

Overview

The aim of the CAVES project is to examine the role of climate variability as a major driver of behavioural change in early modern humans. It focuses on Morocco in northwest Africa which now boasts the earliest fossil evidence for *Homo sapiens* (~300,000 years) and some of the longest archaeological records for modern humans anywhere in Africa. Covering most of this timespan is the Middle Stone Age (MSA), a period characterised by a number of major shifts in cultural and technological innovation, including the emergence of symbolic activity. Climate change is frequently postulated as a major agency in driving these innovatory behaviours but, up until now, high-resolution palaeoenvironmental datasets have been largely lacking for this period in northwest Africa. In order to test these ideas, the project compares environmental records from archaeological cave sites using a range of proxies (including isotopes from small mammal teeth and sediment biomarkers), with biochemical evidence from ocean cores. The terrestrial and marine sequences will be tied together using the dating of tephra particles trapped in the cave and ocean sediments from known age volcanic eruptions. This research will be critical in establishing the environmental conditions in which key cultural transitions took place from ~300,000 to 25,000 years ago during the MSA and Later Stone Age. The key objectives that have been met this year were to complete the first stages of analysing and dating proximal volcanic sources on the Azores and Canaries (Figs, 1 & 2), the preparation and sampling for dating and other studies of the ocean core sequences (ODP-958 and GeoB cores 4216,5559) and continuing the analysis of environmental remains from selected archaeological sites (Taforalt, Harhoura II and El Mnasra).



Fig. 1: View from sampling location looking towards Tiede (volcano on Tenerife, Canary Islands). Fieldwork to collect volcanic ash samples was undertaken during July 2024.

1) Tephrochronology

Dr Danielle McLean, Early Leverhulme Fellow, and Dr Emma Horn, the RA postdoc on the CAVES project, under the direction of the Tephra group's leader Prof. Victoria Smith, have made substantial progress in identifying tephra in distal records and developing comprehensive glass chemistry databases for major explosive eruptions from source volcanoes in the Azores and Canary Islands over the past 300,000 years.

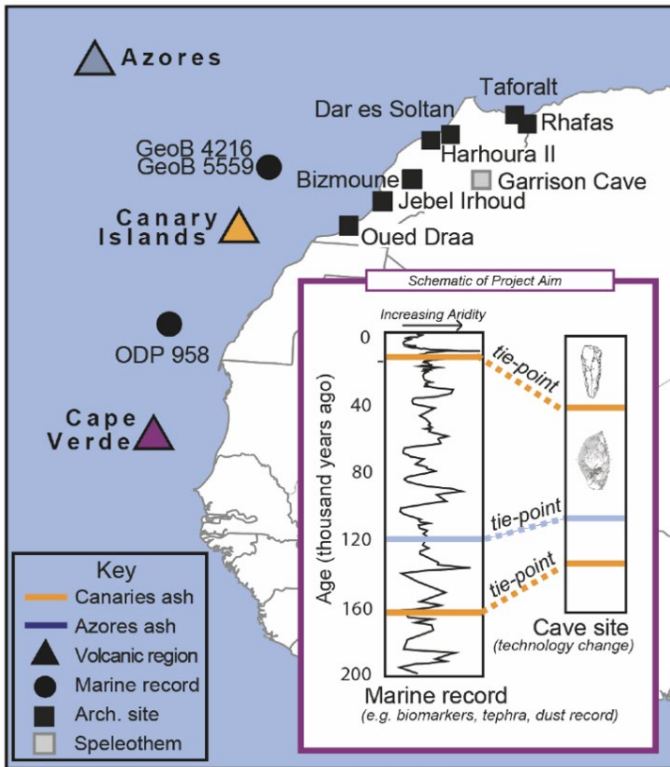


Fig. 2 The location of archaeological sites, volcanic ash sources and Ocean cores in the CAVES project

In the distal records, this year they were able to successfully identify and characterise tephra within samples from both marine archives (ODP 958 and GeoB cores) and archaeological sites (Taforalt and Bizmoune). All samples from these locations have been processed, and cryptotephra layers (non-visible volcanic ash) have been identified. To date, they have also completed the acquisition of major and trace element glass compositions for the Azores eruption deposits. These data are now being prepared for publication. One paper focuses on the composition of the largest eruptions from the Azores. We aim to submit this by the end of September. The other incorporates all Azores data, and is planned for submission in the new year, once new $^{40}\text{Ar}/^{39}\text{Ar}$ ages (in collaboration with Dr. S. Nomade, Laboratoire des Sciences du Climat et de l'Environnement) and ^{14}C (Oxford) dates for the Azores eruptions have been completed.

