

TEM ANALYSIS OF HYDROXYAPATITE AND WHITLOCKITE OBTAINED UNDER HYDROTHERMAL CONDITIONS.

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Some calcium phosphates are of great importance because their characteristics and properties are very similar to those of bones and teeth, such as hydroxyapatite ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$) [1, 2]. Besides, hydroxyapatite has two important characteristics as biomaterials: biocompatibility and bioactivity. In some natural systems and calcifications magnesium ions are also presented additional to the hydroxyapatite elements. Nevertheless, these magnesium ions have a marked effect on nucleation and growth of calcium phosphates when they are synthesized. However it is also very well established that magnesium disallows the hydroxyapatite nucleation and promotes whitlockite [3].

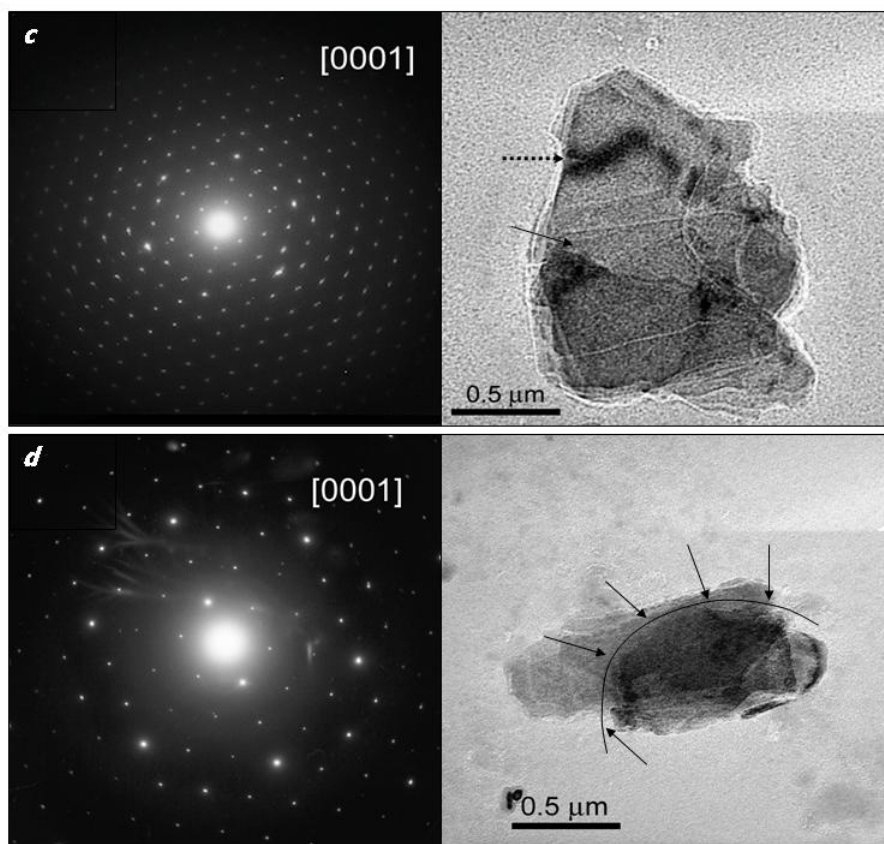
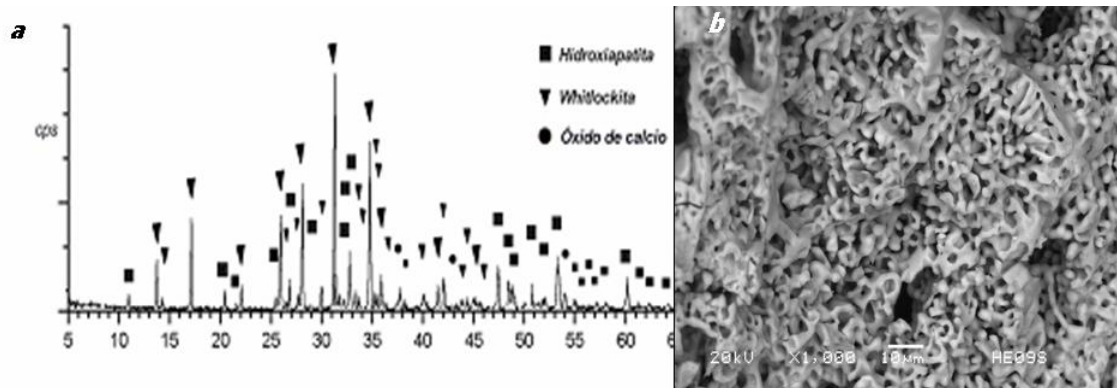
In this work we comment the structural characteristics of both hydroxyapatite and whitlockite obtained by the hydrothermal method. The process includes calcium oxide (lime) and diammonium phosphate in an autoclave. The product was characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) [4].

For X-ray diffraction, a BRUKER-AXS Diffractometer was used. For SEM observation a JEOL 5600LV, that has attached NORAN characteristic x ray energy dispersive spectrometer (EDS), was used. For TEM analysis, a JEOL 100CX was used.

Whitlockite and hydroxyapatite were easily identified by X-ray diffraction (Figure **a**). In all cases, the powder obtained was compound by agglomerates of not very defined particles. These irregular structures have less than 5 μm in size, as observed with SEM (Figure **b**). Small particles corresponding to each phase were analyzed by TEM and confirmed their structural characteristics by electron diffraction patterns (Figure **c** and **d**).

References

- [1] Bu Park J. Biomaterials Science and Engineering. Plenum Press. N.Y. 1984.
- [2] Smith D. K. Calcium Phosphates Apatites in Nature. Hydroxyapatite and related materials. Paul W. Brown and Brent Constant Editors. C. R. C. Press. 1994.
- [3] Hamad, M. The growth of Whitlockite. JCG. 79 (1986) 192-197.
- [4] Williams D. B. and Carter C. B. Transmission Electron Microscopy. A Text Book for Materials Science. Plenum Press. N. Y. 1996.
- [5] The authors thank to Pedro Mexia, Roberto Hernández, Luis Rendón, Jacqueline Cañetas, Samuel Tehuacanero, Cristina Zorrilla, Manuel Aguilar, and Carlos Magaña for technical support.



a) XRD spectrum indicating the presence of hydroxyapatite, whitlockite and calcium oxide. **b)** SEM image of particles of the product. **c-d)** Electron diffraction patterns and TEM bright field images of hydroxyapatite and whitlockite particles respectively.