

9th International Bone Diagenesis Meeting

21-24 September 2021 Évora - Portugal

Programme and Abstracts









Content

Sponsors	2
Conference organizers and scientific committee	3
Conference location	4
Presentation format	5
Scientific program	6
Meeting schedule	7
Abstracts – Oral presentations	11
Abstracts – Poster presentations	45
List of BD2021 participants	62

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Conference location

BD2021 will be held in the auditorio of the University of Évora, in the historical Colégio de Espirito Santo from September 21-24 2021.

(Main entrance Rua do Cardeal Rei)





Presentation Format

Instructions for talks and posters are the following:

* Talks: 15 min maximum (you will have 20 min including questions)

* Posters: one flash talk of 3 min maximum (1-2 slides max) <u>AND</u> one poster A0 Portrait in pdf (which should not exceed 5MB) that we will display on a virtual platform in order to be visible from all the participants, whether online or present in Évora.

Also, we would be grateful if you could provide us:

* For online presenters: one version of the **pdf of your talk** and/or **pdf of your flash talk the day before your presentation** (the latest). We expect the presentations to be given live but in case you encounter any problem sharing your presentation, we will display your pdf and advance the slides as you talk.

* For onsite presenters: your presentation will be uploaded to the conference computer before the morning or afternoon sessions.

Scientific Sessions

- 1. Taphonomy, preservation, environment
- 2. Preservation of organic molecules in bones in deep times
- 3. Advance in sample preparation, analytical technique and experiments on skeletal tissues
- 4. Non-traditional and new chemical proxies applied to ancient bones and teeth
- 5. Diagenesis in forensics

Invited Speakers

<u>Tim Thompson</u>, Professor of Applied Biological Anthropology and Associate Dean (Academic) in the School of Health & Life Science, Teesside University, UK Keynote session 5

<u>Valéry Zeitoun</u>, Researcher at the Centre de Recherche sur la Paléobiodiversité et les Paléoenvironnements (CR2P), Sorbonne-Université UMR7207, Paris, France Guest speaker (Tribute to Alain Person)

<u>Julia Lee-Thorp</u>, Emeritus Professor of Archaeological Science, Research Laboratory for Archaeology, Oxford, UK Guest speaker (state of the art and proceedings of the meeting)

Meeting Schedule

(**Time is Lisbon time**; respective time for the participants who will present remotely is provided)

Tuesday, 21 September

18:00-20:00 Registration and welcome reception

Wednesday, 22 September

08:30-09:10	Registration
09:10-09:30	Opening
Session 1 –	Taphonomy, preservation, environment Chair – Christophe Snoeck
09:30-09:50	Oleg Shilovsky et al. (presented by Daria Kiseleva) (+5h GMT) Diagenesis in Permian tetrapod bones from the Kotelnich and Sundyr localities (Russia)
09:50-10:10	Kévin Rey et al. δ ¹⁸ O _p geographical variability within the South African Karoo Basin during Permo-Triassic based on tetrapod apatite
10:10-10:30	loannis Kontopoulos et al. (+2h GMT) Linking bone histology to burial type at St. Rombout cemetery (Mechelen, Belgium)
10:30-10:50	Kirsten Mandl et al. (+2h GMT) The diagnostic utility of μCT in comparison to transverse thin sections for assessing bioerosion in archaeological bone samples
10:50-11:20	Coffee break
11:20-11:40	Fiona Brock et al. (+1h GMT) Investigating diagenesis of archaeological bones from Etton Causewayed Enclosure and Star Carr, UK
11:40-12:00	Ina Reiche et al. (+2h GMT) Comparative trace element analysis using non-invasive microPIXE/PIGE of raw mammoth ivory and worked personal ornaments from different European Palaeolithic key sites
12:00-12:20	Daria Kiseleva et al. (+5h GMT) The diagenesis of subfossil holocene reindeer bones and antlers (Mammalia: Artiodactyla, <i>Rangifer tarandus</i> L., 1758) from the Ust'-Polui archaeological site in Northern Eurasia
12:20-12:40	Maura Pellegrini et al. Stable isotope relationship between biological and sedimentary carbonates in karstic archaeological sequences
12:50-14:00	Lunch Time
14:00-14:20	Paul Ullmann (-4h GMT) Establishing criteria to differentiate mass-death and attritional fossil bonebeds based solely on trace element signatures

14:20-14:40	Rachel Laker (-5h GMT) Authigenic variation as a function of exposure duration in the marine Eocene vertebrates of Wadi Al-Hitan, Egypt: diagenetic insights into the controls of bone preservation
14:40-15:00	Giovanni Magno and Alberto Zanatta (+2h GMT) The tannization of human remains as a preservation technique at the Morgagni Museum (Padua, Italy)
15:00-15:10	Poster Session 1
	Annalisa Ferretti et al. (+2h GMT) Dead, fossil or alive
	Rhy McMillan et al. (-7h GMT) Diagenetic impacts on the Color Alteration Index and geochemical characteristics of conodonts
15:20-15:50	Coffee break
Session 2 –	Preservation of organic molecules in bones in deep times Chair – Federico Lugli
15:50-16:10	Tamara Leskovar et al. (+2h GMT) ATR-FTIR spectroscopy as pre-screening technique for the DNA preservation in human skeletal remains
16:10-16:30	Ismael Rodriguez Palomo et al. (+2h GMT) Site specific deamidation of Asn and GIn assessed by analysis of ancient collagen
16:30-16:40	Poster Session 2
	Christina Ryder et al. (-6h GMT) Saving Old Bones: an application of near-infrared spectroscopy to prescreen bone for collagen
	Dimitris Iliopoulos and Elizabeth Stathopoulou Collagen extraction and evaluation of organic preservation in Greek & Cypriot fossilised bones
Session 3 –	Advance in sample preparation, analytical technique and experiments on skeletal tissues Chair – Lisette Kootker
16:50-17:10	Thomas Tütken et al. In vitro alteration of mammal teeth in isotopically enriched tracer solution: assessing diagenetic stability of bioapatite δ ¹⁸ O values
17:10-17:30	Christine France et al. (-4h GMT) Establishing robust indicators of bone, dentin, and enamel bioapatite preservation using ATR-FTIR

Thursday, 23 September

Session 3 – Advance in sample preparation, analytical technique and experiments on skeletal tissues Chair – Lisette Kootker				
09:05-09:25	Rachel Wood et al. (+10h GMT) The use of radiocarbon dating to assess the impact of diagenesis on δ^{13} C analysis of tooth enamel			
09:25-09:30	Poster session 3 Rachel Wood et al. (+10h GMT) Novel pretreatment strategies to remove contamination from tooth enamel for radiocarbon dating			
09:30-09:50	Laura van der Sluis et al. (+2h GMT) Using XAD resin to remove synthetic contamination from archaeological bone prior to radiocarbon dating			
09:50-10:10	Laurent Tranchant et al. (+2h GMT) Determination of the diagenetic and biogenic elements in Palaeolithic mammoth ivories and bones at PUMA / SOLEIL and NewAGLAE / C2RMF			

10:10-10:30	Tanya Smith et al. (+10h GMT) Developmentally-informed assessments of diagenesis in teeth
10:30-10:50	Frederik Tielens and Flavio Siro Brigiano Biological calcifications characterized at the molecular level
10:50-11:20	Coffee break
11:20-11:40	Hannah James et al. The potential for laser ablation strontium isotope analysis on cremated petrous bones
11:40-12:00	Barbara Veselka et al. Penetrating the pars petrosa: investigating the influence of bone turnover and diagenesis on strontium isotope ratios related to childhood mobility
12:00-12:20	Jason Laffoon and Lisette Kootker Investigating the petrous bone (pars petrosa) as a reservoir of biogenic strontium in unburnt archaeological skeletal remains
12:20-12:40	Maura Pellegrini et al. Newest technology in the analysis of isotopes archaeological remains
12:50-14:00	Lunch time
14:00-14:20	Poster Session 3
	Daria Kiseleva and Elizaveta Pankrushina (+5h GMT) Temperature dependent Raman spectroscopy of modern and archaeological human enamel and dentin
	Héctor Del Valle et al. Assessing the effects of two nanoparticle based consolidants through diagenetic parameters
	Mandi Curtis et al. (+1h GMT) Microscopic Sampling of Bone Collagen
	Diana Moreiras Reynaga et al. (-7h GMT) Effects of consolidants and their removal by polar solvents on the stable isotope compositions of an archaeological caribou bone
Session 4 –	Non-traditional and new chemical proxies applied to ancient bones and teeth Chair – Vincent Balter
14:20-14:40	Noreen Tuross and Linda Reynard (-4h GMT) Perfect as the enemy of good: organic hydrogen and inorganic oxygen isotopes in bones and teeth
14:40-15:00	Federico Lugli et al. Life histories told by fossil teeth: achievements, limitations, and pitfalls
15:00-15:20	John Samuelsen (-5h GMT) A Test of Trace Element Analysis for Detecting Pb and Sr Contamination in Ancient Human Tooth Enamel
15:20-15:50	Coffee break
15:50-16:10	Poster Session 4
	Mateusz Michailow et al. (presented by Annalisa Ferretti) (+2h GMT) Guess who's coming to dinner: Ca isotopes and trace elements of dinosaur tooth enamel from the Late
	Cretaceous of Montana

	Jiménez-Morillo et al. Pyrolysis compound-specific isotope analysis (δ ¹³ C and δ ¹⁵ N PY-CSIA): a novel analytical approach for archaeological studies Marta Hlad et al. Iron, Copper, and Zinc isotope ratios and concentrations in calcined bones: preliminary results and prospects
16:30-22:30	Visit of Alentejo + wine tasting + conference dinner

Friday, 24 September

Session 5 – Diagenesis in forensics Chair – Hannah James				
09:10-09:50	Keynote – Tim Thompson (+1h GMT) Do bone diagenesis studies have a role to play in forensic and medico-legal investigations?			
09:50-10:10	Aida Galiacho Gutiérrez and Gordon Turner-Walker (+2h GMT) Bacterial bioerosion of bones is a post-skeletonisation phenomenon and appears contingent on soil burial			
10:10-10:30	Emese Végh et al. Differences in structure, major and trace elemental concentrations in bioapatite during the late postmortem interval pre- and post-burning			
10:30-10:50	Partha Pratim Biswas and Gordon Turner-Walker (+8h GMT) Physico-chemical and mineralogical transformation, and dissolution behaviour, of experimentally cremated bones			
10:50-11:20	Coffee break			
11:20-11:40	Lisette Kootker et al. Towards a better understanding of the effects of leaching and diagenesis on the antemortem ⁸⁷ Sr/ ⁸⁶ Sr of human hair keratin			
11:40-11:50	Poster Session 5			
	Elizavet Stamataki et al. From diagenesis to cremation: The application of FTIR to investigate post-mortem alteration of burnt bones			
	Edda Guareschi (+8h GMT) The role of marine bioerosion in the diagenesis of terrestrial bone: a pilot study			
11:50-12:10	Valéry Zeitoun et al. (Tribute to Alain Person) (+2h GMT) Multifactorial approach to describe early diagenesis of bones: the case study of the Merovingian Cemetery of Saint Linaire (France)			
12:10-12:50	General discussion and conclusion with Julia Lee-Thorp			
12:50-13:10	Closing remarks			
13:10-14:10	Lunch time			
14:30-18:30	Visit of archaeological sites			