Currently, the group is focused on geochemically characterising the major and trace element compositions in the archaeological cave layers and marine sediments. In the coming months, these compositions will be compared to established glass chemistry databases from Azores and Canary Islands eruptions to verify correlations. Although samples from major Canary Islands eruptions had previously been analysed, new layers were discovered in the marine records that lacked corresponding proximal samples. To address this, the Tephra Group and collaborator Dr Richard Brown from the University of Durham conducted nine days of fieldwork on Tenerife in July, collecting over 50 samples from eruptions within the relevant timeframe (Fig. 3). The fieldwork was supported by a research award to Dr Emma Horn from the British Institute for Libyan and North African Studies (BILNAS).

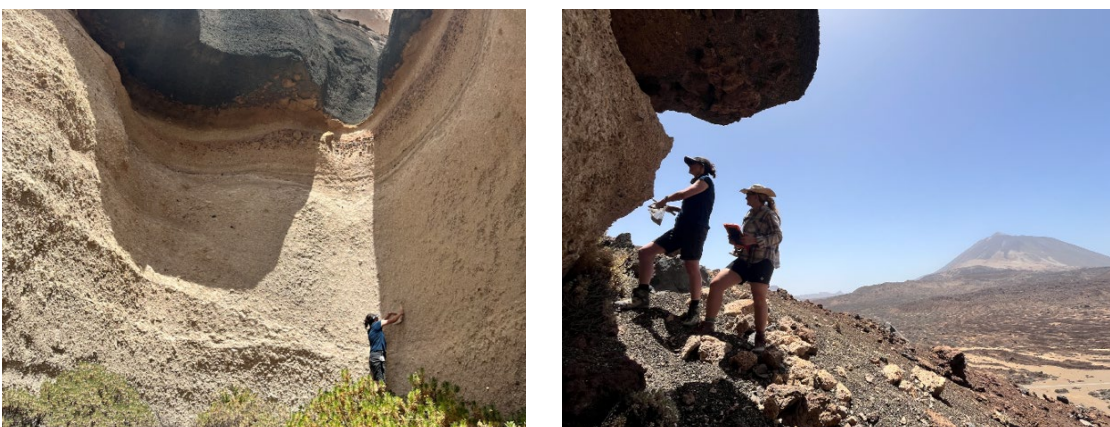


Fig. 3: Sampling the key volcanic deposits (e.g., pumice and ash) within the caldera wall (Tenerife, July 2024).

2) Palaeoecological and isotopic analyses

The objective here is to infer local humidity levels and C3/C4 vegetation composition at archaeological sites during periods of MSA human occupation, using stable isotopes in fossil small rodent teeth. Rodent teeth are found in abundance in stratified sediments at the Moroccan archaeological cave sites but are difficult to analyse for stable isotope composition using conventional pre-treatment and analytical techniques because of their very small size and thin enamel. Over the past year, the RA (Dr Stacy Carolin) has experimented with two methods for isotopically analysing the small samples, (i) a refined pre-treatment and acid dissolution method which reduces required powder sample size from $\sim 1200 \mu\text{g}$ to $\sim 200 \mu\text{g}$ enamel, and (ii) a solid pre-treatment and laser ablation method applicable to enamel as thin as $60 \mu\text{m}$ and sampling surface area as small as 1.5 mm^2 (Fig.4) The refined acid dissolution method is quasi-destructive, requiring a small chip of enamel broken off from the tooth, and is time and labour intensive, requiring ~ 3 hours of meticulous manual cleaning and preparation per tooth to remove all dentine from the enamel before acid dissolution. Further, the individual tooth samples of *Mus sp.* (mouse) and *Gerbillus sp.* (small gerbil) genera are too small for the refined acid dissolution method. The laser ablation method is minimally destructive (Fig. 4), with cleaning and preparation steps taking just 5-10 minutes per tooth. The accuracy of the isotopic analyses on the small rodent tooth samples using both methods was confirmed using external and internal reference materials, and by comparing the results from both methods on enamel material from the same tooth to confirm reproducibility.



