Influence of temperature on obtaining apatites by sol-gel method

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Calcium phosphate-based ceramics (apatites) are synthetic materials that have a chemical composition similar to bone tissue, which makes them interesting to use as biomaterials [1]. In this work, the apatite precursor sols at concentrations of 0.7 mol/L and 1.4 mol/L are prepared by dissolving calcium nitrate and phosphorus pentoxide in ethanol. The sols are kept at rest for 24 hours for hydrolysis (gelatinization) under room temperature and humidity. After hydrolysis, the resulting gels are dried at 80℃ to remove the organic solvent and then calcined under different temperatures (from 500℃ to 1200℃, with increments of 100℃) for 8h [2]. The X-ray diffraction analysis of the samples by the Rietveld method indicates that the samples are composed of monoclinic and hexagonal hydroxyapatite (HAp), the former being predominant, and calcium β-triphosphate (TCP)[3] .For the material started from the sol with a concentration of 0.7 mol/L, the hydroxyapatite fraction increases from 28.8% to 66.5% when the calcination temperature increases from 500℃ to 800℃. Above this temperature, the fraction of this phase decreases. With a concentration of 1.4 mol/L, the hydroxyapatite fraction decreases from 88.2% to 19.5% progressively with increasing temperature from 500℃ to 1200℃. The micrographs revealed the formation of particulate but porous materials that agglomerate without sintering up to a temperature of 900℃, from where the formation of sintered and dense agglomerates is observed. The results of the EDX analysis showed a Ca/P molar ratio ofaround 1.5 for all materials. The cytotoxicity assay, using pre-osteoblast cells (MC3T3-E1,Subclone 4) by non-direct contact of 24h, showed that cell viability and adhesion did notdiffer from those of the control. A decrease in viability and adhesion was observed for samples prepared at a concentration of 0.7 mol/L and calcined at temperatures above 900℃.