Advanced Foam Shell Production

Laser IFE Program Workshop

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Pleasanton, CA
November 13-14, 2001

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An Employee-Owned Small Business

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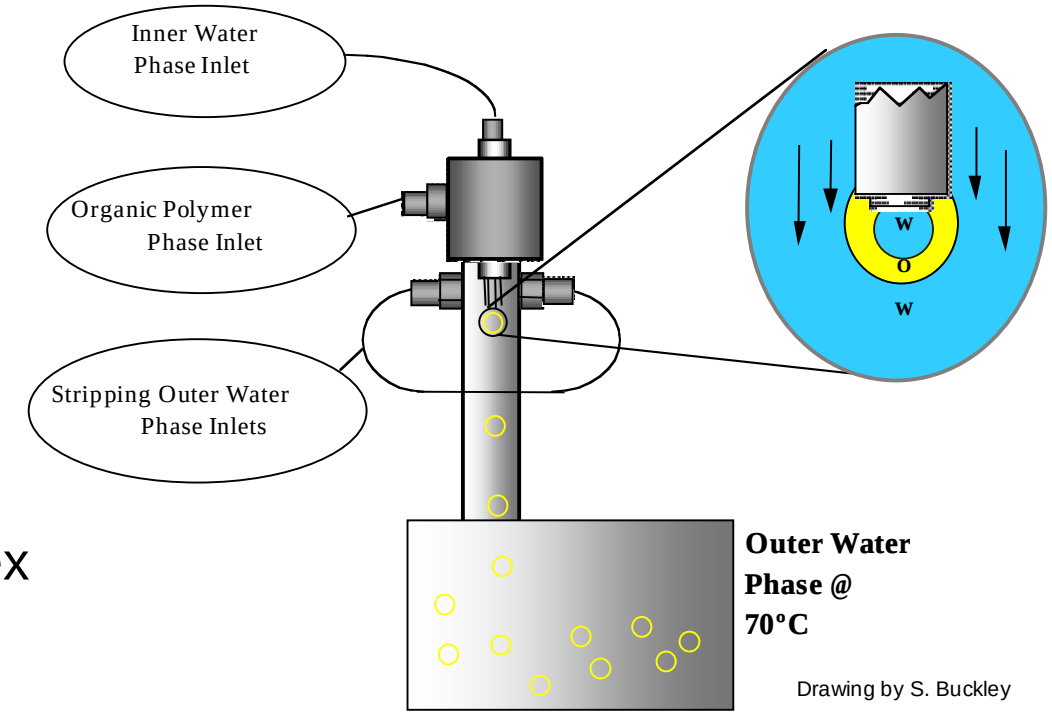
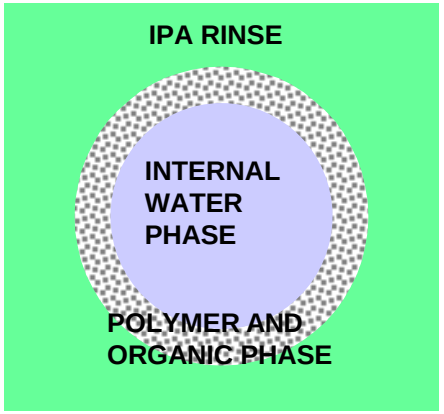
We Proposed Making Overcoated Shells Using A Two Step Approach.

- In the first step, a droplet generator produces uncoated foam shells.
- We have refurbished the droplet generator to accommodate the larger size spheres.
- Gelation of the oil phase needs to occur about 15 minutes after the complex drop is formed.

