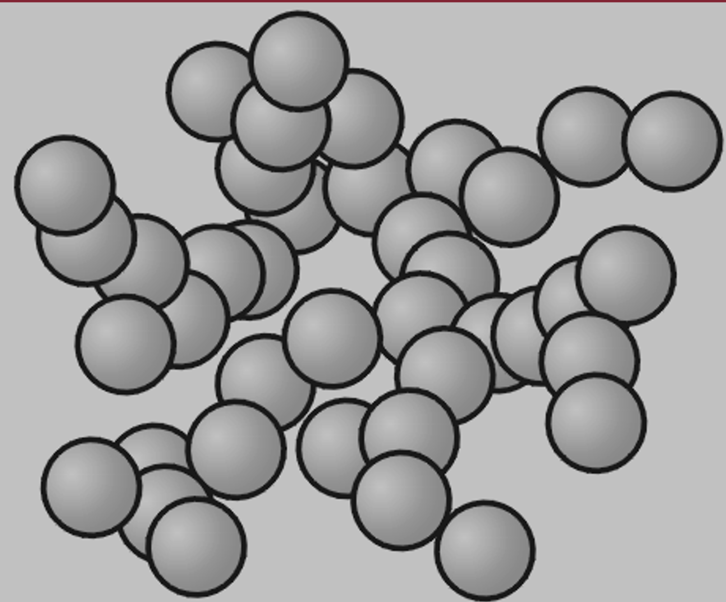

Approaches to Preparing Rhodamine-Dyed Monolithic Silica Aerogels

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Introduction

Silica aerogels are unique nanoporous materials that can be used in a variety of applications. Silica aerogels are attractive for window applications as they are monolithic, thermally insulating, and extremely lightweight. When placed between two panes of glass, the aerogel can serve as a highly effective insulator (Figure 1).[1] They also can be made transparent or translucent. Aerogels can be made using a variety of methods; in this work, a rapid supercritical extraction method (RSCE) is employed. Dyed aerogels can be placed in between windows to create a colored glass window, similar to a stained glass window (Figure 2).[2] This is not just insulating, but aesthetically pleasing as well. We are investigating the usage of dyed monolithic silica aerogels in window applications to improve aesthetic effects. However, the high temperatures and pressures required for the RSCE process can lead to dye degradation. In this presentation, we demonstrate how to incorporate Rhodamine 6B and Rhodamine B into monolithic silica aerogels. Rhodamine B experiences thermal degradation during monolithic aerogel synthesis. Adjusting processing parameters, including temperature and pressure, can minimize thermal degradation while ensuring the structural integrity of the aerogel. Ultraviolet radiation can also lead to dye degradation, which would be problematic for aerogels in a window application. Future work will focus on spectroscopic characterization of the dyed aerogel monoliths.

Figure 1. Aerogel window prototype[1]

Figure 2. Dyed aerogel mosaic, created by Zineb Hijjaj[2]

Research Objectives

- Successfully incorporate Rhodamine dyes into monolithic aerogels.
- Prevent thermal degradation of Rhodamine dyes.
- Investigate whether dyed aerogels photodegrade when exposed to sunlight over an extended period.

Aerogel Fabrication Approach

Precursor solution formula:
- 48.895 g methanol*
- 1432 μL 1.5-M Ammonia
- 6.968 g DI water
- 16.311 g TMOS

*Dye is introduced into the methanol solution.
Ammonia is added last to serve as a catalyst.
Rapid supercritical extraction yields monolithic aerogel.

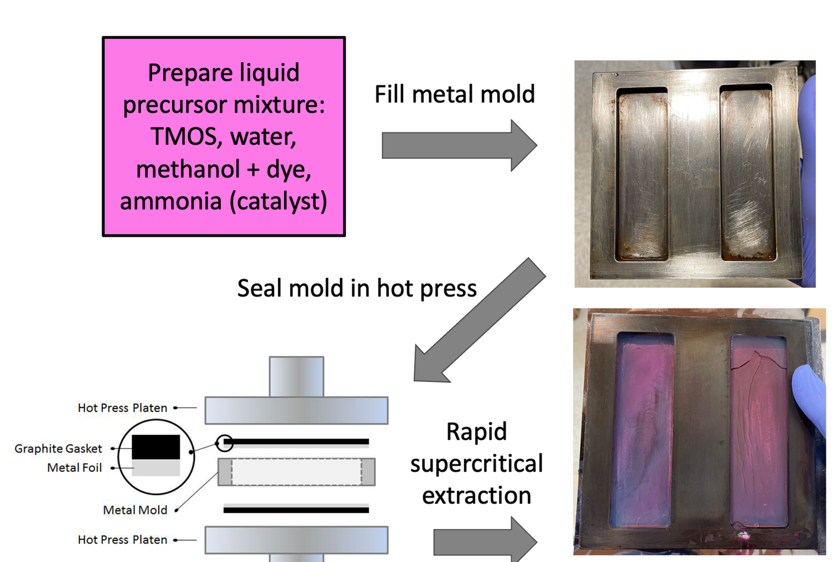

Figure 3. Aerogel fabrication process

Rhodamine 6G Aerogel Fabrication

Rhodamine 6G is an orange fluorescent dye. Rhodamine 6G was dissolved in methanol and was mixed with ammonia, DI water, and tetramethyl orthosilicate (TMOS), as per the prior recipe.
- RSCE processing with a maximum of 600 ºF (316 ºC) was used.
- These aerogels appeared vibrantly orange, and had minimal shrinkage when removed from the hot press (Figure 4)

Repeat measurements are warranted in order to ensure a vibrant color, minimize shrinkage, and create a structurally sound aerogel.

