WIDENING THE FRONTIERS IN FORENSIC IDENTIFICATION: PROBING BURNED HUMAN BONES WITH NEUTRON TECHNIQUES

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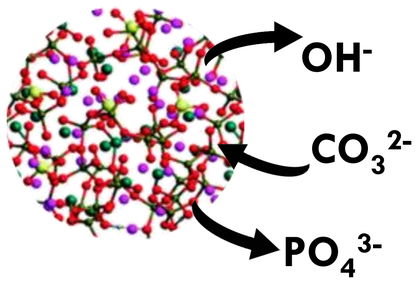
INTRODUCTION

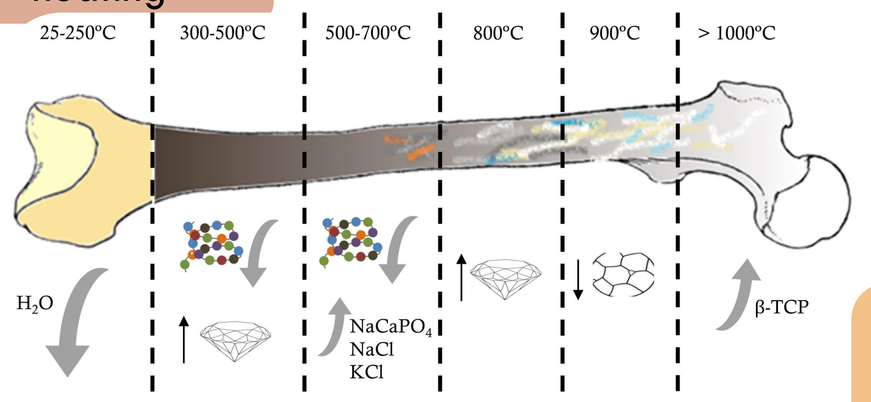
In vivo

Carbonates substitute phosphate and hydroxyl groups

Bioapatite



Exposure to heating

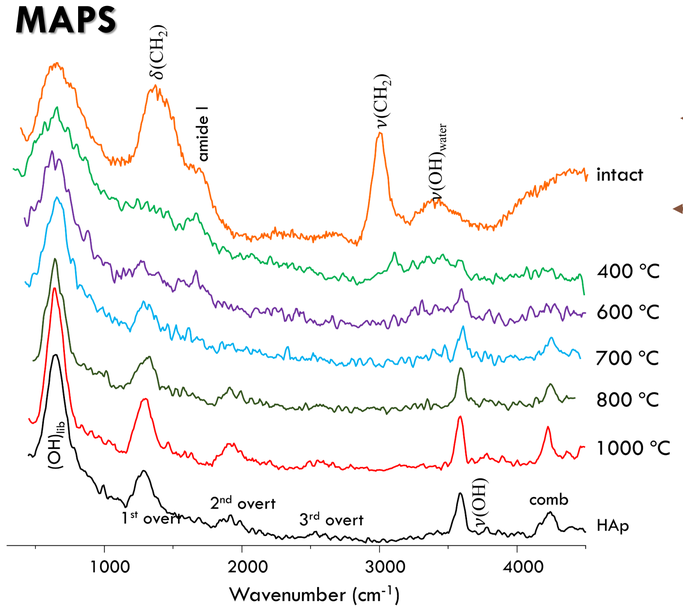


- Increase in Crystallinity
- Organic material degradation
- Changes in the content of carbonates, phosphates and hydroxyls
- Possible formation of new inorganic phases

