

Submission ID #: 70585

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Title: Assembly of Porous Monolithic Materials Using a Pickering Emulsion Strategy Stabilized by Metal–Organic Framework (MOF) Particles

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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? **Yes**

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

If **Yes**, how far apart are the locations? Less than 20 feet

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. **No**

Current Protocol Length

Number of Steps: 12

Number of Shots: 26

Interviews

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

INTRODUCTION:

~~REQUIRED: Summarize your research focus and the main questions it aims to answer.~~

- 1.1. **Ervin PROZSA:** Our research focuses on finding an efficient and easy method to shape porous metal-organic frameworks for larger applications.
 - 1.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera

~~Describe the research contexts or systems in which this protocol can be applied.~~

- 1.2. **Ervin PROZSA:** The protocol provides mild conditions that can be applied to various porous materials.
 - 1.2.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera

CONCLUSION:

~~What does this protocol allow researchers to study or measure?~~

- 1.3. **Ervin PROZSA:** This approach allows researchers to shape porous materials directly as monoliths for direct-flow processes.
 - 1.3.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera

~~Describe the most important consideration or challenge when performing this protocol.~~

- 1.4. **Ervin PROZSA:** The most important consideration in this protocol is getting the mass-to-volume ratios and stirrer angle right to form the emulsion.
 - 1.4.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera

~~How can future studies build on this work? Describe the questions or research directions that can be investigated next.~~

- 1.5. **Ervin PROZSA:** Future studies are already underway to replace the polymer in the formulation with silica and produce stronger monoliths.

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.

Protocol

***Videographer:** Please always record an audio slate while filming each shot mentioned in the shotlist.*

2. Synthesis of Metal-Organic Frameworks

Demonstrator: Ervin PROZSA

- 2.1. To begin, gather the required reagents and materials [1]. Dissolve 15 grams of batteries in 150 milliliters of a 1 to 1 solution of ultrapure water and 37 percent hydrochloric acid in a beaker [2].
 - 2.1.1. WIDE: Talent walking into the room with a tray of materials
 - 2.1.2. Talent adding batteries to a beaker containing the prepared ultrapure water and hydrochloric acid solution.
- 2.2. Place the solution under stirring at 60 g for 24 hours at room temperature [1].
 - 2.2.1. Talent placing the beaker under stirring.
- 2.3. Then, filter the solution to remove carbon and plastic residues [1].
 - 2.3.1. Talent filtering the solution and collecting the filtrate.
- 2.4. Dissolve 2 grams of benzene-1,3,5-tricarboxylic acid or BTC (*B-T-C*) in 500 milliliters of dimethylformamide or DMF (*D-M-F*) [1].
 - 2.4.1. Talent adding BTC to the bottle containing DMF.
- 2.5. Place the solution in an ultrasound bath at 37 kilohertz for 30 minutes at room temperature [1].
 - 2.5.1. Talent placing the solution in the ultrasound bath.
- 2.6. Mix the metallic solution and organic solution in a 1 to 2 ratio [1].
 - 2.6.1. Talent combining the metallic solution and organic solution in the specified ratio.
- 2.7. Place the mixture in an ultrasound bath at 37 kilohertz for 30 minutes at room temperature [1]. Then, place the mixture in an oven at 90 degrees Celsius for 24 hours [2].
 - 2.7.1. Talent placing the mixture in the ultrasound bath.
 - 2.7.2. Talent placing the mixture in the oven.
- 2.8. Wash the metal-organic framework or MOF (*Mof*) 3 times with DMF and 3 times with ethanol via Büchner filtration [1].

- 2.8.1. Talent washing the MOF by Büchner filtration using DMF followed by ethanol.
- 2.9. Dry the MOF in an oven at 40 degrees Celsius for 72 hours [1].
 - 2.9.1. Talent placing the MOF in the oven.

