

EGU22-218, updated on 12 Aug 2022

<https://doi.org/10.5194/egusphere-egu22-218>

EGU General Assembly 2022

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Iron speciation throughout a karst pedosedimentary record in South of Italy

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A 3-m thick sediment was found in a limestone mine located in the Apulia region (south of Italy), at a depth of 25-30 m from the current ground level. Samples from 5 layers were investigated by X-ray Absorption Near Edge Spectroscopy (XANES) and Extended X-ray Absorption Fine Structure (EXAFS) at the iron (Fe) K-edge, paired with Raman spectroscopy (RS) and thermal analysis.

The pedosediment record under investigation represents a peculiar stratigraphic series showing complex features related to the Fe dynamic.

From a technical point of view, the challenges of phase identification in Fe oxides are many, but the application of multiple techniques provides sufficient evidence for the identification and discrimination of Fe phases. The combination of Fe XANES and EXAFS allowed to get information on the Fe speciation and its local structure. In detail, ferrihydrite is the most abundant Fe species, followed by goethite and minor amounts of hematite. Ferrihydrite content decreases with depth, where goethite and hematite occur. The presence of ferrihydrite, only detected by Linear Combination Fitting (LCF) on the first-derivative XANES spectra and on the EXAFS spectra, suggests that redox cycling of Fe occurred in these sediments. In addition, higher contents in Fe(III) complexed by organic matter is found in the top and deepest layer.

Generally speaking, RS and thermal analysis might present a promising tool to unravel some mineralogical components such as calcite, goethite and some Mn-oxides. Unlike for the identification of some Fe-bearing minerals like ferrihydrite, RS seems to be not well suited and this aspect require further investigations.

Definitely, this study confirms that each layer has preserved the distinct features that relate to its time of deposition. We underline how a multidisciplinary approach is strongly required to obtain reliable records when peculiar environments like karst pedosequences are investigated.