Synthesis, sintering and characteristics of hydroxyapatite nanopowders

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INTRODUCTION: Calcium phosphates, including hydroxyapatite (HAP) have been paid attention of intensive research as bone filler, bone graft substitutes do to its biocompatibility and osteo-conduction. The main results of investigations of HAP materials performed in frames of EUREKA project E!3033 BIONANOCOMPOSIT (in period of 2003-2012) are shortly summarised in this report.

METHODS: The chemical interaction between calcium hydroxide and phosphoric acid was chosen for synthesis of the HAP nanopowder. Various methods for manufacturing dense HAP materials from obtained powders were used and compared (Table 1). Biocompatibility was compared by enumeration of the number of osteoblast-like cells to the materials.

RESULTS: The TEM image (Fig. 1) of the synthesized HAP nanopowder shows the agglomerate which consist of nanosized rod-shaped particles in size of 50–100 nm. The powder obtained by spray-drying consisted from quasi spherical granules with average diameter of 20-40 μm.

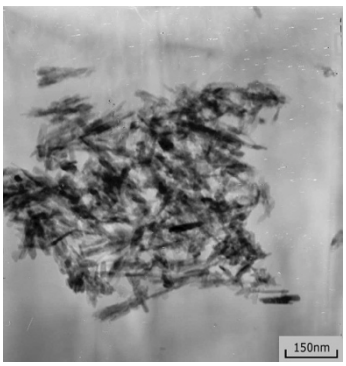

Fig. 1: The TEM micrograph of initial HAP particles

The specific surface area of such powder was in the range of 60-70 m^2/g. The XRD pattern of the powder showed low crystallinity. The only peaks recognized were those corresponding to the calcium hydroxyapatite phase (JCPDS pattern 9–432 for HAP).

All used methods allows to manufacture the dense HAP materials but more promising method seems to be the MW sintering because only this method allows to obtain dense HAP material at relatively low temperature and in short time – at 900 ^oC in 15 minutes (Table 1). This provides the saving of nanosize microstructure of the material and therefore the high values of fracture toughness up to 1.5 $MPa \cdot m^{1/2}$, microhardness up to 5 GPa and improved biocompatibility [1].

Table 1. Sintering conditions for production HAP materials with relative density > 95 % and the average grain size of the obtained material.

Sintering method and conditions	Sintering temperature, ^oC / time	Average grain size, μm
Axially pressed (100 MPa) samples in air in common furnace	1200/ 2 h	3-10
Axially pressed in microwave (MW) furnace in air	900/ 15 min	0,1
	1200/ 15 min	1,6
Could isostatically pressed (CIP) at 400 MPa in common furnace	1200/ 2 h	3
Hot pressing at 20 MPa in Argon of CIP samples	1000/ 2 h	0.4
CIP samples in MW furnace	900/ 15 min	0,1
	1200/ 15 min	3,5
Spark plasma sintering at 40 MPa	1000/ 10 min	1-7

REFERENCES: [1] Dj. Veljovic´, M. Colic´, V. Kojic´, et al (2012) *J Biomed Mater Res Part A* **100A**: 3059–3070.