FINAL SCRIPT: APPROVED FOR FILMING

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Title: Synthesis and Performance Evaluations of ZnCoS/ZnCdS with Twin Crystal Structure for Multifunctional Redox Photocatalysis in Energy Applications

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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**

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

   Lab at 3rd floor and Lab at 5th floor within same building

Current Protocol Length
Number of Steps: 18
Number of Shots: 41

Introduction

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

1.1. **Ong Wee Jun:** Our team studies hybrid nanostructures for clean energy and environment, focusing on nanocatalytic mechanisms to enhance efficiency, selectivity, and scalability in solar-to-chemical conversion and environmental remediation via photocatalysis and electrocatalysis.

1.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B roll: Figure 4*

What are the most recent developments in your field of research?

1.2. **Ong Wee Jun:** Recent advances include 2D nanomaterials, carbon-based nanocomposites, metal sulfide-based heterojunctions for the photoredox applications in hydrogen evolution, CO2 conversion, plastic reforming, and organic synthesis, thus boosting solar-to-chemical efficiency and sustainability.

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

What significant findings have you established in your field?

1.3. **Tan Ji Siang:** My work has focused on creating advanced nanocatalysts for artificial photosynthesis and photothermal catalysis, achieving significant improvements in solar-driven hydrogen and syngas production while revealing fundamental surface chemistry and reaction mechanisms that drive sustainable energy conversion.

1.3.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B roll: 2.9*

What advantage does your protocol offer compared to other techniques?

1.4. **Tan Ji Siang:** Our protocol exploits ZnCoS/ZnCdS twin crystal WZ/ZB junctions and ZnCoS electron reservoirs, enabling superior charge separation, visible-light utilization, and dual-function H_2 evolution–benzaldehyde production with significantly enhanced efficiency.

1.4.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B roll: Figure 1*

What research questions will your laboratory focus on in the future?

1.5. **Ong Wee Jun:** My future research focuses on designing advanced photocatalysts and electrocatalysts for CO conversion, methane conversion, nitrate-to-ammonia transformation, hydrogen production and storage, biomass conversion, and plastic upcycling, with the goal of achieving scalable, efficient, and sustainable solar-to-chemical energy and environmental applications of efficiency more than 5%.

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.

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

1.6. **Tan Ji Siang, Assistant Manager of Center of Excellence for NaNo Energy and Catalysis Technology (CONNECT)**: Publishing with JoVE visually showcases our complex methods, improving clarity, reproducibility, and global reach, ultimately boosting the visibility, impact, and adoption of our research across diverse scientific communities.

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. **Ong Wee Jun, Director of Center of Excellence for NaNo Energy and Catalysis Technology (CONNECT)**: By publishing with JoVE, I expect our laboratory to benefit in several tangible ways. The visual format will significantly streamline the onboarding process for new researchers, reducing training time and minimizing costly experimental errors. Clear, step-by-step demonstrations will enhance the consistency in experimental procedures, leading to improved reproducibility and more reliable results. This clarity will also make our methods more accessible to a global audience, fostering new international collaborations and expanding our research network. In turn, the broader visibility can translate into increased citations and stronger recognition within the scientific community, which can open doors to competitive funding opportunities. Ultimately, publishing with JoVE has the potential to accelerate our discoveries, enhance lab productivity, and amplify the overall impact of our work in sustainable energy and catalysis.

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

Protocol

2. Preparation of ZnCoS/ZnCdS Photocatalyst

Demonstrator: Ong Wee Jun

2.1. To begin, place a 100-milliliter beaker on the work surface **[1]** and pour 40 milliliters of ethylene glycol solution into it **[2]**. Using a spatula, add zinc acetate dihydrate, cobalt acetate tetrahydrate, and thioacetamide into the solution **[3-TXT]**. Subject the solution to ultrasonic treatment for 30 minutes **[4]**, then stir it continuously for 4 hours at ambient temperature **[5]**.

2.1.1. WIDE: Talent placing a 100 milliliter beaker on the work surface.

2.1.2. Talent pouring 40 milliliters of ethylene glycol into it.

2.1.3. Talent adding zinc acetate dihydrate into the beaker with labelled bottles of cobalt acetate tetrahydrate, and thioacetamide in the background. **TXT: $Zn(CH_3COO)_2{\cdot}2H_2O$: 0.22g; $Co(CH_3COO)_2{\cdot}4H_2O$: 0.5 g, CH_3CSNH_2: 0.3 g**

2.1.4. Talent placing the beaker into an ultrasonic cleaner and starts it.

2.1.5. Talent placing the solution on a magnetic stirrer.

2.2. Transfer the resulting mixture into a 100-milliliter synthetic polymer-lined stainless-steel autoclave **[1]**. Then, transfer the solution into a preheated oven and heat at 180 degrees Celsius for 12 hours **[2]**.

