

XEOL study of the kinetics of optical and chemical changes induced during synchrotron X-ray analysis of fossil teeth

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X-ray beams produced by a synchrotron source have properties of high brilliance and spatial coherence that make them highly suitable for studying a range of ancient materials. For example, synchrotron X-ray methods have been used to study fossil teeth to determine the age at death of humans using X-ray micro-tomography [1] and to identify chemical elements as markers of provenance from prehistoric sites and markers of diagenesis of Palaeolithic mammoth ivory using X-ray fluorescence [2]. However, high-flux X-ray measurements can induce chemical changes that can be visible to the naked eye (e.g. darkening of teeth [3]) or invisible (e.g. degradation of ancient DNA measured during quantitative polymerase chain reaction amplification [4]). The parameters governing the chemical mechanism and kinetics involved require further investigation.

In this work, we studied a corpus of fossil teeth (five specimens; 20,000 to 70 million years old) from five distinct animals and monitored changes using full-field multispectral X-ray excited optical luminescence (XEOL) imaging on the PUMA beamline of SOLEIL. After irradiation, evolution with time of the observed changes induced by the beam using photoluminescence (PL) micro-imaging was monitored.

During irradiation, we characterized the decay of the XEOL signal on a minute time scale under the high-flux X-ray irradiation as a function of the absorbed dose, the type of fossilized biological tissue (enamel, dentine), the incident energy, the detection conditions and the use of concomitant UV illumination. In particular, we found that UV illumination during and after the irradiation led to a faster and more complete recovery of the XEOL signal. On the enamel of a Meshippus tooth, PL imaging after cessation of irradiation showed the presence of non-emissive lines correlated with the X-ray irradiation (immediately and 6 months after irradiation; Figure 1); however, no changes were observed in other tissues. The effect of the parameters on the decay of XEOL is being further investigated.

We have therefore identified characterization strategies to optimize the study of the secondary effects of irradiation and to better mitigate them in future experiments. This work is part of the general development of strategies to monitor, mitigate and better visualize the side effects of high-flux irradiation [5,6].

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