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Fabrication of Porous Calcium Phosphate Based Bioceramicsand Its Mechanical Properties

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----------ABSTRACT----------
In this study, porous calcium phosphate (CaP) based bioceramics in hydroxyapatite (HA) and tricalcium phosphate (TCP) form was fabricated for bone graft and porous orthopedic applications. Metal ion doped CaP based powders in HA structure were synthesized by wet chemical methods for pH5.5 and pH8.5. All shaped ceramics were sintered between at1100-1300°C for 1-3 h to possess porous structure. The microstructure and phase formation of the porous ceramics were characterized by X-ray diffraction (XRD) device and scanning electron microscopy (SEM). Depending on the sintering temperature and synthesized pH, the compressive strength of the porous ceramics was tested by the universal test machine. In conclusion, the HA structures were transformed to tricalcium phosphate (TCP) phase for pH5.5 at 1100°C. The synthesized powder for pH8.5 preserved its hydroxyapatite structure for all sintering temperatures. The highest compressive strengths of TCP and HA bioceramics for pH5.5 and pH8.5 were measured as 17.6 and 20.3 MPa at 1200°C and 1300°C, respectively.

KEYWORDS:*Calcium phosphate, Hydroxyapatite, Tricalcium phosphate, sintering, characterization, compressive strength*

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I. INTRODUCTION

Hydroxyapatite (HA) is a calcium phosphate similar to the human hard tissues in composition and morphology [1]. Particularly, it has a hexagonal structure [2, 3] and a stoichiometric Ca/P ratio of 1.67, which is identical to bone apatite [2, 4, 5]. An important property of HA is its stability when compared to other calcium phosphates. HA is the most stable calcium phosphate compound under physiological conditions such as pH, temperature, and composition of the body fluids [2]. HA often preferred because of its extraordinary properties such as bioactivity, biocompatibility, osteoconductivity, non-inflammatory, and non-toxicity nature [6]. HA bioceramics were used in bone void fillers for orthopedic, traumatology, bone tissue engineering, spine, orthopedic and dental implant coating, maxillofacial and dental surgery, and remineralizing agent in toothpaste.

With the development of nanotechnology, a significant impact on materials science has been noticed. The fabrication of nanomaterials has gained considerable attention for catalysis, adsorption, and optical applications, particularly when biomaterials are involved. Nano-hydroxyapatite (nano-HA) is attracting interest as a biomaterial for use in prosthetic applications because of its crystallography, the similarity in size, and chemical composition with human hard tissue. Teeth enamel and bone are largely composed of a form of this mineral [6-8].

The literature survey presents that the risk associated with an exposition to calcium phosphate in doses that are usually applied in health care products, biomedicine, and cosmetics is very low. Also, it also expresses that under all reasonable conditions, calcium phosphate particles can be considered safe for human bodies [9-12].

In this study, porous CaP based bioceramics in HA and TCP form were fabricated at the different sintering temperatures (T_S=1100, 1200, 1300 °C). The effects of sintering temperature and synthesized pH values (pH5.5 and pH8.5) on the porosity, compressive strength, and microstructure of bioceramics were investigated.

II. EXPERIMENTAL PROCEDURE

In this study, metal ion doped CaP-based powders in hydroxyapatite (HA) structure was synthesized by wet chemical methods for pH5.5 and pH8.5 (abbreviated as pH5.5 and pH8.5). Then, organic pore-forming agent (polystyrene-PS ball) and synthesized HA powders were homogeneously mixed in polyvinyl alcohol added water medium. The mixed materials have been transformed into the consistency of play dough. Produced

pasty materials were shaped by press using a cylindrical mold under low load. Ceramic beads were dried at 50 °C during 16 h and the samples were sintered between 1100 and 1300 °C for 1-3 h.

