

# Supported Nanoparticle Stability, Assembly and Extended Charge Neutralization in Deep Eutectic Solvents

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## Abstract

Supported nanoparticles are promising materials for various technologies including fuel cells, catalysis and sensors. Here we employ nanoparticle electrodeposition from Type III Deep Eutectic Solvents (DES) as the method of preparation. Recently, it has been shown that nanoparticle electrodeposition from room temperature ionic liquids (RTIL) can allow for stable nanoparticles of various shapes and sizes to be synthesized. Key to the manipulation of the particle morphology is an understanding of the interaction between the particles and solvent. In this work, *in-situ* synchrotron ultra small angle X-ray scattering (USAXS) is combined with electrochemical techniques, such as: electrochemical impedance spectroscopy (EIS) and cyclic voltammetry (CV), as well as *ex-situ* SEM imaging to study the supported nanoparticles. Using this approach, we show that the state of the electrodeposited nanoparticles is very different *in-situ*, compared with what is observed *ex-situ*. Based on our results, we propose that the deposited nanoparticles assemble into 2-D superstructures, rich in adsorbed species, immediately following the electrodeposition pulse. The abundance of these adsorbed species, within the superstructure, induces an anionic layer above them, which can be observed by USAXS, as well EIS. The surface charge of the particles is, therefore, not neutralized locally, as is the case with traditional colloidal systems. We also show that these otherwise stable nanoparticles readily aggregate when the DES is removed. This study therefore emphasizes the unique opportunities that are possible in nanoparticle electrodeposition from DESs, as well as the value of an *in-situ* approach.