NANOCRISTALLITES OBTAINED THROUGH THE PYROSOL METHOD

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The decrease of particles size from micrometric to the nanometric scale is allowing the development of new materials, with dramatically improved properties.

The pyrolysis of solutions is a very interesting method of production for reactive nanopowders. A great range of composition, size and morphology can be obtained through modifying the characteristics of precursors, the properties of solutions used and the experimental parameters of the selected [1]. The pyrosol process is based on the pyrolysis of an aerosol, produced by ultrasonic pulverization. The vibrations generated on the surface of a solution by a beam of ultrasounds, directed towards the interface gas/liquid, are used for generating the aerosol. The ultrasounds beam is generated by a piezoelectric ceramic. The wavelength of the vibrations depends mainly on the frequency "f" of the ultrasounds and the characteristics of the irradiated solution [2]. The average diameter of drops is inversely proportional to $f^{2/3}$. The aerosol thus formed is pulled by a carrier gas, whose flow is fixed, in a furnace at high temperature, in which the drops undergo a sequence of physicochemical transformations, described into details by Messing and coll. [1, 3, 4, 5, 6]. The high temperature causes evaporation of solvent and also determines the precipitation of soluble species in the volume of a dense and spherical particle. At the exit from the furnace, an electrostatic filter allows the recovery of powders. The schema of a typical pyrolisis installation is presented in Figure 1.

In this study, the influence of the various synthesis parameters on the crystallinity and the composition of calcium phosphate powders were investigated. Firstly, the influence of the chemical nature of precursory calcium salt was studied, by using two types of water soluble calcium salts: $CaCl_2 \cdot 2H_2O$ and Ca $(NO_3)_2$. The precursor used for P_2O_5 is $(NH_4)H_2PO_4$. In the second time, it was studied the effect of the pyrolisis temperature, in the interval 600˚C-1000˚C.

The obtained powders were investigated through thermal analysis: thermo gravimetric-TGA and thermal differential analyses-DTA, X-ray diffraction and electronic scanning microscopy. Whatever the temperature of pyrolysis of the aerosol, the apatite structure is recognizable - JCPDS 09-0432), if the precursor used for the calcium oxide is calcium chloride. If the powders are elaborated starting from calcium nitrate, the only phase identified is β-calcium phosphate.

For the powders synthesized from calcium chloride the microscopic observations highlight a majority of approximately spherical particles, of hundreds of nanometers size. Can also be noticed larger particles, of around 4μm, which we suppose that are containing inside smaller particles (Figure 2a). The general aspect of powders is kept for those obtained starting from calcium nitrate. The smaller spherical particles are however more deformed and the larger ones are highly porous, presenting a rough surface (Figure 2b).

The average size of grains was calculated using the Scherrer formula, from diffraction data. It was found that the lowest crystallite sizes are obtained for the lowest pyrolysis temperature. The size of crystallites for the powder obtained starting from calcium chloride is of 30nm, for a pyrolisis temperature of 600˚C and it is increasing to 50nm at 900˚C and to 66nm for a temperature of 1000˚C.

In all cases, the synthesis by pyrosol makes it possible to obtain powders with various compositions, made up of spherical particles, with sizes in the nanometric range, that can have many applications.

References:

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Figures:

Figure 1: Pyrosol installation

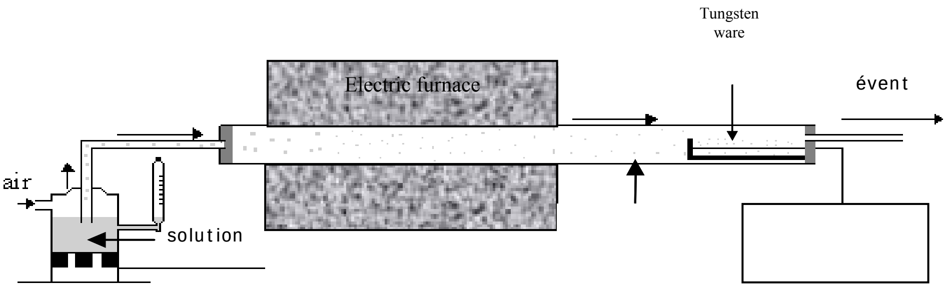
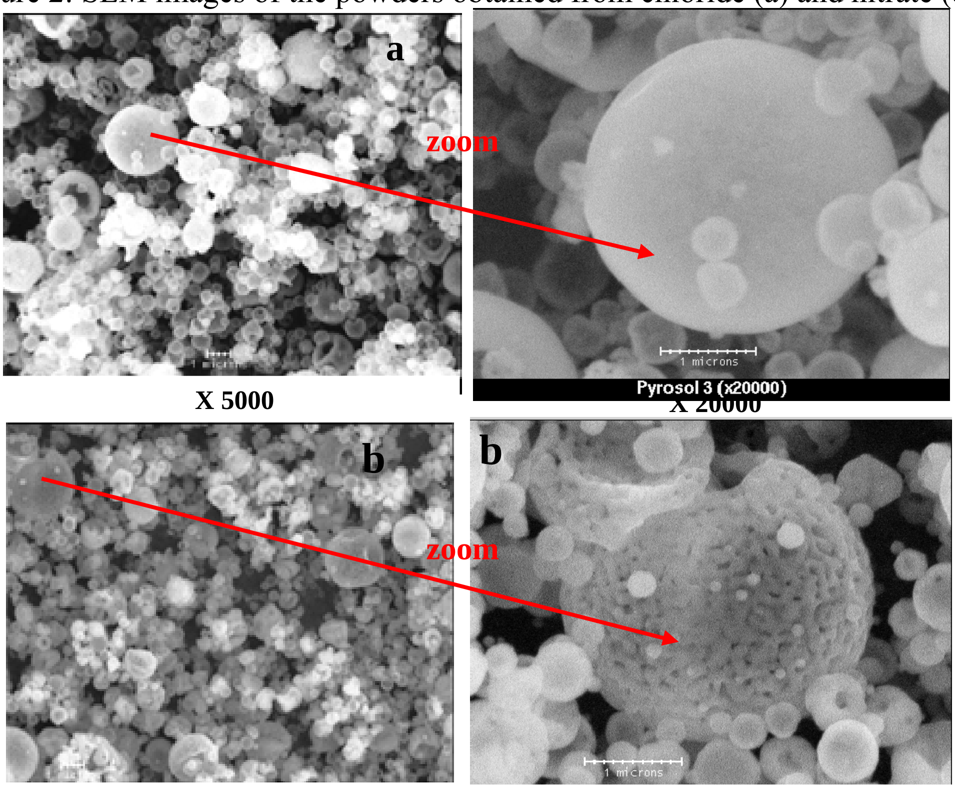


Figure 2: SEM images of the powders obtained from chloride (a) and nitrate (b)



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