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

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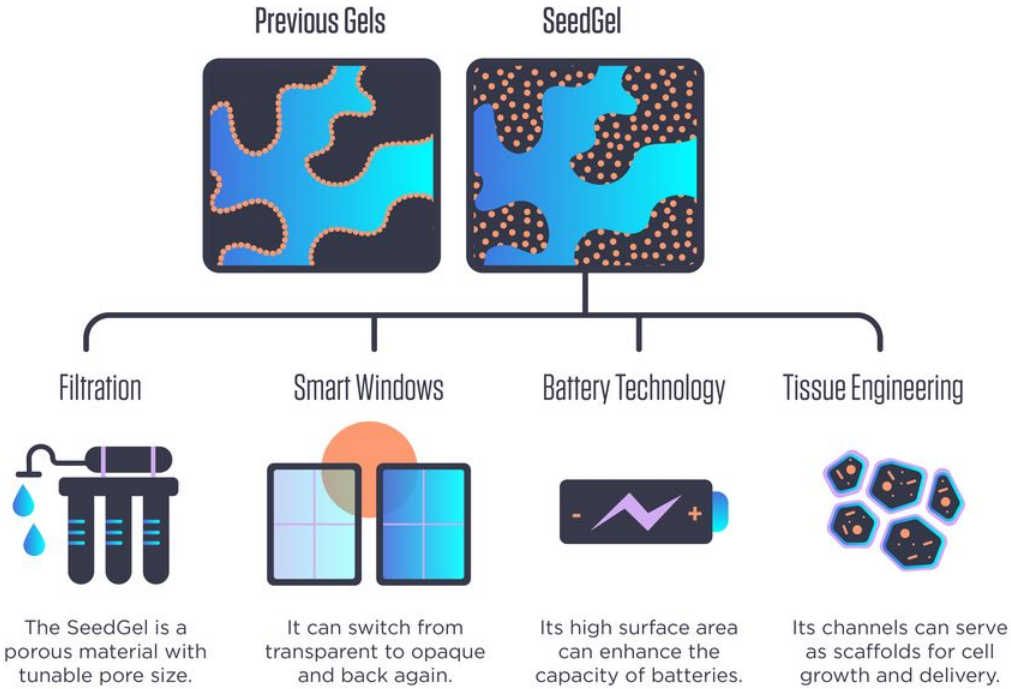
NIST
National Institute of Standards and Technology

BROOKHAVEN
NATIONAL LABORATORY

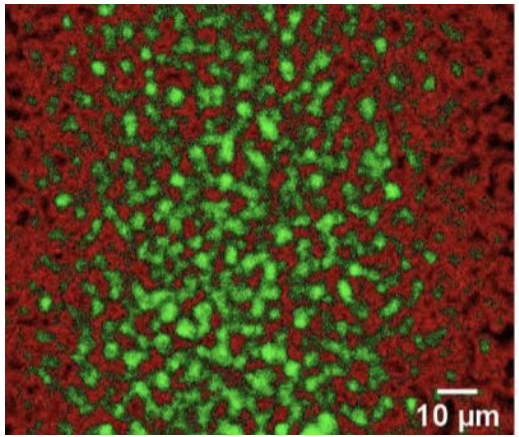
Bicontinuous Structure Applications



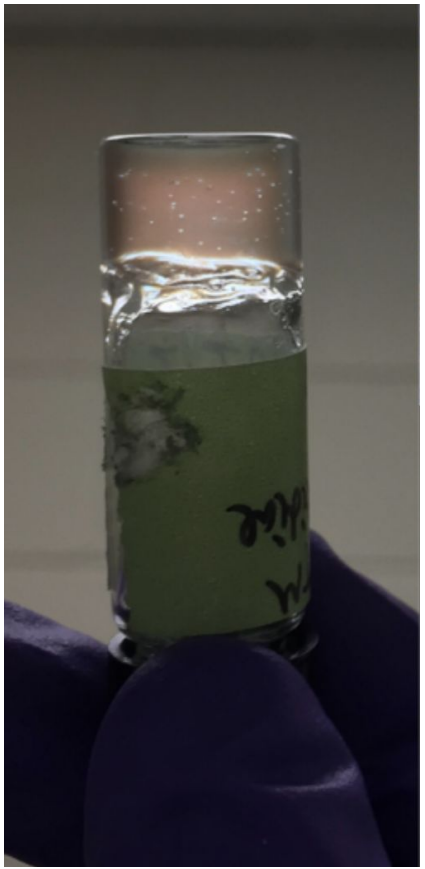
Nanoparticle Gel Unites Oil and Water in Manufacturing-Friendly Approach, NIST, 2021

What is SeedGel?

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



Nature Communications 2021, 12, 910

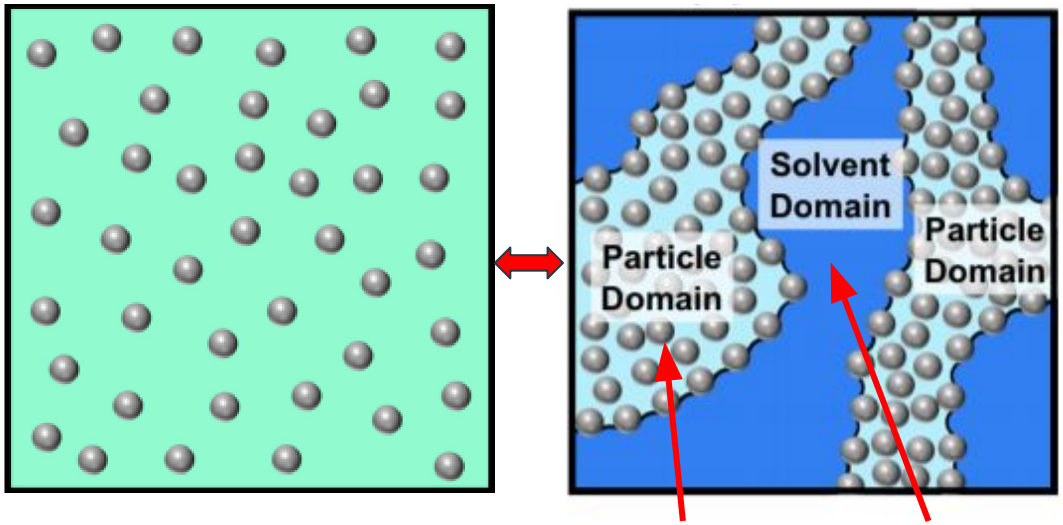


Langmuir 2021, *37* (6), 2170-2178

SeedGel Domains

Liquid Phase

Gel Phase

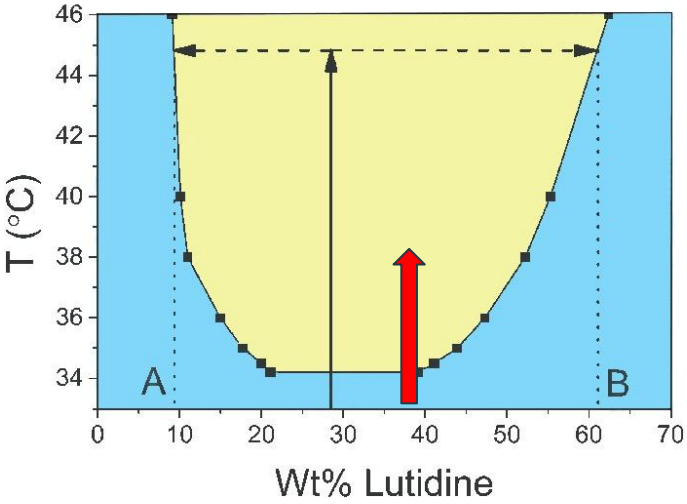


Water and silica particles

2,6-Lutidine or 3MP

Nature Communications 2021, 12, 910

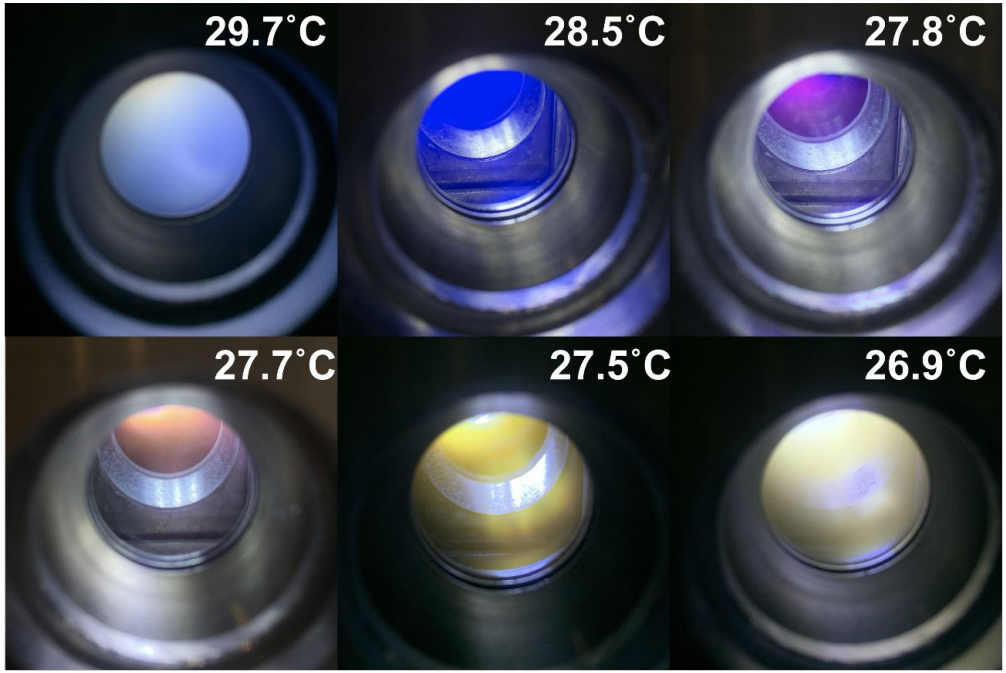
Bulk Solvent Phase Diagram



Soft Matter 2016, 12(18)

