

Standard Operating Procedure

Task: Electrode Polishing

Date: May 29, 2013

Revision Date: Nov 19, 2021

Background:

- Electrode polishing helps to create a structurally consistent surface to facilitate the acquisition of reproducible data.
- Disc electrodes should be polished using 0.3 and 0.05 micron alumina according to this SOP before any electrochemical experiment (e.g. cyclic voltammetry, chronoamperometry, differential pulse voltammetry, controlled potential electrolysis)
- Disc electrodes should be polished *between every cyclic voltammetry scan* using 0.05 micron alumina whenever possible. Assume that some amount of deposition onto the electrode is occurring until proven otherwise. When working in the glovebox where polishing is cumbersome or impossible, bring multiple pre-polished disc electrodes into the glovebox for use in successive scans.

Training Requirements:

- Lab safety training
- Electrochemistry training

Potential Hazards:

- Respiratory irritation due to inhalation hazard of small alumina particles

Special PPE Requirements:

- Standard laboratory attire

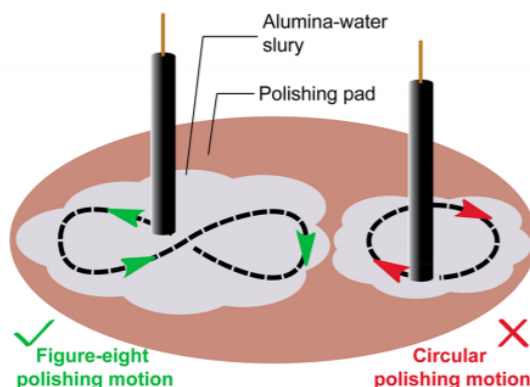
Materials Needed:

- Disc Electrode (glassy carbon, GC, or boron-doped diamond, BDD)
- Polishing kit
 - MicroPolish Powder (0.3 and 0.05 micron)
 - 2-7/8" Mastertex PSA Buehler polishing pads
- High-purity high-resistance ($>18\text{ M}\Omega$) Water, methanol, and acetone squirt bottles

Procedure:

- Inspect the surface of the electrode:
 - The electrode surface should look uniformly shiny and reflective.
 - General lack of luster, or patches of grime, grit, or foreign material can usually be addressed with polishing.
 - If the electrode surface is visibly scratched, consult a senior electrochemist in the group.
- Prepare the polishing pads:
 - Identify the appropriate polishing pad. There are two polishing pads, one for the 0.3 micron powder and one for the 0.05 micron powder. Be careful not to cross-contaminate the polishing pads.

- Add water to each polishing pad to wet the surface.
- Add a pea-sized scoop more polishing powder to the pads if necessary.
- Polish the electrode:
 - Before beginning an experiment, polish with 0.3 micron powder, followed by 0.05 micron alumina
 - Between CV or DPV scans or between CA experiments, polish with 0.05 micron alumina
 - Rinse the electrode with water. Note: Water for polishing and rinsing should be high-purity high-resistance ($>18\text{ M}\Omega$) grade. **Do NOT use DI water from the sink taps!**
 - Keeping the plane of the disc electrode perpendicular to the pad, firmly (but not forcefully) and move the electrode in a figure 8 pattern for 20 seconds (See figure, from *J. Chem. Ed.* **2018**, 95, 197)
 - Thoroughly rinse the electrode with water
 - Repeat with the other alumina powder, if necessary. **Note: If you are polishing between each scan, it is not necessary to add fresh alumina and water each time.**
 - Rinse the electrode with acetone to remove water. Blot with a KimWipe to remove any solvent droplets. Do NOT apply pressure or “scrub” the electrode with the KimWipe, as this could scratch the electrode surface.
 - Inspect the electrode surface in the light to check for scratches or blemishes. If scratches are present, consult an experienced electrochemist.
 - Cover the polishing pads for storage after use, to keep away dust or particulates.



References

J. Chem. Ed. **2018**, 95, 197