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Carbon Spectromicroscopic Studies of Metal Interactions

M. Nachtegaal (U. of Delaware), E.A. Ghabbour (Northeastern U.), G. Davies (Northeastern U.), S. Wirick, C. Jacobsen (SUNY, Stony Brook) and D.L. Sparks (U. of Delaware)

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Introduction: The release of potentially toxic metals into the environment is of worldwide concern. Due to its high specific surface area and cation exchange capacity (CEC), humic substances (HS) can play a significant role in affecting the retention and mobility of metals in the environment. Humic substances are defined as a series of relatively high-molecular-weight, brown- to black- colored substances formed by secondary synthesis reactions. Contrary to their inorganic counterparts, the interactions of metals with humic substances are not well understood. In this study scanning transmission x-ray microscopic analysis was applied to study morphological changes of humic acid (HA) in solution upon metal reaction, and was combined with NEXAFS studies of the metal reacted HA. This study will provide unique spatially resolved chemical information on the affinity of the metals for the different functional groups of HA.

Methods and Materials: The humic acid used in this study was isolated from a peat bog soil and has a low (0.25 wt%) ash content. The freeze dried humic acid was hydrated in double deionized water for 1 week (0.1 g humic acid/ liter), after which it was reacted for 24h with a 0.5 mM metal solution, at pH 5 and $I = 0.1 \text{ M NaNO}_3$. A small droplet of this reactant was placed in between two Si N windows. These two windows were screwed tightly together using a so called 'wet cell' until Newton interference fringes indicated the right sample thickness. Image stacks of the metal reacted humic acid were collected, with 0.05 eV energy steps at the region of interest. NEXAFS spectra were extracted from those image stacks by choosing a region free of humic acid as I_0 and a region with a humic acid particle as I .

Results: A small selection of the collected spectra is shown in Figure 1. These spectra were background corrected and normalized. The most important peaks were assigned to phenolic (285 eV), aliphatic (287 eV) and carboxylic (288 eV) functional groups. Shifts in peak intensity and peak position were observed after reaction of the humic acid with the metal solutions. The shifts in peak intensity can be attributed to the bonding of the metals to these functional groups. Shifts in peak position were only observed for some of the 3d transition metals and indicate a shift of electron negativity from the metal to the HA functional groups or opposite. The image stacks showed a coagulation of the humic substances upon metal reaction.

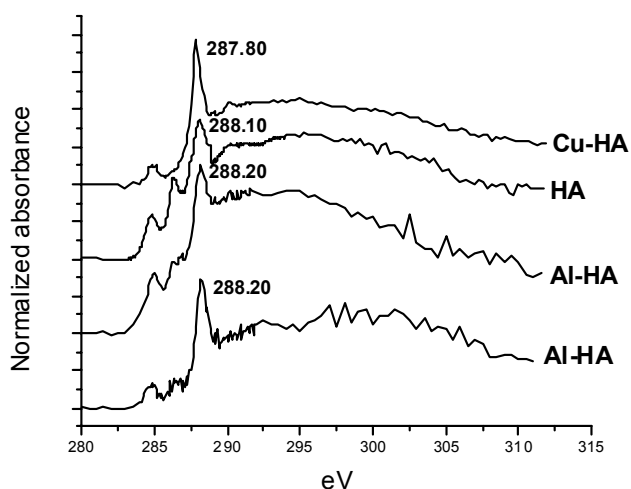


Figure 1: 0.1 g/L HA reacted with a 0.5 mM Cu or Al solution, for 24 h at pH 5 and $I = 0.1 \text{ M NaNO}_3$.