

Supporting Information for

An Easily Fabricated Low-Cost Potentiostat Coupled with User-Friendly Software for Introducing Students to Electrochemical Reactions and Electroanalytical Techniques

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S1. Materials

Disclaimer: we make suggestions in the following sections for possible vendors for purchase of parts to assemble the JUAMI potentiostat. The authors have no financial interest in any of the vendors and are simply providing information for the readers' convenience. There are a number of alternative vendors for all the parts listed.

PCB for Daughter Board Circuit

The bare PCB board for assembling the daughter board can be purchased from several online vendors; some suggested vendors are listed below:

https://www.seeedstudio.com/fusion_pcb.html

<https://www.pcbway.com/>

<https://ecommerce.pcbfabexpress.com/>

Typically, a Gerber file containing the copper layers (.gbr file extension) is the industrial standard for PCB board printing. The Gerber file we used in this project is included in the .zip file in the Supporting Information. Upload the Gerber file to the online vendor of your choice and designate the quantity (usually a minimum of 5 is required) and shipping information.

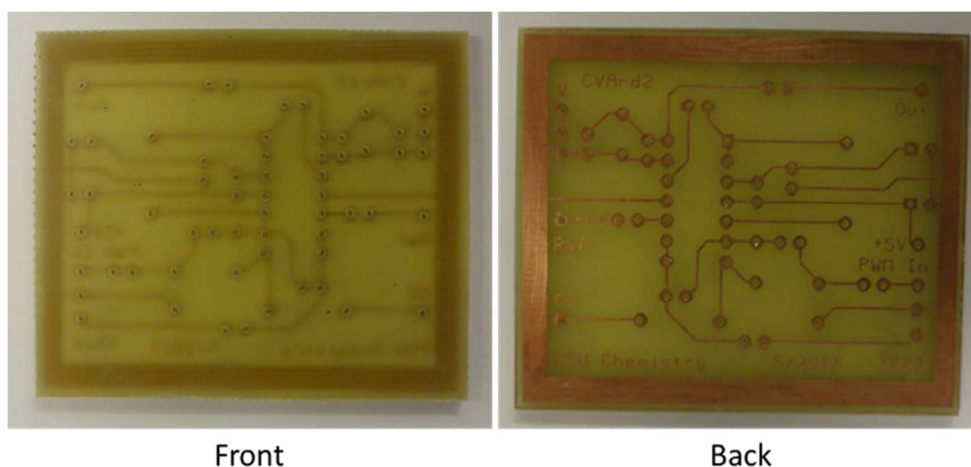


Figure S1. The bare PCB board.

Electronic Components

The electronic components for this project are all readily available. Digikey can provide one-stop shopping for all the components, but they are also available from other vendors as well as from the manufacturers. Detailed information for the components and the Digikey part numbers are provided in Table S1. The components are also labeled according to Table S1 in Figure S4.

Digikey: <https://www.digikey.com/>

Quantity	Components	Value	Digikey Part #
1	Capacitor (C1)	1uF	BC1622-ND
1	Capacitor (C2)	1000pF	BC5256CT-ND
2	Capacitor (C3, C4)	10uF	493-17428-1-ND
1	Capacitor (C5)	100pF	BC5255CT-ND
2	Resistor (R1,R13)	1K	CF14JT1K00CT-ND
4	Resistor (R2,R3,R9,R11)	10K	CF14JT10K0CT-ND
1	Resistor (R4)	4.7K	CF14JT4K70CT-ND
2	Resistor (R5,R6)	100K	CF14JT10K0CT-ND
4	Resistor (R7,R8,R10,R12)	68	CF14JT68R0CT-ND
1	Resistor (jumper)	220	CF14JT220RCT-ND
1	Op amp		LMC6484IMX/NOPBCT-ND

Table S1. List of all the electronics components needed for the daughter board circuit.

3D Printed Casing

The casing for the JUAMI potentiostat can be made with a 3D printer (the authors used a LutzBot printer) or ordered from a number of online vendors. Some suggested vendors are listed below:

<https://www.xometry.com/3d-printing/>

<https://www.makexyz.com/>

The .stl files provided in the .zip file in the Supporting Information contains the CAD drawing required for 3D printing. There are three individual parts to the casing. Upload each .stl file to an online vendor of your choice and specify the quantity and shipping information.



Figure S2. The 3D printed casing from the .stl files. From left to right are the top, middle and bottom pieces, which are assembled together with screws in the corners.

Arduino Uno board

The Arduino Uno circuit board can be purchased directly from the Arduino company, <https://www.arduino.cc/>, or from Amazon, <https://www.amazon.com/>.

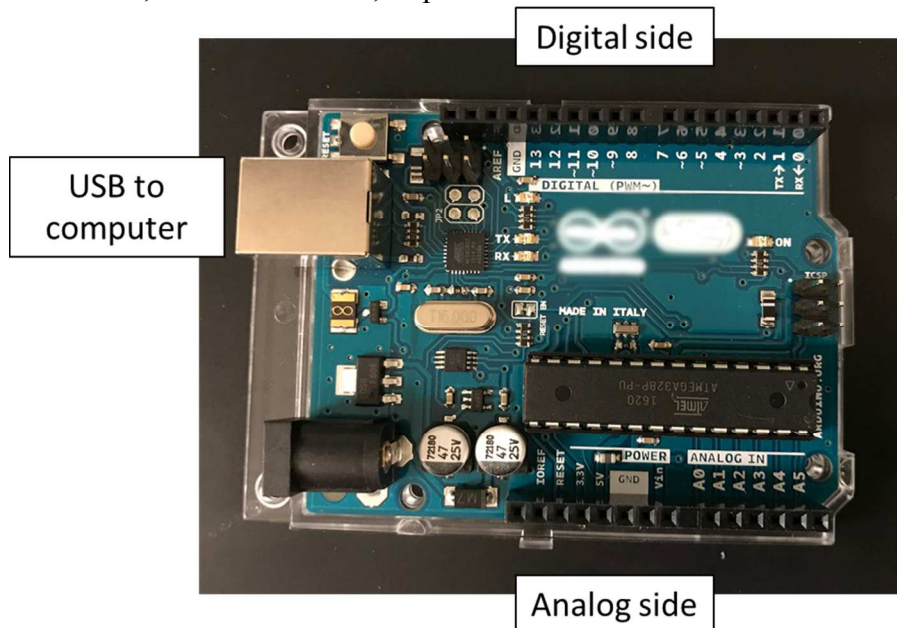


Figure S3. The Arduino Uno circuit board.