SeedGel has Unique Optical Properties

- Structure color
- Dependent on temperature
- Dynamically tunable

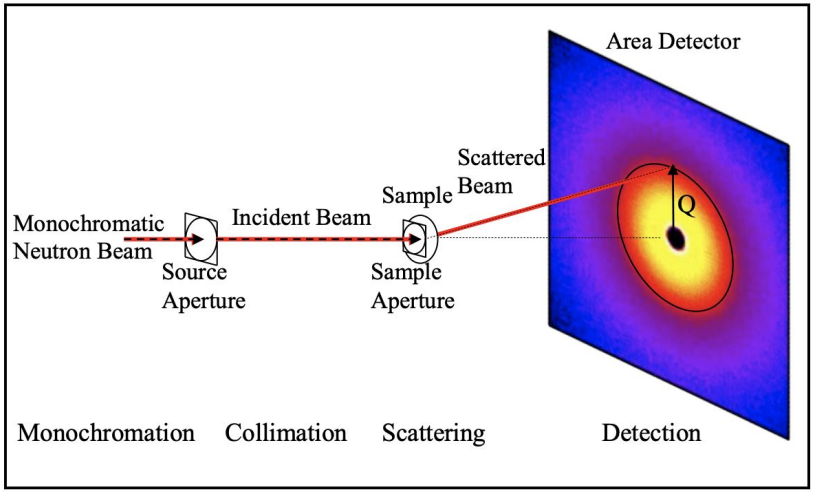


Patent Pending

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)

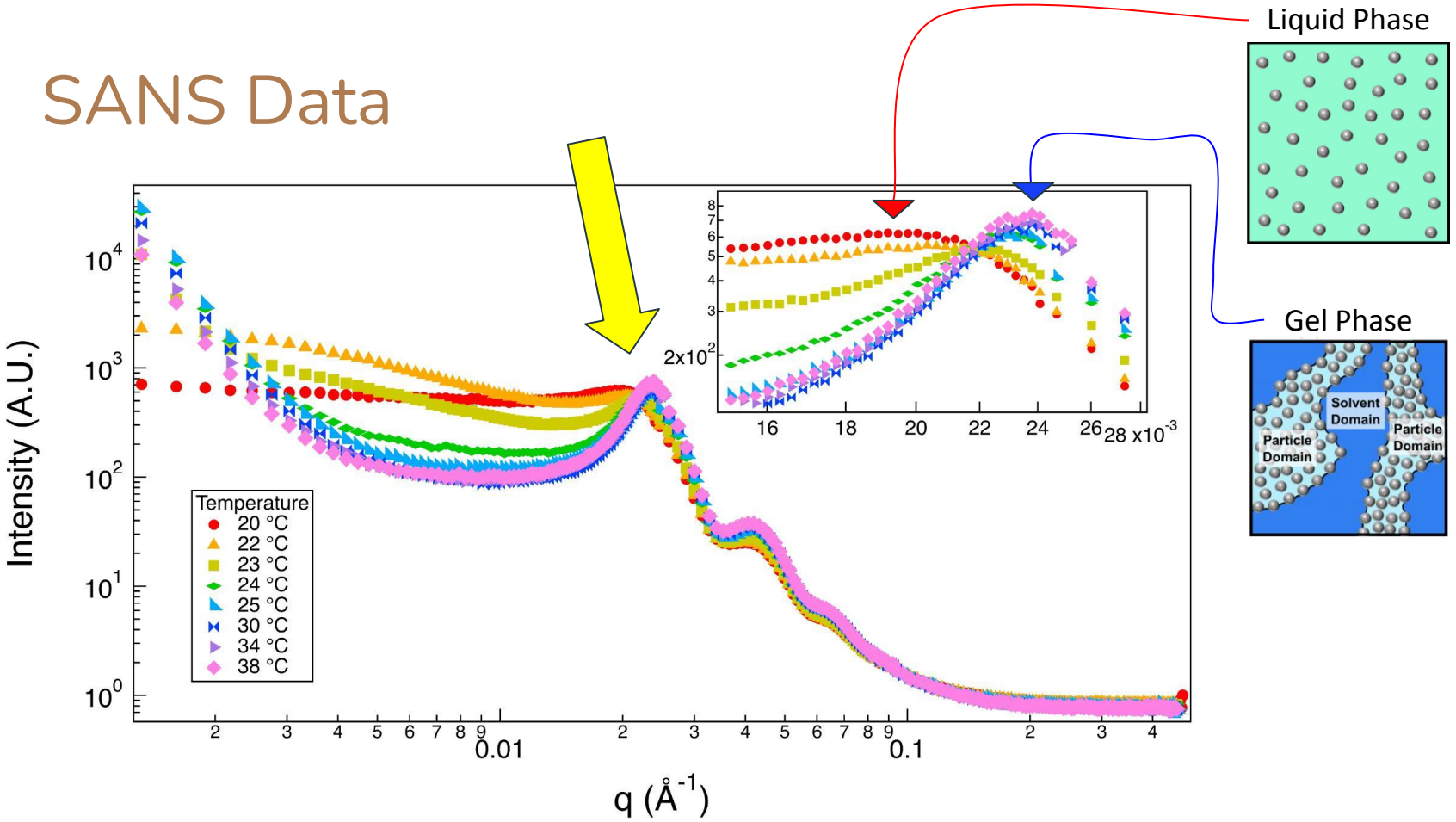
$$q = \frac{4\pi}{\lambda}\sin(\theta_s)$$



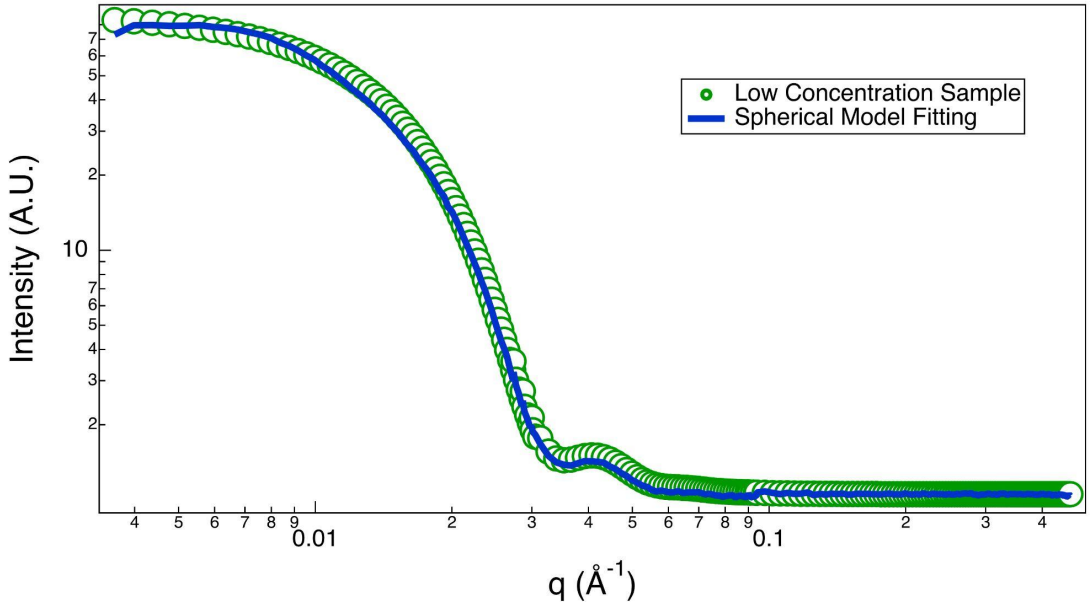
System #1

Water and 2,6-lutidine binary solvent

SANS Data


Liquid Phase
Gel Phase
Solvent Domain
Particle Domain
Particle Domain
Intensity (A.U.)
q (Å^{-1})
Temperature
20 °C
22 °C
23 °C
24 °C
25 °C
30 °C
34 °C
38 °C
2x10^{2}
28 x10^{-3}

