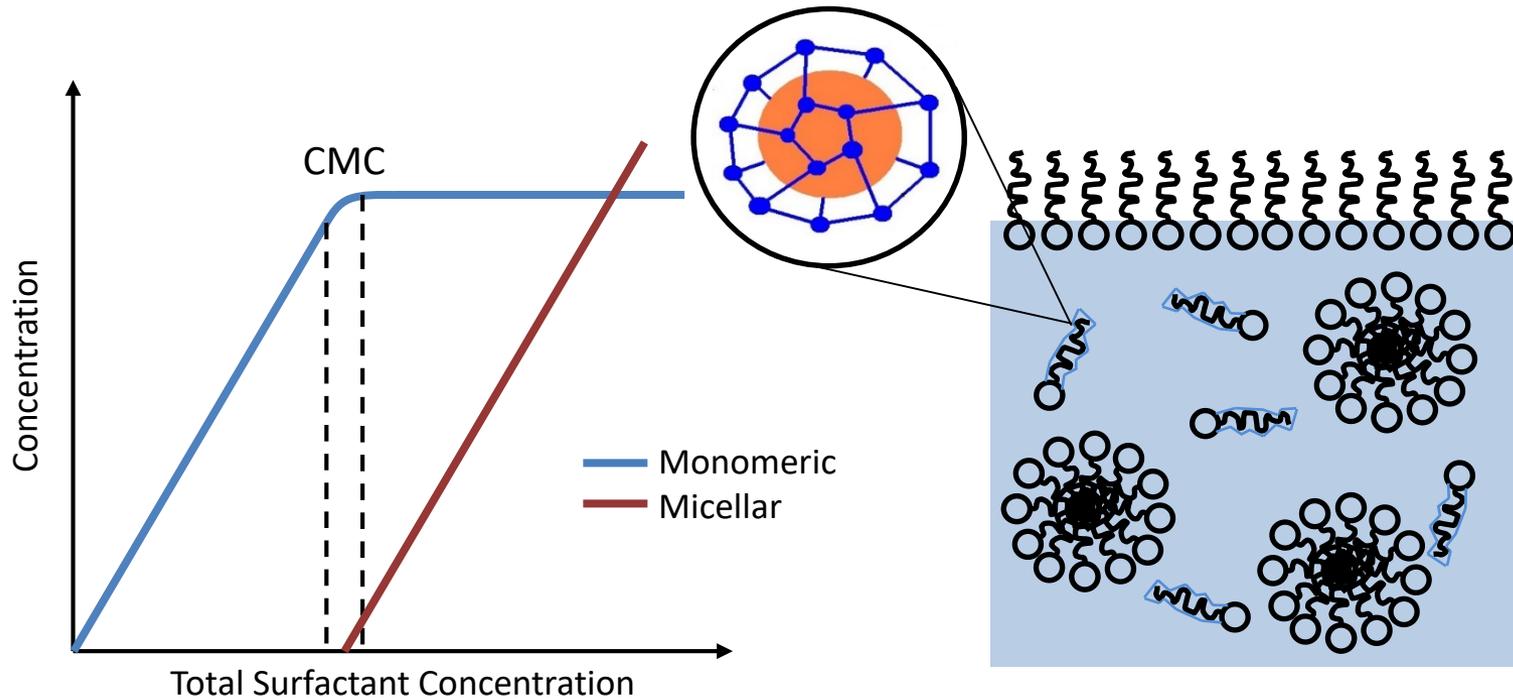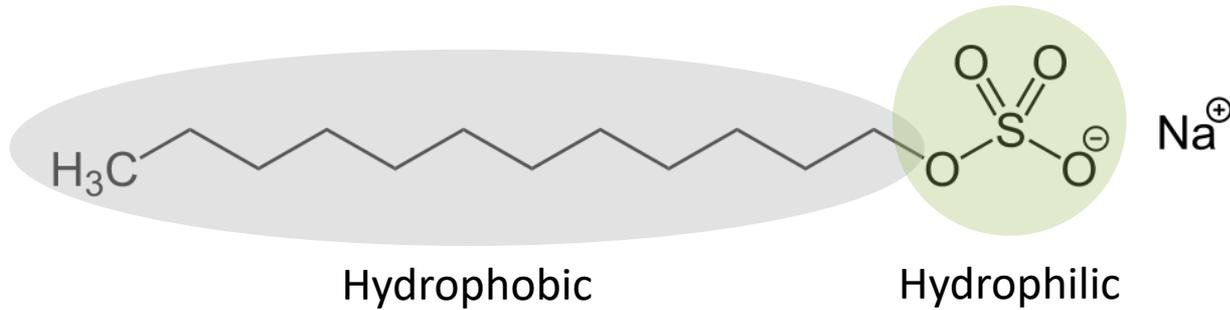# Structural and Mechanical Characterization of HPMC/SDS Aggregation through Rheological and Neutron Scattering Measurements

Douglas Scott
NCNR SURF Associate
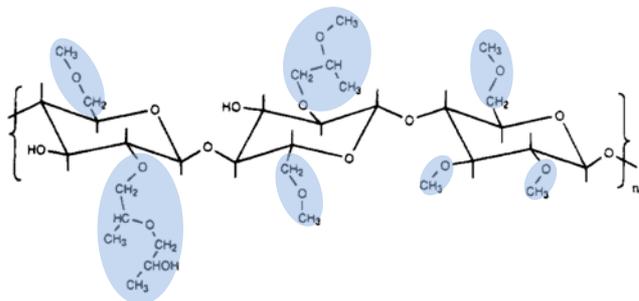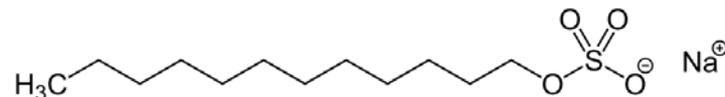
Surface Active Agents & Micellization

Hydrophobic

Hydrophilic

CMC

Concentration

Total Surfactant Concentration

Monomeric
Micellar
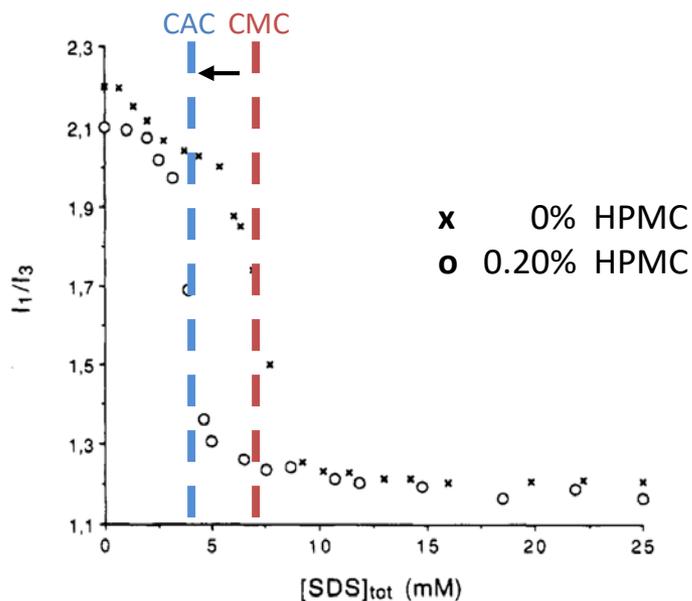
Polymer-Surfactant Interactions

Hydroxypropylmethyl cellulose (HPMC)

Sodium dodecyl sulfate (SDS)