Miscellaneous Parts

4 sets of 10-32 screws and nuts, electrical wire and alligator clips are needed. They can be purchased from a number of online stores or local hardware stores. We purchased all the items from McMaster (<https://www.mcmaster.com/>):

- 10-32 screw, 1-5/8" length, McMaster part # 92185A223
- 10-32 nut, McMaster part # 90257A411
- 28-gauge electrical wire, McMaster part # 8054T31
- Alligator clip: McMaster part # 7236K29

S2. Potentiostat Assembly Instructions

Daughter Board Assembly Instructions

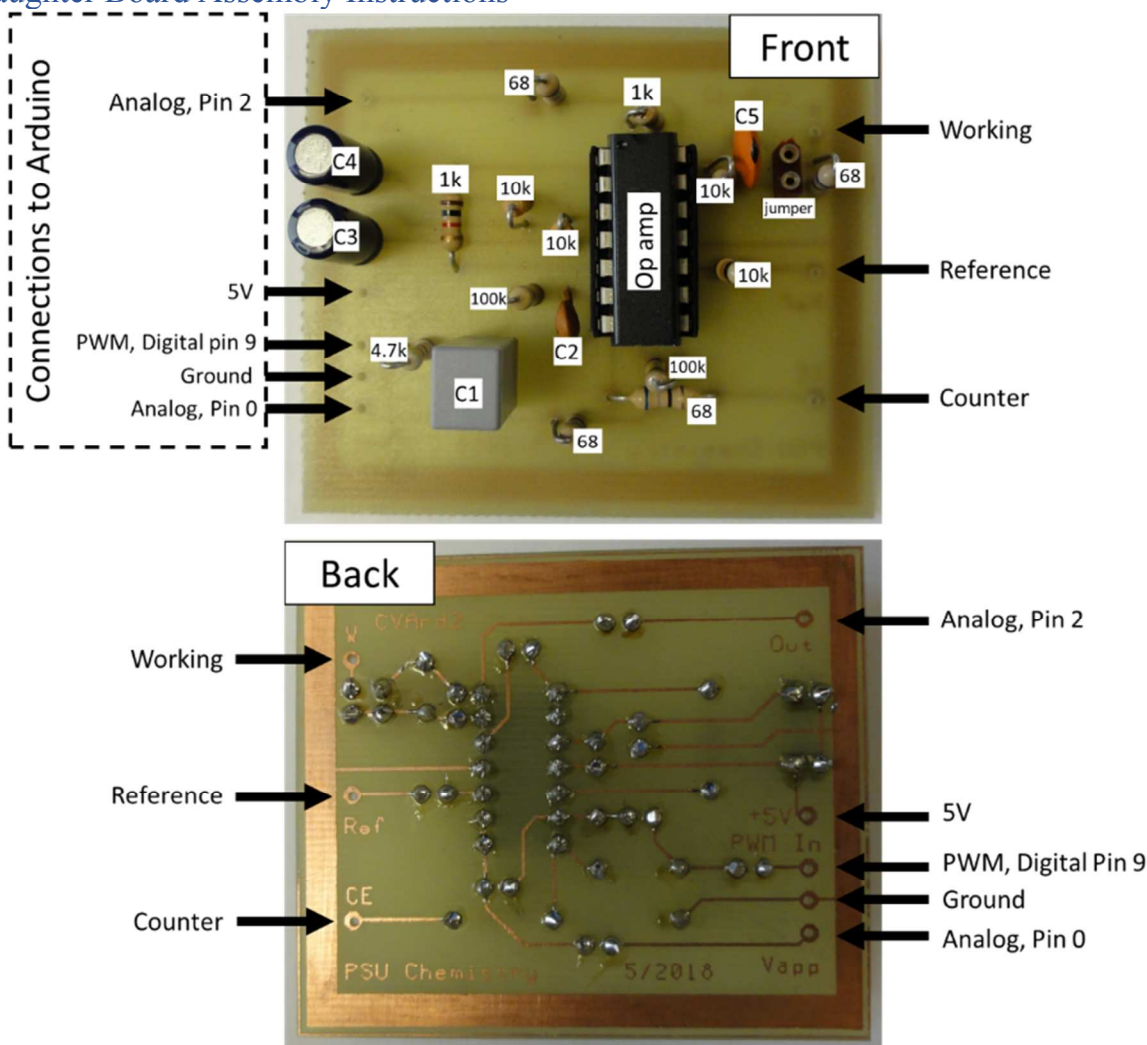


Figure S4. The fully assembled daughter board. The resistors are listed with their values. The capacitors are listed with their labels referenced to Table S1. Inset shows the backside of the daughter board.

The JUAMI potentiostat consists of the main Arduino board and a daughter board. The purpose of the daughter board is to smooth the pulse width modulation (PWM) from the Arduino into an analog potential sweep. The fully assembled circuit is shown in Figure S4. The resistor values are listed in the Figure while the capacitors are labeled according to Table S1.

Solder each of the electronic components onto the bare PCB board. Start with the op amp at the center and work outwards with the electronic components one at a time. The locations of all components are labeled in Figure S4 and pictorial instructions for step by step assembly are provided in Figure S5.

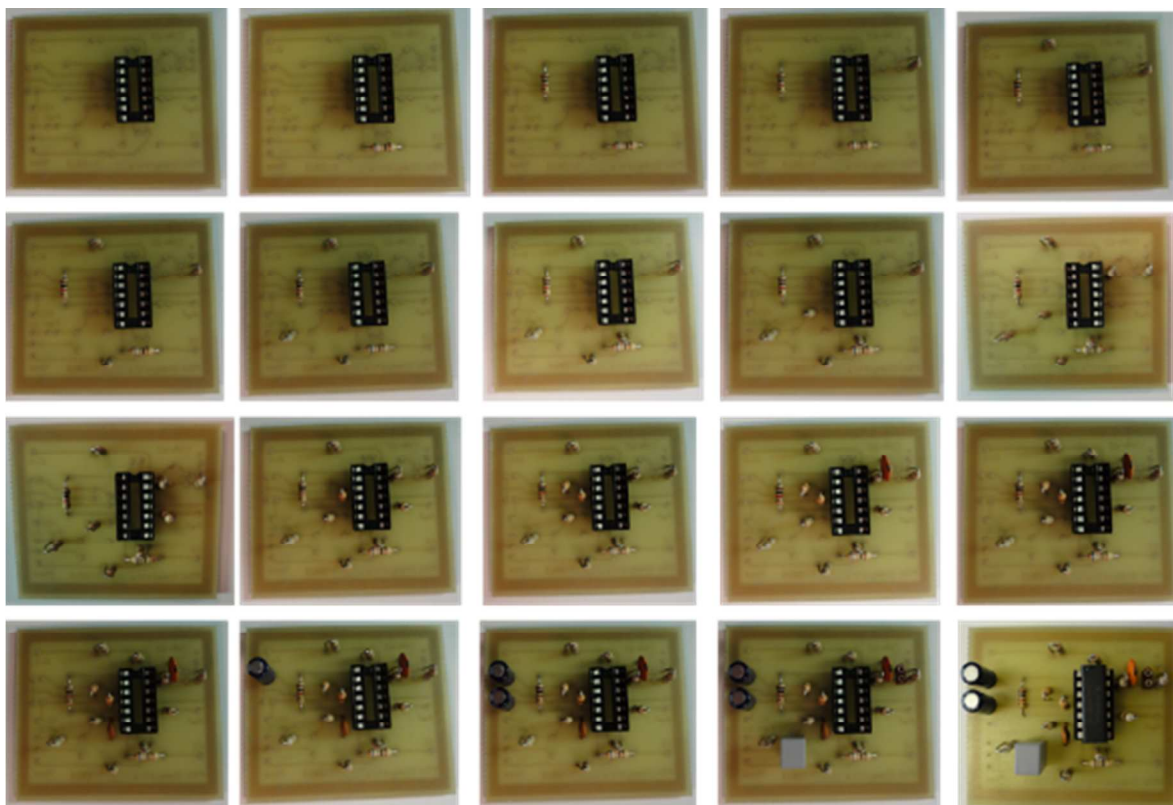


Figure S5. Step-by-step instructions for the daughter board assembly.

The electronic schematic used to generate the Gerber file for the daughter board is provided in Figure S6. The locations of the parts in Figure S6 do not necessarily reflect their physical location. The “jumper for resistor” is an open circuit where the user can insert a resistor. The resistor value will affect the signal amplitude of the working electrode. The maximum current for the JUAMI potentiostat can be adjusted by inserting a different resistor value here. Typically, a higher maximum current will result in lower current resolution, or vice versa. A 220 Ω resistor was used to collect the data reported in the main manuscript.

