Grant Messner

Section 2

Procedure

1. Connect a 365 nm LED to the driver box provided and connect power supply. Set to constant wave (CW) and turn the dial all the way to the right.
2. Take one of the electrode plates and secure a copper foil wire pair to the plate with binder clip. Make sure the foil is contacting conductive side, ensure that wire goes through hole in binder clip. Fill the long pass rectangular cell half full with electrolyte (NaOH .1 M). Place newly constructed electrode into the cell such that metal parts do not contact each other.
3. Place new electrode into cell such that metal parts don’t contact the electrode. Connect potentiostat such that wire from copper-foil wire pair is connected to working electrode, the platinum wire electrode to the counter/auxiliary and the Ag/AgCl to the reference. Make sure that the electrolyte solution does NOT contact metal parts of electrodes. Have to disassemble and rinse cell if this happens!
4. Take LSV data for electrodes with no illumination, starting from just positive of water oxidation potential to just negative of point at which electrode begins to produce cathodic current in light. Several runs might be necessary to get good sense of these bounds.
5. Once current response graph appears, turn LED on, record time at which LED turned on. Observe current response and sample behavior (bubbles). Once there is a current begin experiment on system. Settings and light source/intensity can be altered. For example, turn light on/off a few times, turn knob on LED driver to decrease intensity, change LED sources to longer wavelength.
6. Select bulk electrolysis experiment, set interval time to 10 minutes. Set sampling rate to be ~10 points per second, set 5 sec induction period with .75 V irradiation. Set electrolysis potential to .75 V vs. Ag/AgCl. Measure current as settings change.
7. Obtain .1 M NiNO3 and a solution of NaOH. Dip TiO2 slide in nitrate solution, dip in NaOH solution, and repeat. Repeat approx. 10 times to get good layer of nickel oxide on surface. Repeat linear sweep voltammeter and bulk electrolysis experiments for electrode. Observe differences, as well as whether the slide is more or less efficient for water oxidation.
8. Export all data to Excel. Place graph and data in notebook. Compare responses and record observations. Don’t use any 5x5 cm plates, needed for later lab.