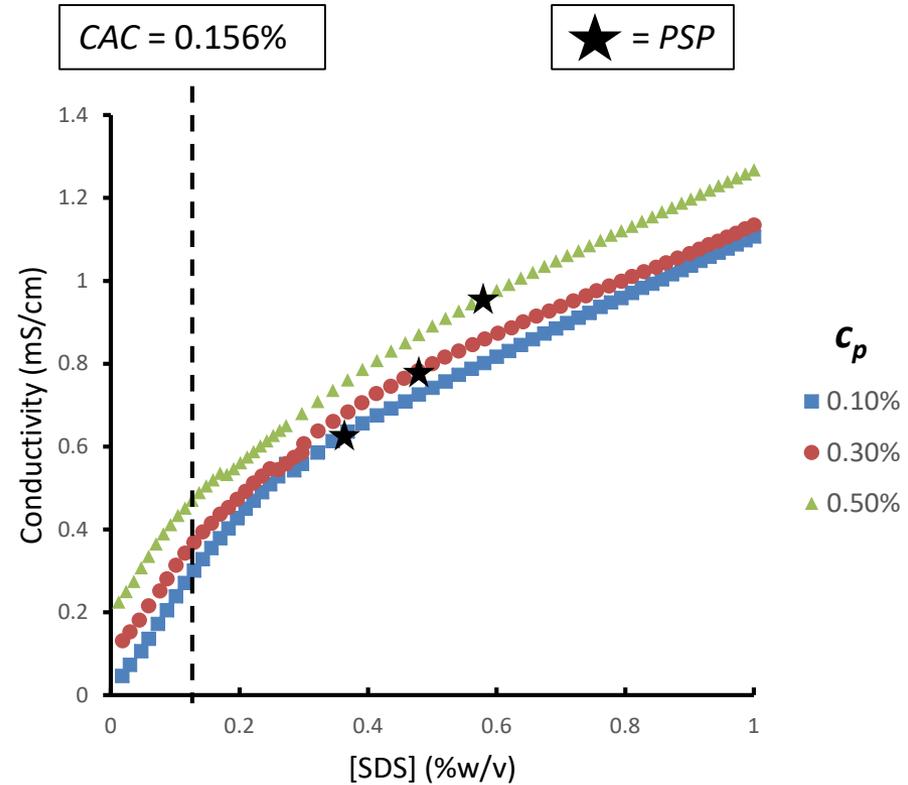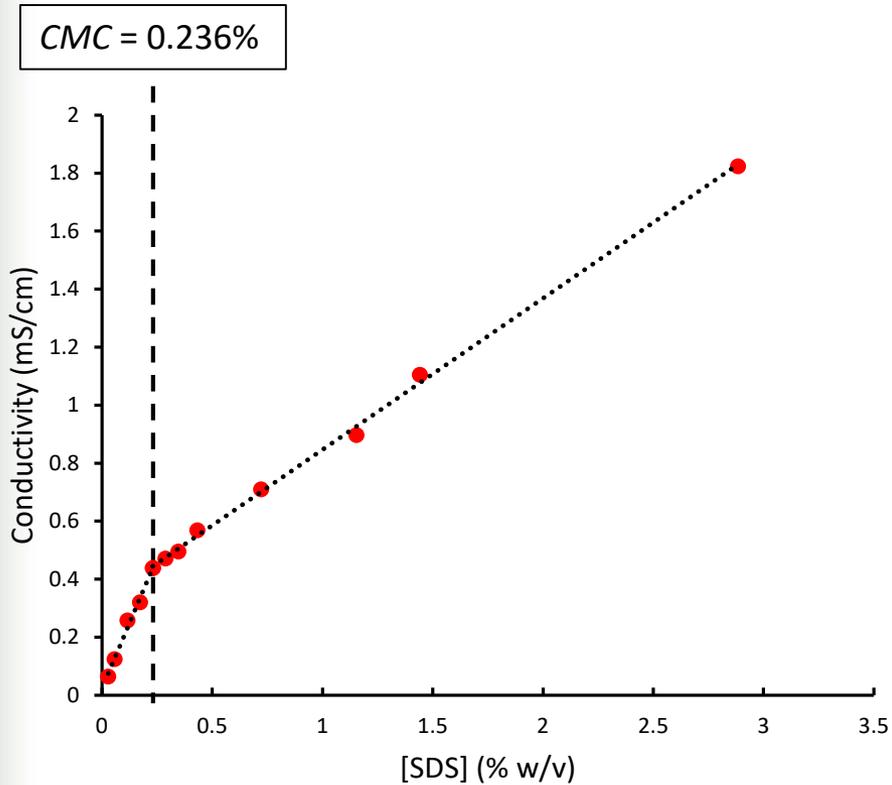## Aggregation at Lower Surfactant Concentrations

Practical Rheological Properties

CAC   CMC

x   0%   HPMC
o   0.20%   HPMC

I_1/I_3 vs $[SDS]_{tot}$ (mM)

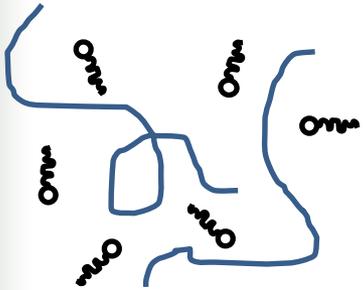Nilsson, S., *Macromolecules* **1995,** *28* (23), 7837-7844.
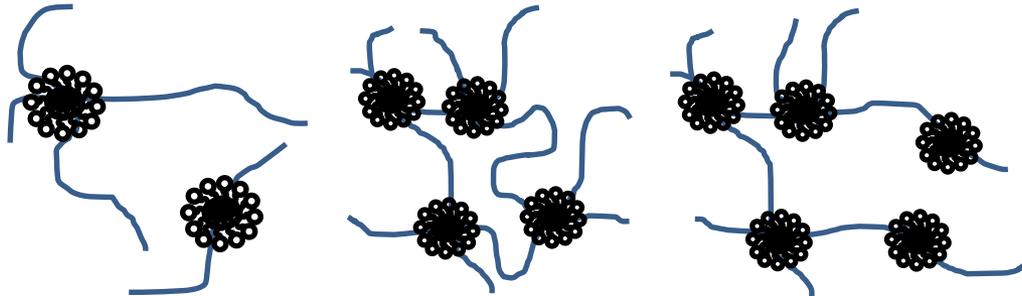
Conductivity Measurements

CMC = Critical Micelle Concentration
CAC = Critical Aggregation Concentration
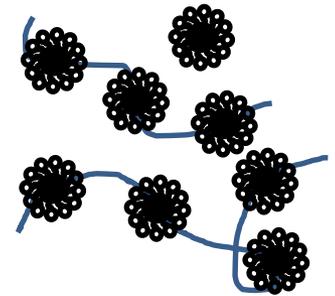PSP = Polymer Saturation Point