Fig. 4. Left: Tooth appearance after two performed sampling methods for enamel isotope analysis: laser ablation and enamel chip removal for acid dissolution to compare the isotopic results between both applied methods. Right: Modern *Mus spretus* mandibles mounted in preparation for laser ablation analysis

This year Carolin has isotopically analysed a total of 121 fossil teeth of *Meriones sp.*, *Gerbillus sp.*, and *Mus sp.* using the laser ablation method and 10 fossil teeth of *Meriones sp.* (large gerbil) using the refined pre-treatment and acid dissolution method. She has also isotopically analysed 52 modern teeth of *Meriones sp.*, *Gerbillus sp.*, and *Mus sp.* using the laser ablation method (e.g. Fig. 4). The results of these analyses are now being prepared for multiple publications. The first publication will discuss the refined methodologies developed for isotope analysis of extremely small rodent teeth, and the application of this methodology to analyse *Mus sp.* tooth samples at multiple Moroccan sites. Combined with previously published work on the isotopic composition of larger modern rodent teeth, we are now able to comment on i) the diets of multiple rodent species from two genera (mouse and gerbil) in Morocco, and ii) the utility of isotopic analysis of various tooth types (molar and incisor) of multiple genera in environmental reconstructions. Both

points have significant implications for palaeo-environmental studies. The subsequent publications will discuss insights gained from the fossil *Meriones sp.*, *Gerbillus sp.*, and *Mus sp.* specimens on local past humidity and vegetation changes. Specimens have been analysed from sediment layers of El Harhoura II cave which contain MSA lithics with proposed cultural attribution to the Aterian; from El Mnasra cave which contain Aterian lithics characterised by tanged tools as well as perforated *Nassarius sp.* shells; and from Taforalt cave Sector 12, believed to include the oldest excavated sediment of Taforalt, possibly > 130 kyBP.

3) Biomarkers as evidence of vegetational change and past human subsistence activities.

Dr Nick O'Mara (Yale University), a partner in the project, visited Kevin Uno's new lab at Harvard in June 2024 to process, extract, and analyse samples from a sequence spanning the MSA-LSA transition at Taforalt Cave for biomarkers (Fig. 5). Data analyses of plant wax distributions are underway. O'Mara plans to return to Uno's Lab this autumn to measure carbon and hydrogen isotope ratios of the plant waxes and to measure PAH concentrations. The distribution and carbon isotope signatures of plant waxes will be evaluated to determine changes in local vegetation. The hydrogen isotope signatures of these waxes will be measured to create a history of local paleohydrological shifts. Third, polycyclic aromatic hydrocarbons (PAHs), which are organic combustion by-products (fire biomarkers), from ashy hearth layers in this section will be used to glean new insights into both changes in fuel use and fire temperatures through time within the cave. These samples will provide new information regarding both the local ecological and



hydrological backdrops for cultural transitions. These data will be paired with measurements of the same proxies in marine core ODP 958, aided by absolute tie-points provided by cryptotephra analysis, to assess both the potential local and regional scale climate drivers of cultural changes observed at Taforalt. This year, a suite of modern plants was collected by Moroccan graduate student Ismail Ziani near Taforalt cave under the guidance of his advisor and project collaborator Jacob Morales (University of the Las Palmas, Canaries). O'Mara will analyse plant wax biomarkers from these modern plants to develop an interpretive framework for the ancient biomarker data from Taforalt cave sediments.

Fig 5 Photo of biomarker samples being taken by Dr Nick O'Mara at Taforalt Cave, Morocco

4) Marine core analyses for North African paleoecology and paleoclimate reconstructions

Dr Bryce Mitsunaga, the RA postdoc in Uno's lab at Harvard University, has spearheaded efforts in marine core biomarker analyses since joining the project in 2024. Mitsunaga and undergraduate Annika Dellinger processed ~70 sediment samples from ODP core 958B over the summer and plant wax biomarker analyses are in progress. Uno and Mitsunaga also started work on a new marine core, GeoB 4216, which spans the last 220,000 years. This core fills in critical gaps in the ODP 958B core record and will strengthen the regional climate reconstructions over the primary study interval for the project (~330 ka to present). After Uno and Mitsunaga obtained permissions and established a collaboration with the German research group who drilled the GeoB 4216 core, Mitsunaga travelled to the core repository in Bremen, Germany to sample the core. He returned with 85 additional biomarker samples that will be processed this fall. Analyses of samples from both cores will include plant-wax carbon isotope ratios of *n*-alkanes to track changes in the relative abundance of C3 and C4 vegetation through time, whereas hydrogen isotope ratios will track

changes in precipitation. PTMEs, a class of biomarkers produced by grasses, will serve as an important vegetation proxy. Lastly, PAHs will be used to reconstruct biomass burning and will yield a paleofire record, similar to other palaeofire records from further south. The cores have been sampled at 3kyr resolution (~7.5 cm) to resolve orbital scale variability. The core age model for ODP 958 has been established through new alkenone sea surface temperature (SST) measurements made by Mitsunaga this summer. In addition, cryptotephra work by Smith and McLean on the ODP 958 core has provided independent verification of the SST age model with excellent agreement. Moreover, the cryptotephra records from both cores (ODP 958 and GeoB 4216) will provide chronological tie points to the cave sites, synchronizing the marine and terrestrial records. Mitsunaga will lead a CAVES co-authored presentation of preliminary organic geochemical data at the annual meeting of the American Geophysical Union in December 2024.