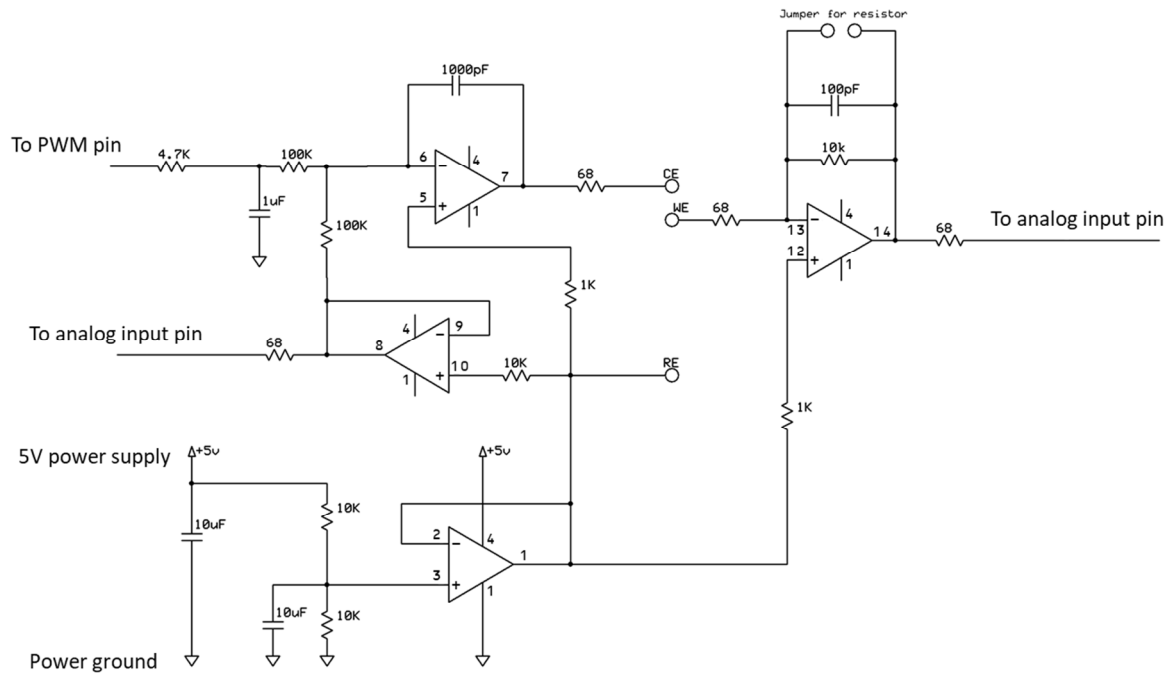


Figure S6. The electronic schematic diagram for the Arduino daughter board. The component parts are listed in Table S1 and the assembly instructions are given in Figure S4 and Figure S5.

Assembly Instructions

Once the daughter board is ready, it can be assembled together with the Arduino Uno to make the JUAMI potentiostat. As shown in Figure S7, the PWM wire on the daughter board connects to digital Pin 9 on the Arduino Uno; +5 V wire on the daughter board connects to the 5 V Pin on the Arduino Uno; The ground wire on the daughter board connects to the GND pin on the Arduino Uno; Analog read 1 and analog read 2 on the daughter board connect to analog Pin 0 and Pin 2 respectively on the Arduino Uno board. The Arduino board is situated in the bottom part of the 3D printed casing and the daughter board is on the middle part. The Top part covers the daughter and finishes the hardware portion of the JUAMI potentiostat, as shown in Figure S8.

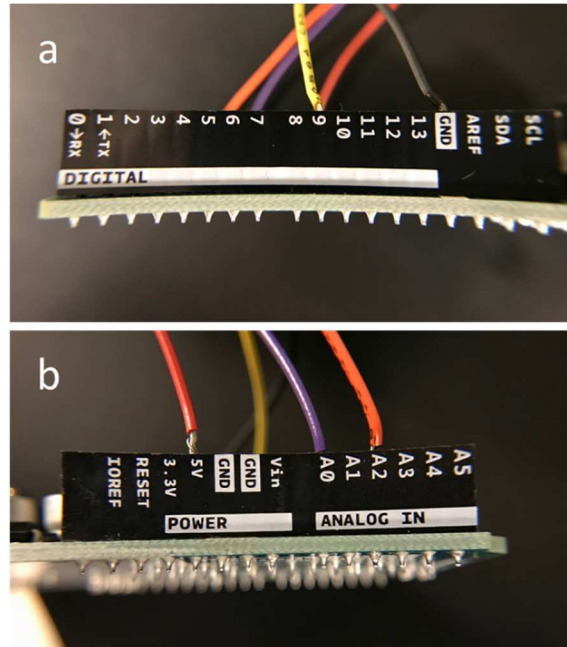


Figure S7. The wiring of the daughter to the Arduino board: a) the digital side and b) the analog side.

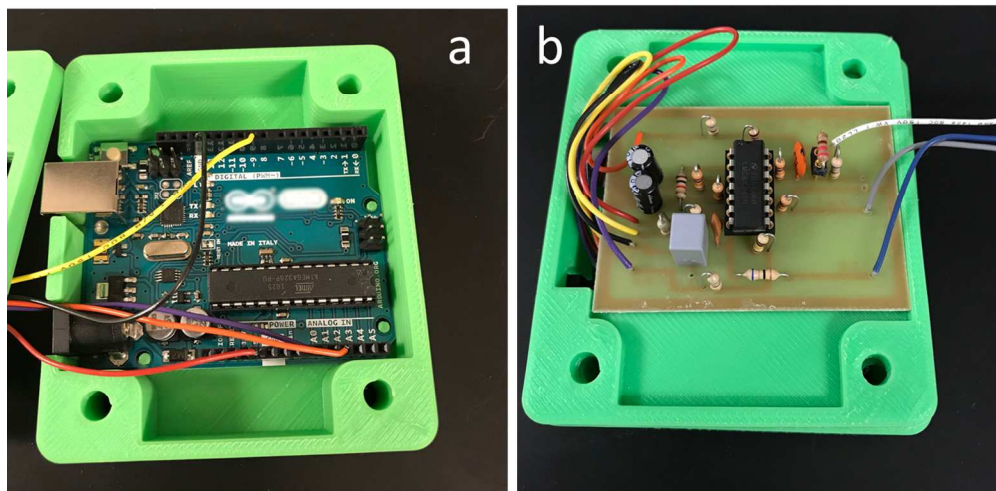


Figure S8. The JUAMI potentiostat in disassembled view: a) the Arduino Uno board and b) the daughter board.

Communications to LabVIEW

Once the JUAMI potentiostat is assembled, it can be connected to a computer via a USB B cable. However, there are a few firmware patches needed to make the JUAMI potentiostat function properly.