Proposed Aggregate Structures

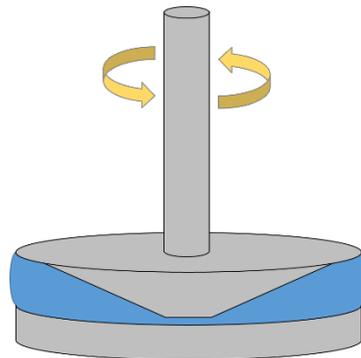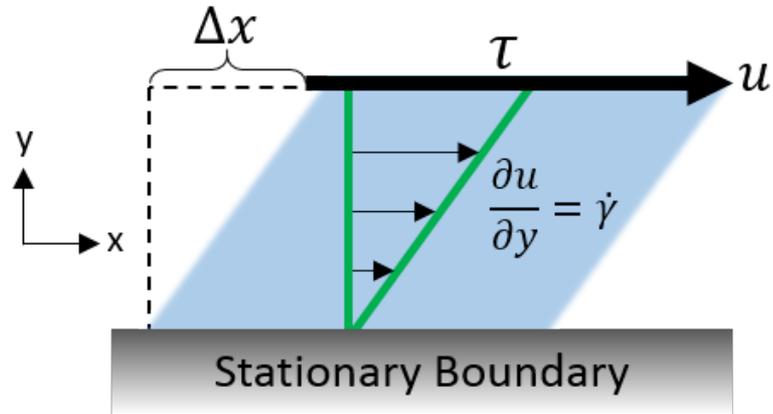$[SDS]_{tot} < CAC$ $CAC < [SDS]_{tot} < PSP$ $[SDS]_{tot} > PSP$

Increasing SDS Concentration

CMC = Critical Micelle Concentration
