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Optimized strategy of 1H and 13C solid-state NMR methods to investigate water dynamics in soil organic matter as well as the influence of crystallinity of poly(methylene) segments

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Water plays a crucial role in soil organic matter (SOM) having various different functions such as transport of material, elution of ,e. g., pollutants in soil, and also the sequestration of humic substances. Furthermore, the generation and quantification of hydrophilic and hydrophobic regions in soil has several effects on SOM which can also include the storage amount and time of certain material, especially chemical pollutants. The importance of water in soil is also documented by the multitude of scientific approaches to characterize soils including diffusion NMR to study the water channel structure in soil. Our focus is on the study of water dynamics and soil structure to elucidate mechanisms of physicochemical aging.

The approach uses the application of various solid-state NMR techniques - including 1H and 13C NMR - to get a multitude of information on SOM. In non-rotating samples, 1H lines are usually very broad and unstructured. Nevertheless, this rather simple technique allows for a differentiation of 1H containing chemicals based on their dynamics in soil. This includes rather solid soil components and solid as well as mobile water molecules. Based on an optimized 1H solid-state NMR strategy to study soil material together with a straightforward lineshape analysis, a series of soils and peats are characterized.

Although even 1H NMR with sample spinning (MAS) often gives only limited information on different structures, we present results on the application of 2D 1H-1H phase-modulated Lee-Goldburg sequences (PMLG), that show already at medium spinning speeds the separation of functional groups. Their quantification can be correlated with sample composition, type of sample conditioning, and other parameters such as cation type or concentration and heat treatment.

We are especially interested to correlate NMR data with DSC measurements based on a certain heat treatment of the soils. Our proposed model describes the presence of water in soil as a matrix linking together organic matter via hydrogen bonding. The measurement profile includes a conditioning step under a certain humidity as a starting point. Then, the soil is heated up to 110°C for 30 minutes to destroy the present water matrix and then follow the regeneration of this matrix over time. Both in 1H static NMR and DSC this physical aging of the water structure in soil can be followed. While the bridges may be easily disrupted, re-formation is slow in the SOM matrix and can be on the order of weeks and even months depending on the soil.

Apart from the water dynamics in soil, additional information can be obtained from 13C solid-state NMR which is the typical measurement technique for the study of soil due to the larger chemical shift range. In our approach, we propose a correlation of the analysis of soils based on functionality and quantification with results based on a special pyrolysis field ionization mass spectrometry optimized for soils to identify molecular mass distribution of soils. Furthermore, the presence of poly(methylene) groups that are present in both crystalline and amorphous morphologies, that can be clearly assigned by their chemical shifts, is interesting. Our heat treatment induces melting of the crystallites and depending on the cooling procedure (slow cooling in air or instant cooling in liquid nitrogen) changes the degree of crystallinity. The results on the NMR characterization show correlations with features observed from DSC measurements.

Our studies were done on a series of different soils and peats with varying organic and water content. The overall goal is to identify certain features that are typical for soils which help to identify correlating results obtained from other characterization techniques and therefore improve the general understanding of soils.