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Organic trace analysis of lignin phenols in speleothems using UHPLC-ESI-HRMS and their use as vegetation proxy

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To understand the climate of the past, it is necessary to get information not only about temperature and precipitation, but also about the vegetation. In contrast to the vegetation proxies used in speleothems so far, like $\delta^{13}C$, the analysis of lignin can provide information not only about the quantity, but also about the type of the regional vegetation. Lignin is widely used as vegetation proxy in sediment samples and natural waters,[1] but there are no methods to analyse lignin in speleothems yet.

Lignin is one of the main constituents of wood and woody plants. It is a biopolymer that consists mainly of three monomers, p-coumaryl alcohol, coniferyl alcohol and sinapyl alcohol. The proportion of these three monomers varies with the type of vegetation, for example gymnosperms, angiosperms or herbaceous plants.[2] 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 monomers, also called lignin phenols, and then to enrich and quantify these lignin phenols.

In the method we are presenting here, stalagmite samples are acid digested 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.[3] The oxidation products are enriched by solid phase extraction and analysed by ultra high performance liquid chromatography coupled to electrospray high resolution mass spectrometry. We present the limits of detection and quantification, reproducibility and recovery rates as well as first proof-of-principle results from stalagmite samples from the Herbstlabyrinth-Adventshöhle in Germany.

References:

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