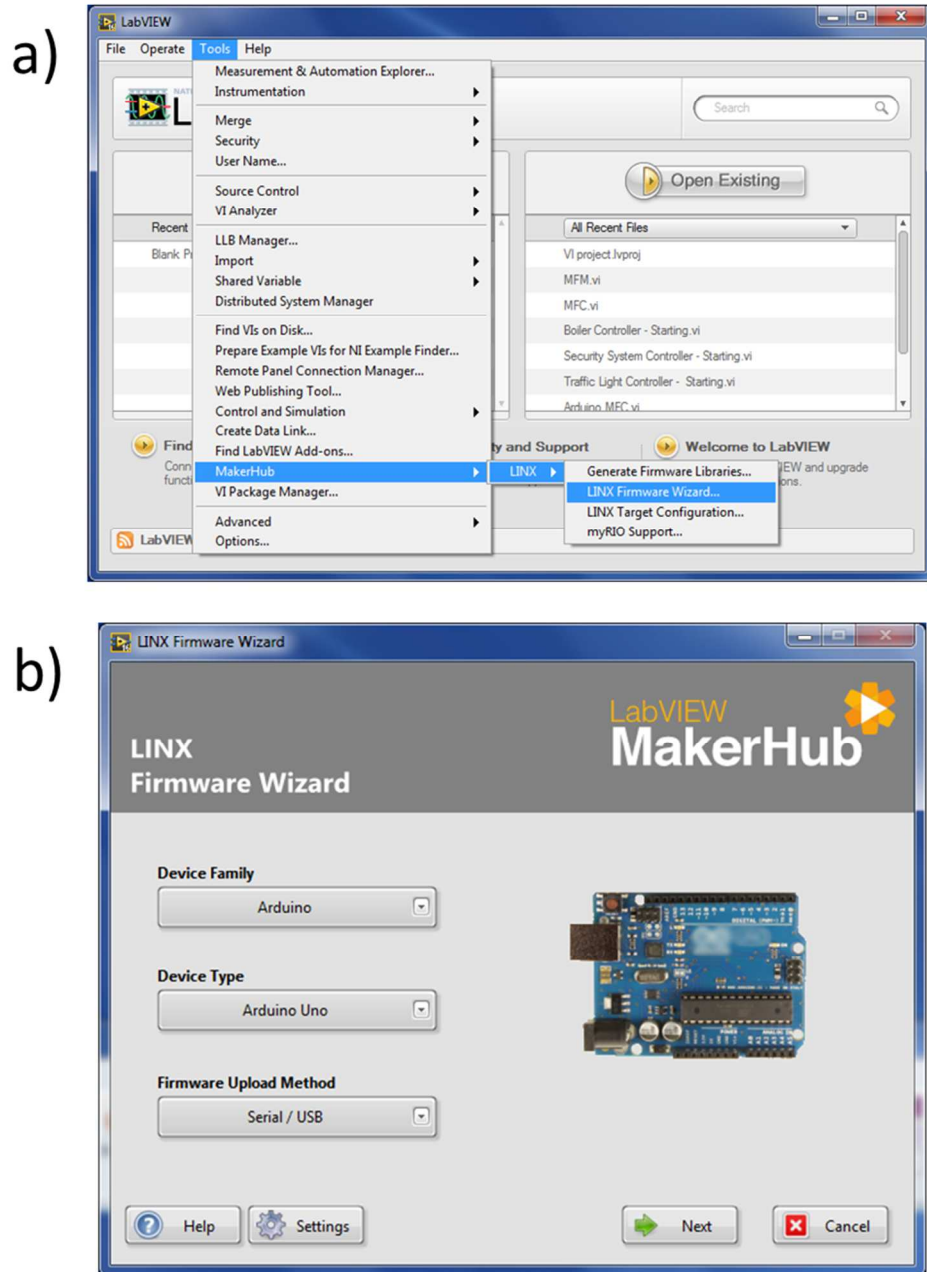


Figure S9. The Linx interface for establishing communication for LabVIEW and an Arduino board.

First, LabVIEW firmware is needed on the Arduino board to enable the software to control the Arduino hardware. General instructions for establishing communications between an Arduino Uno board and LabVIEW are available on the Makerhub website. This part of the firmware update procedure requires LabVIEW, but LabVIEW is not required for the operation of

the JUAMI potentiostat itself. Thus, an evaluation copy of LabVIEW can be used for the firmware update.

Download and install the Linx package for LabVIEW from the website provided below. Connect the fully assembled JUAMI potentiostat to the computer via a USB B cable. Then open LabVIEW as shown in Figure S9a to open the “Firmware Wizard”; Follow the on-screen instructions, shown in Figure S9b, to upload LabVIEW firmware onto the Arduino board.

Makerhub: <https://www.labviewmakerhub.com/>.

Linx package: <https://www.labviewmakerhub.com/doku.php?id=libraries:linx:start>

PWM Frequency Update

The Arduino circuit board generates a voltage ramp via a pulsed width modulation method. The default frequency of the PWM channel on the Arduino Uno board is 490 Hz or 977 Hz, which are too low for application to the potentiostat. Fortunately, the default frequency can be modified with a few lines of simple code, given below. To update the PWM frequency, copy and paste the following code into the Arduino IDE (the native Arduino programming interface, as shown in Figure S10) and hit the “Update” arrow. This will reset the frequency for Pin 9 of the Arduino board to 31 kHz. Without making this modification, the JUAMI potentiostat will still be functional, but the voltage ramp signal will show step-wise features instead of a smooth voltage ramp.

```
#include <SPI.h>
#include <Wire.h>
#include <EEPROM.h>
#include <Servo.h>
#include <LinxArduinoUno.h>
#include <LinxSerialListener.h>
LinxArduinoUno* LinxDevice;

void setup()
{
  LinxDevice = new LinxArduinoUno();

  LinxSerialConnection.Start(LinxDevice, 0);
  TCCR1B = B00000001;
}

void loop()
{
  LinxSerialConnection.CheckForCommands();
}
```

The Arduino IDE is the native programming-based interface for the Arduino family circuit boards. The software is available free of charge from the Arduino website. Copy and

paste the code above into the programming interface and hit “Upload”, as shown in Figure S10. After this final adjustment, the JUAMI potentiostat is fully assembled and ready for action.

Arduino IDE: <https://www.arduino.cc/en/Main/Software>

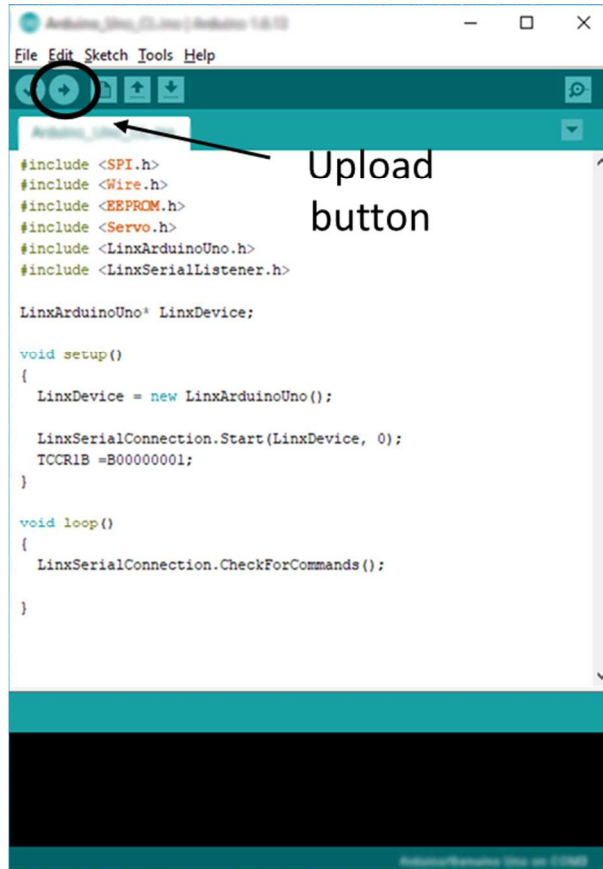


Figure S10. Screen capture of the Arduino IDE programming interface.

