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# MULTIFUNCTIONAL BEHAVIOUR OF MAGNESIUM/CALCIUM PHOSPHATE

**K. Thanigai Arul*[1], J. Ramana Ramya[2]**

[1]Department of Physics, AMET University, Kanathur, Chennai-603 112, Tamil Nadu, India
[2]Crystal Growth Centre, Anna University, Chennai- 600 025, Tamil Nadu, India

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## ABSTRACT

Pure and magnesium/nano hydroxyapatite (HAp) prepared by microwave route. The sample was analyzed by X-Ray Diffraction (XRD), photoluminescence (PL) and bioactivity. The XRD analysis was revealed that pure phase of HAp. As the concentration of magnesium ion increases, the average crystallite was reduced. The PL and *in vitro* bioactivity of the doped sample were enhanced. Hence, magnesium based calcium phosphate could be a suitable candidate for multifunctional applications.

**KEYWORDS**: Magnesium-Calcium Phosphate, Photoluminescence, Bioactivity

## I. INTRODUCTION

Calcium phosphate based bioceramics are promising synthetic material. Among these hydroxyapatite (HAp) has fascinated to researchers because of its resemblance of natural bone and teeth in their composition. It has good properties such as bioactivity, biocompatibility and osteoconductivity [1]. Calcium, phosphate and carbonate were the major bone minerals apart from major elements and other inorganic trace elements were also present in bone such as sodium, fluoride, chloride, magnesium, strontium, etc. [2]. To improve performance of HAp doping is necessary for replacing sublattice with various ions. Minor species which able to affect bone mineral characteristics, such as crystallinity, degradation behaviour, morphology stability, and mechanical properties of the HAp structure. The substitution of calcium by magnesium in HAp is limited could be due to large ionic size difference between $Mg^{2+}$ (Magnesium) and $Ca^{2+}$ (calcium) (0.28 Å) which causes strong distortions of the HAp lattice and decrease its crystallinity [3]. There are various methods used for preparation by mechanochemical-hydrothermal method [4], precipitation method [5], Sol-gel method [6] and microwave [7]. Among above mentioned techniques microwave which is a simple and versatile methods. It is efficient method for the preparation of nano HAp materials [7]. In current work, magnesium/hydroxyapatite was prepared and analyzed for physical and multifunctional behaviour.

## II MATERIALS AND METHODS

Nano HAp and Magnesium (Mg) doped hydroxyapatite were synthesized by microwave route method. Calcium nitrate tetrahydrate ($Ca(NO_3)_2.4H_2O$), diammonium hydrogen phosphate ($(NH_4)_2.HPO_4$) , ammonia solution and triple distilled water were used for the synthesis. 0.6 M diammonium hydrogen phosphate solution was prepared using triple distilled water as solvent with pH 10. 1.0 M calcium nitrate and 1.0 M calcium nitrate mixed with 0.07M $MgCl_2.6H_2O$ was added to diammonium hydrogen phosphate, were employed as pure and doped respectively. This mixture was continuously stirred for two hours and microwave irradiation was done on the samples for about 30 minutes. Then, the pure sample was denoted as HAp and Mg-doped sample with 0.07M are denoted by 1MgHAp respectively.

The as synthesized HAp and Mg-doped HAp were examined by XRD Bruker. The elemental chemical analysis was carried out using PHILIPS MiniPal PW 4025 energy dispersive X-ray spectrometer. Surface morphology of samples was characterized by SEM using JEOL model NO JSM-6360 instrument. Photoluminescence (PL) was done using Horiba Jobin Yvoun spectrofluorometer (at 325 nm) on the samples.

For the bioactivity test, Stimulated Body Fluid (SBF) was employed for sample analysis. It was prepared by dissolving NaCl, $NaHCO_3$, KCl, $K_2HPO_4.3H_2O$, $MgCl_2.6H_2O$, 1N HCl, $CaCl_2.2H_2O$, $Na_2SO_4$ and Tris in

deionized water and buffered to PH 7.4 at 37 ± 0.1 °C with HCl prepared based on the method of kokubo et al [8]. The SBF solution was similar to blood plasma. The 20ml of SBF was taken in an air tight plastic container and the pellets were immersed in it. It was stored at 37 ± 0.1 °C. The solution was renewed for every two days for three weeks.

## III RESULTS AND DISCUSSION

The XRD patterns of the pure and magnesium doped sample were in good agreement with the standard JCPDS value of hydroxyapatite (09-0432) as shown in the Fig.1 (a-b). The average crystallite size was found using a software MAUD (Material Analyzing Using Diffraction). The crystallite size of HAp was 35.02 (±2). As magnesium concentration increases, X-ray peak intensity was found to be decreased with increase in peak broadening which revealing a decrease in average crystallite size (19±1). The ionic radius of magnesium (0.65 Å) which was lesser than the calcium (0.99 Å) [9], but crystallite size was decreased may be due to occupancy of ions in interstitial sites in HAp.

