Probing Ion Distributions in Charged Polymers Using Neutron, Light, and X-Ray Scattering

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Paper draft

PennState
College of Earth and Mineral Sciences

Presentation Outline

- Counterion condensation: the central problem in polyelectrolyte physics
- Light scattering as a tool to study counterion condensation
- Beyond the two-phase model: insight from SANS and SAXS into counterion distributions

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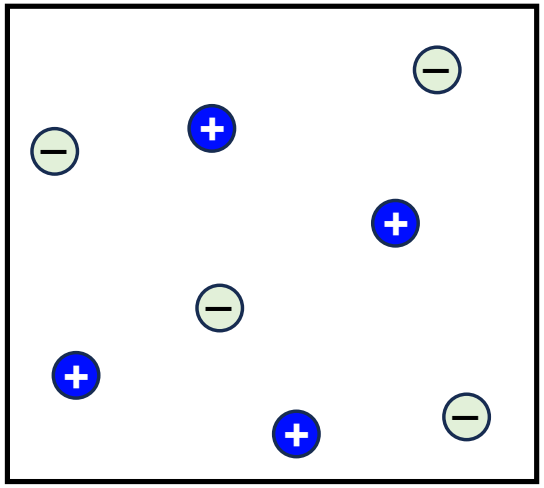
Solutions of simple salts

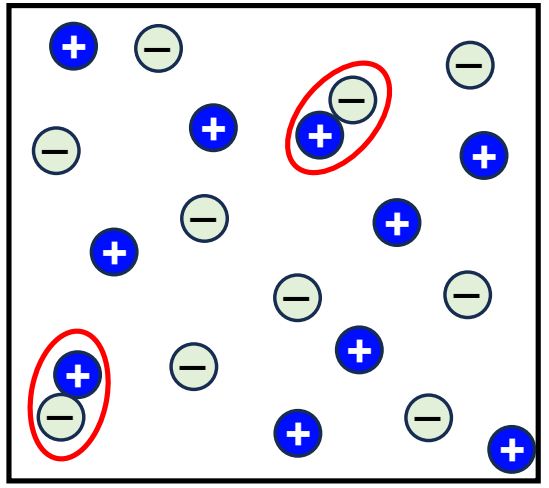
dilute



Individual ions

In distribution is described by Debye-Hückle theory (simple physics)