S3. JUAMI Software User Interface Instructions

Firmware Installations

1. We are continuously improving the software interface, please check with the authors and see if there is an updated version.
2. The JUAMI software provided in the supporting information is compatible with any Window 7 or 10 computer. Two more files are needed, *NIVISA1700full* and *LVRTE2016_f5Patchstd*. Both files are available, free of charge, from the National Instruments website.
<http://www.ni.com/en-us/shop/labview/download.html>
3. These files install the communication port between the JUAMI potentiostat and the PC computer. Order of installation for these two files does not matter.

Software Operations

1. Download and unzip the .zip file provided in the supporting information, Open the “Software” folder and open the JUAMI.exe file. The software interface should come up, as shown in Figure S11. At this point, connect the JUAMI potentiostat to the computer via a USB and it should be operational. Continue for general comments.

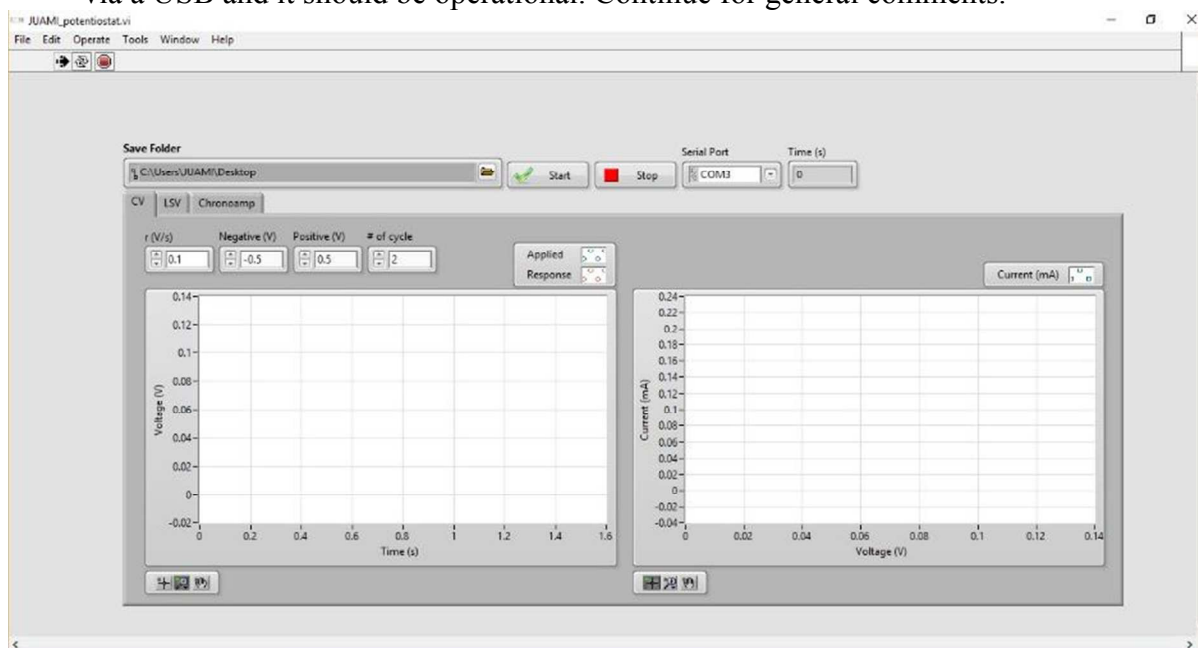


Figure S11. Screen capture of the JUAMI software interface.

2. General comments, as shown in Figure S12:
 - a. On top, the “**Save Folder**” option specify where to save the data file. Click the folder icon to select a fold path on the user’s local computer
 - b. “**Serial Port**” is where the JUAMI potentiostat is plugged into on the computer. It is usually “COM” with a number, such as “COM1”. If there are additional USB connected devices to the same computer, several COM ports may appear in the drop-down menu. Select the COM port designated for the Arduino board by checking with “Device manager” on the computer.

- c. Below on the graphical screen, there are three tabs: “**CV**” – for cyclic voltammetry, “**LSV**”- for linear scanning voltammetry, and “**chronoamp**” – for chronoamperometry. Select the appropriate tab for the desired type of experiment.
- d. Set the parameters in the experiment window and hit “**Start**” to initiate an experiment. “**r (mV/s)**” is the scan rate, “**Negative (V)**” is the negative vortex of the scanning window and “**Positive (V)**” is the positive vortex of the scanning window. A window will pop up after the experiment starts, requesting a file name. Enter a file name and hit ok. The experiment will proceed to start and data will be displayed on the windows on the GUI. There may be a few seconds delay before data collection starts.

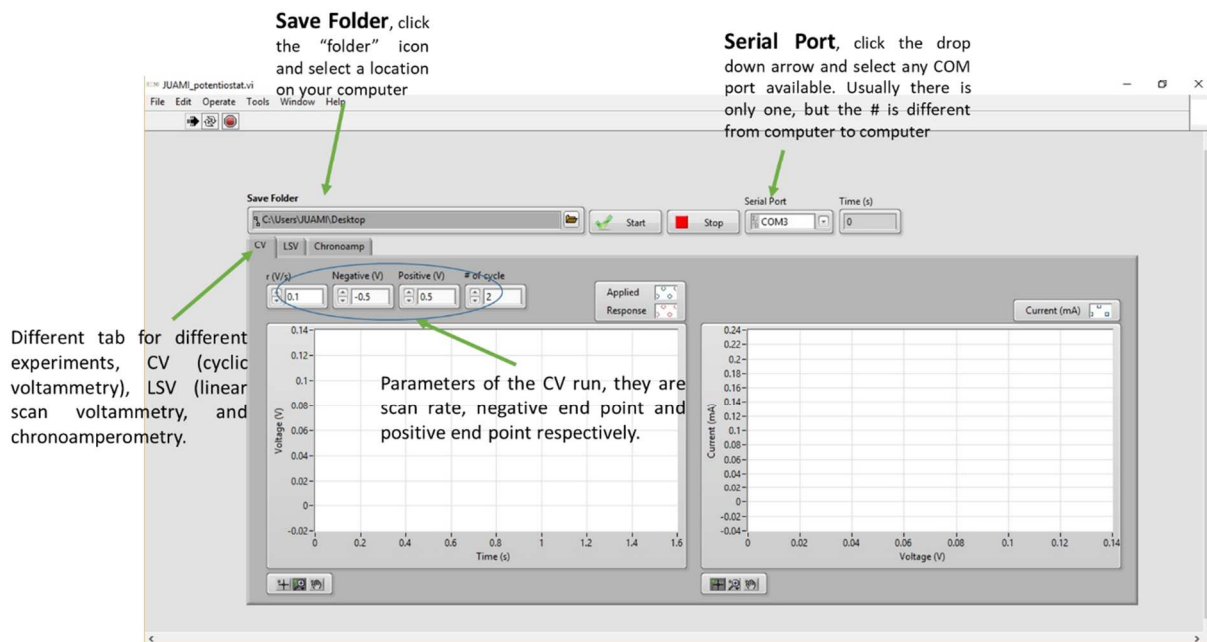


Figure S12. Screen capture of the JUAMI user interface with description for each parameter.

- e. The black arrow circled in Figure S13 (left) below is when the software is ready to run. It defaults back to an idle state, shown in Figure S13 (Right), at the end of every experiment. Click the run arrow again for the next experiment.

