Fine Tuning Sol-gel Synthesis and Further Applications

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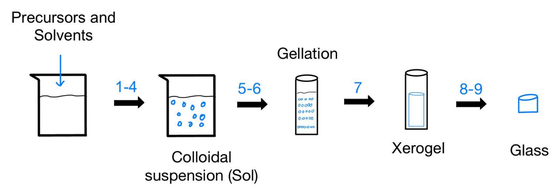
Motivation

Sol-gel glasses have various useful applications, but the process itself is finicky and yield is low.[1]

Aims

1. Systematically adjust various steps of the sol-gel synthesis procedure to optimize yield and pore size
2. Synthesize fluorescent nanoparticles governed by the porosity of the material

Methods[2,3]



1. Add DI water to a solution of TEOS (tetraethyl orthosilicate) and ethanol (12:1:2 molar ratio)
2. Stir for 15 minutes at 40°C in a paraffin oil bath
3. Remove from heat, and then add 1 mmol HCl
4. Stir at room temperature until homogenous
5. Add previous amount of water and ethanol to solution, and 1 mmol NH_4OH to initiate condensation
6. Distribute solutions into polyethylene vials, cap, and age for one week
7. Replace cap with perforated Parafilm, and dry for six to eight weeks
8. Fire gel at ramp rate of 0.05°C/min to 500°C, dwell for 360 min
9. Ramp down at 0.10°C/min to 25°C

Results

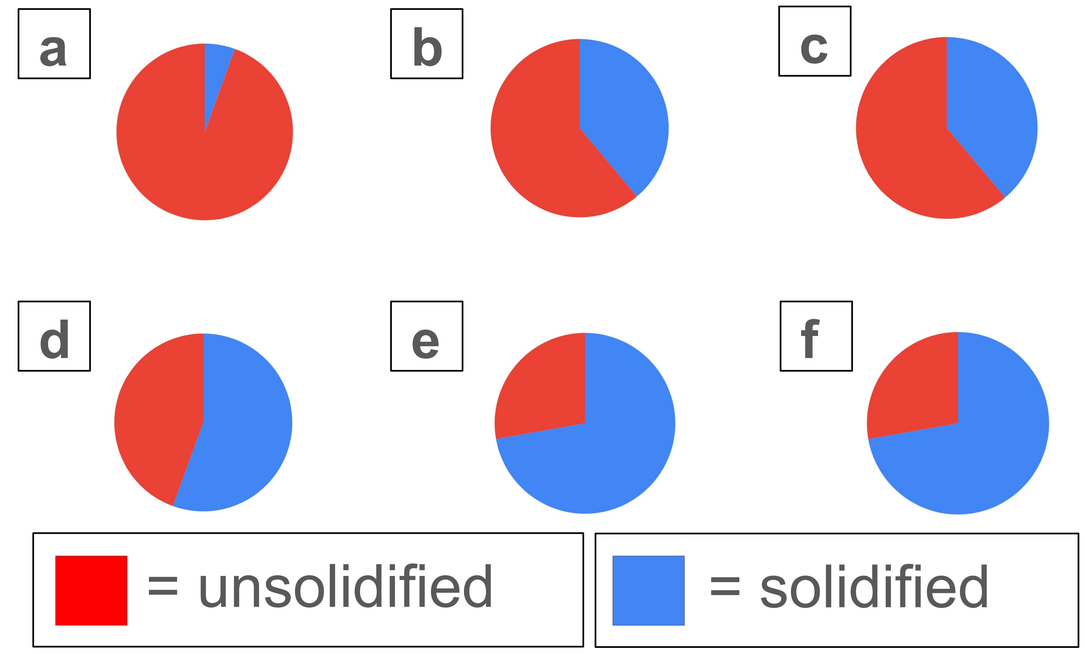


Figure 1: Percentage of solidified vs. unsolidified sol-gels in batch a) 9/5, b) 9/7, c) 9/12, d) 9/20, e) 10/11, f) 10/18.

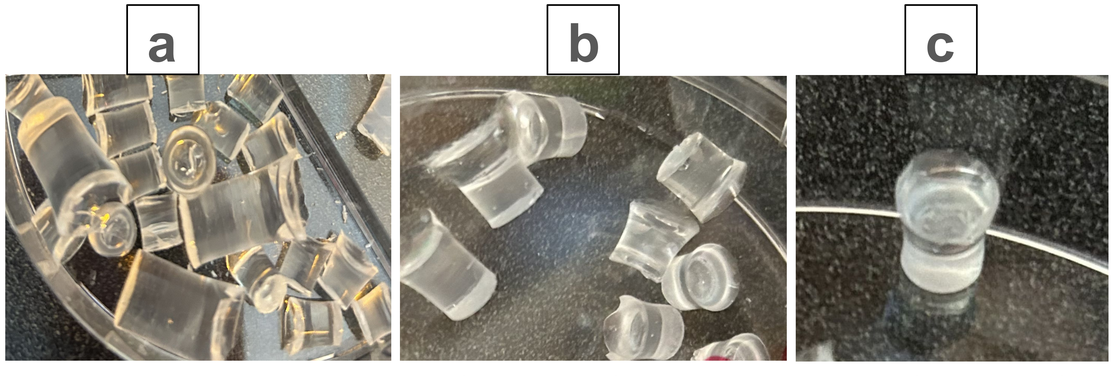


Figure 2: Images of solidified sol-gels from a) 9/20, b) 10/18, and c) 10/18 after oven firing.

Conclusion

- As seen in Results, this method has greatly increased the yield of the solidified sol-gel glasses
  - More successful in producing whole, uncracked glasses
- Our synthesis provides consistent, reproducible results for a procedure known to be unreliable

Future Research

Fluorescent Nanoparticles

1. Soak sol-gel glass in photoresist consisting of 0.025 wt% Rhodamine G in SR499
2. Irradiate sample with 405 nm laser to induce polymerization/solidify the nanoparticles within the sol-gel framework
3. Dissolve the sol-gel glass in HF acid
4. Use UV-Vis spectroscopy to validate the presence of nanoparticles

Can also be relevant to:

- Sol-gel process in different settings: thin film coatings, wet-gel synthesis, etc

References

1. Garino, T. J. The Cracking of Sol-Gel Films during Drying. *MRS Proceedings* **1990**, *180*. DOI:10.1557/proc-180-497
2. Loughnane, B.J. Dynamics of Liquids in Confinement. PhD Thesis, Boston College: Boston, MA, 1999.
3. He, X. Studying Liquid Dynamics with Optical Kerr Effect Spectroscopy. PhD Thesis, University of Maryland, College Park: College Park, MD, 2015.