3. Preparation of Pickering Emulsions and Polymers

- 3.1. Dissolve 1.1 grams of MIL-96 (*Mil-ninety-six*) aluminum in 10 milliliters of ultrapure water in a glass vial [1].
 - 3.1.1. Talent dissolving MIL-96 aluminum in ultrapure water in a glass vial.
- 3.2. Add 13.35 milliliters of paraffin to the solution [1].
 - 3.2.1. Talent adding paraffin to the solution in the glass vial.
- 3.3. Using a homogenizer with a single rotor, homogenize the mixture at 15,000 g for 2 minutes to form an emulsion [1].
 - 3.3.1. Talent homogenizing the mixture using the single-rotor homogenizer
- 3.4. Obtain the required reagents [1], to prepare the Polymerized High Internal Phase Emulsions or PolyHIPE (*Poly-hipe*) [2]. Using a single-rotor homogenizer, homogenize the solution at 15,000 g [3].
 - 3.4.1. Talent arranging the reagents on the workbench.
 - 3.4.2. TEXT ON A PLAIN BACKGROUND:
 - MIL-96(Al) - 375 milligrams
 - Acrylamide - 375 milligrams
 - Methylene bisacrylamide - 18.75 milligrams
 - KPS - 18.75 milligrams
 - Ultrapure water - 3.02 milliliters
 - 3.4.3. Talent homogenizing the solution using the single-rotor homogenizer.
- 3.5. While stirring, add 0.73 milliliters of Polyvinyl alcohol or PVA (*P-V-A*) [1] and 11.25 milliliters of paraffin at a fixed ratio of 0.75 volume per volume [2].
 - 3.5.1. Talent adding polyvinyl alcohol to the stirred solution.
 - 3.5.2. Talent adding paraffin to the stirred solution.
- 3.6. Maintain homogenization at 15,000 g for 2 minutes to form an emulsion [1]. Then, place the mixture in an oven at 80 degrees Celsius for 24 hours to polymerize [2].
 - 3.6.1. Shot of mixture being homogenized.

- 3.6.2. Talent placing the mixture in the oven.
- 3.7. Using a Soxhlet extractor, wash the PolyHIPE with ethanol for 48 hours to remove the oil, excess polymer precursors, and PVA [1].
- 3.7.1. Talent loading the PolyHIPE into a Soxhlet extractor and adding ethanol for solvent extraction.
- 3.8. Dry the material under vacuum at 100 degrees Celsius [1].
- 3.8.1. Talent placing the material in a vacuum drying system.
- 3.9. Prepare the samples [1]. Perform Brunauer–Emmett–Teller or BET (B-E-T) analysis [2]. For powder X-ray diffraction, use a scan range of 5 to 70 degrees [3]. Set the acquisition time to 0.5 seconds per step [4] and use a wavelength of 1.54 angstroms [5].
- 3.9.1. Talent loading a sample holder with the prepared sample
- 3.9.2. TEXT ON A PLAIN BACKGROUND:
- BET Analysis Parameters:**
- Temperature - 77 K
- Adsorption - 20 cm³/g to 0.10 P/P₀
- Adsorption - 60 cm³/g to 0.90 P/P₀
- Desorption - 40 cm³/g between 0.90–0.30 P/P₀
- 3.9.3. SCREEN: 70585_screenshot_1.png *Video editor: Please highlight “Start angle” and “end angle”*
- 3.9.4. SCREEN: 70585_screenshot_1.png *Video editor: Please highlight “time per step”*
- 3.9.5. SCREEN: 70585_screenshot_2.png
- 3.10. For infrared spectroscopy, use a spectral range of 615 to 4000 reciprocal centimeters [1]. Set the scan time to 1 minute and the number of scans to 4 [2]. Analyze the characterization data obtained from the analyses [3].
- 3.10.1. SCREEN: SCREEN: 70585_screenshot_3.png *Video editor: Please highlight “Range”*
- 3.10.2. SCREEN: SCREEN: 70585_screenshot_4.png *Video editor: Please highlight the “Duration”*
- 3.10.3. SCREEN: SCREEN: 70585_screenshot_5.png

