

SYNTHESIS, CHARACTERIZATION AND MECHANICAL PROPERTIES OF POROUS HYDROXIAPATITE

Altair Contreras, Gema González, Javier Ochoa, Rafael Villalba

Instituto Venezolano de Investigaciones Científicas. Departamento de Ingeniería. Laboratorio de Ciencia e Ingeniería de Materiales. Altos de Pipe. Venezuela

The use of biomaterials has shown a large development in the last years, specially for bone implants biologically similar to natural bone. On this group of materials Hydroxyapatite (Hap) is considered highly compatible due to its chemical and structural similarity to the mineral part of natural bone. The main applications are in facial traumas, as a filler of osteolytic zones generated by tumors and also as agents of slow liberation of medicaments.^(1,2)

The objective of this study was the synthesis and consolidation of porous HAp by sintering at different temperatures (900, 1000, 1100, 1150, 1200, 1250 and 1300°C) and times (2, 5, 10, 15 and 20 h). For the control of porosity mixtures of HAp and Polyvinil alcohol (PVA), in volume proportions of 25%, 50% and 75% were prepared.

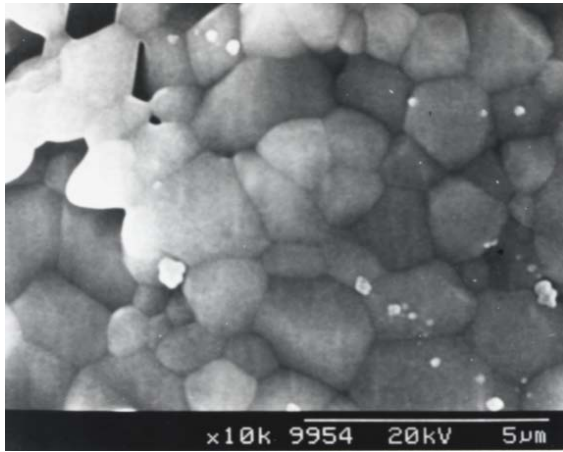
The structural characterization was carried out by x-ray diffraction (XDR) and infrared spectroscopy (IR) while the morphological characterization was done by Scanning electron microscopy (SEM). Porosity and density were measured by the liquid immersion method.

The structural characterization by XDR and IR showed the stability of HAp with the treatments, only a small transformation above 1200 °C to some phosphates groups (C₃P y C₄P) was observed by the presence of very small peaks in the XRD patterns. Samples that show the best consolidation results and a porous interconnected structure were obtained for temperatures in the range of 1150°C-1300°C and times between 5 and 20 h and HAp. High concentrations of PVA (75%) show detrimental mechanical properties due to insufficient sintering. Therefore, the recommended PVA concentration is in the range of 75% HAp- 25% PVA and 50% HAp-50% PVA. For these samples the mechanical properties were in the range of 1,20 to 7 MPa and 0,44 to 3,5 MPa.

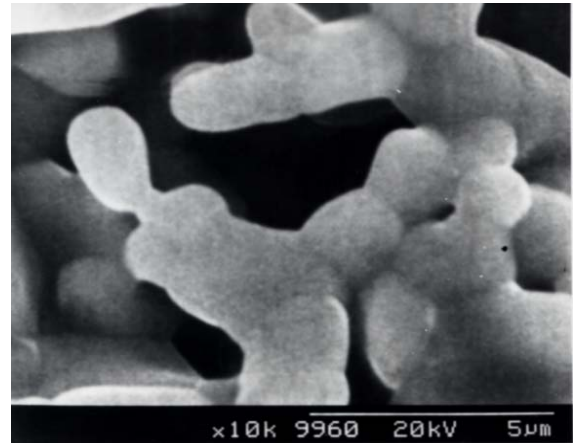
A structure of interconnected porosity with the presence of macropores of 100 µm diameter and micropores between 0.30 and 1.4 µm for 5 and 20 h of sintering was obtained. The porosity was between 20 and 75% for the lower and higher PVA content, respectively.

Fig. 1 shows the SEM micrographs of the different samples obtained varying %PVA for 1250°C and 5h sintering conditions. The effect of PVA in the structure is clearly observed. The highest the concentration, the more open the pore structure. Fig. 2 shows the effect of sintering temperature and time on the microstructure. Grain growth is remarkable at this conditions and the presence of a new morphology showing steps and terraces is observed. This is attributed to the phosphates transformation found by XDR and IR.

From the results of chemical composition, microstructure, porosity and compression strength is shown that the materials have similar properties to the trabecular bone. Therefore, these materials can be recommended for possible applications as bone graphs or implants.



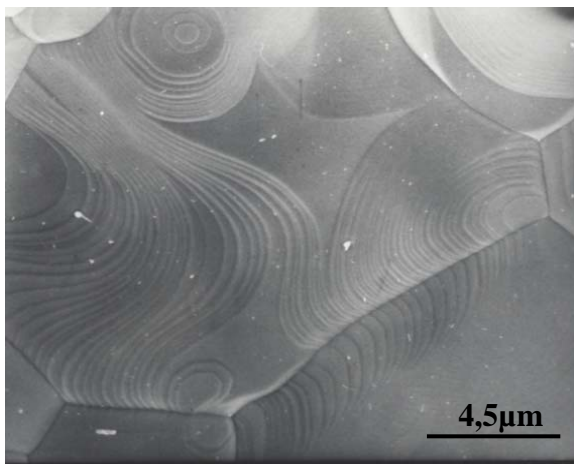
(a)



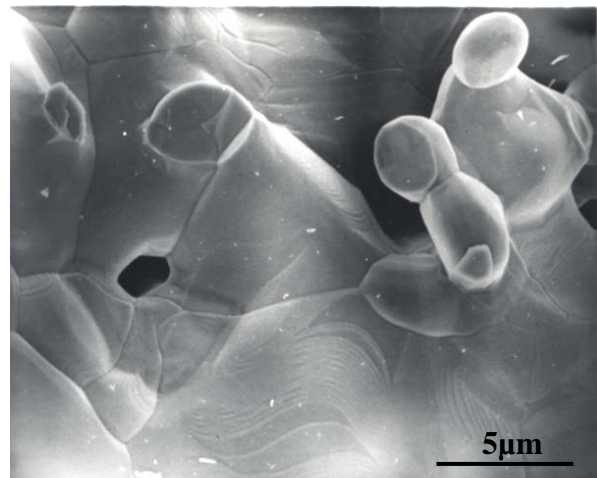
(b)

Fig 1. Photomicrographs of samples with different PVA content, sintered at 1250°C for 5 h

- a) 75% HAp-50% PVA
- b) 50% HAp-50% PVA



(a)



(b)

Fig 2. Photomicrographs of samples with different PVA content, sintered at 1300°C for 20 h

- a) 75% HAp-50% PVA
- b) 50% HAp-50% PVA

References

1. HING, K. et al. Characterization of Porous Hydroxyapatite. J. Mater. Sci.10: 135-145 (1999).