CAC = Critical Aggregation Concentration
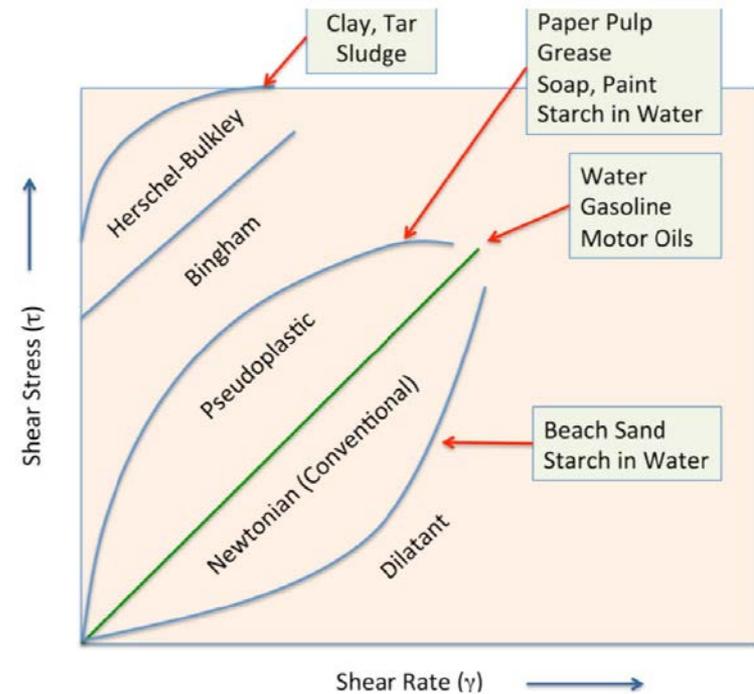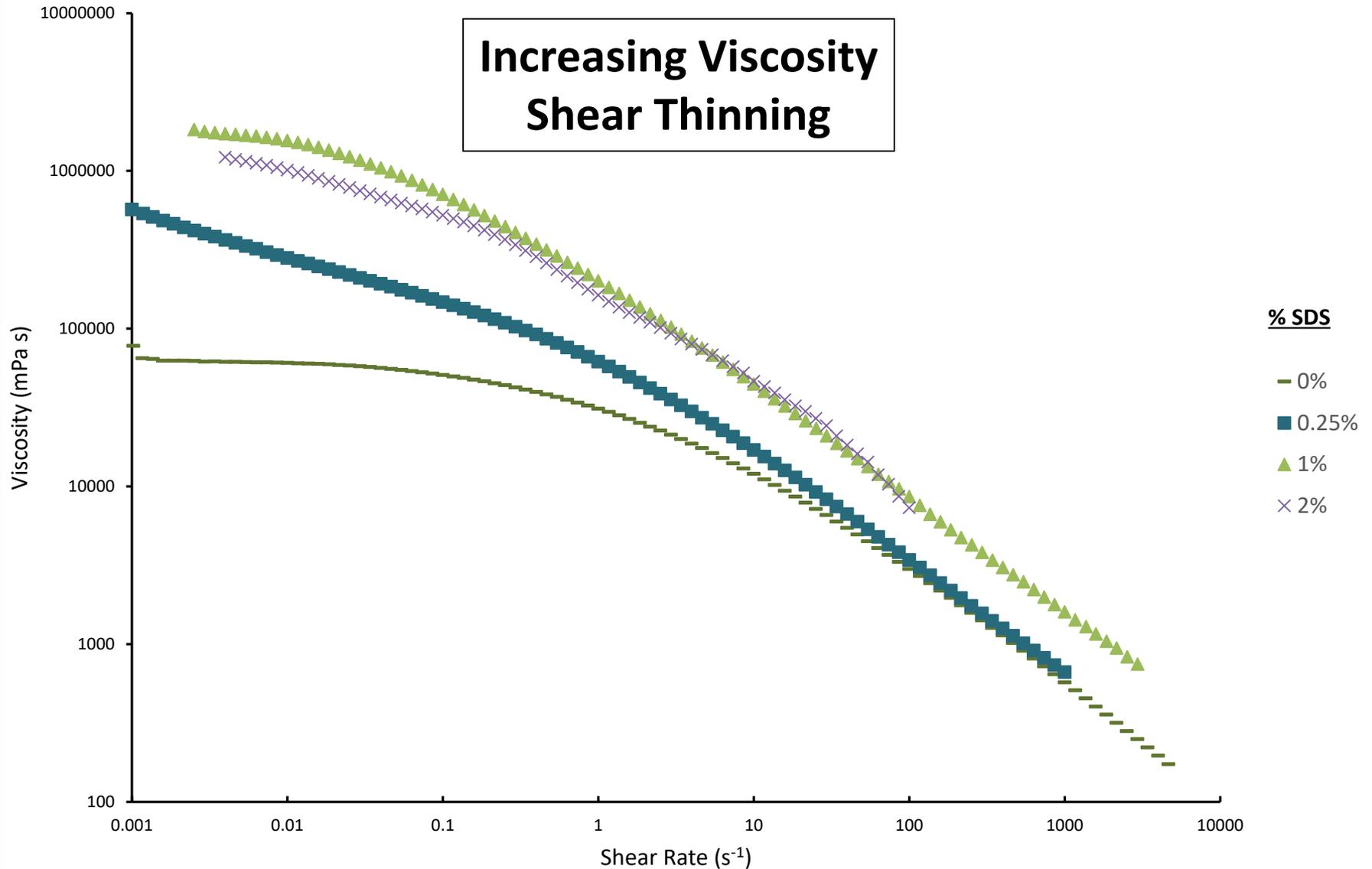**PSP** = Polymer Saturation Point
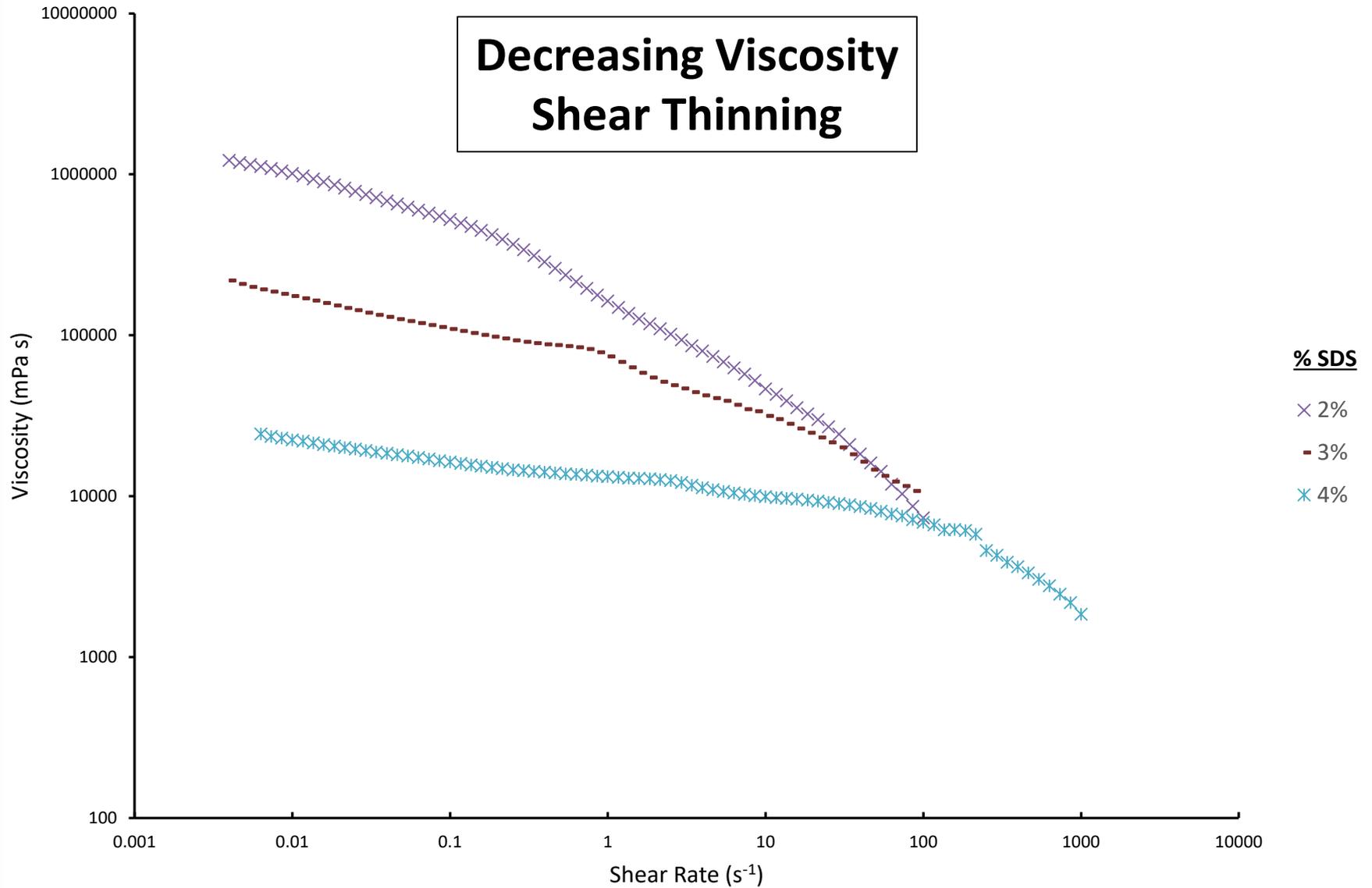
Rheology 101