concentrated



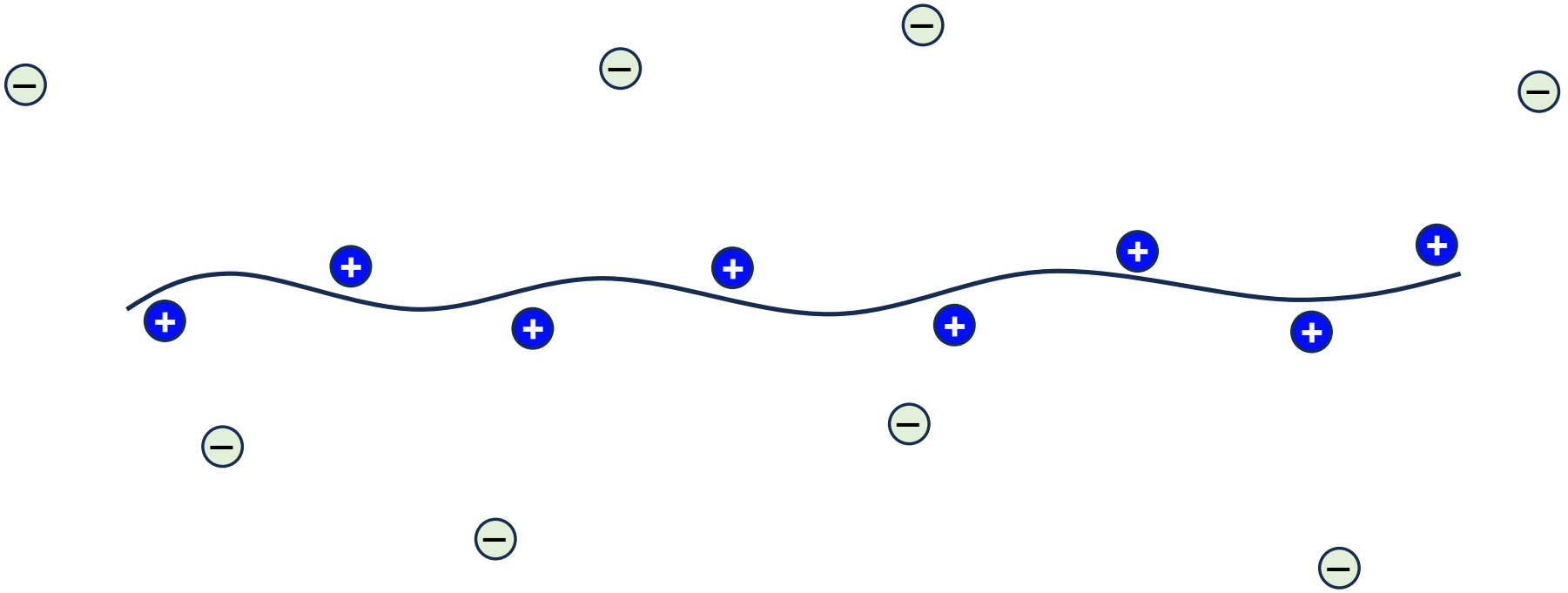
Ion-pair formation

Ion-pairing is set by solvation shells and follows a mass-action law (chemistry becomes important)

Marcus, Y. and Hefter, G., 2006. Ion pairing. *Chemical reviews*, *106*(11), pp.4585-4621.

Polyelectrolyte solutions