Results

4. Results

- 4.1. Pickering emulsions containing polyvinyl alcohol and polymer precursors produced substantially smaller droplets [1] than emulsions without PVA [2].
 - 4.1.1. LAB MEDIA: Figure 1. *Video editor: Emphasize panel F.*
 - 4.1.2. LAB MEDIA: Figure 1. *Video editor: Emphasize panel C.*
- 4.2. Higher MOF content affected the composite specific surface area [1].
 - 4.2.1. LAB MEDIA: Figure 2. *Video editor: Emphasize the blue curve labelled MIL-96 with the value "655 m²/g" on the right side and emphasize the black curve labelled M25/10/0.5 with the value "15 m²/g" on the right side.*
- 4.3. Samples containing 0.5 weight percent PVA exhibited higher specific surface areas [1] than samples without PVA [2].
 - 4.3.1. LAB MEDIA: Figure 2. *Video editor: Emphasize the red curve labelled "M100/10/0.5" and the red value "91 m²/g".*
 - 4.3.2. LAB MEDIA: Figure 2. *Video editor: Emphasize the green curve labelled "M100/10/0" and the green value "51 m²/g".*
- 4.4. Incorporation into the polymer matrix reduced the specific surface area [1] compared with pristine MIL-96 [2].
 - 4.4.1. LAB MEDIA: Figure 2. *Video editor: Emphasize red, green, and black curves and their values on the right side.*
 - 4.4.2. LAB MEDIA: Figure 2. *Video editor: Emphasize the blue "MIL-96" curve and the value "655 m²/g" on the right side.*
- 4.5. PVA-containing monoliths displayed unobstructed flow through interconnected pores [1].
 - 4.5.1. LAB MEDIA: Figure 3. *Video editor: Emphasize panel D.*
- 4.6. MOF particles were deposited on the pore surfaces of the polymer structure [1].
 - 4.6.1. LAB MEDIA: Figure 3. *Video editor: Emphasize panel C.*
- 4.7. Modification of the MOF particles altered their position at the paraffin–water interface [1].
 - 4.7.1. LAB MEDIA: Figure 4. *Video editor: Emphasize the yellow arrows and the label MOF layer in the right and left images.*
- 4.8. Controlled adsorption of fluorinated molecules [1] reduced embedding of MOF

particles within the polymer wall [2].

4.8.1. LAB MEDIA: Figure 4. *Video editor: Emphasize the purple crystal structure at the top center and the black chemical structure below that.*

4.8.2. LAB MEDIA: Figure 4. *Video editor: Emphasize the three orange circles inside the grey blocks at the bottom center.*

4.9. Polymerization and paraffin removal yielded a hierarchically structured monolith integrating highly accessible MOF particles [1].

4.9.1. LAB MEDIA: Figure 4. *Video editor: Emphasize the glass vial containing the white emulsion in the top-left, grey arrows, and the right white column.*