Abstracts – Oral presentations

Listed alphabetically

Presenting authors are underlined

Physico-chemical and mineralogical transformation, and dissolution behaviour, of experimentally cremated bones

Biswas Partha Pratim¹ and Turner-Walker Gordon²

1 National Cheng Kung University, Department of Earth Science, Tainan, Taiwan

2 National Yunlin University of Science and Technology, Graduate School of Cultural Heritage Conservation, Taiwan

In our study, we explored different synchrotron-based spectroscopic and imaging techniques, including FTIR, XRD, TXM, and XPS to investigate the physicochemical properties of cremated bones (300-1200 °C) and their dissolution behaviour at different pHs (4 and 6). The mineral transformation from B-type to A,B-type carbonate substitution occurred mainly <700 C, while the transformation from carbonated hydroxyapatite (CHAp) to more mineralogically- and chemically stable HAp occurred >800 °C. The signal for the structural OH- band increased with increasing temperatures, due to the simultaneous reverse reaction of inorganic CO³²- and H2O. The increasing band ratio of (v1+v2)-PO4³-/v³-CO3²- with increasing temperature could be explained by the thermal decomposition of inorganic CO3²and its reaction with hydrogen-phosphate (2HPO4²- + CO3²- \rightarrow 2PO4³- + H2O + CO2), whereas organic C decomposition occurred within the range 300-600 °C. As the temperature increased up to 1100 °C, the centroid of the v3c PO4³⁻ peak exhibited a blue-shift (FTIR, 1029-1051 cm⁻¹) and red-shift (XPS), resulting from the rearrangement of P-O bonds in the vacancies vacated by evolved H2O and CO2. The surface consolidation was obvious at 700 °C. The pH of immersed cremated bone dominated over the initial pH of aqueous solutions over 140 hours' incubation. The P dissolution was relatively high for 300>500 °C cremated bone at both pH 4 and 6, due to the anaerobic degradation of organic C which maintained the solution pH acidic. The pH of 500 °C cremated bone solutions (4 and 6) achieved nearequilibrium within an hour and remained fairly constant (≈ 7.5) through the incubation period. A higher dissolution was observed in 700 °C cremated bone at higher pH 6 (6.03±0.03 ppm), compared to that at pH 4 (3.489 ±0.07 ppm) due to the formation of a negative surface complex at pH<8.2, which readily occurred in the pH 6 solution due to its higher alkaline nature may prohibit the PO4³⁻ re-adsorption. Our results have implications for isotopic and radiometric dating analyses on cremated bones.

Investigating diagenesis of archaeological bones from Etton Causewayed Enclosure and Star Carrm UK

Brock Fiona¹, Loy Charlotte¹, Rogers Keith¹, Snow Tim², Greenwood Charlene³ and Hiller Bardsley Jen⁴

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2 Diamond Light Source Ltd, Diamond House, Harwell Campus, Didcot, Oxfordshire OX11 0DE, UK

3 School of Chemical and Physical Sciences, Keele University, Keele ST5 5BJ, UK

4 Higher Education & Science Directorate, Cultural Engagement, British Council, 1 Redman Place, London, E20 1JQ, UK

Diagenesis of archaeological bones proceeds via a complex combination of chemical, physical and/or microbial processes over many millennia. These processes are influenced by the depositional environment, including fluctuations in pH, mineral availability and water table.

This study investigates diagenetic alterations to the organic (collagen) and mineral phases of bones from two UK archaeological sites, Etton Causewayed Enclosure (Cambs) and Star Carr (North Yorkshire).

Bones from the Neolithic gravel site at Etton exhibit unusual staining patterns, including iron-rich layers underneath the bone surface and manganese speckling throughout the bone, alongside variable collagen preservation. In comparison, specimens from the peat-rich Mesolithic site at Star Carr exhibit homogeneous dark brown staining throughout the bone. Zinc and manganese are detected within these samples, as are regions of iron in association with sulphur.

A range of techniques, including FTIR, SEM, and simultaneous synchrotron SAXS, WAXS and XRF were employed to analyse bones from both sites, to investigate changes in bone mineral crystallinity and elemental composition and their relationship to collagen preservation.

Establishing robust indicators of bone, dentin, and enamel bioapatite preservation using ATR-FTIR

France Christine A.M.¹, Sugiyama Nawa² and Aguayo Esther²

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Bioarchaeological analyses of bones and teeth require accurate information about preservation to determine diagenetic state and make appropriate interpretations of diet, provenance, demographics, health, and life histories. This study used Attenuated Total Reflectance Fourier Transform Infrared spectroscopy (ATR-FTIR) to examine the bioapatite mineral in archaeological bones and teeth. ATR-FTIR is rapidly becoming a preferred method for inorganic bioapatite analysis due to its speed, minimal sample requirements (< 1 mg), and relative user-friendliness. However, parameters defining well-preserved samples are variable between laboratories, are not necessarily comparable between raw and chemically pretreated samples, and are particularly lacking for teeth. We examined a large suite of modern and archaeological human and deer bones and teeth to examine differences between modern, well-preserved archaeological, and poorly-preserved archaeological materials; examine inter-species differences; compare raw and chemically processed samples; increase the body of dentin and enamel data; and establish an index of ATR-FTIR peak height ratios (C/P, IRSF, C/C, BPI, API) beyond which bioapatite is likely altered. Results show peak height ratios were similar between species. Raw and processed samples must be considered separately as chemical removal of secondary carbonates can alter the crystallinity. The C/P ratio was the most distinctive between well- and poorly-preserved processed samples. The IRSF is the best ratio to identify poorly-preserved bioapatite in raw samples. However, the IRSF in raw samples will classify a small number of well-preserved samples with the poorlypreserved group. Results from this study are combined with reported literature ATR-FTIR peak height ratios to provide a distinct range of values, which we term a preservation index, to distinguish between well- and poorly-preserved archaeological bone, dentin, and enamel.

Bacterial Bioerosion of Bones is a Post-skeletonisation Phenomenon and Appears Contingent on Soil Burial

Gutiérrez Galiacho Aida^{1,2}, Turner-Walker Gordon³

- 1 Unitat d'Antropologia Biològica, Departament de Biologia Animal, Biologia Vegetal i Ecologia, Universitat Autònoma de Barcelona, Spain
- 2 Grup de Recerques de les Terres de Ponent, the Institut d'Estudis llerdencs and Universitat Autònoma de Barcelona, Spain
- 3 Graduate School of Cultural Heritage Conservation, National Yunlin University of Science & Technology, Taiwan

The physico-chemical post-mortem changes that take place during the decomposition of animal soft tissues are of obvious interest to forensic scientists. As a result, considerable advances have been made in understanding the interplay of these changes, and the timescales over which they operate.

Similarly, the changes to the chemistry and microstructure of bones, post-skeletonisation, are of interest to archaeological scientists. The past decades has seen tremendous advances in the recovery and interpretation of trace evidence from archaeological bones and teeth: e.g. trace elements, stable isotopes and biomolecular evidence such as DNA. These advances have been matched by an improved understanding of how diagenesis may impact endogenous evidence, often through field and laboratory studies.

One of the most important mechanisms in the post-depositional degradation of bones and teeth is microbial bioerosion, particularly by bacteria. The question of the origin of these bacteria, and why some archaeological bones are bioeroded while others are not has received considerable attention in recent years. The assertion that the bacteria responsible originate in the gut, and infiltrate bones tissues during the putrefactive stages of decay, has led to a number of claims as to what can be inferred by the presence or absence of these tunneling bacterial. Such claims include evidence for human mummification, identification of stillborns and infanticide, animal sacrifices, etc.

Here we present histological examinations of bones from field experiments using pig carcasses interred in brick lined tombs, a forensic case of a body exposed on a concrete floor for ten years, together with de-fleshed, bovine bones buried in tropical soils for 1-10 years. None of the decomposing pig carcasses, nor the human corpse, showed any signs of bacterial tunneling in their bones. By contrast, the bones buried directly in soils showed evidence of microbial bioerosion after as little as one year.

The potential for laser ablation strontium isotope analysis on cremated petrous bones

James Hannah^{1,2,3}, Wood Rachel^{1,2}, Frieman Catherine², Grün Rainer^{1,4}, Valera Antonio, Enge Gabriel¹ and Knowles Brett¹

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Laser ablation multi collector inductively coupled plasma mass spectrometry (LA-MC-ICP-MS) allows for fast and targeted ⁸⁷Sr/⁸⁶Sr analysis on archaeological material. A method for applying laser ablation on tooth enamel has been developed and allows for analysis spots 200µm square and ~20µm deep. The petrous bone has emerged as an alternative target for Sr analysis in cremated remains, and the differing tissue turnover rates within this bone may allow for isotopic histories within a person to be uncovered. The small spot size of laser ablation could allow for multiple measurements on a variety of tissues within the petrous bone structure.

This paper will present the use of LA-MC-ICP-MS on cremated petrous bones alongside human teeth from individuals buried at several Portuguese sites. Interesting patterns of ⁸⁷Sr/⁸⁶Sr variation within bones and within measurements of the same spot emerged. Methodological challenges and data corrections will also be discussed.

The diagenesis of subfossil holocene reindeer bones and antlers (Mammalia: Artiodactyla, *Rangifer tarandus* L., 1758) from the Ust'-Polui archaeological site in Northern Eurasia

<u>Kiseleva Daria</u>¹, Kosintsev Pavel², Shagalov Evgeny^{1,3}, Chervyakovskaya Maria¹, Ryanskaya Anastasia¹, Pankrushina Elizaveta¹ Lepekha Svetlana¹, Cherednichenko Nadezhda¹ and Bachura Olga²

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One of the main topics addressed in the archaeological studies is the relationships between human mobility patterns and reindeer migrations. The migratory herds of reindeer demonstrate clear cyclical seasonal movements, moving at rapid rates over large areas on specific trajectories, with individuals returning to the same locations year after year. ⁸⁷Sr/⁸⁶Sr isotopic ratios in fossil and modern bone and dental tissues have been successfully used for the reconstructions of human and animal mobility. Nevertheless, the diagenetic changes occurring almost immediately after the death and burial of an individual in a sediment can affect the correctness of the isotopic signatures.

The Ust'-Polui sanctuary settlement is one of the most important archaeological sites of the Bronze-Iron Age in the Yamal region of Russia with its northeastern part represented by permafrost deposits. The assemblage of faunal remains from the Ust'-Polui excavations includes a huge collection of reindeer remains (bones and antlers). The reindeers from the north of Western Siberia migrate in spring (April - June) from south to north from northern taiga to tundra zone via forest-tundra, and backwards in autumn. With constant migration routes within one or close geochemical provinces, the ⁸⁷Sr/⁸⁶Sr ratio in reindeer bones and antlers should be the same, while when migrating through different provinces, it can vary significantly.

The fragments of four skulls with unshed antlers, two skulls without antlers, four shed antlers from different individuals from the Ust'-Polui site and a recent unfossilised skull and antler of a modern reindeer from the Polar Urals were studied.

Based on a study of the elemental, isotopic and structural characteristics of subfossil reindeer bones and antlers by SEM-EDS, Q-ICP-MS and MC-ICP-MS, XRD, IR and Raman spectroscopy the effect of diagenetic changes on skeletal tissues was estimated. The features of fossilization under the permafrost taphonomic conditions were revealed.

