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P10-Dechlorination of ferrous archaeological artefacts in subcritical conditions: understanding of the corrosion layer transformation thanks to a multi-scale characterisation

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Dechlorination of ferrous archaeological artefacts is rendered necessary after excavation in order to remove chloride species present at the metal/corrosion layer interface. Indeed, when artefacts are exposed to air after a long period of burial they can suffer severe damages. This is due to the reactivation of the corrosion processes through the migration of those chlorides that provokes local acidification. In the conservation field, treatments are based on the immersion of the artefacts in alkaline bath in order to transform the chloride containing phases (akaganeite and β-$Fe_2(OH)_3Cl$). But specifically for artefacts of high dimensions the process can last from several months to years. An innovating approach based on treatment in subcritical conditions (180°C, 35 bars) allows to reduce by a factor of 10 the treatment duration. The optimisation of this treatment requires the determination of the reaction pathways between the initial corrosion layers and the transformed ones after treatment. Therefore synthetic phases representative of archaeological corrosion products formed in aerated environments (goethite, lepidocrocite, magnetite, akaganeite, maghemite) have been treated separately to study their transformation during the treatment. Specifically in-situ experiments under synchrotron radiation have been performed on akaganeite in order to determine the different transformation steps of this phase. The behaviour of two akaganeite, the first one obtained by laboratory synthesis and the second one from archaeological origin has been compared. Thanks to XANES at the Fe K-edge in sub-critical conditions it has been highlighted that depending on the heating rate the akaganeite transforms either in hematite or in a mix of goethite and hematite. High resolution X-ray diffraction under synchrotron has allowed to determine the evolution of the crystalline structure of the remaining akaganeite depending on the chloride content of this phase. Last the results show that the transformation kinetics depend on the origin of the precursor and that archaeological phases transform much slower than synthetic ones.