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Micro-scale elemental composition of organo-mineral associations collected in situ from a Calcaric Gleysol

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Evaluating the temporal dynamics of the chemical composition and spatial distribution of organo-mineral associations is crucial for understanding their role in cycling and stabilizing soil organic matter (SOM).

Especially Fe oxides and hydroxides play an important role for the stabilization of SOM due to their high reactivity. However, it is not straight forward to study the formation of organo-mineral associations in situ at intact soil structures. In groundwater-affected redoximorphic soils, precipitation of Fe oxides occurs directly at the soil-water interface of coarse pores. Therefore, these soils are particularly suitable to observe the dynamics of Fe oxides regarding interactions with dissolved organic matter and redox-induced aging of initially short-range ordered Fe oxides. We therefore chose a dyked marshland site with a Calcaric Gleysol formed from marine sediments for collecting freshly precipitated and aged Fe oxides in situ. Sampling was carried out without physical and chemical disturbance using a special rod with attached glass slides or silicon wafers. The application of slides enabled us to use a wide range of microscopic and spectroscopic techniques to analyze the chemical and mineralogical composition of these precipitates.

Three rods were inserted for half a year $(R_{0.5})$, one year (R_1) and two and a half years $(R_{2.5})$. Wafers and glass slides within the Bg-Horizon (oxidizing conditions) were analyzed starting with field emission scanning electron microscopy (FE SEM) to select suitable spots for subsequent chemical mapping using nano-scale secondary ion mass spectrometry (NanoSIMS).

For $R_{0.5}43$ spots on two glass slides were measured, for R_1 39 spots on four silicon wafers and for $R_{2.5}$ 34 spots on four glass slides. For the NanoSIMS measurements seven masses were chosen, mainly: $^{16}O^-$, $^{12}C^-$, $^{12}C^{14}N^-$, $^{31}P^-$, $^{32}S^-$, $^{27}Al^{16}O^-$, $^{56}Fe^{16}O^-$.

One plane with 10 ms per pixel $(R_{0.5}+_{2.5})$ and 40 planes with 1 ms per pixel (R_1) were measured. For evaluating a triangle threshold was applied via ImageJ. Look@NanoSIMS was used to define regions of interest (ROIs). For statistical analysis ratios of different masses will be calculated and statistically evaluated.

First preliminary results based on selected spots of $R_{0.5}$ and $R_{2.5}$ show a patchy distribution of organic material on aggregates. The spots of SOM increase over time, which suggests that larger organo-mineral associations are formed. A tendency for the competition of adsorption sites between sulfate and organic anions can be assumed for $R_{0.5}$ and $R_{2.5}$, demonstrated by larger ratios of $^{32}S^-$ to $^{56}Fe^{16}O^-$ instead of $^{12}C^-$ or $^{12}C^{14}N^-$ to $^{56}Fe^{16}O^-$. The further evaluation of the data set will give us insight, how organo-mineral associations are altered over time, e.g. whether the association of organic matter with Fe oxides becomes more important than the association with phyllosilicates after a larger number of redox cycles. This would contribute to a long-term SOM stabilization.