SYNTHESIS AND CHARACTERIZATION OF HYDROXYAPATITE NANOPOWDERS WITH HIGH STABILITY

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Hydroxyapatite (HAp: $Ca_{10}(PO_4)_6(OH)_2$) is chemically similar to the mineral component of bones and hard tissues, being one of the few synthetic materials that shows bioactivity [1-3]. However, its applications are limited, because of HAp instability when processed at elevate temperatures [1-4]. Preparation of dense HAp ceramics with superior stability is possible if the starting powders are very fine [1-5]. Synthesis methods that are imitating the physiological conditions seem also to lead to materials with improved properties [1-6].

The hydroxyapatite powders were obtained through chemical methods, from $Ca(NO_3)_2 \cdot 10H_2O$ and $(NH_4)_2HPO_4$, mixed in stoichiometric proportions. The reaction was performed in SBF (Simulated Body Fluid) solution, in the presence of urea. There were used two different ways for mixing the precursors: "initial" addition and "successive" addition [7-10]. For the "initial" addition method the solutions of precursors were mixed instantly, and for the "successive" addition, the solutions were mixed in droplets.

The powders were calcinated at 600^0C and characterized in what it concerns the mineralogical composition (using an X-ray difractometer *SCHIMADZU XRD 6000)* and the microstructure (using a scanning electronic microscope *EDAX - HITACHI S2600N*). The diffraction images showed the presence of hydroxyapatite (JCPDS 09-0432) as unique phase, for all experimental conditions, that proved to be stable even after sintering treatments at temperatures between 1000 and 1500˚C, for 6 hours.

The scanning electron microscopy showed that the powder obtained by "initial" addition, thermally treated at 1000°C for 2h, is constituted of particles with elongated shape, needle-like, with dimensions between 0.5 and 1.0 μm. Intergranular porosity is present, with dimensions of approximately 0.5μm (figure 2A). If the temperature increases to 1500°C, the SEM images are demonstrating a higher densification of samples, with importantly reduced porosity (figure 2B).

The powder synthesized by the „successive" addition, thermally treated at 1000˚C/2h, it has the same morphology, but this time the length of needles is bigger, reaching even 20 μm (figure 3A). Higher densification is achieved also for this powder at higher temperature.

We might conclude that all experimental conditions considered led to the formation of hydroxyapatite nanopowders, stable at high temperature thermal treatments. That will permit better densification of hydroxyapatite compacts, which could potentially develop better mechanical properties.

References:

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Figures:

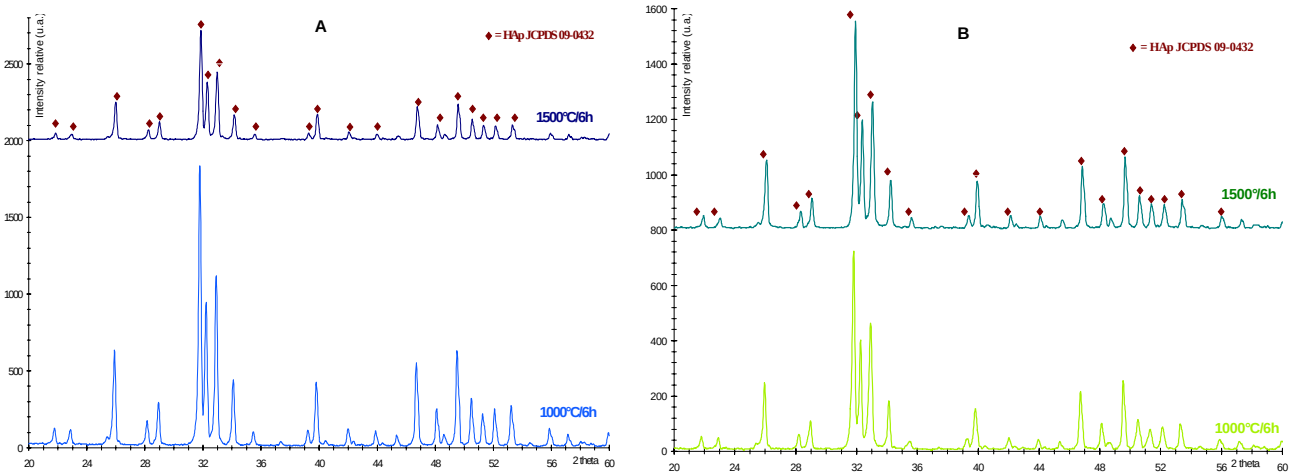

Figure 1. The X-ray diffraction spectrum for powders synthesized by “initial” addition (A) and “successive” addition (B), for temperatures of thermal treatment of 1000 and 1500°C