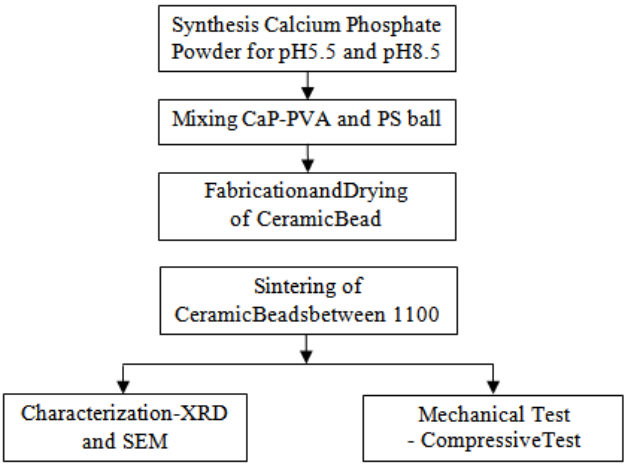

Fig. 1: Fabrication scheme of porous CaP based ceramics

The images of fabricated porous CaP based ceramics (TCP-pH5.5, HA-pH8.5) were shown in Fig. 2. As seen from the figure, the sizes of the sintered samples were decreased due to the sintering temperature. The microstructure and the phase formation of the porous ceramics were characterized by scanning electron microscopy (SEM) and X-ray diffraction (XRD) device, respectively. The compressive strength of the porous bioceramics was determined by the compressive test unit (GUNT WP300). By using this test machine (load capacity: 2 ton), the load was applied manually.

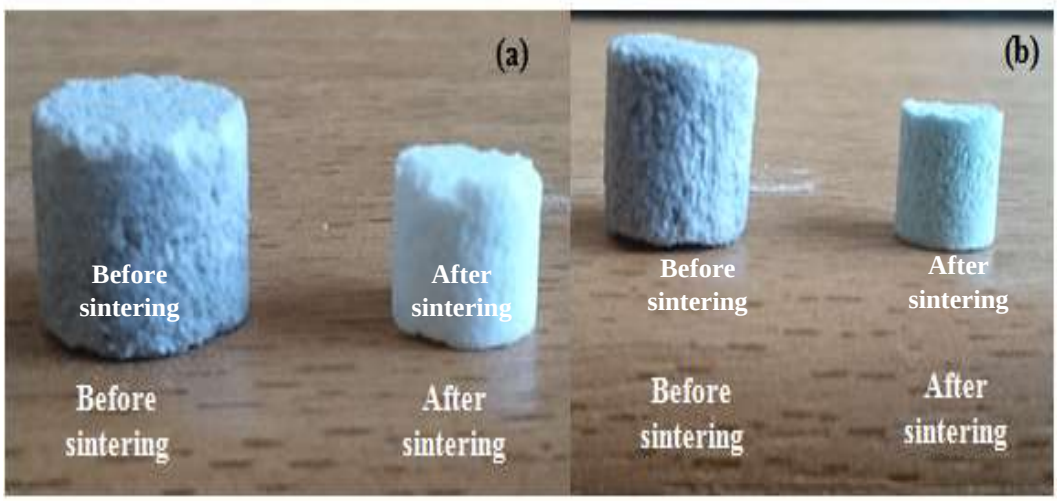

Fig. 2: Fabricated porous CaP based bioceramics: (a) TCP-pH5.5, (b) HA-pH8.5 (T_S=1300°C)

III. RESULTS AND DISCUSSION

The SEM image of CaP-based powders in hydroxyapatite (HA) structure for pH5.5 was presented in Fig. 3a. The image shows the uniform distribution of HA powders. The uniform distribution positively affects the mechanical properties of CaP based bioceramics. The average particle size of synthesized CaP-based powders in hydroxyapatite for pH5.5 is nearly 40 nm as shown in Fig 3b. From the results of the XRD analysis, it can be clearly seen that synthesized CaP based powders in hydroxyapatite form for pH5.5 transformed into tricalcium phosphate after heat treatment (Fig. 3c).

