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Title: Zinc-Sponge Battery Electrodes that Suppress Dendrites

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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. Interview statements: Considering the COVID-19-imposed mask-wearing and social distancing recommendations, which interview statement filming option is the most appropriate for your group? **Please select one**.

☒ Interviewees wear masks until videographer steps away (≥6 ft/2 m) and begins filming, then the interviewee removes the mask for line delivery only. When take is captured, the interviewee puts the mask back on. Statements can be filmed outside if weather permits.

4. Filming location: Will the filming need to take place in multiple locations? **No**

Current Protocol Length

Number of Steps: 24
Number of Shots: 43

Introduction

1. Introductory Interview Statements

REQUIRED:

1.1. **Brandon J. Hopkins:** This protocol provides an easy, low-cost method of creating a zinc electrode that suppresses dendrites when cycled in an alkaline battery **[1]**.

1.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 2.13., and 3.11.*

1.2. **Brandon J. Hopkins:** The main advantage of this technique is that one can easily tune the structure of the zinc electrode by changing zinc particle size, type of porogen and its size, or heat-treatment protocol **[1]**.

1.2.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 2.4.3., 3.3.2., 2.3.1., 3.3.1., and 3.8.1.*

OPTIONAL:

1.3. **Brandon J. Hopkins:** The metal sponge created using this protocol falls into a relative density and cell size that can be difficult to achieve using standard metal-foam manufacturing methods **[1]**.

1.3.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 4.1.1.*

Protocol

2. An Emulsion-based Method to Create Zn-sponge Electrodes

2.1. Begin by adding 2.054 milliliters of deionized water **[1]** and 4.565 milliliters of decane to a 100-milliliter glass beaker **[2].** Then, dissolve 0.1 gram of SDS by stirring the beaker **[3-TXT]**.

2.1.1. WIDE: Talent holding a glass beaker and adding deionized water to it.

2.1.2. Talent adding decane into the beaker, with the decane container in the shot.

2.1.3. Talent adding SDS and stirring the beaker, with the SDS container in the shot. **TEXT: SDS: Sodium dodecyl sulphate**

2.2. Next, add 5 milligrams of water-soluble medium viscosity CMC sodium salt to the beaker **[1-TXT]** and stir for 5 minutes or until dissolved **[2].** Use plastic or plastic-coated stirring tools because a metallic surface can adversely affect resulting zinc sponges **[3]**.

2.2.1. Talent adding CMC in the beaker. **TEXT: CMC: Carboxymethyl cellulose.**

2.2.2. Talent stirring the beaker to dissolve CMC.

2.2.3. Talent using plastic stirring tools to stir the mixture in beaker

2.3. Add and stir 0.844 grams of water-insoluble preswollen carboxymethyl cellulose resin to the beaker **[1]**.

2.3.1. Talent adding carboxymethyl cellulose resin in the beaker.

2.4. Keep the beaker on an overhead paddle stirrer equipped with a plastic **[1]** and stir the entire mixture at 1000 rpm for 5 minutes **[2]**. Add 50 grams of zinc powder and continue stirring for 5 more minutes **[3]**. *Videographer: This step is difficult and important!*

2.4.1. Talent placing the beaker on an overhead paddle stirrer.

2.4.2. Talent setting the rpm of the stirrer.

2.4.3. Talent adding zinc powder into the beaker.

2.5. After 5 minutes, stop the stirrer and remove the beaker **[1]**. Place the beaker in a desiccator for 5 minutes at room temperature to outgas the mixture and its contents under vacuum **[2]**.

2.5.1. Talent stopping the stirrer and removing the removing the beaker.

2.5.2. Talent placing the beaker in the desiccator.

2.6. Portion the zinc paste into polypropylene molds of approximately 10-millimeter diameter and 5-millimeter height to air dry the paste overnight **[1]**. *Videographer: This step is important!*

2.6.1. Talent portioning the zinc paste in polypropylene molds to air dry.

2.7. After drying, carefully remove the zinc paste from the molds **[1]** and place them into a mesh casing that rests on a notched alumina holder **[2]**. *Videographer: This step is important!*

2.7.1. Talent removing the zinc paste from the molds.

2.7.2. Talent placing the paste on mesh casing.

Videographer's NOTE: Shot 2.7.1 and 2.7.2 were filmed in one shot.

2.8. Place the assembly into a tube furnace with ports to flow gas in and out of the tube **[1]**. Pipe nitrogen gas into the furnace for 30 minutes at a rate of 5.7 centimeters per minute to purge the furnace of air **[2]**.

2.8.1. Talent placing the assembly into a tube furnace.

2.8.2. Talent attaching pipe to furnace ports to purge in and out nitrogen gas.

2.9. After 30 minutes of purging, throttle the nitrogen gas to a constant rate of 2.8 centimeters per minute **[1]**.

2.9.1. Talent changing the rate of nitrogen gas flow.

2.10. Program the temperature of furnace to increase linearly from 20 to 369 degrees Celsius over the course of 68 minutes, hold at 369 degrees Celsius for 5 hours, rise linearly from 369 to 584 degrees Celsius over the course of 105 minutes, and then turn off **[1]**.

2.10.1. Talent programming the temperature of furnace

2.11. Start the furnace program as the nitrogen continues to flow **[1]**. Stop the nitrogen gas flow manually after the 5 hours of temperature hold and pipe in breathing air at 2.8 centimeters per minute **[2]**.

2.11.1. Talent starting the furnace program.

2.11.2. Talent stopping the nitrogen gas flow manually.

2.12. Let the furnace cool to room temperature once the heating program stops while keeping the breathing air flowing **[1]**.

