



	<b>Experiment title: Alterations in fossilization process in teeth of small vertebrates by infrared spectrometry (step 2)</b>	<b>Experiment number:</b> ES-42
<b>Beamline:</b> ID21	<b>Date of experiment:</b> from: 15/04/2013 (08 am) to: 20/04/2013 (08 am)	<b>Date of report:</b> 26 april 2013
<b>Shifts:</b> 12	<b>Local contact(s):</b> Hiram Michel-Castillo	<i>Received at ESRF:</i>

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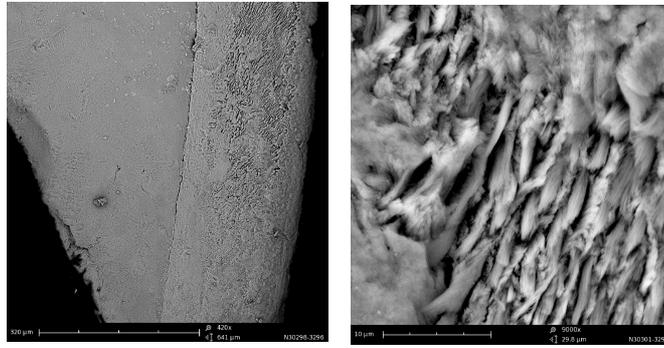
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**Report:**

A “long term” three-step study was planned to identify alteration processes occurring during fossilization in teeth of small vertebrates (Rodents). First, modern fresh teeth of Rodents will be studied to build a set of references for the mineralogy, organo-mineral ratios, crystallinity, fluor contents of enamel and dentine in molars and ever-growing incisors. Then, modern teeth extracted from regurgitation pellets or carnivore faeces will be studied to estimate the changes induced by the digestion of the predator. At last, fossil teeth from archaeological sites in Morocco will be analysed. Changes in crystallinity and organo-mineral ratios... will be determined using FTIR maps and spectra, then will be compared with microstructural changes (SEM and AFM), changes in the bulk organo-mineral contents (laser confocal UV and fluorescence), and elemental chemical composition modifications (electron microprobes). Additionally, the composition of the enamel and dentine will be compared with that of the sediment.

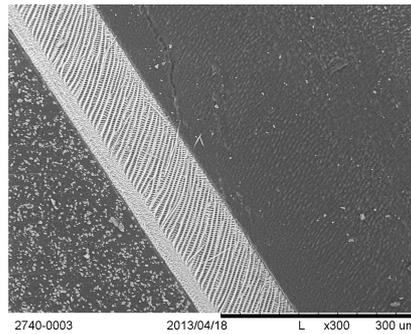
The first experiment (EC 792) was dedicated to the first stage: studies of fresh teeth. Incisors from wild animals (*Meriones*) and a breed one (*Rattus rattus*) were selected for FTIR analyses. This second experiment was dedicated to incisors extracted from modern regurgitation pellets and fossil teeth. All samples are incisors from *Meriones*. Fossil samples were selected from levels 1, 2, 3, 7 and 8 collected in the El Harhoura 2 cave (Morocco) where hominid samples have been found. The cave shows 12 archaeological levels, and the studied levels were chosen from taphonomic and stratigraphic criteria. The SEM preliminary examination of the outer surface (enamel and dentine) of the teeth have shown that the microstructures were preserved.

Outer surface of enamel before the polishing process

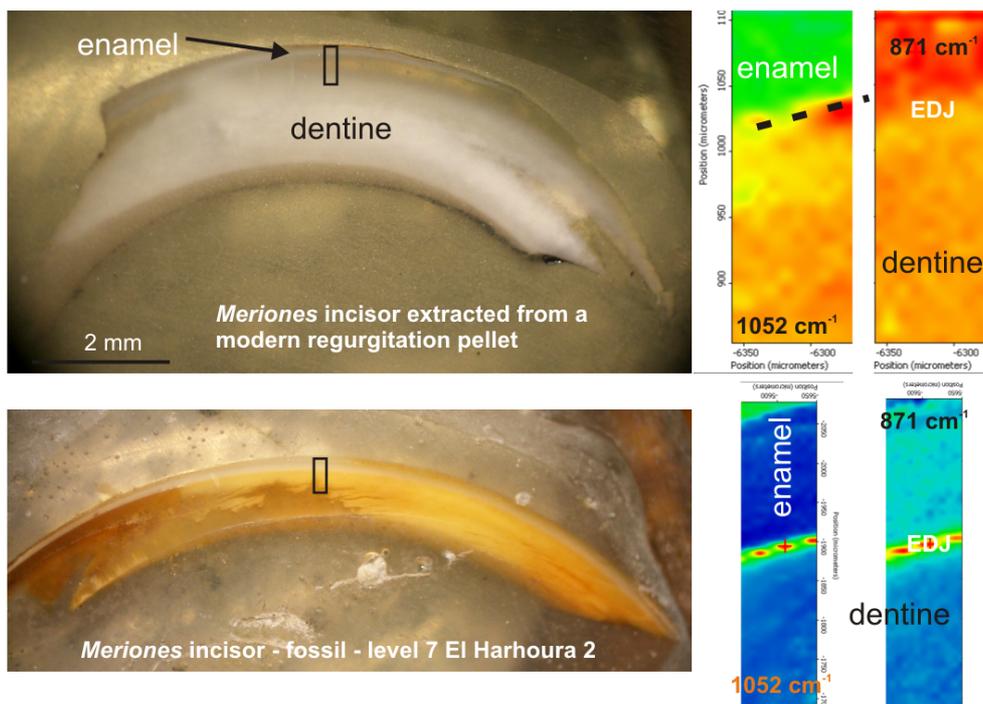


Then, teeth were polished to obtain a flat surface, with a very light etching to clean the surface and to reveal the structure of enamel and dentine.

SEM image showing the enamel and the dentine (right)



In all incisors, “large” maps (> 8 hours) were done on both enamel and dentine, the EDJ (enamel dentine junction) being included. Despite some “lost beam” periods at the beginning of the experiment, all the samples have been successfully mapped. Tuesday was a day without synchrotron beam. Thus, we use the internal source of the FTIR spectrometer to acquire spectra on freshwater pearls to test the feasibility to distinguish aragonite – calcite - vaterite for a possible future proposal. A very first look at the maps shows that all the studied fossil teeth are different. Most samples are still apatite, but variations occurs between the enamel and dentine in a given tooth, and between enamels of the different samples as well as dentines.



Polished sections of two studied samples (left) and FTIR maps at two  $\text{PO}_4^{3-}$  bands. In the fossil tooth, the enamel – dentine junction (EDJ) is clearly visible. In both teeth, enamel and dentine differ.

Detailed analyses of the maps for specific bands will be done in the next months, to quantify crystallinity, organic – mineral ratios, F and Sr contents, etc. Are the changes induced by the digestion of various birds of prey distinguished using FTIR analyses is one of the question? In such small teeth (thickness < 2 mm), localized analyses are the only way to have data on the enamel on one hand, and dentine on the other hand. Observation of the mapped zones will be done using scanning electron microscopy, epifluorescence and electron microprobe chemical analyses.

Teeth have been collected thanks to a French ANR grant (MOHMIE, 6è extinction), the aim of which is to detect the potential role of human colonization of wild fauna. The results of this experiments will be compared to the chemical composition of the sediments and soils in which the teeth were embedded, and with the taphonomic data.

All these observations will be used in future manuscripts.