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A ptychographic spectromicroscopy workflow for marine nanoparticles

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Synchrotron scanning transmission X-ray microscopy (STXM) is a well-developed analytical technique that has become widely available at facilities globally [1]. Applications of STXM to Earth and environmental science research questions have yielded discoveries of submicron processes affecting the transport and reactivity of atmospheric aerosols, marine trace metals, soil minerals and organic matter, and more. STXM can be strategically placed within a portfolio of microprobe and bulk synchrotron measurements to scale-up observations and add the context of co-occurring elements [2]. However, it is often observed that features of interest within natural samples are below the practical spatial resolution of STXM (ca. 50 nm). While electron microscopy is frequently included in studies using STXM for pre-analysis characterization and for adding finer spatial resolution of imaging, the spectromicroscopy features of synchrotron X-ray microscopy are more sensitive to the speciation of elements such as carbon in natural organic matter. Our goal was to increase spatial resolution of imaging while retaining the many advantages of synchrotron X-ray microscopy for analysis of natural particles by integrating synchrotron X-ray ptychographic spectromicroscopy with STXM for the study of marine nanoparticles (<100 nm). Samples from two hydrothermal vent sites, Rainbow (Mid Atlantic Ridge) and Endeavour (Juan de Fuca Ridge) were investigated. The STXM and ptychography data were collected at the Advanced Light Source, Lawrence Berkeley National Laboratory on beamlines 5.3.2.2 and 7.0.1.2. Sample preparation, data collection, and data analysis will be discussed and placed into context with complementary observations. We found that iron-bearing nanoparticles are aggregated with organic matter at both vent sites within the particulate size fraction. We will discuss best practices for ptychographic spectromicroscopy for marine nanoparticles and generalize recommendations for natural samples containing metals, minerals, and organic matter. This research used resources of the Advanced Light Source, which is a U.S. Department of Energy (DOE) Office of Science User Facility under contract number DE-AC02-05CH11231.

[1] Feggeler et al. 2023 https://doi.org/10.1016/j.elspec.2023.147381
[2] Toner et al. 2016 https://doi.org/10.1021/acs.accounts.5b00282