

Experiment title:

Characterisation by means of μ -diffraction and μ -fluorescence analyses of Fe-oxyhydroxides heterogeneous aggregates formed by supergenic alteration of natural Fe-Cu-sulfides

Experiment number: CH2095

Beamline: ID18f

Date of experiment: from: 18 Nov 05 to 21 Nov-05

Date of Report:

Shift: 12

Local contact: Sylvain Bohic

Name and affiliation of applicants

Carbone C., Marescotti P., Lucchetti G., DIP.TE.RIS. University of Genoa, Italy

Martinelli A., INFM-LAMIA Italy

Report

In the last years, several techniques based on synchrotron radiation have been applied to environmental sciences and gave the possibility for non-destructive investigations with micron spatial resolution. These studies applied a combination of synchrotron methods to determine the elemental distribution and speciation of metal elements in soils and waste rock using several synchrotron-based techniques, such as μ -XRF, μ -XRD, and μ -EXAFS (Manceau et al., 2003; Morin et al., 1999; 2001). The results showed that the elements that circulate in solution are trapped in the different phases through adsorption or coprecipitation.

We report preliminary results obtained using simultaneous μ -diffraction and μ -fluorescence analyses performed on the ID18F beamline at ESRF (Grenoble) using polymer compound refractive lenses (CRL) achieving a x-ray spot size of 2 μ m (Nazmov et al., 2004). The aim of the present experiments is to correlate the crystallochemical analyses and mineralogical data on Fe-oxyhydroxides-bearing samples formed during Acid Mine Drainage processes.

The studied samples are: a) mineralised fragment which represents the transition from the unaltered sulfides to authigenic Fe-rich assemblages (Fig. 1a); b) stratified crust (Fig. 1b) formed by ageing of stream sediments precipitated from acid mine waters discharged at mine adits.

Simultaneous μ -XRF (excitation energy = 28 keV; spectrum collection using a Si(Li) solid state detector; detection limit = 0,01 ppm for $30 < Z < 35$ and $< 0,1$ ppm for $Z > 25$) and μ -XRD (monochromatic X-rays of $\lambda = 0.44285$ Å; spectrum collection using a high resolution MAR CCD camera), allowed to correlate the chemical and mineralogical variations across 2 to 4 mm transects characterised by submicrometric to micrometric mineralogical and textural heterogeneities.

μ -diffraction analyses performed on mineralised fragment (Fig. 1a) allowed to characterise the transition from unaltered to completely oxidised sulfide-bearing crusts. The transition is marked by the disappearance of the sulfide mineralisation assemblages (pyrite + chalcopyrite + quartz) that are progressively replaced by goethite (Fig. 2a). μ -fluorescence patterns evidenced that most of the metals released during sulfides alteration are efficiently uptaken by the new formed authigenic phases (Fig. 2b)

Results from μ -diffraction patterns (Fig. 3a) show that stratified crusts are characterised by mineralogical heterogeneity even at the micrometric scale; the different coloured layers mark the transition from goethite-rich zones (containing significant amounts of 6-line ferrihydrite) to almost pure hematite-rich zones (without 6-line ferrihydrite). Minor amounts of other mineral species, such as lepidocrocite and phyllosilicates, are occasionally present.

Chemical analyses obtained with μ -XRF, combined with μ -XRD patterns, evidenced a significant mineralogical control on the mobility of chemical elements of environmental concern (such as Ni,

Cu, Zn, As, Se, Rb, Sr, Nb, Mo, and Ag); in particular goethite-rich layers are enriched in Ni, Zn, Rb, whereas hematite-rich precipitates selectively concentrate As, Se, Mo, Cu (Fig. 3b).

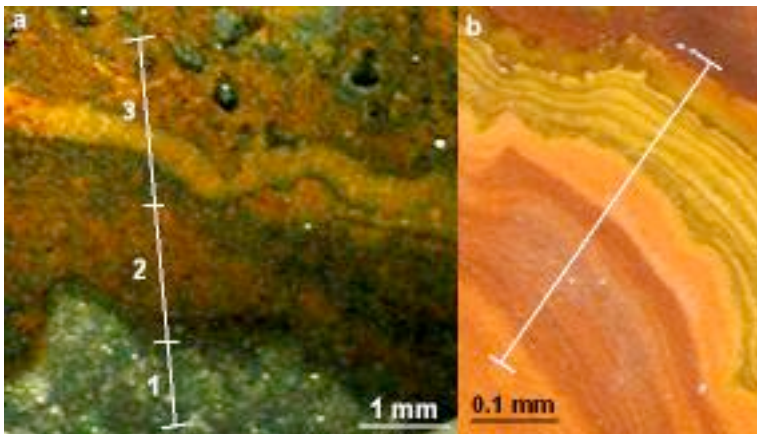


Figure 1. Optical microscopy images of investigated samples: a) transition from unaltered sulfide mineralisations (1) to completely altered layer (3). The number 2 indicate the transition zone. b) Stratified Fe-oxyhydroxides crusts.

