Mechanical properties of sintered hydroxylapatite (HAp)

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INTRODUCTION

In biology and medicine different types of calcium phosphates materials (CaP) play a very significant role. They can be found in human body as one of the crystalline components of dental enamel, dentine, and bones. Hydroxylapatite (HAp) based calcium phosphate ceramics due to their chemical composition, excellent biocompatibility, bioactivity and osteoconduction can be used as reparation materials in maxillofacial, dental and orthopedic surgery. Hydroxylapatite $Ca_5(PO_4)_3OH$, crystallographically and chemically similar to the mineralised constituent of hard tissues (Fig. 1.), was synthesized by a simple precipitation method. The HAp powders were compacted by uniaxial pressing at 78 MPa and sintered at 1000 °C for 2h. The effects of high temperature sintering conditions on the microstructure, phase composition stability, density and microhardness of sinthesized HAp material were investigated.

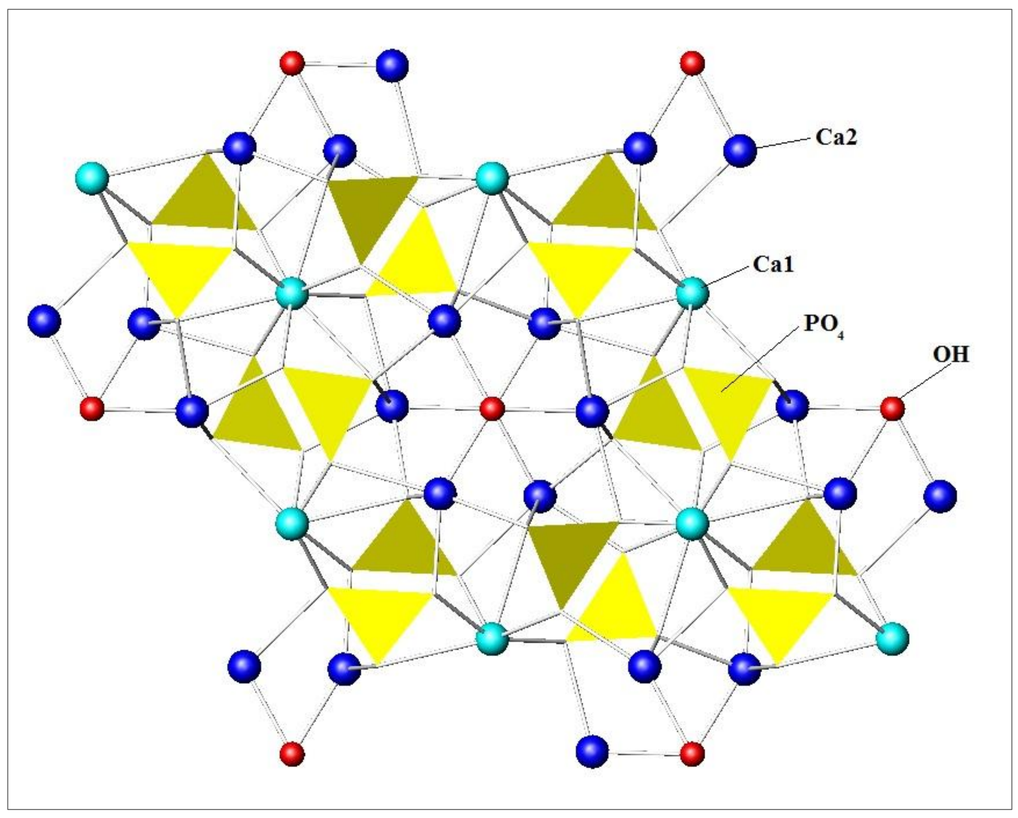


Fig. 1. Structure of Hydroxylapatite (HAp), s.g. $P6_3/m$

MATERIALS AND METHODS

❖Pure HAp was prepared by solution-precipitation method (Fig. 2.).

❖The phase composition before and after sintering treatment were determined by X-Ray powder diffraction (XRPD) analysis using Ultima IV Rigaku diffractometer, 40.0 kV, 40.0 mA; in range of 5–60° 2θ with a scanning step size of 0.02° and at a scan rate of 5°/min.

❖The Vickers' hardness of the HAp compacts was measured using Buehler MicroMet Microindentation Tester. The micro hardness of sintered compacts was examined with applied load of 0.3 kg with an indentation time of 30 s.

❖The morphology of compact was examined using Scanning electron microscopy (SEM), and analysis was carried out on the untreated sample surface using a JEOL –JSM – 500LV microscope.