Form Factor Modeling



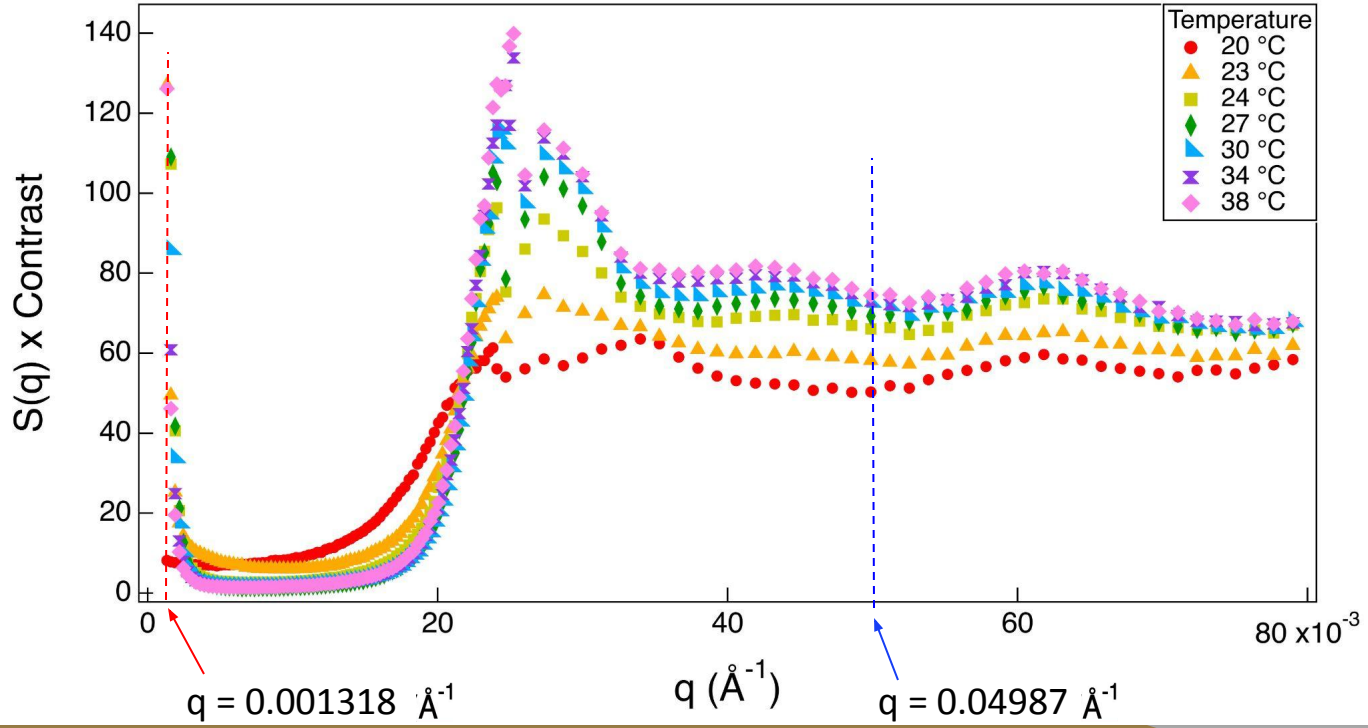
$$I(q) = \frac{\text{scale}}{V} \cdot \left[3V(\Delta\rho) \cdot \frac{\sin(qr) - qr\cos(qr))}{(qr)^3} \right]^2 + \text{background}$$

SasView Version 5.0.4, Zenodo

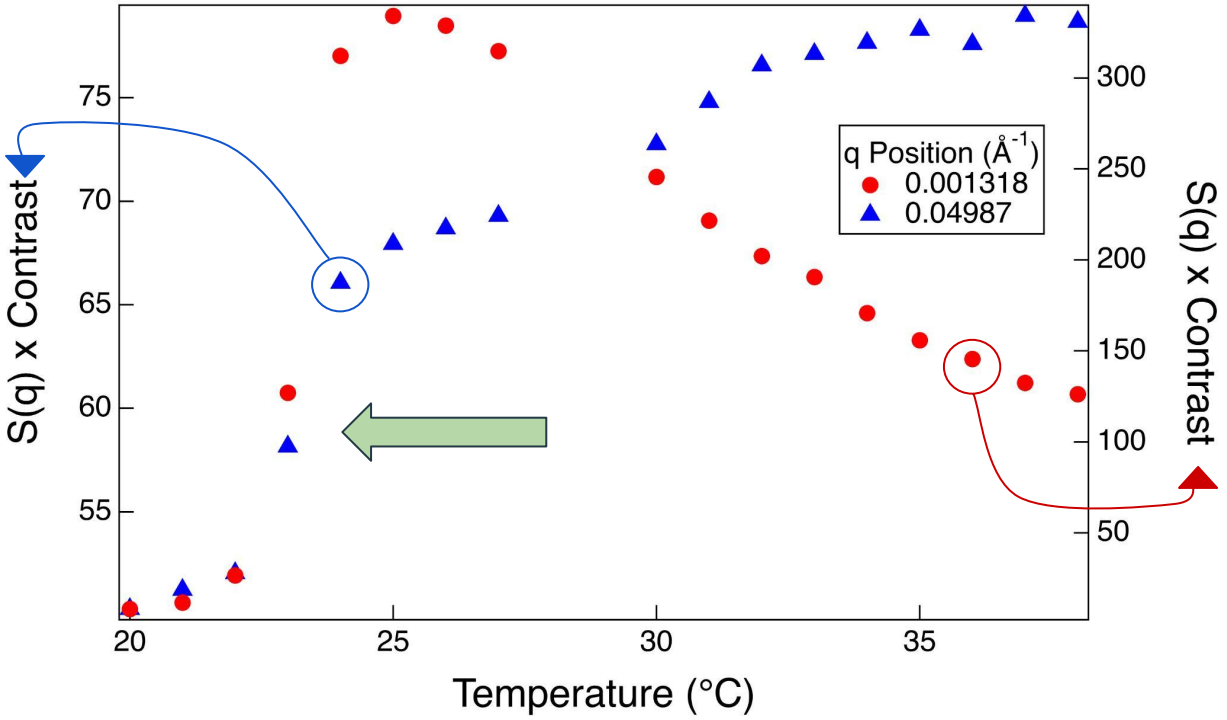
Small-Angle Scattering of X-Rays, 1955

Structure Factor

$$I(q) = n * v^2 * \Delta\rho^2 * P(q) * S(q)$$



Temperature Trends at Select q Positions



$$d = 2\pi/q$$

length scale

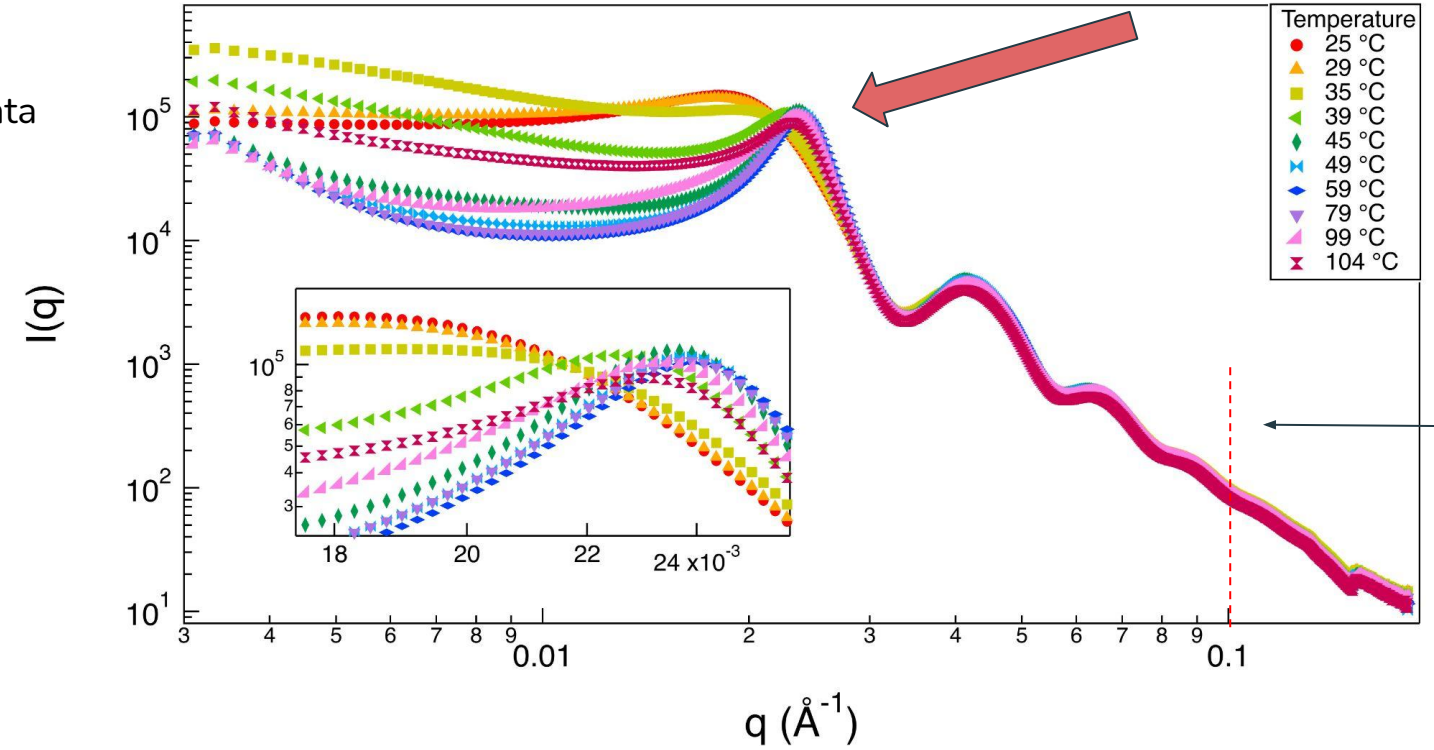
q value (Å^{-1})		length scale (nm)
0.001318	=	476.721
0.04987	=	12.599

