Sintering of different hydroxyapatite nanopowders

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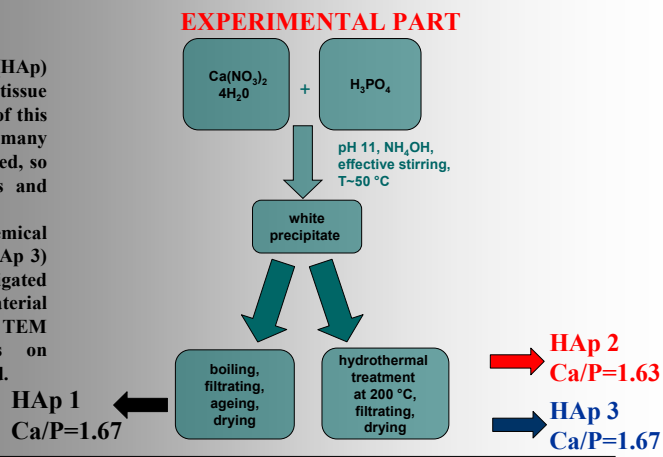
INTRODUCTION

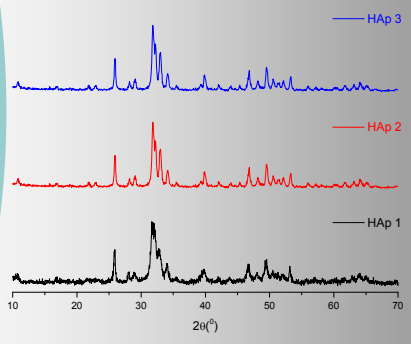
Among the large number of ceramic biometerials, dense hydroxyapatite (HAp) bioceramics belong to the most promising candidates for reconstruction of bone tissue large defects due to its combined biocompatibility and bioactivity.[1] Final quality of this material, like mechanical properties and biological response, depend on many parameters related to the powder synthesis procedure and sintering cycle employed, so it is very important to understand the connection of powder characteristics and properties of sintered materials.

In this study, three different HAp nanopowders were synthesized via chemical precipitation (HAp 1) and hydrothermal processing of precipitate (HAp 2 and HAp 3) with different Ca/P ratio of the precursors. Their sintering behavior was investigated through various two-step sintering (TSS) cycles.[2] Characteristics of starting material and sintered bioceramics are investigated through XRD, BET, FE SEM, TEM measurements. The influence of starting nanopowders characteristics on microstructural properties, density, average grain size and uniformity, is discussed.

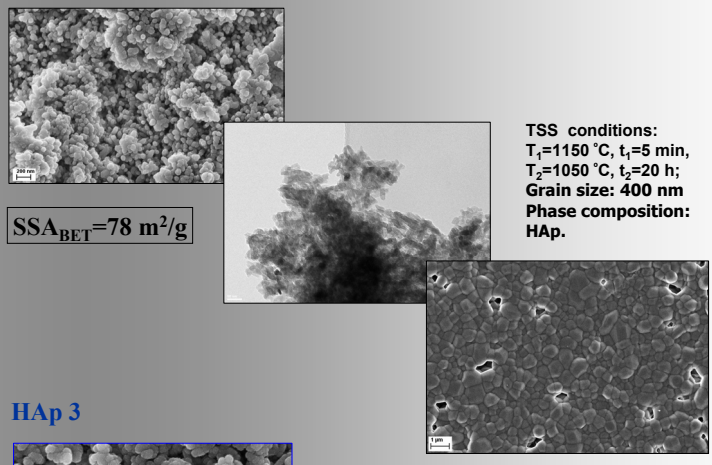
EXPERIMENTAL PART



RESULTS AND DISCUSSION



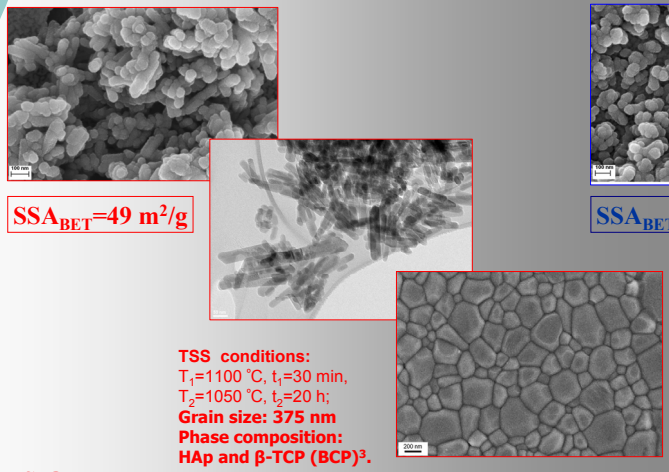
HAp 1



SSA_{BET}=78 m^2/g

TSS conditions:
T_1=1150 °C, t_1=5 min,
T_2=1050 °C, t_2=20 h;
Grain size: 400 nm
Phase composition: HAp.

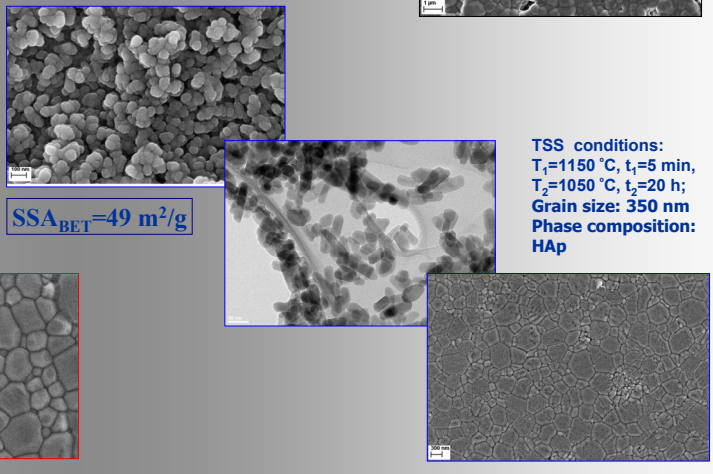
HAp 2



SSA_{BET}=49 m^2/g

TSS conditions:
T_1=1100 °C, t_1=30 min,
T_2=1050 °C, t_2=20 h;
Grain size: 375 nm
Phase composition: HAp and β-TCP (BCP)[3].

HAp 3



SSA_{BET}=49 m^2/g

TSS conditions:
T_1=1150 °C, t_1=5 min,
T_2=1050 °C, t_2=20 h;
Grain size: 350 nm
Phase composition: HAp

CONCLUSION

This study showed that hydroxyapatite nanopowders with average particle size lower than 100 nm can be successfully synthesized via both chemical precipitation and hydrothermal processing. Degree of crystalinity of hydrothermally produced materials is significanty higher, having at the same time lower SSA. Their sintering ability is strongly influenced by these parametars as well as initial Ca/P ratio. TSS approach yielded in all three cases to fine-grained ceramics, with average grain size of 400 nm or lower. Hydrothermally produced nanopowders (HAp 2 and HAp 3) exhibited better sinterability while the final phase composition is determined by initial Ca/P ratio and sintering temperature.

REFERENCES

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2. I.-Wei Chen, X.-H. Wang, Sintering dense nanocrystalline ceramics without final- stage grain growth, *Nature*, **404**, 168-71 (2000).
3. M. Lukić, Z. Stojanović, S.D. Škapin, M. Maček-Kržmanc, M. Mitrić, S. Marković and D. Uskoković, Dense fine-grained biphasic calcium phosphate (BCP) bioceramics designed by two-step sintering, J. Eur. Ceram. Soc., **31**, 1-2, 19-27, (2011).