ῥέω, "flow"

Viscosity
$$\tau = \eta\dot{\gamma}$$

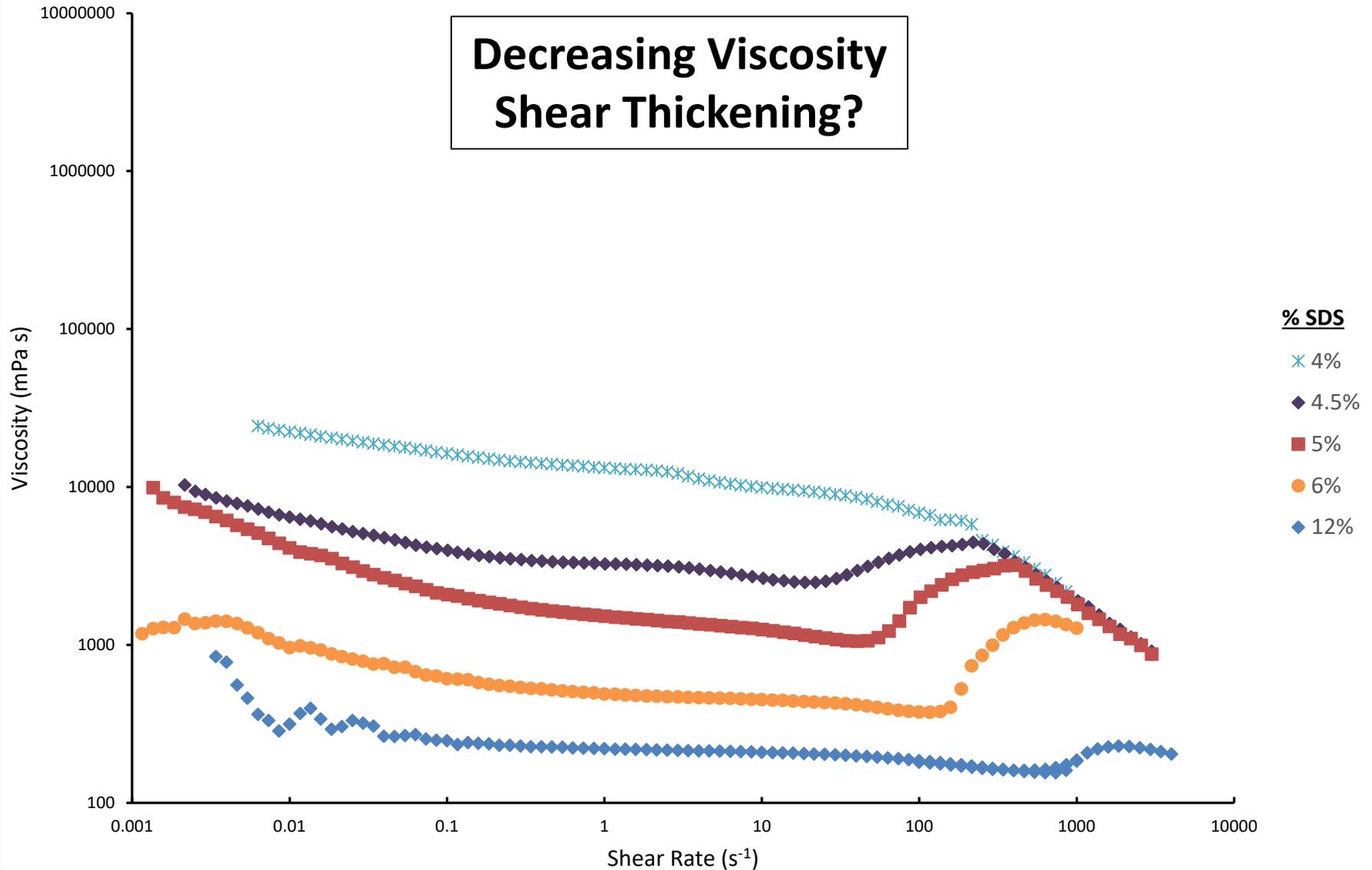Image from: materialssolutions.com.au

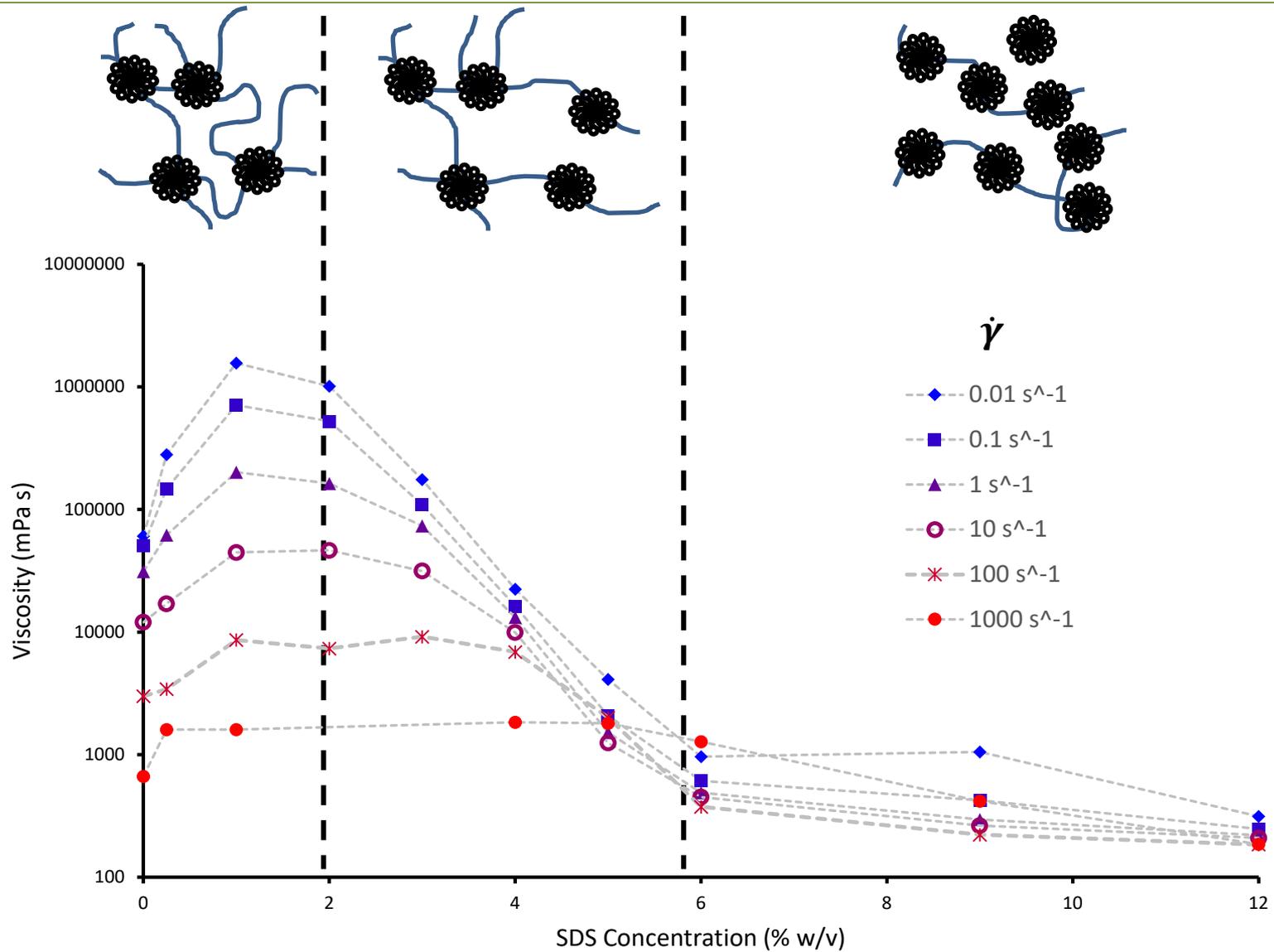# Shear Profiles: 3% HPMC Solutions with SDS
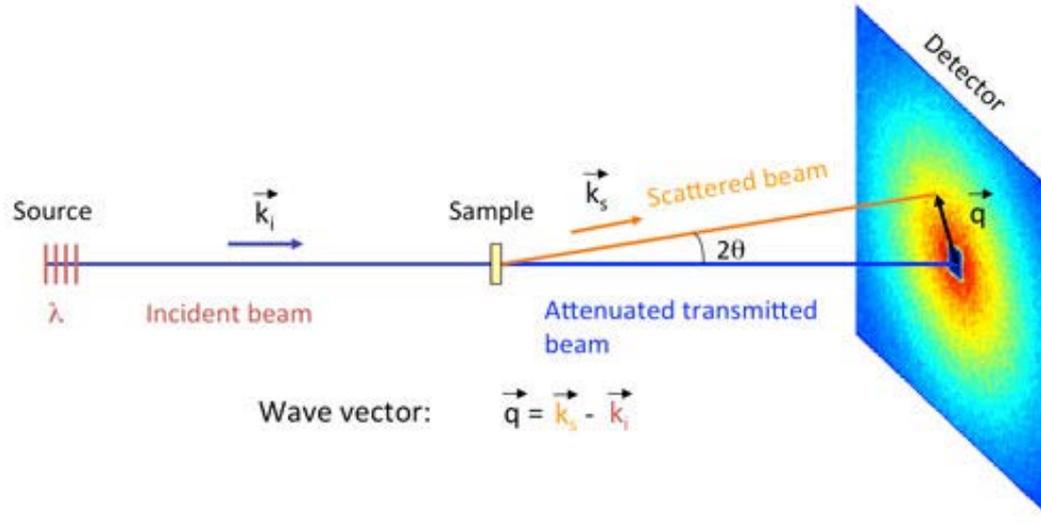
Shear Profiles: 3% HPMC Solutions with SDS