1. Ultrapure
Pronunciation link: <https://www.merriam-webster.com/dictionary/ultrapure>
IPA: /ˌʌl.trəˈpjʊr/
Phonetic Spelling: ul·truh·pyoor
2. Hydrochloric
Pronunciation link: <https://www.merriam-webster.com/dictionary/hydrochloric>
IPA: /ˌhaɪ.droʊˈklɔːr.ɪk/
Phonetic Spelling: hy·droh·klor·ik
3. Benzene-1,3,5-tricarboxylic
Pronunciation link: <https://www.howtopronounce.com/benzene-1-3-5-tricarboxylic-acid>
IPA: /ˈben.ziːn ˌtraɪ.kɑːr.bɑːkˈsɪl.ɪk/
Phonetic Spelling: ben·zeen try·kar·bok·sil·ik
4. Dimethylformamide
Pronunciation link: <https://www.merriam-webster.com/dictionary/dimethylformamide>
IPA: /daɪˌmeθ.əl.fɔːrˈmæm.ɪd/
Phonetic Spelling: dye·meth·uhl·for·mam·id
5. Ultrasound
Pronunciation link: <https://www.merriam-webster.com/dictionary/ultrasound>
IPA: /ˌʌl.trəˈsaʊnd/
Phonetic Spelling: ul·truh·sownd
6. Metal-organic framework
Pronunciation link: <https://www.howtopronounce.com/metal-organic-framework>
IPA: /ˌmeɪ.əl ɔːrˈɡæn.ɪk ˈfreɪm.wɜːk/
Phonetic Spelling: met·uhl or·gan·ik fraym·werk
7. Büchner
Pronunciation link: <https://www.howtopronounce.com/büchner>
IPA: /ˈbyk.nə/
Phonetic Spelling: byook·ner
8. Pickering
Pronunciation link: <https://www.howtopronounce.com/pickering>

- IPA: /'pɪk.ər.ɪŋ/
Phonetic Spelling: pik·uh·ring
9. Homogenizer
Pronunciation link: <https://www.merriam-webster.com/dictionary/homogenizer>
IPA: /hə'mɑː.dʒə.naɪ.zər/
Phonetic Spelling: huh·mah·juh·ny·zer
10. Emulsion
Pronunciation link: <https://www.merriam-webster.com/dictionary/emulsion>
IPA: /ɪ'mʌl.ʃən/
Phonetic Spelling: ih·mul·shuhn
11. Acrylamide
Pronunciation link: <https://www.merriam-webster.com/dictionary/acrylamide>
IPA: /ə'krɪl.ə.mɑɪd/
Phonetic Spelling: uh·kril·uh·myde
12. Methylene bisacrylamide
Pronunciation link: <https://www.howtopronounce.com/methylene-bisacrylamide>
IPA: /'meθ.ə.liːn baɪs.ə'krɪl.ə.mɑɪd/
Phonetic Spelling: meth·uh·leen bys·uh·kril·uh·myde
13. Polyvinyl
Pronunciation link: <https://www.merriam-webster.com/dictionary/polyvinyl>
IPA: /pɑː.li'vaɪ.nəl/
Phonetic Spelling: pol·ee·vye·nul
14. Soxhlet
Pronunciation link: <https://www.howtopronounce.com/soxhlet>
IPA: /'zɑːks.lət/
Phonetic Spelling: zoks·luht
15. Brunauer–Emmett–Teller
Pronunciation link: <https://www.howtopronounce.com/brunauer-emmett-teller>
IPA: /'bruː.nəʊ.ə 'em.ət 'tel.ə/
Phonetic Spelling: broo·now·er em·uht tel·er
16. Diffraction
Pronunciation link: <https://www.merriam-webster.com/dictionary/diffraction>
IPA: /dɪ'fræk.ʃən/
Phonetic Spelling: dih·frak·shuhn
17. Spectroscopy
Pronunciation link: <https://www.merriam-webster.com/dictionary/spectroscopy>
IPA: /spek'trɑː.skə.pi/
Phonetic Spelling: spek·trah·skuh·pee
18. Fluorinated
Pronunciation link: <https://www.merriam-webster.com/dictionary/fluorinate>
IPA: /'flɒr.ə.neɪ.tɪd/
Phonetic Spelling: floor·uh·nay·tid
19. Hierarchically
Pronunciation link: <https://www.merriam-webster.com/dictionary/hierarchical>

IPA: /ˌhaɪ.əˈɑːr.kɪ.kəl.i/

Phonetic Spelling: hy·uh·ar·ki·kuh·lee

20. Monolith

Pronunciation link: <https://www.merriam-webster.com/dictionary/monolith>

IPA: /ˈmɑː.nəˌlɪθ/

Phonetic Spelling: mon·uh·lith