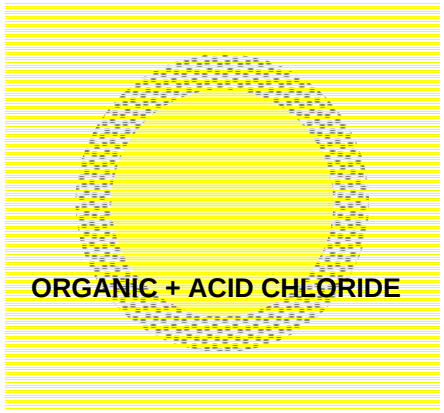


Drawing by S. Buckley

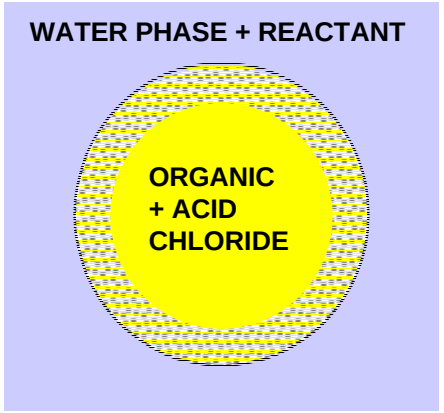
In the Second Step, Interfacial Polymerization Produces The Polymer Overcoat.



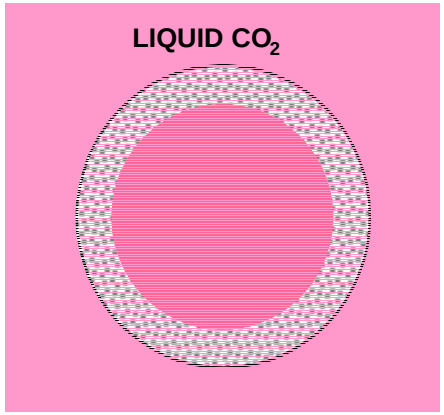
The organic phase and internal water phase must be removed.



The shell is placed in organic solvent + acid chloride.



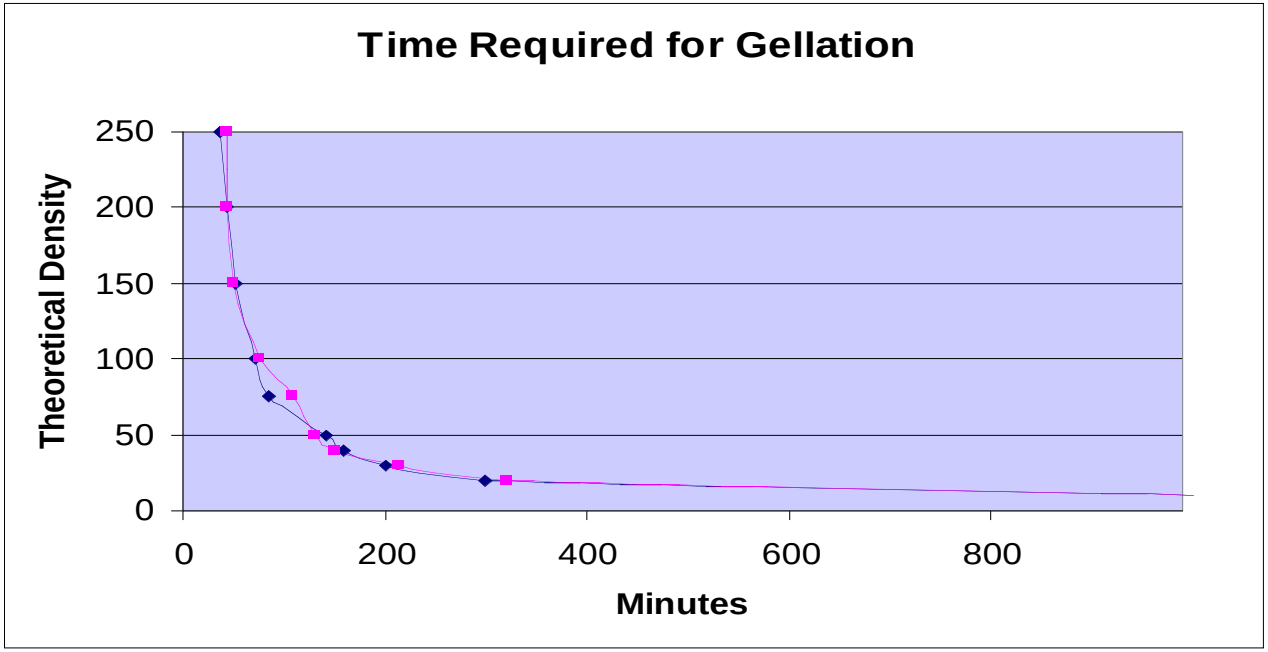
The shell is placed in a reactive aqueous solution. A wall is built at the interface.