In parallel with the macroscopic alterations: color, shape and size

Compromising the application of the Bioanthropological methods

MAPS

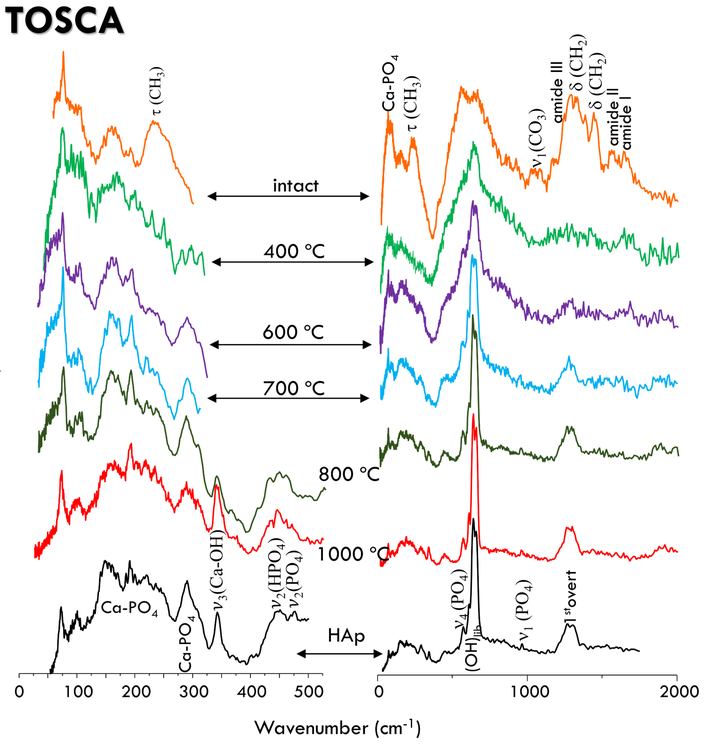


MAPS data for human femur burned 400-1000ºC, and HAp refence from NIST *

- No organic material above 600ºC
- All HAp vibrational modes observed <u>confirming the hydroxylation of bioapatite</u>
- The narrowing of the signals with increasing temperatures evidences dimensional changes of the bioapatite crystals (<u>increasing crystallinity</u>)
- Crystal lattice ($Ca\text{-}PO_4$) signals are related with the short-range order and hydrogen-bonding profile within the crystalline framework, changes in these suggest crystal dimensional alterations as temperature increased

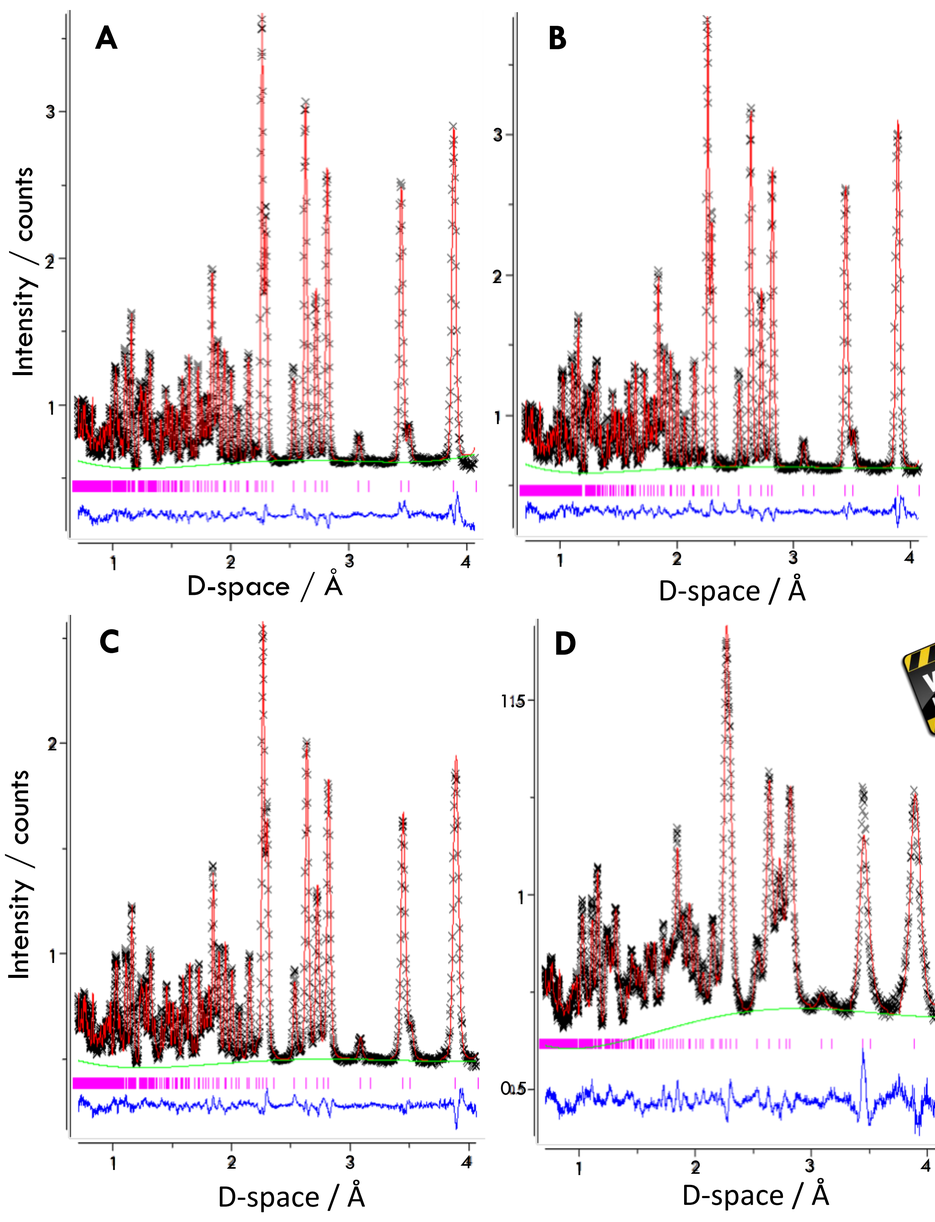
*M.P.M. Marques, et al. (2018) Heat-induced Bone Diagenesis Probed by Vibrational Spectroscopy, *Scientific Reports* **IN PRESS**