Polyelectrolytes are polymers with ionic groups along their backbone



Side groups are chemically fixed onto the backbone → no mobility

Polyelectrolyte solutions

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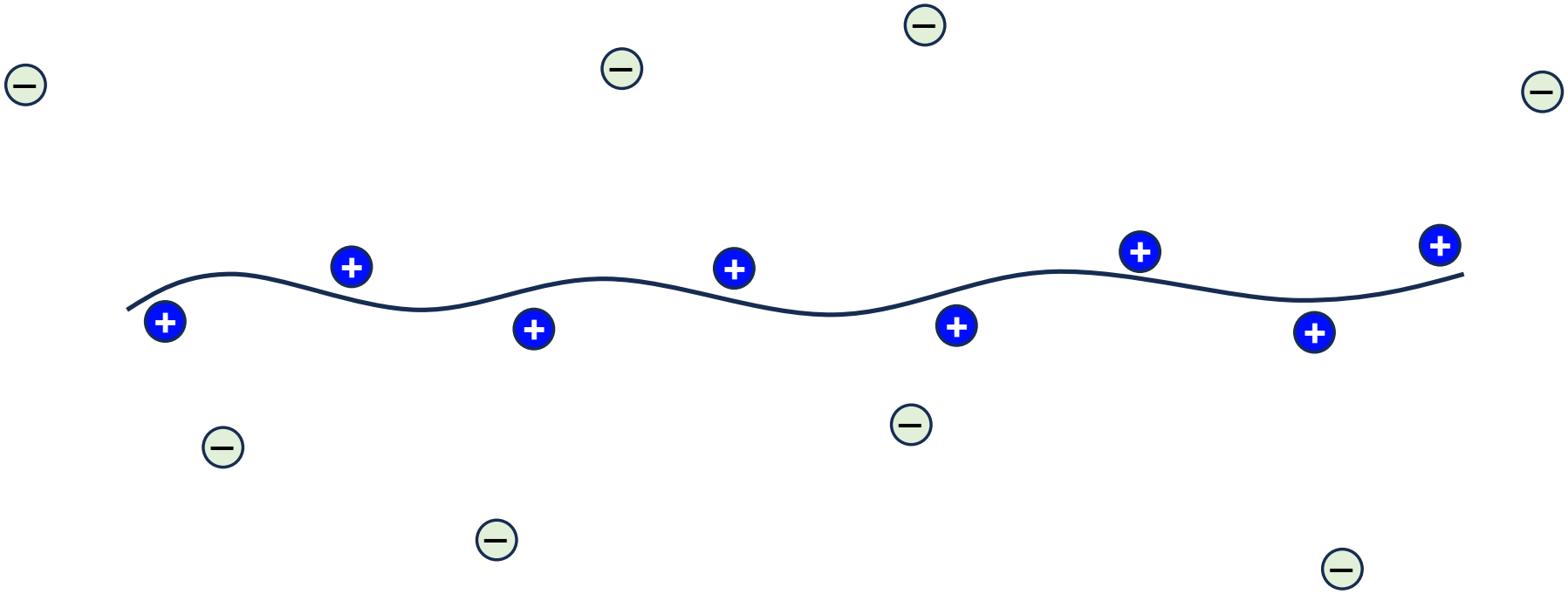
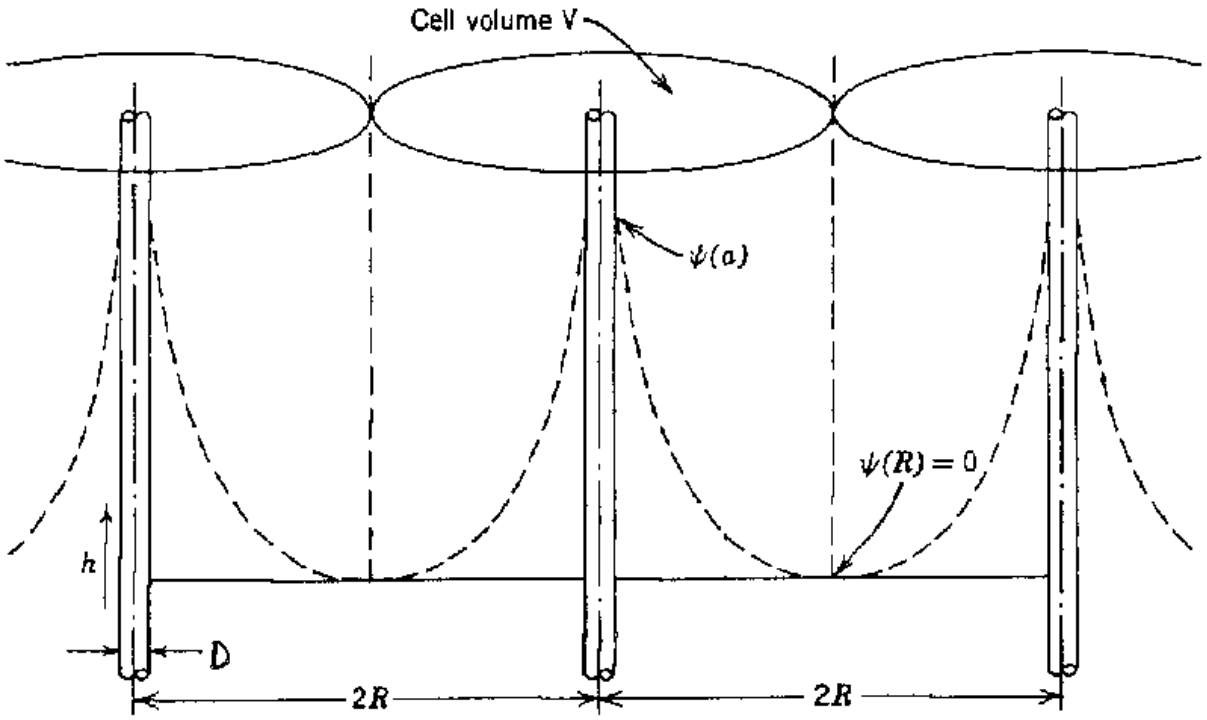