The shell is rinsed with IPA, then liquid CO_2. The CO_2 is taken supercritical and vented.

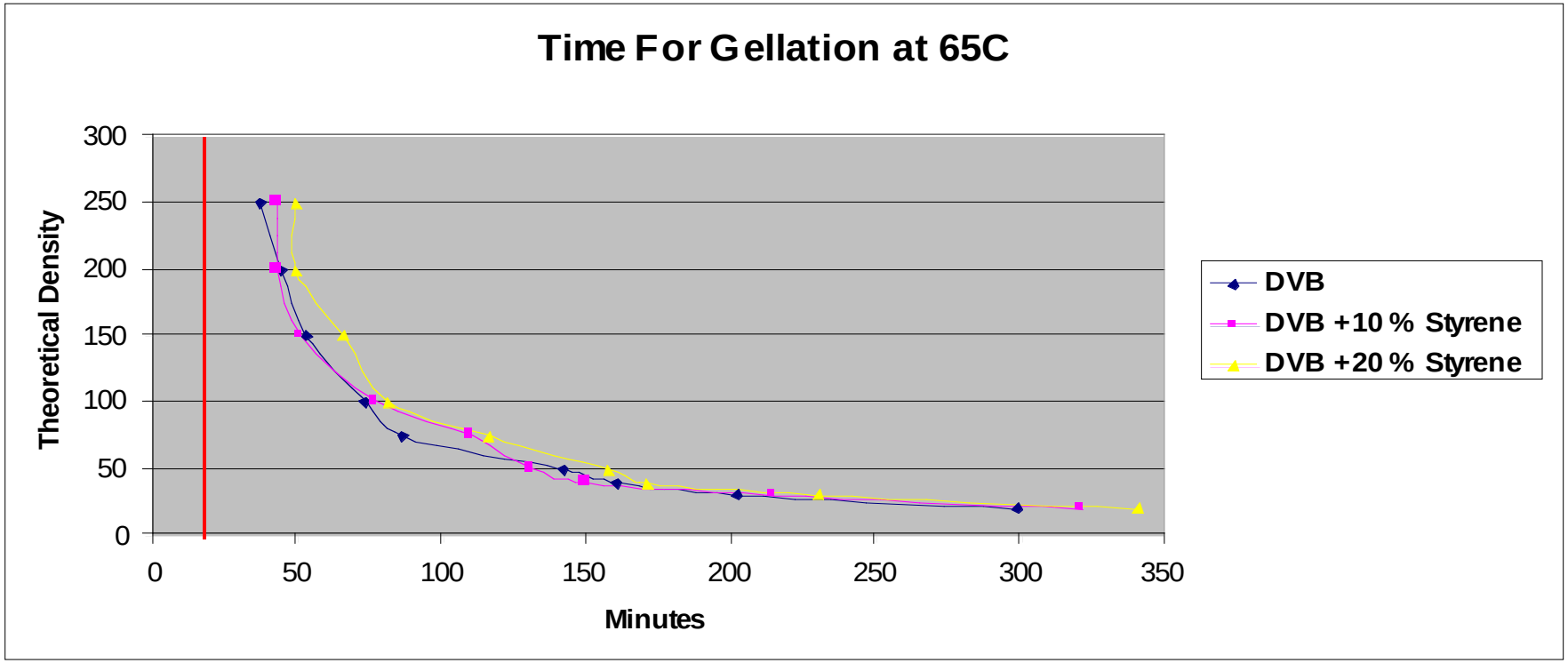
I Presented This Slide Last Time.

