

## Scientific report

### **of short visit grant for the workshop: Magnetic Methods in Biogeochemistry – from field to microscopy and Mössbauer spectroscopy**

The workshop held in Copenhagen was organized by Christian Bender Koch from University of Copenhagen, Denmark; Mihály Pósfai from University of Pannonia, Hungary; Rafal Dunin-Borkowski from Center for Electron Nanoscopy (CEN), Denmark; Barbara A. Maher from Lancaster University, England and Ian Snowball from Lund University, Sweden. The workshop was organized within an European research program entitled 'The Functionality of Iron Minerals in Environmental Processes'. The main themes of the workshop were Mössbauer spectroscopy and electron microscopy techniques. The participants got acquainted with various techniques utilizing magnetic phenomena at different size and time scale to the studies of biogeochemical processes of iron. Classes and practical exercises were held in the laboratories at Lund (Quaternary Geology), Lyngby (Center of Electron Nanoscopy), and Copenhagen (Department of Basic Sciences and Environment). 12 participants with various backgrounds took part in the workshop. Most of the participants were PhD students working in iron-oxides related fields, such as synthesizing magnetite nanocrystals through bacterial mediated synthesis methods, synthesizing iron-sulfides, studying green rust, studying biogenic magnetite and its usability for bioremediation, biomineralization of lepidocrocite and goethite by nitrate-reducing Fe(II)-oxidizing bacteria, iron oxidation under neutrophilic and oxygen limited conditions in the Rhizosphere of wetlands. Most of the students had their own samples for the measurements, thus most of them had their results by the end of the workshop.

My research area is synthesizing magnetite ( $\text{Fe}_3\text{O}_4$ ) nanocrystals with controlled size and morphology. Among various iron oxides magnetite has special importance both because of its widespread occurrence in soils and sediments and its nanotechnological applications.

My purpose of the visit was first of all to improve my knowledge about electron microscopy techniques. I use this technique for studying the size, morphology, structure and composition of my synthesized magnetite nanocrystals. On the other hand I take scanning electron microscopy course at University of Pannonia and I consider it important to be up to date about the newest available electron microscopy techniques with which I could become acquainted in the electron microscopy laboratories at CEN. Not only the conventional scanning (SEM) and transmission electron microscopes (TEM) were presented, but focused ion beam microscopes,

a high-resolution dual-beam SEM/FIB and electron backscattered diffraction for determining crystallographic orientation, new generation environmental field-emission TEM and an analytical TEM for holography and tomography.

The other part of my work is about nanocrystalline maghemite ( $\gamma\text{-Fe}_2\text{O}_3$ ). Concerning maghemite and magnetite structures, the two minerals are similar and thus difficult to distinguish using conventional transmission electron microscopy techniques. I have already used high-resolution transmission electron microscopy (HRTEM), selected-area electron diffraction (SAED) and electron crystallography for the structural characterization of synthetic maghemite nanocrystals. These methods are for studying individual nanocrystals. I was interested in techniques which are capable to distinguish these two mineral phases by measuring the whole sample. Mössbauer spectroscopy is one of these methods, thus my further purpose was to learn more about it and measure some of my magnetite and maghemite samples.

Two of my samples were measured by Mössbauer spectroscopy at room temperature, a magnetite and a maghemite sample. During the practical exercises we learnt how to calibrate, fit and get results from the Mössbauer spectra. In my case the room temperature Mössbauer spectra of both magnetite and maghemite could be fitted. So the size dependent magnetic behaviour did not arise in the samples, which mean the crystals in the sample are large enough to be magnetic one domain. On the magnetite spectra two sextets could be seen separating from each other and a doublet which refers to goethite impurity. The first sextet is due to the  $(\text{FeIII})_{\text{tet, oct}}$ , the second sextet is due to the  $\text{Fe(II)}_{\text{tet}}$ . We determined from the areas of the two sextets that the magnetite sample is not a stoichiometric magnetite. From the room temperature Mössbauer spectra it was possible to identify the maghemite sample. Maghemite spectra showed one sextet due to the only Fe(III) content.

Further studies of both magnetite and maghemite with various size and morphology would be interesting using Mössbauer spectroscopy. For example determining the stoichiometry in the case of a solid solution between magnetite and maghemite, the size dependence of magnetic behaviour and the effect of a polycrystalline magnetite sample onto the Mössbauer spectra.

Possibilities for collaboration with Christian Bender Koch (for Mössbauer measurements) and Rafal Dunin Borkowski (for TEM measurements) have been offered.