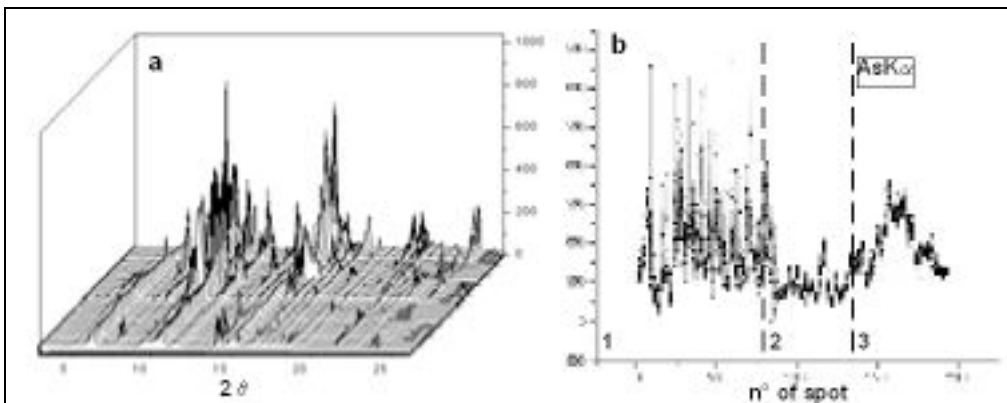


Figure 2. a) μ -XRD analyses showing mineralogical variations along the transect of Fig 1.a (analytical step 2 μ m); b) selected μ -XRF line scan evidencing the chemical variation (As-K α) along the same transect.

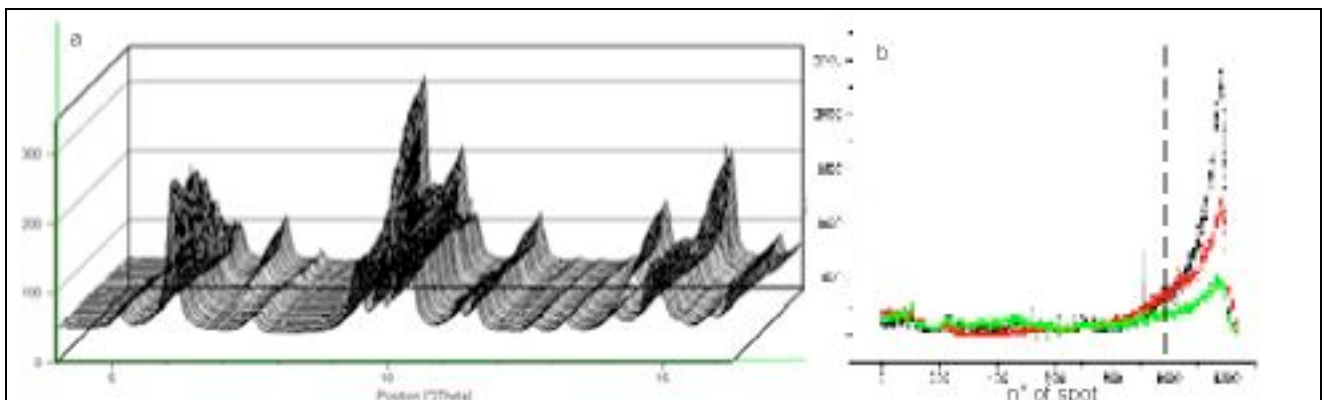


Figure 3. a) μ -XRD analyses evidencing mineralogical variations at the micrometric scale along the transect in Fig 1b (analytical step 2 μ m). It also shows the relationships between goethite and hematite. b) μ -XRF line scan evidencing the chemical variations of Mo (black), As (red), and Se (green) at the transition (dashed line) from goethite-rich to hematite-rich layers.

References

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