Linking bone histology to burial type at St. Rombout cemetery (Mechelen, Belgium)

<u>Kontopoulos Ioannis^{1,2}</u>, Van de Vijver Katrien², Robberechts Bart⁴, von Tersch Matthew¹, Turner-Walker Gordon⁵, Penkman Kirsty⁶ and Collins Matthew J.^{7,8}

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- 4 Archaeology Service, Mechelen, Belgium
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- 7 Centre for Evogenomics, Globe Institute, University of Copenhagen, Copenhagen, Denmark
- 8 McDonald Institute for Archaeological Research, Department of Archaeology, University of Cambridge, Cambridge, United Kingdom

Post-mortem modifications in bone microstructure arise from a multiplicity of factors relating to whole-body decomposition and the external depositional environment. These modifications (microscopic foci of destruction, cracking, staining) have been attributed to various physical, chemical and biological processes but their connection to the early post-mortem history of the body is still not well understood. Recently, some researchers have attempted to link e.g. the lack of microscopic foci of destruction to specific treatment of the body (mummification). However, the conflicting evidence on the origins of the bacteria responsible for the post-mortem erosion of bone, and the limited data from experimental studies hinder deeper understanding.

This study compares histological preservation in bones from different burial types (single coffin, single wrapped, and multiple burials) in an effort to unravel the histotaphonomy-to-funerary practices relationship. We collected data from 61 specimens (20 individuals) from the medieval/post-medieval (10th-18th c. CE) cemetery of St. Rombout (Belgium). Using optical and scanning electron microscopy, we provide an insight into the relationship between the burial type and the histological modifications observed in archaeological bone. We further highlight the implications for the origins of bacteria responsible for bioerosion, and we discuss the capabilities and limitations of commonly used microscopy techniques in the field of histotaphonomy in an effort to increase awareness in future studies.

Towards a better understanding of the effects of leaching and diagenesis on the antemortem ⁸⁷Sr/⁸⁶Sr of human hair keratin

Kootker Lisette M.¹, Ammer Saskia T.M¹. and Davies Gareth R.¹

The application of isotope analysis to aid human identification processes in forensic cases has become more common. In contrast to dental enamel, hair and nail keratin can potentially provide valuable insights into more recent diet and health status (e.g., C-N-S), and recent geolocation (e.g., Sr-Pb). Recent studies, however, have shown that it is difficult to separate diet-related endogenous Sr-Pb signatures from environment-specific exogenous signatures in human hair keratin. Moreover, the effectiveness of hair keratin as a means of evidence for reconstructing recent mobility patterns depends greatly on its susceptibility to diagenetic alterations (e.g. burial context). To gain a better understanding of the optimal pre-treatment of hair keratin samples and the applicability of hair keratin samples in various forensic contexts, an experimental leaching project was combined with actualistic experiments at the ARISTA (Amsterdam Research Initiative for Sub-surface Taphonomy and Anthropology) facility in the Netherlands. The data show a close connection between hair colour, i.e., the amount of melamine present in the sample, and the effectiveness of the leaching protocol on retrieving the antemortem Sr isotope composition of hair keratin. More importantly, following previously reported data [1] from FARF (Forensic Anthropology Research Facility) in Texas, US, the data from the actualistic experiments at ARISTA accentuate the extreme difficulty or even impossibility to recover the hair's biogenic Sr isotope compositions after 306 days of burial, despite the application of the most effective pre-treatment protocol (2:1 MC-MQ-0.1M HCL-MQ).

[1] Kootker, L.M., von Holstein, I.C.C., Broeders, J., Wescott, D.J., Davies, G.R., Mickleburgh, H.L., 2020. The effects of decomposition and environment on antemortem H-Pb-Sr isotope compositions and degradation of human scalp hair: Actualistic taphonomic observations, Forensic Science International 312, 110336.

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Investigating the petrous bone (*pars petrosa*) as a reservoir of biogenic strontium in unburnt archaeological skeletal remains

Laffoon Jason¹ and Kootker Lisette M.²

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Strontium isotope (87Sr/86Sr) analysis of skeletal remains has become a powerful tool in archaeological studies of human migration and mobility. Owing to its resistance to postmortem alteration, dental enamel is the preferred sampling material used for ⁸⁷Sr/⁸⁶Sr analysis in bioarchaeological research, although recent research has demonstrated that cremated bone is also generally resistant to diagenesis. This paper presents the results of a pilot study exploring the potential of unburnt (uncalcined) petrous bone (pars petrosa) as a reservoir of biogenic strontium, as the otic capsule or bony labyrinth, within the petrous bone is extremely dense and often contains high quantities of endogenous aDNA. This study focuses on an individual from a colonial era (18th century) site on the island of Saba in the Caribbean for whom previous enamel ⁸⁷Sr/⁸⁶Sr results had indicated nonlocal origins. This small island (13km²) has been extensively mapped for bioavailable strontium isotopes (n=50) with 87Sr/ 86Sr varying between ~0.706 to 0.709, whereas enamel ⁸⁷Sr/⁸⁶Sr (n=3) ranged from 0.7104 to 0.7112. Based on osteological analysis, cultural context, and these elevated enamel ⁸⁷Sr/⁸⁶Sr ratios this individual was identified as an enslaved, first generation (forced) migrant from Africa. For the current study, multiple locations (n=4) on the petrous were sampled and measured for strontium isotope composition. All four petrous ⁸⁷Sr/⁸⁶Sr ratios (0.711-0.712) are consistently and considerably higher than the local bioavailable range, and very similar to the enamel ⁸⁷Sr/⁸⁶Sr. These results provide initial evidence that unburnt petrous bones may preserve biogenic strontium, at least in this specific burial context. While more research in diverse burial conditions is needed to validate this observation, if confirmed it would have broader implications for sample selection strategies in bioarchaeological studies using the strontium isotope method.

Authigenic variation as a function of exposure duration in the marine Eocene vertebrates of Wadi Al-Hitan, Egypt: diagenetic insights into the controls of bone preservation

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The infilling material of fossil bones can vary significantly among specimens from the same formation, suggesting that early-diagenetic, authigenic processes may be as or more important to preservation than pervasive, late-diagenetic lithification. This suggests that early-diagenetic conditions, and factors such as exposure duration or burial rate, may drive preservation.

Sequence stratigraphy can provide a framework to compare burial rates and exposure duration: bones that experience rapid burial from steady sedimentation should have a simple infills, due to a shorter window for alteration, compared to bones from the maximum flooding surface (MFS), which should experience prolonged exposure at or near the sediment-water interface in the taphonomically active mixed layer; the expected result is chemically complex multigenerational infills and higher taphonomic damage. Bones from a sequence boundary (SB) should experience reworking and subaerial exposure, and thus show signs of erosion, drying, or oxidized mineral infills.

To test these predictions in a fully lithified record, whale bones from different positions within a 3rd-order depositional sequence (Peters et al. 2009 Palaios) from the Eocene of Egypt (Wadi Al-Hitan/Valley of the Whales) were analyzed for taphonomic heterogeneity.

The dominant authigenic composition does vary significantly. Samples from within systems tracts are dominated by partial or complete iron-rich calcite infills of varying textures; those from the MFS yield complex geopetal iron primary infill, compaction, and a celestine-dominated secondary infill; and those from the SB are dominated by siliciclastic sediment infill cemented by calcite, with rimmed iron-oxides and a significant amount of microscopic bone fracturing.

This variation suggests that early post-depositional conditions and rapid authigenesis survive late-diagenetic overprinting and can influence the preservational outcomes of fossil bone in marine settings, and that sequence stratigraphy provides a valuable independent framework for analyzing diagenetic variation on timescales beyond the reach of Holocene records.

ATR-FTIR spectroscopy as pre-screening technique for the DNA preservation in human skeletal remains

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Skeletal remains are commonly used for various analyses, including DNA. As the remains are subjected to numerous taphonomic processes after death, their chemical and physical structure is altered. The success and integrity of DNA analyses therefore depend on the state of preservation of the sample.

Several techniques have been tested as preliminary screening methods for assessing DNA preservation in skeletal remains. Although some show potential, they are financially challenging and time consuming and have limited predictive power. In our research, we are investigating relatively fast and inexpensive ATR-FTIR spectroscopy with further data manipulation as a potential pre-screening technique. We seek to correlate spectra and chemometric indices with the quality and quantity of DNA in the samples, also considering the skeletal element, absolute age, and environmental conditions to which the remains have been exposed.

In our research we studied human bones and teeth derived from archaeological, WWII or forensic contexts. The samples were cleaned and pulverised according to established methodological procedures for DNA extraction. The DNA was extracted and quantified, while the remaining powder was analysed by ATR-FTIR spectroscopy. The spectra obtained were manipulated to extract physical and chemical information, which were then subjected to statistical analysis, machine learning, and prediction algorithms.

Although there are still many questions that need to be answered, the results obtained suggest that the combined ATR-FTIR and statistical analyses have a high potential as a pre-screening method for assessing DNA preservation in human skeletal remains.

Life histories told by fossil teeth: achievements, limitations, and pitfalls

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The advent of laser ablation mass spectrometry (LA-(MC-)ICPMS) kicked off the microchemical investigations of mineralized tissues at unprecedented spatial-resolution. In particular teeth – due to their high resistance of enamel to diagenesis and their characteristic micro-structural features that register events at a daily scale – can reveal a wealth of information about past life events.

Yet, the current scant knowledge about specific elements' behaviour during mineralization and possible diagenetic overprints hamper a seemingly-straightforward interpretation of '*invivo*' elemental/isotopic signals. Utilizing modern as well as archaeological/fossil human and faunal teeth ranging from Medieval times to the Middle Palaeolithic, we showcase the flaws and merits of such investigations when combining LA-(MC-)ICPMS and histomorphometric analyses.

In detail, we suggest that for (sub)fossil specimens a strong control on the micro-elemental distribution of diagenetic markers can help elucidating the 'goodness' of the retrievable biogenic information. Specifically, uranium – being highly soluble in water as uranyl yet initially present at the ng/g level in modern teeth – is a key diagenetic indicator, as it is readily adsorbed after burial due to the high distribution coefficient in hydroxyapatite. Indeed, U matches the fluctuations observed for some biogenic elements in enamel compositional profiles. Barium, in particular, seems to frequently co-vary with U, shedding doubt on its reliability as a dietary indicator. Instead, our data indicate that Sr, thanks to higher concentrations relative to Ba and lower susceptibility to diagenesis, is less commonly altered, better understood in modern samples and thus overall reflects better shifts in infant diets – i.e. from (breast)milk to weaning foods – rather than Ba. What is more, Sr also provides time-resolved mobility information via ⁸⁷Sr/⁸⁶Sr isotope ratio profiles.

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The tannization of human remains as a preservation technique at the Morgagni Museum (Padua, Italy)

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Lodovico Brunetti (1813-1899), professor of pathological anatomy at the University of Padua (1855) and founder of the Morgagni Museum of Pathological Anatomy (1860s), believed that anatomical preparations were essential for the practice and teaching of pathological anatomy. At his arrival in Padua in 1855 there were about three hundred specimens made by other professors of medicine, including some by Giovanni Battista Morgagni, preserved either in liquid or dry. These conservation methods did not satisfy Brunetti, as they drastically altered the shape of the anatomical pieces (reduced by mummification and dilated in liquid), thus he decided to create a new method called "tannization", for the use of tannic acid.

