

CRYSTAL ANALYSIS OF HYDROXYAPATITE ROD-LIKE PARTICLES OBTAINED BY HYDROTHERMAL SYNTHESIS FROM COMPLEX Ca(II)-EDTA

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Calcium based phosphate ceramics have been proved to be excellent material for biological applications. The hydroxyapatite ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$), main mineral component of bones and teeth, has been studied due to its excellent biocompatibility. One of the hydroxyapatite preparation methods is the hydrothermal synthesis. This method allows to obtain crystal in several sizes and shapes which range from spherical nanoparticles, to sticks, prisms and needles even with micrometer sizes. In this study, hydroxyapatite crystals with uniform morphology were synthesized through the hydrothermal method using the Ca (II)-EDTA complex as the starting solution and phosphate ions at pH 9.0; The synthesis was carried out at 170 °C for 24 hours. The product obtained was characterized through X-ray diffraction and absorption spectroscopy in the infrared region and scanning electronic microscopy. The crystalline structure analysis and physical characteristics of the crystals were carried out through X-ray diffraction and the Rietveld Method. The microstrain distribution was performed through the phenomenological model of Peter Stephens incorporated to the Rietveld Method in the program GSAS+ExpGui. The hydroxyapatite was identified as an only phase, with refined cell parameters $a = b = 9.43575$ (8) Å $c = 6.8834$ (1) Å. Numbers in parenthesis indicate the standard deviation of the last decimal place. Refining quality statistical indicators were $S = 1.17$, $R_{wp} = 12.14\%$ and $RB = 2.74\%$. These values show the good quality of the profile fitting and the crystal structure. The crystallite exhibited anisotropic medium size, with 15.4 Å and 13.9 Å in the parallel and perpendicular directions to the planes (0 k 0), respectively. The distribution of microstrains also indicated an anisotropic defects distribution. Micrographs obtained through SEM revealed that the particles obtained are uniform as rod-like microcrystal, which confirmed the results obtained for the crystallite size anisotropy and reticulum microstrains.

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