Synthesis, characterization and electrochemistry of water soluble palladium nanoparticles

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Introduction:

In this work we present a new method for aqueous synthesis of water soluble palladium nanoparticles using bicinchoninic acid (BCA) as a capping ligand. The synthesized particles were characterized using x-ray powder diffraction, energy dispersive x-ray spectroscopy and transmission electron microscopy. Obtained nanoparticles were electrochemically deposited on glassy carbon electrode and electrochemistry of palladium nanoparticles was studied.

Synthesis of bicinchoninic acid (BCA) capped water soluble palladium nanoparticles:

BCA is a water soluble analogue of bipyridyl containing two quinoline rings. The carboxylate moieties are responsible for water solubility. BCA capped Pd NPs were prepared by reduction of H_2PdCl_4 solution using $NaBH_4$ as a reducing agent. The carboxylate moieties are responsible for water solubility.

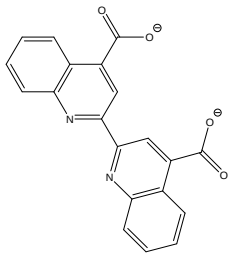

Figure 1: Structure of bicinchoninic acid (BCA).

Transmission electron microscopy of Pd-BCA NPs:

TEM samples were prepared by drop coating a dilute solution of the BCA-Pd NPs onto a formvar coated 400 mesh copper grid. The sample showed particles of average diameter~ 2.0 ± 0.8 nm.

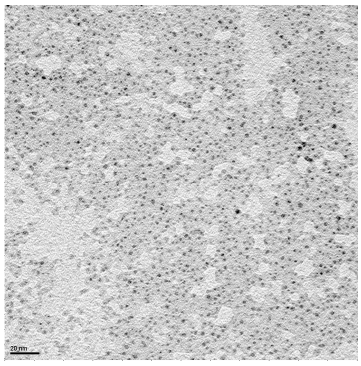

Figure 2: TEM image of water soluble Pd-BCA NPs.

Electrochemical deposition of Pd NPs on glassy carbon electrode:

Pd NPs were electrochemically immobilized on GCE using Pd-BCA NPs in 0.1 M K_2SO_4 solution at 100 mV/s. The appearance of cathodic peak at around -0.2 V, which is attributed to the reduction of PdO to Pd indicates deposition of Pd NPs on GCE. This peak increases with increasing number of cycles indicating further deposition of Pd NPs on the GCE.

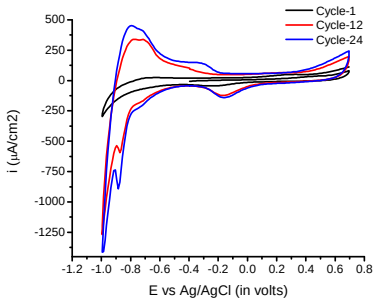

Figure 3: Electrochemical deposition of Pd NPs on glassy carbon electrode.

Cyclic voltammetry of deposited palladium nanoparticles:

The cyclic voltammetry of the Pd NPs immobilized on GCE was studied at different scan rates in 1 M KOH. The stripping of the PdO and the formation of PdO can be seen at -0.4 V and was found to linearly increase with scan rate. At higher scan rates hydrogen insertion peaks are shifted to more negative potential whereas hydrogen desorption peaks shifted to less negative potential as compared to low scan rates leading to increased separation between insertion and desertion at higher scan rates. This behavior is characteristic of an adsorption process that is associated with slow electron transfer.

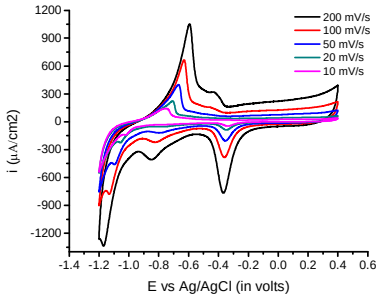

Figure 4: Cyclic voltammogram of Pd NPs on glassy carbon electrode in 1 M KOH for scanning rates of 10, 20, 50, 100 and 200 mV/s.

Future plans: Purification of Pd-BCA NPs of different size ranges. Size-dependent anodic dissolution and catalytic properties studies with Pd BCA NPs.

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