ABSTRACT: Advanced mechanistic understanding of carbon (C) biogeochemical cycling is essential for the development of Earth System Models. Critical for development of that mechanistic understanding is the determination of the physical characteristics and chemical speciation of C in subsurface and root zone environments. Hard x-ray microtomographic imaging of soil pore structure in unaltered soil aggregates is a powerful approach to understanding the physical structure of soil and the physical controls of C partitioning within soils (Kemner, et al., 1998). In addition, x-ray spectroscopy- and microscopy-based investigations of the constituents of subsurface and root zone materials can provide critical insights into the chemical nature of C in these materials. Although soft x-ray scanning transmission x-ray microscopy (STXM) can provide spatially resolved chemical information about C in samples, the thickness and hydration state of environmental samples often preclude the utility of soft x-rays. This limitation can be overcome with the non-resonant inelastic x-ray scattering (NIXS) technique, which enables measurement of C 1s x-ray absorption spectra using high energy x-rays. Applying NIXS at an insertion device beam line at the Advanced Photon Source (APS) enables measurement of these types of samples without the need for containment in vacuum. We are integrating x-ray microtomographic, NIXS, and STXM approaches to develop an advanced mechanistic understanding of C cycling.

Our initial NIXS investigations of C speciation in a variety of standards, microbial biomass, and soil constituents from peats, humic materials, and Alaskan permafrost (Mishra, et al., 2013) showed that NIXS measurements can distinguish important C moieties like aromatic-C, amide-C, phenol-C, carbonyl-C, and carboxyl-C. Additionally, we have shown that NIXS can enable quantitative determination of the relative percentage composition of these moieties. Comparisons of results determined with NIXS to those determined with nuclear magnetic resonance (NMR) measurements indicate agreement and complementarity of the two approaches. Integration of soft x-ray STXM approaches (presently being developed at the APS) into these investigations will provide the spatial resolution information that will be complementary to NIXS measurements of bulk samples.

We have also investigated the pore space and physical structure of three size classes of soil aggregates (250-425, 425-841, and 841-100 μm) collected from a grassland field to determine correlations between aggregate size, internal pore structure, and microbial community composition within the aggregates (Bailey, et al., 2013). X-ray transmission microtomographic measurements showed that a greater proportion of the pore space in the small- and medium-sized macroaggregates is present as relatively smaller pores, resulting in greater overall porosity and pore-mineral interface area.