

Development of high strength bioceramic materials for potential biomedical applications

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ABSTRACT

Calcium phosphate ceramics are known for their biocompatibility and osteoconductivity. In particular, Hydroxyapatite (HAP) is considered as the best suited material for coating orthopaedic implants. has been widely studied as an important bioceramic material because of its chemical and biological similarity to inorganic component of bone and tooth, as well as good biocompatibility, osteoconductivity, and bone-bonding property. Owing to its bioactive, biodegradable and osteoconductive properties, HAP has been widely used in many biomedical applications. It was also proven that doping zinc into calcium phosphate structure gave better mechanical strength of calcium phosphate implants. In this research, zinc-doped hydroxyapatite powder was synthesized through sol-gel method. The obtained powders were physically characterized using FTIR, XRD, and SEM. It was shown that synthesized Zn-doped HA powders resulted in highly crystalline powders after calcination. The as-synthesized powders are to be tested further for their biocompatibility.

Keywords: hydroxyapatite, hydrothermal, sol-gel, biocompatible, orthopaedic.

1. INTRODUCTION

Calcium hydroxyapatite (HAP), $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$, which is commonly referred to as HA, is one of the best suited calcium phosphate based bioceramic material bones and teeth related replacement therapies. Synthetic HAP is known to be one of most important bioceramics material due to its biocompatibility, bioactivity and osteoconductivity [1-4]. Although these materials can closely replicate the structure of human bone the inherent brittleness and low tensile strength of HA samples confine their medical applications in the field of orthopedics and dentistry. The specific chemical, structural and morphological properties of HA bio ceramics are highly sensitive and changes with chemical composition and processing conditions [5-9]. One of the convenient ways to effect the morphological and physical strength of HA ceramics is the method of synthesis and recently sol-gel synthesis has recently attracted much attention, due to its many advantages, which include high product purity, homogeneous composition, and low synthesis temperature [10]. Additionally, incorporation of Zinc ions in to HAP structure is attempted since zinc is found in all tissues of the human body, and most of the zinc is stored in the skeleton. Moreover zinc intercalation of hydroxyapatite can promote the deposition of biological apatite and inhibit osteoclast bone resorption; and stimulate new bone formation. Therefore, the addition of zinc phosphate and hydroxyapatite are of great interest to many researchers [11-13]. Hence In this study, zinc-doped HA (ZHA) was synthesized by the sol-gel method. The objective of this research is to synthesize the monophasic Zn doped hydroxyapatite nanopowders with high crystallinity and to carry out the structural characterisation of it.

2. MATERIALS AND METHODS

2.1 CHEMICALS AND REAGENTS

$\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, $(\text{NH}_4)_2\text{HPO}_4$ and $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, Ethanol and Ethylene glycol were obtained from Merck, India. All the received chemicals were of analytical grade and were utilized without further purification. Deionized water was used throughout the synthetic process as a solvent to mix the salts as described in stoichiometric quantities.

2.2 SYNTHESIS OF PURE AND ZINC DOPED HA BY SOL-GEL METHOD

Pure and Zn-doped HA powder were synthesized under atmospheric conditions by a sol-gel method. 0.5 mol $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ and 0.3 mol $(\text{NH}_4)_2\text{HPO}_4$ and 0.1 mol $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ were dissolved into 50 ml of deionised water and Ethylene glycol (1:1 and 1:2). The phosphate solution and zinc was added drop wise to the calcium solution by a constant stirring. All reactions were kept in an oven at 80 °C for 6 h and then the pH was maintained between 9.5 and 10 using NaOH solution. The precipitates were filtered and washed three times by deionised water and ethanol (1:1). Then the precipitates were dried in an oven at 90 °C for 10 h. finally Zn-doped HA powder was calcined for 4 hrs. The powders obtained were further characterized with different analytical techniques.

3. RESULTS AND DISCUSSION

3.1 FTIR analysis of pure and Zn-doped HA powder

The FTIR spectrum (Fig.1) of the synthesized powders shows all characteristic peaks for HAP. The peak at 876 cm^{-1} can be assigned to ν_1 mode of phosphate group and the bands at 568 cm^{-1} and 603 cm^{-1} are due to ν_4 mode. The broad band around 3448 cm^{-1} is due to the adsorbed water, while the peak at 3570 cm^{-1} is due to the OH stretching. The ν_3 and ν_2 bands of phosphate groups occur at 1034 cm^{-1} and 463 cm^{-1} respectively. The substitution of zinc is evident from the peak observed at $1420 - 1440 \text{ cm}^{-1}$ spectral range [14].

