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Title: Preparation of Expanded Chitin Foams and Their Use in the Removal of Aqueous Copper

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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? **N**

2. **Software:** Does the part of your protocol being filmed include step-by-step descriptions of software usage? **N**

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: 13
Number of Shots: 30

Introduction

1. Introductory Interview Statements

REQUIRED:

1.1. **Blaine Berrington**: This protocol allows the novel physical transformation of chitin, a high-volume waste material that is notoriously difficult to work with **[1]**.

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

1.2. **Kris Bosch**: The main advantage of this technique is the simplicity of the processing methodology, as it does not require specialized materials, only the basic equipment commonly found in chemistry laboratories **[1]**.

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

OPTIONAL:

1.3. **Katelyn Alley**: We demonstrate this method with chitin, but the technique can be modified to create expanded versions of other polymers and biopolymers that form gels **[1]**.

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

1.4. **Kris Bosch**: In the simplest version of this technique, performed as demonstrated, patience is important. The gelling rate will change with daily and seasonal changes to humidity **[1]**.

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

Protocol

2. Preparation of Expanded Chitin

2.1. One day before preparing the expanded chitin, dry at least 1.2 grams of chitin flakes for 24 hours in an 80-degree Celsius oven **[1]**.

2.1.1. WIDE: Talent placing flakes into oven

2.2. The next morning, in a fume hood and wearing chemical resistant gloves and goggles **[1]**, add 15 grams of lithium chloride to 285 grams of D-MAc (D-mac) in a 500 milliliter Erlenmeyer flask containing a 50-millimeter PTFE (P-T-F-E) lined magnetic stir bar **[2-TXT]**.

2.2.1. WIDE: Talent at fume hood, wearing gloves and putting on goggles

2.2.2. Talent adding lithium chloride and dimethylacetamide into flask. *Videographer: This step is important!*

2.2.3. Talent placing magnetic bar into the flask. **TEXT: D-MAc: dimethylacetamide**

2.3. Cap the flask with a rubber septum **[1]** and place it on a heating stir plate **[2]**. Place a temperature probe into the mixture through the septum **[3]** and stir the mixture at 400 rotations per minute at 80 degrees Celsius for about 4 hours **[4]**.

2.3.1. Talent placing rubber septum on the flask.

2.3.2. Talent placing flask on heating stir plate.

2.3.3. Talent placing temperature probe into the mixture.

2.3.4. Shot of the flask on the heating stir plate.

2.4. When all of the lithium chloride has dissolved, add 1 gram of the oven-dried chitin flakes to the 5% lithium chloride-D-MAc solution **[1]** and transfer the resulting solution into a 500-milliliter round bottom flask containing a 50-millimeter, PTFE-line magnetic stir bar **[2]**.

2.4.1. Talent adding chitin flakes to lithium chloride solution. *Videographer: This step is important!*

2.4.2. Talent adding solution to flask.

2.5. Place the flask, capped with a rubber septum, on a stirring heat block **[1]**. Pierce the septum with a needle to allow the flask to vent **[2]** and heat the solution at 80 degrees Celsius with stirring at 400 rotations per minute for 24-48 hours **[3]**.

2.5.1. Talent placing the flask on stirring heat block.

2.5.2. Talent piercing the septum.

2.5.3. Shot of solution being stirred

2.6. When all of the chitin has dissolved, allow the resultant chitin sol-gel to cool to room temperature for about 1 hour with stirring **[1]**. Once the solution reaches room temperature, place the flask in an ice bath with continued stirring for approximately 20 minutes until the temperature stabilizes **[2]**.

2.6.1. Shot of the flask rotating on the heat block with heating off. *Videographer: This step is important!*

2.6.2. Talent placing the flask in ice bath.

2.7. To prepare a 100-milliliter slurry of sodium hydride in DMAc, in a fume hood wearing chemical resistant gloves and goggles, wash approximately 1 gram of sodium hydride removed from mineral oil storage three times with 10 milliliters of hexane per wash **[2].**

2.7.1. Talent washing sodium hydride.

2.8. Add 0.82 grams of the washed sodium hydride to a fresh 250 milliliter Erlenmeyer flask containing 100 milliliters of D-MAc and a PTFE-lined magnetic stir bar **[1]** and swirl the mixture to produce a sodium hydride D-MAc slurry **[2]**.

2.8.1. Talent adding sodium hydride to the flask.

2.8.2. Talent swirling the flask.

2.9. To form the chitin gel, add the entire volume of sodium hydride slurry to the cooled sol-gel while vigorously stirring the gel solution **[1]**. Then replace the cap and continue to stir the mixture at 400 rotations per minute until a gel forms **[2]**.

2.9.1. Talent adding sodium hydride slurry to the flask. *Videographer: This step is difficult and important!*

2.9.2. Shot of the solution being stirred.

2.10. After the gel has formed, add 100 milliliters of deionized water to the flask in a fume hood **[1]** and remove the expanded chitin foam from the flask, breaking the foam into pieces if necessary **[2]**. Place the foam in a crystallization dish sufficiently large enough to hold the foam along with 1000 milliliters of deionized water **[3]**.