2.2.1. Talent places the prepared mixture into a stainless-steel autoclave.

2.2.2. Talent places the solution in a laboratory oven set to 180 degrees Celsius.

2.3. Using a centrifuge, collect the dark gray precipitate **[1]**. Then, wash the precipitate three times, each with deionized water and ethanol **[2]**.

2.3.1. Talent shows the precipitate after centrifugation.

2.3.2. Talent washing the precipitate with deionized water and ethanol alternately.

2.4. Dry the washed, dark gray sample in an oven overnight at 60 degrees Celsius to obtain a dark gray zinc-cobalt sulfide powder **[1].**

2.4.1. Talent placing the washed sample into a drying oven set at 60 degrees Celsius.

2.5. To synthesize zinc cadmium sulfide, pour 40 milliliters of deionized water into a 100-milliliter beaker **[1]**. Using a spatula, add zinc acetate dihydrate, cadmium acetate

dihydrate, sodium sulfide hydrate, and thioacetamide into the solution **[2][3]**. Subject the solution to ultrasonic treatment for 30 minutes **[4]**, followed by stirring for 3 hours at ambient temperature **[5]**.

2.5.1. Talent placing a 100 milliliter beaker and pouring 40 milliliters of deionized water into it.

2.5.2. Talent adding zinc acetate dihydrate into the beaker.

2.5.3. TEXT ON A PLAIN BACKGROUND

$Zn(CH_3COO)_2 \cdot 2H_2O$: 0.22g

$Cd(CH_3COO)_2 \cdot 2H_2O$: 0.27g

$Na_2S \cdot xH_2O$: 0.16 g

CH_3CSNH_2: 0.15g

2.5.4. Talent places the beaker into an ultrasonic cleaner.

2.5.5. Talent placing the mixture on a magnetic stirrer.

2.6. Next, add 0.2 molar sodium hydroxide aqueous solution drop-wise into the stirred solution to adjust the pH to 7.0 **[1]**. Transfer the adjusted solution into a 100-milliliter synthetic polymer-lined stainless-steel autoclave **[2]**. Then, place the solution in an oven and heat it at 180 degrees Celsius for 24 hours **[3]**.

2.6.1. Talent using a pipette to add 0.2 molar sodium hydroxide solution drop-wise into the beaker while monitoring the pH.

2.6.2. Talent transferring the adjusted solution into a stainless-steel autoclave.

2.6.3. Talent placing the solution into an oven preheated to 180 degrees Celsius.

2.7. Using a centrifuge, collect the yellowish precipitate **[1],** then wash the precipitate three times, each with deionized water and ethanol **[2]**.

2.7.1. Talent shows the pellet formed after centrifugation.

2.7.2. Talent washing the resulting yellowish precipitate sequentially with deionized water and ethanol.

2.8. Transfer the washed yellowish precipitate into an oven and dry it overnight at 60 degrees Celsius to obtain zinc cadmium sulfide solid powder **[1]**.

2.8.1. Talent placing the washed yellowish precipitate into a drying oven set at 60 degrees Celsius.

2.9. To synthesize the photo catalyst, dissolve 4 milligrams of zinc cobalt sulfide and 0.196 grams of zinc cadmium sulfide into 40 milliliters of deionized water **[1]**. After ultrasonication, collect the yellowish precipitate **[2]**, then wash the sample three times each with deionized water and ethanol **[3]**.

2.9.1. Talent adding zinc cobalt sulfide and zinc cadmium sulfide powders into a beaker containing 40 milliliters of deionized water.

2.9.2. Talent shows the pellet formed after centrifugation.

2.9.3. Talent washing the precipitate alternately with deionized water and ethanol three times.

2.10. Transfer the washed yellowish precipitate into an oven and dry it overnight at 60 degrees Celsius **[1]**. The final product is a yellowish zinc cobalt sulfide and zinc cadmium sulfide solid powder **[2]**.

2.10.1. Talent placing the yellowish precipitate into an oven set at 60 degrees Celsius.

2.10.2. Close-up of the dried yellowish zinc cobalt sulfide/zinc cadmium sulfide powder in a container.

3. Photoredox Dual Reaction of Benzyl Alcohol Oxidation and H_2 Production Measurement

Demonstrator: Tan Ji Siang

3.1. Add 20 milligrams of the synthesized photocatalyst and 60 milliliters of benzyl alcohol aqueous solution into a 100-milliliter beaker **[1]**. Place the beaker in an ultrasonic cleaner and perform ultrasonication for 30 minutes **[2]**.