Brunetti's new method was based on dissection and injection techniques, and it had the substantial advantage of maintaining unchanged the shape and texture of the anatomical specimens, even microscopically, as well as being not so expensive. Another important advantage consisted in the fact that the different stages of the preparation could be put into practice even at different times and at a considerable distance from each other. His specimens seemed to be mummified, but they maintained a remarkable elasticity and softness, as well as almost completely unaltered proportions.

The method was abandoned in the twenties of the twentieth century also due to new Italian laws that limited the collection of human remains for public display. Today, the Morgagni Museum of Pathological Anatomy of the University of Padua still preserve several tanninised preparations attributable to Brunetti and his successors.

The current study aims to show the quality of the method showing the results of Brunetti's method nowadays both in specimens with tissue and with bone parts evaluating a possible replicability in modern samples.

The diagnostic utility of μ CT in comparison to transverse thin sections for assessing bioerosion in archaeological bone samples

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Microbial activity is one of many crucial factors that can affect bone microstructure and lead to the loss of original histological features. Microscopic focal deconstruction can be identified by characteristic Wedl tunneling, which is most probably caused by saprophagous organisms, and by non-Wedl tunneling, caused by bacterial activity. In recent decades, several systematic studies have shed light on how to qualitatively assess these tunnels in archaeological bone. In addition to conventional light and scanning electron microscopy, X-ray microtomography (microCT) is increasingly being used to assess bioerosion. Nevertheless, this method is still underutilized and has yet to be validated. Although the organic phase and some histological features cannot be clearly identified with the resolution provided by microCT, the advantage is that the direction and extent of bioerosion in and around the Haversian canals can be visualized in 3D. An additional advantage is that it is a non-destructive method.

Here, we assess contextualized 3D reconstructions of human long bones from archaeological sites in Austria by comparing them to transverse thin sections of the same specimens for which we determined the Oxford Histological Index (OHI) using light microscopy. The aim is to establish whether the OHI can be applied one-to-one to microCT images or if the index needs to be modified. Furthermore, we present a protocol for identifying and visualizing microscopic taphonomic bioerosion in mammalian bone from a temperate European environment. Our protocol provides standard guidelines and an easily reproducible method for visualizing and assessing the level of bioerosion, which can aid in prescreening for future sampling strategies. We will also present some unexpected results and discuss how 3D histological models can be used as a guide to refine the method of histological indexing.

Stable isotope relationship between biological and sedimentary carbonates in karstic archaeological sequences

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As a mean for investigating the complicated interaction (or lack of it) between buried skeletal components and their burial environment in karstic contexts, we investigated and compared the isotopic composition of carbon and oxygen isotopes in carbonate.

Bone and tooth enamel apatite measurements (13C/12C and 18O/16O) of 85 biological samples were compared to the same compositions in sedimentary carbonate from four different central Italian archaeological limestone caves. Human and faunal remains cover a temporal interval ranging from the Pleistocene to the Bronze age, with different taphonomic contexts.

The results show that, at least in some cases, the chemical compositions of the skeletal samples may have been modified towards to composition of the hosting environment by circulating fluids, with a critical impact on the pristine biological signature that could bias the interpretation of the isotopic signature.

Newest technology in the analysis of isotopes archaeological remains

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Isotope Ratio Mass Spectrometry (IRMS) is increasingly used in archaeological sciences to source people, animals and materials, and to study past human's lives.

Isotopic fingerprinting of elements such as C, N, O, H, S and Sr is based on the analysis of small differences of isotopic ratios in the chemical compounds of various archaeological materials. The measured isotopic differences are originating from the physical and biochemical isotope fractionation occurring in nature and can provide information about the biochemical and environmental origin of substances that can be used for paleo diet and migration studies.

This presentation will provide an overview of the state of the art of analysis in Isotope Ratio Mass Spectrometry applied to organic materials in archaeological sciences. Recent advances in instrumentation and in applications will be highlighted.

Comparative trace element analysis using non-invasive microPIXE/PIGE of raw mammoth ivory and worked personal ornaments from different European Palaeolithic key sites

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The systematic use of osseous raw materials such as antler, bone, and ivory is a distinctive human behaviour that emerges in Europe for the first time in the early Upper Palaeolithic (40,000-28,000 BP). In the Aurignacian, exotic raw materials such as steatite, marine and fossil shells, and mammoth ivory were used in the production of ornaments that follow regional patterns of standardization. Although the use of ivory in the production of art and ornaments is of of great interest for studies of the Upper Palaeolithic, relatively few physico-chemical studies have been conducted on mammoth ivory from archaeological sources [1-2]. Until recently, most analytical methods were considered unsuitable for the analysis of rare archaeological materials because of their destructive effects. However, recent applications showed that techniques such as microPIXE/PIGE present the opportunity to enhance our knowledge of archaeological ivory artefacts with little to no risk to the materials [3-4]. In this way, more than one hundred ivory artefacts from historic and prehistoric contexts were examined non-destructively. The results of these chemical analyses can help answer questions about the procurement and use of ivory in the Upper Palaeolithic and the diagenetic processes that change ivory in archaeological contexts over time. The current study was built on this long-term research and be expanded to include the analysis results of ivory fragments and artefacts from other Upper Palaeolithic archaeological sites, in particular Sungir in Russia. The objective of this presentation is to study trace elemental difference between mammoth ivory raw material and personal ornaments made of mammoth ivory. We expect to obtain additional information on taphonomic changes that may be related to the processing of the material.

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$\delta^{18}O_p$ geographical variability within the South African Karoo Basin during Permo-Triassic based on tetrapod apatite.

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The Beaufort Group of the Main Karoo Basin of South Africa represents an uninterrupted accumulation of fluvio-lacustrine sediments from the Middle Permian to the Middle Triassic and covers approximately 200000 km², about 20% of the country's surface area. A diverse vertebrate fauna that includes fish, therapsids, amphibians, archosauromorphs and parareptiles, has been recovered from this rock succession and allows geochemical studies over a large temporal and geographical range.

Questions relating to temporal and biological variation of oxygen stable isotope ratios ($\delta^{18}O_p$) have been addressed, but geographical variation of the bioapatite-recorded $\delta^{18}O_p$ has not yet been studied on the Permo-Triassic of South Africa.

Here we highlight possible variations in $\delta^{\rm 18}{\rm O}_{\rm p}$ recorded on a large-scale basin during the Permo-Triassic.

Several tetrapods, mainly therapsids, were sampled from four different biozones, two Permian (*Cistecephalus AZ* and *Dicynodon-Theriognathus AZ*) and two Triassic (*Lystrosaurus declivis AZ* and *Trirachodon-Kannemeyeria AZ*). For each of these biozones, fossils were sampled from several localities to compare their recorded $\delta^{18}O_n$ values.

While the results showed no variation for both Permian AZ, restricted to the south-western part of the Basin, a north to south decrease of the $\delta^{18}O_p$ values was noted for both Triassic AZ. Also, a 'high' set of values can be separated from a 'low' set for each AZ.

The lack of variation for the Permian samples may be the result of the sampled localities being in close proximity as well as a unique source for the $\delta^{18}O_p$.

Triassic latitudinal variation of the $\delta^{18}O_p$ values results from either a latitudinal gradient of temperature, the continental effect or even the distance from the sources of the watershed or a combination of those. The two set of values suggest two different sources of water, one from the north-east and the other from the south.

Site specific deamidation of Asn and Gln assessed by analysis of ancient collagen

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The need for authentication of proteins in ancient samples is becoming a central issue in palaeoproteomics as risks of contamination from modern proteins are increasingly recognised. Moreover, dating based upon the level of decay is important due to the necessity of building chronological scales in archaeological studies. Glutamine deamidation has been proposed before as an authenticity and chronological marker. Using Python, we mined published and in-house MS2 data analyzed with MaxQuant from collagen type I rich ancient samples from bone, dental calculus, parchment and leather. We derived the site specific asparagine (Asn) and glutamine (Gln) deamidated fraction and calculated the relative rates of deamidation for different tripeptide combinations of the form Xxx-Asn/Gln-Zzz in order to sort them. The order of Xxx-Asn-Zzz tripeptides in our bone and dental calculus samples is correlated with that in previously studied solubilized Gly-Xxx-Asn-Zzz-Gly pentapeptides, while Xxx-Gln-Zzz patterns do not show correlation with their corresponding pentapeptides. In parchment and leather samples, the correlation is disrupted due to chemical treatments and environmental factors. We performed a principal component analysis on the tripeptides deamidated fraction data. The loadings of the tripeptides to the principal components is structured by these features. Moreover, we show a relation between the first principal component and Thermal age, resembling previous studies. We hypothesize that these patterns are due to different deamidation reaction mechanism domination, either via a fast cyclic intermediate formation or a slow direct hydrolysis, in mineralized versus solubilized collagen, which is rapidly lost due to leaching.

A Test of Trace Element Analysis for Detecting Pb and Sr Contamination in Ancient Human Tooth Enamel

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Trace element analysis evaluated soil Pb/Sr contamination of ancient human teeth at the Crenshaw site in southwest Arkansas, USA. Crenshaw is multiple-mound Caddo ceremonial center with a skull and mandible cemetery dating between A.D. 1253 and 1399. This study follows and tests the trace element analysis method outlined by Kamenov et al. (2018) for ancient human tooth enamel. This is one of the first studies to attempt to use this method, so the method is evaluated for effectiveness. Three potential issues were discovered.

(1) The original study did not evaluate any correlations between Pb and other elemental concentrations. If they are to detect Pb contamination, they must be shown to have a correlation between higher concentrations of these elements and higher concentrations of Pb. (2) Thresholds are based on modern human tooth enamel, so potential differences between modern and ancient peoples' exposure to some elements may not be incorporated in the defined thresholds. (3) The concentrations of some elements in human tooth enamel are likely to be different in other regions based on the natural concentrations of these elements in the soil.

Correlation analysis and comparisons to burial soil trace elements suggest that this population had greater in-vivo exposure to rare earth elements and vanadium than modern populations. The trace element analysis was largely successful at assessing contamination and is recommended for future studies, but some elements and universal thresholds may not be useful for ancient populations in different areas.

Diagenesis in Permian tetrapod bones from the Kotelnich and Sundyr localities (Russia)

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The Kotelnich locality of pareiasaurs (the Vyatka river, Kirov region, Russia) is characterized by unique taphonomic and geochemical conditions of sedimentation and fossilization. In the Sundyr locality with similar lithological characteristics (the Volga River, Republic of Mari El, Russia), whose faunal complex has been described only in 2011, the accumulation of sediment most likely took place under the conditions of periodically wetted floodplain, similar to those of Kotelnich. However, unlike Kotelnich, temporary reservoirs in the Sundyr were connected with permanent ones.

Fossil biogenic phosphate formations (bones and teeth of vertebrates, etc.) in sedimentary rocks can be indicative of sedimentation conditions. The paper considers the features of the composition and structure of the fossil bone tissue (rib fragments) of the Permian tetrapods from the Sundyr-1 and Kotelnich localities. The bone remains were examined using optical microscopy, scanning electron microscopy, micro-X-ray fluorescence analysis, and laser ablation quadrupole mass spectrometry (LA-ICP-MS).

