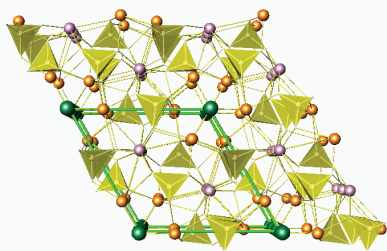


Introduction

The main inorganic component of bones and teeth is an impure form of hydroxyapatite, $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$. The major difference between natural bioapatites and hydroxyapatites is in the presence of small amount of F and about 3 wt.% CO_3 in bioapatites. The chemical composition of biogenic apatites usually range from carbonate-hydroxyapatite, $\text{Ca}_{10-x}(\text{PO}_4)_{6-y}(\text{CO}_3)_y(\text{OH})_z$, to $\text{Ca}_{10-x}(\text{PO}_4)_{6-y}(\text{CO}_3)_y\text{F}_z$. These end-members of two isomorphous series of carbonate-apatites have also been referred to as minerals dahllite and francolite, respectively. In this work, we analyzed natural hydroxyapatite extracted from alveolar human bone by thermal treatment with hot H_2O_2 .



1. Representation of crystal structure of hydroxyapatite along c-axis. The connection of Ca1 (purple-gray) and Ca2 (gold) cations with PO_4 tetrahedra (yellow). OH^- groups in channels are green.

Methods and Materials

Powder diffraction data for structural and microstructural analysis of investigated sample were collected at room temperature in Bragg-Brentano geometry using Philips 1050 diffractometer ($\text{CuK}\alpha$ radiation, $\lambda = 1.54178 \text{ \AA}$) and scan range from 8 to $110^\circ 2\theta$ at every $0.02^\circ 2\theta$. The counting time was 12 s/step. The crystal structure was refined by the Rietveld method using the Fullprof (Rodríguez-Carvajal, 1990) computing program. From the X-ray line broadening analysis, the apparent crystallite sizes in different directions and strain were calculated. The Raman spectra were taken in the backscattering geometry using a μ -Raman system with a Jobin Yvon T64000 triple monochromator, equipped with a liquid nitrogen cooled charge-coupled-device (CCD) detector. The morphology of the powders, previously gold-coated, was studied by scanning electron microscopy (JEOL SEM, JSM 6390), operating at 20 kV.

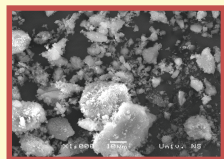
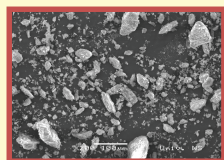


Fig. 6. SEM images of humne aleolar bone

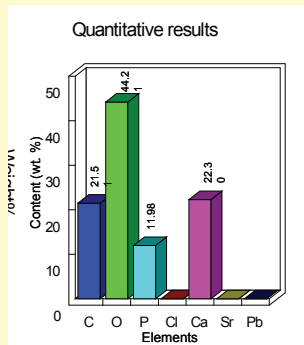


Fig. 5. EDS analysis of humne aleolar bone

Results

X-ray powder diffraction analysis as well as Raman spectroscopy confirmed that mammalian bones and teeth are an impure form of hydroxyapatite, (HAp), $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$. The collected XRPD data of investigated powder (Fig. 2) was used to refine its structure and calculate structural and microstructural parameters. The reliability factors (Table 1) show good agreement between the observed and calculated powder pattern. The refined cell dimensions are in accordance with literature data (Table 1). The structure refinement indicated X-ray line broadening anisotropy as a consequence of anisotropic growth of crystallites. Based on the studied values of crystallite size (Table 1) along different $[hkl]$ directions, the morphology of crystallites was assumed (Fig. 3).

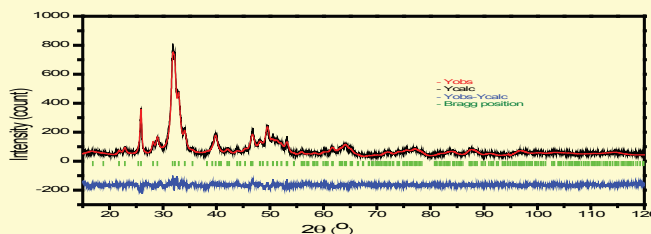


Fig. 2. Rietveld refinement plot of humne aleolar bone powder

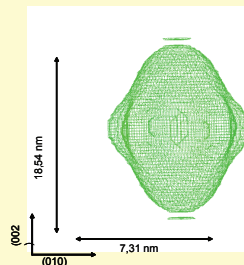


Fig. 3. The shape of crystallite size of the natural bone

Table 1. Crystall data and R factors

Space group	$P6_3/m$
Unit cell parameters	
a (Å)	9.41842(1)
c (Å)	6.88374(1)
Microstructural parameters	
crystallite size (Å)	99. (0.1)
strain (%)	0.22 (0.054)
Agreement factors	
R_w	12.10
R_p	9.12
R_f	3.20

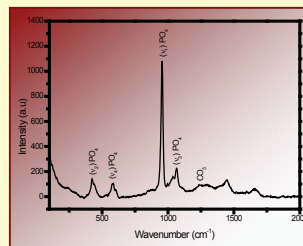


Fig. 4. Raman spectra of humne aleolar bone

The Raman spectroscopy clearly indicates on existence the CO_3 group in the structure of natural bone (Fig. 4). The EDS analysis gives definitive justification of the presence of C atoms in the investigated powder (Fig. 5). The SEM images of human alveolar bone powders show structures consisting of non-uniform agglomerates with various average particle sizes (Fig. 6).

Conclusion

X-ray powder diffraction, EDS analysis and Raman spectroscopy show that mammalian bones and teeth are an impure form of hydroxyapatite, (HAp) $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$. The Raman and EDS analysis confirmed the presence of CO_3^{2-} ions in the natural bone structure. The position of CO_3^{2-} ions in the apatite structure are not precisely defined and this will be the theme of our future work.