

**CHAPTER 10**  
**THE FABRICATION OF NANOPOROUS HYDROXYAPATITE**  
**CERAMICS**

**Overview** - Nanoporous hydroxyapatite (HA) ceramic were fabricated using polyvinyl alcohol (PVA) as pore former and vibro-milling method for making nanocomposite powder. The mixing powder was uniaxial pressed in stainless steel mold. The product was then sintered at 1200°C for 3 h with heating rate of 4°C/min. The average porosity of final products is 64.6±1.4% and the main morphology are existence of open and interconnected pores with average pore size less than 100 nm. The bending strength of 14.7±3.2 MPa was obtained offering with high potential for bone repair. It is also believed that the received nanopores in the prepared ceramic may enhance the efficiency of the controlled drug delivery devices.

### **10.1 Introduction**

In recent years, nanomaterials have received much attention from various researchers to the development of modern health care industry and have improved the quality of human life. Prostheses and implants are being developed from nanostructured materials with the unique properties that can lead to novel tissue engineering. Hydroxyapatite (HA) ceramics have been used as tissue engineering materials for cell culture and tissue due to the excellent biocompatibility and bioactivity with the human body<sup>47</sup>. In addition, HA can be use of controlled drug

delivery systems, such as in the delivery of anti-tumor agent and antibodies for the treatment of osteomyelitis<sup>48</sup>. The control of three-dimensional (3-D) pore structure is of great importance for the development of HA materials which enhance the possibility of tissue in-growth of natural bone, and this offer greater drug-loaded regions and larger surface to volume ratio<sup>47-48</sup>.

In this work, we have fabricated the nanoporous HA ceramics by using simple method that can made many products for large scale application, the use of polyvinyl alcohol (PVA) as pore former and vibro-milling method to making HA nanocomposite powder before sintering has not to our knowledge been previously demonstrated.

## 10.2 Materials and methods

The hydroxyapatite powder was derived from extracted deproteinized bovine bone with calcinations at 800°C for 3 h. After that it was crushed to small pieces and milled in a ball mill pot for 24 h and then sieved to average particle size less than 45 µm. The composite was obtained by mixed PVA powder with hydroxyapatite powder and ball milled in ethanol for 24 h, dried and sieved. Nanosized HA nanocomposite particles were prepared by vibro-milling method for 4 h. The powder was uniaxial pressed in stainless steel mold. The product was sintered at 1200°C for 3 h with heating rate of 4°C/minute. The thermal stability was characterized by X-ray diffraction (XRD). The nanoporous structure was examined using scanning electron microscopy (SEM). The physical properties of porosity and bending strength were investigated by the Archimedes' method and ball-on-ring test<sup>34</sup>, respectively.

### 10.3 Results and Discussions

Fig. 10.1 shows XRD pattern of the resulting the sintered porous HA ceramic corresponded to that of pure hydroxyapatite phase of JCPDS File No.9432. It is indicating that the sintered porous HA has no decomposition to another phase. The SEM images of resulting HA composite powder in Fig. 10.2 clearly showed the nano-rod shape of HA and PVA granules were well dispersed with a diameter of less than 100 nm. Therefore, vibro-milling is considered to be a successful method for producing HA nanocomposites powder for use as raw materials to fabricate nanoporous with all about holes diameters less than 100 nm.

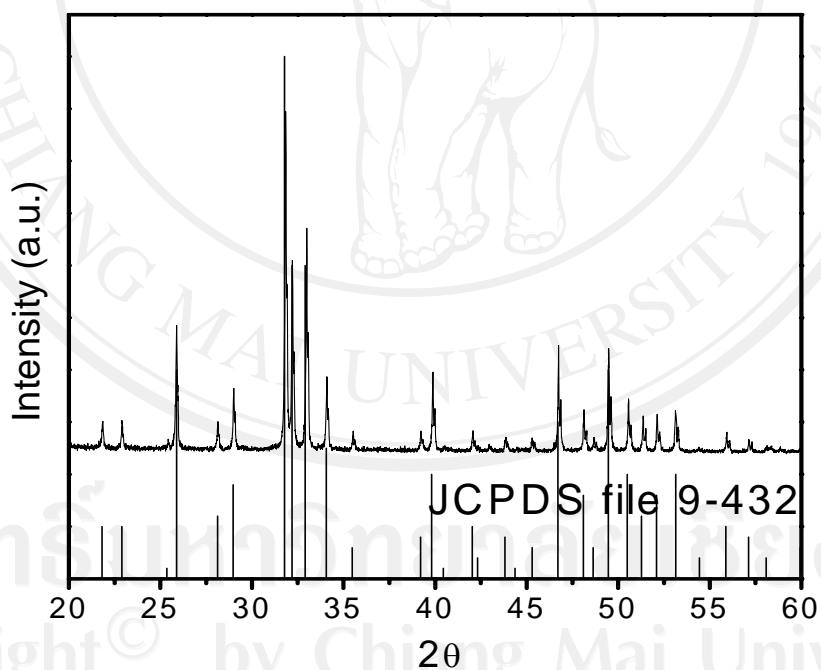


Fig. 10.1 XRD patterns of sintered samples at 1200°C.