3.1.1. Talent adding 20 milligrams of photocatalyst and pouring 60 milliliters of benzyl alcohol aqueous solution into a 100 milliliter beaker.

3.1.2. Talent operating the ultrasonic cleaner with the beaker inside for 30 minutes.

3.2. Then, transfer the solution into a three-necked top-irradiation reactor cell **[1]** and insert a magnetic stirrer bar **[2]**. Maintain the solution under slow stirring throughout the entire reaction process **[3]**.

3.2.1. Talent transferring the ultrasonicated solution into a three-necked top-irradiation reactor.

3.2.2. Talent placing a magnetic stir bar inside.

3.2.3. Shot of the solution stirring slowly during the reaction.

3.3. Next, connect a moisture trap to the downstream side of the reactor cell **[1]**. Then

connect the outlet to the gas sampling loop inlet of the gas chromatography system **[2]**. Further, connect the gas sampling loop outlet to the reactor cell inlet to form a closed gas circulation system **[3]**.

3.3.1. Talent attaching a moisture trap to the downstream port of the reactor cell.

3.3.2. Talent connecting tubing from the moisture trap to the gas sampling loop inlet of the gas chromatography system.

3.3.3. Talent connecting the gas sampling loop outlet back to the reactor cell inlet to complete a closed-loop system.

3.4. Seal the reactor with a glass window **[1]**. Then purge nitrogen gas at a flow rate of 50 milliliters per minute through the reactor for 30 minutes to remove all air inside **[2]**.

3.4.1. Talent placing a glass window on top of the reactor and securing the seal.

3.4.2. Talent turning on the nitrogen supply and adjusting the flow rate to 50 milliliters per minute for purging.

3.5. Now, turn on the peristaltic pump and set the flow rate to 20 milliliters per minute to circulate the nitrogen gas within the closed gas circulation system **[1]**.

3.5.1. Talent switching on the peristaltic pump and setting the digital flow controller to 20 milliliters per minute.

3.6. Switch on the Xenon lamp at 15 volts and position it so that the light passes through the glass window and reaches the solution inside the reactor **[1-TXT]**.

3.6.1. Talent switching on the Xenon lamp and adjusting its position in front of the reactor glass window. **TXT: Light intensity: 84.6 mW/cm^2; Distance: 15 cm**

3.7. Once the reaction is complete, using a 0.22-micrometer nylon syringe filter, filter 1 milliliter of the suspension **[1]**. Dilute the filtered suspension with deionized water in a ratio of 1 to 9 **[2]**.

3.7.1. Talent drawing 1 milliliter of the reaction suspension into a syringe and attaching a 0.22 micrometer nylon filter.

3.7.2. Talent diluting the filtered sample in a tube with deionized water at a 1 to 9 volume ratio.

3.8. Finally, use a high-performance liquid chromatography system equipped with a photodiode array detector and a high-performance 100 Angstrom column **[1-TXT]**.

3.8.1. Show the HPLC setup with a photodiode array detector and Poroshell 120 EC-

C18 column installed. **TXT: Mobile phase: H_2O/ACN (60:40); Detection: 254 nm**

Results

4. Results

4.1. The high-resolution transmission electron microscopy images confirmed the coexistence of zinc blende and wurtzite phases in Zinc cadmium sulphide **[1]**, and an interphase boundary clearly distinguished the two crystalline domains **[2]**.

4.1.1. LAB MEDIA: Figure 1A and B. *Video editor: Highlight the labels and lines showing "ZB ZnCdS (111) 0.32 nm" and "WZ ZnCdS (100) 0.36 nm" on the left and right sides of the image.*

4.1.2. LAB MEDIA: Figure 1A and B. *Video editor: Highlight the region marked as "ZB/WZ interphase" in the center of the image*.

4.2. The interface structure of the Zinc cobalt sulphide and Zinc cadmium sulphide heterojunction was distinctly observed **[1],** demonstrating the successful incorporation of Zinc cobalt sulphide on the zinc blende and wurtzite interphases of Zinc cadmium sulphide **[2]**.

4.2.1. LAB MEDIA: Figure 1C.

4.2.2. LAB MEDIA: Figure 1C. *Video editor: Trace or outline the wavy interface separating the ZnCoS and ZnCdS regions*.

4.3. The UV-Visible absorbance spectra showed that Zinc cadmium sulphide had a higher absorbance in the visible region compared to Zinc cobalt sulphide **[1]**, and the Zinc cobalt sulphide and Zinc cadmium sulphide heterojunction exhibited slightly enhanced absorbance over Zinc cadmium sulphide alone **[2]**.

