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Tracing Fe reaction pathways in tephra-rich deep subseafloor sediments from the Nankai Trough offshore Japan by using sequential extractions and stable Fe isotopes

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The deep subseafloor biosphere represents one of the Earth's largest, but also least understood ecosystems with diverse species and mostly uncharacterized microbial communities. International Ocean Discovery Program (IODP) Expedition 370 (Temperature Limit of the Deep Biosphere off Muroto) established Site C0023 down to 1180 mbsf in the Nankai Trough off Japan to explore the upper temperature limit of microbial life in the deep sedimentary biosphere [1]. Site C0023 is characterized by a complex lithostratigraphic and depositional history with strongly changing sedimentation rates. Volcanic ash layers are ubiquitous in all lithological units. However, the highest abundance of ash layers could be observed between 400 and 700 mbsf. Previous studies have shown that volcanic ashes represent hotspots for microbial life [2] and are commonly characterized by high Fe(III) and Mn(IV) contents [3]. Onboard measurements show a release of dissolved Fe in the depth interval associated with the highest abundance of ash layers [1]. Therefore, we hypothesized that the release is related to microbial Fe reduction fueled by the mineralogy of the volcanic ash. In order to identify the source and reaction pathway of the liberated Fe, we applied sequential extractions of differently reactive Fe oxide pools on mud rock and ash layer samples as well as stable iron isotope ($\delta^{56}\text{Fe}$) analyses on pore-water and solid-phase samples. Microbial Fe reduction leads to Fe isotope fractionation with an enrichment of light isotopes in the released Fe and a respective enrichment of heavy isotopes in the residual ferric substrate. Therefore, the $\delta^{56}\text{Fe}$ signals of different reactive Fe pools and the pore water are used to identify the pools actually involved in microbial respiration processes. Our results show that the total Fe content in mud rock of Site C0023 is relatively constant at ~4.2 wt%. Reactive Fe oxides represent 25% of the total Fe. The bulk Fe content in the ash layers varies between 1.4 and 6.8 wt%. Surprisingly, most ash samples contain less total Fe (3.35 wt% on average) compared to the surrounding mud rock. Similarly, the contents of the reactive Fe oxides are significantly lower.

This indicates that either (1) ash layers do not represent the energy substrate for microbial Fe reduction, or (2) reactive Fe in ash samples has already been used up by microbes. The bulk Fe content in recent volcanic material from an active volcano on the Japanese island arc is ~4.4 wt% [4]. The higher Fe content in fresh volcanic material compared to ash samples at Site C0023 might suggest that reactive Fe in ash layers is already reduced. Alternatively, the dissolved Fe release might be related to microbial reduction of structural Fe(III) in smectite promoting the smectite-to-illite transition, which has previously been proposed for Site C0023 [5].

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