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Title: In Situ Photo-Rheology Monitors Viscoelastic Changes in Photo-Responsive Polymer Networks

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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, all done**

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

   If **Yes**, how far apart are the locations? N/A

Current Protocol Length
Number of Steps: 21
Number of Shots: 39 (14 SC)

Introduction

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

1.1. **Danielle Mai:** We engineer polymers to advance materials for human health and planetary health. We investigate how molecular design impacts material properties, including mechanical strength, stimuli-responsive behavior, and recyclability **[1]**.

1.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 2.3.1*

What technologies are currently used to advance research in your field?

1.2. **Michael Burroughs:** In polymer engineering, mechanical tests are used to understand material strength and durability. Integrating mechanical tests with chemical or optical measurements allows us to study molecular features underlying these properties **[1]**.

1.2.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 3.1.2*

What research gap are you addressing with your protocol?

1.3. **Eleanor Quirk:** We hope to reduce the barrier to entry for researchers who want to use photo-rheology. We describe how we designed our instrument and how others can design their own system **[1]**.

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

What advantage does your protocol offer compared to other techniques?

1.4. **Michael Burroughs:** In situ photo-rheology measures a material's mechanical response during photo-stimulation. This approach allows us to track materials as they are formed or as they change in response to light **[1]**.

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

What new scientific questions have your results paved the way for?

1.5. **Eleanor Quirk:** Our studies shed light on the design of photo-responsive polymer networks, which invite new questions about tuning their mechanical properties. For example, how might we formulate recyclable networks **[1]**?

1.5.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll:5.3.1*

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

Protocol

2. Setting up the Rheometer

Demonstrator: Eleanor Quirk

2.1. To begin, install the upper Peltier plate and geometry on the rheometer **[1]**. Next, install the lower optics plate to enable uniform irradiation of samples during photo-rheology measurements **[2]**. Use an optics plate that is transparent to the wavelengths of interest to allow light transmission through the plate **[3]**.

2.1.1. WIDE: Talent showing the rheometer installed with peltier plate.

2.1.2. Talent placing the lower optics plate onto the setup area.

2.1.3. Close-up of the transparent lower plate installed.

2.2. Wipe the lower optics plate clean and dry **[1]**. Then, center the light source under the transparent plate **[2]**, and secure it using posts, holders, or other appropriate supports **[3]**.

2.2.1. Talent wiping the optics plate with a lint-free cloth.

2.2.2. Talent aligning the light source beneath the plate. Note: 2.2.2 and 2.2.3 are shot together as 2.2.2

2.2.3. Talent securing the light source using support structures.

2.3. Now, place a photodiode power sensor connected to an optical power meter on top of the transparent plate, with the sensor facing downward toward the light source **[1]**. If necessary, take measures to prevent exposure to light **[2]**. Then, turn on the light **[3]** and measure the light intensity using the power meter **[4-TXT]**.

2.3.1. Talent positioning the photodiode sensor on the plate.

2.3.2. Talent placing UV-blocking sheets to block the light. Note: Mislabeled as 2.3.1. Please match the shot action.

2.3.3. Talent activating the light source.

2.3.4. Shot of the optical power meter showing the readings. **TXT: Repeat the intensity measurement periodically as the light source ages**

2.4. If required, adjust the light intensity using a light source driver **[1]**. Mark the positions of the light source supports to allow reproducible placement for future experiments **[2-**

TXT].

2.4.1. Talent rotating a knob or adjusting settings on the light source driver.

2.4.2. Talent marking the positions of light source support structures with a marker or tape.

3. Loading the Sample onto the Rheometer

3.1. Estimate the required sample volume using the formula for the volume of a cylinder and dispense slightly more to avoid under-filling **[1-TXT]**. For a low-viscosity liquid sample such as 100 milligrams per milliliter of PEG *(peg)*-anthracene in water, pipette 75 microliters onto the center of the optics plate **[2]**.

3.1.1. Talent calculating and writing in a notebook. **TXT: Determine a suitable gap height that allows uniform UV irradiation**

3.1.2. Talent pipetting the liquid sample onto the center of the transparent optics plate.

3.2. Next, lower the rheometer head until the geometry contacts the sample, then pause to avoid forming bubbles **[1]**.

3.2.1. Talent lowering the geometry slowly as it makes contact with the liquid.

3.3. View the sample through the optics plate to check for bubble formation **[1]**. If bubbles are visible, raise the geometry to break the connection between the plates **[2]**, then gently rotate and lower the geometry again to reestablish contact **[3]**.

