

The Effect of Mg Supplement on Hydroxyapatite Produced by Chemical Precipitation

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Hydroxyapatite (HAp) exhibits excellent biocompatibility but the low mechanical strength of normal HAp ceramics generally restricts its use to low load-bearing applications. Magnesium is an attractive material for use in biodegradable implants due to its low density, non-toxicity and mechanical properties similar to those of human tissue such as bone. Its biocompatibility makes it amenable for use in a wide range of applications from bone to cardiovascular implants. The aim of this work was to produce hydroxyapatite powder with chemical precipitation method and supplement Mg and investigate the effect of magnesium on structure of HAp powders. The Mg content ranged between 1 and 2 mol.%. The Mg+HAp powders were examined for morphology. The scanning electron microscopy, X-ray diffraction, energy-dispersive X-ray spectroscopy were used to characterize the specimen powders.

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1. Introduction

HAp is the main mineral component of bones and teeth [1]. With the precipitation, sol-gel, hydrothermal synthesis methods, hydroxyapatite can be produced synthetically. However, synthetic hydroxyapatite has a similar composition to inorganic component of bone [2], natural hydroxyapatite has extra compounds like magnesium oxide, sodium oxide etc. Hydroxyapatite is used for dental and surgery applications with its high biocompatibility effects. Due to the poor mechanical properties of hydroxyapatite [3], it can be used with metallic-based implants like coating. Hydroxyapatite, on implantation, allows good bone ingrowth and eventually is replaced by endogenous bone [4]. Magnesium comprises 0.5% to 1% of bone ash and it is an important component of the hydroxyapatite lattice. Magnesium can also directly influence on hydroxyapatite crystal formation and growth [5]. Magnesium is an important bivalent ion associated to biological apatites [6].

2. Experimental procedures

Pure and Mg substituted HAp were produced with chemical precipitation method [7]. $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ (Merck, 98%), H_3PO_4 (Merck, 85%), $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (Merck, 99.99%) were used for Mg substituted HAp synthesis and NH_4OH were used for pH adjustment. $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, H_3PO_4 , $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ were dissolved in deionized water separately and NH_4OH was added to all of the solutions to increase the pH level to 10–11. Then magnesium nitrate solution was added into

calcium nitrate solution which was continuously stirred by magnetic stirrer. After a while later, finally, orthophosphoric acid solution was added slowly. Final pH level was measured as about 10. Liu pointed out that increasing pH brought about low crystallinity [8] which stands for nanostructure. With adding orthophosphoric acid solution, simultaneously, gelatinous solution was produced. After the solution was stirred for 24 h at room temperature, hydroxyapatite particles were precipitated for 24 h. Then the solution was filtered with adding deionized water to remove remaining residuals. Finally wet product was dried in the furnace at 105 °C for 24 h. After drying product was grinded with agate mortar.

For characterization, scanning electron microscopy (SEM) (TESCAN VEGA II) was performed to investigate the microstructure of powders, X-ray diffraction (XRD) (RIGAKU D/MAX/2200/PC) was performed to identify phases in the structure, energy dispersive X-ray spectroscopy (EDX) analyses (BRUKER Quantax) were performed for elemental analysis, particle size analyses (MICROTRAC S3500) were performed.

3. Results and discussions

SEM micrographs and EDX analyses results were shown in Fig. 1. It was obvious that particles had sharp-edged shape because of grinding with agate mortar.

With EDX analysis, it can be seen in Fig. 2 that there were Mg elements in 1, 2 mol.% Mg substituted HAp. Mg content was added successfully to HAp structure. The other elements were the same, calcium, phosphate and oxygen which were in HAp structure.

Chaudhry investigated that adding 1 to 2 wt% Mg contents to hydroxyapatite structure led to biphasic calcium-phosphate mixtures as hydroxyapatite-Mg whitlockite [9]. Similar situation was shown in Fig. 3.

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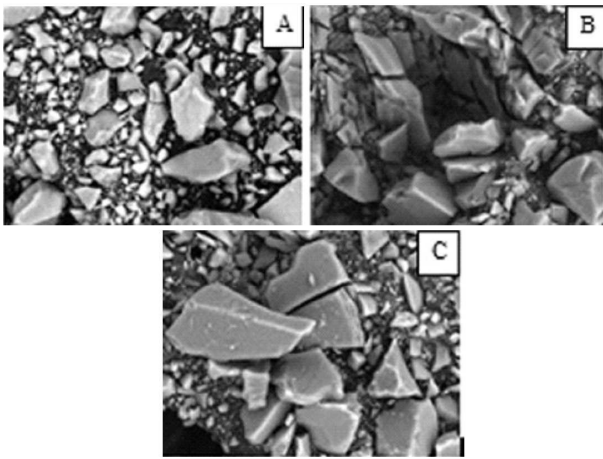


Fig. 1. SEM micrographs of hydroxyapatite particles: (a) pure HAp, (b) 1 mol.% Mg substituted HAp, (c) 2 mol.% Mg substituted HAp.

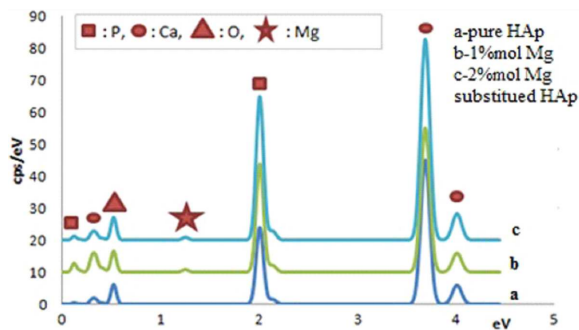


Fig. 2. EDX analysis results: (a) pure HAp, (b) 1 mol.% Mg substituted HAp, (c) 2 mol.% Mg substituted HAp.

It is shown in Fig. 4 that 1% Mg substituted HAp peak fit into 2% Mg substituted HAp peak. Particle size was connected with grinding. Whole particle size ranges between 1 to 700 μm .

4. Conclusion

In this study, the effect of magnesium supplement was investigated. High crystallinity was provided after sin-

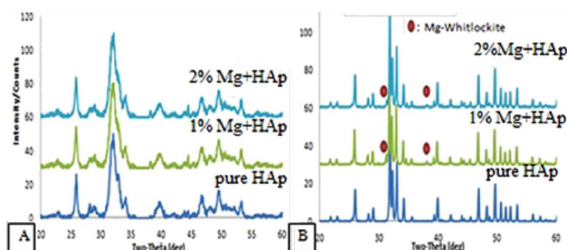


Fig. 3. XRD patterns: (A) before sintering, (B) after sintering at 900 °C/1 h at air atmosphere.

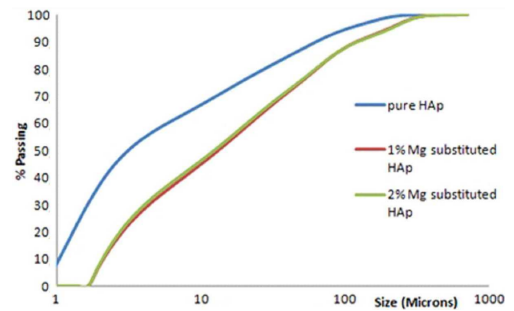


Fig. 4. The results of particle size analysis.

tering hydroxyapatite. It was shown that magnesium addition in hydroxyapatite led to biphasic mixture of hydroxyapatite and Mg-whitlockite in XRD results. Magnesium, silver, and some other cations can be added with chemical precipitation to hydroxyapatite structure easily. SEM results showed that microstructures were sharp-edged because of grinding.

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