Mineralogy of reactive iron as driving force for microbial activity in marine sediments

Patrick Meister1, Aude Picard1,2, Christian März3, Anna Lichtschlag4

1Max Planck Institute for Marine Microbiology, Celsiusstrasse 1, 28215 Bremen, 2MARUM, Center for Marine Environmental Sciences, Bremen, 3ICBM, Universität Oldenburg, Carl-v.-Ossietzky-Str 9-11, 26111 Oldenburg

Beamline and settings:

This experiment is new and we report analyses performed during the first beamtime in November 2009. The purpose of the experiment was to measure the oxidation state of iron and to determine which Fe-minerals are dominant in deep sea-sediments. We used the A1 beamline at the DORIS III facility for this purpose. EXAFS spectra were recorded at the Fe K-edge in known iron minerals and in sediments in the fluorescence mode using a PIPS detector for fluorescence. The size of the beam on the samples is quite large (5 mm horizontal x 0.8 mm vertical) but it allows the measurement of good average signal in Fe minerals and sediments.

Sample preparation:

Sample and standard materials were prepared by different methods. Standard minerals were measured as entire fragments when available or as pellets (sea preparation below). Anoxically-stored sediments were sub-sampled and loaded on a sample holder in an anaerobic chamber to maintain anaerobic conditions. Dry sediment or standard samples were finely ground, mixed with cellulose and compressed to form a pellet. For comparison, selected samples were prepared using both methods.

Completed measurements:

During our beamtime, we recorded EXAFS spectra at the Fe K-edge of 19 Fe-containing minerals (standards) and 30 deep-sea sediment samples. The sediment samples came from the following locations: Eastern Equatorial Pacific (Ocean Drilling Program Site 1226), Nankai Through (International Ocean Drilling Program Leg 319), Bering Sea (IODP Leg 323, sites 1341, 1343, 1344), Peru Margin (ODP site 1229), Arctic Sea, Black Sea.

Results:

Spectra were recorded between 6960 and 8000 eV in 677 points of 1s each. The resolution was of 5 eV before the edge, 0.25 eV around the edge and 0.8 eV after the edge. Fe concentration was high enough to produce good-quality EXAFS spectra at the Fe K-edge (Fig. 1). All measurements were done at least in duplicate and duplicate spectra from samples and standards were identical, indicating no photo-oxidation or photo-reduction. Standard Fe-minerals show clearly distinct spectra. Moreover, preliminary results allow to distinguish between Fe(III) and Fe(II) minerals in sediments. Sample preparation had no significant influence on the spectra since spectra of pellets and fragments were identical. A difference was noticed between spectra of anoxic sediment samples dry/frozen sediment samples. Analysis of the spectra was attempted using the ATHENA software [1], however some standard components are still missing to proceed the complete analysis of the spectra in the sediments. They will be measured with other samples during the second round of beamtime in May 2010.
Figure 1: Fe K-edge XANES spectra of selected Fe minerals. In pyrite, the Fe oxidation state is II, in goethite the oxidation state is III, while in magnetite and vivianite the oxidation states are mixed.

References