FINAL SCRIPT: APPROVED FOR FILMING

Submission ID #: 68870
Scriptwriter Name: Pallavi Sharma
Project Page Link: https://review.jove.com/files_upload.php?src=21005398

Title: Solar-Driven Electrochemical Green Fuel Production from CO_2 and Water Using $Ti_3C_2T_x$ MXene-Supported CuZn and NiCo Catalysts

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Author Questionnaire

1. **Microscopy**: Does your protocol require the use of a dissecting or stereomicroscope for performing a complex dissection, microinjection technique, or something similar? **NO**

2. **Software:** Does the part of your protocol being filmed include step-by-step descriptions of software usage? **No**

   Videographer: Please film the computer screen/display screen for all shots labelled as SCREEN.

3. **Filming location:** Will the filming need to take place in multiple locations? **Yes**

   0.5 mile

4. **Testimonials (optional):** Would you be open to filming two short testimonial statements **live during your JoVE shoot**? These will **not appear in your JoVE video** but may be used in JoVE's promotional materials. **Yes**

Current Protocol Length
Number of Steps: 27
Number of Shots: 56

Introduction

Videographer: Obtain headshots for all authors available at the filming location.

INTRODUCTION:

1.1. **Mojtaba Abdi-Jalebi:** The scope of our work broadly addresses the synthesis, characterization, and analysis of novel disruptive binder-free catalyst films to produce solar fuels from water and carbon dioxide.

1.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B roll: 5.2.1*.

What are the current experimental challenges?

1.2. **Nusrat Rashid:** The main challenges in the field include low faradaic efficiencies, inadequate production rates, long-term catalyst durability, and scalability of system.

1.2.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

CONCLUSION:

What significant findings have you established in your field?

1.3. **Zahra Albu:** A conformal bi-metallic system lowers overpotentials, performs well at industrial current densities, and has long operating hours.

1.3.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

What research gap are you addressing with your protocol?

1.4. **Shurui Yang:** We are addressing the synthesis of novel, economical, scalable, and binder-free functional bi-metallic anodes and cathodes for carbon dioxide and water reduction.

1.4.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B roll: 2.7.2, 2.7.3*

How will your findings advance research in your field?

1.5. **Mojtaba Abdi Jalebi:** our findings pave the way to new one-step film depositions that can lead to multi-carbon products and green hydrogen generation at low power consumption.

1.5.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

Videographer: Obtain headshots for all authors available at the filming location.

Testimonial Questions (OPTIONAL):

Videographer: Please capture all testimonial shots in a wide-angle format with sufficient headspace, as the final videos will be rendered in a 1:1 aspect ratio. Testimonial statements will be presented live by the authors, sharing their spontaneous perspectives.

NOTE: There is no clapperboard for the testimonials (shots 1.6.1 & 1.7.1) because I didn't have enough time to get my tablet from the meeting room where I filmed the interviews. I was also not too sure how the frame these shots. I didn't fully understand what was wanted when I read the script. Hopefully I've framed it correctly.

How do you think publishing with JoVE will enhance the visibility and impact of your research?

1.6. **Mojtaba Abdi-Jalebi, Associate Professor:** (authors will present their testimonial statements live)

1.6.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

Can you share a specific success story or benefit you've experienced—or expect to experience—after using or publishing with JoVE? (This could include increased collaborations, citations, funding opportunities, streamlined lab procedures, reduced training time, cost savings in the lab, or improved lab productivity.)

1.7. **Mojtaba Abdi-Jalebi, Associate Professor**: (authors will present their testimonial statements live)

1.7.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

Protocol

2. Fabrication of MXene-Supported Electrodes

Demonstrators: Shurui Yang, Nusrat Rashid, Zahra Albu

2.1. To begin, clean the nickel foam using acetone **[1]**. Then, sonicate the nickel foam in deionized water for 5 minutes **[2]**.