- We have begun gellation studies
  - **Time to gelation is a function of density.**
  - **Clearly the lower the density the more difficult microencapsulation becomes.**
  - **We will vary the polymerization conditions, radical initiators, and try adding small amounts of other CH monomers.**

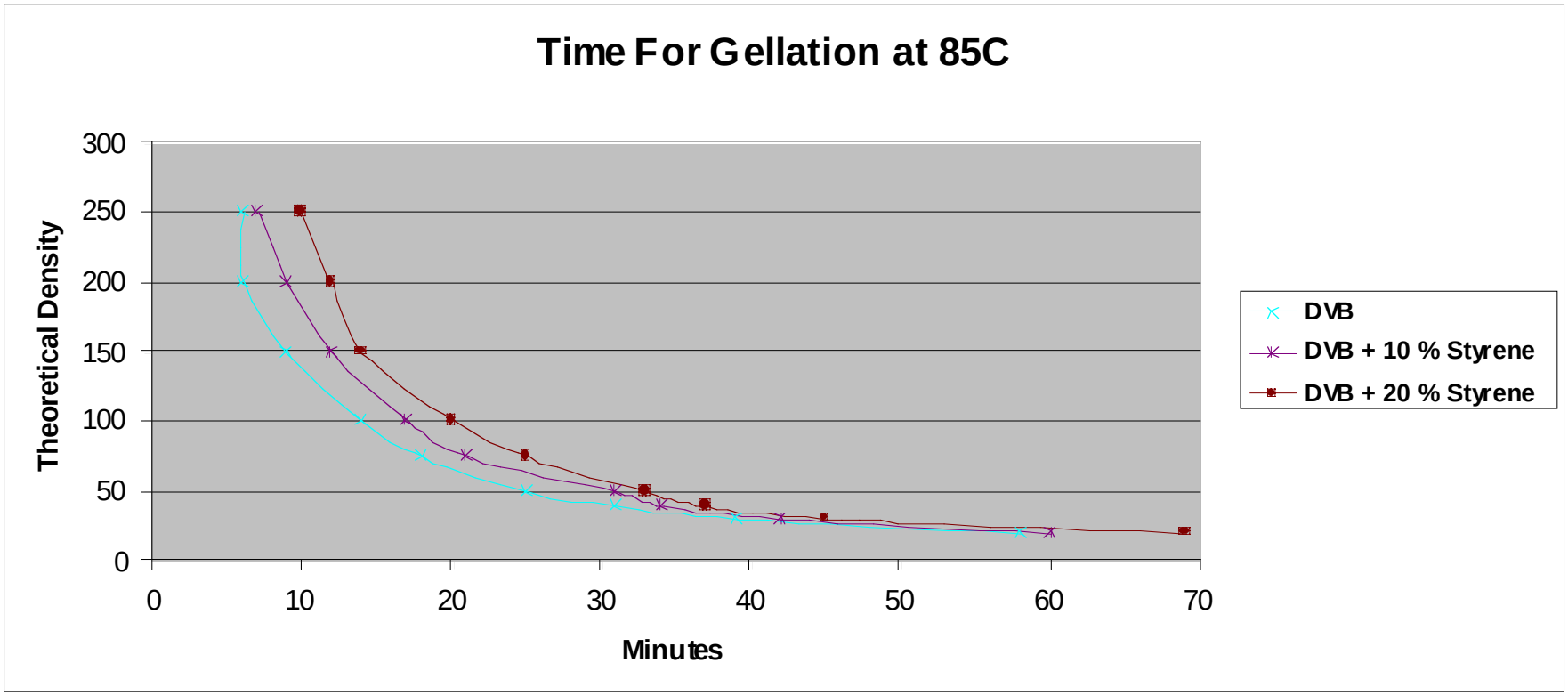


We Have Varied All Of those Parameters.

- I still want to try one more radical initiator - but it is difficult to obtain because of its high reactivity.
- The only valuable parameter is temperature.



