

Microscopic Methods for Evaluating Ceria Nanoparticles in Thin Tissue Sections

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Cerium oxide nanoparticles have a wide range of uses in industry, including applications as polishing and chemical planarization media, catalysts in automotive fuel additives, catalytic converters and gas sensors. [1] The widespread use of these materials has raised concerns about potential occupational hazards. There is a growing need to characterize and identify the key properties of these materials that have adverse health impacts. High resolution transmission electron microscopy (HRTEM) coupled with advanced detectors allows one to probe materials in unprecedented detail, providing both local chemical information and their structural properties. Electron energy loss spectroscopy (EELS) and energy dispersive x-ray spectroscopy (EDS) in scanning (STEM) mode enable elemental point analysis for profiling and mapping of composition variation, material phases, and oxidative states. It has been hypothesized that surface interactions play an important role in the toxicity of some nanoscale materials, and these methods can distinguish the surface versus bulk properties of nanomaterial particles. [2] Application of advanced characterization methods to cytotoxicity studies in cell cultures and tissues will improve the quality of toxicological data and exposure standards derived from them. [3]

We performed EELS analysis and collected spectrum images on a 200 kV JEOL 2100F TEM/STEM to investigate morphologies, size distribution and oxidation states of ceria nanoparticles in rat spleen and liver tissues. Thin sections were prepared from selected processed tissue blocks and analyzed on carbon coated grids, without tissue staining. A small probe size (0.2 nm) in STEM mode was used to minimize the sample damage under the electron beam. The oxidation states of cerium, respectively Ce(IV) and Ce(III) in oxides CeO₂ and Ce₂O₃, were determined by the fine structures of M_{4,5} edges in EELS. Example results for these analyses are shown in Figs. 1 and 2. Different valance states of ceria were characterized by core loss EELS. Relative composition and spectral mapping of Ce, P, C, Ca, N and O were determined by spectrum imaging.

References:

- [1] Cassee, F. R. *et al.*, *Crit. Rev. Toxicol.*, **41**, (2011), 213.
- [2] De Marzi, L. *et al.*, *Int. J. Mol. Sci.*, **14**, (2013), 3065.
- [3] Graham U. M. *et al.*, *ChemPlusChem*, (2014), accepted.

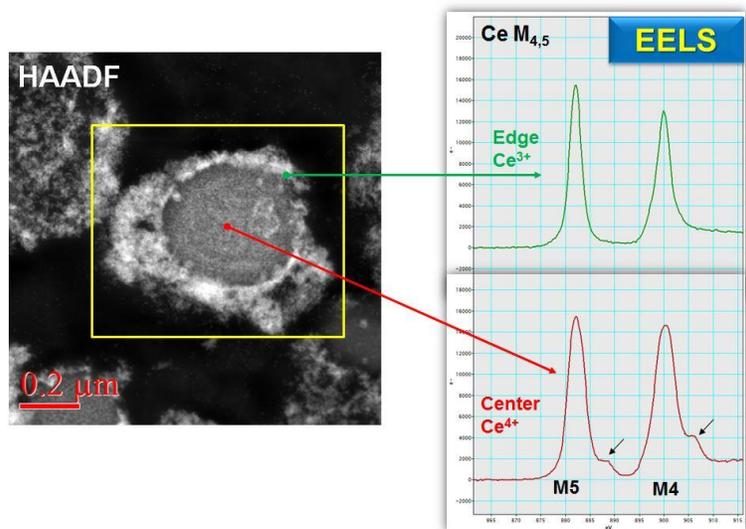


Figure 1. Z-contrast image (left) shows the localization of ceria particles (bright contrast) in mouse spleen cells. Oxidation states of Ce (Ce^{4+} and Ce^{3+}) on the edge and center of ROI can be identified by M_4/M_5 ratio of ELNES (right).

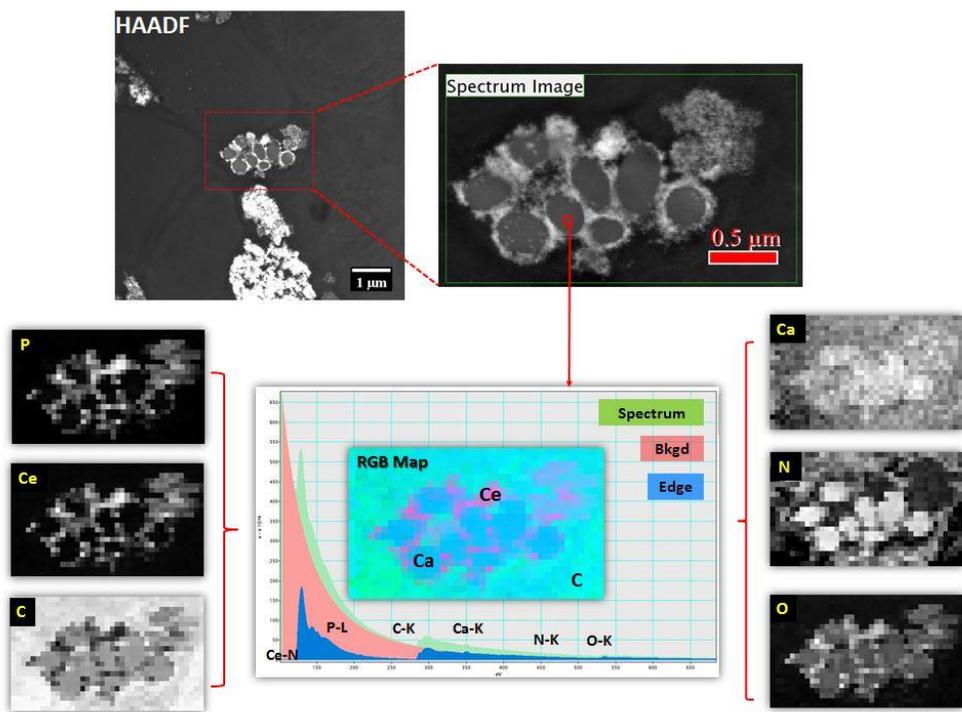


Figure 2. HAADF spectrum images show composition maps of P, Ce, C, Ca, N and O in spleen tissue.