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Detection of Insulating Nanoparticles at Pt and Hg Electrodes

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Abstract

Using chronoamperometry, it is possible to detect the adsorption events of single nanoparticles at an ultramicroelectrode (UME).[1,2] When a nanoparticle strikes the electrode surface, it blocks part of the electroactive area, resulting in a decrease to the electrode current. The extent to which UME surface properties affect the nanoparticle response is still poorly understood. This research compared collision data for polystyrene nanoparticles using a solid Pt UME to new experiments using a Hg UME. Blocking of the electrode area by nanoparticles was detected at the Hg UME, although individual particle impacts could not be resolved, possibly due to mobility of the particles on the Hg surface. The electrodes used were also characterized using cyclic voltammetry (CV).

Project Summary

- Characterization of Pt UME; making and characterization of Hg UME
- Pt UME Nanoparticle collision experiments (950, 500 nm diameter polystyrene)
- Compare collision data at Pt and Hg UMEs to better understand influence of surface on particle detection
- Future applications: Fundamental studies of nanopaticle electrochemistry; chemical sensors

Electrode Preparation

Pt Electrode Preparation

- 10 µm diameter Pt disk UME, sealed in glass
- UME sanded, polished and checked under microscope accordingly
- Potential Cycling in 0.5 M Sulfuric Acid (+1.5 to –1.0 V vs. Pt wire for 30 minutes)
- Ran CVs to ensure it worked properly

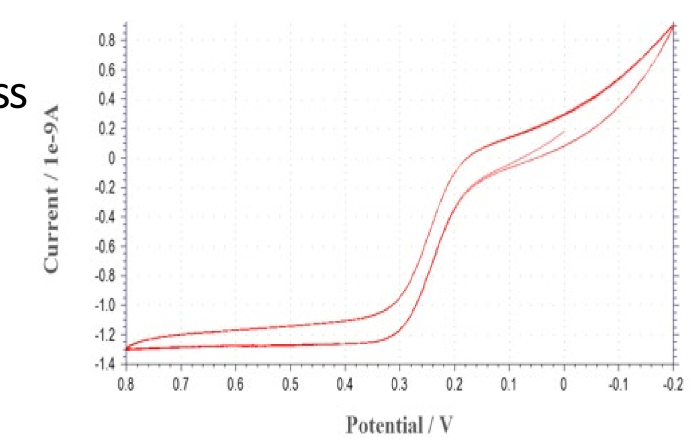
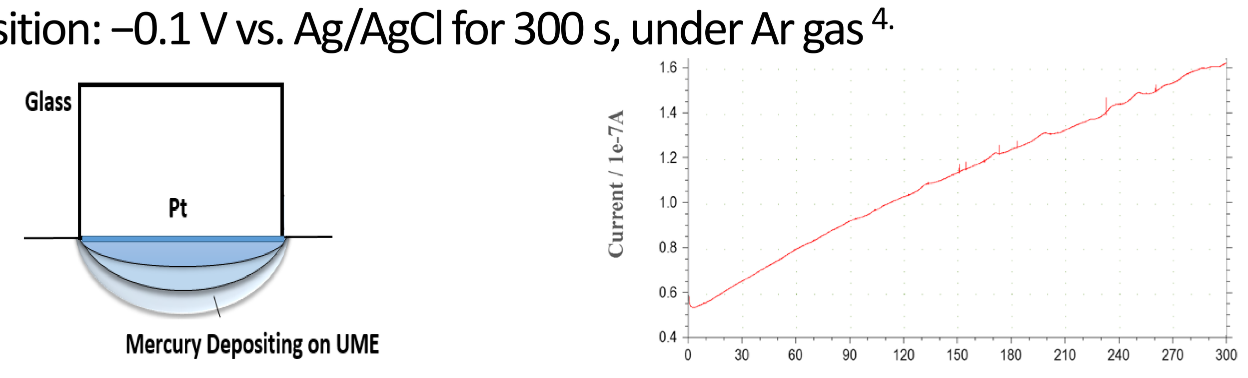


Figure 1: CV Characterization of Pt UME in 1mM FcDm with1 mM KCl
Parameters include -0.2 to +0.8 V vs. Pt reference electrode, 0.05 V/s scan rate

Hg Electrode Preparation

- Sanded and polished Pt UME to remove remnants of mercury
- UME in 10 mM $Hg_2(NO_3)_2$•$2H_2O$ and 0.1 M KNO_3/1 M HNO_3 solution
- Hg deposition: –0.1 V vs. Ag/AgCl for 300 s, under Ar gas [4]