2.1.1. WIDE: Talent placing nickel foam into a beaker containing acetone.

2.1.2. Talent placing the cleaned nickel foam into a sonicator filled with deionized water.

2.2. Under the fume hood, soak the carbon fiber paper in 1 molar nitric acid for 20 minutes to activate it while wearing gloves and appropriate personal protective equipment **[1]**.

2.2.1. Talent placing a piece of carbon fiber paper into a beaker containing 1 molar nitric acid under a fume hood.

2.3. Immerse the cleaned nickel foam in MXene *(Mak-Xene)* solution for 5 minutes **[1]**. After drying the sample, label it as MXene-Nickel-Foam **[2]**. NOTE: The VO is edited for the deleted shot.

2.3.1. Talent immersing the nickel foam into a beaker containing MXene solution.

2.3.2. ~~Talent placing the soaked nickel foam inside a vacuum chamber at room temperature~~. NOTE: Not filmed

2.3.3. Talent labeling the dried sample as MXene-Ni-Foam.

2.4. Now, load 50 milliliters of MXene solution into a spray gun **[1]**. ~~Spray the MXene ink from 5 centimeters onto the activated carbon fiber paper, ensuring the entire surface of the 2 centimeter by 2 centimeter piece is covered **[2]**. Label the coated sample as MXene-CFP *(Mak-Xene-C-F-P)* **[3]**.~~ NOTE: The VO is edited for the deleted shot.

2.4.1. Talent filling a spray gun reservoir with 50 milliliters of MXene solution.

2.4.2. ~~Talent holding the spray gun 5 centimeters above the carbon fiber paper and spraying evenly~~. NOTE: 2.4.2 and 2.4.3 are not filmed

~~2.4.3. Talent labels the sample as MXene-CFP.~~

2.5. Next, add copper and zinc precursor solutions into a glass electrochemical cell **[1]**. Insert a silver or silver chloride reference electrode, a platinum counter electrode, and the MXene-carbon fiber paper working electrode into the cell **[2]**. Then, connect each electrode to its respective terminal on the potentiostat **[3-TXT]**.

2.5.1. Talent pouring copper and zinc solutions into a glass electrochemical cell. Videographer'sNOTE: Shots 2.5.1, 2.5.2 & 2.5.3 were combined at first but the procedure by the Author didn't go to plan. The Author told me that shot 2.5.1 went to plan and was captured in this take. We did shots 2.5.2 & 2.5.3 again but in separate takes.

2.5.2. Talent placing Ag/AgCl reference electrode, platinum counter electrode, and MXene-CFP working electrode into the solution. Videographer'sNOTE: Shot 2.5.2 had an error in the extra information section on the board. I forgot to delete the information that showed it being a combined shot. It was a single shot.

2.5.3. Talent connecting each electrode to its corresponding port on the potentiostat. **TXT: Electrolyte bath: 20 mL DI water with 30 mM Cu citrate + 10 mM Zn oxalate**

2.6. Apply the given pulsed current deposition sequence and repeat for 1,000 times to deposit the copper-zinc layer **[1].** ~~Label the final electrode as Copper-zinc at $Ti_3C_2T_x$ MXene *(tie-TAY-nee-um CAR-bide Mak-ZEEN)* on carbon fiber paper **[2]**.~~ NOTE: The VO is edited for the deleted shot

2.6.1. TEXT ON A PLAIN BACKGROUND

-10 mA/cm² for 1 s

0mA/cm² (null pulse) for 0.5 s

+10 mA/cm² for 0.5 s

~~2.6.2. Talent labeling the completed electrode.~~ NOTE: Not filmed

2.7. In a separate electrochemical cell, add nickel and cobalt precursor solutions **[1]**. Use the same electrode configuration but replace the working electrode with the MXene-Nickel-Foam **[2]**. Connect all electrodes to the appropriate inputs on the potentiostat, ensuring the MXene-Ni-Foam is connected as the working electrode **[3]**. Prepare the electrolyte bath with 50 milliliters of 50 millimolar nickel nitrate and cobalt nitrate dissolved in deionized water **[4-TXT]**.

