THE XRD ANALYSIS OF THE CALCIUM PHOSPHATES PHASE COMPOSITION DEPENDING ON THE POWDER SYNTHESIS METHODS AND HEATING RATES

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BACKGROUND

- The hydroxyapatite [$Ca_{10}(PO_4)_6(OH)_2$] (HAp) belongs to large family of calcium phosphate minerals, which have a wide application in medicine, catalysis and ecology. In the comparison with other calcium phosphate phases, hydroxyapatite possesses the highest stability at room temperature and pH between 4 and 12. However, with increasing the temperature, HAp can be partially or total transformed in the tricalcium phosphate [$Ca_3(PO_4)_3$] (TCP). The phase transformation of HAp into low-temperature (beta) modification of tricalcim phosphate usually starts on the temperature above 700 °C. Furthermore, the increasing of temperature above 1100 °C, leads to appearance high temperature (alpha) modification of tricalcium phosphate. Dynamics of the emergence of the particular phases as well as their relative ratio in the systems strongly depend on the heating regime, dwell time as well as the properties of the investigated powders.
- In this work the influence of the different heating rates (5, 10 °/min) on the HAp powders synthesized by precipitation method and hydrothermal treatment was analyzed. The dwell time at certain temperatures was 5 minutes for both systems.

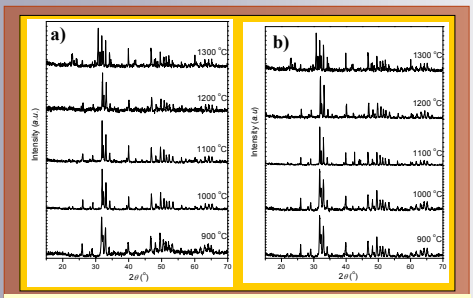

Fig. 1 *XRD patterns of HAp powders synthesized by hydrothermal method and heated with heating rates (a) 5 °/min and (b) 10 °/min.*

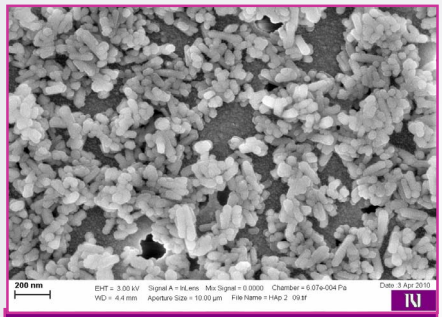

Fig.3 FE SEM image of the starting HAp powder synthesized by hydrothermal treatment.

Table 1:The influence of the different heating rates and sintering temperature on the HAp powders synthesized by hydrothermal treatment.

Sintering temperature (°C)	heating rates					
	5 °/min			10 °/min		
	HAp	β-TCP	α-TCP	HAp	β-TCP	α-TCP
900	100	0	0	100	0	0
1000	100	0	0	100	0	0
1100	100	0	0	100	0	0
1200	92	8	0	100	0	0
1300	55	0	45	45	0	55

Table 2:The influence of the different heating rates and sintering temperature on the HAp powders synthesized by precipitation method

Sintering temperature(° C)	heating rates					
	5 °/min			10 °/min		
	HAp	β-TCP	α-TCP	HAp	β-TCP	α-TCP
900	100	0	0	100	0	0
1000	100	0	0	100	0	0
1100	100	0	0	100	0	0
1200	100	0	0	100	0	0
1300	100	0	0	91.5	8.5	0

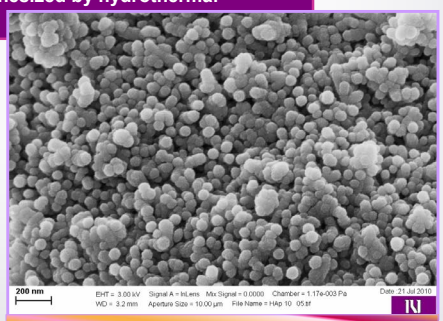

Fig. 4 FE SEM image of the starting HAp powder synthesized by precipitation method.

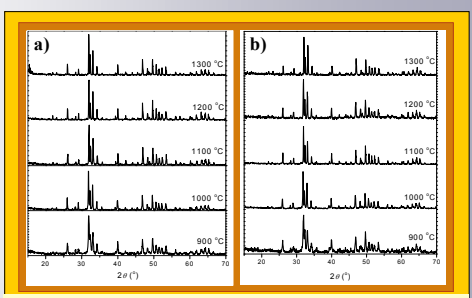

Fig. 2 *XRD patterns of HAp powders synthesized by precipitation method and heated with heating rates (a) 5 °/min and (b) 10 °/min.*

MATERIALS AND METHODS

The evolution of the phases and their mutual ratio in the investigated systems was estimated by the XRD analysis. Powder diffraction data for structural and microstructural analysis of investigated sample were collected at room temperature in Bragg-Brentano geometry using Philips 1050 diffractometer (CuKα radiation, λ = 1.54178 Å) and scan range from 10 to 70º 2θ at every 0.05º 2θ. The counting time was 3 s/step. The morphology of the starting HAp powders was performed by scanning electron microscopy (FE SEM).

RESULTS

The influence of the different heating rate (5, 10 °/min) as well as sintering temperature on the HAp powders synthesized by precipitation method (Fig. 1) and hydrothermal treatment (Fig. 2) was estimated by the XRPD analysis. Based on intensity ratio of the diffraction maximums, the relative amount of the phases was calculated in the investigated powders (Table 1 and Table 2). Obtained data indicate on significant influence of the heating rate as well as of synthesis procedure on the phase transformations and stability of the investigated powders. It is evident that HAp powder synthesized by precipitation method shows higher stability than powder synthesized by hydrothermal treatment. Lower heating rates result in higher stability of the HAp powders with increasing temperature.

CONCLUSIONS

The X-ray powder diffraction analysis was used to estimate evolution of the phases and their mutual ratio in the HAp powders synthesized by precipitation method and hydrothermal treatment. Obtained data clearly indicate on significant influence of heating rate and synthesis procedure on the phase transformation and evolution of the phases in the investigated samples.

It is evident that powders synthesized by precipitation method show higher degree of stability compared with powders synthesized by hydrothermal treatment.