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Towards multi-element characterization of marine metal nanoparticles: First results from the suboxic zone of the Black Sea

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Transition metals serve as electron acceptors in microbial respiration and as electron donors in chemosynthesis to drive ecosystems. Furthermore, they act as catalytic centers for metabolic processes in all areas of life. Deep-sea redox interfaces continue to be a major supply of metals to the upper marine layers in the current oxic state of aquatic systems. Nonetheless, there is an urgent need for a fundamental knowledge of how deep-sea originating metals are delivered and transported in their nanoparticle states to various ecosystems within oceans. Methods tailored towards marine samples are scarce, but due to the increasing interest in engineered nanoparticles entering aquatic systems, as well as the emerging relevance of NPs in therapeutic and diagnostic applications in medicine, several analytical approaches for nanoparticle detection are attaining maturity. One method is single-particle inductively coupled mass spectroscopy (sp-ICP-MS), which can be extended to multi-element detection by including time-of-flight spectroscopy (TOF-MS). In this study, we test this approach for marine samples and show that Fe and Mn co-exist in nanoparticle phases that occur naturally in the Black Sea's suboxic zone. We extensively characterized the biogeochemical redox transition in the open waters of the western Black Sea during the R/V Bilim expedition in 2021, and undertook sampling for size-fractionated particle and colloidal/nanoparticle pools. The samples were analyzed using a TOF-ICP-MS (IcpTOF, TofWerk). The nanoparticles observed ranged in size from 10 nm to 200 nm and were concentrated in the water column of the Black Sea at sigma-theta values of 16.0 to 16.4, consistent with rapid redox cycling at the lower end of the suboxic zone and at the onset of the sulfide zone. The discovered nanoparticle accumulation density layers do not necessarily match with the turbidity layers identified by the oceanographic CTD-rosette system, indicating the presence of nanoparticle-rich layers across marine redox gradients that is concealed and more prevalent. When combined with other novel detection and separation approaches, the approach presented here has the potential to detect and quantify various tracers of redox-driven abiotic processes and biotic, sub-micromolar level transformations across redox gradients and extreme environments such as deep-sea vents.