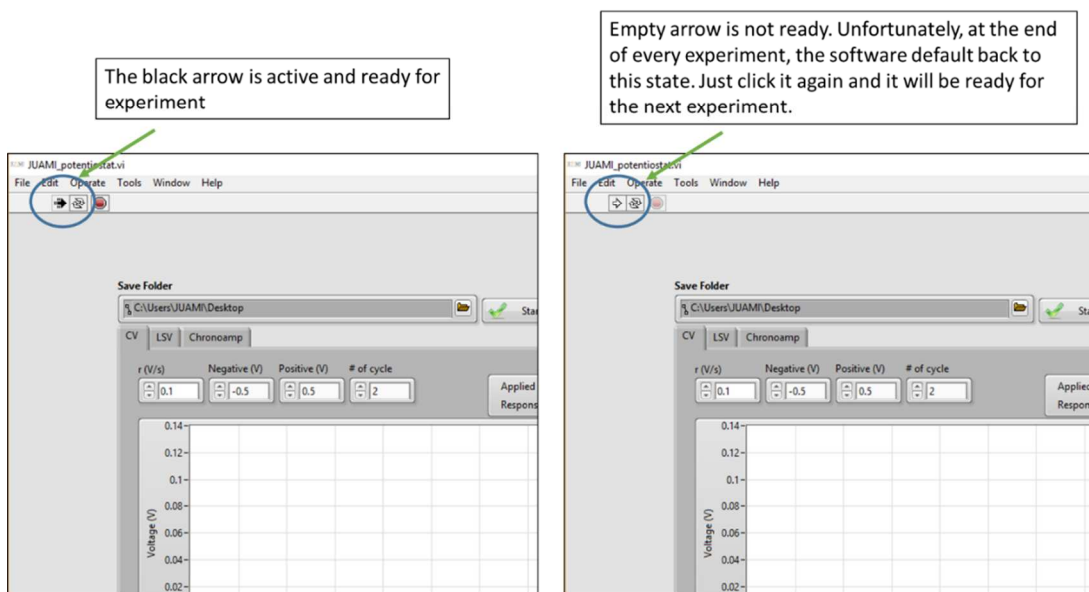
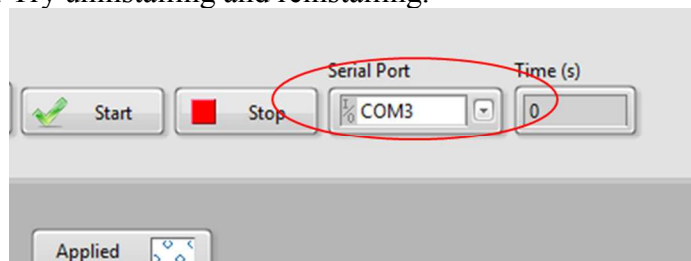


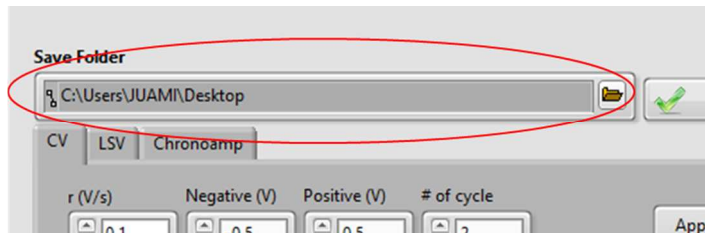
Figure S13. Screen capture of the user interface.

Troubleshooting

1. If the COM port is not selected correctly, the software will indicate an error.
2. If there is no COM port at all to select, there may be problem with the firmware installation. Try uninstalling and reinstalling.



3. Make sure to change the file path for the “Save Folder” points to the local computer, otherwise the software won’t be able to save the file and will also display an error.



S4. Experimental Procedures

Making and Cleaning a Pt Electrode

Materials

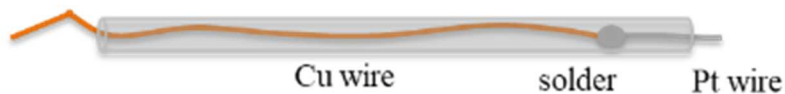
- Pt wire, ~7 cm length, 0.25 – 1 cm diameter
- Electrical wire, ~15 cm length, 26 gauge
- Pt mesh for counter electrode
- Ag/AgCl (sat'd KCl) reference electrode
- 1 M H₂SO₄ acid

Procedures

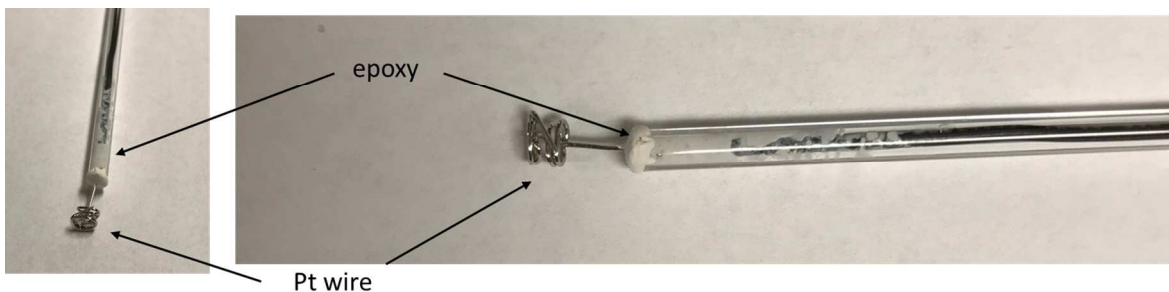
1. Prepare a 1 M H₂SO₄ solution by diluting an appropriate amount of concentrated H₂SO₄ acid in DI water.
2. Coil up one end of the Pt wire and solder the opposite end to the electrical wire using rosin-core solder. Be careful not to get any solder near the working end of the Pt wire.



3. Encapsulate your soldered wire in a section of 3-4 mm OD glass tubing as shown below, so that the working end of Pt is protruding from one end and the electrical wire from the other.



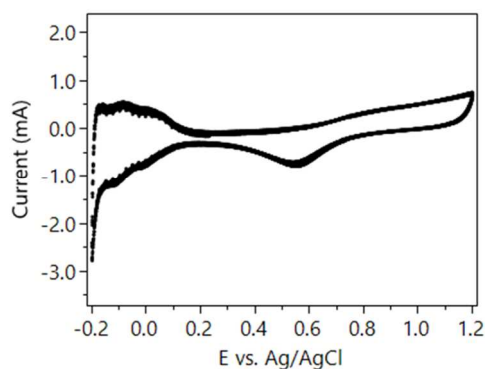
4. Seal the Pt wire completely with epoxy as shown in the picture below, and also push some epoxy into the open end of the glass tube so that it covers the solder joint. Let the epoxy harden for several hours.



5. Soak the Pt electrode in H_2SO_4 acid to clean the surface. Sonicate the Pt end of the electrode in deionized (DI) water afterward and then rinse with DI water and allow to air dry.

6. Connect the Pt electrode to the WE, Pt mesh to the CE and Ag/AgCl to the RE lead of the JUAMI potentiostat. Submerge all the electrodes in the 1 M H_2SO_4 solution. There are two steps for the Pt cleaning. The first step is to hold the working electrode at +2 V vs. Ag/AgCl for 2 min to burn off organic residue. Open the JUAMI software interface and select the “chronoamp” tab. Set the potential to 2V and the “stop time” to 120 seconds. Hit “start” to initiate the experiment.

7. Next, cycle the Pt electrode between -0.2 V and +1.2 V vs. Ag/AgCl at 100 mV/s for ~20 cycles. Select the “CV” tab and set the scan rate at 100 mV/s, number of cycles to 20 and voltage window between -0.2 and 1.2 V. At the end of the process, you should get a CV that looks like the one below.