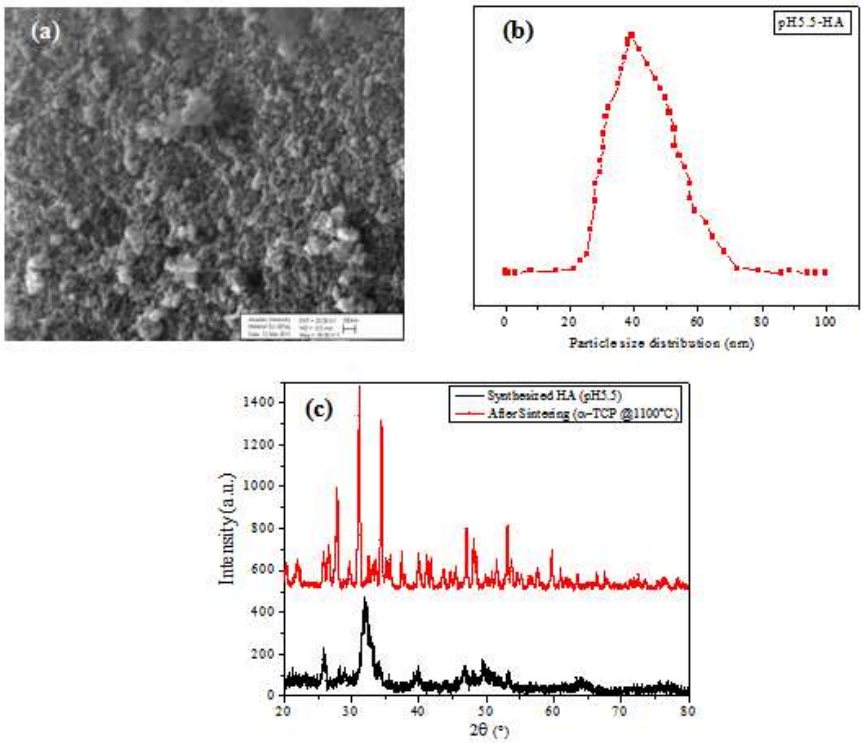

Fig. 3: SEM image of HA for pH5.5 powders (a), the particle size distribution of HA powders for pH5.5 (b), and X-ray diffraction plot of HA and TCP (c)

Fig. 4a gives the SEM image of CaP-based powders in hydroxyapatite (HA) structure for pH8.5. From the SEM image, the homogeny distribution of ceramic powders was observed. The mean particle size of synthesized CaP based powders in hydroxyapatite form for pH8.5 was determined as nearly 70 nm as given in Fig. 4b. It was seen that the particle size of synthesized HA at pH8.5 is bigger than synthesized HA at pH8.5 bioceramics. X-ray diffraction analysis test results show that CaP based powders in hydroxyapatite form for pH5.5 preserve its structure as HA. Also, the crystal phase of HA is seen more clearly due to the increased crystallinity of the powder with sintering temperature.