The concentrations and distributions of trace elements and REE (lack of bell-shaped REE patterns), as well as Ce, Eu, and Gd anomalies and $(La/Yb)_{N'}$, $(La/Sm)_{N}$ and Y/Ho ratios have indicated the absence of bone apatite recrystallization during late diagenesis and the predominant adsorption mechanism of REE fractionation during early diagenesis. REEs in the Sundyr bone correspond to modern seawater ($(La/Sm)_{N} = 0.6-1.6$), while the bone from the Kotelnich locality – to modern fresh water ($(La/Sm)_{N} = 0.2-1.2$). Nevertheless, both bones are shifted towards larger ($La/Yb)_{N}$ values (3.63-5.27 for the Sundyr and 3.67-4.87 for the Kotelnich samples), which may be due to both secular variations in the composition of seawater and diagenetic transformations of bioapatite during early diagenesis, including REE fractionation via adsorption mechanism. For the studied samples, the sedimentation in shallow (in the case of Kotelnich well oxygenated) alkaline water basins with the input of material from continental sources were reconstructed.

Developmentally-informed assessments of diagenesis in teeth

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Analyses of the tooth chemistry of captive and wild nonhuman primates, as well as human children with prospective nursing and health records, reveal the nutritional and physiological experiences of individuals in unprecedented detail [1-3]. Despite a suggestion that calciumnormalized barium (Ba/Ca) patterns in teeth relate to dietary stress rather than dietary transitions [4], we have demonstrated Ba/Ca increases with the advent of milk intake and decreases with the cessation of nursing in multiple species. This pattern of element distribution in teeth is not substantially altered by mineral incorporation after secretion, yielding the first precise ages for the cessation of nursing (weaning) in Neanderthals [1,5].

Recent assessments of elemental chemistry in bones and teeth often employ laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) sampling of discontinuous spots or linear tracks, which may fail to identify precise changes in body chemistry during tooth mineralization, or diagenesis after burial. By contrast, mapping the entirety of tooth crowns and roots with LA-ICP-MS provides comprehensive longitudinal developmental records of dietary behavior, health, and neurotoxicant exposure [3]. These maps are superior to conventional LA-ICP-MS approaches; they permit the comparison of developmental and elemental geometry in multiple tissues and across successive teeth, and reveal differential element-specific preservation and diagenesis [3, 5]. This is possible because biogenic elemental geometry can be distinguished from diagenetic alteration as the later often shows a more localized diffuse pattern, particularly when multiple elements are considered in tandem [1, 5]. Such studies of trace element distributions from sections of recent human and hominin teeth offer a potent mechanism for the detection of diagenetic alteration at high spatial resolution, extending studies of ancient behavior and health further into the past.

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Biological Calcifications Characterized at the Molecular Level

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Solids of biological origin such as hydroxyapatite or calcium oxalates are directly involved in chemistry of "life": Hydroxyapatite as the main compound of human bones and calcium oxalate as main compound of kidney stones. In this broad study around chemistry of life, materials chemistry and computational chemistry we focus here on Calcium Oxalate and Hydroxyapatite.

We have performed DFT studies on different interfaces and investigated the interaction with a series of bio-organic molecules, and characterized them by calculating vibration frequencies and chemical shifts, which were compared to experimental data.

From the bulk crystal structures of calcium oxalate polymorphs obtained through DFT methods[1, 2, 3] the low index surfaces were build. Since the thermodynamic stability depends on the medium in which the surface is introduced, the calculation of the interaction of the calcium oxalate surface with water, urea, and other small molecules gives us the possibility to understand the change in crystal morphology of the final oxalate crystal in its natural medium. The final aim is the prediction of the shape of the kidney stone in its natural medium.

Another biological mineral that we have studied is hydroxyapatite, being the major mineral component of tooth enamel, dentin and bone in which it is currently associated to biomolecules and various biopolymers. Organic nanosized particles are described as comprising a core surrounded by a crystalline hydrated amorphous layer. In this study the characterization of the surface of hydroxyapatite nanoparticles is essential to better understand their formation, and dissolution mechanisms and interactions existing at the interface between the crystalline and amorphous phases. The purpose of this study is to model a surface(s) of hydroxyapatite and analyze the physicochemical properties using the methods of quantum chemistry (periodic DFT).

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Do bone diagenesis studies have a role to play in forensic and medicolegal investigations?

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Those working in the forensic sciences often adopt methods and techniques that have been developed in other disciplines and apply them to the medico-legal context. This is a very fruitful approach since the range of forensic contexts can be large, varied and complex and require myriad data and intelligence to resolve. Forensic anthropology is an example of one such field, which has routinely applied methods and approaches developed within the archaeological and palaeoecological arenas. Likewise, there are examples of the reverse, where methods developed to address specific forensic questions are then applied to remains from the past. Despite the transfer of methods from one field to another, there are times when the underlying principles and tenets do not flow in the same direction. Thus methods can be applied without suitable appreciation of the underlying assumptions that can influence one's interpretation of the results. Further, the specific demands that the medico-legal context places upon the interpretation of these data and the application of the methods provides additional pressures. This talk will explore some of the considerations with applying research, data and methods used for studying bone diagenesis in the past to the modern forensic sciences, and *vice versa*.

Determination of the diagenetic and biogenic elements in Palaeolithic mammoth ivories and bones at PUMA / SOLEIL and NewAGLAE / C2RMF

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During the palaeolithic era mammoth ivory has been used for the first body ornaments, sculptures or music instruments in human history [1]. It is a precious witness about ancient human behavior but is fragile due to complex alteration processes occurring over time. Therefore, it is necessary to understand the diagenetic phenomena in order to improve the conservation of ivory and bone artefacts. Moreover, the determination of the biogenic elements is of high importance to track the origin of the samples and to detect potential exchange of ivory and bone artefacts between sites at the prehistoric times. In this work we studied 12 mammoth ivory samples and 10 bone samples coming from the Hohle Fels prehistoric site, located in South-west of Germany.

The strong diversity in trace elements in archaeological ivories and bones makes their study relevant to highlight the potential of complementary analysis at PUMA / SOLEIL and at NewAGLAE / C2RMF. Previous studies of ivory artefacts have shown that Fe and Mn belong to diagenetic elements whereas Zn, Br and Sr to the biogenic elements and thus can be used as potential tracer of ivory origin [2].

X-ray fluorescence (XRF) mapping at PUMA is more sensitive to heavier elements (Z > 17) while particle-induce X-ray emission (PIXE) mapping at NewAGLAE is well suited to localize and quantify light elements (Z < 30). These techniques coupled with others available at these facilities (XAS at PUMA, PIGE and RBS at NewAGLAE) allow to confirm the previous determination of diagenetic/biogenic elements and also to investigate if other elements belong to one of these categories.

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Perfect as the enemy of good: organic hydrogen and inorganic oxygen isotopes in bones and teeth

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Isotopic data from bones and teeth has proven useful in dietary, environmental, migration and life history studies. Diagenesis is never far from the mind of practitioners. Some would assert that for carbon (both organic and inorganic) and nitrogen, diagenetic factors are under control. For newer isotopic applications such as hydrogen in collagen and oxygen in both collagen and hydroxyapatite, diagenetic concerns range from nonchalant to panicked.

We present data on a controlled heating experiment on bone to address issues of hydrogen and oxygen isotopic change in collagen. We discuss these data in the context of the molecular configuration from the new "collagen" isotope standards [1]. We further explore proxies for isotopic fidelity including elemental values and experimentally imposed exchange exposures.

We suggest that comprehensive analyses within and between ecosystems and the constituent fauna will provide important insights from hydrogen and oxygen isotopes. Finally, we address the unique, and to some, important issue—humans.

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In vitro alteration of mammal teeth in isotopically enriched tracer solution: assessing diagenetic stability of bioapatite δ^{18} O values

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Oxygen isotope compositions in enamel bioapatite, both of the phosphate ($\delta^{18}O_{PO4}$) and carbonate ($\delta^{18}O_{CO3}$) group are considered as robust proxy archives for ingested drinking water and/or vertebrate body temperature. However, only few alteration experiments have tested this assumption.

Here we present the results of in-vitro alteration experiments of dental tissues cut from a modern African elephant molar (AG-Lox) reacted in vitro in a closed system at temperatures of 30 to 90 °C for up to two months under extreme physico-chemical conditions surpassing typical low temperature burial settings. Dental cubes with about 3 mm edge length, comprised of both enamel ($\delta^{18}O_{PO4} = 20.3 \%$; $\delta^{18}O_{CO3} = 29.3 \%$ VSMOW) and dentine, were placed into 2 mL of acidic aqueous solution enriched in different isotopes (²⁵Mg, ⁴⁴Ca, ⁶⁷Zn, ⁸⁶Sr) and highly enriched in ¹⁸O (3.43 mols/mol%) and ²H (96.6 mol/mol%) and reacted in sealed Teflon vials. Oxygen isotope ($\delta^{18}O_{SIMS}$) distribution across the reacted dental cubes were measured in-situ with a Cameca 1280-HR secondary ion mass spectrometer (SIMS) using a 30 µm raster size to determine the degree of bioapatite alteration via profiles with step distance as short as 20 µm.

In the 30 °C experiments only slight enrichment in $\delta^{18}O_{SIMS}$ of a few permil occurred at the outer enamel rim and EDJ. In contrast, for 90 °C experiments enamel of all dental cubes was strongly enriched in ^{18}O ($\delta^{18}O_{SIMS} \approx 100$ to 600 ‰) throughout the complete enamel thickness, progressively increasing with experimental duration reaching extreme values >1000 ‰. In contrast, the isotopically labelled major and trace elements Mg, Sr, Zn and Ca were only altered in the outer 200-300 µm of the enamel [1]. It is clear that the ^{18}O -enriched tracer solution completely penetrated the enamel and isotopically exchanged with the bioapatite. Thus, even mm-thick mammalian enamel seems to be prone to pervasive oxygen isotope alteration.

Oxygen in bioapatite occurs in three different moieties: PO_4 ($\delta^{18}O_{PO4}$), CO_3 ($\delta^{18}O_{CO3}$) and OH ($\delta^{18}O_{OH}$) group. The SIMS measures the bulk bioapatite composition ($\delta^{18}O_{SIMS}$). Therefore, analyses of the different oxygen-bearing moieties are necessary to assess which of those were altered. Serial enamel sampling of an intensely altered dental cube (90 °C, 21 days) reveals an oxygen isotope exchange (i.e. ¹⁸O enrichment) of the CO_3 -group ($\delta^{18}O_{CO3} = 540$ to 936 ‰); The PO₄-group is generally considered to be robust against abiogenic low temperature oxygen isotope exchange due to strong P-O bonds while the OH-group is prone for such alteration. Implications for the robustness of enamel bound oxygen isotopes as palaeoclimatic and palaeoecologic proxy as well as potential alteration mechanisms will be discussed.