5) Initial synthesis of results

Well-preserved cryptotephra layers have now been confirmed in the main cave sequences and marine records (Fig 6) that will be essential for synchronising archaeological and palaeo-environmental datasets. Once the new results of tephra sourcing and dating have been fully integrated, the project will be able to present first generation models that will test hypotheses concerning continuity of human occupation and conditions surrounding key cultural transitions in northwest Africa in the last 300,000 years.

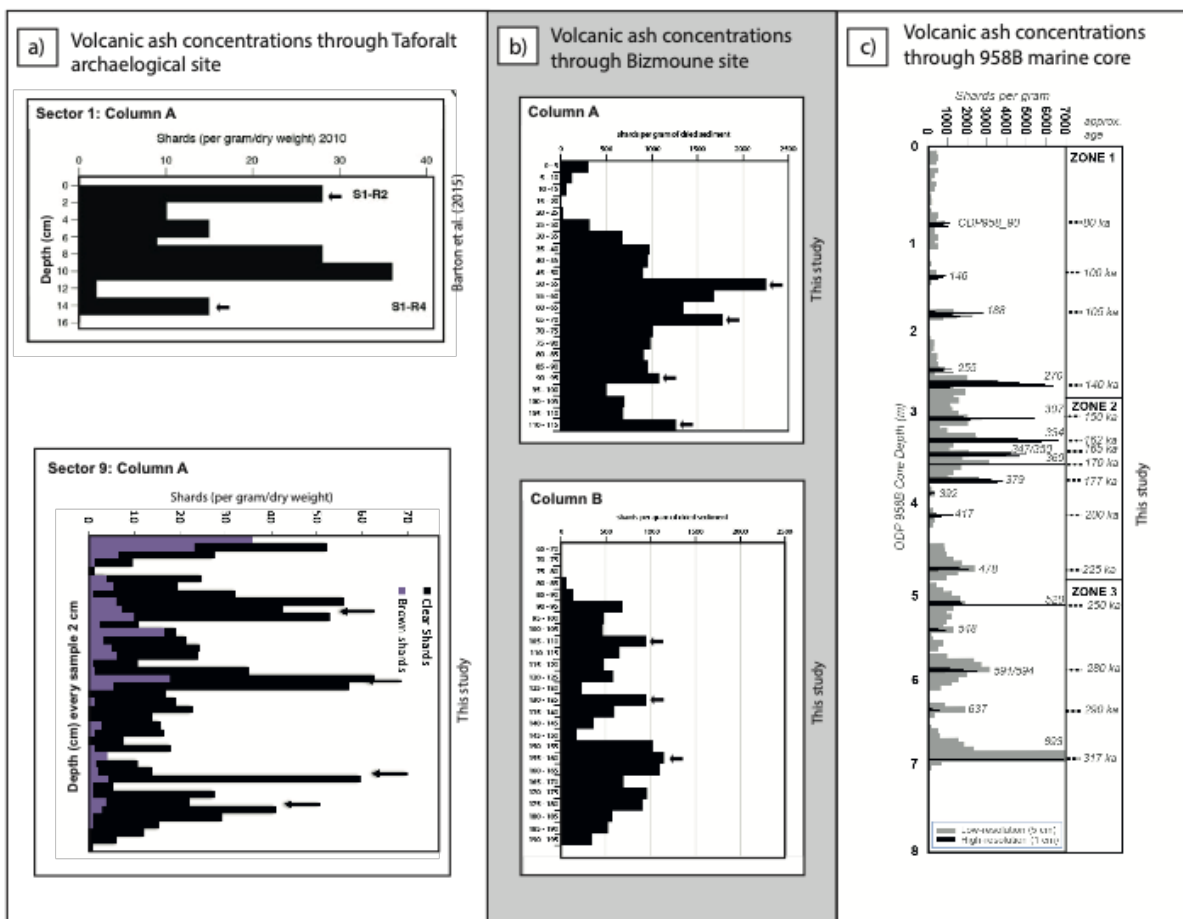


Fig. 6: Volcanic ash stratigraphy through selected columns at: (a) Tavoralt, (b) Bizmoune (archaeological sites), and (c) marine core 958B. The peaks in shard concentrations show the position of the volcanic eruptions in the different records.

Acknowledgements

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