Characterization of Hydroxyapatite by Electron Microscopy

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Bones and teeth of vertebrate animals are constituted by a mineral phase very similar to hydroxyapatite (Ca$_{10}$(PO$_4$)$_6$(OH)$_2$), for this reason, this material receives a great attention in the industrial and biological area. The structure and particular properties of the synthetic hydroxyapatite used in the production of artificial bones and teeth are very sensitive to the conditions of synthesis, for which, the studies carried out on the methods of synthesis of hydroxyapatite are very important because they determine the growth of the crystal, morphology, aggregation, nucleation and dissolution properties of hydroxyapatite. The present work was developed in order to establish the effects of reaction time in morphological and structural characteristics of hydroxyapatite.

The synthesis was carried out starting of brushite and starfish, the last one like source of calcium carbonate, under hydrothermal conditions using stoichiometric amounts of both reagents, varying times of reaction and maintaining pressure and temperature constant. The products of reaction were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

The growth of grouping crystals in form of fibers of the order of 1 µm to 370 µm long and 0.15 µm to 10 µm diameter with a composition of C, O, P and Ca was observed (Figure 1). It is possible also observe the growth of fibers of the order of 5 µm of longitude and the same composition that last. Besides particles of the order of 3 µm to 10 µm with an elementary composition of C, O, P Mg and Ca and prisms of the order of 17 µm to 43 µm were also observed.

By TEM, fibers with a size of 800 nm long and 160 nm diameter were observed and they show a growth in groups (Figure 2a). Each fiber exhibits the typical hexagonal structure of the hydroxyapatite. The diffraction pattern (Figure 2b) corresponds to hydroxyapatite fibers according to the indexation.

References

Figure 1. a) Hydroxyapatite fibers in order about 30 µm. b) EDS spectrum of hydroxyapatite fibers.

Figure 2. a) Bright file image of hydroxyapatite fibers forming a group in order to 800 nm long and 160 nm diameter. b) Diffraction pattern in [0 0 0 1] direction.