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Synthesis and characterization of HAP and beta-TCP ultrafine powders

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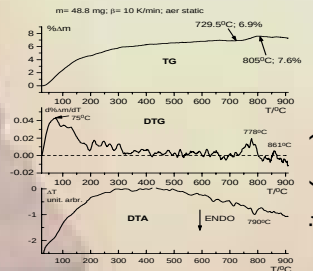
AIM OF THE WORK

To obtain ceramic ultra fine powders of HAP and β –TCP, by wet chemical synthesis, from salt solutions.

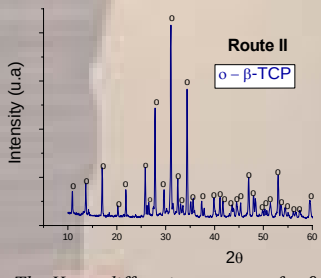
EXPERIMENTS

- **Hydroxyapatite (HAP) and beta-tricalcium phosphate (β-TCP) powders were obtained by precipitation synthesis, from salt solutions.**
- **For experiments was used $(NH_4)_2HPO4$ and $Ca(NO_3)_2 \cdot 4H_2O$, all made by Fluka.**
- **The first step was to obtain the precursor phase of $Ca_9(HPO_4)(PO_4)_5(OH)$, which is named ,,apatitic tricalcium phosphate", and then converting it to β -TCP by calcinations at low temperature.**
- **Hydroxyapatite was obtain by thermal treatment at 1000°C for 2hour.**
- **The formation of the β - TCP and the temperature transformation of precursor (apatitic tricalcium phosphate) was evidenced from X-ray diffraction analysis (XRD) and thermal analysis (TG/DTA/DGA). The same characterizations were carried out for analyzing the hydroxyapatite powders.**

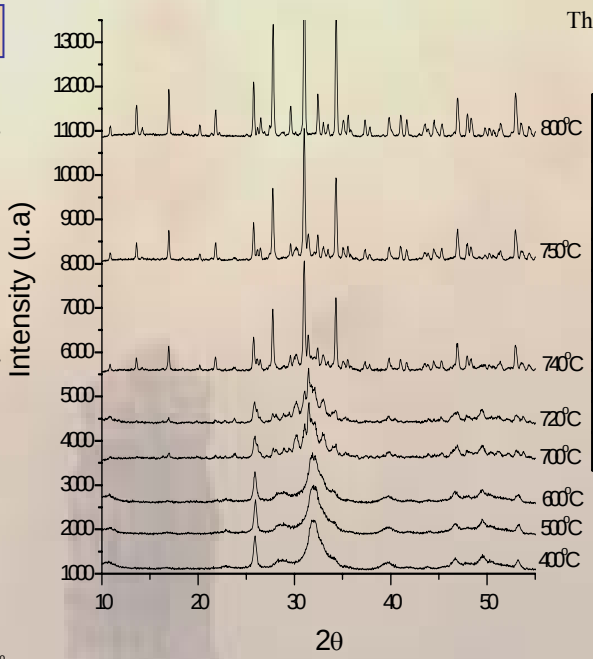
RESULTS



The curves of TG/DTG/DTA for formation of the β-TCP compound.



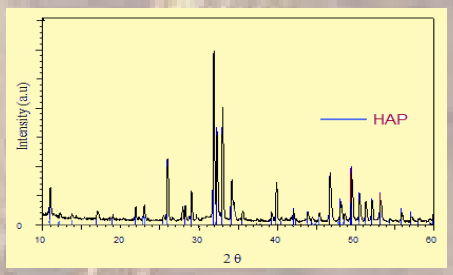
The X-ray diffraction patterns for β-TCP



XRD patterns showing the transformation of apatitic tricalcium phosphate to crystalline β – TCP that occurs at a temperature between 700 – 800 °C.

The average crystallite size values for the precursor and samples calcined between 400 – 800 °C.

Calcination temperatures (°C)	Crystallite size (nm)
precursor (apatitic tricalcium phosphate)	38
400	36
500	39.2
600	36.6
700	23.5
720	23.1
740	38.1.
750	57.1
800	91.7



The X-ray diffraction patterns for HAP

CONCLUSIONS

• Ceramic nanopowders based on hydroxyapatite and tricalcium phosphate were obtained by precipitation chemical synthesis, from salt solutions (in accordance with ASTM 09-169 files for beta – TCP and ASTM 09-0432 for HAP)
▪ Differential thermal analysis (DTA) indicate an endothermic peak at 790°C, corresponding to the transformation from precursor to β – TCP, in correlation with X-ray diffraction measurements.
▪ After calcination at 800 °C, when the beta – TCP compound (unique phase) was formed, the average crystallite sizes for nanopowders was of 91.7 nm.

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