Investigation on corrosion of iron archaeological artefacts using microfocused synchrotron X ray absorption spectroscopy and imaging

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abstract Micro x-ray absorption and elemental cartography have been used to study the corrosion products of ferrous ancient artefacts and particularly the role of the chlorinated phases.

Synchrotron based micro X Ray Absorption Spectroscopy was used in the present study to obtain micro scale chemical information such as coordination and oxidation state of phases constituting corrosion products within archaeological iron artefacts buried in soil. This iron corrosion process after excavation is related to the presence of chlorine, and its knowledge is particularly important for restoration and conservation of these metallic artefacts.

The samples available for X ray microprobe analyses are cross section from iron corroded objects coming from archaeological excavation sites dating from 12th to 16th century AD. Previously, several analytical techniques such as µXRD and µRaman have been employed to reveal morphological, compositional information of corrosion products. X-Ray Absorption Near Edge Structure (µXANES) was used to determine the spatial variation of the predominant Fe oxidation state and the corresponding crystallographic phase. The micro-XAS and imaging experiments reported here were conducted on the LUCIA beam line at SLS. The analyses performed at Fe and Cl K-edge (µXANES) revealed the correlation between the Fe^{2+} and Fe^{3+} distribution in the corrosion products, and the evolution of the chlorine concentration. In addition to the presence of beta iron hydroxide β–FeOOH: akaganeite, in iron corrosion product, we highlights the presence of an other important phase, the β–$Fe_2(OH)_3Cl$ hydroxychloride. This result is particularly interesting because, to our knowledge, this phase has never been identified in archaeological artefacts corrosion products. These finding help to gain new insights concerning the influence of such phases in iron corrosion mechanism within their precise characterisation.

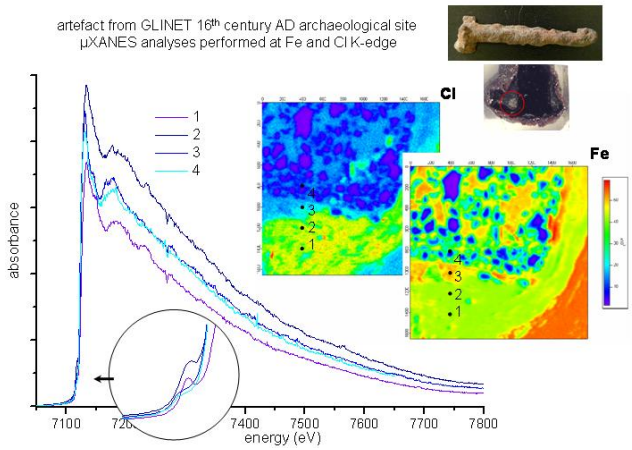

Figure 1: iron XAS spectra for different spots of the images (100*100µm^2) where are presented the elemental repartition of Cl and Fe.

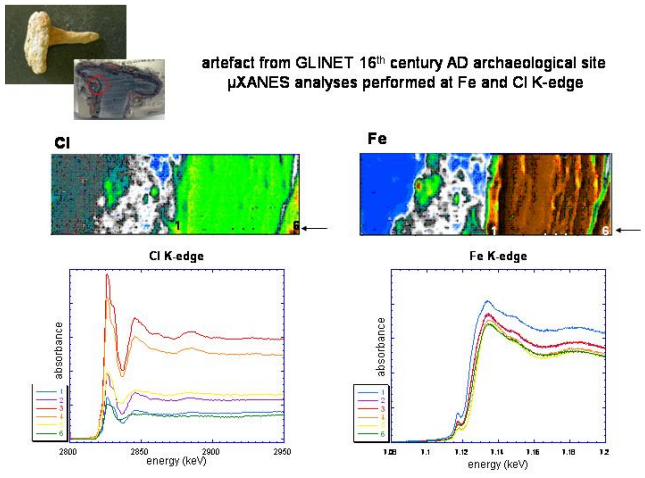

Figure 2: chlorine and iron XAS spectra taken at different locations on the image shown on top and separated by 50µm.