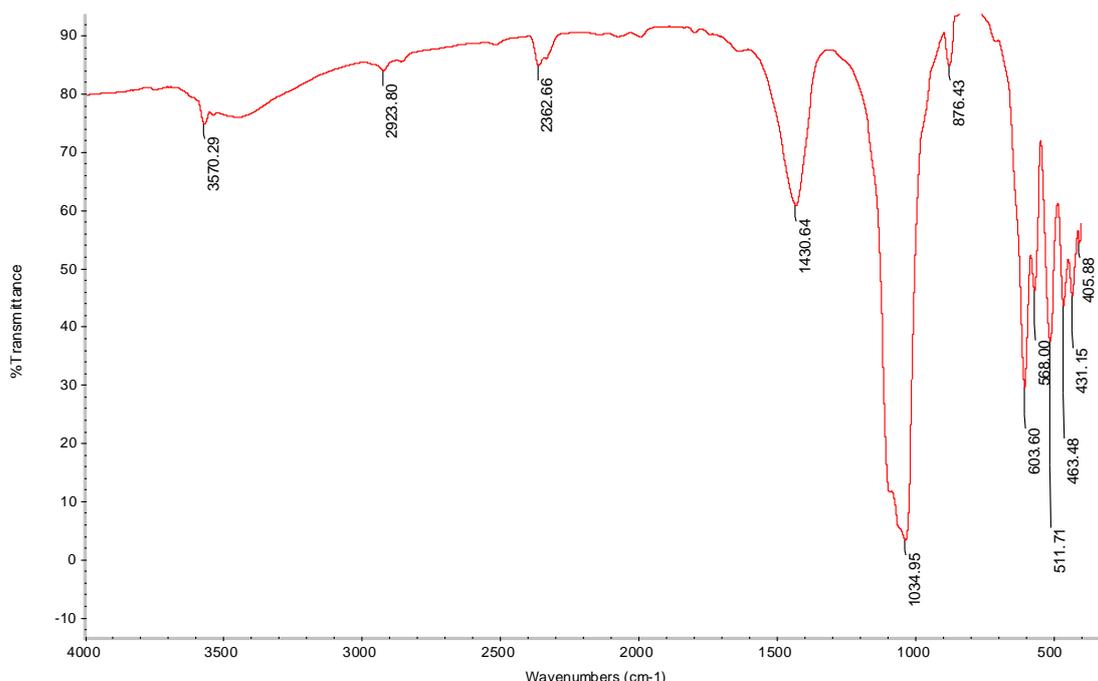


Fig. 1 FTIR spectrum Zn-doped HAP powders

3.2 XRD characterization of pure and Zn-doped HA powder

The powder diffraction and phase analyses were carried out by powder X-ray diffraction technique and a good correlation with stoichiometric hydroxyapatite (JCPDS No. 09-0432) was found (**Fig.2**). It was observed that there was a small shift to higher angles which may be due to zinc substitution [15].

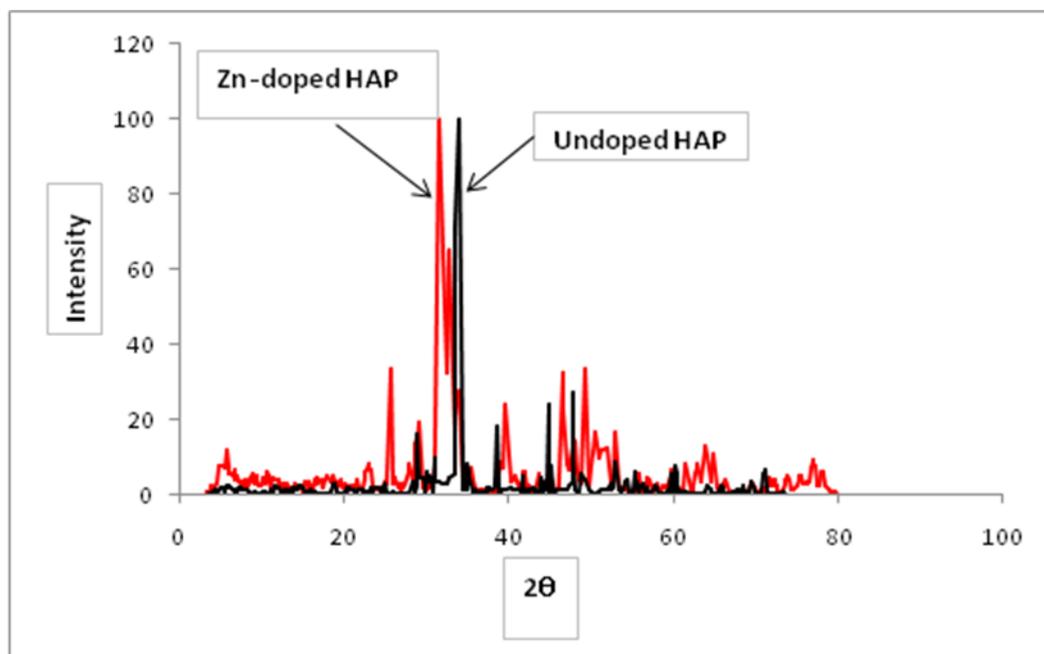


Fig. 2 XRD pattern of undoped and Zn-doped HAP powders

It may also be inferred that zinc affects the structure since it partially replaces calcium in the hydroxyapatite crystal and the results presented here are logical and in agreement with those previously reported [16], due to similar ionic radii of Zn^{2+} (0.074 nm) and Ca^{2+} (0.099 nm).

3.3 Scanning electron microscopic studies

The SEM images (**Fig. 3**) obtained for the Zn-doped HAP powders showed particles of elongated morphology.

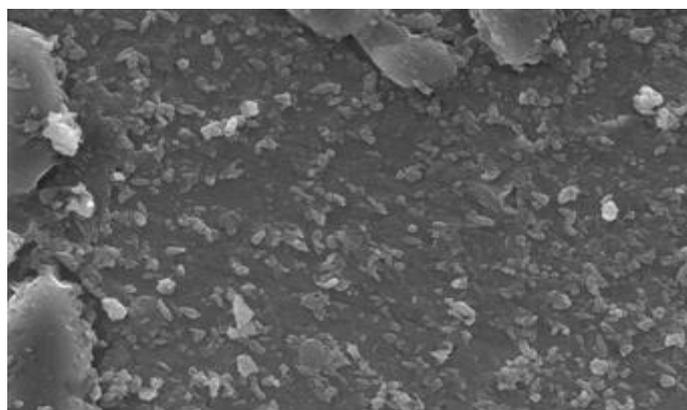


Fig. 3. SEM image Zn-doped HAP powders.

This elongation of particles are may be due to the high pH maintained during the reaction. Nevertheless the particles are of almost uniform size and morphology. EDXA analysis are further awaited which may reveal the extent of doping in HAP crystals.

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