

Mineral, metal and microbial mapping of Antarctic soil habitats: Validating soft X-ray

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Synchrotron-based soft X-ray scanning transmission x-ray microscopy (STXM) is a spectroscopic and imaging technique that produces element-specific component maps for important biological elements (i.e. C, N, O, P, S), the alkaline metals (i.e. Na, Mg, K, Ca), all of the 1st row transition metals (e.g., Fe, Mn, Ti), Al and Si, at high spatial resolution (<25 nm), with minimal sample preparation and reduced radiation damage (i.e. spectral distortion as a result of bond breaking) compared to electron microscopies. Moreover, the chemical speciation of an element (e.g., oxidation state) can be determined by collecting a sequence of images at various photon energies (i.e. newline stack), producing an X-ray absorption near-edge structure (XANES) spectrum for each pixel. Quantification of the chemical species in each pixel is possible since the mass absorption coefficient (μE) is measured directly in transmission and the resulting transmittance images can readily be converted to absorbance/optical density images. By collecting element specific stacks (e.g., C and Fe) and/or image resonance difference maps for the same area, the relationship between different elements and/or chemical species can be determined through co-localization analysis. Hence, soft X-ray STXM is extremely powerful, providing spatial chemical information at the nanometer-scale for the elements most relevant for soil and biogeochemical systems. However, it is not clear how samples should be prepared and processed to allow the results from these nano-scale imaging and spectroscopic tools to be scaled to field scale biogeochemical results. We used a simple sample preparation technique that allows one to assess detailed mineral, metal and microbe spectroscopic information at the nano and micro scale in a soil sample. We then evaluated three common approaches to process nano and micron scale information by STXM and the correspondence of these approaches to millimeter scale soil measurements. Finally, we assessed the reproducibility and spatial autocorrelation of nano and micron scale organic, Fe(II) and Fe(III), Mn, Ca, Al and Si densities in a soil sample. We demonstrate that linear combination fitting of the stacks using reference spectra slightly underestimates mineral densities compared to image resonance difference mapping but that STXM results are highly reproducible between soil and processing replicates. Further, STXM results scale to the mm scale in complex systems with an approximate geospatial range of 3 μm in these soil samples.