

## Linking Iron and Nitrogen Cycles in Lake Sediment

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In anoxic environments, iron reduction can function as a significant pathway for organic matter oxidation, resulting in the production of ferrous iron. This Fe<sup>2+</sup> can potentially be re-oxidised by nitrogen species formed in surface sediment (e.g. nitrate; NO<sub>3</sub><sup>-</sup>, nitrite; NO<sub>2</sub><sup>-</sup>). The fate of such oxidized nitrogen compounds is of particular importance to nitrogen availability in microbial communities – determining whether nitrogen is retained in the environment in a bioavailable form (NH<sub>4</sub><sup>+</sup>) or effectively ‘lost’ as N<sub>2</sub>. Although both freshwater and marine microbial strains have been isolated which are capable of coupling NO<sub>3</sub><sup>-</sup> reduction to iron oxidation, the role of such processes for either iron or nitrogen cycling in natural environments is largely unknown.

The potential interactions of iron with NO<sub>3</sub><sup>-</sup> reduction pathways were investigated in anoxic incubations with iron-rich sediment from a shallow, mesotrophic Danish lake. <sup>15</sup>N tracing methods were used to determine contributions of denitrification and dissimilatory nitrate reduction to ammonium (DNRA) and how Fe<sup>2+</sup> potentially influences the partitioning between these processes. DNRA was found to be the dominant nitrate reduction process and appeared to be further stimulated by addition of Fe<sup>2+</sup> to incubations. These results suggest that NO<sub>3</sub><sup>-</sup> reduction coupled to Fe<sup>2+</sup> oxidation in this sediment yields NH<sub>4</sub><sup>+</sup> as opposed to N<sub>2</sub> as previously described in several culture studies; resulting in nitrogen being retained in the system as a bioavailable substrate. Whether our findings are more generally applicable to aquatic systems such as marine sediments and anoxic waters where Fe<sup>2+</sup> and oxidized nitrogen species co-occur is currently being explored.

## Identifying carbon pools in heterogeneous materials: Use of peak fitting and TGA-DSC-MS data

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Thermogravimetry-differential scanning calorimetry (TG-DSC) is an effective tool for the analysis of modelable carbon pools (including labile, recalcitrant, resistant and inorganic carbon) in soils [1,2]. Coupling of the TG-DSC instrument with a quadrupole mass spectrometer to give a TG-DSC-MS arrangement allows the temperature dependent evolution of low molecular weight species such as carbon dioxide and methane to be monitored at the same time as mass losses [2].

A TG-DSC-MS analytical run from 100 to 1000 °C in an atmosphere of 20% O<sub>2</sub> 80% He<sub>2</sub> produces data that can be interpreted using GRAMS-AI software (Thermo Scientific). After ruling out potential interferences, monitoring the m/z 44 ion shows the evolution of CO<sub>2</sub> at different temperatures during the TG-DSC-MS run and a semi-automated peak fitting process has been developed, whereby the proportions of labile, recalcitrant, resistant and inorganic carbon in a sample can be estimated. By defining a range of temperatures over which each carbon pool might be expected to thermally decompose, and running multiple iterations of GRAMS-AI's peak fitting algorithm, a composite “best fit” of the four individual carbon pool peaks to the m/z 44 trace can be produced. The relative peak areas indicate the proportion of each type of carbon in the sample and parallel use of an elemental analyser allows estimation of carbon masses.

The approach adds utility to the TG-DSC-MS approach where heterogeneous materials are under investigation and has been used to investigate man-made soils [3] and to study organic rich residuals from the drinking water industry [4]. It appears to have general applicability in cases where rapid estimation of the size of the four carbon pools is required.

[1] Lopez-Capel *et al.* (2005) *Soil Sci. Soc. Am. J.* **69**, 136-140. [2] Lopez-Capel *et al.* (2006) *J Anal. Appl. Pyrolysis* **75**, 82-89. [3] Manning *et al.* (2013) *Int. J. Greenhouse Gas Control*, submitted. [4] Finlay *et al.* (2013) *MinMag*, this volume.