Already in the 1940s experimentalists noticed that the were ‘missing counterions’

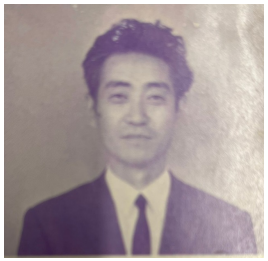
Katchalsky's cell model



Aharon Katchalsky
(1914-1972)

Counterions accumulate near the polyion and become **osmotically inactive**

Polyelectrolytes:
Oosawa-Manning theory of condensation



Fumio Oosawa
(1922-2019)

Gerald Manning
(b.19??)

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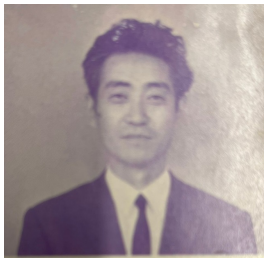

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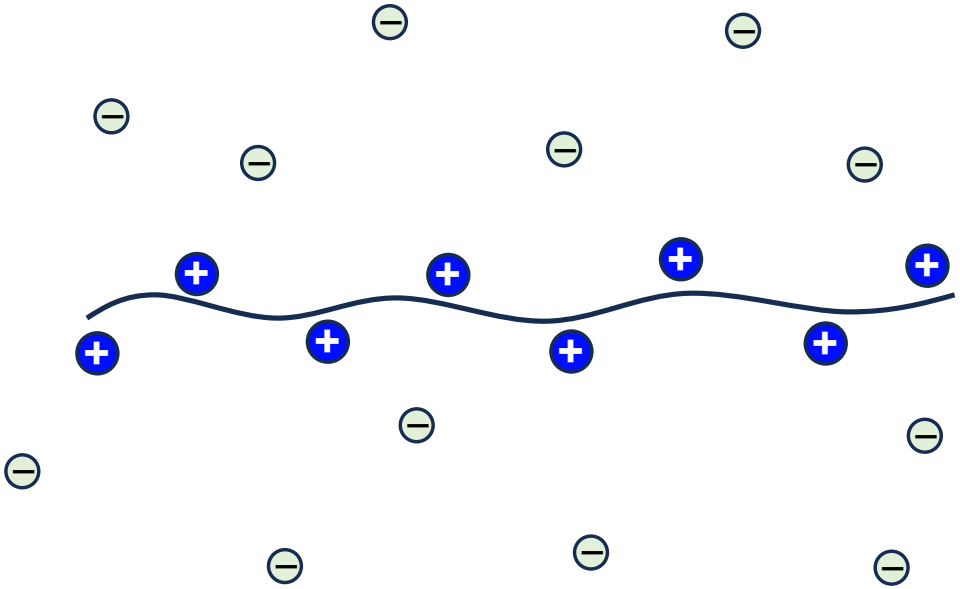

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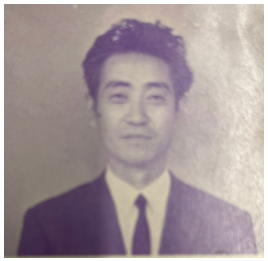

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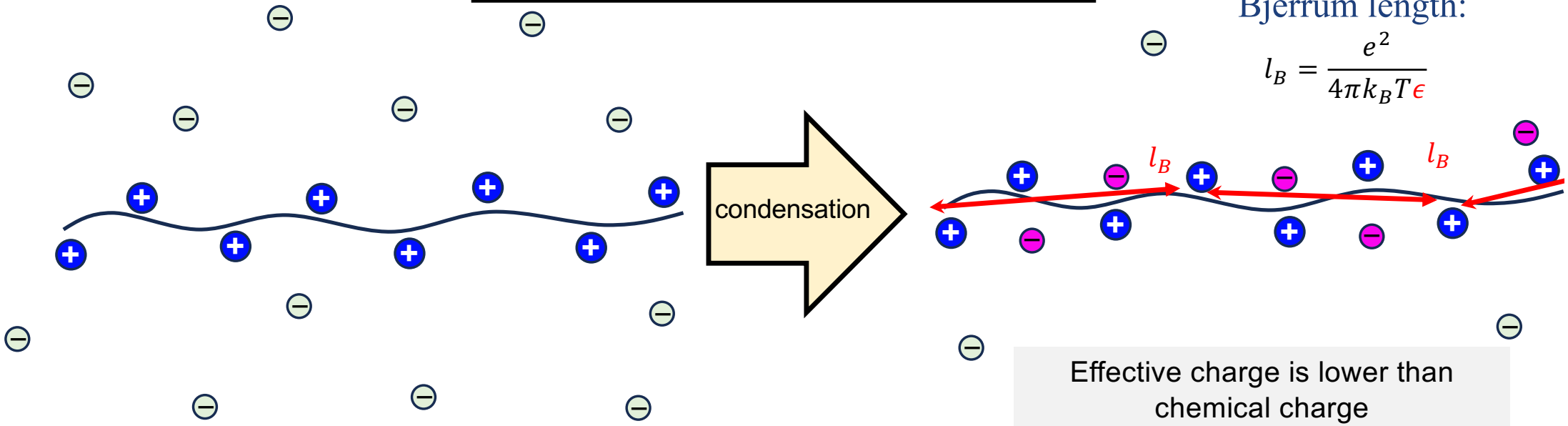
⊖ Free counterion