System #2

Water and 3-methylpyridine (3MP) binary solvent

SeedGel with Hydrogenated 3MP

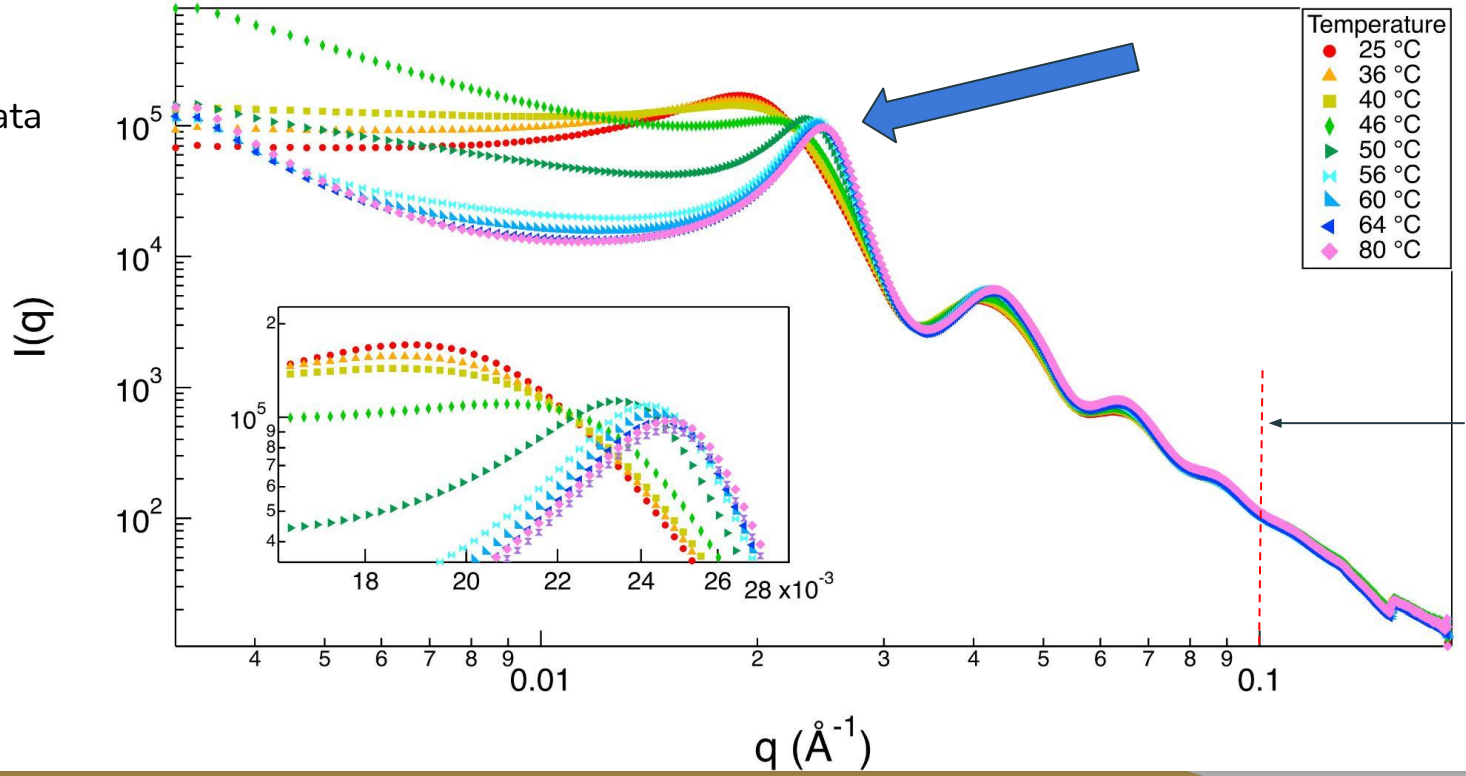
SAXS Data



Length scale of further analysis

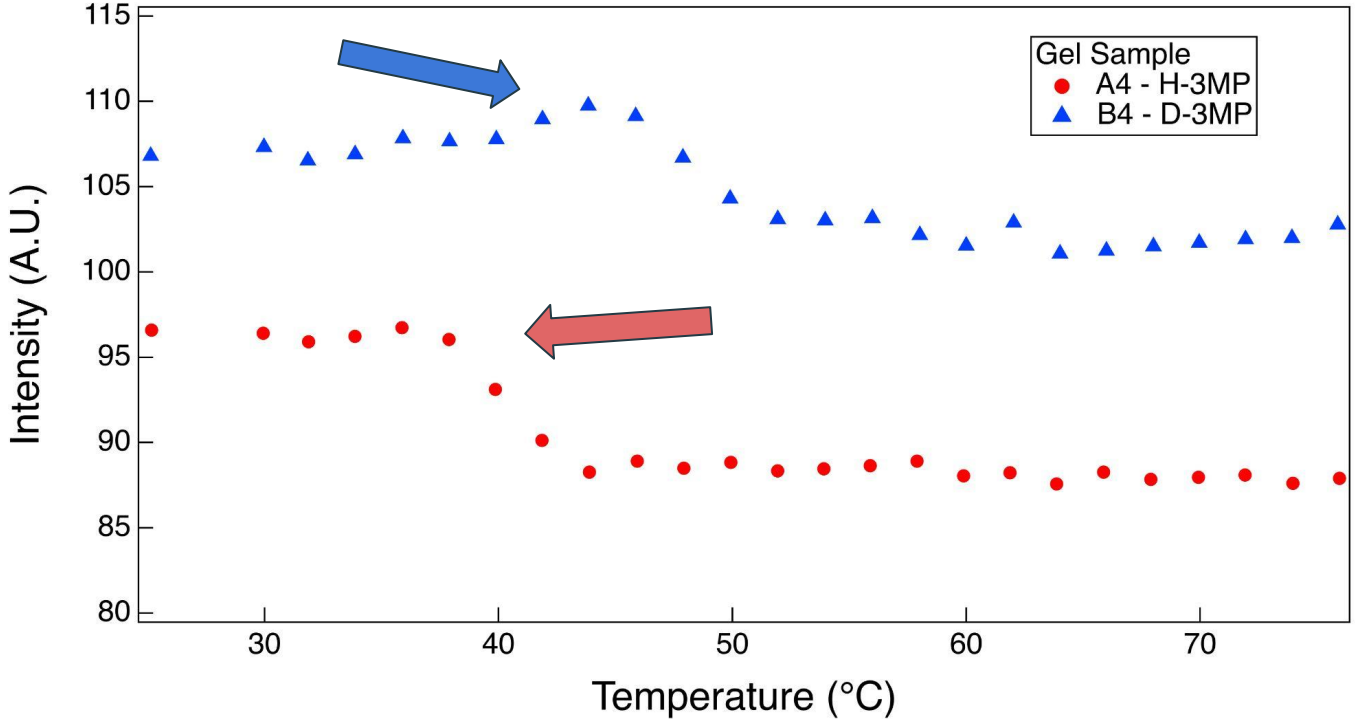
SeedGel with Deuterated 3MP

SAXS Data



Length scale of further analysis

Deuterated vs Hydrogenated 3MP



Conclusions

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

Acknowledgements

- Yuyin Xi, NCNR / University of Delaware
- Yun Liu, NCNR / University of Delaware
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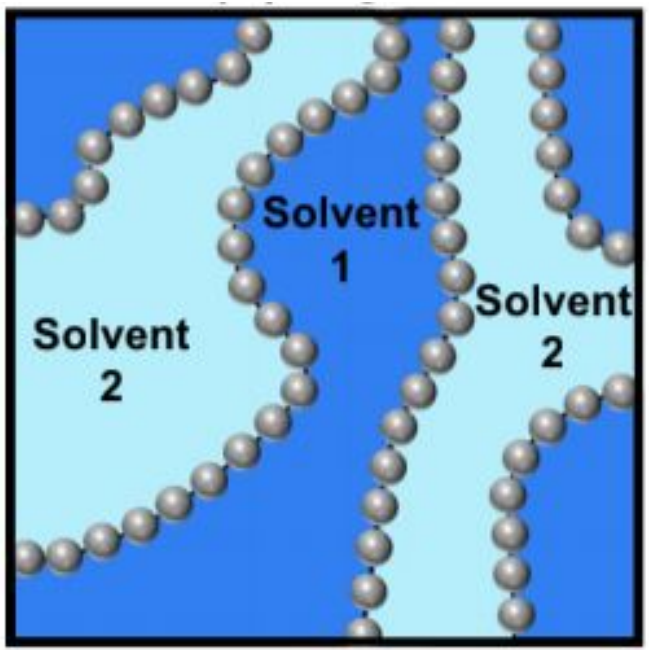
- Joseph Dura, NCNR
- Julie Borchers, NCNR