Figures 2 & 3: Deposition of Hg on 10 µm Pt UME at –0.1 V vs. Ag/AgCl for 300 seconds
Figure on left shows more and more Hg adding onto UME to create larger surface area
Figure on right shows constant decrease in current due to increasing surface area

Particle Collision Experiment at Pt UME

- Pt UME: Characterization and Collision Experiments
  - 3 electrode cells in 1mM KCl and 1 mM Ferrocenedimethanol (FcDM) solution put inside Faraday Cage
  - Working = Pt UME; Counter = Pt electrode; Pt wire quasi-reference
  - Polystyrene nanoparticles were washed to eliminate surfactant

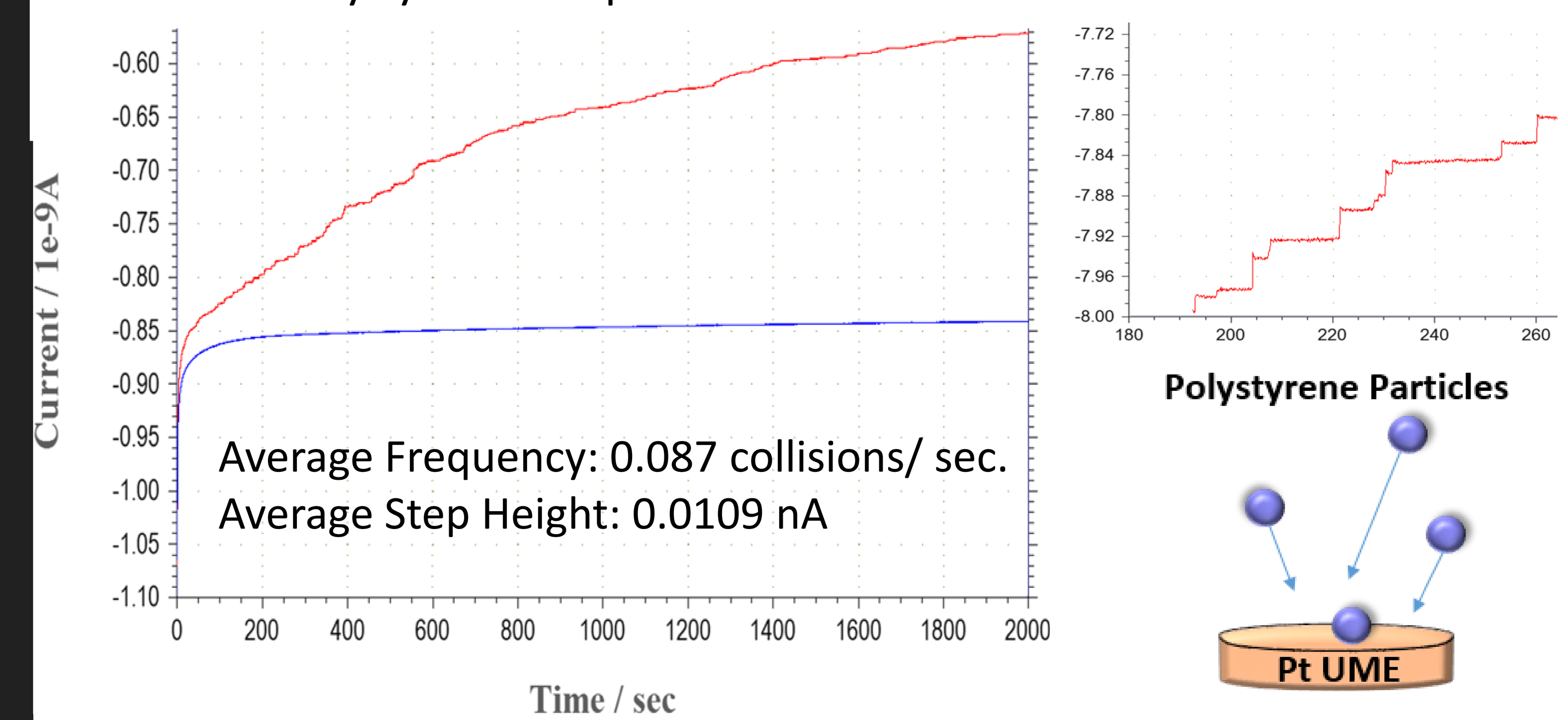


Figure 4 & 5: 960 nm diameter particle collision data on 10 µm Pt UME at +0.4
Figure on left shows control (blue) and nanoparticle collision data (red)
Figures on right shows close-up of single collisions by the stairstep pattern

- Pt UME showed single steps for each particle collision
- Constant decrease in current shows the blockage of electroactive area