2.7.1. Talent pouring nickel nitrate and cobalt nitrate solutions into a glass electrochemical cell. NOTE: 2.7.1, 2.7.2 and 2.7.4 are filmed in a single shot

2.7.2. Talent placing the MXene-Ni-Foam as the working electrode.

2.7.3. Talent connecting each electrode to the correct port on the potentiostat.

2.7.4. Talent adding electrolyte solution to the cell. **TXT: Repeat 1000 sets to obtain NiCo@$Ti_3C_2T_x$-MXene-Ni-Foam**

2.8. ~~Then, fill a spray gun with 50 milliliters of platinum on carbon solution **[1]**. From 5 centimeters, spray coat the platinum on carbon onto a nickel mesh **[2]**. Dry the coated mesh in a vacuum oven at 60 degrees Celsius to create the reference cathode for water electrolysis **[3]**.~~ NOTE: Not filmed

~~2.8.1. Talent filling the reservoir of a spray gun with 50 milliliters of platinum on carbon solution.~~

~~2.8.2. Talent spraying platinum on carbon evenly over a piece of nickel mesh.~~

~~2.8.3. Talent placing the sprayed nickel mesh into a vacuum oven and setting the temperature to 60 degrees Celsius.~~

3. Electrochemical CO_2 Reduction

3.1. To assemble a hydrogen cell, insert an alkaline exchange membrane to separate the two chambers **[1]**.

3.1.1. Talent inserting an alkaline exchange membrane into the slot.

3.2. Use nickel foam as the anode and a 2-centimeter by 2-centimeter Copper-Zinc at $Ti_3C_2T_x$-MXene on carbon fiber electrode as the cathode **[1]**. Then, fill both chambers with 1 molar potassium hydroxide solution as the electrolyte **[2]**. Check the membrane junction thoroughly for any electrolyte leaks and tighten the junction if needed **[3]**.

3.2.1. Talent placing nickel foam into the anode compartment and the CuZn-MXene-CFP into the cathode compartment. NOTE: Shots 3.2.1, 3.2.2 & 3.2.3 were combined together

3.2.2. Talent pouring 1 molar potassium hydroxide into both chambers.

3.2.3. Talent inspecting the membrane junction closely.

3.3. Now, insert a mercury or mercury oxide reference electrode into the cathode chamber **[1]** and seal the entire system to ensure it is gas tight **[2].** Add one tube for carbon dioxide inlet and another for the gas outlet in the cathodic chamber **[3]**.

3.3.1. Talent inserting a mercury/mercury oxide electrode into the cathode side. Shots 3.3.1, 3.3.2 & 3.3.3 were combined in a single shot

3.3.2. Talent sealing the system.

3.3.3. Talent connecting two flexible tubes to the cathodic chamber—one labeled for

CO_2 inlet and one for gas outlet.

3.4. Then, purge carbon dioxide into the cathodic chamber at a rate of 30 milliliters per minute for 15 minutes to saturate the electrolyte **[1]**.

3.4.1. Talent opens the carbon dioxide gas line and setting the flowmeter to 30 milliliters per minute.

3.5. Illuminate the photovoltaic cell with 1-sun intensity **[1]** and connect it to the cell **[2]**.

3.5.1. Talent illuminating the cell NOTE: Shots 3.5.1, 3.5.2 & 3.7.2 were combined together

3.5.2. Talent connecting the cell.

3.6. Now, record cyclic voltammetry from 0 volts to minus 2.5 volts at a scan rate of 50 millivolts per second and EIS at open circuit potential **[1]**.

3.6.1. Cyclic voltammetry and EIS are being recorded.

3.7. Perform a zero-ampere chrono potentiometric measurement for 2 hours **[1]**. Record the current periodically using a multimeter to calculate the faradaic efficiency of the products **[2]**.

3.7.1. SCREEN: Show the potentiostat interface with the current set to 0 ampere.

Videographer: Please film the computer screen/display screen for all shots labelled as SCREEN.

