Goldschmidt 2025 Abstract
https://doi.org/10.7185/gold2025.30222

Nano-scale Investigation of Carbon, Iron, and Sulfur Speciation at Sediment-Water Interfaces from Prairie Pothole Region Ponds with Synchrotron Spectromicroscopy

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The Prairie Pothole Region significantly contributes to greenhouse gas fluxes, namely methane and carbon dioxide, from high biogeochemical activity at the sediment-water interface in millions of small freshwater lakes and wetlands. Oxidation of iron sulfide minerals in sediment by dissolved oxygen generates reactive oxygen species. The reactive oxygen species interact with natural organic matter to produce low molecular weight organic acids and alcohols that promote microbial processes which generate greenhouse gases. In this study, the speciation of carbon, iron, and sulfur in sediments from the sediment-water interface were characterized as an initial condition for future controlled experiments.

Sediment from two ponds within the Prairie Pothole Region were collected from the upper 5 cm of the sediment-water interface. Sediment slurries were centrifuged to separate porewater and frozen in glass jars with an inert headspace. Scanning transmission X-ray microscopy (STXM) and ptychography were used to compare carbon, iron, and sulfur chemical speciation and spatial relationships between the two pond sediments. The STXM and ptychography data were collected at the Advanced Light Source, Lawrence Berkeley National Laboratory on beamlines 5.3.2.2 (C 1s and Fe 2p) and 7.0.1.2 (Fe 2p and S 1s). Sediment suspensions were dropcast onto silicon-nitride membranes and TEM grids for analysis.

Carbon spectra for both ponds were similar and showed evidence for proteinaceous and carbonate-rich material, matching peak positions for bovine serum albumin and calcium carbonate reference standards. Particles of ferrous, ferric, and mixed-valent iron were present in both ponds. Ferrous iron was not linked to sulfide minerals, despite identification from bulk S XANES analysis, but rather dominated by phyllosilicate minerals, as suggested by the bulk Fe K-edge XANES and by the presence of potassium L-edge features (297.3 eV) in the carbon spectra.[1] Future research will utilize an *in-situ* liquid flow STXM cell to observe oxidation of iron minerals by dissolved oxygen in real-time and the subsequent reaction with model organic matter compounds.

[1] Pacheco, M.; Stenberg, C.; Arnold, W.; Toner, BM. Dark Fenton reactive oxygen species generation at the sediment water interface of prairie pothole wetlands. Poster presented at American Chemical Society Conference: March 17-21, 2024; New Orleans, Louisiana.