2.12.1. Talent observing the furnace getting cooled at room temperature.

2.13. Finally, remove the cooled zinc sponges **[1]** and either saw or sand them to the desired dimensions **[2]**.

2.13.1. Talent removing the zinc sponges from the furnace.
2.13.2. Talent scraping the sponges as desired.

3. An Aqueous-based Method to Create Zn-sponge Electrodes

3.1. Add 10.5 milliliters of deionized water to a 100-milliliter glass beaker **[1]**.

3.1.1. WIDE: Talent holding a glass beaker and adding deionized water to it.

3.2. Then, add 0.12 grams of water-soluble high-viscosity cellulose gum and stir using a plastic-coated stirring tool **[1]**. Vortex and stir this mixture by hand for 5 minutes or until the cellulose gum is dissolved **[2]**.

3.2.1. Talent adding CMC to the beaker.

3.2.2. Talent vortexing the mixture to dissolve CMC.

3.3. Add 2.4 grams of corn starch while vortexing the beaker for 2 minutes **[1]** and then add 120 grams of prepared zinc powder while vortexing for an additional 2 minutes **[2]**. *Videographer: This step is difficult and important!*

3.3.1. Talent adding corn starch to the beaker.

3.3.2. Talent adding the prepared zinc powder to the vortexing beaker.

3.4. Once done, press the resulting zinc paste into the desired mold cavities **[1]** and leave them to dry overnight at 70 degrees Celsius in open air in a furnace **[2]**. *Videographer: This step is important!*

3.4.1. Talent pressing the zinc paste into the molds.

3.4.2. Talent placing the mold in the furnace.

3.5. After drying, carefully remove the zinc paste from the molds **[1]** and place them into a mesh casing that rests on a notched alumina holder **[2]**. *Videographer: This step is important!*

3.5.1. Talent removing the zinc paste from the molds.

3.5.2. Talent placing the paste on mesh casing.

Videographer's NOTE: Shot 3.5.1 and 3.5.2 were filmed in one shot.

3.6. Place the assembly into a tube furnace with ports to flow gas in and out of the tube **[1]**. Pipe nitrogen gas into the furnace for 30 minutes at a rate of 5.7 centimeters per minute to purge the furnace of air **[2]**.

3.6.1. Talent placing the assembly into a tube furnace.

3.6.2. Talent attaching pipe to furnace ports to purge in and out nitrogen gas.

Videographer's NOTE: Shot 3.6.1 and 3.6.2 were filmed in one shot.

3.7. After 30 minutes of purging, throttle the nitrogen gas to a constant rate of 2.8 centimeters per minute **[1]**.

3.7.1. Talent changing the rate of nitrogen gas flow.

3.8. Program the temperature of the furnace to increase linearly from 20 to 369 degrees Celsius over the course of 68 minutes, hold at 369 degrees Celsius for 5 hours, rise linearly from 369 to 584 degrees Celsius over the course of 105 minutes, and then turn off **[1]**.

3.8.1. Talent programming the temperature of furnace

Videographer's NOTE: For shot 3.8.1, use shot 2.10.1.

3.9. Start the furnace program as the nitrogen continues to flow **[1]**. After 5 hours of temperature hold, stop the nitrogen gas flow manually and pipe in breathing air at 2.8 centimeters per minute **[2]**.

3.9.1. Talent starting the furnace program.

3.9.2. Talent stopping the nitrogen gas flow manually.

3.10. Allow the furnace cool to room temperature once the heating program stops while keeping the breathing air flowing **[1]**.

3.10.1. Talent observing the furnace getting cooled at room temperature.

3.11. Finally, remove the cooled zinc sponges **[1]** and saw or sand them **[2]** to the desired dimensions **[3]**.

3.11.1. Talent removing the zinc sponges from the furnace.
3.11.2. Talent sawing or sanding the sponges as desired.
3.11.3. Finished sponge.

Results

4. **Assessment of Morphological and Functional Characteristics of the Prepared zinc-sponge Electrodes.**

    4.1. This protocol was used to produce emulsion-based zinc sponges with densities of 2.8 grams per cubic centimeter and aqueous-based sponges with densities of 3.3 grams per cubic centimeter **[1]**.

        4.1.1. LAB MEDIA: Figure 1a & 1b

    4.2. The zinc sponges should be rigid and brittle, their cross-sections should look similar to the ones shown here. All properties of the resulting zinc sponges should fall within the ranges provided in the text manuscript **[1].**

        4.2.1. LAB MEDIA: Figure 2a & 2b

    4.3. The zinc-sponge electrode shows a cycling stability at a gravimetric capacity of 328 milliampere hours per gram of zinc oxide, which maps to a 43% depth of discharge **[1]**.

        4.3.1. LAB MEDIA: Figure 2c

    4.4. After extensive electrochemical cycling of the constructed zinc electrodes to estimate their efficiency, no dendrites were observed in scanning electron micrographs of the zinc-sponge electrodes **[1]**.

        4.4.1. LAB MEDIA: Figure 3a &3b

    4.5. The surface of the zinc sponge undergoes restructuring during cycling. The deeper the level of discharge and the greater the cycle life, the greater the amount of restructuring **[1]**.

        4.5.1. LAB MEDIA: Figure 3b

Conclusion

5. Conclusion Interview Statements

5.1. **Brandon J. Hopkins:** When attempting this protocol, remember to stir the mixture with a plastic-coated tool **[1]**.

5.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 2.4.2.*

5.2. **Brandon J. Hopkins:** Similar fabrication processes could be used to create other types of metal foams based on different metals for numerous applications **[1]**.

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