8. The anodic waves near the positive limit correspond to oxidation of the Pt surface, and the cathodic wave at +0.6 V (vs Ag/AgCl) corresponds to stripping the oxide to regenerate the elemental Pt surface. Initially, this wave will be very large, because you grew a thick oxide at 2.0V, but upon repetitive scanning the CV will start to look like the one above. The cathodic and anodic waves near the negative limit of the scan correspond to adsorbing H atoms onto the Pt surface and stripping them off. A clean Pt surface will show these features and none other. If you have extra peaks in your CV, discard the H_2SO_4 solution and start over with the anodization step in fresh 1 M H_2SO_4 . Once you have a CV that looks good, stop the scan at 1.2 V, remove your electrode from the solution, rinse it with DI water, and allow it to air dry.

Cyclic Voltammetry

Materials

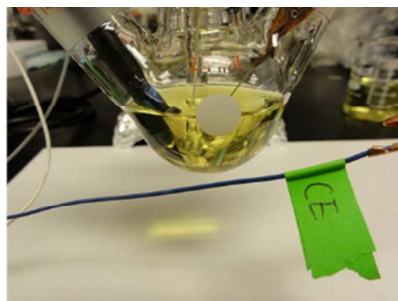
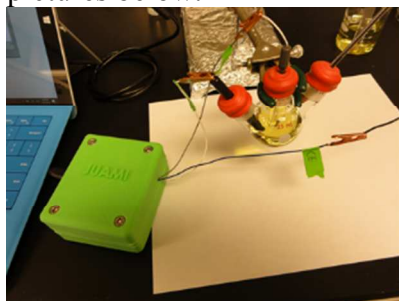
- $\text{K}_3\text{Fe}(\text{CN})_6$
- KCl
- 1 glassy carbon electrode as working electrode
- 1 Pt electrode as counter electrode
- Ag/AgCl reference electrodes

Procedures¹⁻²

1. Prepare a 20 mM $\text{K}_3\text{Fe}(\text{CN})_6$ and 0.5 M of KCl solution by dissolving the appropriate amount of each chemicals in DI water.



2. Rinse all the electrodes thoroughly with DI water before use. Connect the glassy carbon electrode to the WE, Pt electrode to the CE and Ag/AgCl electrode to the RE, as shown in the pictures below.



3. Open the JUAMI software interface and select the "CV" tab. Set the scan rate as 50, 100 or 200 mV/s with voltage vortexes between -0.1 to 0.9 V. Hit "Start" to initiate the experiment. Data is saved to the designated folder specified on the front panel.

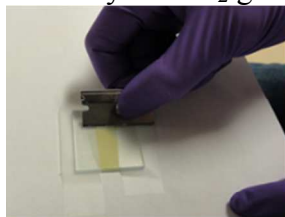
Dye Sensitized Solar Cell

Materials

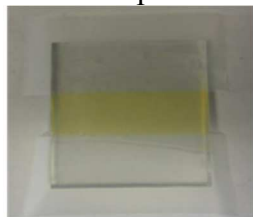
- Electrolyte (Sigma-Aldrich, 791482)
- FTO glass
- TiO₂ paste (Sigma-Aldrich, 791547)
- Sensitizer dye (Sigma-Aldrich, 703206)
- Cathode: Pt coated glass
- Tooth pick
- Small piece of parafilm
- Razor blade
- Pipette
- 2X binder clip
- 2X alligator lead

Procedures³

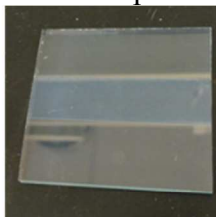
1. Tape down a clean FTO glass with scotch tape with the conductive side facing up and leave about 1 cm gap. Doctor blade the TiO₂ paste onto the FTO glass. The slide is then dried on a hot plate at 300 °C for 20 min, 350 °C for 10 min and 500 °C for 30 min. The TiO₂ anode is sensitized with 100 μM of N3 dye in anhydrous ethanol. After 24 hours, take out the anode and blow dry with N₂ gas. The anode processes at each step is shown in the pictures below.



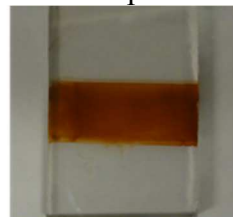
Doctor blade



Unannealed

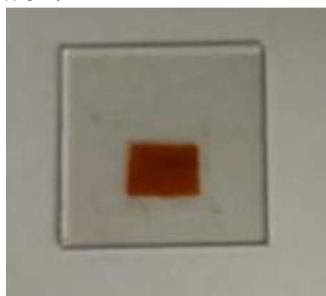


Annealed

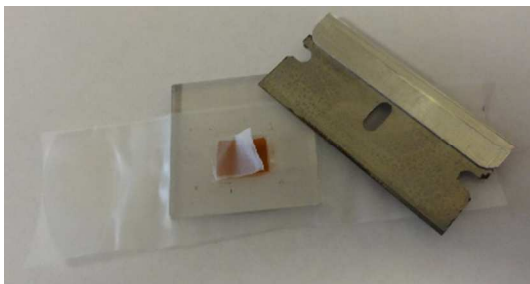
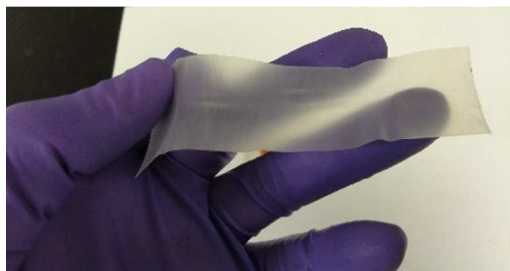


Sensitized, 24hr

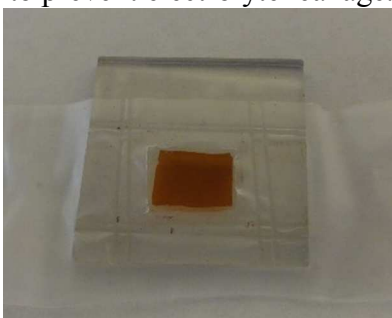
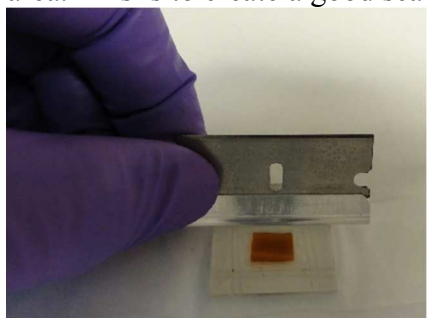
2. Use a tooth pick to scratch off the dye area until an active area of about 0.6X0.8 cm is left, as showed in the picture below. However, as long as the area is known, any other size is fine as well.



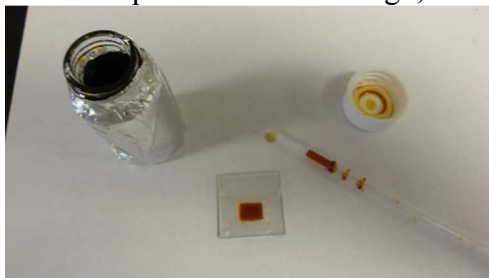
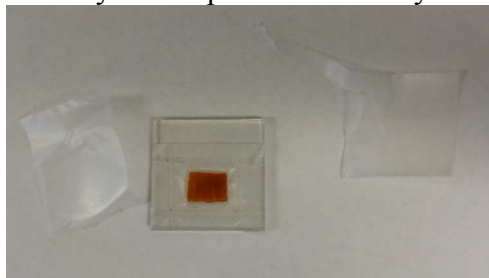
3. Stretch a parafilm a little so that is more flexible. Cover it on the dye area and gently press the surrounding area of the dye for good adhesion. DO NOT press the parafilm on the dye area. Use the razor blade to cut out a hole exposing only the active dye area.



