

Metal-Aid Secondment Report

German Research Centre for Geosciences (GFZ)

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The focus of my studies lies on the investigation of siderite as a potential material for *in situ* remediation of contaminated groundwater. Therefore, I have conducted experiments in the laboratory to test the efficacy of siderite to remove lead (Pb) and copper (Cu) from acidic solutions. Over the past months, I have achieved to develop a batch-experiment method in which Pb and Cu were removed from solution almost entirely over the course of the experiment. This, however, was done under ambient, oxic, laboratory conditions. In order to assess how the mechanisms of heavy metal sequestration are affected by oxygen in the acidic aqueous solution, equivalent experiments required the use of an oxygen-free environment, i.e. an anaerobic chamber (glovebox). Because the glovebox in the UCL geochemistry laboratory is not yet fully functional for this purpose, it was decided to conduct the planned experiments during a secondment at the German Research Centre for Geosciences (GFZ) in Section 4.4 “Interface Geochemistry”, Potsdam, Germany, where they have an anaerobic chamber perfectly suitable for my work. Liquid chemical element and solid analyses via various techniques would also be available.

During the first part of my secondment at the GFZ, I have completed a series of batch experiments on siderite dissolution in the presence of Pb and Cu under anaerobic conditions as direct analogues to batch experiments under ambient (oxic) laboratory conditions. Thanks to the kind support from Jeffrey Perez (ESR 7) and other research scientists from the group, I obtained respective chemical (inductively coupled plasma optical emission spectroscopy, ICP-OES) and morphological (field emission scanning electron microscope with energy dispersive X-ray spectroscopy, FE-SEM/EDS) analyses to detect the chemical composition of the reacted solution and to characterise the precipitates that have formed during siderite dissolution in both oxic and anoxic conditions by electron microscopy.

Since the particle size of the precipitates produced during the siderite dissolution reaction, particularly in the oxic environment, range from only a few tens up to a few hundred nanometres, high-resolution techniques are required for proper phase characterisation and investigation of the interphase between the etched siderite grain surfaces and the secondary phases (how are the precipitates attached to the siderite?). Therefore, thin micro-scale foils of selected samples were prepared via the focused ion beam method in the scanning electron microscope (FIB-SEM) for high resolution analysis by transmission electron microscopy (TEM), both at the GFZ.

The main purpose of the second part of my secondment at the GFZ was using the TEM for solid analysis. Therefore, further samples were prepared and investigated together with previously prepared foils in the TEM for microstructural (selected area electron diffraction, SAED; fast Fourier transform, FFT) and chemical (EDS) characterisation and high-resolution imaging. The data obtained were partially processed during my visit, however, most of the data yet need to be processed, which requires experience and training on the analytical software.

The results obtained during my secondment are very satisfactory, as they suggest that siderite dissolution leads to effective Pb and Cu sequestration from acidic solutions under oxic as well as anoxic conditions. This is demonstrated by a significant decrease in Pb and Cu concentrations relative to their initial concentrations in the starting solution; particularly Pb was

removed almost entirely in both systems. For both metals, however, the controlling mechanisms for their removal become distinctly more dissimilar if no dissolved oxygen is available in the aqueous system, which can be explained by the different degrees of redox sensitivity of these two metals – eventually confirming the strong reducing potential of siderite.



Figure 1 (above) Batches of the long-term experiment of siderite dissolution in the presence of Pb (left) and Cu (centre) under exception of oxygen on a rotary shaker in the geochemistry laboratory of Section 4.4, GFZ.

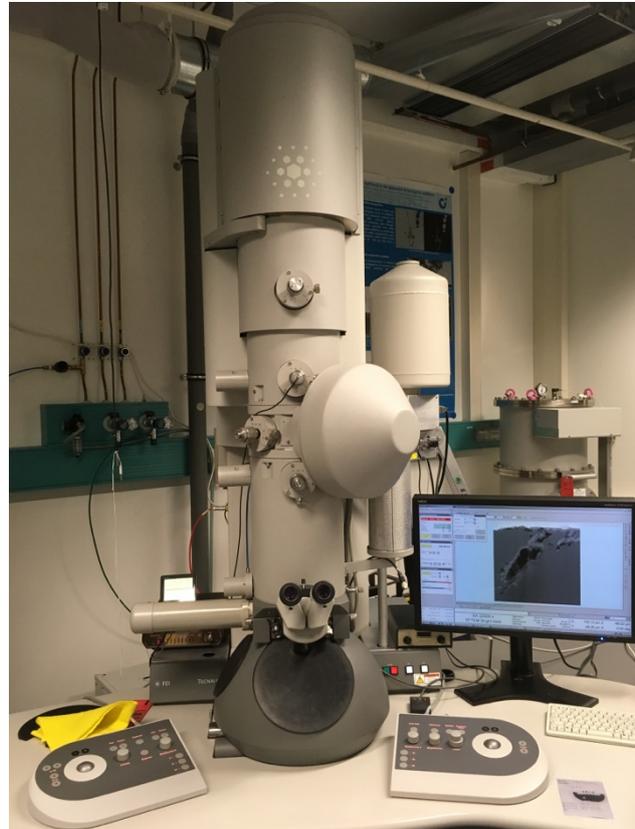


Figure 2 (to the right) The FEI Tecnai G2 F20 X-Twin transmission electron microscope (TEM) in the laboratory of Section 4.4, GFZ Potsdam. Specimen were prepared in the adjacent electron microscopy laboratory with a FEI FIB200TEM Focused Ion Beam device.

Besides the tightly scheduled laboratory and analytical work, I also had the opportunity to join several seminars hosted by the Section 4.4 as well as to present my work in the section seminar. I furthermore received a short training on geochemical modelling of my experiments using PHREEQC. In order to better understand the role of Cu in the redox processes, it has been suggested and offered to use X-ray photoelectron spectroscopy (XPS) at the GFZ in the near future to determine the oxidation state of Cu on the etched siderite surface and secondary iron hydroxides formed in the oxic environment.

Overall, this secondment at the GFZ has been a great opportunity for me in respect of great progress in my work and for networking. I am very grateful for the kind support I received and am very much looking forward to future collaborations.