Shear Profiles: 3% HPMC Solutions with SDS

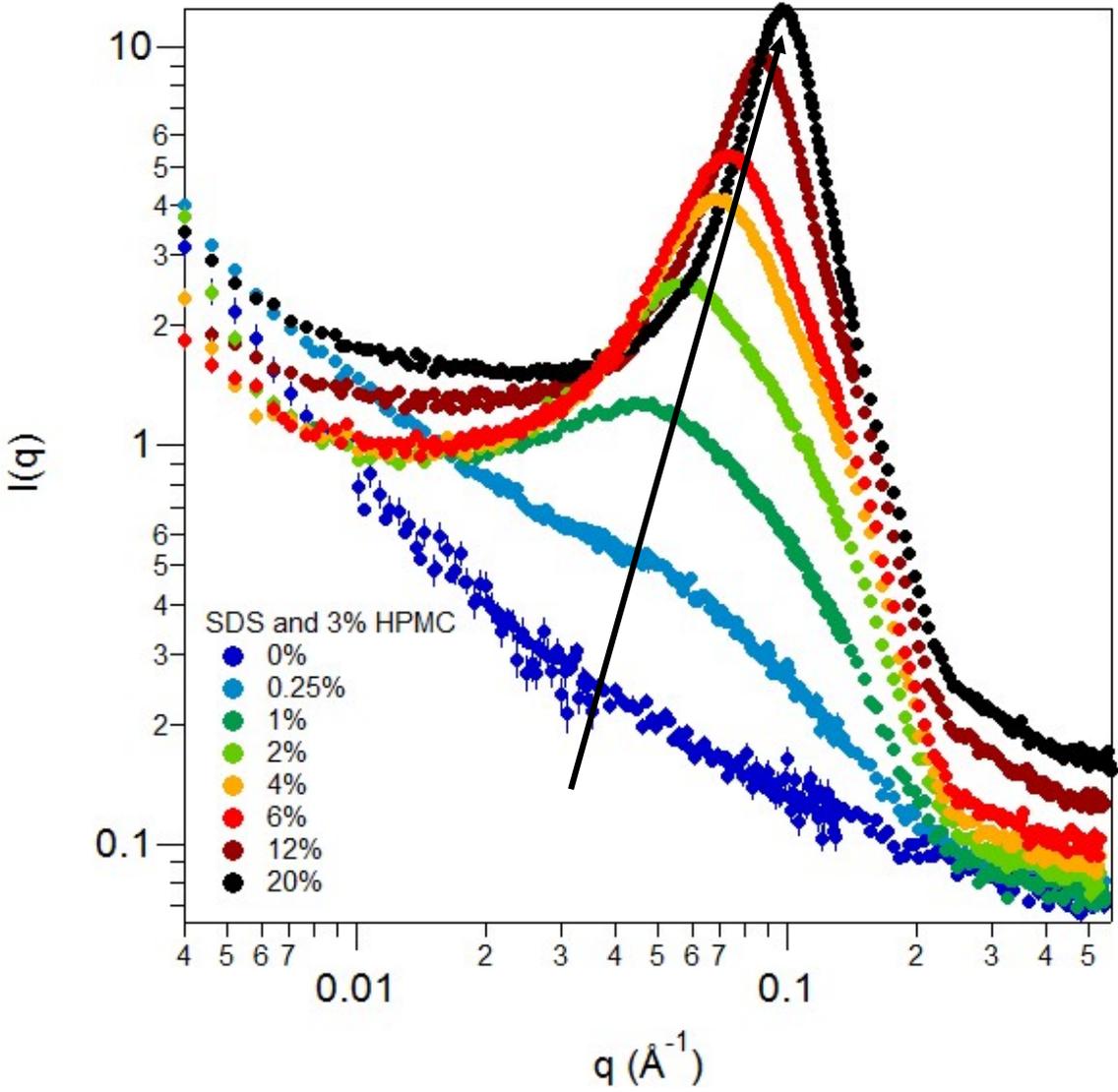# Viscosity Dependence on Concentration

Small Angle Neutron Scattering (SANS) 101

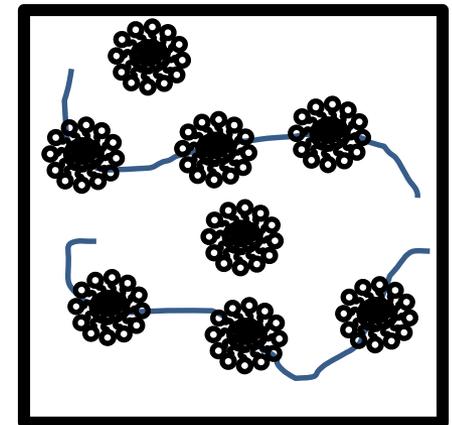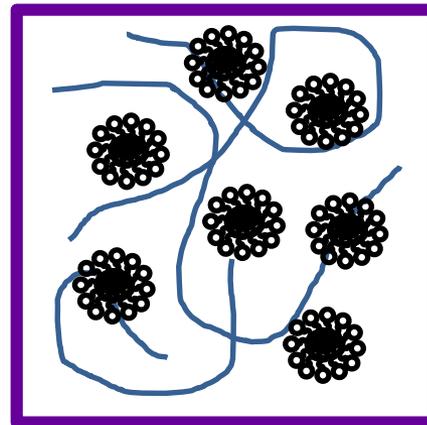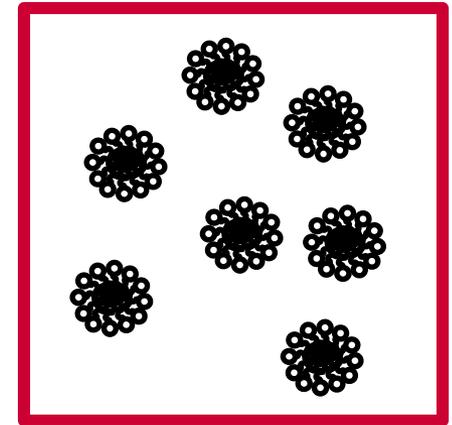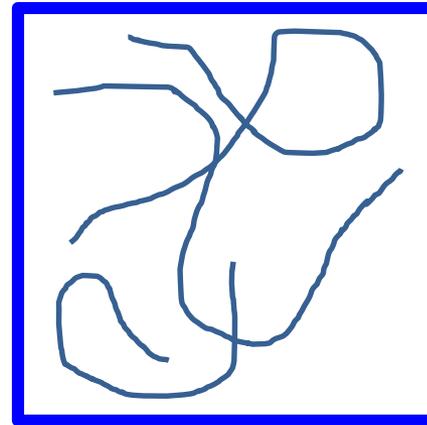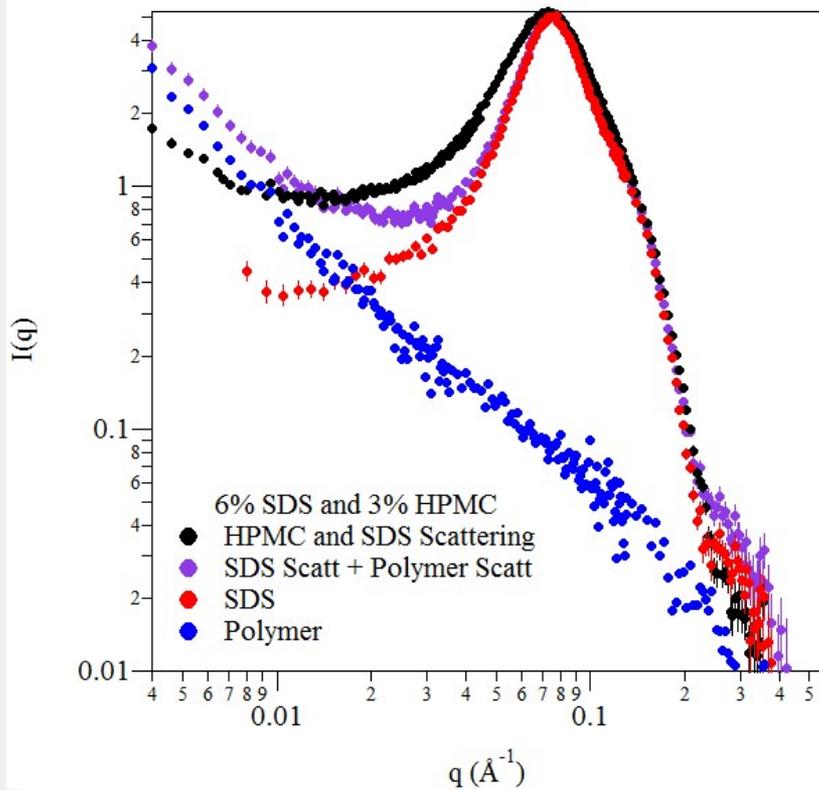$$Q = \frac{4\pi}{\lambda} \sin \theta$$

$$I(Q) = \frac{d\Sigma(Q)}{d\Omega} = \left(\frac{N_A}{V}\right)(\rho_A - \rho_B)^2 {V_A}^2 P(Q)\, S_I(Q)$$

cross section	number density	contrast factor	particle volume	form factor	structure factor

