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Application of environmental cell transmission electron microscopy (EC-TEM) and electron energy loss spectroscopy (EELS) to mineral reactions and redox chemistry

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Electron energy loss spectroscopy (EELS), energy filtered transmission electron microscopy (EFTEM), and high resolution transmission electron microscopy (HRTEM) were employed to investigate mineral transformation associated with microbial Fe(III) reduction in magnetite. *Shewanella putrefaciens* strain CN32, a dissimilatory metal-reducing bacterium was incubated with magnetite as a sole electron acceptor and with lactate as an electron donor for 14 days under anaerobic conditions in a bicarbonate buffer. The high resolution TEM revealed that Fe-reduction proceeded via close association between bacterial cells and nanometer sized magnetite crystals, where cells were coated with magnetite crystals, and that siderite were formed apparently in the space previously occupied by a bacterial cell. The elemental map of Fe, O, and C and RGB composite map were created by EFTEM technique. The interface between magnetite and siderite were also investigated using EELS technique to understand the Fe oxidation state in each mineral. The Fe oxidation state was determined based on the integral ratio of L_3 to L_2. The integral ratios of L_3 to L_2 of magnetite(6.29) and siderite(2.71) corresponded to 71% of Fe(III) in magnetite, and 24% of Fe(III) in siderite based on van Aken et al. study (1998). Fe-L_{23} edges of Annite(11% of Fe(III)) and Muscovite(41% of Fe(III)) standards were newly obtained and the integral ratio of L_3 and L_2 of each standard was plotted as a function of Fe-oxidation state(van Aken et al. (1998)). Chemical shift (about 1.9 eV) of Fe-L_3 edges of magnetite and siderite was detected indicating the major difference in the oxidation state of Fe between the two minerals. Furthermore, EELS spectrum images of magnetite and siderite were extracted from the electron energy loss ranging from 675 to 755 eV, showing the oxidation state of magnetite-siderite interface. Application of EC-TEM to study clay mineral reactions were also described in this presentation.