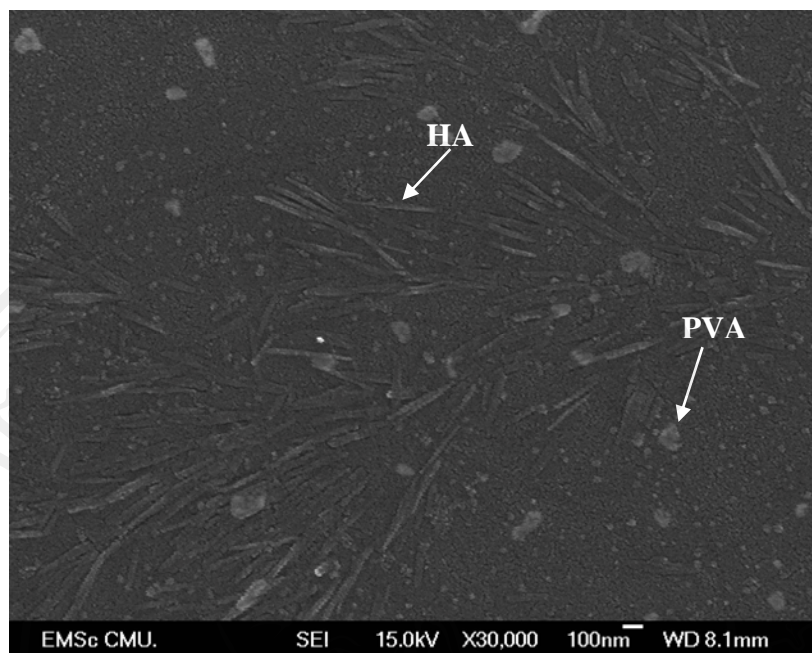
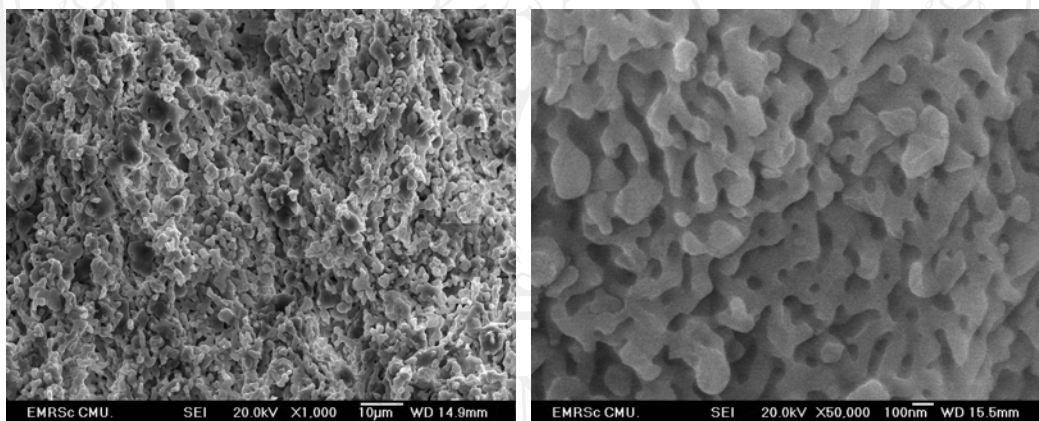


Fig. 10.2 SEM micrograph of HA-PVA nanocomposite powder.

The fracture surfaces of the sintered porous HA ceramics at 1200°C in Fig. 10.3 demonstrated that the final products had high porosity of  $64.6 \pm 1.4\%$  and the main morphology was existence of open with interconnected pores averaging less than 100 nm in pore size. It has been reported that nanoporous structure of the biomaterials tremendously enhance the cells adhesion, proliferation and differentiation required for tissue functions<sup>7</sup>. Therefore, it can be expected that nanoporous HA ceramic would have the capability to support the bone tissue in-growth upon implantation. Moreover, the mechanical tests revealed a bending strength of  $14.7 \pm 3.2$  MPa which is offering high potential for bone repair. This mechanical property allow the easy handing and shaping of porous sample for implantation as well as load bearing after surgery, which are often problematic<sup>17</sup>. Fig. 10.3(b) shows the struts between the nanoporous appear strong, consolidated and the nanoporous walls were characterized by liquid phase sintering. It is indicating that the

nanostructure of started HA powder improved densification of HA porous samples, which could improve mechanical properties. Further, the porous architecture with nanopores could enhance the efficacy of HA materials scaffold for potential use in tissue engineering applications. It is also worth mentioning that the nanostructure features of HA promotes osteoblast adhesion, differentiation and proliferation, osteointegration, and the deposition of calcium containing minerals on its surface, which leads to enhanced formation of new bone tissue within a short period<sup>2-3</sup>. Thereby the processed nanoporous HA ceramic may be highly beneficial for bone repair and augmentation to controlled drug delivery device.