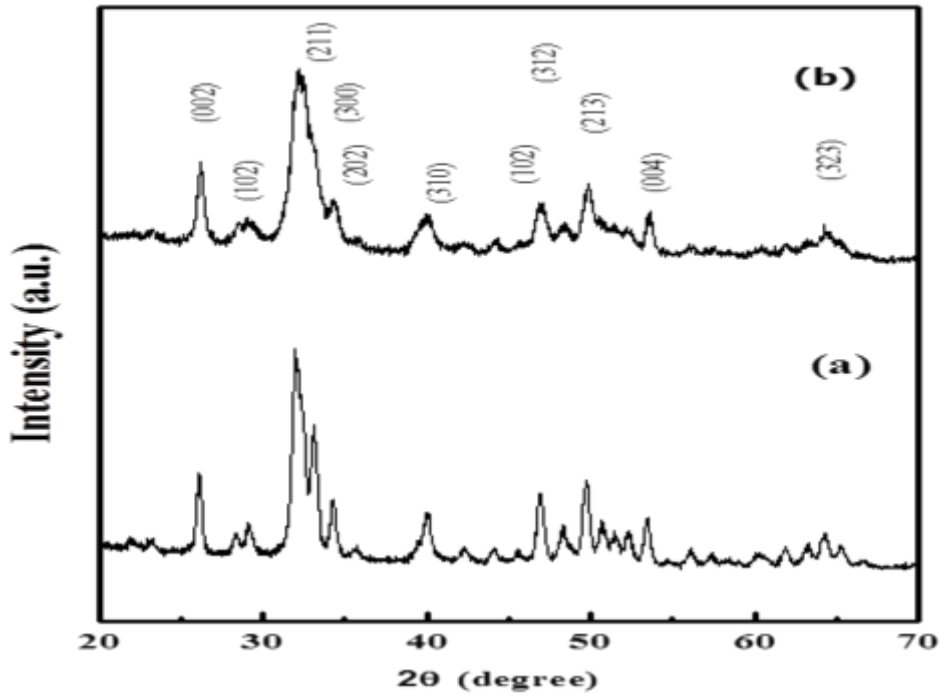


**Fig. 1** XRD patterns of a) HAp and b) 1Mg-HAp

PL studies of samples were shown in Fig.2. On magnesium ion doping in HAp, the PL intensity was enhanced could due to increase in number of defect's which able to increase recombination of electron/hole [10].

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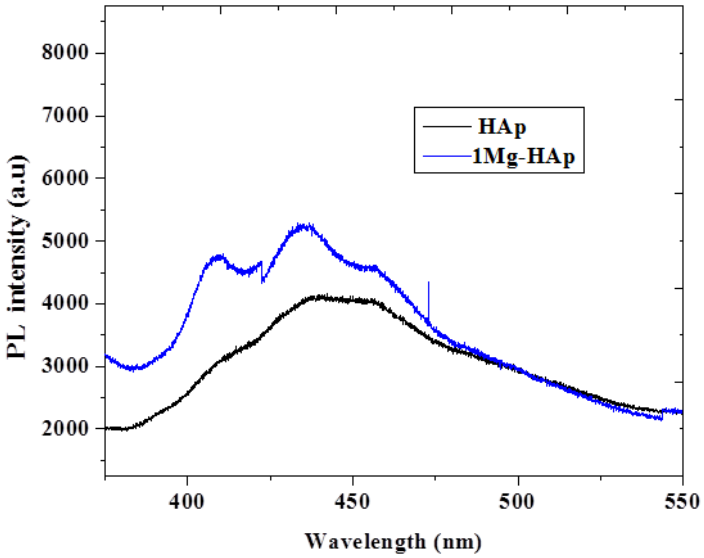


**Fig. 2** PL of HAp and 1Mg-HAp

SEM micrographs of (a) without SBF soaked HAp, (b) with SBF soaked HAp and (c) with SBF soaked 1MgHAp were as shown in Fig.3. HAp unsoaked sample revealed no apatite formation [Fig. 3(a)]. In magnesium ion doped sample, apatite formation on the surface was enhanced observed in Fig.3(c). It may be due to increase in surface charge and decrease in hydroxyl groups compared to soaked HAp sample.

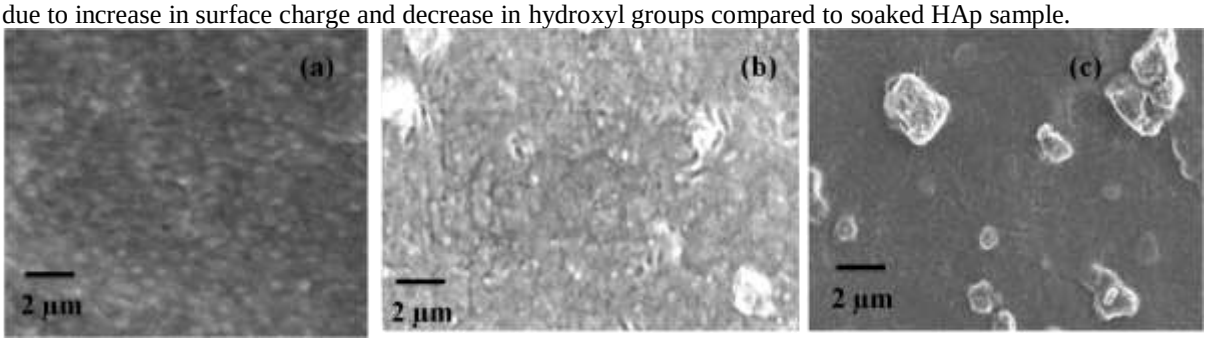


**Fig.3** SEM of (a) without SBF soaked HAp, (b) with SBF soaked HAp and (c) with SBF soaked 1MgHAp

## 1V CONCLUSION

Pure and magnesium-calcium phosphate were prepared by microwave route. XRD analysis revealed the decrease in crystallite size on magnesium doping. There was enhanced PL intensity and *in vitro* bioactivity attained on Mg-doped sample. In summary, magnesium doped sample could be an outstanding candidate for bone replacement applications.

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