



Structural and mechanical characterization of boron doped biphasic calcium phosphate produced by wet chemical method and subsequent thermal treatment

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ABSTRACT

In the current study, boron doped biphasic calcium phosphate bioceramics consisting of a mixture of boron doped hydroxyapatite (BHA) and beta tricalcium phosphate (β -TCP) of varying BHA/ β -TCP ratios were obtained after sintering stage. The effects of varying boron contents and different sintering temperatures on the BHA/ β -TCP ratios and on the sinterability of the final products were investigated. Particle sizes and morphologies of the obtained precipitates were determined using SEM. XRD and FTIR investigation were conducted to detect the boron formation in the structure of HA and quantitative analysis was performed to determine the BHA/ β -TCP ratio before and after sintering stage. In order to determine the sinterability of the obtained powders, pellets were prepared and sintered; the rates of densification were calculated and obtained results were correlated by SEM images. Also Vickers microhardness values of the sintered samples were determined. The experimental results verified that boron doped hydroxyapatite powders were obtained after sintering stage and the structure consists of a mixture of BHA and β -TCP. As the boron content used in the precipitation stage increases, β -TCP content of the BHA/ β -TCP ratio increases but sinterability, density and microhardness deteriorate. As the sintering temperature increases, β -TCP content, density and microhardness of the samples increase and sinterability improves.

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1. Introduction

Hydroxyapatite (HA) and beta tricalcium phosphate (β -TCP) have been extensively used in medical and dental applications [1] since “HA exhibits strong affinity to host bone tissues because of its close similarity in chemical composition with natural bone and its high biocompatibility” [2,3] and “ β -TCP is proved to be resorbable in vivo with promoting bone growth to the implanted β -TCP” [2]. In spite of the stated advantages of HA and β -TCP, there are some limitations of these bioceramics. Low resorption of HA within the body obstructs the formation of new bones into the implant and bone remodeling; moreover since only a limited amount of HA is replaced with bone, local instability and stress concentrations arise [1,4]. On the other hand, absorption rate of β -TCP does not allow required time to bone ingrowth, resulting in insufficient bone replacement [1,5]; moreover β -TCP lacks osteogenic property [1, 6]. In order to overcome or decrease these types of deficiencies of single HA and single β -TCP, there is a growing interest in developing HA/ β -TCP biphasic calcium phosphate (BCP) ceramics [7].

BCP bioceramics, consisting of a mixture of HA and β -TCP of varying HA/ β -TCP ratios, are more effective in bone repair and bone

regeneration than pure HA or pure β -TCP [7], and have the benefits of combining the stability of HA with the reactivity of β -TCP [8]. The concept is to obtain a balance of the more stable phase of HA and more soluble phase of β -TCP [9] and to control the degradation rate to a certain degree [7,10]. This situation encourages the bone remodeling and new bone formation by allowing bone ingrowth as partial dissolution of BCP releases calcium and phosphate ions in the local environment [9,11]. In-vitro and in-vivo tests conducted on the BCP granulates with the specific content of 62% HA and 38% β -TCP indicated that BCP granulates have no cytotoxic effects on MG-63 cells, show good biocompatibility and provide a strong positive effect on bone regeneration [12].

These mentioned advantages of BCP can be improved by doping the material with foreign elements. Boron is one of the promising foreign elements which is used for doping because of its positive effect on bone formation [13–15] and its possible antibacterial effect [16,17]. Jain et al. [13] mentioned that boron has beneficial effect on bone cell differentiation when added to bioglass bone-replacement scaffolds. Hakki et al. [14] mentioned that molecular level B has an important role on bone metabolism and may find novel usage at the regenerative medicine; furthermore Gorustovich et al. [15] stated that boron addition to 45S5 bioactive glass has positive effect on enhancing bone formation. Xue et al. [16] and Wang et al. [17] mentioned that boron doping to titania enhanced antibacterial efficiency compared with non-doped titania.

Abbreviations: B, Boron; HA, hydroxyapatite; BHA, boron doped hydroxyapatite; β -TCP, beta tricalcium phosphate; BCP, biphasic calcium phosphate.

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In literature, there is limited number of studies on the subject of boron doping to HA. In the study of Ternane et al. [18] calcium borohydroxyapatite was obtained by the solid-state reaction of CaCO_3 , $(\text{NH}_4)_2\text{HPO}_4$ and H_3BO_3 and subsequent heat treatment. They mentioned that the ratio of P/B affects the final structure; when the ratio is equal to 7.22 boron atoms will totally be introduced into the matrix lattice, however if the ratio is greater than 11, $\text{Ca}(\text{OH})_2$ will be observed besides borohydroxyapatite and if the ratio is below 7.22, $\text{Ca}_3(\text{BO}_3)_2$ will also be observed [18]. Barheine et al. [19] prepared calcium borohydroxyapatite by the high temperature solid-state reaction of CaCO_3 , $\text{Ca}_2\text{P}_2\text{O}_7$ and B_2O_3 . They reported that besides the primary HA phase trace amounts of CaO and α -TCP were observed in the XRD spectra and BO_3 , BO_2 and B–O groups were observed in the FTIR spectra [19]. When boron is added to the structure, borate groups would partially replace the PO_4 and OH groups [18–21]. Guler et al. [22] obtained calcium borohydroxyapatite without any other phases by the solid-state reaction using the starting materials $\text{Ca}_2\text{B}_6\text{O}_{11} \cdot 5\text{H}_2\text{O}$ and $(\text{NH}_4)_2\text{HPO}_4$, and applying a sintering step at 1200°C for 12 h. Although they indicated that BO_3 would partially substitute the PO_4 groups, they did not observe any BO_2 groups in the FTIR spectrum [22]. Hayakawa et al. [23] synthesized boron-containing HA particles by using wet chemical method and subsequent heat treatment process. They stated that the chemical reaction between HA and $\text{B}(\text{OH})_3$ has taken place above 900°C [23].