3.3.1. Shot of the view through the optics plate.

3.3.2. Talent raising the geometry slightly. Note: 3.3.2 and 3.3.3 are shot together

3.3.3. Talent gently rotating the geometry and re-lowering it to contact the sample. NOTE: Added extra optional shot: 3.3.2-A and 3.3.3-A (with break in liquid)

3.4. Then, gradually lower the geometry until reaching the final experimental gap height **[1-TXT]**.

3.4.1. Talent lowering the head in small steps while monitoring the sample. **TXT: Continue observing for bubble formation throughout the process**

3.5. Now, gently rotate the geometry to homogenize the liquid sample **[1]**.

3.5.1. Talent manually rotating the top geometry part of the rheometer.

3.6. To prevent evaporation over short timescales, soak lint-free wipes in water **[1-TXT]** and place them near the sample to create a high-humidity environment around the sample **[2-TXT]**.

3.6.1. Talent soaking a lint-free wipe in water. **TXT: Minimize evaporation during the experiment** Note: 3.6.1 and 3.6.2 are shot together

3.6.2. Talent positioning the moist lint-free wipe close to the sample on the stage. **TXT: Ensure the wipes do not touch the sample or the geometry**

3.7. For longer measurements, seal the sample with an immiscible layer such as mineral oil to isolate it from the ambient environment **[1-TXT]**.

3.7.1. Talent carefully pipetting mineral oil around the perimeter of the aqueous sample. **TXT: Mineral oil prevents evaporation during longer experiments**

3.8. Close the upper Peltier plate jacket to protect the sample from ambient light and temperature changes **[1]**. Then, add additional shielding like UV-blocking sheets as needed to protect from harmful stray irradiation **[2]**.

3.8.1. Talent closing the jacket around the rheometer's upper plate.

3.8.2. Talent placing UV-blocking panels around the instrument.

3.9. To determine an appropriate strain for dynamic experiments, perform preliminary strain amplitude sweeps on the sample to identify the linear viscoelastic region **[1]**. Sweep the strain from 1 percent to 1000 percent at a constant frequency of 10 radians per second before and after UVA-induced network formation **[2]**. Use a 10 percent strain for this sample to ensure linear viscoelasticity both before and after network formation **[3]**.

3.9.1. SCREEN: 68394_3.10.1.-3.10.2._t1_precrosslink.mp4 00:18-00:22.

3.9.2. SCREEN: 68394_3.10.1.-3.10.2._t3_precrosslink.mp4 03:35-03:55. *Video editor: Highlight the sample title "pre-crosslink"*

3.9.3. SCREEN: 68394_3.10.1.-3.10.3._t1_postcrosslink.mp403:10-03:32. *Video editor: Highlight the sample title "post-crosslink"*

4. Conducting a Photo-Rheology Experiment

4.1. Set the sample temperature to 22 degrees Celsius **[1]**. Pre-shear the sample for 10 seconds, then allow it to equilibrate for another 60 seconds **[2]**.

4.1.1. SCREEN: 68394_4.1.1._4.1.2._4.2.1._4.3.1._4.6.1._t1.mp4 00:05-00:10 *Video editor: Highlight the temperature value of 22 degrees Celsius*

4.1.2. SCREEN: 68394_4.1.1._4.1.2._4.2.1._4.3.1._4.6.1._t1.mp4 00:05-00:10 *Video editor: Highlight the "equilibration box" covering the duration 60 seconds*

4.2. Perform a frequency sweep before irradiation, ranging from 100 radians per second to 0.1 radians per second **[1]**. Use the sweep results to confirm the initial physical state of the sample **[2]**.

4.2.1. SCREEN: 68394_4.1.1._4.1.2._4.2.1._4.3.1._4.6.1._t1.mp4 00:15-00:20 *Video editor: Highlight the "angular frequency" line showing 100 to 0.1 rad/s*.

4.2.2. SCREEN: 68394_4.1.1-4.6.2._t1.mp4 03:53-04:03.

4.3. Set an oscillation time sweep to span the irradiation process, including buffer periods before and after light exposure **[1]**.

4.3.1. SCREEN: 68394_4.1.1._4.1.2._4.2.1._4.3.1._4.6.1._t1.mp4 00:25-00:40

4.4. Take measurements for 60 seconds prior to UVA irradiation, continue for 1 hour during irradiation, and measure for 10 seconds after irradiation **[1]**. Use 10 radians per second frequency and 10 percent strain amplitude throughout the sweep **[2]**.