❖Mass of pellets was measured and the volumes were calculated using formula: $V = r^2\pi \cdot h$. The volumetric mass densities were calculated by using the theoretical density ($\rho = 3.156\ gcm^{-3}$).

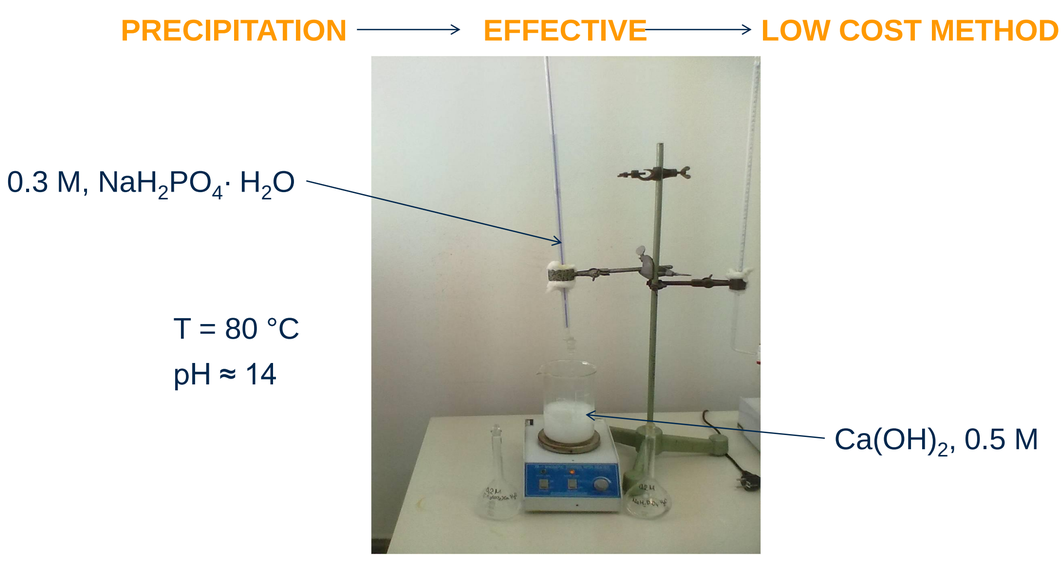


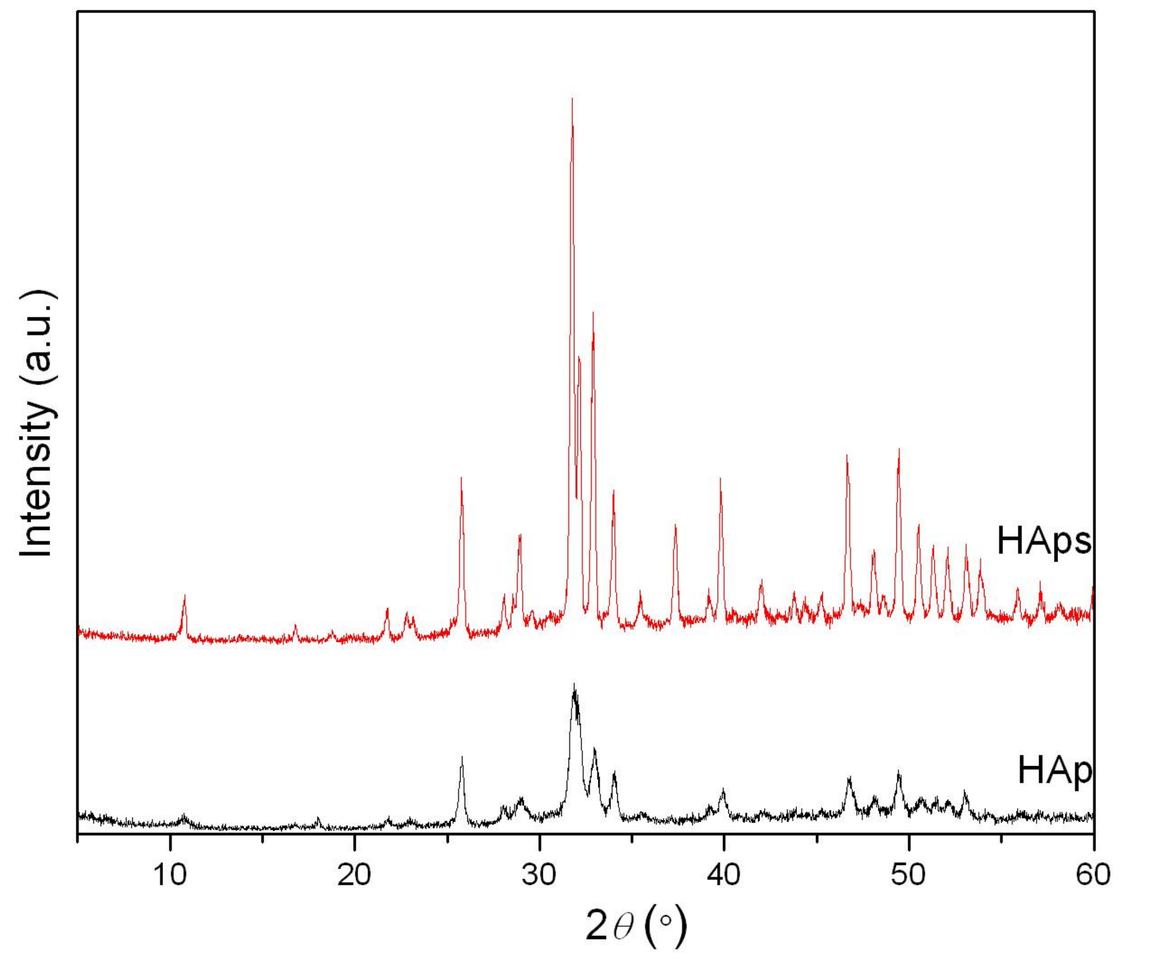
Fig. 2 . Synthesis of Hydroxylapatite material (HAp)

