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Structural and Chemical Characterization of Janus Nanoparticles with 2D and 3D Electron Microscopy

Jesus Valdez[1*], Wanting He[2], Yannan Liu[2], Dongling Ma[2], and Audrey Moores[3]

[1.] Facility for Electron Microscopy Research (FEMR), McGill University, Montréal, QC, Canada.
[2.] Institut National de la Recherche et Scientifique, Varennes, QC, Canada.
[3.] Chemistry Department, McGill University, Montréal, QC, Canada.
* Corresponding author: jesus.valdez@mcgill.ca

Nanotechnology is a very active research area with applications over vast technology domains. [1] Nanoengineering has been key in designing and synthesizing new nanomaterials with controllable structural and chemical features to afford tunable physical and chemical properties. [2] These properties have enabled remarkable advances for applications in the biomedical, catalysis and optical areas.

The combination of two materials with different properties at the nanoscale has allowed scientists to open new discovery paths. Janus nanoparticles (JNPs) have been used as the next promise for nanotechnology thanks to the combination of complex structural nano-systems that make them suitable for a wide range of applications. [3] However, because of their inherent complexity, JNPs characterization has been challenging.[4] Electron microscopy (EM) and coupled techniques such as electron diffraction, energy dispersive X-Ray spectroscopy (EDS), and electron energy loss spectroscopy (EELS) are powerful tools in the nano scientist's toolbox, [4], [5] but they need to be combined with 3D analysis to fully tackle this question, especially using low background and high tilt angle holders.

In this work, we employed EM techniques of characterization to study JNPs made from Au and (4-hydroxyphenyl) zinc poly-porphyrin THPG nanoparticles. Scanning transmission electron microscopy (STEM) and high angle annular dark field detector (HAADF) show the conformation of the JNPs where one single gold nanoparticle is attached to a single-larger THPG nanoparticle. Figure 1 shows the electron tomography (ET) performed using the HAADF detector. This technique reveals the unique structure that is difficult to elucidate by 2D imaging, showing the hollowed nature of the THGP nanoparticle. These structural details obtained by 2D and 3D imaging, provide valuable information on the interaction of the two phases (metal-polymer) in JNPs and the advantages and limitations for the application of the JNPs.

The chemical characterization was performed using STEM-EDS. The use of a low background double-tilt holder increased the signal for the characteristic elements in high resolution. The tilt feature also helped in finding the correct position of the AuNPs to avoid an overlap with the THPG NP signal. The electron energy loss spectroscopy (EELS) confirmed the observations made on EDS spectroscopy as seen in Figure 2. Additionally, a high-resolution EELS map revealed a diffusion of elements characteristic of the THGP polymer. The structural and chemical interaction between the single AuNPs with the polymer is critical for the application of these nanoparticles. The use of electron microscopy techniques of characterizations was essential to elucidate the physical and chemical properties of the JNPs.

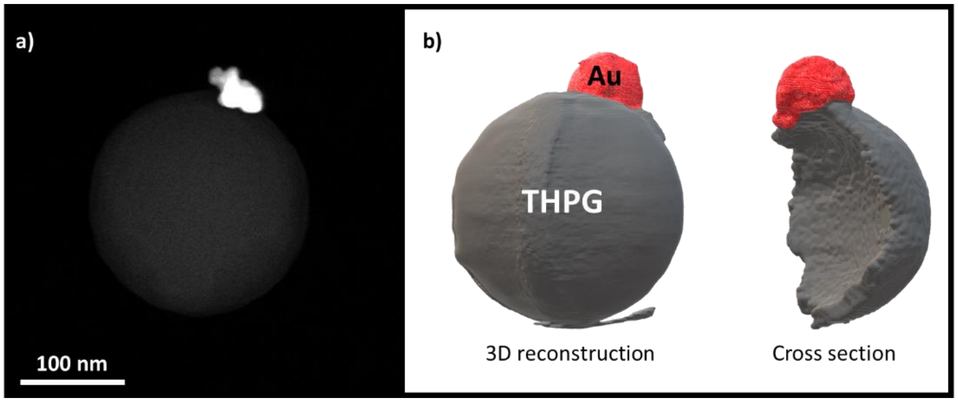

Figure 1. 2D and 3D characterization of a single JNP. STEM-HAADF image of a single JNPs showing two different phases of a small metal NP and a large THPG polymer NP (a). ET for a single JNPs and its cross-section (b) showing the complete and hollow structure of the JNP.

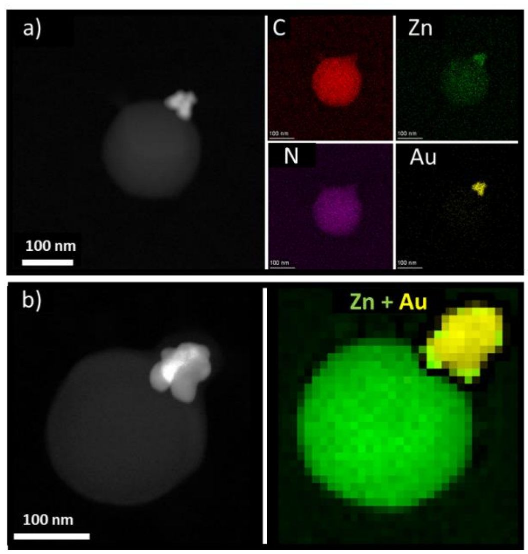

Figure 2. STEM-EDS image and elemental mapping for a single JNPs (a) showing the characteristic elements for the Janus nano-system. STEM-EELS mapping of a JNP, showing and supporting the information obtained by STEM-EDS (b).

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