Hg UME Characterization

- Hg UME: Characterization and Collision Experiments
  - Solutions: 1mM FcDM with 1mM KCl; 1mM FcDM with 1mM KNO_3
  - Recorded CVs and Amperometric Collision Data

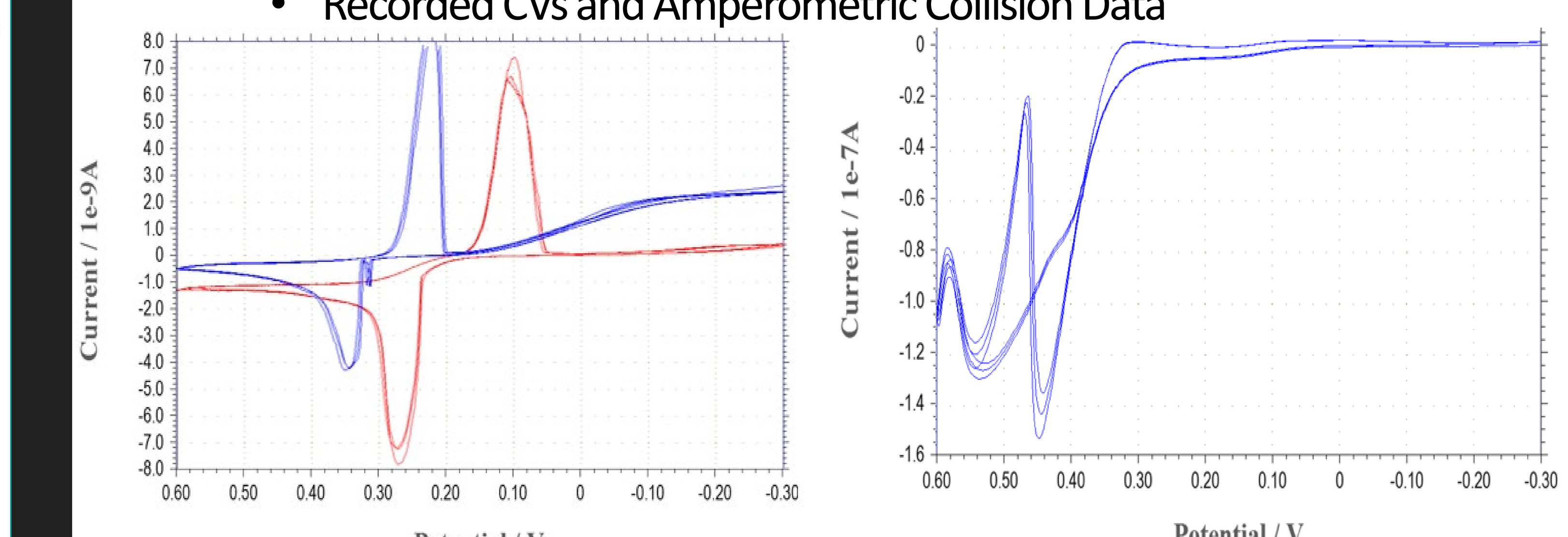


Figure 6 & 7: CV Characterization of Hg UME in different solutions
Parameters include -0.3 to +0.6 V vs. Ag/AgCl, 0.05 V/s scan rate
Figure on Left shows 1mM FcDm with1 mM KCl (red) or with 1 mM KCl solution (blue)
Figure on right shows 1 mM FcDM with 1 mM KNO_3 solution

- Both KCl solutions ,with and without FcDM, showed large peaks in the CVs
- With the KNO_3 solution, mercury becomes unstable past +0.4 V