RESULTS

❖**XRPD** patterns of synthesized sample and sample sintered at 1000°C for 2h, are shown on Fig. 3. The XRPD patterns show clearly defined peaks of Hydroxylapatite.

❖Significantly higher intensity of the peaks of the sintered material (HAps) than those of synthesized sample (HAp) suggest that the high temperature treatment leads to structural ordering. Based on XRPD results the materials remains stable and pure at 1000°C, there is no secondary CaP phase.

❖**SEM** results (Fig. 3.) of obtained materials confirm the XRPD results. Synthesized sample (HAp) reveals loosely packed particles, while the sintered sample (HAps) shows larger and more compact grains oriented in same direction.



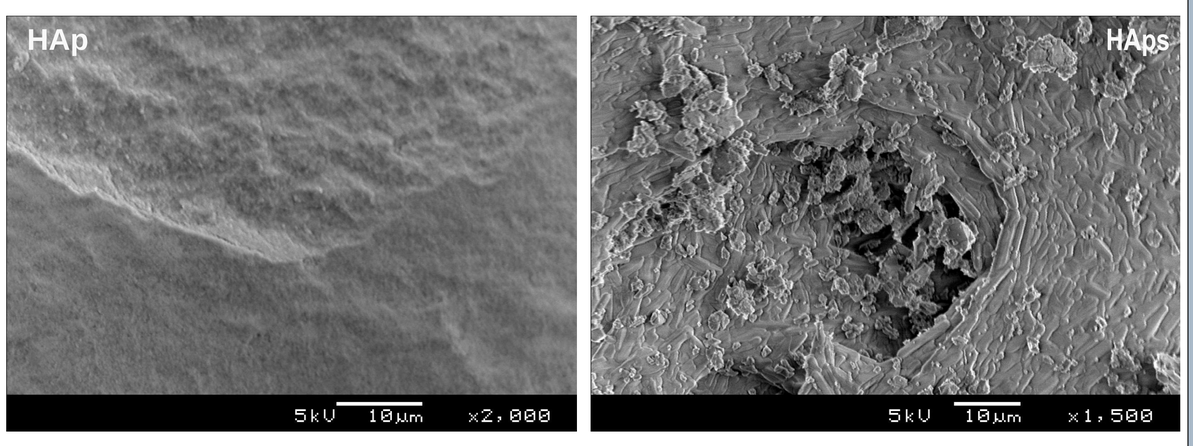


Fig. 3 . XRPD patterns and SEM microphotographs of HAp (untreated sample) and HAps (sample sintered on 1000 °C for 2h)

❖Results of **relative density and micro hardness** of obtained materials are shown in Table 1. Based on these results it is evident that the increase in temperature leads to density increase and better mechanical properties.

Table 1: Relative densities and micro hardness of HAp and HAps

Sample	Relative density (%)	Temperature (°C)	Retention (h)	Vickers' hardness (MPa)
HAp	39.38			4.3
HAps	86.08	1000	2	11.5

CONCLUSIONS

Pure HAp material was successfully synthesized using solution-precipitation method. Obtained material has low degree of crystallinity, relative density and hardness. At higher temperatures (sintered sample) grain size increases as does the crystallinity. Sintered sample shows two and a half times higher values of density and hardness than thermally untreated sample. Structure of the obtained material remains stable during sintering procedure. For further use of these materials for biological purposes biocompatibility and cytotoxicity tests should be conducted.

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