2.10.1. Talent uncapping the flask and adding water.

2.10.2. Talent removing chitin foam from the flask. *Videographer: This step is important!*

2.10.3. Talent placing foam in a crystallization dish.

2.11. Rinse the isolated gel three times with 500 milliliters of deionized water per wash **[1]** before soaking the gel in 1000 milliliters of deionized water **[2]**, 500 milliliters of methanol **[3],** and 1000 milliliters of fresh deionized water for 24 per immersion **[4].**

2.11.1. Talent rinsing the gel with water. *Videographer: This step is important!*

2.11.2. Gel soaking in water.

2.11.3. Talent adding methanol to gel.

2.11.4. Talent adding water to gel.

2.12. After the last deionized water wash, allow the gel to air dry for 24 to 48 hours **[1]**. The gel can then be dried in the oven for 48 hours at 85 degrees Celsius under ambient air or **[2]** in a lyophilizer at minus 43 degrees Celsius and 0.024 millibars of pressure for 48 hours **[3]**.

2.12.1. Talent removing chitin foam from water/placing foam to dry

2.12.2. Talent placing the gel in an oven

2.12.3. Talent placing the gel in a lyophillizer.

2.13. When a solid chitin foam has formed, use a mortar and pestle to grind the foam into a fine powder **[1].**

2.13.1. Talent grinding the chitin foam in a mortar.

Results

3. Results: Analysis of Crystalline Structure and Cu Adsorption Capacity of Expanded Chitin

3.1. Before drying, the neat chitin flakes exhibit a coarse sand appearance **[1]**. After drying, the expanded chitin morphology resembles a kernel of popped corn regardless of the method **[2]**. Scanning electron micrographs reveal the neat chitin as a compact, dense structure **[3]**, while the expanded chitin resembles crinkled paper **[4]** or wrinkled sheets **[5]**.

3.1.1. LAB MEDIA: Figures 3 A1-C1 *Video Editor: please emphasize A1 image*

3.1.2. LAB MEDIA: Figures 3 A1-C1 *Video Editor: please emphasize B1 and C1 images*

3.1.3. LAB MEDIA: Figures 3 A2-C2 and A3-C3 *Video Editor: please emphasize A2 and A3 images*

3.1.4. LAB MEDIA: Figures 3 A2-C2 and A3-C3 *Video Editor: please emphasize B2 and B3 images*

3.1.5. LAB MEDIA: Figures 3 A2-C2 and A3-C3 *Video Editor: please emphasize C2 and C3 images*

3.2. In X-ray diffraction studies, neat chitin displays a strong peak at 19.3 degrees corresponding to its crystal plane **[1]**, which decreases in intensity after baking **[2]** or lyophilizing, suggesting that drying changes the crystallinity index of the chitin **[3]**.

3.2.1. LAB MEDIA: Figure 4 *Video Editor: please emphasize highest peak of black data line*

3.2.2. LAB MEDIA: Figure 4 *Video Editor: please emphasize highest peak of blue data line*

3.2.3. LAB MEDIA: Figure 4 *Video Editor: please emphasize highest/middle peak of orange data line*

3.3. Measurement of the specific surface area obtained from nitrogen-physisorption isotherms shows the greatest uptake volume for the expanded foams **[1]**, confirming the more open and porous structure of these samples **[2]**.

3.3.1. LAB MEDIA: Figure 5A *Video Editor: please emphasize orange and blue data lines*

3.3.2. LAB MEDIA: Figure 5A

3.4. Despite these changes in morphology, the expansion process does not appear to affect the chemical structure of the chitin, as observed in these representative IR (eye-R) spectrograms **[1]**.

3.4.1. LAB MEDIA: Figure 6 *Video Editor: please sequentially add/emphasize data lines from top to bottom of graph*

3.5. Similar observations are noted after thermogravimetric analysis **[1]**, with the onset of thermal decomposition of all three samples occurring at 260 degrees Celsius **[2]** and the maximum decomposition rate occurring at a higher temperature for chitin flakes due to its more compact morphology **[3]**.

3.5.1. LAB MEDIA: Figure 7 *Video Editor: please emphasize data lines in Figure 7A*

3.5.2. LAB MEDIA: Figure 7 *Video Editor: please emphasize 260 °C data point for all three lines in Figure 7B*

3.5.3. LAB MEDIA: Figure 7 *Video Editor: please emphasize black data line peak in Figure 7B*

3.6. The increase in specific surface area is accompanied by an expected increase in the maximum copper uptake by chitin **[1]**. However, these differences in uptake disappear when the copper uptake is normalized by the surface area **[2]**.

3.6.1. LAB MEDIA: Figures 8A and 8B *Video Editor: please emphasize orange and blue data lines in both graphs*

3.6.2. LAB MEDIA: Table 1 *Video Editor: please emphasize Cu Uptake data row*

Conclusion

4. Conclusion Interview Statements

4.1. **Kris Bosch**: It is important to remember that DMAc and NaH are dangerous chemicals that must be handled carefully. Always work in a fume hood and wear appropriate PPE.

4.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 2.2.1 for "fume hood and wear appropriate PPE".*

4.2. **Katelyn Alley**: The limited N_2-adsorption isotherms we collected can only provide specific surface areas. Full N_2-adsorption isotherms can give porosity information that is critical to determine usefulness in size-selective applications.

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