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Thermal stability of hydroxyapatite prepared by mechano-chemical reaction

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Abstract – The main goal of the present work is the study of the thermal stability of hydroxyapatite prepared by means of mechanochemical process. For this purpose we employ calcium carbonate and ammonium phosphate as raw materials. The characterization of the samples were made by infrared spectroscopy (FT–IR), X-ray diffraction technique (XRD), Induced coupled plasma spectroscopy (ICP) and scanning electron microscopy (SEM). The results show that after 30 hours of mechanochemical process was can be obtained a mixture of nanostructured powder of non- stoichiometric Hap and a calcium carbonate remain. The thermal studies revealed that the carbonate ions are present in the Hap phase as a substitute in the phosphate sites. Furthermore, the results show that the samples start to decompose above 600^{o}C reaching a total decomposition in two phases, stoichiometric Hap and Tricalcium phosphate, at 900^{o}C.

The main inorganic component of hard tissues on vertebrates is a phase of calcium phosphate whose structure closely resembles hydroxyapatite, $Ca_{10}(PO_4)_6(OH)_2$, [1]. In this sense the study of synthetic hydroxyapatite (Hap) has been attracting considerable attention because is widely used in biomedical applications. Between the different processing techniques developed to prepare Hap [2] the mechanochemical technique has shown to be the most suitable due to its versatility and low production cost. This technique takes advantage of the perturbation by pressure of surface-bonded species to enhance thermodynamic and kinetic reactions in powders. The present investigation shows the thermal stability of Hap prepared by means of mechanochemical process employing hen's eggshell as the Ca source. The reaction was made following the equation in the equilibrium:

$$10\ CaCO_3+6(NH_4)H_2PO_4 \rightarrow Ca_{10}(PO_4)_6(OH)_2+8H_2O+10CO_2+6NH_3$$

The characterization of the samples was made by infrared spectroscopy (FT–IR), X-ray diffraction technique (XRD), Induced coupled plasma spectroscopy (ICP) and scanning electron microscopy (SEM). The results indicate that after 30 hours of milling applied over a mixture of $CaCO_3$ and $(NH_4)H_2PO_4$, can be obtained a mixture of nanostructured powder of non- stoichiometric Hap and a calcium carbonate remain. The average crystallite size was estimated within the range of 10 to 30 nm. The thermal studies revealed that carbonate ions were present into the Hap phase as a substitute in the phosphate site. This study also shows that the samples were metastables because they started to decompose above 600^{o}C. At 900^{o}C we observed the decomposition of the sample into stoichiometric Hap (JCPD: 89-6439) and a phase of tri-calcium phosphate (TCP) (JCPD: 79-2186).

References

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