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

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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 techniques were required in order to answer questions about iron corrosion process related to the presence of chlorine, particularly for restoration and conservation of metallic heritage application. Indeed, the deterioration after excavation of archaeological artefacts buried in soil is often associated to the presence of chlorine ions in corrosion products.

The samples available for X ray microprobe analyses are cross section from iron corroded objects coming from particular archaeological excavation sites dating from 12^{th} to 16^{th} century AD. Previously, various analytical techniques are employed to reveal morphological, compositional information of corrosion products; some others techniques are based on structural investigation of artefacts, such as µXRD and µRaman. [1-3] In this specific study, we present a number of case studies applying synchrotron-based micro absorption spectroscopy to iron artefacts. 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 experiments reported here were conducted on the ID21 beam line at ESRF and 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 the well known beta iron hydroxide $\beta-FeOOH$: akaganeite, in iron corrosion product, we highlight the presence of an other important phase, the $\beta-Fe_2(OH)_3Cl$ 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.

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