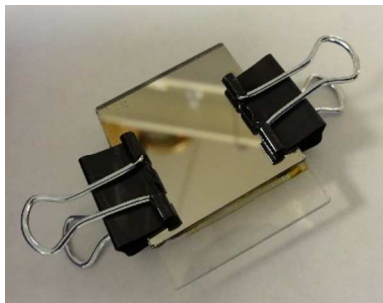
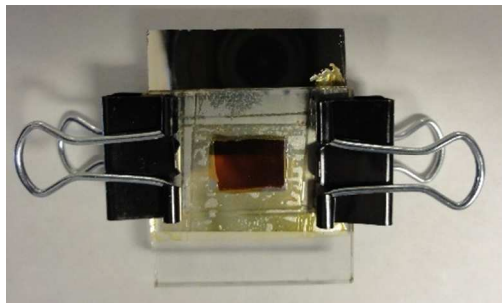
4. Use the back of the razor blade to press several lines on all the edges of the active dye area. This is to create a good seal to prevent electrolyte leakage.



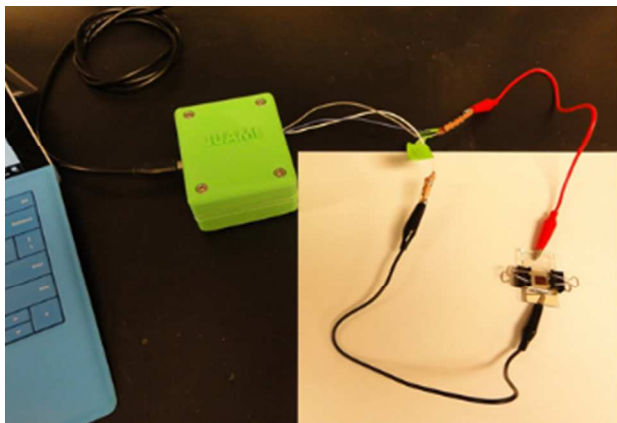
5. Use the razor blade to trim off any excess parafilm. Use a pipette to put one drop of electrolyte on top of the active dye area. One drop is more than enough, don't try to put more.



6. Cover the anode assembly so far with the Pt coated glass, offset the anode/cathode as showed in pictures below to allow room for the alligator clips. Pt side must be facing the anode! The Pt electrode is deposited with an e-beam evaporator in this study. Alternative Pt deposition methods or carbon based cathode electrode are available in the literatures.⁴⁻⁵



7. With alligator clips, connect the Pt electrode to the WE and TiO₂/dye anode to the CE/RE of the JUAMI potentiostat, as shown in the picture below.



8. Place the DSSC in an AM1.5 solar simulator or direct solar irradiation.
9. Open the JAMI software interface and select the “LSV” tab. Set the scan rate as 100 mV/s with voltage vortex between 0 to 0.75 V. Hit “Start” to initiate the experiment. Data is saved to the designated folder specified on the front panel.

Water Oxidation Reaction

Materials

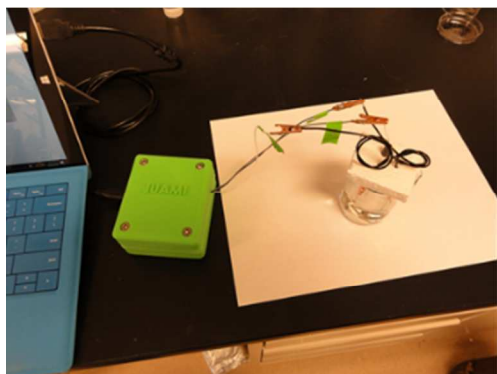
- KOH
- 2 Pt electrodes as both working and counter electrode
- Ag/AgCl reference electrode

Procedures

1. Prepare a 0.5 M KOH solution by dissolving the appropriate amount of KOH in DI water.



2. Rinse all the electrodes thoroughly with DI water before use. Connect the different Pt electrodes to the WE and CE of the JUAMI potentiostat separately. And connects the Ag/AgCl electrode to the RE.



3. Open the JUAMI software interface and select the “CV” tab. Set the scan rate as 100 mV/s with voltage vortex between 0 to 1.5 V. Hit “Start” to initiate the experiment. Visible amount of bubbling should be observed on both the WE and CE. Data is saved to the designated folder specified on the front panel.

Cu Deposition

Materials

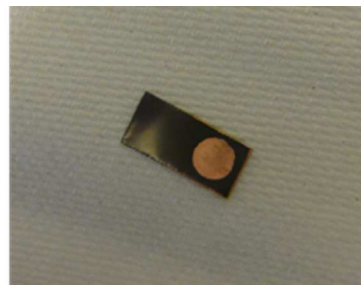
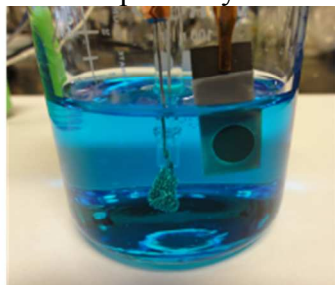
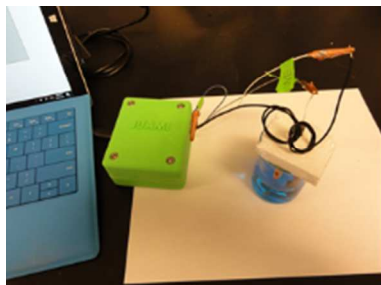
- CuSO_4
- Concentrated H_2SO_4
- A conductive substrate as working electrode (carbon paper or Ti foil)
- 1 Pt electrode as counter electrode
- Ag/AgCl as reference electrode

Procedures

1. Prepare a 0.5 M CuSO_4 and 0.5 M H_2SO_4 solution by dissolving an appropriate amount of CuSO_4 and H_2SO_4 in DI water.



2. Connect the Ti foil to the WE of the JUAMI potentiostat. Pt mesh and Ag/AgCl are used as the counter and reference electrode respectively.



3. Open the JUAMI software interface and select the “Chronoamp” tab. Set the reducing potential at -1 V and the stop time as 1800 s. The deposition voltage and time can be adjusted to modify the copper film thickness and morphology. Hit “Start” to initiate the experiment. Data is saved to the designated folder specified on the front panel.

References

- (1) Kissinger, P. T.; Heineman, W. R. Cyclic Voltammetry. *J. Chem. Educ.* **1983**, *60* (9), 702.
- (2) Elgrishi, N.; Rountree, K. J.; McCarthy, B. D.; Rountree, E. S.; Eisenhart, T. T.; Dempsey, J. L. A Practical Beginner's Guide to Cyclic Voltammetry. *J. Chem. Educ.* **2018**, *95* (2), 197-206.
- (3) McCool, N. S.; Swierk, J. R.; Nemes, C. T.; Saunders, T. P.; Schmuttenmaer, C. A.; Mallouk, T. E. Proton-Induced Trap States, Injection and Recombination Dynamics in Water-Splitting Dye-Sensitized Photoelectrochemical Cells. *ACS Appl. Mater. Inter.* **2016**, *8* (26), 16727-16735.
- (4) Smestad, G. P.; Gratzel, M. Demonstrating Electron Transfer and Nanotechnology: A Natural Dye-Sensitized Nanocrystalline Energy Converter. *J. Chem. Educ.* **1998**, *75* (6), 752.
- (5) Kloke, A.; von Stetten, F.; Zengerle, R.; Kerzenmacher, S. Strategies for the Fabrication of Porous Platinum Electrodes. *Adv. Mater.* **2011**, *23* (43), 4976-5008.