films, which do not severely affect the substrate morphology and topography.
- Composite preparations with other materials like chitosan collagen and other polymers able to reinforce the matrix while promoting osteoconduction; therefore, providing scaffolding properties required in tissue engineering applications.
- Nanosized n-HAp can be used in drug delivery systems like intestinal delivery of insulin or other drugs such as antibiotics.
- Further examples studied include its use in genetic therapy for certain types of tumors.

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