Geophysical Research Abstracts
Vol. 18, EGU2016-8235, 2016
EGU General Assembly 2016
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Dynamic Nuclear Polarization (DNP) solid-state NMR spectroscopy, a new approach to study humic material?

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Compared to solution NMR spectroscopy, solid-state NMR spectra suffer from broad resonance lines and low resolution. This could be overcome by the use of 2-dimenstional solid-state NMR pulse sequences. Until recently, this approach has been unfeasible as a routine tool in soil chemistry, mainly because of the low NMR sensitivity of the respective samples. A possibility to circumvent those sensitivity problems represents high-field Dynamic Nuclear Polarization (DNP) solid-state NMR spectroscopy (Barnes et al., 2008), allowing considerable signal enhancements (Akbey et al., 2010). This is achieved by a microwave-driven transfer of polarization from a paramagnetic center to nuclear spins. Application of DNP to MAS spectra of biological systems (frozen solutions) showed enhancements of the factor 40 to 50 (Hall et al., 1997). Enhancements of this magnitude, thus may enable the use of at least some of the 2D solid-state NMR techniques that are presently already applied for pure proteins but are difficult to apply to soil peptides in their complex matrix. After adjusting the required acquisition parameters to the system “soil organic matter”, lower but still promising enhancement factors were achieved. Additional optimization was performed and allowed the acquisition of 2D 13C and 15N solid-state NMR spectra of humified 13C and 15N enriched plant residues. Within the present contribution, the first solid-state DNP NMR spectra of humic material are presented. Those data demonstrate the great potential of this approach which certainly opens new doors for a better understanding of biochemical processes in soils, sediments and water.

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