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Investigating the Chemical Composition of Soil Aggregates to Inform Changes in the Sulfur Cycle

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In California vineyards, high amounts of elemental sulfur (S^0) are applied annually as a fungicide to control powdery mildew. Elemental S oxidizes immediately to sulfate. Research to date has shown that sulfate reduction occurs within vineyard soils, likely within aggregates, however the forms of reduced S and their association with other metals, including iron (Fe), remain unknown. Here we use a combination of photopaper imaging and micro-X-ray fluorescence (μ-XRF) mapping and micro-X-ray absorbance near-edge structure (μ-XANES) spectroscopy to determine the speciation and co-association of S and Fe in soil aggregates within intact soil blocks (5 cm diameter x 30 cm deep, cut in half lengthwise) from both Napa Valley vineyards receiving S^0 inputs and non-agricultural areas receiving background S during the wet/dormant and dry/growing seasons of 2023-2024. Locations of anoxic regions within the soil blocks were initially identified with silver photopaper incubated on the exposed cutting surface of the halved cores inside an anoxic glovebag. The photopaper darkens in the presence of reduced S, providing a “picture” of the reduced S locations that exist on the soil block face. These regions were subsequently investigated with μ-XRF mapping and K-edge μ-XANES to determine the specific chemical form of S and Fe in the reduced regions along with the spatial relationship between S, Fe, and other metal constituents. Determining the association of reduced S with Fe and other soil constituents is an important step in gaining a complete picture of the fates of agricultural S applications, such as whether it is sequestered as iron sulfides (FeS, FeS_2), mercuric sulfide (HgS), or associated with MeHg, a potent neurotoxin. These data will be useful to motivate future field studies across larger geographic regions and provide important information to farmers and regulators in California and beyond.