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Analysis of lignin oxidation products in a stalagmite from the Herbstlabyrinth-Adventshöhle in Germany and comparison with δ^{13}C and other vegetation proxies

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Lignin oxidation products (LOPs) are widely used as vegetation proxies in sediment cores and natural waters and can be found in speleothems, too. They not only indicate the quantity of vegetation, but also allow to differentiate between angiosperm and gymnosperm plant sources and woody and herbaceous plant material. Lignin is one of the main constituents of wood and woody plants. It is a biopolymer that mainly consists of three monomers, p-coumaryl alcohol, coniferyl alcohol and sinapyl alcohol. The proportion of these three monomers varies with the type of vegetation. To analyse the composition of lignin particles in speleothems, it is necessary to extract the speleothem samples, to digest the lignin polymer in order to split it into its monomeric oxidation products, and to quantify these LOPs.[1]

In the method we are presenting here, stalagmite samples are dissolved in hydrochloric acid and the acidic solution is extracted by solid phase extraction. The resulting organic fraction is submitted to an alkaline cupric oxide oxidation using a microwave digestion system.[2] The LOPs are enriched by solid phase extraction and analysed by ultra-high performance liquid chromatography coupled to electrospray high resolution mass spectrometry (UHPLC-ESI-HRMS). We used this method to analyse the 11000 years old stalagmite NG01 from the Herbstlabyrinth-Adventshöhle cave in Germany.[3] The stalagmite slab was cut in oblong pieces following the growth lines so that the sample weight was ca. 3-5 g and the sample length along the growth axis was ca. 1 cm, corresponding to a time resolution of ca. 200 years per sample.

We present the first LOP record of a stalagmite as well as the method validation. The results of the LOP analysis were compared with records of δ^{13}C, δ^{18}O and trace elements of the same stalagmite analysed by Mischel et al. (2016).[3]

References

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