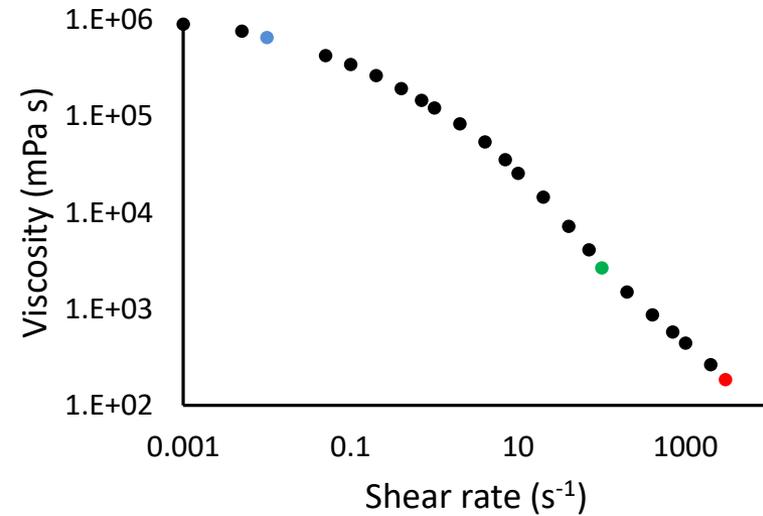Grillo, I. *Small-Angle Neutron Scattering and Applications in Soft Condensed Matter*; 2008
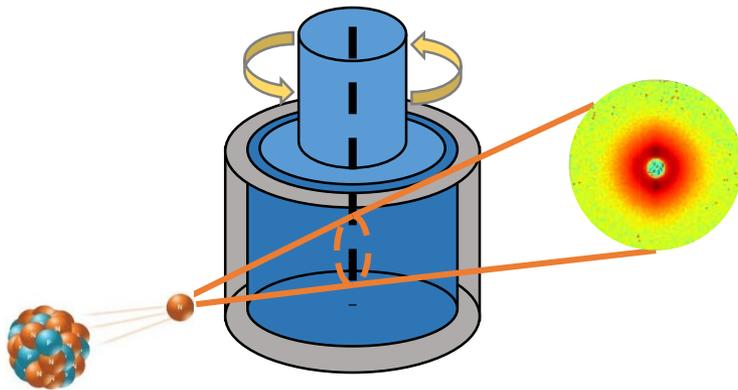
Static SANS: HPMC/SDS Mixtures

Static SANS: More than the sum of its parts…

RheoSANS

$$\dot{\gamma} = 0.01 \ s^{-1} \qquad \dot{\gamma} = 100 \ s^{-1} \qquad \dot{\gamma} = 3000 \ s^{-1}$$
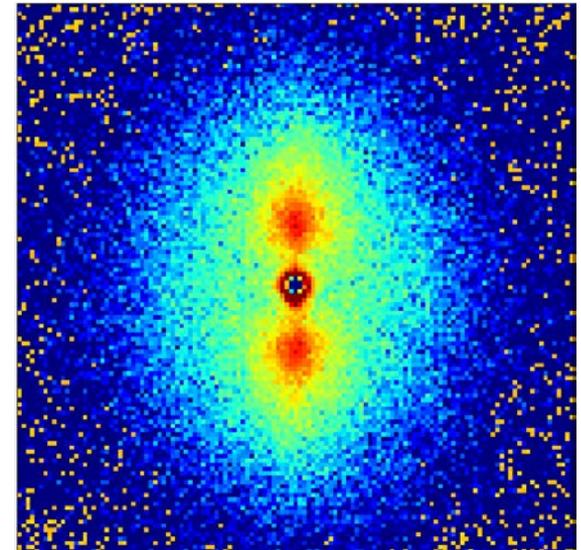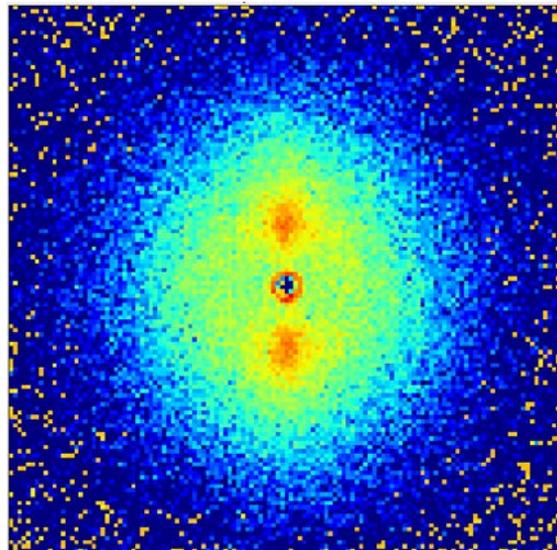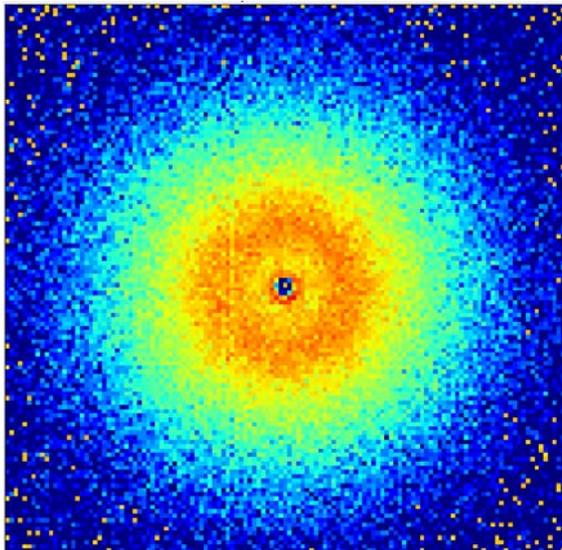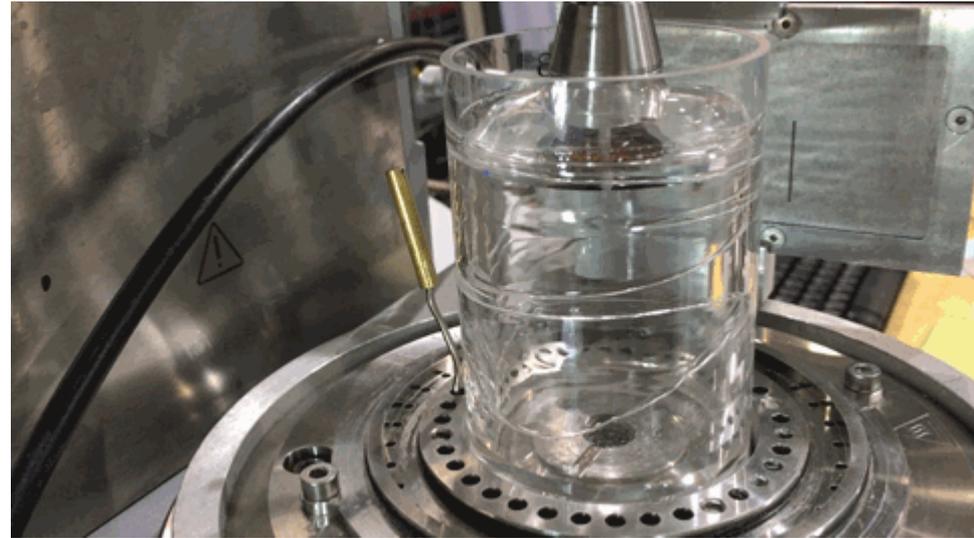
Shear Thickening or Foaming?

