Spatial distribution of Al and Fe in synthetic Al-substituted Goethite

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Goethite (α-FeOOH) is a common iron oxide in temperate soils. It is mainly formed during weathering of Fe-containing minerals. Depending on the pristine mineral or the chemistry of the surrounding solution, the formation can be influenced by coprecipitation of different metal ions. Due to its high geochemical abundance and its similar ionic radius, Al^{3+} is the most commonly observed substituent cation. In nature, Al can exchange up to one third of the Fe in goethite. Under these circumstances, no adjacent Al-atoms occur and the incorporation is described as homogenous. In contrast, other research proposed the theory, that even in low concentrations, the formation of isolated Al-cluster within the goethite is favoured. The present study aimed at visualizing the spatial Al distribution in synthetic goethite with low Al-concentrations at the nanoscale by using advanced spectroscopic methods in combination with state-of-the-art imaging techniques.

We synthesized Al-substituted goethites with Al concentrations of 0.1, 3 and 7%. First, the goethite-samples were mineralogically characterized using Fourier Transform Infrared (FTIR) Spectroscopy, X-ray diffraction (XRD) and Mössbauer spectroscopy. To visualise the spatial Al distribution, subsamples of each concentration were analysed with energy dispersive X-ray spectroscopy (EDX) and nanoscale secondary ion mass spectrometry (NanoSIMS). Especially the low detection limit and high spatial resolution of NanoSIMS allows to visualise the Al-distribution, even at trace metal concentrations.

The FTIR and XRD spectroscopy demonstrated the increase of defects in the crystal pattern with increasing Al-content, whereas the absence of pure Al-oxide related signals were used as an indicator for homogenous Al incorporation. Additionally, no Al-enriched clusters could be visualised at the nanoscale using NanoSIMS. We conclude, that the Al is homogenously incorporated in the goethite-structure within the investigated concentration range.