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► **To cite this version:**

Robin Le Penglau, M. Bouhier, Delphine D. Neff. Description of archeological corrosion layers thanks to multivariate analysis. Eurocorr 2017, Sep 2017, Praha, Czech Republic. cea-02341039

HAL Id: cea-02341039

<https://cea.hal.science/cea-02341039>

Submitted on 31 Oct 2019

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Description of archeological corrosion layers thanks to multivariate analysis

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Predict how corrosion evolves for centuries in iron materials is decisive as well in nuclear field in order to evaluate the nuclear waste containers thickness as in cultural heritage for conservation of metal items. Our goal is to describe the Corrosion Product Layers (CPLs) which grew up under natural conditions in order to understand the corrosion mechanisms and the role of the layer in those phenomena. The aim of this study is to correlate both elementary and structural data thanks to chemometrics. The development of a reliable methodology is based on the case of archeological artefacts corroded in atmosphere. CPLs contain goethite, lepidocrocite, akaganeite and ferrihydrite ($\text{Fe}_2\text{O}_3, n\text{H}_2\text{O}$). Ferrihydrite is an electrochemical reactive phase which can see its reactivity decrease by adsorption of chemical species coming from the environment like phosphate and sulfate [1]. Phases and chemical elements are heterogeneously distributed at micrometric scale and these phenomena have to be taken into account to describe the long term corrosion processes in atmosphere.

In order to obtain a representative description on large areas, Raman and SEM-EDX hyperspectral images were acquired on the same zones. Hyperspectral images are about several tens of thousands spectra. Raman data have been analyzed by Multivariate Curve Resolution–Alternating Least Squares (MCR-ALS). This algorithm allows to detect every crystalline phases in the analyzed zone and to quantify phase proportion [2].

To evaluate the correlations between structural and chemical data, Raman and EDX hyperspectral images (micrometric resolution) have been examined together. A first approach consists in merging Raman and EDX spectra together after several preprocesses. Then a PCA is used on merged spectra. A second approach is based on the use of component images obtained from the analysis of both datasets separately. For Raman spectroscopy, the quantification images of component extracted by MCR-ALS are used. For EDX, intensity signals of each elements are extracted. Multiblock treatments are performed on the images obtained from these data [3, 4] to specify the possible correlation between elementary and structural information in CPLs.

First results on merged spectra, thanks to PCA, seems to show a correlation between phosphorus and other minor elements as sulfur with ferrihydrite on one of the principal components.

1. Benali, O., et al., *Effect of orthophosphate on the oxidation products of Fe(II)-Fe(III) hydroxycarbonate : The transformation of green rust to ferrihydrite*. *Geochimica and Cosmochimica Acta*, 2001
2. de Juan, A., et al., *Chemometric Tools for Image Analysis*, in *Infrared and Raman Spectroscopic Imaging*. 2009
3. Tucker, L.R., *An inter-battery method of factor analysis*. *Psychometrika*, 1958
4. Hanafi, M., A. Kohler, and E.-M. Qannari, *Connections between multiple co-inertia analysis and consensus principal component analysis*. *Chemometrics and Intelligent Laboratory Systems*, 2011