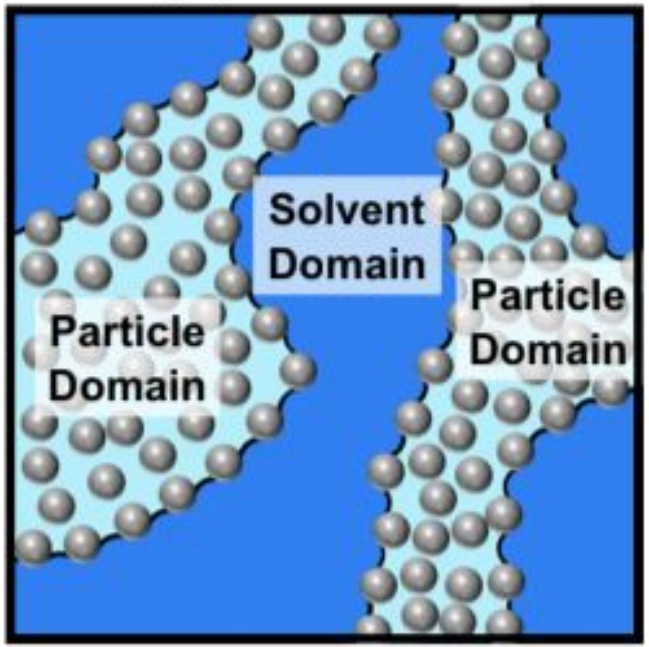
- Summer Undergraduate Research Fellowship
- National Institute of Standards and Technology
- NIST Center for Neutron Research
- Center for High Resolution Neutron Scattering
- Brookhaven Synchrotron Light Source II

Remove bijel half, include phase diagram for water/lut online

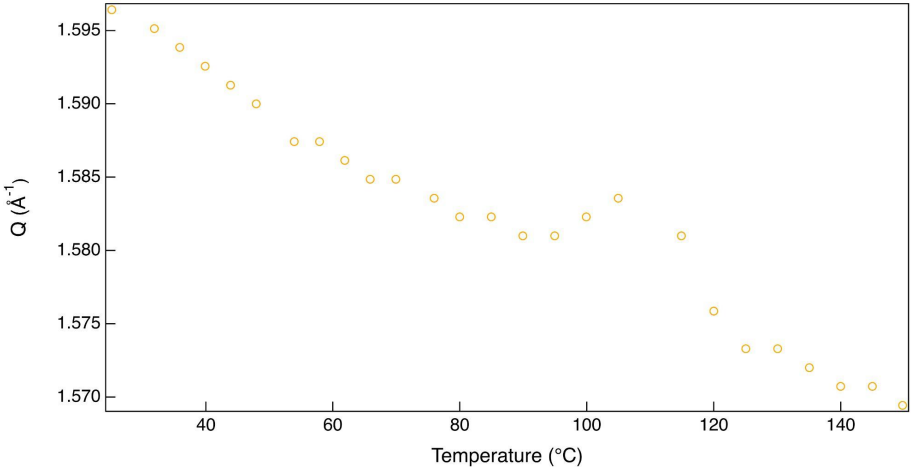
Bijel vs SeedGel

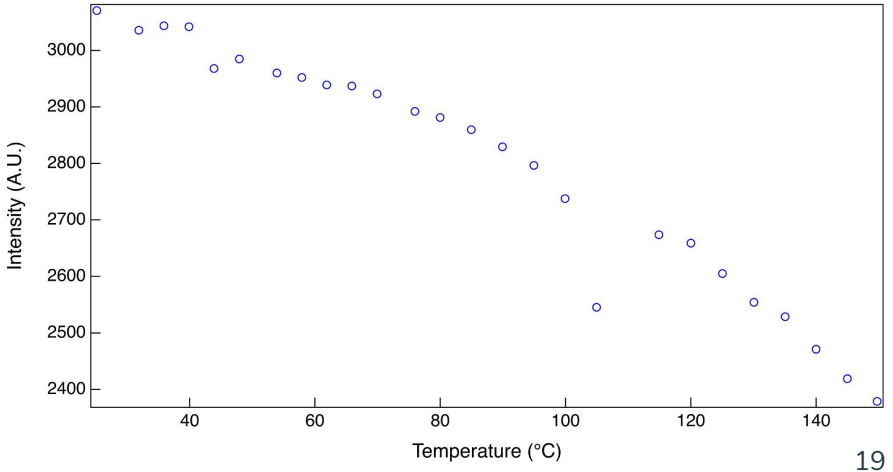
Reference





A4 WAXS Data Peaks

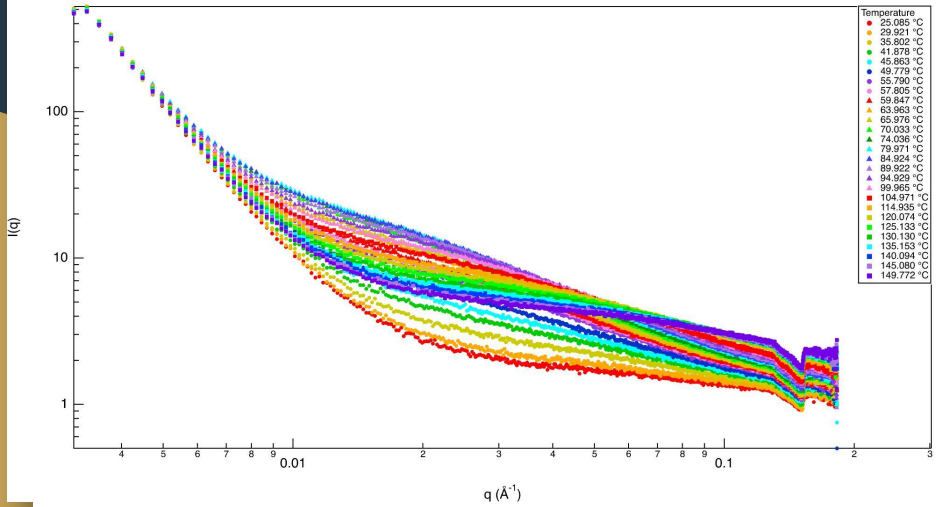


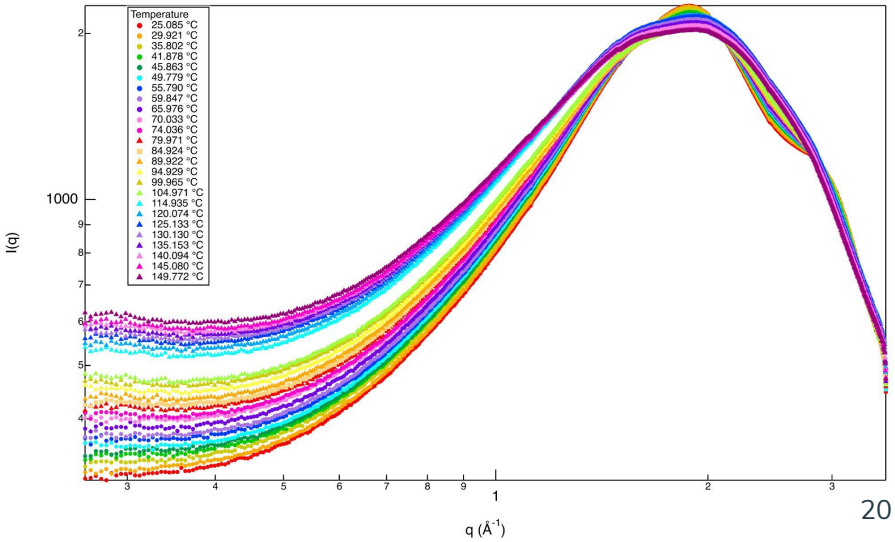


A1 - A binary solvent of 73.7 vol% H2O and 26.3 vol% H-3MP

Maybe skip

Go up to what temp?