Particle Collision Experiment at Hg UME

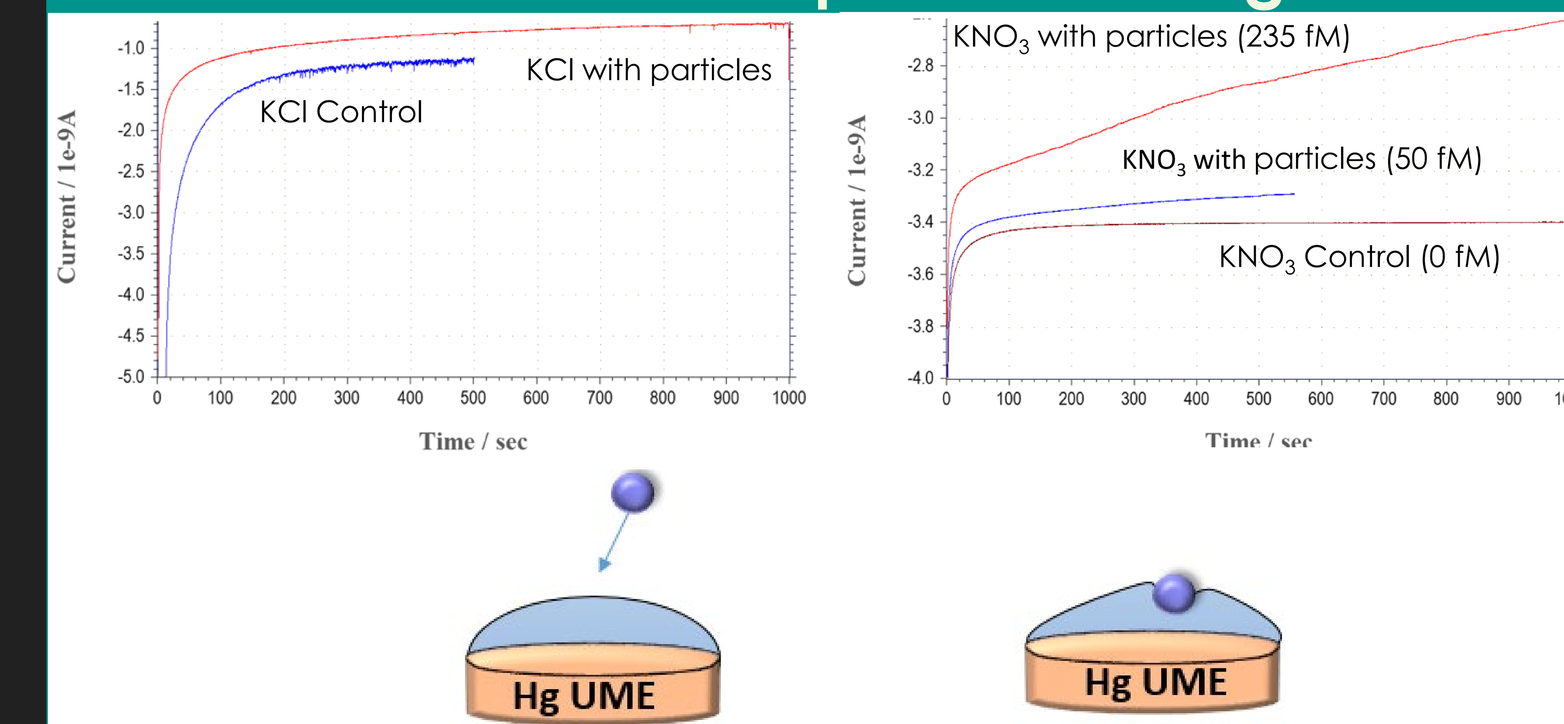


Figure 8 & 9: Nanoparticle Collision Data on Hg UME in 2 solutions at +0.3 V vs. Pt wire
1 mM FcDM/1 mM KCl and 1 mM FcDM/1 mM KNO_3 solutions
Figures on top show nanoparticles interacting with Hg UME in different solutions
Figure on bottom shows single particle interacting with Hg UME

- Shows blocking of electroactive area by decrease in current, but not single particle steps
- No effect of the particles in KCl solution; surface reaction possibly disturbs particle adsorption

Conclusions and Future Work

- Detected single insulating nanoparticles on Pt UME
- Successfully made a Hg UME using electrodeposition
- Saw blocking effects of nanoparticles on Hg UME
- Collected CVs and chronoamperograms for Pt and Hg UMEs

- Future Work
  - Collect more CVs and collision data for Hg UMEs
  - Understand Hg CVs and collision data
  - Try new electrolyte; 1 mM $KClO_4$, which may be less reactive with mercury
  - Run more trials using 500 nm diameter polystyrene particles

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References

1. Quinn, B. M.; van 't Hof, P. G.; Lemay, S. G. Time-Resolved Electrochemical Detection of Discrete Adsorption Events. *J. Am. Chem. Soc.* **126,** 8360–8361 (2004).
2. Boika, A.; Thorgaard, S. N.; Bard, A. J. Monitoring the Electrophoretic Migration and Adsorption of Single Insulating Nanoparticles at Ultramicroelectrodes. *J. Phys. Chem. B* **117,** 4371–4380 (2012).
3. Fosdick, S; Anderson, M; Nettleton, E; Crooks, R. Correlated Electrochemical and Optical Tracking of Discrete Collision Events. *J Phys. Chem.* 5994-5997 (2013).
4. Mauzeroll, J; Hueske, E; Bard A.J. Scanning Electrochemical Microscopy. 48. Hg/Pt Hemispherical Ultramicroelectrode: Fabrication and Characterization. J. Am. Chem. 75.15, 3880-3889 (2003).