Geophysical Research Abstracts
Vol. 16, EGU2014-14137, 2014
EGU General Assembly 2014

Can X-ray photoelectron spectroscopy (XPS) relate changes in wetting properties of biogeochemical interfaces to surface chemical composition?

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Under natural conditions soil particles are coated by biogeochemical interfaces (BGI), a nm to μm thick layer of inorganic and organic components that govern all processes involving liquid and colloid transport. A crucial factor here are the wetting properties that can be quantified in terms of contact angle (CA), visible at the three phase boundary after placing a drop of water on the surface. The CA is determined by the kind (polar, non-polar) and orientation of functional groups present in the BGI, where only the first nm determines the wetting properties of the whole material (“CA-interphase”).
To relate CA to surface chemical composition, a bulk analysis will be inappropriate, especially due to the distinct dilution of BGI-specific elements. Common surface analysis techniques like ATR-FTIR or EDX have an analysis depth of about 1 μm which distinctly exceeds the CA-interphase. A promising alternative is X-ray photoelectron spectroscopy (XPS) whose maximum analysis depth of only 10 nm is considerably closer to the extension of the CA-interphase. Although originally designed for homogeneous flat surfaces, we will reveal XPS as a tool to chemically characterize changes in CA of soil particle surfaces by showing examples for BGI modification due to heat treatment and soil development within a soil chronosequence.
The wetting properties of a sandy podzol topsoil were modified by treatment at 40°C, 60°C, and 105°C for 24 h in open glass beakers. The non-treated material used as reference showed an initial CA around 100° that within 5 seconds decreased to about 60°. With increasing treatment temperature the initial CA increased to about 106° (40°C), 113° (60°C), and 125° (105°C), indicating distinct changes especially after treatment at 105°C. At the same time, CA stability increased and for the 105°C-treatment, CA after 5 seconds still was 123°, i.e. the sample became permanently hydrophobic (CA>90°). XPS analysis revealed some characteristic changes in surface chemical composition, especially after treatment at 105°C: the oxygen concentration decreased and the carbon concentration increased.
Within the chronosequence (Damma glacier, Swiss Alps) BGI development was followed by decreasing wettability as CA increased from 0° (0 yr; hydrophilic) to about 98° for a soil age of 120 yr. XPS analysis showed increasing carbon and nitrogen concentration that both could be related to CA. Oxygen concentration decreased with soil age.
As a means to relate wetting properties and chemical composition the O/C ratio can be used. It decreased within the chronosequence from around 5 to around 1.5 which correlated very well with the increase in CA. The linear regression (r2=0.932) found for the chronosequence fitted excellently with the common regression including the heat treated podzol samples and further sandy and silty samples (r2=0.930).