⊖ Condensed counterion

Do not contribute to thermodynamic or charge transport properties

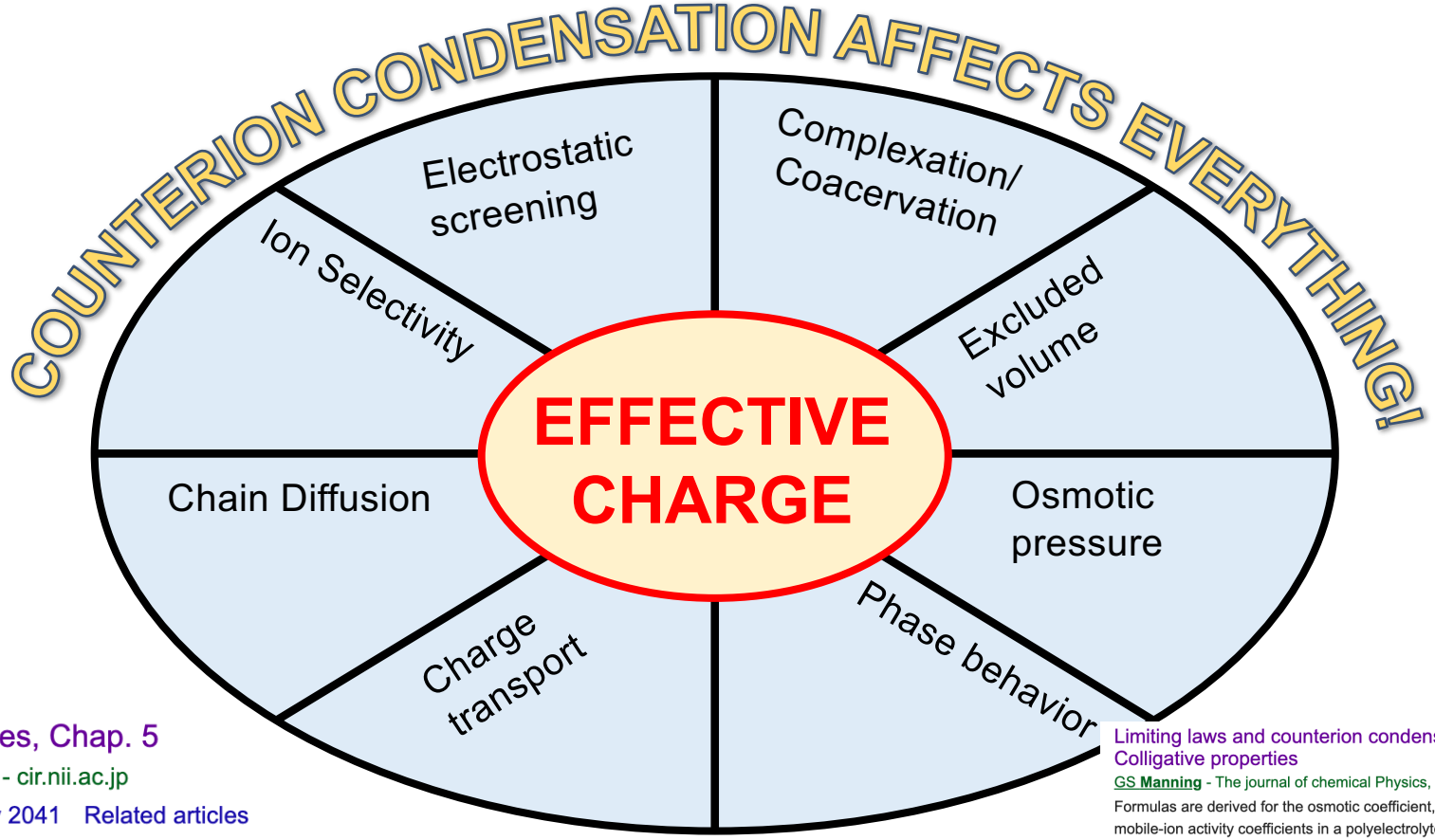
Bjerrum length:

