



	Experiment title: X-ray absorption Studies of chlorinated phases formed ferrous archaeological artefacts	Experiment number: ME-1104
Beamline: ID21	Date of experiment: from: 27.04.05 to: 02.05.05	Date of report: 24/06/05
Shifts: 15	Local contact(s): Doctor Marine COTTE	<i>Received at ESRF:</i>
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Report:

Synchrotron based micro X Ray Absorption Spectroscopy and elemental cartography have been used in the present study to obtain micro scale chemical information of chlorinated phases constituting corrosion products within archaeological iron artefacts buried in soil. 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 analyse all the thickness of the corrosion products..

Indeed, chlorine levels of specifics chlorinated phases vary considerably, from a sample to another and even in the same artefact. We have to understand the evolution of such phases containing chlorides and particularly the possible release of chlorides ions.

The first part of the experiment was dedicated to the collection of references samples of iron oxihydroxides including chlorides as akaganeite. Thus, a complete set of reference of akaganeite powder synthesised in our laboratory and containing different Cl amounts was analysed with macrobeam. The akaganeite powder was synthesised and wash in order to observe desalinisation effect on cristal structure. To our knowledge, it seems that it is the first time that this phases is investigated with this technique and procedure.

The observations at Cl K edge, that we can see on figure 1, show important variations due to the release of Cl ions :

- The XANES amplitude of the oscillations increase when chlorine content decrease.
- The XANES pre edge decrease with the chlorine content. These results have to be understood as structural variation and must be compared to the variations observed on archaeological artefacts.

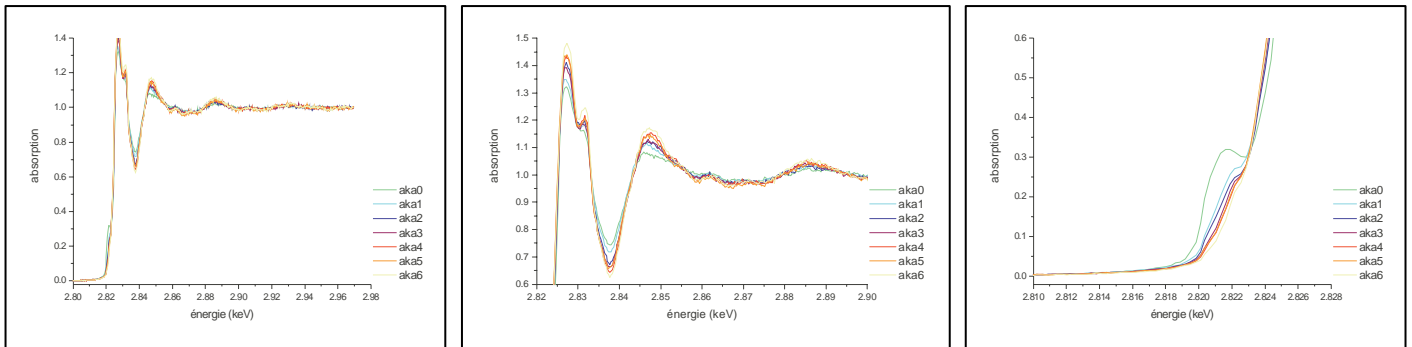


Figure 1 : XANES spectra at Cl K edge of akaganeite containing different amount of chlorine

In a second step, the beam was focused on a $1 \times 1 \mu\text{m}^2$ surface in order to localise and analyse the different chlorinated phases in the layers of the archaeological artefacts. Fluorescence profiles and/or images were collected in order to localise precisely the Cl in the layers. Then, μXANES profiles were made on the same profile at the Cl K-edge. In this second part, seven samples were analysed. Figure 2 presents results obtained on artefact from GLINET 16th century AD archaeological site.

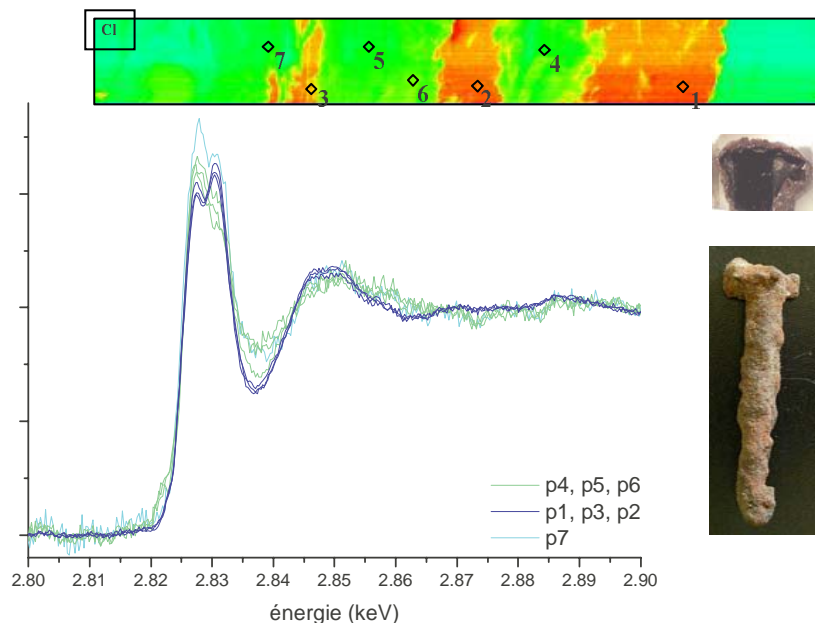


figure 2 : artefact from GLINET 16th century AD archaeological site - μXANES spectra performed at Cl K-edge for different spots of the images where are presented the elemental repartition of Cl.

As we can see, it was possible to distinguish different pre-edge and edge structures in the zones containing different amounts of chlorine.

All of these results have to be associated with the results obtained by XANES studies at Fe K edge (see ME 949).