Even at 85ºC Only One 100 mg/cm^3 Foam Meets the Time Requirement.



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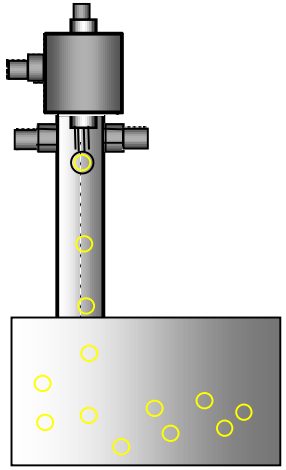
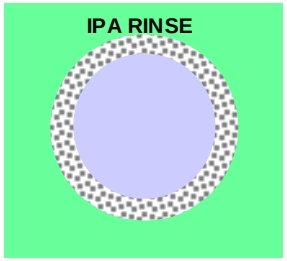
We Tried Two Approaches To Solve This Problem.

1. **Starting the polymerization in a method analogous to the RF microencapsulation procedure.**
   - **This helps, but the polymerization does not have the dramatic viscosity increase of RF, so results are difficult to reproduce.**
2. **Starting the overcoating chemistry at the initial microencapsulation stage. (Lloyd Brown concept)**
   - **The overcoating reaction is fast - done in about 5 minutes.**
   - **We have tried this two ways**
     - **Run the overcoating chemistry first, the polymerize. This could be "cleaner" from an interaction point of view.**
     - **Partially polymerize the DVB, microencapsulate into overcoating solution, complete the polymerization.**

There Are Many Potential Advantages.

- **Process is much more streamlined.**

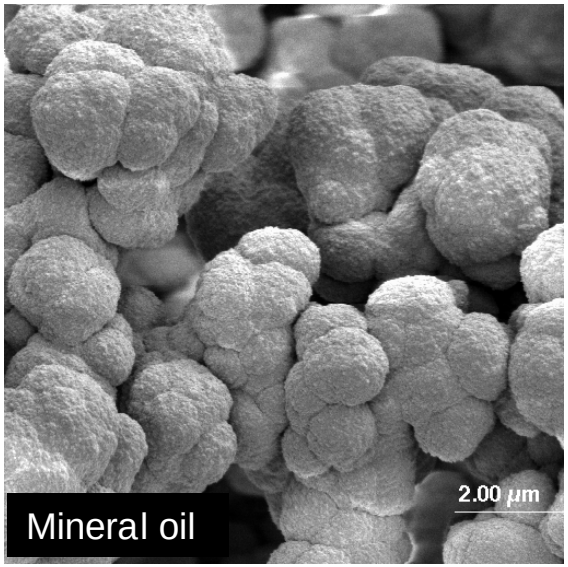


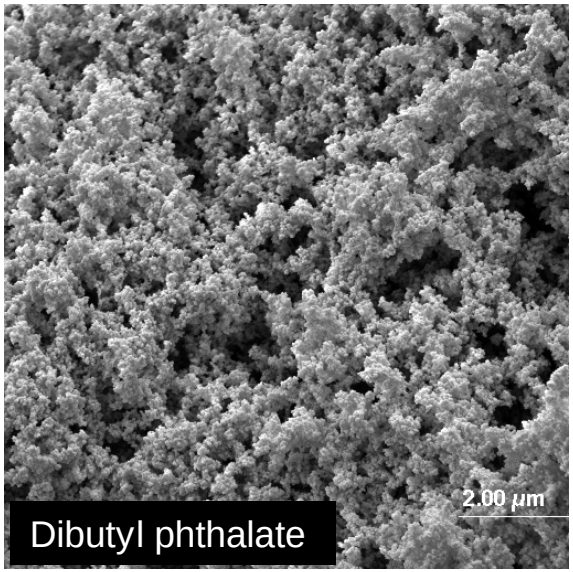


- **Only one step risks agglomeration.**
- **Disadvantages:**
  - **It may be more difficult to optimize for both processes simultaneously; for example, best solvent.**
  - **Uniformity does not look as good in these early trials.**

We Need To Characterize Foam Processed With The Acid Chloride In The Organic Phase.