$$l_B = \frac{e^2}{4\pi k_B T \epsilon}$$



Effective charge is lower than chemical charge

COUNTERION CONDENSATION IS THE CENTRAL PROBLEM OF POLYELECTROLYTE SCIENCE



[CITATION] Polyelectrolytes, Chap. 5
F Oosawa - (No Title), **1971** - cir.nii.ac.jp
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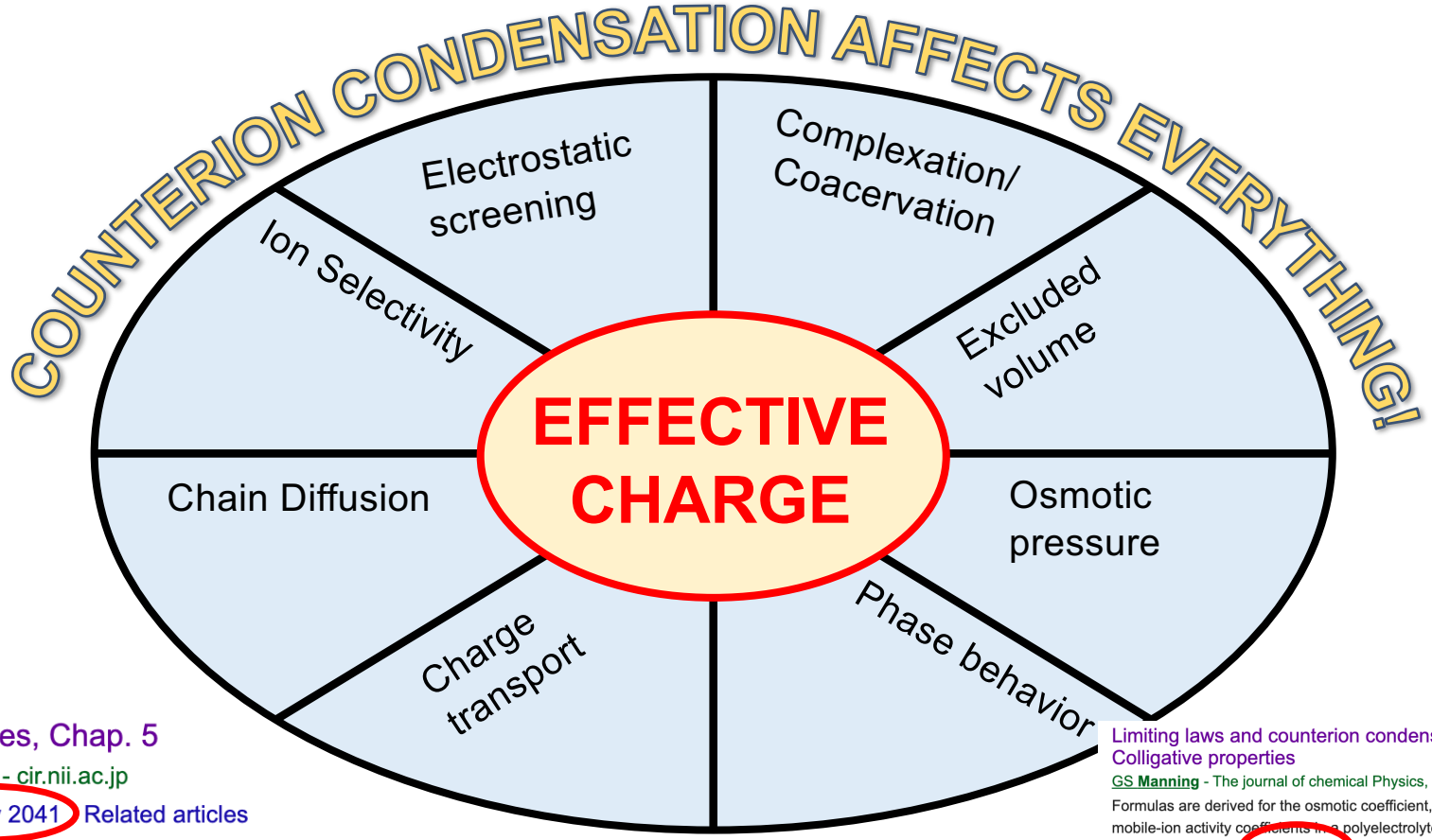

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METHODS FOR DETERMINING COUNTERION CONDENSATION

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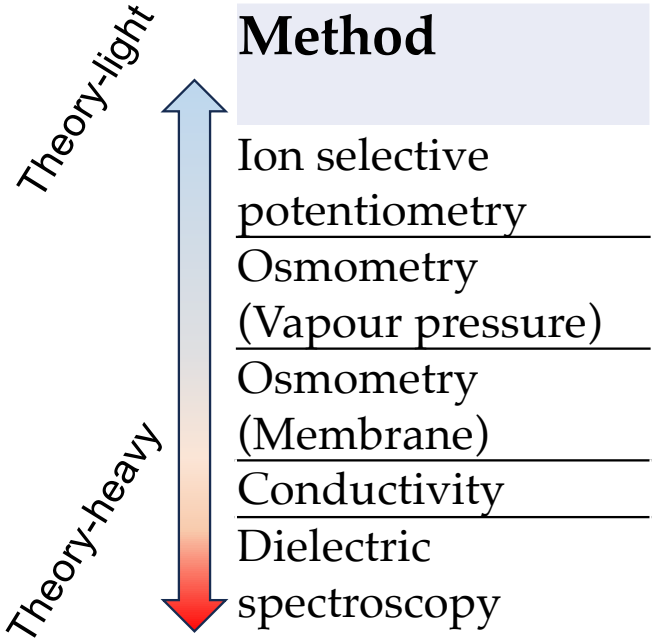

