FEG-SEM and XRD Studies of Aging Time in the Hidroxiapatite Synthesis by the Sol-Gel Method using Biological Precursor

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Compounds derived from calcium phosphate have been the subject of numerous researches for new material system development and technological application, such as, coating systems for metal structures, support structure for catalysts, fertilizers, and water treatment media [1-2]. The sol-gel method is considered one of the best methods for the synthesis of hydroxyapatite by conducting a molecular mixing of the precursors of calcium and phosphorus. These mechanism induces the formation of hydroxyapatite of high purity and homogeneity using low processing temperatures with the production of nanometric particles. Some aspects of the HAP microstructure and of the synthesis process can be characterized by the surface morphology, the presence of porosity, the particle size distribution, the particle shape and crystallographic orientation and the identification of the formed phases. In this work, hydroxyapatite (HAP) was obtained by the sol-gel method, using eggshells as precursors of calcium, where the aging time was varied for the observation of its influence amount of hydroxyapatite phase obtained and in the influence of the particle size. The reactions were performed in aqueous medium with control of reagent pH and temperature and the reaction time. The gels obtained during the synthesis were treated in three different conditions: calcined directly after their production, aged for 24 hours and aged for two weeks, to observe the HAP conversion rate and the particle size and morphology. High resolution Scanning Electron Microscopy has been performed over those samples for morfology and particule size observation using a JEOL 7100FT. For those observations, It has been used 0,30 kV for electron acceleration and inlens SE/BS detectors to achieve higher magnifications. XRD measurements were carried out on these samples using a Panalytical X'PERT PRO diffractometer with CuKα radiation, a scanning step of 0.05^{o} and a collecting time of 300 seconds per step with Quantitative Rietveld calculations. The high resolution FEG-SEM images have revealed, for all aging conditions, a submicron HAP particule size with a faced like morphology, as shown in figure 1. This behavior could be inferred with the monoclinic structure of the HAP phase identified with Rietveld calculations. This HAP crystal structure is related with the use of natural calcium precursor for HAP synthesis[3]. The Rietveld phase quantification has revealed the aging time influences the HAP phase content in the calcinated particles. The aging time decreased the HAP content from 76.15% for the as synthetized samples, to 72.76% for the 2h aged samples and to 0.0% of HAP content for 2 week aged samples. The EDS mapping (Fig. 2) have revealed the presence of Ca, P, O and H and for the 2 week aged sample, also the incorporation of N.

References

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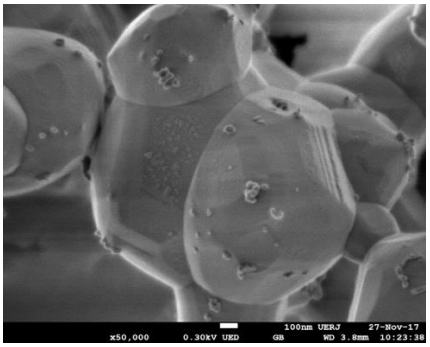

Fig. 1. FEG-SEM image of as-synthetized HAP using SE inlens detector with 0.30 kV electron acceleration

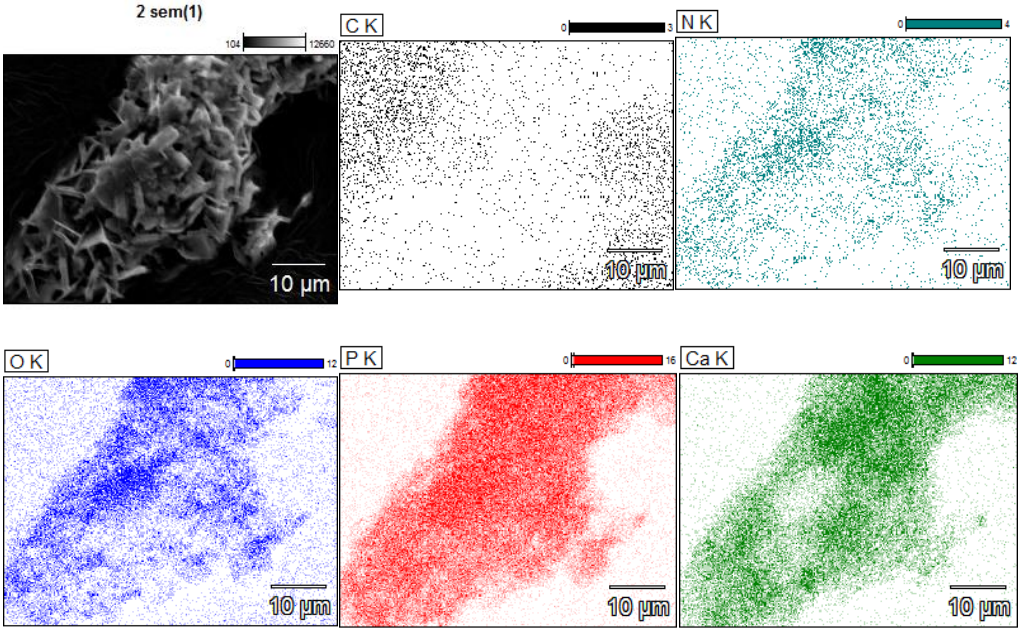

Fig. 2. EDS mapping of as-synthetized HAP