- 4.5%
- 5%
- 6%
- 12%

Viscosity (mPa s)

Shear Rate (s^{-1})

*Videos courtesy of Daniel Seeman
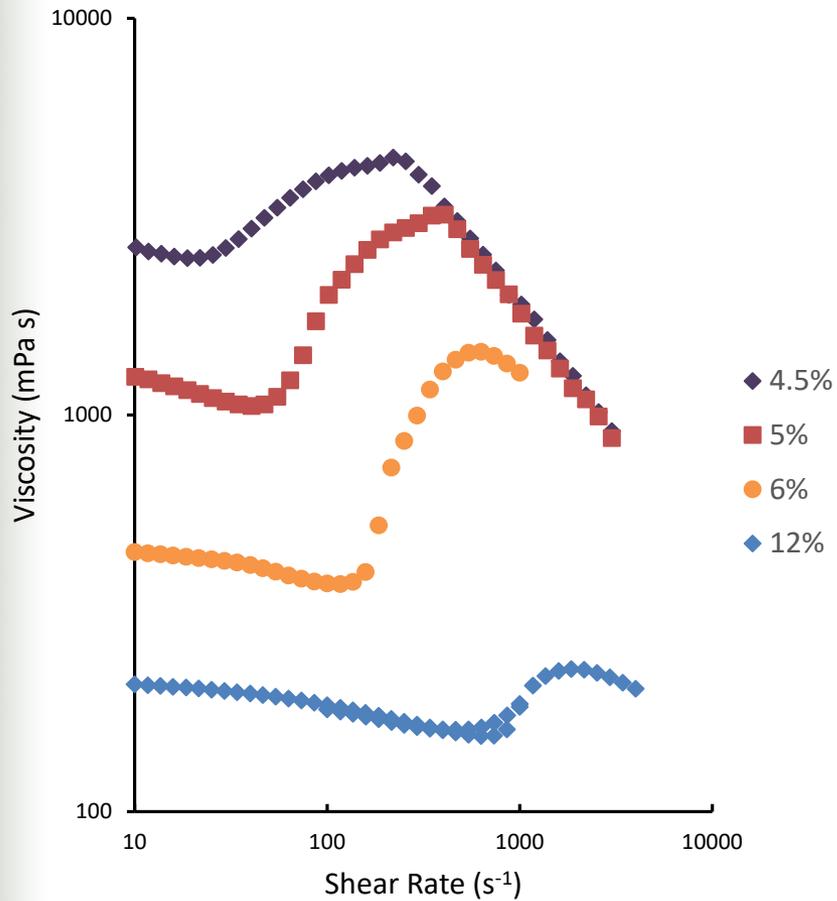
Looking Forward

Contrast Matching

Deuterate SDS

Enclosed Rheology

2D Detector

Sample Ports

Neutron Beam

Quartz Window

N. Wagner (UD)
M. Liberatore (UD)
F. Nettesheim (UD)
L. Porcar (NIST)

Extensional Flow

K. Weigandt (NIST)
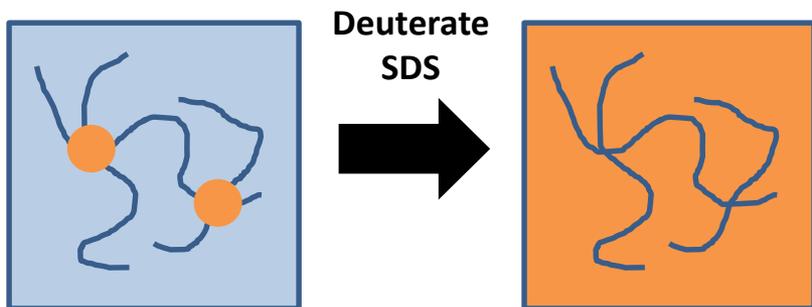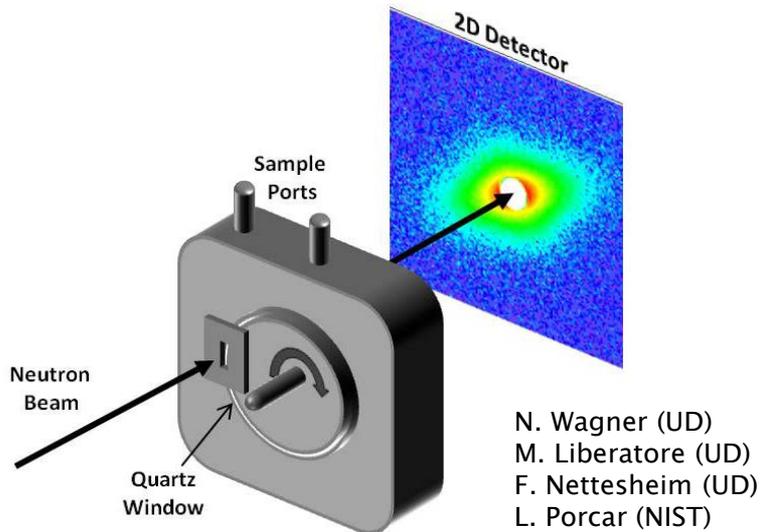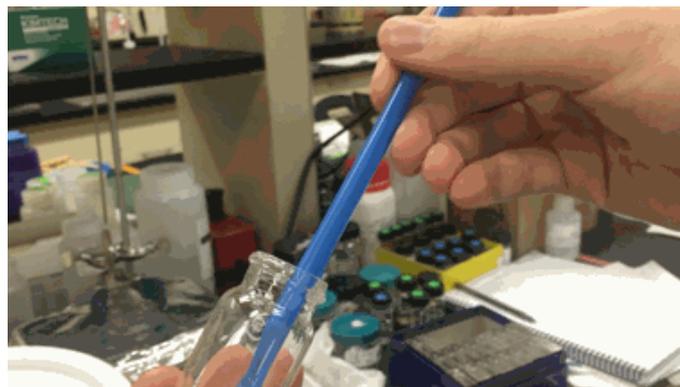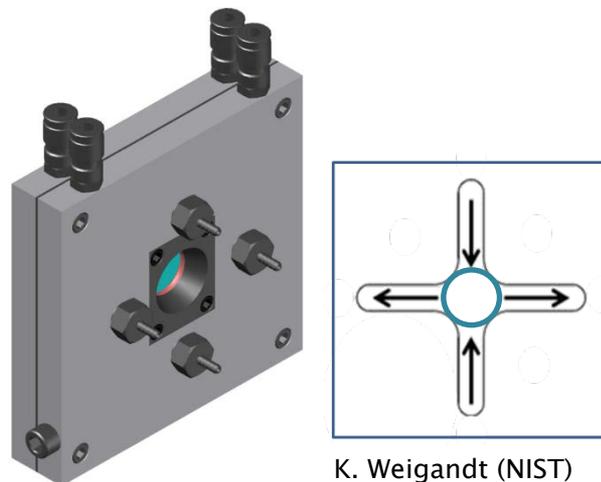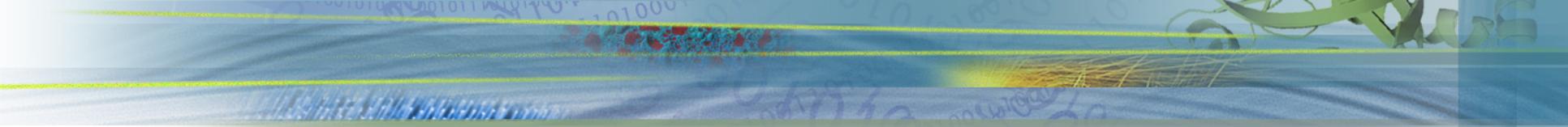
Acknowledgements

- Katie Weigandt
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  - Joe Dura
  - Julie Borchers

- Proctor & Gamble
  - Mark Hammersky
  - Seth Lindberg

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Thank you for listening!

Questions?

NIST
National Institute of
Standards and Technology
U.S. Department of Commerce