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Establishing criteria to differentiate mass-death and attritional fossil bonebeds based solely on trace element signatures

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Rare earth elements (REE), uranium, and other trace elements have long been recognized as valuable tools for deciphering the taphonomic history of fossil assemblages. These elements are readily adsorbed by bone hydroxyapatite after burial, and under favorable conditions, early-diagenetic trace element signatures can persist throughout diagenesis and serve many uses. Here, I contrast the geochemistry of two Cretaceous bonebeds representing nearly-ideal end members along a taphonomic continuum: a mass-death, monospecific Edmontosaurus bonebed from the Hell Creek Formation of South Dakota and an attritional, multitaxic bonebed from the Lance Formation of Wyoming. Despite modest late-diagenetic overprinting, the contrasting accumulation histories of these assemblages remain readily apparent from their trace-element signatures. The South Dakota bonebed has been identified as a mass-death assemblage and the Wyoming bonebed as an attritional accumulation, and these assignments can be made based solely on the trace element compositions of the bones (i.e., independent of their sedimentologic and physical-taphonomic context). By combining observations from these sites with prior studies, it is apparent that mass-death assemblages are often characterized by consistency among specimens in: (1) shale-normalized trace element compositions (including REE fractionation patterns); (2) type of concentration-depth profiles from the cortical margin, and; (3) redox signatures (e.g., cerium anomalies). Massdeath assemblages also often exhibit greater trace element enrichment in bones with greater tissue porosity and low concentrations of middle REE and elements with slow diffusivities through bone middle cortices. In contrast, attritional assemblages are often characterized by heterogeneity in the three attributes above, as well as: (1) greater overall trace element uptake, and; (2) variable trace element enrichment among bones of similar size and histology. These conclusions are independently corroborated by sedimentologic, stratigraphic, and traditional taphonomic data at all sites analyzed, making the geochemical distinctions identified herein robust indicators of the formational history of fossil assemblages.

Using XAD resin to remove synthetic contamination from archaeological bone prior to radiocarbon dating

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Extraction protocols for the radiocarbon dating of bone collagen samples are continuously being tested and improved. Even small amounts of modern carbon can affect the ¹⁴C age, especially in old samples. Contamination can originate from the burial environment as well as post-excavation treatments, for example in the use of synthetic consolidants. While most contamination can be removed using the ABA method, this is not the case for cross-linked contamination or certain consolidants. Single amino acid dating can eliminate this type of contamination, although large sample sizes are required. The most suitable method to remove contamination from small and/or badly preserved bone samples seems to be the XAD method, which was originally developed by Tom Stafford [1].

The XAD protocol was implemented at the MNHN radiocarbon laboratory and the setup was tested using bone blanks and standards. As difficulties were encountered at different stages of the sample preparation, different setups were tested and these results will be presented here.

After validation, the method was applied to a relatively large whale bone from the Late Palaeolithic site of Santa Catalina (Spain) that was visibly contaminated with some type of consolidant and also produced an anomalous peak in the FTIR-ATR spectra at 1720 cm⁻¹. Four ¹⁴C dates from two different collagen extractions followed by XAD resin treatment of the whale bone are in agreement, ranging from 13 060 to 13 090 ± 60 BP, while a sample from the same extraction without the XAD treatment produced an older radiocarbon date of 13 300 ± 60 BP. This suggests that the contaminant was not fully removed by the classical treatment and may contain fossil carbon. This study demonstrates the interest of the XAD treatment to clean heritage bone samples stored in museums prior to geochemical analyses.

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Differences in Structure, Major and Trace Elemental Concentrations in Bioapatite During the Late Postmortem Interval Pre- and Post-Burning

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Chemical profiles of bones affected by contamination from the burial environment can give information on the early postmortem state of skeletons. The postmortem environment and treatment of remains, such as burning, inhumation, or sub-aerial exposure, can heavily alter bone chemistry. Chemical degradation of the inorganic phase happens along alterations to the crystal structure due to the abundant soluble and exchangeable ions from the environment. This transformation can happen either during early diagenesis during or following bioerosion, encouraging the dissolution/movement of the minerals/ions eventually resulting in a more thermodynamically stable mineral phase. This paper looks at whether the chemical profile of bioapatite could be used as a proxy for decomposition prior to burning in bones through an actualistic taphonomic study.

Fleshed pig (*Sus scrofa*) tibiae were left exposed on a field, then collected at 14, 34, 91, 180, 365 day intervals before being burnt in an outdoor fire (≤750 °C bone temperatures). Fresh tibiae acted as unburnt and burnt controls. Fresh, decayed, and post-burnt bone samples were sectioned, mounted and polished and then spatially analysed for major and trace elements by energy wavelength-dispersive spectroscopy (EMPA-WDS) and Fourier Transform Infrared Spectroscopy (FTIR). Linear regression and Principal Component Analysis (PCA) were performed on both datasets.

Results show that K significantly decreases in a linear fashion in both unburnt and burnt bones throughout the postmortem interval (PMI). Al and Mg increases in unburnt bones, while Na, K, Fe, and Ca have the potential to estimate PMI prior to burning. Lower variations of concentrations were found further from the outer cortical bone, with elements associated with bioapatite (Ca, P) and the extracellular fluids (K, Mg) being the most reliable. Carbonates (A and B) decreased while the crystallinity index increased in unburnt bones during the first 1 year of PMI. PCA differentiated between pre- and post-burnt bones, but not PMI groups.

Penetrating the pars petrosa: investigating the influence of bone turnover and diagenesis on strontium isotope ratios related to childhood mobility

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Strontium isotope analysis can be applied to the calcined human otic capsule in the petrous part (pars petrosa ossis temporalis; PP) to gain information on childhood mobility in archaeological and forensic contexts. However, only a thin layer of the otic capsule, the inner cortex, demonstrates virtually no remodelling and would be suitable for obtaining childhood ratios, while the external cortex (the layer directly surrounding the inner cortex) and other parts (i.e. the apex) of the PP have higher bone turnover rates and may exhibit different ⁸⁷Sr/ ⁸⁶Sr. Applying our improved sampling method, calcined PP from ten cremation deposits are sampled for strontium isotope analysis, thereby sampling both the inner and external cortex and the apex of two PP. For comparison, diaphysis and rib fragments from each cremation deposit were also sampled. Forty percent (4/10) of the calcined PP show marked differences in ⁸⁷Sr/⁸⁶Sr (0.00035-0.00065) between the inner and external cortex. One of the apices yielded a ⁸⁷Sr/⁸⁶Sr that differed > 0.0009 from any of the other samples from the same cremation deposit. While the differences in ⁸⁷Sr/⁸⁶Sr between the inner and external cortex could be explained by dissimilar bone turnover rates, the large difference in ⁸⁷Sr/⁸⁶Sr between the apex and the rest of the elements may be attributed to other factors, such as diagenesis. Our study highlights the problematic nature of the external cortex and the apex, suggesting that despite being calcined, diagenesis may influence ⁸⁷Sr/⁸⁶Sr ratios in general but especially in the apex and that more research is needs to be undertaken by sampling various locations of the PP to further improve our understanding of bone turnover and diagenesis in calcined PP.

The use of radiocarbon dating to assess the impact of diagenesis on δ^{13} C analysis of tooth enamel

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 $\delta^{13}C$ in ancient tooth enamel is often thought to be minimally impacted by diagenetic alteration. This is at odds with the enormous quantities of carbonate contaminantion identified by radiocarbon dating. Enamel from teeth known to be more than 50,000 years is often radiocarbon dated to 15,000 BP, and after an acid leach barely exceeds 20,000 BP. Assuming the contaminant is modern in age, this implies that at least 8% of the carbonate in enamel is a contaminant after pretreatment. The true amount is likely substantially more as the contaminant is unlikely to be modern. In the past it has been difficult to accurately and precisely establish 'known $\delta^{13}C$ values' for ancient teeth, and so to establish the impact of contaminants with a precision of more than a few ‰.

We use radiocarbon dating of teeth from karstic environments in Vietnam, known to be more than 50,000 years old, to identify the presence of carbonate contaminants. By progressively removing contamination we can create a relationship between F¹⁴C and δ^{13} C. As samples of this age should not contain any ¹⁴C, we can use the relationship to estimate the accurate δ^{13} C.

We find that ancient enamel can be more than 2.5 ‰ higher than the estimated δ^{13} C. After applying an acid leach typical of many stable isotope laboratories, we find enamel is still at least 1 ‰ higher than the estimated δ^{13} C. In every case examined, the enamel is enriched in ¹³C. FTIR, commonly used to establish whether enamel is reliable for isotope analysis, cannot identify this contamination. Whilst this effect is relatively small in comparison to the difference between C3 and marine/C4 systems, caution must be applied when attempting to assess the small differences which result from e.g. the canopy effect. In these cases, novel pretreatment methods are required.

Multifactorial approach to describe early diagenesis of bones: the case study of the Merovingian Cemetery of Saint Linaire (France)

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The excavation of the Merovingian cemetery of Saint Linaire (France) was an opportunity to describe the completeness of the tombs preserved from soil erosion.

An anthropobiological study was carried out on the osteological material and the different categories of graves were dated. On the basis of a complete archaeo-anthropological corpus we have undertaken an analysis of the differential conservation of the bones according to the different archaeobiological parameters such as the type of bone, the individual age, the sex but also the type of grave such as open ground, coffin, dolomitic or limestone sarcophagus and their dating.

Analyses on the carbonate content of the bones, the amount of fluorine, the carbonate content, the microporosity and macroporosity of the bones, the δ^{13} C, the residual content of Carbon and Nitrogen were mobilized in order to establish whether and how funerary practices had impacted the early diagenesis of the bones. We propose to expose the different characteristics identified between the different biological, geochemical and mineralogical parameters in order to describe an early process of bone diagenesis.

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Abstracts – Poster presentations

Listed alphabetically

Presenting authors are underlined

Microscopic Sampling of Bone Collagen

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The sampling of bone collagen for carbon and nitrogen isotopic analysis has been widely used to research past populations' health and nutrition. The study of bone collagen, with few exceptions, is done using bulk collagen analysis. Using bulk collagen limits the amount of information that can be acquired as the isotopic input for 5-25 years is averaged into a single sample.

A method was developed using a scanning electron microscope (SEM) imaging and microscopic sampling of bone collagen to gain more dietary information by detecting isotopic changes during the formation and remodelling of bones. The technique only requires a small 'core' of bone to be removed for sampling. The sampling method is aimed at osteons and interstitial lamellae within the compact bone. The technique was developed using modern faunal bone and tested on archaeological human remains. The preservation of the archaeological remains was varied in order to determine limitations on the method.

The isotope analysis of samples used in the development of the method shows that variation in the isotope values can be detected, beyond that observable from bulk sampling. Variations in dietary input were seen within both the faunal and human skeletal samples. However, even with the increased data that is available, the method is limited by bone preservation. It has been determined that the method may not provide usable data if the bone collagen and histological integrity is compromised by poor preservation.

The developed method allows for a more detailed analysis of bone collagen sampling that is not obtainable when using bulk collagen analysis and limits the destructive nature of isotopic analysis.

Assessing the effects of two nanoparticle based consolidants through diagenetic parameters

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Bone is often subjected to conservation treatments prior to study. Some treatments, amongst others consolidation, may interfere with some analysis. This research is aimed to analyze the influence of nanoparticle products, which emerged in the last decade as consolidants for archaeological bones. Specifically, this study characterizes the effect of two products, calcium hydroxide nanoparticles (Nanorestore®) and silica nanoparticle (NanoEstel ®), used on archaeological samples from the early to middle Pleistocene archaeological sites of Galería (Sierra de Atapuerca, Burgos, Spain) and Barranc de la Boella (La Canonja, Spain).

Samples were divided into three parts: the control sample and two more pieces to be consolidated with Nanorestore ® and NanoEstel ®. There were two processes of consolidation, one by entirely immersing the sample in the nanoparticle solution, and the other by consolidating the bone powder, the latter to enable a deeper study of the chemical modifications and the mineralogical phases formed.

