



First Multi-Shot thermochemolysis applied to the lipid-free organic fraction isolated from a peat (Jura Mountains, East France)

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Thermochemolysis was applied to a wide variety of polymeric organic materials, including complex and intractable samples (i.e. soils and sediments) for rapid structural characterization, chemical profiling or source determination of the original material.

Thermochemolysis using tetramethylammonium hydroxide (TMAH) is commonly used for the analysis of both free and bound carboxylic and alcoholic moieties because of its relatively strong alkaline character. As a consequence, the discrimination between the different moieties originally present in samples were done using TMAH combined alternatively with less reactive reagents [1,2 and references therein].

“Multi-shot” pyrolysis was used in the last few years for the analysis of complex natural organic matters [3 and references therein], by the sequential examination of the products released successively by thermal desorption and by thermal cracking from the same sample. Here, we present the results obtained after a multi-shot thermochemolysis performed on the lipid-free fraction of a peat sample from a wooded Sphagnum-dominated mire (Frasne, Jura Mountains, France) based on reactivity differences with selected chemical agents.

Reactants with increasing strength were used: hexamethyldisilazane (HMDS) followed by trimethylsulfonium hydroxide (TMSH) and finally TMAH. Products generated from each successive step were recovered in cold traps before being analysed by GC/MS. In order to determine the additional information provided by the multi-shot method, the results obtained by multi-shot thermochemolysis were compared with those obtained for the same peat sample using classical thermochemolysis done separately with each reagent.

Analysis of the thermolysates showed conspicuous differences between the shots regarding the nature and/or the distribution of the numerous products identified in the three thermolysates. The material thus appears to exhibit an expected complex and heterogeneous composition, including various types of tightly trapped and/or labile compounds released during the first shot with HMDS (mostly carbohydrates), covalently linked compounds released during the second shot with TMSH (mainly aromatic units from tannins) and units with a relatively higher thermal stability (monomers from biopolymers such as lignin and cutins, suberins or waxes) during the third shot with TMAH.

Most of these components originates from higher plants and exhibit low to moderate alteration due to the anoxic conditions of the studied sediment. Important differences were observed between the three shots and thus afforded important additional information in relation to the composition and sources of the original material, the nature of its components, and the large differences in reactivity between some of the (macro)molecular constituents.

Multi-shot thermochemolysis thus appears as an interesting tool to help deciphering the composition, origin and nature of soil and sediment organic matter.

References

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