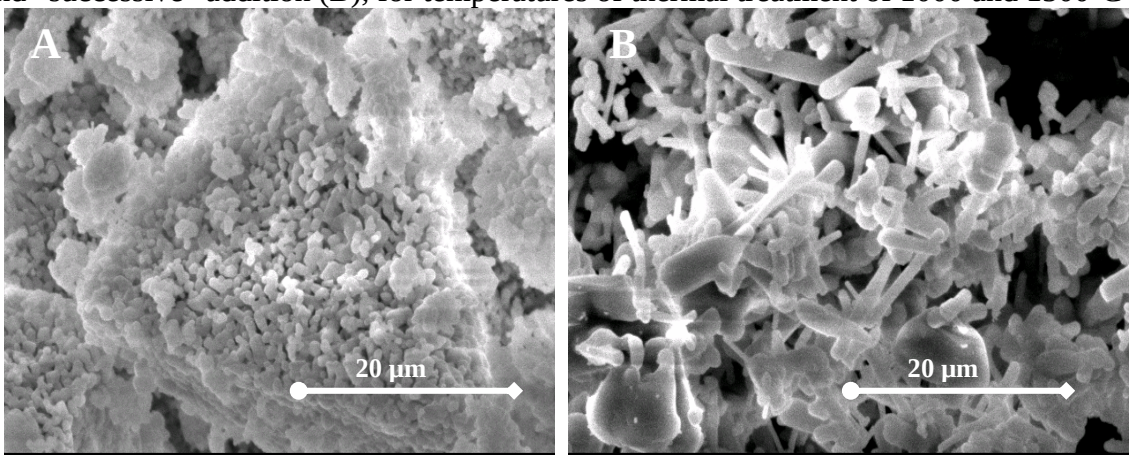

Figure 2: Scanning electron microscopy images of powder synthesized by “initial” addition (A) and “successive” addition (B) thermally treated at 1000°C/6h (X 2000)

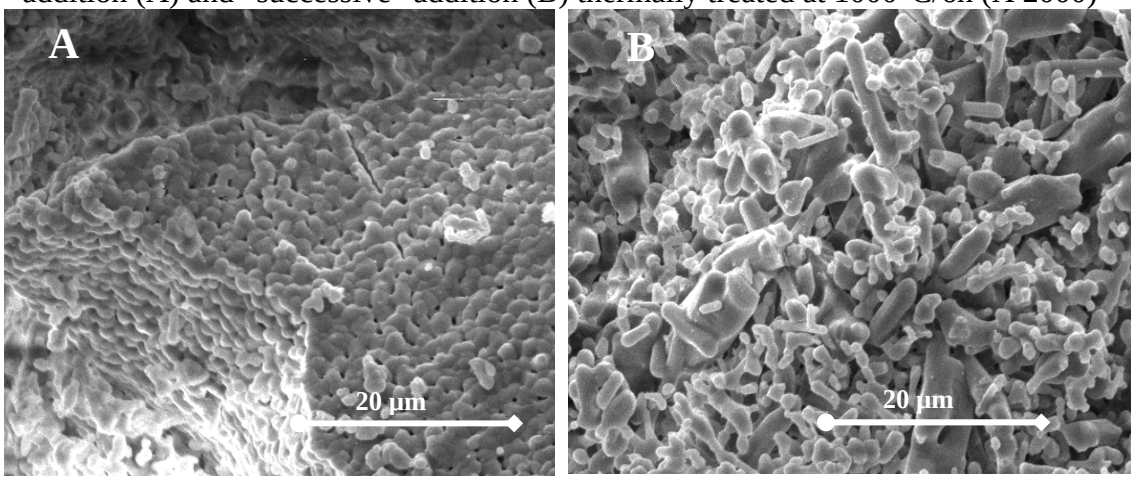

Figure 3: Scanning electron microscopy images of powder synthesized by “initial” addition (A) and “successive” addition (B) thermally treated at 1500°C/6h (X 2000)