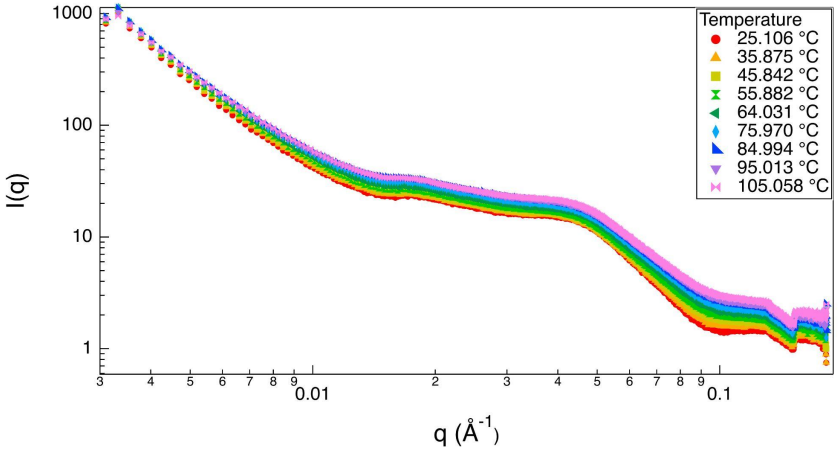


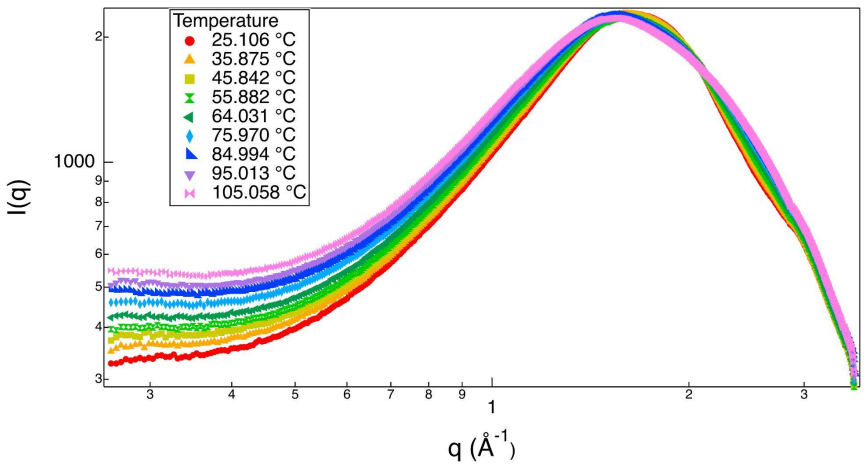


A3 - A binary solvent of 53.8 vol% H2O and 46.2 vol% H-3MP

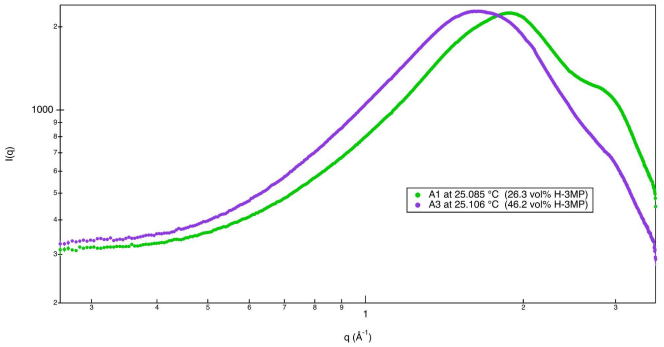
Add insets

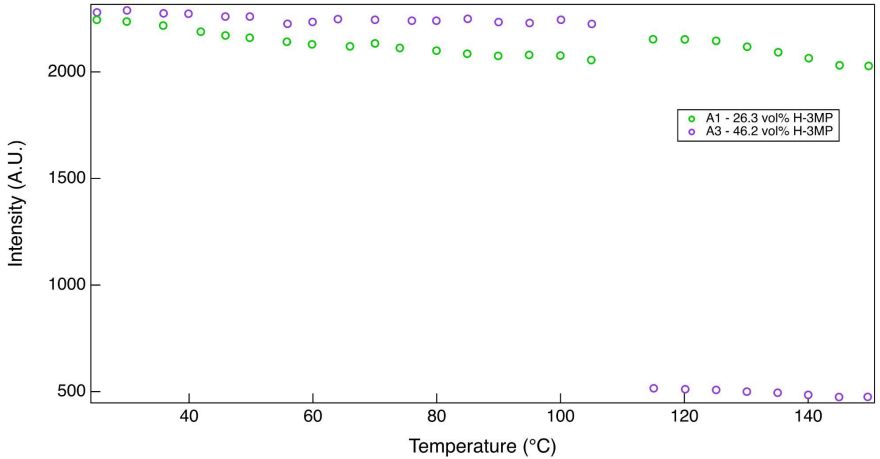
Maybe skips

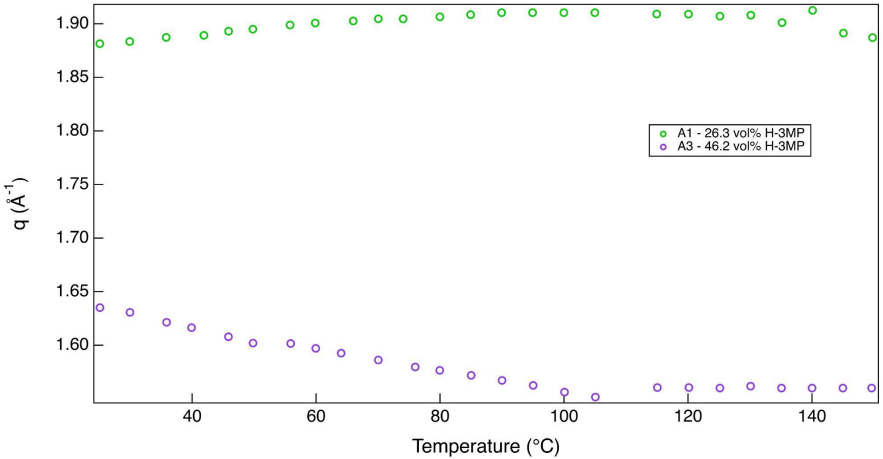




Solvent Comparison Graphs

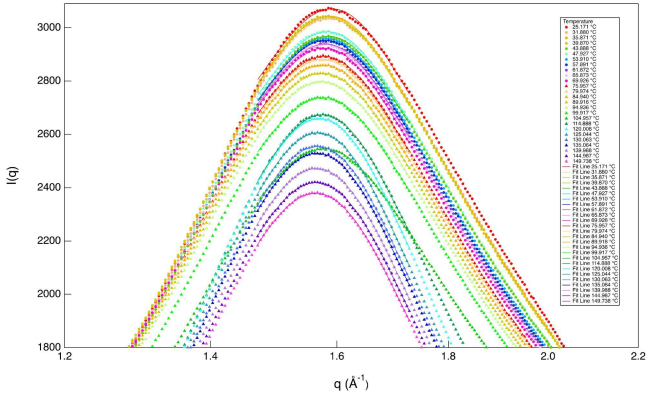




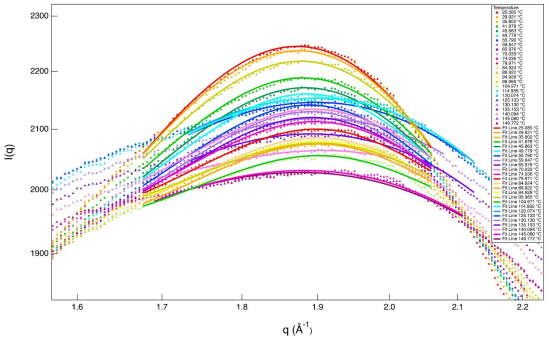


Gauss fittings to obtain the peak positions

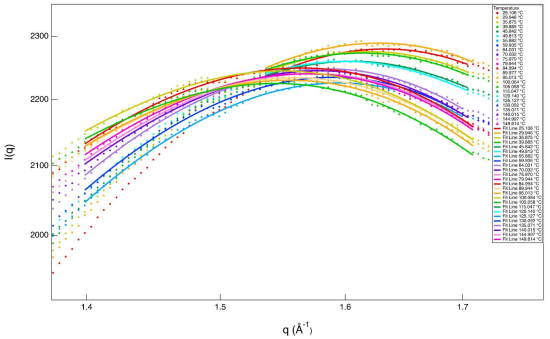
A4 - Gel



A1



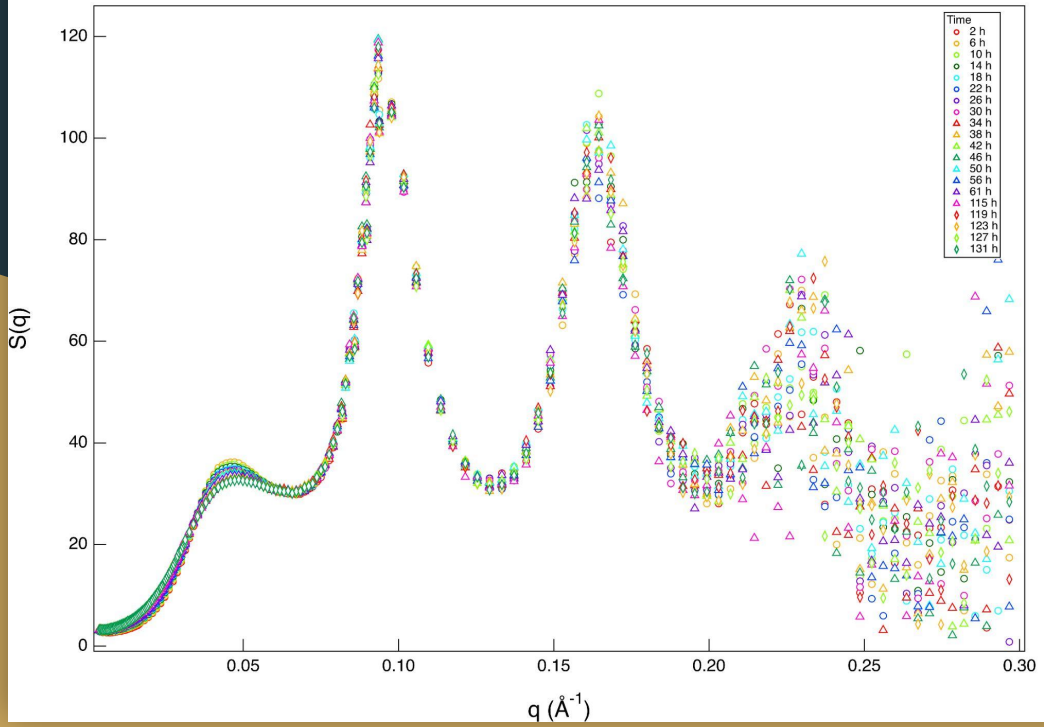
A3



C2

Add in graphs and comments

SM


Time
2 h
6 h
10 h
14 h
18 h
22 h
26 h
30 h
34 h
38 h
42 h
46 h
50 h
56 h
61 h
115 h
119 h
123 h
127 h
131 h