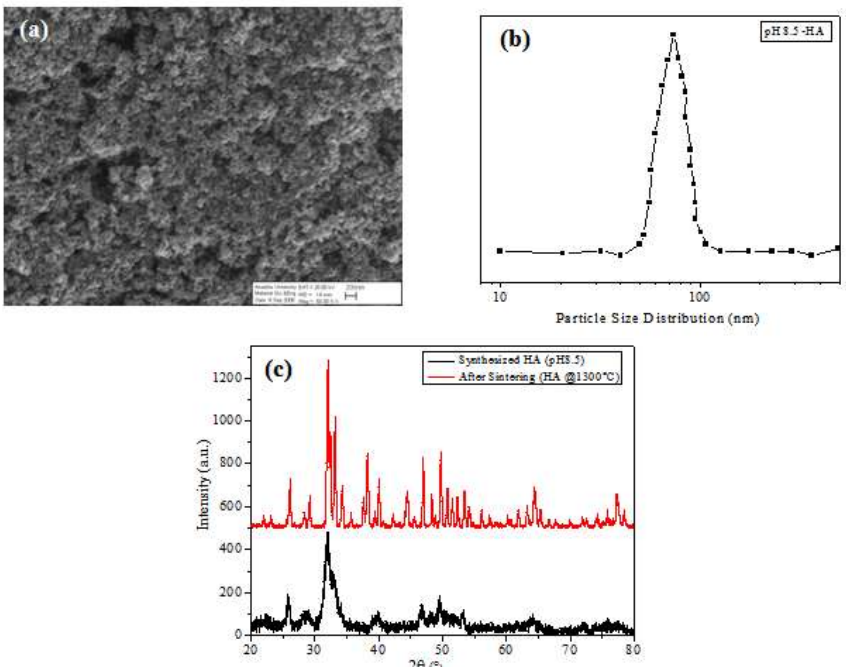

Fig. 4: SEM image of HA powders at pH8.5 (a), the particle size distribution of HA powders at pH8.5 (b), and X-ray diffraction plots of HA and TCP after synthesized and heat treatment (c)

The variation of %porosity with sintering temperature for porous CaP based bioceramics at pH5.5 and pH8.5 were shown in Fig. 5a. The lowest porosity (13±0.9%) value was obtained at HA for pH8.5 bioceramics and T_S=1300 °C sintering temperature. The increase in pH and sintering temperature decreased the porosity of bioceramics. Similarly, the compressive test results show that the compressive strength increased with sintering temperature for HA at pH8.5 bioceramics (Fig. 5b). The maximum compressive strength was determined as 20.4±0.7 MPa at T_S=1300 °C for HA at pH8.5 bioceramics. In summary, increase in sintering temperature increases the mechanical properties of CAP-based HA bioceramics.

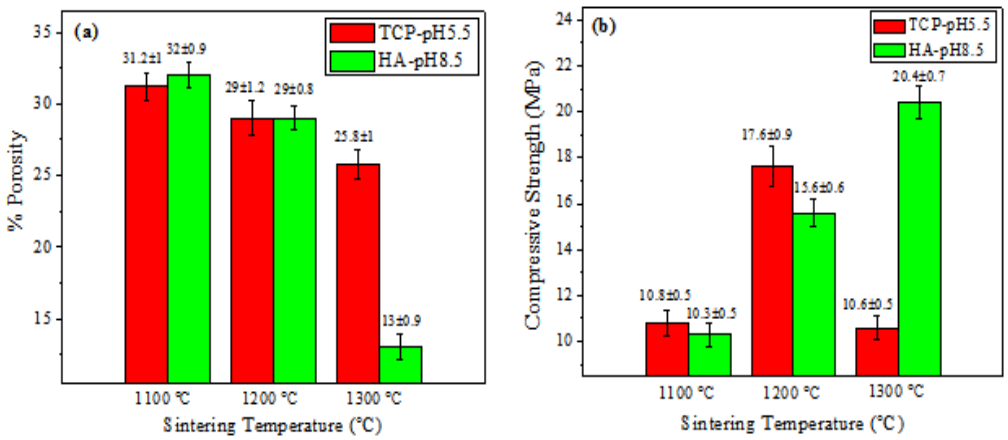

Fig. 5: The variation of porosity and compressive strength with sintering temperature for porous CaP based HA bioceramics

Fig. 6 gives the scanning electron microscope images of porous CaP based bioceramics (TCP-pH5.5, HA-pH8.5). Weak particle bonding and large porosity in macroscopic pores were observed at TCP for pH5.5 bioceramics (Fig. 6a). Also, dense microstructure and small porosity in macroscopic pores were detected at HA for pH8.5 bioceramics (Fig. 6b). It was concluded that SEM analysis results are suitable with the compressive test results.