3.7.2. Talent using a multimeter to manually record current readings at regular intervals from the output terminals.

3.8. Connect the gas outlet of the cathodic chamber to a gas chromatograph for in-line sampling every 10 minutes **[1]**. Program the gas chromatograph to detect and quantify permanent gases using a packed column such as a molecular sieve **[2]**. Set the oven temperature to start at 150 degrees Celsius with a 2-minute hold, then ramp to 200 degrees Celsius with a 1-minute hold for effective separation and elution of gases **[3]**.

3.8.1. Talent attaching tubing from the cathodic chamber to the inlet of a gas chromatograph. NOTE: Shots 3.8.1 and 4.6.1 are the same shot . The Authors told shot 3.8.1 can be reused for shot 4.6.1 in editing

3.8.2. SCREEN: Show the GC software interface displaying the selection of a molecular sieve packed column and settings for gas identification. NOTE: Use take 2

3.8.3. SCREEN: Show the oven temperature programming—initial temperature of 150

degrees Celsius held for 2 minutes, ramped to 200 degrees Celsius with a 1-minute hold.

4. Electrochemical Water Electrolysis (OER)

4.1. Repeat the H-cell assembly using Nickel cobalt at $Ti_3C_2T_x$-MXene-Nickel-Foam as the anode and platinum on carbon-coated nickel mesh as the cathode **[1]**. Insert a mercury or mercury oxide reference electrode into the anodic chamber along with the working electrode **[2]**.

4.1.1. Talent placing NiCo-MXene-Ni-Foam in the anodic chamber and Pt/C-Ni mesh in the cathodic chamber of a new H-cell setup. NOTE: Shots 4.1.1, 4.1.2 & 4.2.1 were combined together

4.1.2. Talent inserting the Hg/HgO electrode into the anodic chamber and adjusting its position next to the working electrode.

4.2. Fill both chambers of the H-cell with 1 molar potassium hydroxide solution **[1]**.

4.2.1. Talent pouring 1 molar potassium hydroxide solution into both the anodic and cathodic chambers using a beaker.

4.3. Record cyclic voltammetry from 0 volts to 1.2 volts at a scan rate of 50 millivolts per second **[1]** and EIS *(E-I-S)* under open circuit conditions **[2]**. Use the OCP *(O-C-P)* determination function on the Autolab potentiostat to record the open circuit potential **[3]**.

4.3.1. SCREEN: Show CV scan parameters being set—range from 0 to 1.2 volts at 50 millivolts per second. NOTE: Shots 4.3.1, 4.3.2 & 4.3.3 were combined together.

4.3.2. SCREEN: Show EIS parameters.

4.3.3. SCREEN: Show selection of OCP determination function on the Autolab software interface and activation of the recording.

4.4. Now, illuminate the photovoltaic cell using a solar simulator positioned 5 centimeters away, set to 1-sun light intensity **[1]**.

4.4.1. Talent aligning the solar simulator 5 centimeters from the PV cell and switching it on. NOTE: Shots 4.4.1 & 4.5.1 were combined together

4.5. Connect the terminals of the photovoltaic cell to the electrodes **[1]** and perform a zero-ampere chrono potentiometric measurement. Monitor and log the current continuously for efficiency calculations **[2]**.

4.5.1. Talent connecting the photovoltaic cell leads to the anode and cathode of the

H-cell. NOTE: Shot 4.5.2 has an error with the take number on the board. This was a technical issue with the app I believe. Rather than do another take, I just amended this during the filming.

4.5.2. SCREEN: Show potentiostat display recording a chrono potentiometric curve at 0 ampere while current readings are logged periodically.

4.6. Now, connect the gas outlet of the cathode chamber to a gas chromatograph **[1]**. Then, analyze hydrogen gas production every 10 minutes using a thermal conductivity detector and nitrogen as the carrier gas **[2]**.

4.6.1. Talent attaching tubing from the cathode outlet to the inlet of a gas chromatograph.

4.6.2. Show the thermal conductivity detection panel.

5. Zero-Gap Electrolyzer Assembly

5.1. After washing the components with water, start assembling a zero-gap electrolyzer cell **[1]**. Prepare clean polyvinyl propylene tubing and attach compatible push and pull valves to create junctions between different parts of the alkaline water electrolysis setup **[2]**.