TOSCA



TOSCA data for human femur burned 400-1000ºC, and HAp refence from NIST *

RESULTS



Rietveld refinement of neutron diffraction data of human femur burned at A)1000ºC (using hexagonal hydroxyapatite as model); B) 900ºC; C) 800ºC; D) 700ºC under controlled conditions

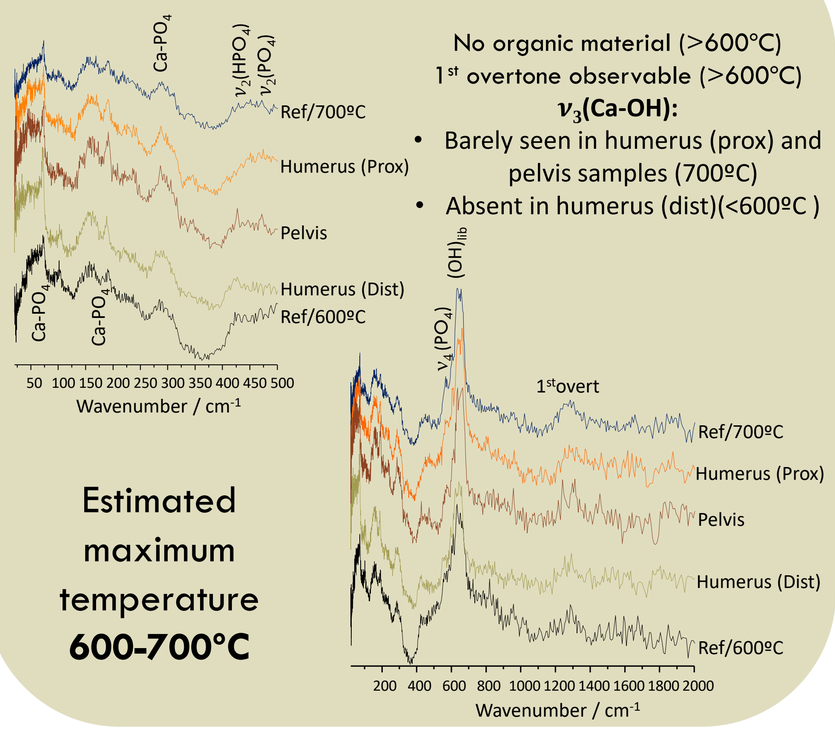
- GEM data of the bone samples burned under controled conditions fit the HAp model <u>except the 700ºC burned sample</u> because at this temperature traces of bioapatite (carbonates) is present (suggesting 700-800ºC to be a crucial interval to the understanding of the heat-induced changes within bone's submicrostructure)
- Two different models needed for the refinement at temperatures below 700ºC, <u>HAp & carbonated apatite</u>

SYSTEMATIC ERRORS in this model unable to complete the refinement

CONCLUSIONS

- Neutron techniques provided unprecedented and valuable information on heat-induced changes within bone's inorganic structure and composition
- Dimensional changes in bioapatite´s crystal definitely occurring between 700-800ºC
- The results are not compromised by the presence of contaminants
- INS at TOSCA allowed us to estimate the maximum temp reached in the caravan's experiments

Simulation of a real fire in a caravan



No organic material (>600ºC)
1st overtone observable (>600ºC)
ν_3(Ca-OH):
- Barely seen in humerus (prox) and pelvis samples (700ºC)
- Absent in humerus (dist)(<600ºC)

Estimated maximum temperature **600-700ºC**

EXPERIMENTAL

- Modern human femora and humeri were sectioned and burned in an electric oven for 120min. at 400 to 1000ºC (100ºC increment)
- Emperimentally burned samples under controlled consitions were grinded and analysed through INS (MAPS and TOSCA) and neutron diffraction (GEM), at ISIS-Rutherford Appleton Laboratory.
- GEM data was analysed with GSAS-EXPGUI to perform the Retveld refinement
- Different regions of human humeri and pelvis were burned in a caravan in order to simulate a real fire situation. These samples were also grinded and analysed on TOSCA.

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STFC Rutherford Appleton Laboratory – ISIS Neutron Facility, RB 1400034, 1510054, 1710008