Iron Substituted Hydroxyapatite by Two Powder Preparation Method: a Comparison

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The aim of this work is the characterization of hydroxyapatite (HAP), nominal composition $[(Ca_{10-x}Fe_x)(PO_4)_6(OH)_y]$, x=0.0; 0.05; 0.1; 0.5; 1.0; 1.5, obtained by high temperature solid state reaction (HTS) and wet precipitation (WPS). The ceramic samples were prepared by firing appropriate quantities of the metal carbonate, iron(III) oxide and ammonium hydrogen phosphate at 1200 °C (x=0.05, 0.1, 0.5 and 1.0) for 72 hours or at 1450 °C (x=1.5) for 48 hours. The heat treatment was repeated two more times with intermediate grinding. Materials of the same composition were prepared with a wet chemical synthesis by mixing appropriate quantities of solutions of the metal nitrate and ammonium phosphate at pH=10.5. The precipitates were dried at 80 °C and fired at 550°C in O_2 flux for 6 hours. The X-ray diffraction patterns show for all powders obtained by HTS sharp and narrow apatite peaks, while WPS samples present broad peaks indicating a low degree of crystallinity; crystallite sizes result to be 200 and 20 nm respectively. All HTS samples show the presence of HAP and/or tricalcium phosphate, the relative amounts depending on the theoretical composition. Hydroxyapatite decreases from 100 to 39% for x varying from 0.0 to 0.5, while for x=1.0 and 1.5 no hydroxyapatite peaks were detected. With decreasing hydroxyapatite content, tricalcium phosphate increases up to 96% for x=1.0 and 1.5. Mössbauer spectra show the presence of iron oxidic phases, such as hematite and magnetite for HTS samples and ferrihydrite for WPS ones, not detectable by X-ray diffraction.

Keywords: apatites, Mössbauer, x-ray diffraction