

EGU2020-2413

<https://doi.org/10.5194/egusphere-egu2020-2413>

EGU General Assembly 2020

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Trace analysis of levoglucosan and lignin-phenols in speleothems by HILIC-UHPLC-ESI-HRMS: A new method

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Secondary mineral deposits in caves like stalagmites, stalactites, or flowstones are valuable paleoclimate archives. Advantages of organic trace analysis in such deposits are stable conditions in a cave, protecting compounds from external influences, as well as the possibility to precisely date samples up to 600,000 years using the uranium/thorium method.[1]

Lignin, a biopolymer, is one of the main constituents of higher plants and consists of three monomeric units: sinapyl-, coniferyl-, and coumaryl alcohol. Lignin can be degraded into its monomeric units by alkaline CuSO_4 -oxidation. The oxidized monomer units can be analysed by UHPLC-ESI-HRMS with limits of quantification in the ng/g range. By determination of the ratios among different oxidation products in a speleothem, conclusions can be drawn on the type of vegetation above the cave. [2,3]

Levoglucosan, an anhydrosugar, naturally only originates from the combustion of cellulose and can thus be used as a biomass burning marker. Analysis of levoglucosan in sediments shows good correlation with traditional burning markers like black charcoal. [4] Mannosan and galactosan, stereoisomers of levoglucosan, are formed during the combustion of hemicellulose. Literature suggests that the ratio of levoglucosan to its isomers rather than absolute levoglucosan concentrations should be considered when characterizing burning events. [5] To date, no data on levoglucosan or its isomers in speleothems is published.

As the anhydrosugars are highly polar molecules, extraction and analysis with traditional reversed phase systems proved difficult. An optimized sample preparation to access both lignin and levoglucosan in speleothems is presented. Furthermore, a HILIC-UHPLC-ESI-HRMS method was developed to analyze the lignin oxidation products (LOPs) and anhydrosugars.

The methods were applied to a flowstone from a cave of the Dolomites in Southern Tyrol.

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