FTIR-ATR analysis was used to evaluate the effect of the two consolidants by using the established diagenesis parameters such as collagen wt.%, IRSF, C/P, calcite wt.%, API, BPI, BAI, FWHM, C/C. In addition, XRD was used to characterize the mineralogical phase formed.

Results show that Nanorestore® affects specially the IRSF and carbonates rates such as API, BPI, BAI, as well as the carbonates area. Meanwhile, NanoEstel® produces a remarkable increasing in FWHM, and presents some frequencies in the natural silica compounds range, such as opal (798cm⁻¹), affecting to collagen quantification. By contrast, XRD diffractogram show amorphous silica compounds present in the sample of bone powder consolidated with NanoEstel®.

Despite this fact, it is remarkable that the depth of penetration of these consolidants is limited to a few mm, and simply removing a thin external layer of the bone would also remove the products. Finally, further research on this issue should be car- ried out.

Dead, fossil or alive

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Calcium carbonate, silica and calcium phosphate have been selectively used by organisms in the production of mineralized hard parts throughout the Phanerozoic. Among these materials, bioapatite has enabled fundamental acquisitions in the evolution of life. Despite the remarkable biological success, the crystallography of bioapatite and the eventual modification of lattice parameters over a wide range of geologic time have in contrast been scarcely investigated.

In our study we analyzed living, dead and fossil remains of apatite biomineralizing organisms, both vertebrates and invertebrates, ranging from the Cambrian to the Recent, a time-lapse spanning over 500 million years. We detected the bioapatite crystal features of the major phosphatic phyla (Brachiopoda, Arthropoda, Bryozoa, and Chordata: the latter including conodonts, cartilaginous and bony fishes, amphibians, reptiles, birds and mammals). Groups were investigated using either fossil or recent material (dead and alive, the latter referring to material extracted from living organisms).

We applied a consolidated protocol of analyses [1-3] integrating optical and scanning electron microscopy coupled with chemical microanalyses (ESEM-EDX and SEM-EDS) and micro X-ray diffraction (μ -XRD). Our data reveals that living and dead organisms, and their fossil remains, have a distinct geometric signature in terms of bioapatite lattice cell parameters mirroring atom re-arrangements within the crystal lattice which drive to a general reduction of the cell volume (i.e., the volume of the hexagonal crystalline cell frame) over time.

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The role of marine bioerosion in the diagenesis of terrestrial bone: a pilot study

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Bioerosion is the collective word describing the removal of bone by living organisms. The principal mechanisms of bone removal are abrasion and tunneling. Many marine organisms bioerode bone, but the study of bone bioerosion in the marine environment has been hitherto limited, especially in the case of bone of terrestrial mammalian origin. The anatomy of terrestrial species differs from marine vertebrates', and leads to different patterns of taphonomy and diagenesis [1]. In this pilot study, two submerged and macro-bioeroded terrestrial mammalian bones were investigated, with the aim of characterizing their taphonomy, discussing its influence on diagenesis and identifying the eroding organism.

Two archaeological bones with a known post-submersion time (169 and 316 years) were characterized, both macroscopically, by visual observation, and microscopically, by Scanning Electron Microscopy (SEM) and Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS).

The bones were fragmented and porous. SE microscopy revealed a heavy alteration of their microstructure, with rarefied cortex and trabecular flimsy remnants. LA-ICP-MS showed a haphazard distribution of diagenetic trace elements.

The macroscopic appearance of both bones was peculiar. Saltwater had flown unimpeded through their disordered microstructure, possibly promoting dissolution. The morphology and the regular pattern of the widespread surface borings, small round holes of different sizes connected by canals to a network of internal cavities, suggest the action of a sponge [2-4]. Most likely, the sponge belonged to Clionaidae, a taxonomic family living in tropical and temperate seas and preferring shallow environments, like the one where both bones were recovered. The identification of borings produced by a marine organism on bones of terrestrial mammalian origin enables to include marine submersion in their taphonomic history, with relevant consequences in multiple disciplines, such as paleontology, archaeology and forensics.

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Iron, Copper, and Zinc isotope ratios and concentrations in calcined bones: preliminary results and prospects

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The use of isotopic proxies can provide information otherwise unavailable in human remains. Previous studies have demonstrated that Fe, Cu, and Zn isotope ratios and concentrations can be used as trophic level tracers on unburned mammal bones, including humans. Additionally, Fe and Cu isotopes have shown the potential of discerning sex in humans.

No research has so far been conducted using these elements on calcined bones. The application of these methods to the analysis of calcined bones could greatly improve our understanding of funerary practices and the lifestyles of populations that practiced cremation as funerary rite. Calcined bones are often fragmented, deformed and lack organic material, so the use of the routine C and N diet proxies is not applicable to them. Isotopic sexing could greatly improve the rates of identified cremated individuals, which are currently under 20% for most archaeological sites. However, methodological considerations and experiments are necessary to establish the potential diagenetic changes due to burning at high temperatures and/or contamination from the burial environment that could affect the respective isotopic signatures.

In this study, concentrations of Fe, Cu, and Zn were measured in experimentally burned samples of modern cow tibia, archaeological human samples from Belgium, and a modern, identified human sample. The preliminary results for concentrations indicate that samples become depleted in Zn at temperatures higher than 800°C, indicating a limited utility of this element for paleodietary reconstructions from calcined bones. However, coupled with other indicators of temperature such as infra-red splitting factor and C/C ratio, Zn has potential for use as an indicator for cremation temperatures. Meanwhile, Fe and Cu differ from Zn by showing no variation with temperature, but important differences in concentrations between modern and archaeological bone. Future experiments can preclude contamination or diagenetic changes of the bone affecting the isotopic ratio and concentration of these elements.

Collagen extraction and evaluation of organic preservation in Greek & Cypriot fossilised bones

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In the last decades research has shown positive results regarding the preservation and extractability of organic molecules in both archeological and paleontological material [1]. The study of such molecules via extraction and/or isotopic and IR analyses [2] can provide useful information in evolutionary, environmental and diagenetic studies. Collagen is one of the most abundant proteins in bones and its presence in fossils can be indicative of the preservation of other more complex biomolecules such as DNA.

This research focuses on the extraction and quantification of collagen and the evaluation of organic preservation in fossils in relation to the age, location and type of samples. The methods used for the extraction of collagen are modifications of the Longin method as suggested in related studies [3], [4], [5]. IR analysis was also applied in an attempt to examine the correlation between collagen preservation and the samples' diagenetic profile [2]. The samples used in this research vary in age and geographical location: Upper Miocene (Pikermi, Samos, Kerasia), Pliocene – Pleistocene (Sesklo), Upper Pleistocene (Megalopoli), Upper Pleistocene – Holocene (Tilos, Aghia Napa, Vraona), Holocene (Dispilio).

Hopefully, this research could be used as the stepping stone in creating a much larger scheme correlating the presence/ absence of collagen with fossilization variables such as taphonomy – diagenesis, age, location, climate, ground geochemistry etc. This could then act as a tool of predicting the possibility of finding organic remains in any fossil, given that those values can be determined.

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Pyrolysis compound-specific isotope analysis (δ^{13} C and δ^{15} N PY-CSIA): novel analytical approach for archaeological studies

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Stable isotope analysis has become an important tool within the field of archaeology. The isotopic composition of human and animal tissues (bone, teeth, keratin, muscle, fat etc.) allows insight into the diet of our ancestors in a specific period of time. Stable isotopes also inform about population migrations, food origin and possibly the commercial routes of food products.

Pyrolysis-compound specific isotope analysis (Py-CSIA) is a cutting-edge analytical approach able to provide, not only a precise identification of organic compounds in different complex matrices, but also additional valuable information about the nature and origin of the materials based on their isotope composition. This technique is based on the coupling of a micro-furnace pyrolysis unit to a gas chromatograph equipped with an isotope ratio mass spectrometer (IRMS). The individual volatile pyrolysis products separated by gas chromatography are directed to a combustion or pyrolysis micro-reactor (GC-Isolink system) and the isotopic composition of the gases produced is measured in a continuous flow IRMS via an interface unit. With this technique it is possible to make direct determination of stable isotope ratios (i.e. δ 13C, δ 15N) of specific compounds with minimum sample handling and pre-treatment, thus minimizing the chance of contamination and artefacts productions.

In this communication, we will show δ 13C and δ 15N values of a lizard scale obtained using the Py-CSIA technique. We will then show the potential of applying this technique into the field of archaeology considering the preliminary results obtained from medieval human skeletons from the Iberian Peninsula.

Temperature dependent Raman spectroscopy of modern and archaeological human enamel and dentin

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Teeth and bones are often found as burnt (cremated) skeletal remains survived in fire caused by deliberate or accidental fires, cremation practices or campfire cooking, both in ancient times and nowadays. Cremation might severely limit the data obtained from burnt skeletal remains, e.g. alter the stable carbon and oxygen isotope ratios, exchange large amounts of carbon with its surrounding environment leading to biased radiocarbon ages, etc. Nevertheless, fully calcined bone (up to 1000°C) retains the original strontium ⁸⁷Sr/⁸⁶Sr isotopic composition [1], while tooth enamel, considered the most resistant to diagenesis, spalls and deteriorates when exposed to burning. It is known, that low temperature heating (^{550°C}) and diagenetic processes may induce similar effects on bone mineral [2].

The study is devoted to the investigation of the thermal behavior of modern and archaeological (Bronze Age) human enamel and dentine using *in situ* temperature dependent Raman spectroscopy in temperature range 270–870 K in order to assess structural changes in organic and mineral components. Raman spectroscopic techniques are widely used to study the local molecular structure and its defects of modern and archaeological human and animal bone and teeth. Moreover, temperature dependent (hot stage) Raman spectroscopy allows the *in situ* experimental studies of bone or tooth fragments to be conducted under controlled heating conditions.

Raman spectra were obtained using a Horiba LabRam HR800 Evolution spectrometer equipped with an Olympus BX-FM confocal microscope, a He-Ne laser (radiation wavelength 633 nm) and a Linkam TSM 600 heating/cooling stage. Statistical methods (autocorrelation function and skewness) used for the parametrization of obtained Raman spectra had provided more accurate diagnosis of temperature-induced spectral changes as compared to conventional peak fitting [3].

The results obtained contribute to the knowledge of thermal behavior of tooth tissues, their in situ micro-structural alterations with high spatial resolution, and diagenetic processes.

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Diagenetic impacts on the Color Alteration Index and geochemical characteristics of conodonts

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The conodont Color Alteration Index (CAI) has been widely used to determine maximum temperature in carbonate rocks. Observable color variations in these phosphatic microfossils are commonly thought to be a result of the thermally-induced structural evolution of organic carbonaceous matter (CM), despite recognition that conodont color can be affected by other factors, such as diagenetic alteration. Raman spectroscopy of carbonaceous material (RSCM) allows thermally-induced structural changes in CM, and thus thermal maturity, to be quantified independent of conodont color. We applied RSCM to conodont specimens of varying CAIs (1.5 - 6.0) from the Canadian Cordillera to estimate maximum temperatures based on the transformation (structural reorganization) of disordered carbon to graphite. Trace element characteristics in the same conodonts show that specimens with the most anomalous CAI with respect to independent RSCM temperature estimates also have the highest concentrations of transition metals.

