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Original Research Paper

Effect of low temperature on formation mechanism of calcium phosphate nano powder via precipitation method

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ABSTRACT

Calcium phosphate powders were synthesized with Ca/P molar ratios of initial reagents ranging from 1.660 to 1.667 using wet precipitation method. This work deals with allocating a specific temperature level to each H_3PO_4 solution and $Ca(OH)_2$ suspension prior to the mixing process, and studying their influences on powder composition. A high pH value of the synthesis medium and the incorporation of numerous carbonate ions into the structure were attained by dropping the temperature of the $Ca(OH)_2$ suspension down to 5 °C. X-ray diffractometry and FTIR spectroscopy showed that heating samples that had a medium temperature exceeding 25 °C resulted in the dominant HAp phase, regardless of the initial acid solution temperature. By maintaining the medium temperature at 5 °C, a sudden formation of the β-TCP phase occurred after thermal treatment at 1300 °C, and this trend continued with the concurrent decrease in the temperature of the initial acid solution. An interpretation of the formation mechanism under these low-temperature conditions is proposed in terms of the temperature and pH value of the medium and the state of the phosphate and carbonate ions.

1. Introduction

Calcium phosphate (Ca–P) based ceramics found in the ternary system $CaO–P_2O_5–H_2O$, vary considerably in their phase composition [1–3]. Their synthetic products have received much attention as bone substitutes due to their chemical similarity to natural bone [4–7]. These ceramics are usually described by their Ca/P molar ratio [2,8].

Among these ceramics, two of the most important inorganic phases of synthetic bone applications-namely hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$, HAp, Ca/P = 1.667) and β-tricalcium phosphate (β-$Ca_3(PO_4)_2$, β-TCP, Ca/P = 1.5) have been widely applied in biomedical fields [2–4].

The literature on the elaboration and characterization of stoichiometric HAp (s-HAp) and β-TCP is considerable, whereas only a few works concerned with non-stoichiometric based compounds are available [9,10]. Only a few authors [11,12] have reported results on the synthesis of Ca-deficient apatite Ca-dHAp, $Ca_{10-x}(PO4)_{6-x}(HPO_4)_x(OH)_{2-x}$ [$0 \leqslant x \leqslant 2$]. Therefore, the characteristics of Ca-deficient apatites, when dissociated into a mixture of β-TCP and HAp of controlled ratios while being heated are the least studied [12]. The major reason for conducting these research is as a response to the demand for regulating the rate of biodegradation of β-TCP [10].

Several different synthesis techniques for producing gram quantities of these compounds have been developed in recent years. Most of these methods suffer from complex and time consuming procedures and the high cost of the raw materials [13,14].

The most common and widely used technique for the preparation of Ca-dHAp powders is still precipitation using a wet-chemical method in aqueous solutions, despite its shortcomings [8–10,12,13,15]. The advantages of this method include simple equipments and uniform, fine particles, and low cost of raw materials [16]. Raynaud et al. [2,9], Destainville et al. [8] and Descamps et al. [13] reported that light variations of the Ca/P molar ratio of the powder resulted in substantial changes in the powder composition and characteristics after thermal treatment, and the Ca/P ratio of the precipitate was not directly dependent on that of the initial reagents. The results of the researchers in the literature commonly contradict each other, and although each investigator obtained the required HAp/β-TCP ratios with the same precursors, their results showed unpredictable different initial Ca/P molar ratios at different calcination temperatures. This may be due to the small variations in the experimental conditions [12]. Finally, it was claimed that relative deviations of 1% or 2% in the Ca/P are generally considered acceptable [2], whereas a relative deviation of 1% can lead to the formation of 10 wt% of a second phase [8,13].

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