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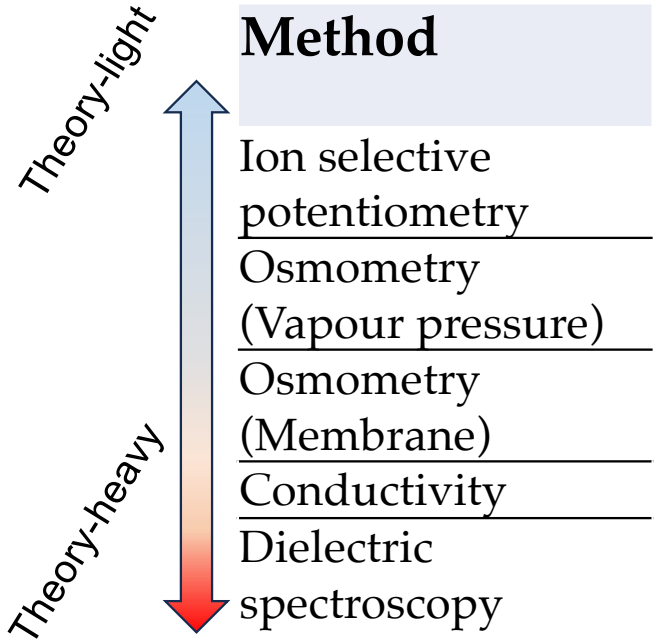

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$$\Pi = k_B T f c$$

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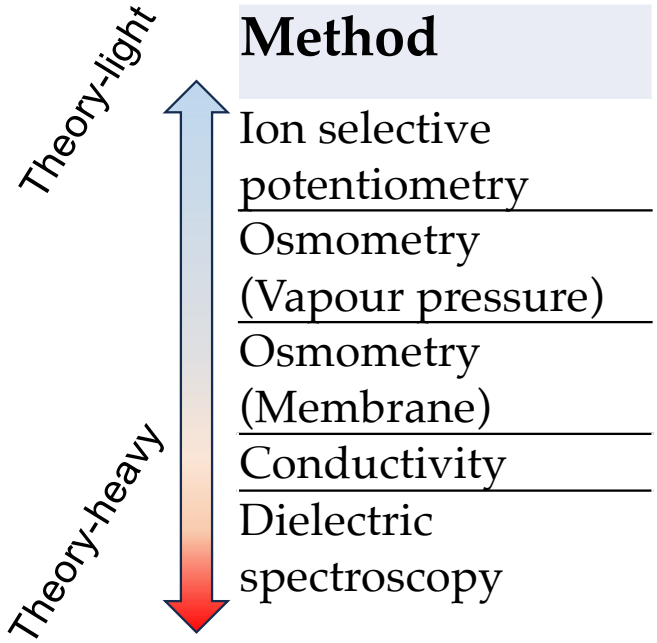

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DRAWBACK: very difficult for non-aqueous systems.

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LIGHT SCATTERING FROM SALT-FREE POLYELECTROLYTES

Static structure factor:

$$S(\mathbf{q}) = \frac{1}{N} \sum_{j=1}^{N} \sum_{k=1}^{N} \mathrm{e}^{-i\mathbf{q}\cdot(\mathbf{R}_j - \mathbf{R}_k)}$$

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Osmotic pressure is $k_B T$ per *free* counterion

Light scattering provides a way to measure the osmotic compresibility of a two-component solution

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Polyelectrolytes should scatter weakly at low q!

Light scattering provides a way to measure the osmotic compresibility of a two-component solution