We propose the adsorption of transition metals such as iron onto bioapatite crystals and permineralization by their oxides in conodont elements as mechanisms for the modification of CAI during diagenesis. Furthermore, the trace element characteristics of conodonts, primarily rare earth elements (REEs), have frequently been used as a proxy for paleoceanographic conditions. However, we document that the impact of early diagenetic processes post-burial obscures this marine signal, and instead the trace element characteristics of conodonts likely reflect the characteristics of pore waters.

Several geochemical 'tools' have been proposed to test for such overprinting of paleoceanographic information, including Y/Ho vs. Σ REE, MREE/MREE*, U concentrations, and La/Yb. Both geochemical and Raman structural characteristics indicate that these 'tools' cannot be systematically applied, suggesting that the tools are unable to discriminate between diagenetic alteration induced before and after burial. As a result, we strongly suggest investigations of both CAI and the geochemical characteristics of conodonts be accompanied by Raman spectroscopic analyses before interpretation.

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Guess who's coming to dinner: Ca isotopes and trace elements of dinosaur tooth enamel from the Late Cretaceous of Montana

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Tyrannosauridae and Ceratopsidae are iconic dinosaurs that once inhabited the vast coastal floodplains of what is now North America. The fossiliferous Judith River Formation of Montana (79-76 Ma, Campanian, Cretaceous) represents an ideal opportunity to track potential changes in trophic relationships among dinosaurs as it preserves a relatively stable fauna over time, and through environmental and geospatial change.

We used LA-ICPMS and MC-ICPMS to study trace elements for Ca-isotopes of tooth enamel to explore the feasibility of these proxies in investigating biological and environmental signals.

Trace elements show diagenetic differences between families, with Tyrannosauridae having typically more pristine enamel than Ceratopsidae. Notably, the U content of Tyrannosauridae enamel is on average below 1 μ g/g, ~200-fold lower than for Ceratopsidae. Similarly, REE are two orders of magnitude higher in Ceratopsidae.

Ca-isotopic signals are also distinct, with the highest $\delta^{44/42}$ Ca observed in Ceratopsidae, as expected for herbivores feeding at a lower trophic level than carnivorous Tyrannosauridae. However, we cannot exclude a (partial) diagenetic overprint of the in-vivo signal.

These results represent an important first step in understanding the ecological niche occupied by Tyrannosauridae and Ceratopsidae during the Cretaceous and demonstrate the great possibility offered by combined trace element and isotopic studies to assess diet versus diagenesis.

Effects of consolidants and their removal by polar solvents on the stable isotope compositions of an archaeological caribou bone

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Although conservation practices have facilitated the establishment of well-preserved museum collections, the procedures used to stabilize ancient materials can alter their chemical and isotope compositions. In this study, we tested two common consolidants – polyvinyl acetate (PVAc) and acrylic resin (Paraloid B-72TM) – followed by two consolidant removal procedures to investigate the impact of these processes on the stable isotope compositions and structural characteristics of a series of subsamples taken from a representative archaeological faunal bone. We examined the collagen ($\delta^{13}C_{col}$ and $\delta^{15}N_{col}$), structural carbonate ($\delta^{13}C_{sc}$ and $\delta^{18}O_{sc}$), and phosphate ($\delta^{18}O_{p}$) isotope compositions of the bone sample before consolidation and after consolidation and consolidant removal procedures. We also measured $\delta^{13}C_{sc'}$, $\delta^{18}O_{sc}$ and structural characteristics before and after the pre-treatment commonly used to remove organic matter and secondary carbonate prior to isotopic analyses.

Our results produced five main outcomes. First, an acetone treatment shorter than 48 hours was sufficient to remove PVAc and acrylic resin from archaeological bone without altering the original $\delta^{13}C_{col}$ and $\delta^{15}N_{col}$. Second, prolonged exposure (>48 hours) to polar solvents during the consolidant removal procedure and collagen extraction modified collagen isotope compositions, in our case, lowering its original $\delta^{13}C_{col}$ by 0.3 to 0.4 ‰ and increasing its original $\delta^{15}N_{col}$ by up to 0.9 ‰. Third, the consolidant application and removal procedures altered the original $\delta^{13}C_{sc}$ and $\delta^{18}O_{sc}$. Fourth, the change in $\delta^{18}O_{sc}$ was likely caused by the bleach (2 % NaClO at 20 °C for 72 hours)/acetic acid (0.1 M for 4 hours at 20 °C) pre-treatment applied prior to isotopic analyses. Fifth, our results confirm and expand on previous studies reporting that $\delta^{18}O_p$ remained unaltered after consolidant and solvent procedures. The outcomes of our study thus provide a framework for obtaining viable stable isotope compositions from previously consolidated archaeological bones.

Mapping in-vivo Pb concentrations in late medieval Ypres to identify health, mobility and diagenesis

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In Europe, an increased use of lead (Pb) is observed from the early medieval period to the late medieval period as evidenced by historical research and the recovery of a large range of lead and lead-coated objects during excavations. These include lead pipes used for water distribution, different roofing materials, Pb-glazing used on different vessels, and other everyday household goods. How this increase in Pb use in the late medieval period impacted the health remains unclear but it is likely to have influenced both the humans and the environment. By investigating Pb-concentrations in human remains, it might become possible to shed light on human health and even mobility between societies with higher or lower lead concentrations. Our study focuses on the Pb concentrations in the skeletal remains of the late medieval city of Ypres (13th- 14th century) and the early medieval population of Koksijde (7th-8th century), to investigate if *in-vivo* exposure to Pb can be detected.

Traditional Pb measurements on archaeological remains focus on enamel rather than on bone apatite due to the possible post-mortem exchange of Pb with the soil. However, as dental enamel only represents a childhood signal, the lifestyle during adulthood often remains vague. In this study, using micro–X-ray fluorescence, we explore the Pb concentrations in bone material of twenty-five individuals from the population of Ypres by comparing them with twenty individuals from the early medieval site of Koksijde where exposure to anthropogenic Pb would have been minimal.

The application of an aluminium (Al) 630 µm source filter during the semi-quantitative element mapping, instead of traditional mapping without filters, allows for the detection of Pb by reducing the X-ray signal for light elements such as P and Ca. A clear difference in Pb concentrations is observed between Koksijde and Ypres, with the latter site being significantly more enriched. In addition, our pre-liminary results show intra-site variations for Ypres and raises the question if these differences could be caused purely by post-mortem processes or (partly) by *in-vivo* exposure to lead.

Saving Old Bones: an application of near-infrared spectroscopy to prescreen bone for collagen

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Well-preserved bone collagen can reveal much about the human past. Archaeological collagen can illuminate the history of past populations, but diagenetic processes can result in rapid and sometimes inconspicuous collagen degradation. Consequently, there has been considerable interest in potential methods to prescreen bone for collagen content. Preservation of bone is not the sole obstacle researchers in archeological science must overcome; consolidant contamination also introduces a unique challenge. Thus identification of the specific bone consolidant applied is an invaluable resource. Often the presence of contamination is detected with visual inspection (sticky or shiny appearance), but identification of the exact substance requires FTIR [1-2], Raman spectroscopy [3], or pyrolysis-GC/MS [4-5].

Here we use near-infrared analyses to characterize collagen content and contamination to find suitable samples for radiocarbon dating among bone from Zafarraya, Spain. Specifically, we compare the efficacy of whole bone percent nitrogen (%N) and near-infrared analyses (NIR) to find well-preserved samples. We also present preliminary work identifying the contaminated Zafarraya samples and the specific contaminant applied (paraffin, Paraloid B-72, or polyvinyl acetate-derived polymers).

Whole bone %N measurements were previously collected using an automated carbon and nitrogen elemental analyzer (Carlo Erba EA1108) [6]. A sample must yield 0.76 %N and 1.0% weight collagen to be selected for radiocarbon dating at the Oxford Radiocarbon Accelerator Unit. NIR spectra were collected with an ASD LabSpec 4 (Malvern Panalytical). NIR analyses employ chemometric models, built using PCA and PLSR from specimens of known collagen yield, to characterize well-preserved specimens [7]. We compare whole bone %N to NIR spectra from specimens from Zafarraya. NIR correctly characterizes collagen content in 82.86% of samples compared to 72.0% for %N. NIR proves to be advantageous because the NIR light penetrates deeply, accurately predicts collagen yield, and identifies contamination in a timely, cost-efficient, and non-destructive manner.

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From diagenesis to cremation: The application of FTIR to investigate post-mortem alteration of burnt bones

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Over the last two decades, Fourier Transform Infrared spectroscopy (FTIR) has been extensively used to investigate bone diagenesis. However, post-mortem alteration caused by cremation has received much less attention, even though burnt human bones are frequently found in the archaeological record. Due to high combustion temperatures (<1000°C), the organic components of bone tissues (as well as water) are lost during cremation. Further, the burning process causes significant structural and chemical changes of the inorganic fraction of bone (bone apatite). These changes, along with post-burial diagenetic alteration, make interpreting spectroscopic data obtained from burnt human bone extremely challenging.

In this study, we used FTIR in Attenuated Total Reflectance Mode (ATR) to investigate how structural and chemical changes in cremated bones relate to, and/or are influenced by, different burning conditions. We examine both experimentally burnt animal bones and burnt human bones from various archaeological contexts from across Belgium.

Burned bone specimens from four archaeological sites distributed in two culturally distinct river basins and dating from Middle Bronze Age to Early Iron Age (1500-450 BCE) were selected (207 samples). In addition, a series of laboratory and outdoor burning experiments were performed on the bodies of domestic pigs (Sus scrofa; 87 samples). These were systematically conducted under various burning conditions to create calibration and reference samples useful for archaeological interpretations.

Our results show that by using a specific set of infrared indices (IRSF, C/C, C/P, OH/P, CN/P, and Am/P) it is possible to investigate the circumstances under which cremations occurred (e.g., temperature, duration, fuel, pyre size, pyre location, the position of the body on the pyre, etc.). The combination of archaeological and experimental data opens up the possibility to investigate the changes in cremation practices through the study of structural and chemical changes of bone apatite.

Novel pretreatment strategies to remove contamination from tooth enamel for radiocarbon dating

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Radiocarbon dating bone that is more than a few thousand years old in tropical and arid regions is hampered by the rapid degradation of the protein normally targeted for dating. Sometimes, a few bones with a little protein can be found after screening large numbers of bones. However, we often find that no collagen remains. This can result in low quality chronologies where the only samples available for radiocarbon dating are poorly associated with the event of interest, such as charcoal from burial contexts. Tooth enamel may provide an alternative skeletal material to radiocarbon date. Unfortunately, relatively little work has examined how carbonate contaminants can be removed. Dates are rarely accurate: typically at least 8 % of the carbon in enamel is a contaminant after routine pretreatment. This causes a sample of >50 ka to appear 20 ka. This poster will explore new methods to clean tooth enamel based on a more thorough understanding of enamel diagenesis. Methods have been developed to break and separate enamel into its individual crystallites, allowing leaching of the surfaces, and preliminary work on the protein component will be presented. It will show that ages on tooth enamel can be drastically improved, but that accurate dates are not yet obtained. Typically the equivalent of around 1% of the carbon in enamel is modern in age after the most successful pretreatment attempted. This means that enamel can be radiocarbon dated from Holocene contexts in the tropics with a reasonable level of confidence, but that Pleistocene ages are likely to be grossly underestimated.

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