5.1.1. Shot of all the required components placed on a clean bench.

5.1.2. Talent attaching push and pull valves at junctions to create secure connectors.

5.2. Stack the cell components sequentially, starting with the cell anode plate, Nickel cobalt at $Ti_3C_2T_x$-MXene-Ni-Foam anode, gasket, alkaline exchange membrane, another gasket, Platinum-carbon cathode, and finally the cathode cell plate **[1]**. Use alignment rods if available or firmly position the layers manually on a table to keep them in place **[2]**.

5.2.1. Talent laying down the cell components in sequence.

5.2.2. Talent aligning all components on a flat surface while keeping them stationary by applying even pressure. NOTE: Shots 5.2.2 & 5.3.1 were combined together.

5.3. To assemble the zero-gap electrolyzer, align all layers and end plates properly and secure it using screws **[1]**.

5.3.1. Talent aligning the cell layers and securing them with screws.

5.4. Connect the assembled cell to peristaltic pumps circulating 30 percent potassium

hydroxide at a flow rate of 30 milliliters per minute **[1-TXT]**.

5.4.1. Talent attaching inlet and outlet tubing from the peristaltic pump to the electrolyzer. **TXT: Adjust flow rate as needed for gas removal and performance**

5.5. Maintain the electrolyte reservoir at 60 degrees Celsius using an oil bath **[1]**. Monitor the temperature with a probe and avoid touching the cell or reservoir without thermal gloves **[2]**.

5.5.1. Talent placing the reservoir containing potassium hydroxide into an oil bath set at 60 degrees Celsius. NOTE: Shots 5.5.1 & 5.5.2 were combined together.

5.5.2. Talent inserting a digital temperature probe into the reservoir.

Results

6. Results

6.1. The diffraction pattern of Ti_3AlC_2 MAX *(Ti-tay-nium al-moo-nium caar-bide Mak-zeen)* exhibited characteristic peaks for metal aluminum carbide structure **[1].** After selective etching and delamination, the peaks shifted to lower angles, suggesting the synthesis of $Ti_3C_2T_x$ MXene **[2]**.

6.1.1. LAB MEDIA: Figure 1A. *Video editor: Highlight the top panel showing the (002) peak for Ti_3AlC_2 MAX with a diamond symbol*.

6.1.2. LAB MEDIA: Figure 1A. *Video editor: Highlight the bottom panel showing the shifted (002) peak for $Ti_3C_2T_x$ MXene with a star symbol*.

6.2. The scanning electron microscope image of the Ti_3AlC_2 MAX phase revealed its layered morphology **[1].**

6.2.1. LAB MEDIA: Figure 1B.

6.3. After selective etching and delamination, the scanning electron microscope image of $Ti_3C_2T_x$ MXene deposited on an alumina anodic template exhibited a flake-like morphology consisting of single to few layers **[1].**

6.3.1. LAB MEDIA: Figure 1C.

6.4. X-ray diffraction **[1]** and scanning electron microscope images confirmed the structures of copper zinc at $Ti_3C_2T_x$ MXene at carbon fiber paper and Nickel cobalt at $Ti_3C_2T_x$ MXene at nickel foam **[2]**.

6.4.1. LAB MEDIA: Figure 2A.

6.4.2. LAB MEDIA: Figure 2B.

6.5. Cyclic voltammetry measurements in 1 molar potassium hydroxide showed distinct curves for carbon dioxide reduction by copper-zinc at MXene and carbon fiber paper **[1]** and for water electrolysis by nickel-cobalt at MXene and Nickel-Foam **[2]**

6.5.1. LAB MEDIA: Figure 4A. *Video editor: Highlight the blue CV curve labeled "NiCo@MXene@Ni-Foam" on the upper half of the plot.*

