



## **Formation mechanism of calcified roots in terrestrial sediments: insights from a multitechnique and multiscale characterization strategy**

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Root remains encrusted by secondary carbonates, e.g. carbonated rhizoliths, are common in many soils and terrestrial sediments from various environmental settings. Rhizoliths usually exhibit a cylindrical shape and may have different sizes (from a few  $\mu\text{m}$  up to several cm). These objects have been known for ages and intensively used as proxies for paleoenvironmental reconstruction. It is generally assumed that such encrustation is controlled or induced by complex organic-mineral interactions at the plant tissue scale, even though this has never been investigated in detail.

The aim of this work was to better constrain the mechanisms of rhizolith formation, which remain unclear so far. Rhizoliths at different stages of encrustation and surrounding sediment were sampled at different depths from a loess-paleosol sequence (Nussloch, SW Germany). They were characterised using a multi-scale and multi-technique approach. The use of SEM and TEM to investigate rhizolith samples has offered a unique combination of chemical and structural information with submicrometer spatial resolution, while solid-state  $^{13}\text{C}$  NMR of decarbonated rhizoliths along with liquid and gas chromatography analyses of organic extracts have provided information at a molecular level.

SEM and TEM reveal that the precipitation of secondary carbonates does not only occur around, but also within the plant root cells and evidence the close relationship existing between organic and inorganic phases within these complex systems. The fine-scale preservation of root cellular ultrastructure with remarkable integrity observed for samples at all stages of encrustation has likely been promoted by this intra-cellular carbonate precipitation. In parallel, gas and liquid chromatography analyses showed that microbial biomarkers were predominant in the former roots, in contrast with the surrounding sediment, dominated by plant biomarkers. This suggests that the molecular signatures of the organic matter differ between calcified roots and the surrounding sediment, as also confirmed by  $^{13}\text{C}$  NMR analyses. Fresh and calcified roots present similar  $^{13}\text{C}$  NMR spectra, showing the good preservation of the root-derived organic matter in carbonated rhizoliths. Altogether, the results allow us to propose a general scenario for the mechanism of plant root encrustation by secondary carbonates in terrestrial sediments.