Microscale Reconstruction of Biogeochemical Substrates Using Combined X-Ray Tomography and Scanning Electron Microscopy

Research Team: Micah Miller, Erin Miller, Jun Liu, James McKinley

Pacific Northwest NATIONAL LABORATORY

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Project 1.7; May 2012 – May 2015

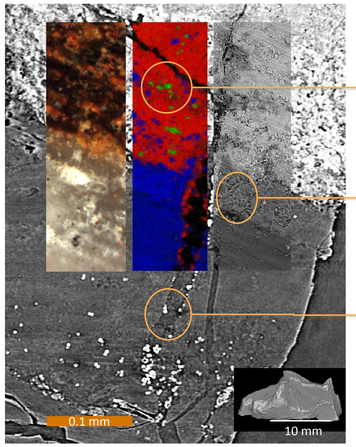
Purpose

- Gain insights for hydrology, geochemistry, and microbial ecology that rely on knowledge of phase and pore space distributions for research on contaminant mobility and natural transport and phase transformations
- Develop technology for 3D reconstruction of the physical and chemical nature of natural or synthetic sample at sub-micrometer scale
- Evaluate ability to combine data across collection methods

Key Successes

- Acquired and registered data from Advanced Photon Source (APS), EMSL computed tomography (CT), and scanning electron microscopy (SEM) of natural samples
- Multiscale, multimodal fusion of 2D and 3D imagery
- Enhanced contrast X-ray imaging provides sub-resolution texture

Research Accomplishments



Composite image: Slice from an X-ray tomogram of hot-spring mineral assemblage. Overlays (L to R): Optical microscopy, SEM elemental map (Red = Fe, Green = S, Blue = Si), SEM backscatter. *Sample courtesy of J Zachara.*

Tomographic depiction of a microbially mediated hematite/goethite composite (bright) on a silicate substrate (dark). This dataset includes 3D X-ray tomography acquired at APS and in-house 2D SEM microprobe and optical microscopy imagery.

SEM elemental analysis detects sulfur deposits indicative of sulfate reduction

Higher resolution SEM reveals physical features in backscattered electron image

Geometry of flow channels may be calculated from 3D structure

Computational manipulation of disparate data can be used to propagate higher resolution SEM analysis through a tomographic volume to provide structural and chemical insights for the entire sample without the expense and information loss incurred by step-wise sample dissection and imaging.

Process Steps

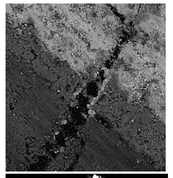
1. Sample is embedded in epoxy for stability
2. Low-resolution CT imagery at EMSL
3. Cut and polish sample into 1-mm-wide columns
4. High-resolution CT imagery at APS
5. SEM microprobe at EMSL
6. Additional analyses at PNNL (µXRF, LM)
7. Imagery registered
8. High-resolution values propagated

The samples represented here originated from two DOE projects at PNNL:

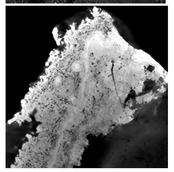
- DOE's Genomic Science Program; Foundational Scientific Science Focus Area (JK Fredrickson)
- DOE's Subsurface Biogeochemical Research; PNNL's Scientific Focus Area (JM Zachara)

This work represents an approach that could be useful in addressing their research objectives.

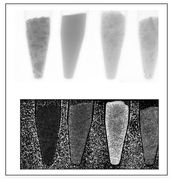
Imaging Modes



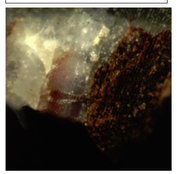
Electron Microscopy. Provides high-resolution 2D imagery of exposed surfaces as well as elemental composition. This instrumentation is available at EMSL.



X-Ray Computed Tomography. Allows for non-destructive 3D imaging resulting in volumetric data containing structural information. Low-resolution imagery is available at EMSL, while APS provides higher.



X-Ray Scattering for Texture Information – Preliminary. Top: Absorption image of size-segregated soil. From left: >150 µm; 25-150 µm; 2-25 µm; <2 µm. Bottom: Scatter coefficient normalized by absorption. Signal appears sensitive to texture size variations.



Light Microscopy. Inexpensive 2D surface inspection tool available at PNNL.

CT image (acquired at APS) of Integrated Field Research Challenge sample taken at a depth of 69 ft. Sample revealed distribution of microbial metabolic byproducts and their relation to physical properties such as pore distribution.

Extension of SEM to 3D

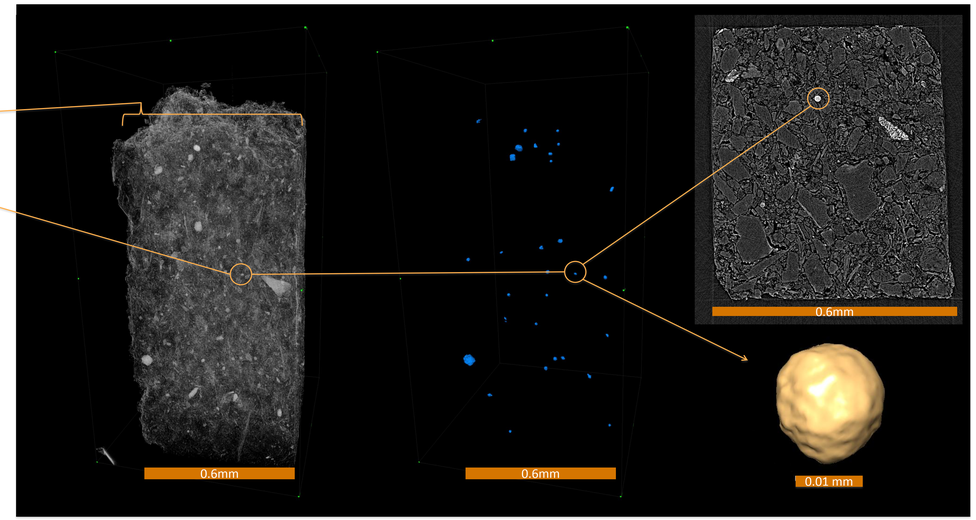
Up to ~1 mm^3 can be examined without damaging the sample.

Spherical objects indicate in situ sulfate reduction.

Volume search for specific objects

A volume can be searched for discrete objects that match a given profile producing a census and map of their abundance. X-ray CT exposes heterogeneity, or lack thereof, within a sample that can help identify areas to analyze with SEM.

This project contributed to the creation of unique, rapid software for reconstruction of phase-contrast data ('AmeriCT'). This GPU-accelerated software can also process standard tomography data from APS many times faster than the APS-provided programs, which do not include any code for phase-contrast data.



CT volume displaying relative X-ray absorption with wireframe denoting whole sample volume

Segmented and filtered volume showing spherical particles

Above: 2D CT slice with target highlighted. Below: Surface rendering of target particle.

Next Steps

- Continue working at APS for higher quality CT on new sample types
- Evaluate 2D-3D propagation algorithms and performance
- Incorporate lower resolution EMSL CT scans and gratings-based contrast enhancement

PNNL-SA-96825