Experimental report

10/12/2019

Proposal: 9-10-1590 **Council:** 10/2018

Title: Unraveling mechanisms of soil carbon sequestration: organic matter-mineral interactions

Research area: Chemistry

This proposal is a resubmission of 9-13-716

Main proposer: Luigi GENTILE

Experimental team: Ulf OLSSON
Erika ANDERSSON
Elin TORNQUIST
Viktoriia MEKLESH
Luigi GENTILE

Local contacts: Ralf SCHWEINS

Samples: Fe2O3x0.5H2O
Soil organic matter (carbohydrates)

Instrument	Requested days	Allocated days	From	To
D11	3	1	24/06/2019	25/06/2019
D22	3	0		

Abstract:

Dissolved organic matter (DOM) plays an influential role in terrestrial and aquatic ecosystems. DOM is made of molecularly dispersed compounds and suspended objects with a size below 0.2 μm. The interaction between minerals and DOM leads to the formation of organic matter-mineral colloids. Recently, the colloidal fraction has been proposed to play a central role in the stability of organic matter towards microbial decomposition. Yet, very little is known about their colloidal properties. The aims of this proposal are:
(i) Investigate DOM with SANS to obtain size and shape of the aggregates extracted from forest soil, and also obtain chemical information from contrast variation.
(ii) Investigate DOM-mineral interactions by adding a known amount of ferrihydrite (Fe2O3x0.5H2O) and observe its effect on aggregate structures. We will primarily focus on ferrihydrite since iron plays a key role in oxidative decomposition of organic matter; Here we will explore contrast variation to highlight the scattering from DOM and mineral, individually.
The results will allow us to characterize DOM and its interaction with relevant mineral particles.

Unraveling mechanisms of soil carbon sequestration: organic matter-mineral interactions

Background

Dissolved organic matter (DOM) is an important component in the natural cycling of carbon and also plays an important role for the transportation of nutrients and contaminants in soil. The interaction between DOM and soil minerals regulates to a major extent the release to the atmosphere or availability to plants of the dissolved carbon and nutrients in the DOM but these interactions are still poorly understood. The DOM has previously been characterised by small angle x-ray scattering (SAXS), elemental analysis and NMR, showing a main content of carbohydrates in the form, as obtained through fitting, of large (> 100 nm) polymer chains and dense clusters. When using techniques such as SAXS or light scattering on mixtures of DOM and minerals the minerals will dominate the scattering and the behaviour of the DOM remains invisible. The purpose of this project is to investigate the interactions between organic matter and different kinds of soil minerals and for doing this the contrast matching possible in SANS is desirable. During this one day beam time the focus was to determine the contrast variations of DOM extracted from forest soil and thereby conclude on its main constituents as a proof of concept for future studies.

Experiment

Freeze-dried DOM was re-dissolved in different concentrations of D_2O/H_2O to vary the solvent contrast. SANS was performed at 25°C at three different sample to detector distances (1.4 m, 8 m and 39 m) to obtain as large of a q-range as possible. Quartz banjo cells with a path length of 2 mm were used. To find the match point of the sample, and thereby its main constituents, the mean of the scattering intensity (I) was plotted as a function of the solvent D_2O content.

Results

The solvent subtracted scattering curve for 100% D_2O can be seen in figure 1. Good data was obtained for 100%, 80% and 60% D_2O but closer to the contrast match point the data is very noisy and longer acquisition time would have been preferable. Despite measuring at the lowest q achievable, no plateau is visible in the scattering curve indicating that the particles have a radius of gyration larger than 600 nm. The shape of the curve agrees well with what has previously been obtained by SAXS and is thought to originate from a mixture of dissolved polymer chains and more densely packed clusters.

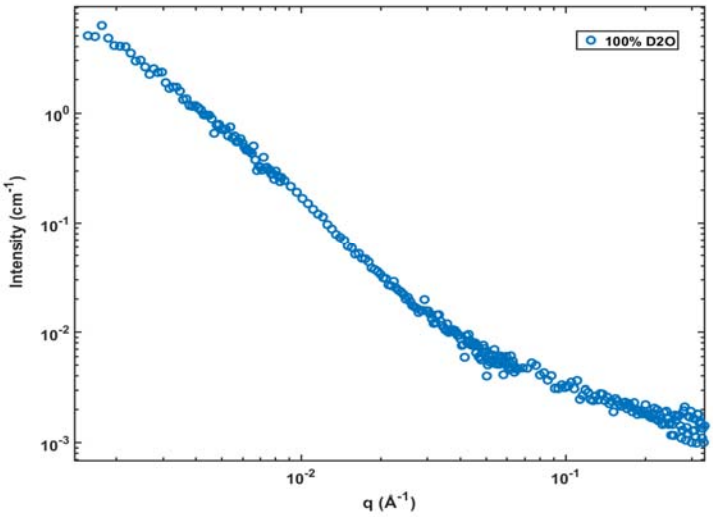

Figure 1. Solvent subtracted scatting curve for DOM in 100%D_2O

Figure 2 shows the obtained $\sqrt{I}$ vs $\%D_2O$ plot from which a contrast match point of 30% D_2O can be determined as this is the only minima. This agrees with the contrast match point of carbohydrates calculated from scattering length density. The irregularity at 50% is considered an experimental error due to noisy data.

The results will be presented together with an analysis of the different sugars making up the carbohydrates in a paper which is not yet submitted. Based on these results, future studies will continue looking at the interactions between the DOM and different soil minerals.

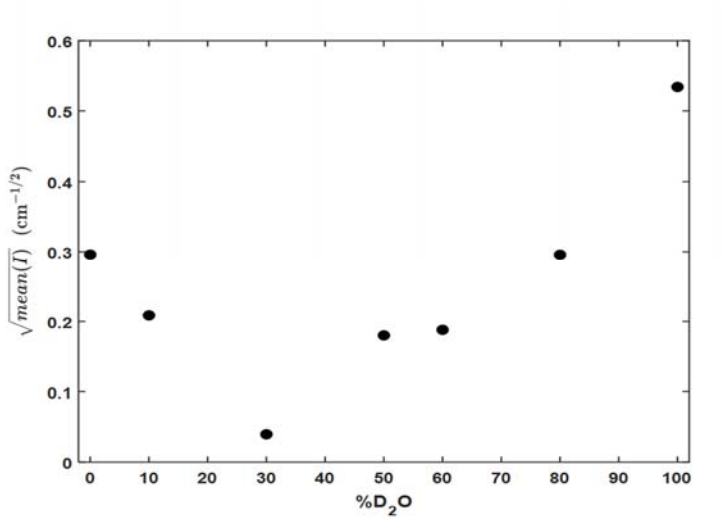

Figure 2. $\sqrt{I}$ vs $\%D_2O$ showing the contrast match poit