Temperature-dependent binary solvent structure of solvent segregation driven gel (SeedGel)

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Bicontinuous Structure Applications

Previous Gels

SeedGel

Filtration

Smart Windows

Battery Technology

Tissue Engineering

The SeedGel is a porous material with tunable pore size.

It can switch from transparent to opaque and back again.

Its high surface area can enhance the capacity of batteries.

Its channels can serve as scaffolds for cell growth and delivery.

Nanoparticle Gel Unites Oil and Water in Manufacturing-Friendly Approach, NIST, 2021
What is SeedGel?

- Bicontinuous channels
- Thermo-reversible
- Gel phase at higher temperatures

Langmuir 2021, 37 (6), 2170-2178
Nature Communications 2021, 12, 910
SeedGel Domains

Liquid Phase

Gel Phase

Water and silica particles

2,6-Lutidine or 3MP

Bulk Solvent Phase Diagram

Nature Communications 2021, 12, 910

Soft Matter 2016, 12(18)
SeedGel has Unique Optical Properties

- Structure color
- Dependent on temperature
- Dynamically tunable

Patent Pending
Types of Data

- Wide-Angle X-ray Scattering (WAXS)
- Small Angle X-ray Scattering (SAXS)
- Small Angle Neutron Scattering (SANS)
- Ultra-Small Angle Neutron Scattering (USANS)

\[ q = \frac{4\pi}{\lambda} \sin(\theta_s) \]
System #1

Water and 2,6-lutidine binary solvent
SANS Data

Intensity (A.U.)

Temperature
- 20 °C
- 22 °C
- 23 °C
- 24 °C
- 25 °C
- 30 °C
- 34 °C
- 38 °C

q (Å⁻¹)

Liquid Phase

Gel Phase

Solvent Domain

Particle Domain

Particle Domain
Form Factor Modeling

\[ I(q) = \frac{\text{scale}}{V} \cdot \left[ 3V(\Delta \rho) \cdot \frac{\sin(qr) - qr \cos(qr))}{(qr)^3} \right]^2 + \text{background} \]
Structure Factor

\[ I(q) = n \times v^2 \times \Delta \rho^2 \times P(q) \times S(q) \]
Temperature Trends at Select q Positions

\[ d = \frac{2\pi}{q} \]

length scale

<table>
<thead>
<tr>
<th>q Position (Å⁻¹)</th>
<th>Length scale (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.001318</td>
<td>476.721</td>
</tr>
<tr>
<td>0.04987</td>
<td>12.599</td>
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</tbody>
</table>
System #2

Water and 3-methylpyridine (3MP) binary solvent
SeedGel with Hydrogenated 3MP

SAXS Data

Length scale of further analysis
SeedGel with Deuterated 3MP

SAXS Data

I(q) vs. q (Å⁻¹)

Temperature:
- 25 °C
- 36 °C
- 40 °C
- 46 °C
- 50 °C
- 56 °C
- 60 °C
- 64 °C
- 80 °C

Length scale of further analysis
Deuterated vs Hydrogenated 3MP

Temperature (°C)

Intensity (A.U.)

Gel Sample

- A4 - H-3MP
- B4 - D-3MP
Conclusions

- Various binary solvents
- Tunable gel transition temperature
- Solvent separation and gelation coincide
- Deuteration increased gelation temperature
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Bijel vs SeedGel

Unlike bicontinuous interfacially jammed emulsion gel (Bijel) which has its particles jammed at the interphase between the two solvents in the gel phase, SeedGel has its silica particles jammed into one of the solvent domains in gel phase.

Reference

Remove bijel half, include phase diagram for water/lut online
A4 WAXS Data Peaks
A1 - A binary solvent of 73.7 vol% H2O and 26.3 vol% H-3MP

Go up to what temp?

Maybe skip
A3 - A binary solvent of 53.8 vol% H2O and 46.2 vol% H-3MP
Solvent Comparison Graphs

Graph 1: Intensity (A.U.) vs. Temperature (°C)
- A1 - 26.3 vol% H-3MP
- A3 - 46.2 vol% H-3MP

Graph 2: q (Å⁻¹) vs. Temperature (°C)
- A1 - 26.3 vol% H-3MP
- A3 - 46.2 vol% H-3MP
Gauss fittings to obtain the peak positions
C2

Add in graphs and comments
To touch on:

-reversibility

-gelation occurs with increase in temp

-
Gauss Fitting for solvents
Q vs Temp for WAXS data peaks

A2
Previous Findings

- Transition temp of 26C

  So what were we looking for within the structure factor
A2- A binary solvent of 63.6 vol% H2O and 36.4 vol% H-3MP

SAXS

WAXS Data
Correct: $S(q) \ast \text{contrast}$

Which TM graphs to use?

Ignore
A4 - 22.7 vol% Particle and 77.3 vol% binary solvent (63.6 vol% H2O and 36.4 vol% H-3MP)

Draw line for B4 vs A4 comp q

Remove WAXS bc not including further analysis

SAXS Data

WAXS Data
B4 - 22.7 vol% Particle and 77.3 vol% binary solvent (63.6 vol% H2O and 36.4 vol% D-3MP)

**SAXS Data**

**WAXS Data**