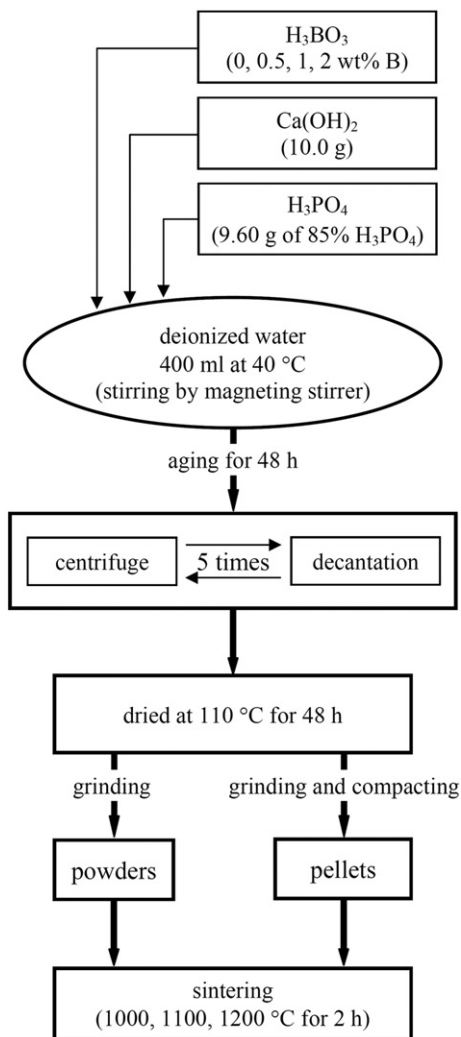


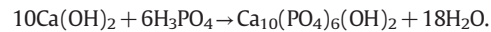
Fig. 1. Flowchart depicting the procedure to obtain undoped and boron doped BCP bioceramics.

To the best of knowledge of the author, the present work is the first study to obtain boron doped biphasic calcium phosphate bioceramics. In order to combine the advantages of boron and BCP, boron doped BCP bioceramics consisting of a mixture of boron doped hydroxyapatite (BHA) and TCP was produced in the present work. Boron doped BCP bioceramics with varying BHA/TCP ratios were obtained by using the wet precipitation method and then sintering consecutively. The effects of the boron contents used in the precipitation stage and the effects of sintering temperatures on the BHA/TCP ratios, densification, sinterability and microhardness of the final products were investigated.

2. Experimental

2.1. Production of powders and pellets

In this study, acid–base method was performed to synthesize non-doped HA powders since the reaction involves no foreign elements and the only by-product is water [2,24]. In this method, calcium hydroxide, $\text{Ca}(\text{OH})_2$, and phosphoric acid, H_3PO_4 were used as Ca and P source, respectively, based on the following reaction:



10.00 g of $\text{Ca}(\text{OH})_2$ powder (~99%, Merck, Germany) was dissolved in 400 ml of deionized water at 40°C using a magnetic stirrer for 30 min. 9.242 g of H_3PO_4 (85%, Merck, Germany) was added slowly at a rate of about 0.5 g/min to the continuously stirring $\text{Ca}(\text{OH})_2$ solution to obtain of the stoichiometric Ca/P ratio of 1.67. After H_3PO_4 addition, the reaction mixture was aged at 40°C for 48 h while stirring. Aged suspension

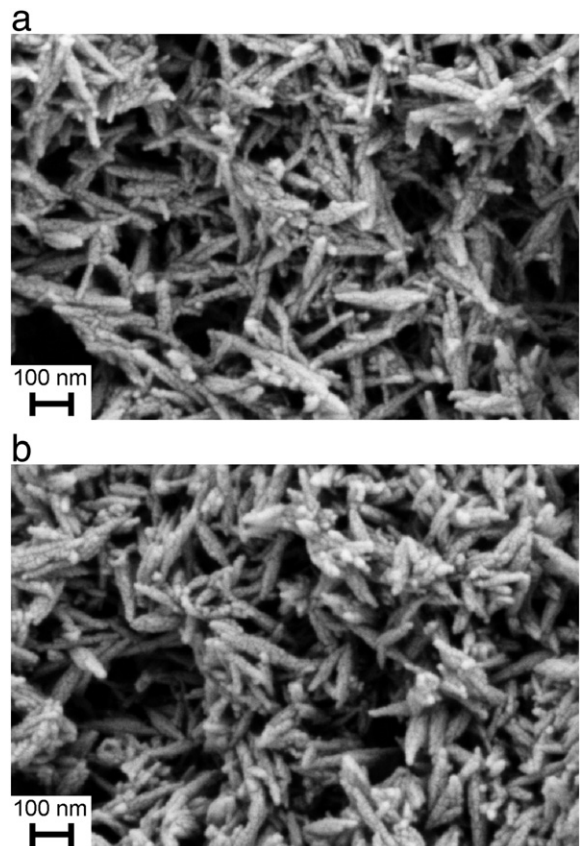


Fig. 2. FESEM micrographs (equal magnification) of synthesized powders at 40°C for 48 h: (a) control sample (0 wt.% B addition), (b) 0.5 wt.% B addition.

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