S(q)
q (Å^{-1})
0
20
40
60
80
100
120
0.05
0.10
0.15
0.20
0.25
0.30

Background

Keep at least journal name, page number
Include authors if enough space
Keep consistent throughout

To touch on:

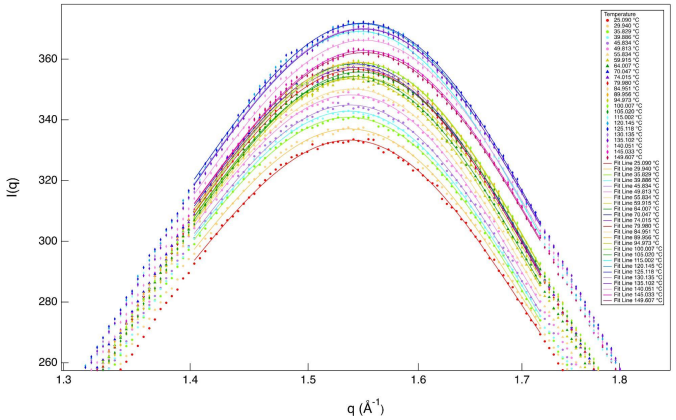
-reversibility

-gelation occurs with increase in temp

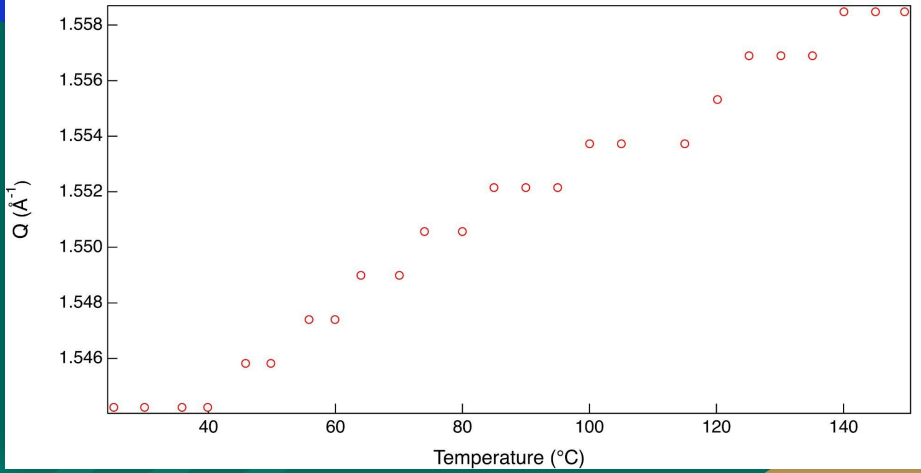
-

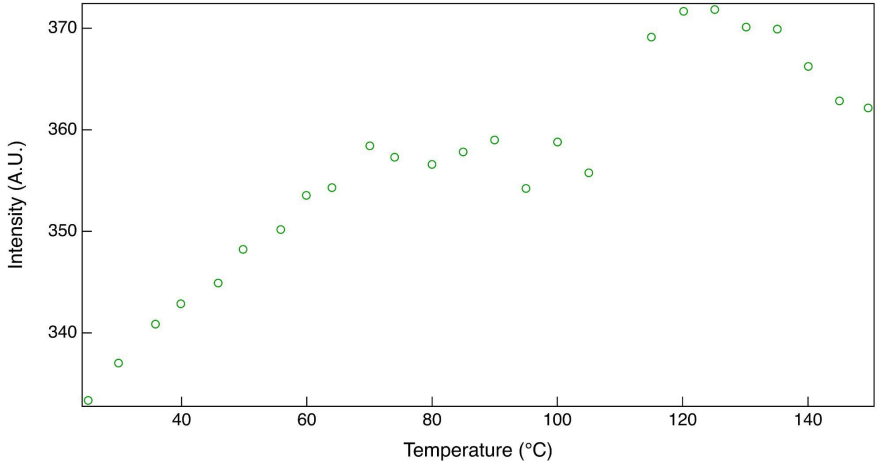
Gauss Fitting for solvents

Q vs Temp for WAXS data peaks



A2





Previous Findings

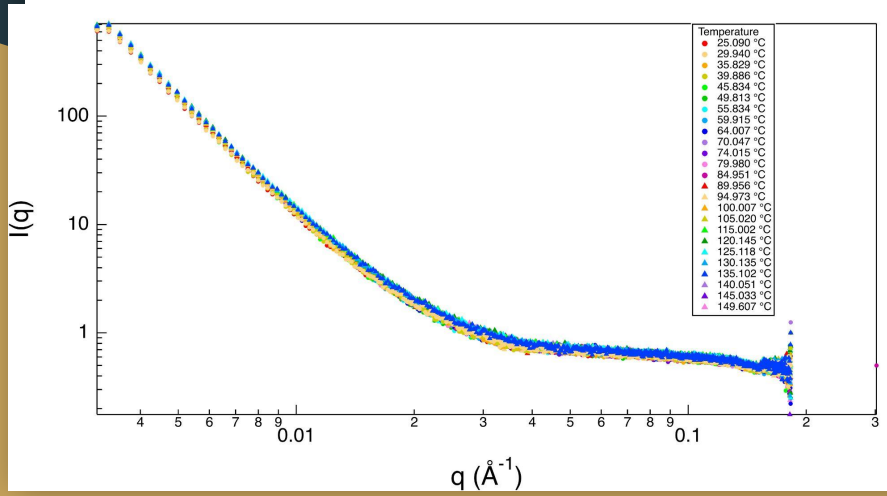
- Transition temp of 26C

-So what were we looking for within the structure factor

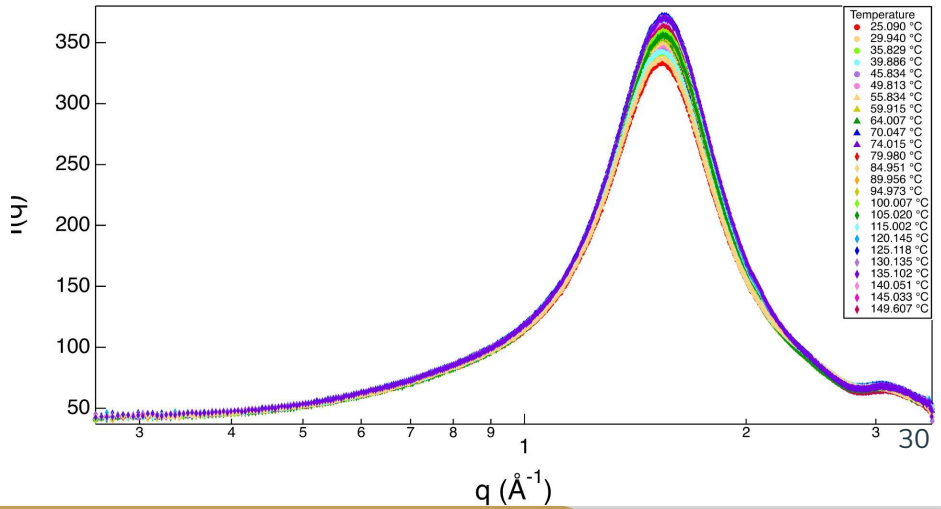
A2- A binary solvent of 63.6 vol% H2O and 36.4 vol% H-3MP

Possibly skip?