Analysis in a UV spectrometer showed a peak Absorbance of >2.0, hence indicating that lower quantities of Rhodamine 6G dye may be necessary for quantification.

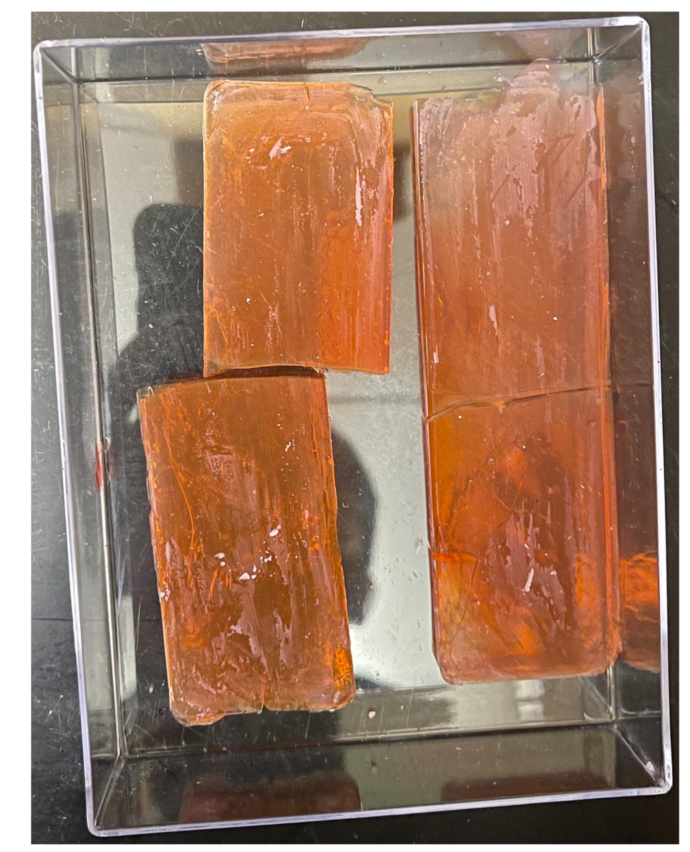

Figure 4. Rhodamine 6G dyed monolithic silica aerogels

Rhodamine B Aerogel Fabrication

Rhodamine B is a pink fluorescent dye. Using the method described above, we attempted to synthesize monolithic Rhodamine B aerogels.

Attempt #1: 550 ºF (288 ºC)
- Aerogels were completely clear and it appeared as if the dye was ingrained on top of the mold.

Attempt #2: 525 ºF (274 ºC)
- Similarly clear aerogels were made. Tiny aerogel particles containing rhodamine were found on the edge of the gasket.
- Decided to lower temperature 10 ºF.

Attempt #3: 515 ºF (268 ºC)
- Slightly cracked in the mold
- Color was present in the aerogel.
- Optimal recipe as this allowed a translucent aerogel to be made. The gasket when removed ripped off a piece of the aerogel, creating a divot in it.
- Visual analysis revealed that the Rhodamine dye was concentrated evenly throughout the edge of the aerogel, and minimal dye was in the center of the aerogel. This result was unexpected and the experiment will be repeated.

Synthesis at lower temperatures and pressures created cracked and cloudy aerogels.

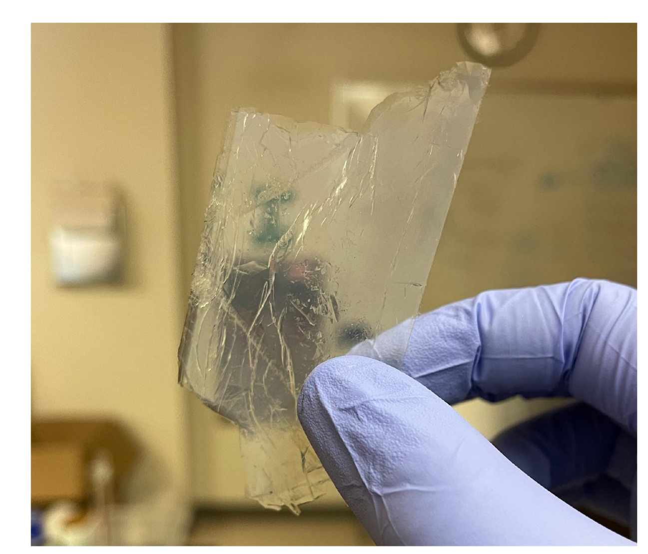

Figure 5. Silica aerogel monolith prepared with Rhodamine B is colorless, indicating thermal decomposition of the dye occurred

