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X-ray Spectromicroscopy Studies of Nanoparticles in the Environment

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Motivation: In recent time, carbon nanotubes (CNTs) have drawn a lot of attention due to their unique properties and due to that possible application, for instance in pharmacology, material sciences or as semiconductors. CNTs are tubes with diameters in the nanometer scale, but with lengths up to several millimeters. Their walls consist of carbon atoms, each bound to three other carbon atoms (sp2-hybridization), which results in a hexagonal honeycomb-like structure. They can also be functionalized, e.g. with carboxyl- or hydroxyl groups.
Although the production and modification of CNTs in sizable quantities have been improved continuously, the characterization of these nano-particles still needs to be advanced. Additionally, the ecological aspect comes into account. Since most of these new materials consist of particles too small to be biodegraded, it is important to analyze the impact of CNTs on the environment (and biomolecular matter in general).
Here we present the result of a study of pristine and functionalized carbon nanotubes (CNTs) using the x-ray scanning transmission microscope (STXM) at the storage ring BESSY II in Berlin for a NEXAFS (near edge x-ray absorption spectroscopy) analysis with spatial resolution.

Experiment and results: We characterized three types of multi-walled CNTs (3-15 walls, outer diameter of 13-16 nm and length distribution 1-10 nm) by x-ray spectromicroscopy. To be more specific, we have investigated different CNT-samples with energies around the C1s K-shell edge ($\sim$284 eV) dry and in aqueous environment at ambient conditions. Using the STXM, the spatial information from the x-ray image with a pixel size of 50 nm can be combined with NEXAFS-spectra[5] of each pixel of the image area.
The differences between the species are observable both in the microscopic images and the spectral data. The evaluation[1][2] of the NEXAFS-spectra yields information about the chemical bindings in the sample.
Discussion The difference between the reference sample and its modifications, CNTs with carboxyl-groups, and CNTs with COOH-groups loaded with depleted uranium are clearly visible.
The x-ray-microscopic images show that the expected curly-network structure expected for all samples is only observable in the case of the pristine CNTs (reference). With the additional binding of depleted uranium, the network structure has vanishes totally. Instead, the particulate character becomes the dominant form. In all three cases, the NEXAFS-spectra unambiguously revealed the binding forms C=C and, if present, C=O. The comparison of their spectra makes furthermore clear, how the changed environment of a bond influences its position on the energy scale.
In addition to that, we recently studied the behavior of pristine and carboxylated CNTs in aqueous media and their interaction with soils.
Comprising, the STXM is ideally suited for examinations like this, since it combines x-ray imaging techniques with NEXAFS-spectroscopy[3][4][6], and the samples can stay at ambient conditions throughout the whole experiment.
Future experiments will concentrate on CNTs in combination with other materials to reveal further insights into the interaction of nano-particles with other substances, for instance soils and in building materials.

Literature:
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