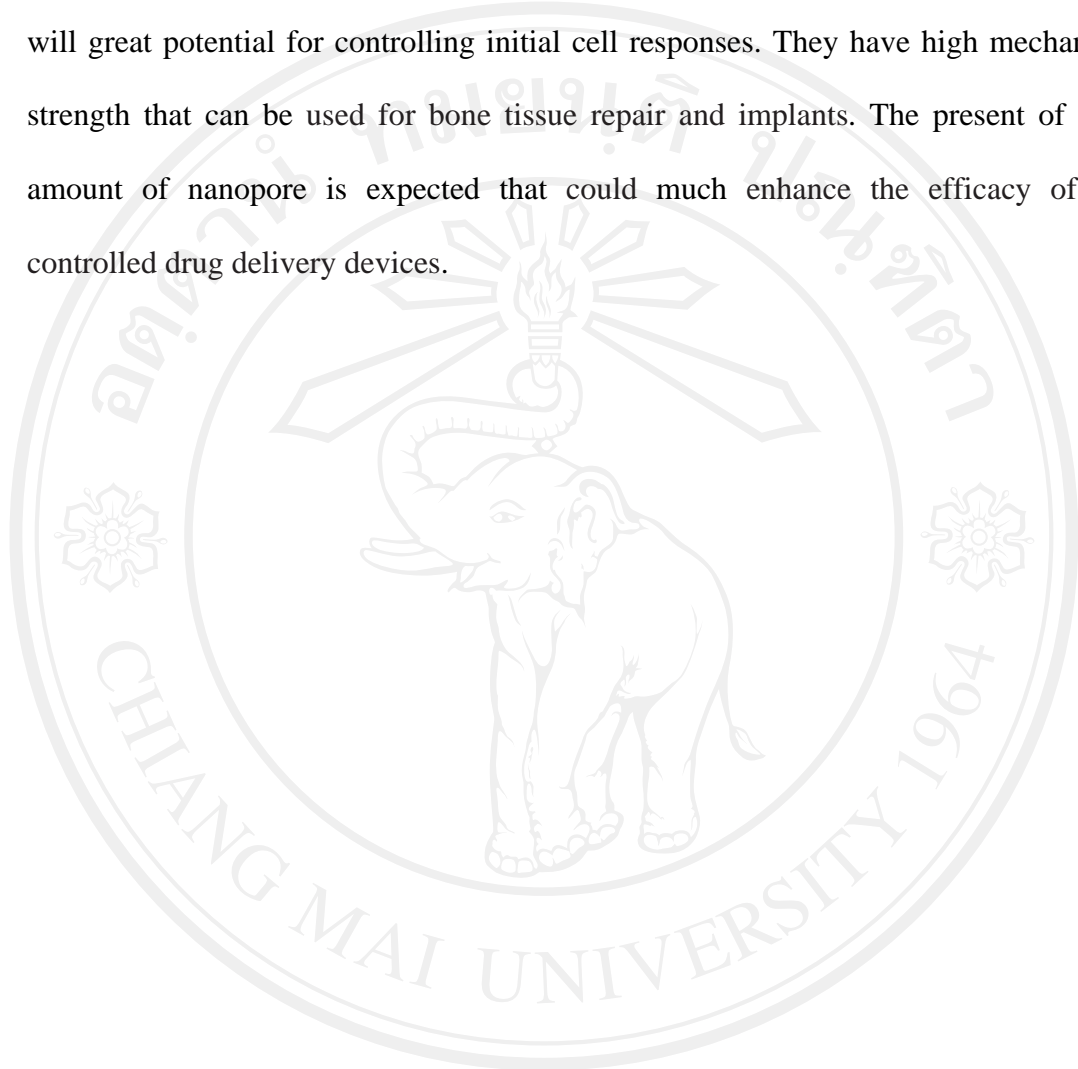
(a)

(b)

Fig. 10.3 SEM micrograph of fracture surface of nanoporous HA ceramics: (a) low magnification and (b) high magnification.

#### 10.4 Conclusions

The porous HA ceramics have nanoscopic interconnecting porosity, which will great potential for controlling initial cell responses. They have high mechanical strength that can be used for bone tissue repair and implants. The present of high amount of nanopore is expected that could much enhance the efficacy of the controlled drug delivery devices.



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