4.3.1. LAB MEDIA: Figure 2A. *Video editor: Highlight the green dashed line labeled ZnCdS that shows a higher drop across 400–600 nm.*

4.3.2. LAB MEDIA: Figure 2A. *Video editor: Highlight the purple dashed line labeled ZnCoS/ZnCdS that follows above ZnCdS in the visible range*.

4.4. The optical bandgap of Zinc cadmium sulphide was calculated to be approximately 2.49 electron volts based on Tauc plot analysis **[1]**.

4.4.1. LAB MEDIA: Figure 2B.

4.5. Nitrogen adsorption-desorption isotherms revealed that the Zinc cobalt sulphide and Zinc cadmium sulphide samples exhibited mesoporous characteristics, with a sharp

increase in adsorption at relative pressure near 1.0 **[1]**.

4.5.1. LAB MEDIA: Figure 3A. *Video editor: Highlight the steep upward curve on the far right of the graph around $P/P_o = 1.0$.*

4.6. The pore size distribution of Zinc cobalt sulphide and Zinc cadmium sulphide was concentrated primarily between 25 and 35 nanometers, confirming the mesoporous nature of the material **[1]**.

4.6.1. LAB MEDIA: Figure 3B. *Video editor: Highlight the two peak regions within the 25–35 nm range of the x-axis*.

Pronunciation Guide:

- Photocatalysis

Pronunciation link: https://www.merriam-webster.com/medical/photocatalysis
IPA: /ˌfoʊtoʊkəˈtæləsɪs/
Phonetic spelling: *FOH-toh-kuh-TAL-uhsis*

- Wurtzite

Pronunciation link: https://www.merriam-webster.com/dictionary/wurtzite Merriam-Webster
IPA: /ˈwɜːrtsaɪt/
Phonetic spelling: *WURT-syt*

- Zinc blende

Pronunciation link: https://www.merriam-webster.com/dictionary/zinc%20blende Merriam-Webster
IPA: /ˈzɪŋk blɛnd/
Phonetic spelling: *ZINK-blend*

- Heterojunction

No confirmed pronunciation link found
IPA: /ˌhɛtərəʊˈdʒʌŋkʃən/
Phonetic spelling: *HEH-tuh-roh-JUNK-shun*

- Photoredox

No confirmed pronunciation link found
IPA: /ˌfoʊtoʊˈriːdɒks/
Phonetic spelling: *FOH-toh-REE-doks*

- Catalyst / Catalytic

Pronunciation link: https://www.merriam-webster.com/dictionary/catalyst
IPA: /ˈkætəlɪst/ (catalyst), /ˌkætəlˈɪtɪk/ (catalytic)
Phonetic spelling: *KAT-uh-list* / *kat-uh-LIT-ik*

- Mechanism

Pronunciation link: https://www.merriam-webster.com/dictionary/mechanism
IPA: /ˈmɛkəˌnɪzəm/
Phonetic spelling: *MEK-uh-niz-um*

- Ultrasonication

No confirmed pronunciation link found
IPA: /ˌʌltrəˌsoʊˈneɪʃən/
Phonetic spelling: *UL-truh-soh-NAY-shun*

- Synthesis

Pronunciation link: https://www.merriam-webster.com/dictionary/synthesis
IPA: /ˈsɪnθəsɪs/
Phonetic spelling: *SIN-thuh-sis*

- Mesoporous

No confirmed pronunciation link found
IPA: /ˌmɛsoʊˈpɔːrəs/
Phonetic spelling: *MEH-soh-POR-us*

- Adsorption / Desorption

Pronunciation link: https://www.merriam-webster.com/dictionary/adsorption
IPA: /ædˈsɔːrpʃən/ (adsorption), /diːˈsɔːrpʃən/ (desorption)
Phonetic spelling: *ad-SORP-shun* / *dee-SORP-shun*

- Photodiode

No confirmed pronunciation link found
IPA: /ˈfoʊtoʊdaɪˌoʊd/
Phonetic spelling: *FOH-toh-DY-ohd*

- Chromatography

Pronunciation link: https://www.merriam-webster.com/dictionary/chromatography
IPA: /ˌkroʊməˈtɑːgrəfi/
Phonetic spelling: *kroh-muh-TOG-ruh-fee*

- Nanostructure

No confirmed pronunciation link found
IPA: /ˈnænoʊˌstrʌktʃər/
Phonetic spelling: *NAN-oh-STRUK-cher*

- Polymorphic / Polymorphous

No confirmed pronunciation link found
IPA: /ˌpɒliˈmɔrfɪk/ or /ˌpɒliˈmɔrfoʊs/
Phonetic spelling: *pol-ee-MOR-fik* / *pol-ee-MOR-fuhs*