6.5.2. LAB MEDIA: Figure 4A. *Video editor: Highlight the red CV curve labeled "CuZn@MXene@CFP" on the lower half of the plot.*

6.6. EIS revealed different resistance profiles for the cathode copper-zinc at MXene and CFP in carbon dioxide reduction **[1]** and the anode nickel cobalt at MXene and nickel-Foam in water electrolysis **[2].**

6.6.1. LAB MEDIA: Figure 4B. *Video editor: Highlight the red curve labeled "NiCo@MXene@Ni-Foam" with smaller arc.*

6.6.2. LAB MEDIA: Figure 4B. *Video editor: Highlight the blue curve labeled "CuZn@MXene@CFP" with larger arc.*

6.7. The CV profile and chrono amperograms were observed at different applied cell potentials **[1].**

6.7.1. LAB MEDIA: Figure 4C and D.

1. **acetone**
   Pronunciation link: https://www.merriam-webster.com/dictionary/acetone
   IPA: /ˈæsɪˌtoʊn/
   Phonetic Spelling: AS-uh-tone
2. **sonicate / sonication**
   Pronunciation link: https://www.merriam-webster.com/dictionary/sonicate
   IPA (sonicate): /ˈsɑnɪˌkeɪt/
   Phonetic Spelling: SON-ih-kate
3. **deionized**
   Pronunciation link: https://www.howtopronounce.com/deionized
   IPA: /diːˈaɪəˌnaɪzd/
   Phonetic Spelling: dee-EYE-uh-nyzd
4. **nitric**
   Pronunciation link: https://www.merriam-webster.com/dictionary/nitric
   IPA: /ˈnaɪtrɪk/
   Phonetic Spelling: NYE-trik
5. **precursor**
   Pronunciation link: https://www.merriam-webster.com/dictionary/precursor
   IPA: /prəˈkɜːrsər/
   Phonetic Spelling: pruh-KER-sur
6. **potentiostat**
   Pronunciation link: https://www.howtopronounce.com/potentiostat
   IPA: /pəˈtɛnʃiˌoʊstæt/
   Phonetic Spelling: puh-TEN-shee-oh-stat
7. **chrono potentiometric**
   - **chrono**: Pronunciation link: https://www.merriam-webster.com/dictionary/chrono
     IPA: /ˈkrɑnoʊ/
     Phonetic Spelling: KRON-oh
   - **potentiometric**: Pronunciation link: https://www.howtopronounce.com/potentiometric
     IPA: /ˌpoʊtɛnʃiəˈmɛtrɪk/
     Phonetic Spelling: po-TEN-shee-uh-MET-rik
8. **electrolyte**
   Pronunciation link: https://www.merriam-webster.com/dictionary/electrolyte
   IPA: /ɪˈlɛktrəˌlaɪt/
   Phonetic Spelling: ih-LEKtroh-lyt
9. **electrolysis**
   Pronunciation link: https://www.merriam-webster.com/dictionary/electrolysis
   IPA: /ɪˌlɛkˈtrɑləsɪs/
   Phonetic Spelling: ih-lek-TROH-lihsis
10. **MXene**
    Pronunciation link: https://www.howtopronounce.com/mxene

IPA: /ˈɛmˌksiːn/ or /ˈmæksiːn/ (often “max-een”) (howtopronounce.com)
Phonetic Spelling: em-X-een or max-een

11. **peristaltic**
    Pronunciation link: https://dictionary.cambridge.org/us/pronunciation/english/peristaltic
    IPA: /ˌperəˈstɑːl.tɪk/ (U.S.)
    Phonetic Spelling: puh-ruh-STAWL-tik (Cambridge Dictionary)
12. **delamination**
    Pronunciation link: https://www.merriam-webster.com/dictionary/delamination
    IPA: /ˌdɛləˈmeɪʃən/
    Phonetic Spelling: deh-luh-MAY-shun
13. **diffraction**
    Pronunciation link: https://www.merriam-webster.com/dictionary/diffraction
    IPA: /dɪˈfrækʃən/
    Phonetic Spelling: di-FRACK-shun

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