4.4.1. SCREEN: 68394_4.1.1-4.6.2._t1.mp4 00:10:45-00:11:05 *Video editor: Highlight the 60 seconds tick mark on the x-axis (step time)*

4.4.2. SCREEN: 68394_4.1.1-4.6.2._t1.mp4 *Video editor: Highlight the "angular frequency" line showing 10 rad/s on the right panel*.

4.5. Confirm that the sample temperature is stable during irradiation despite the energy input **[1]**.

4.5.1. SCREEN: 68394_4.1.1-4.6.2._t1.mp4 00:47:38-00:47:47. *Video editor: Highlight the rows "temperature showing 22 degrees" and "set temperature showing 22 degrees celsius" on the* ***right panel***

4.6. Finally, perform a second frequency sweep after irradiation, using the same frequency range as before **[1]**, to compare post-irradiation viscoelastic properties with the initial measurements **[2]**.

4.6.1. SCREEN: 68394_4.1.1-4.6.2._t1.mp4 01:11:00-01:11:10.

4.6.2. SCREEN: 68394_4.1.1-4.6.2._t1.mp4 01:11:10-01:11:20 *(Video Editor: Keep this continuous with 4.6.1)*

Results

5. Results

5.1. Before irradiation, the amplitude sweep revealed a strain-independent region across the full range of 1% to 1000%, indicating a broad linear viscoelastic region **[1]**. After 1 hour of irradiation, the linear viscoelastic region extended only to 100% strain, beyond which a yielding event occurred **[2]**.

5.1.1. LAB MEDIA: Figure 6. *Video editor: Focus on the left graph labeled "Before crosslinking"*

5.1.2. LAB MEDIA: Figure 6. *Video editor: Focus on the right graph labeled "After crosslinking"*

5.2. Before irradiation, the frequency sweep showed a slight frequency dependence, consistent with a viscoelastic liquid **[1]**. After 1 hour of irradiation, the moduli showed minimal dependence on frequency with elastic moduli exceeding viscous moduli, confirming the material's solid-like behavior **[2]**.

5.2.1. LAB MEDIA: Figure 5. *Video editor: Highlight the left graph labeled "Before crosslinking"*

5.2.2. LAB MEDIA: Figure 5. *Video editor: Highlight the left graph labeled "After crosslinking"*

5.3. Increasing the polymer concentration from 20 to 100 milligrams per milliliter led to faster cross-linking, as indicated by an earlier crossover between the elastic and viscous moduli **[1]**. This change also resulted in stiffer networks, reflected by higher plateau values of the elastic modulus **[2]**.

5.3.1. LAB MEDIA: Figure 7. *Video editor: Sequentially show red, green and blue line curves* .

5.3.2. LAB MEDIA: Figure 7. *Video editor: Highlight the blue curve*.

Pronunciation guide:

1. Rheometer

Link: https://www.merriam-webster.com/dictionary/rheometer
oed.com+13definitions.net+13howtopronounce.com+13
IPA: /ˈriːəˌmɛtər/
Phonetic: REE-ə-meh-tər

2. Peltier

Link: https://www.merriam-webster.com/dictionary/Peltier%20effect
IPA: /ˈpɛltiər/
Phonetic: PEL-tee-er

3. Photodiode

Link: https://www.merriam-webster.com/dictionary/photodiode
IPA: /ˌfoʊˈtoʊˌdaɪ.oʊd/
Phonetic: foh-TOH-die-ohd

4. Photometer (related term)

Link: https://www.merriam-webster.com/dictionary/photometer
IPA: /fəˈtɒmətər/
Phonetic: foh-TAW-muh-tər

5. Photorheology

Link: No confirmed link found
IPA: /ˌfoʊtoʊˌriːˈɒlədʒi/
Phonetic: foh-toh-ree-AW-luh-jee

6. Geometry (rheometer geometry)

Link: https://www.merriam-webster.com/dictionary/geometry
IPA: /dʒiˈɑmətɹi/
Phonetic: jee-AW-muh-tree

7. Equilibrate

Link: https://www.merriam-webster.com/dictionary/equilibrate
IPA: /ɪˈkwɪlɪbreɪt/
Phonetic: ih-KWIL-ih-brayt