SAXS



WAXS Data

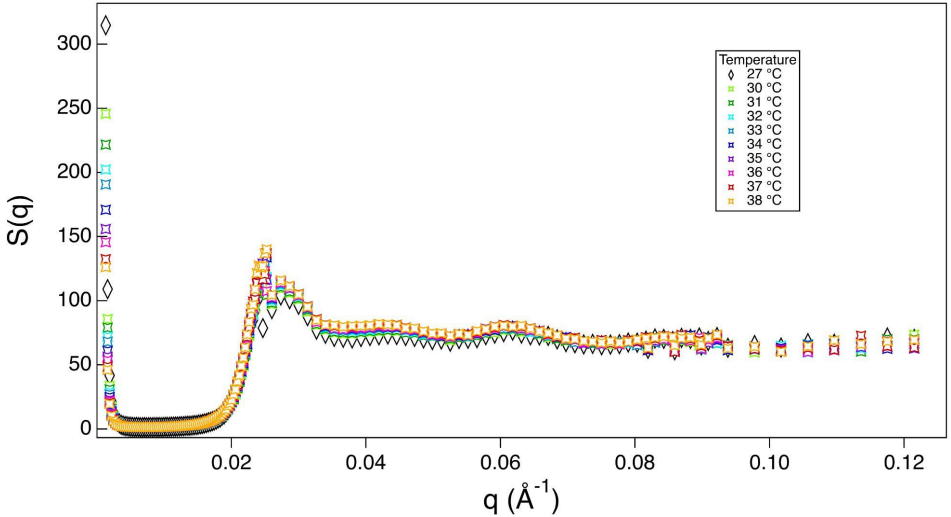


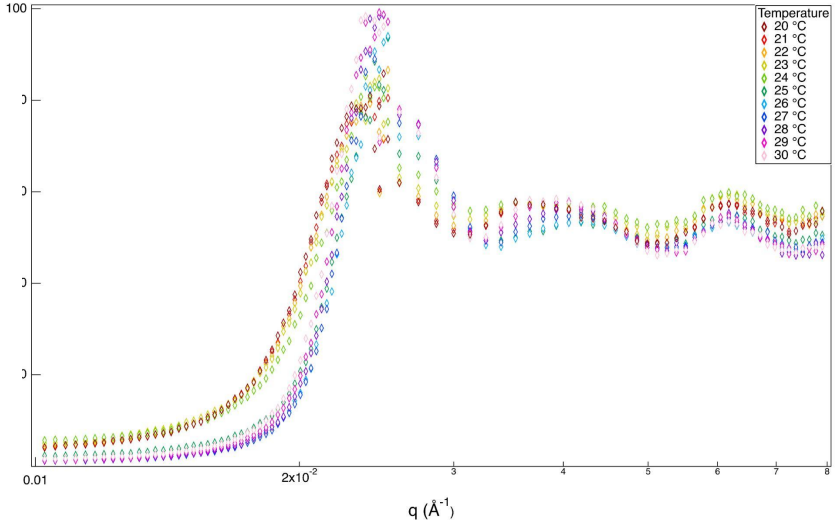
Correct: S(q)*contrast

TM

Which TM graphs to use?

Ignore



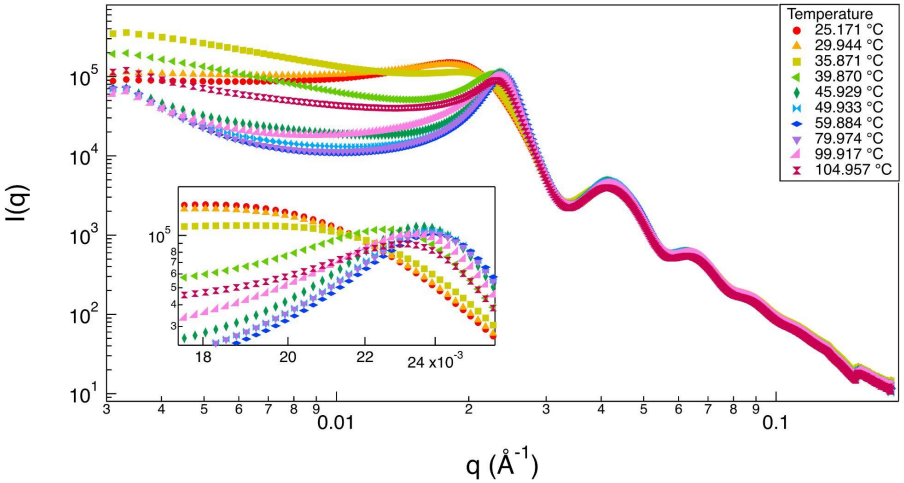


A4 - 22.7 vol% Particle and 77.3 vol% binary solvent (63.6 vol% H2O and 36.4 vol% H-3MP)

Draw line for B4 vs A4 comp q

Remove WAXS bc not including further analysis

SAXS Data



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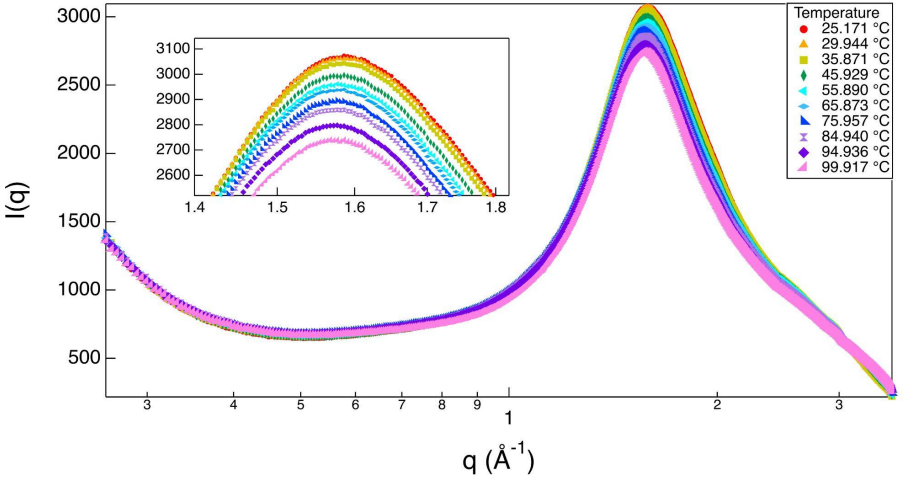
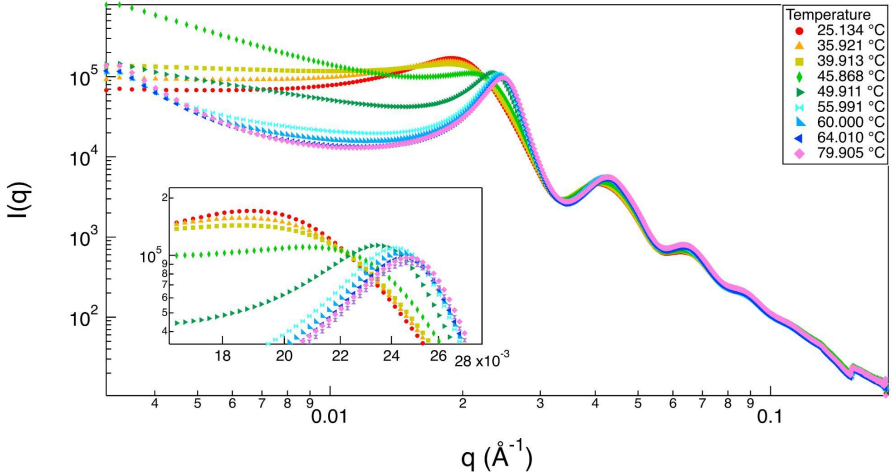


B4 - 22.7 vol% Particle and 77.3 vol% binary solvent (63.6 vol% H2O and 36.4 vol% D-3MP)

SAXS Data



WAXS Data