Direct detection of solid-phase phosphorus speciation in agricultural soils using paired μ-XRF mapping and μ-XANES

KATHRYN D. SZERLAG[1], PAUL NORTHRUP[2], RYAN TAPPERO[3], MATTHEW G. SIEBECKER[4], DEB P. JAISI[1], AND DONALD L. SPARKS[1]

[1] Dept. of Plant and Soil Science, Univ. of Delaware, Newark, DE 19716-7310, USA. (*correspondence: kszerlag@udel.edu)

[2] Dept. of Geosciences, Stony Brook University, Stony Brook, NY 11794, USA.

[3]Brookhaven National Laboratory, Bldg 743, Upton, NY 11973, USA.

[4]Dept. of Plant and Soil Science, Texas Tech University, Lubbock, TX 79409, USA.

Phosphorus (P) in soil is a critical plant nutrient but detrimental to aquatic ecosystems. Determining the solid phase P speciation in soil is crucial to understand its biogeochemical behaviour, yet it remains poorly understood. This uncertainty in P speciation is rooted in the lack of direct methods to accurately determine P speciation in soils. Improved understanding of the solid phase soil P speciation would eventually allow for improved fertilization, updated best management practices, and informed remediation strategies.

This work leverages recent developments in synchrotron-based analyses, utilizing high resolution, micro X-ray fluorescence (μ-XRF) mapping to characterize P speciation in two agricultural soils located on the Delmarva peninsula (Delaware, USA) with high legacy P (>800 mg/kg). Pairing two beamlines at the National Synchrotron Light Source II (NSLS-II), beamlines 8-BM (TES, 2-6 keV) and 4-BM (XFM, 5-20 keV), μ-XRF maps of the same soil thin section and powder mount locations revealed the spatial distribution and co-location of P with Al, Si, S (8-BM), Fe, Ca, Mn, and As (4-BM). Determining this co-location aided in the identification of P species and revealed the spatial heterogeneity in speciation at the micron scale. Following μ-XRF mapping, 8-BM was used to probe P hotspots with P K-edge micro X-ray absorption near edge structure (μ-XANES) to determine P speciation at the micron scale. With the μ-XANES spectra paired with the elemental co-location, we were able to directly and precisely determine P species. This work demonstrates that μ-XRF mapping at two energies paired with μ-XANES is an effective tool for the direct solid phase speciation of legacy soil P.