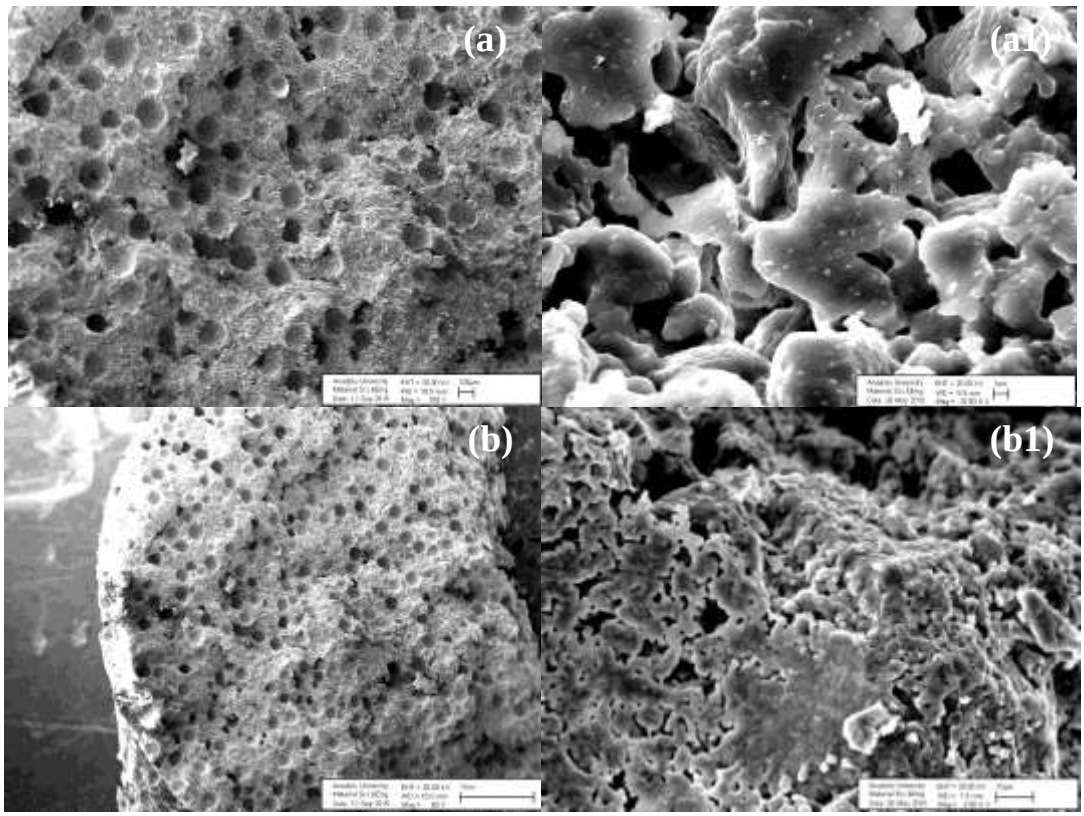

Fig. 6: SEM images of porous CaP based bioceramics: (a, a1) TCP-pH5.5, (b, b1) HA-pH8.5

IV. CONCLUSIONS

In this study, metal ion doped CaP based powders in hydroxyapatite (HA) structure was synthesized by wet chemical methods for pH5.5 and pH8.5. The effects of sintering temperature on the porosity, compressive strength, and microstructure of TCP for pH5.5 and HA for pH8.5 bioceramics were investigated. The conclusions were summarized as below.

- After ball milling, the uniform distribution was detected at metal ion doped HA powders. This uniform distribution affected the mechanical properties of bioceramics positively.
- The minimum porosity (13±0.9%) and the maximum compressive strength (20.4±0.7 MPa) was determined at HA for pH8.5 bioceramics and T_S=1300 °C sintering temperature.
- From SEM analysis, the denser microstructure was detected at HA for pH8.5 bioceramics. Among fabricated bioceramics, the high porosity was observed atTCP for pH5.5 bioceramics. It was concluded that an increase in sintering temperature decreased the porosity and increased the mechanical properties of HA bioceramics.

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