- We need to see if we have changed
  - the foam density (measure bulk pieces, radiograph spheres)
  - composition (Cl or O added)
  - or foam structure (SEM)

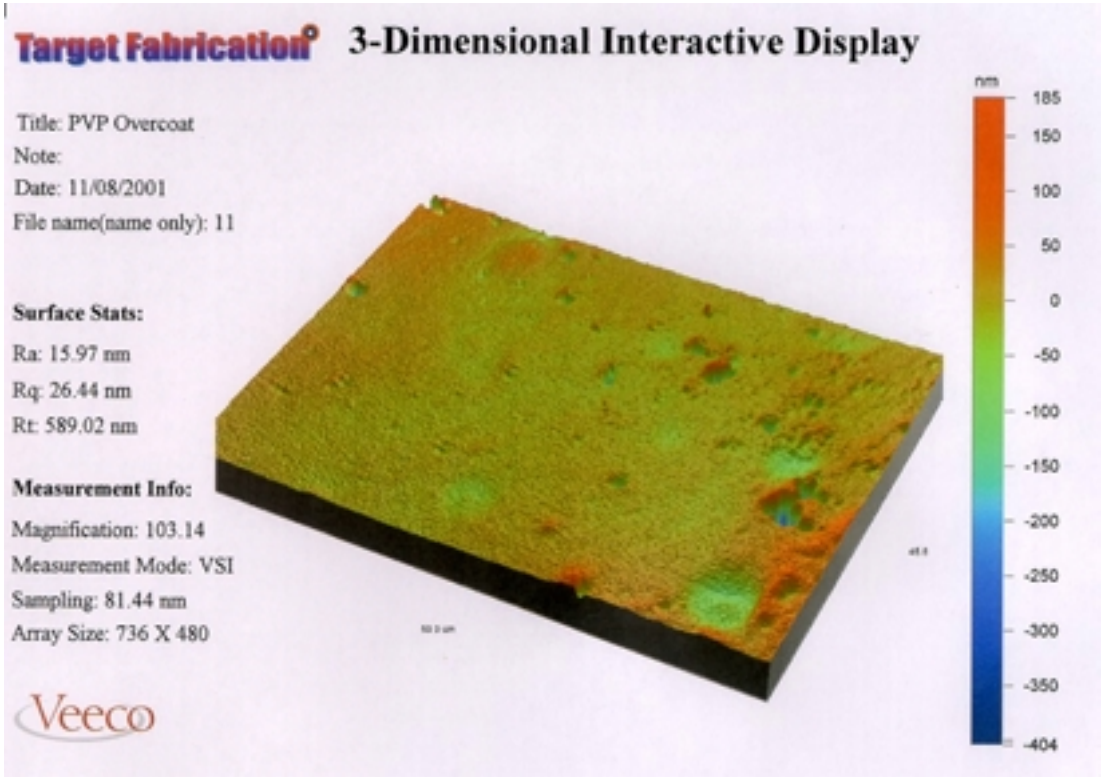




Both micrographs are of 70 mg/cm^3 foam. The only difference in their production was the polymerization solvent.

We Need To Characterize The Overcoat.

- To begin, we wanted to determine the intrinsic surface finish.
  - Made empty (no polymer) shell, ripped it open and laid it on a silicon wafer.
  - Need to dry capsule or bead and compare.



Progress To Date

- Studied gellation rates.
- Polymerized DVB beads $\geq$ 4 mm in diameter with densities down to 50 mg/cm^3.
- Created overcoated polymerized beads in a single step.
  - Need to decide between two approaches, pre-polymerization or overcoat followed by polymerization.
- Need to make capsules.
  - With overcoat chemistry capsule preforms will be more stable.
  - May need to adjust density match for cooler solutions (toluene, D_2O).
- Need to characterize product, both foam and overcoat.