Figure 6. Rhodamine B successfully dyed into silica aerogel

Spectroscopic Analysis

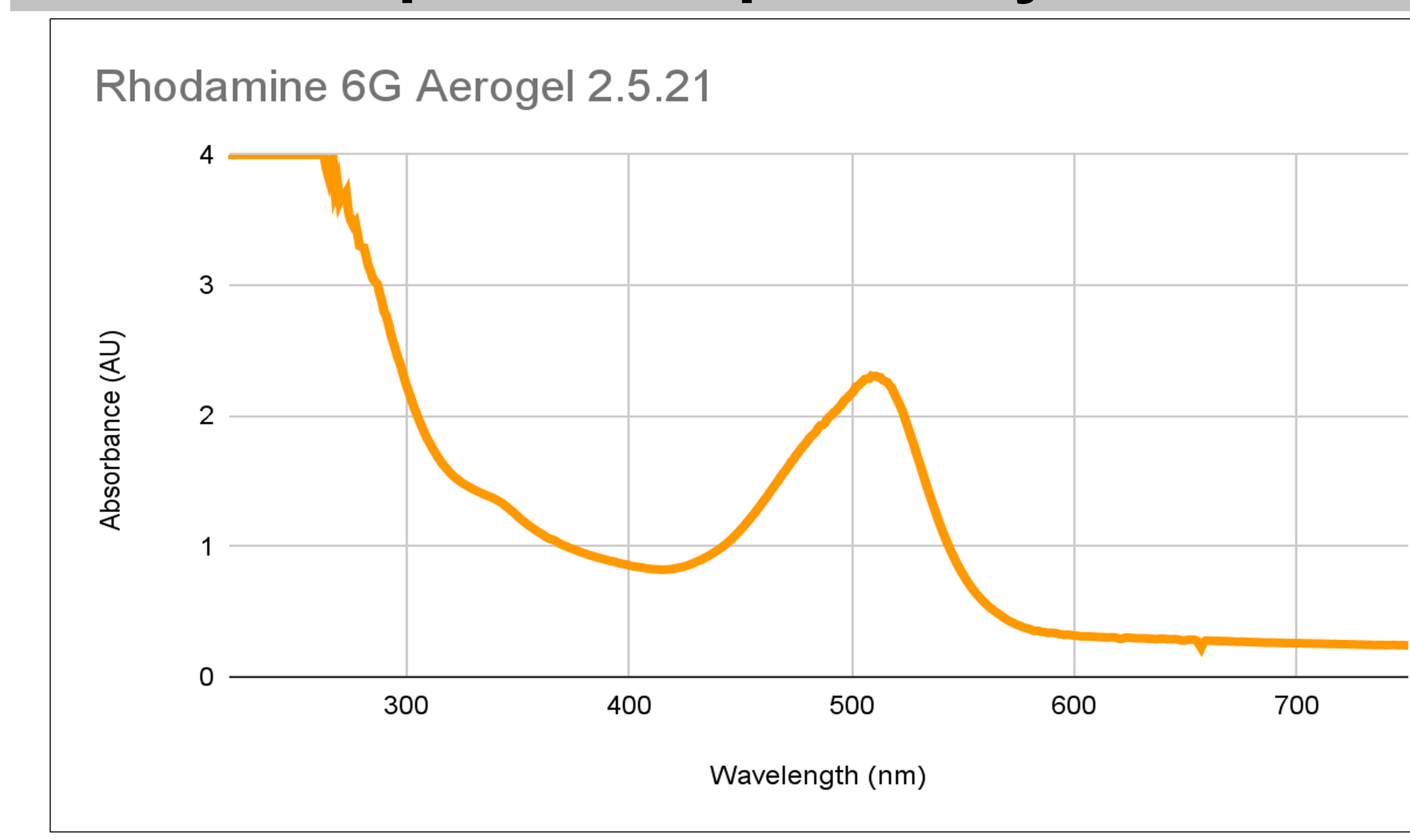

Figure 7. Absorption spectrum of Rhodamine 6G aerogel

- Silica aerogel dyed with Rhodamine 6G has peak absorbance at 509 nm
- Baseline does not have A = 0 because the aerogel is scattering light

Conclusions

- Aerogel monolith containing Rhodamine 6G can be readily fabricated.
- Lower temperatures are required for successful fabrication of Rhodamine B dyed aerogel.
- Dyed aerogel monoliths absorb visible light strongly.
- Some inconsistencies in distribution of dye within the aerogel monoliths are observed.

Future Work

Quantitative investigation of the extent to which UV light degrades the dyes in the aerogel will be conducted. Solutions and aerogels will be placed in a solar simulator, a device that artificially simulates sunlight in a condensed time frame. A UV-visible spectrophotometer will be used to quantify the amount of dye in the aerogel as a function of UV light exposure time.

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Acknowledgements

The authors are grateful to Zineb Hajjaj for prior work, and to Joana Santos and Sri Teja Mangu for assistance with the hot press process. Funding for this project was provided through National Science Foundation Grant No. IIP-2025800 (awarded to SunThru LLC with subaward to Union College) and the Union College Chemistry Department Kane Fund.