Exploring Marine Iron Colloids with Scanning Transmission X-ray Microscopy

KYOUNGLIM KANG¹, JESSICA N. FITZSIMMONS², LARAMIE JENSEN³, COLLEEN L. HOFFMAN⁴, LEE PENN¹ AND DR. BRANDY M TONER. PH.D. ¹

Colloidal Fe (< 200 nm) is increasingly recognized as a key player in elemental transport and carbon stability within marine environments. Marine colloidal Fe constitutes up to 90% of the dissolved Fe pool, with its size and composition varying across coastal waters, open-ocean regions, and hydrothermal plumes. Iron colloids can exist in inorganic forms- such as Fe(III) (oxyhrdr)oxides and Fe(II)-sulfides. However, the majority are thought to be complexed by organic carbon compounds including humic acids, polysaccharides, and siderophores. The chemical form of Fe and carbon within these colloidal composites influences their bioavailability and aggregation behavior.

However, there is no "one size fits all" method for investigating the speciation of marine Fe colloids, as different methods are variably hindered by trace colloidal Fe concentrations in seawater, methodological artifacts from size separation and analytical constraints. Here, we utilize Scanning Transmission X-ray Microscopy (STXM) to image model colloidal particles (latex bead) of different sizes (28, 80, and 220 nm) and to distinguish organic and inorganic Fe species in marine colloids from Galveston Bay (<200 nm fraction). Three ultrafiltration techniques (20 nm Anopore, 50 nm Nucleopore, and ~3 nm cross flow filtration) and two sample preparation methods (dry and wet loading) are compared to evaluate their effectiveness for imaging colloidal Fe.

With the 30 nm spatial resolution determined by the outer width of the focusing optic, focused STXM images were obtained for dried 220 and 80 nm model latex beads, while the size and shape of the 28 nm colloids could not be resolved. Galveston Bay colloid samples, filtered through Nucleopore and prepared as a drop-mount followed by room-temperature drying, revealed Fe(III) species surrounded by aliphatic organic carbon compounds, with a mean diameter of ~120 nm. Our results show that STXM can chemically image marine Fe colloids, but has limitations related to size (< 80 nm). Future studies should explore higher-resolution techniques like ptychography that can resolve the size and shape of colloids in the range of 10-20 nm at the Fe absorption edge. In-situ measurements can be considered to investigate the real-time chemical reactivity and physical fate of Fe colloids under flow conditions.

¹University of Minnesota

²Texas A&M University

³University of Washington

⁴Leidos, National Energy Technology Laboratory