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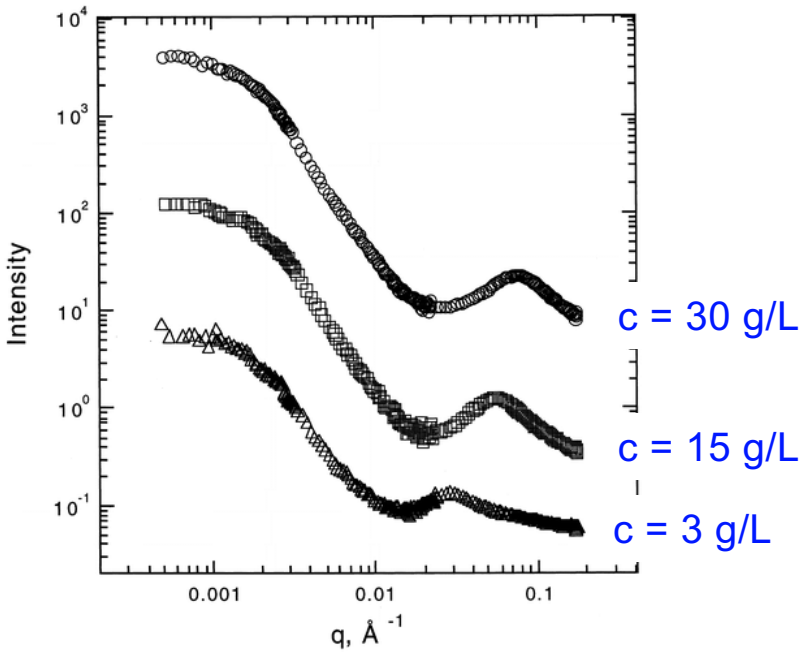
SCATTERING FROM SALT-FREE POLYELECTROLYTES

SANS/SLS



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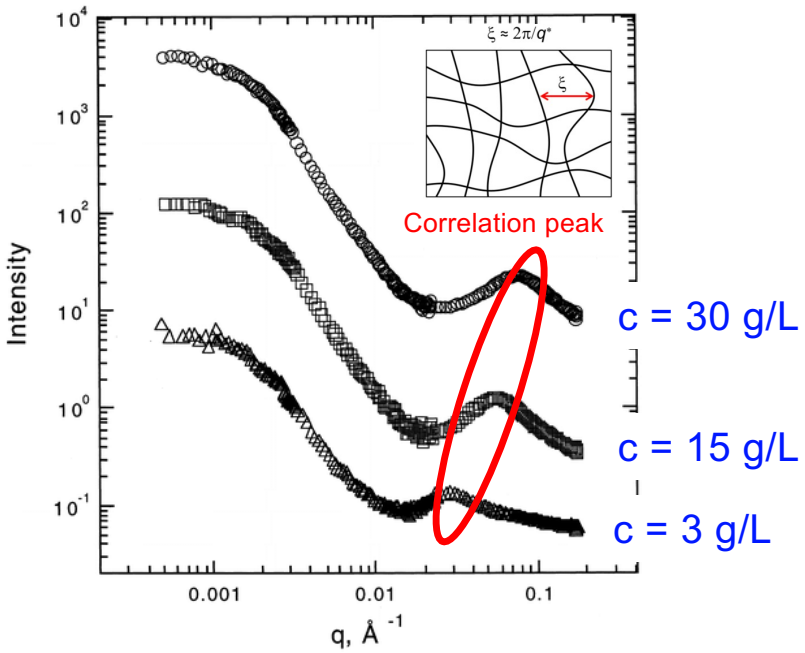

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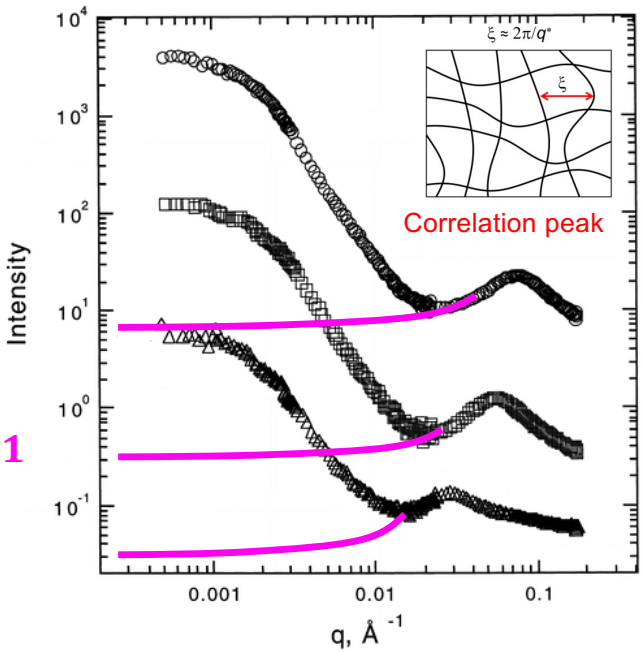

$$S(0) = f^{-1}$$

Theory expects extremely weak scattering by polyelectrolytes at low q but experiments show a huge low-q upturn

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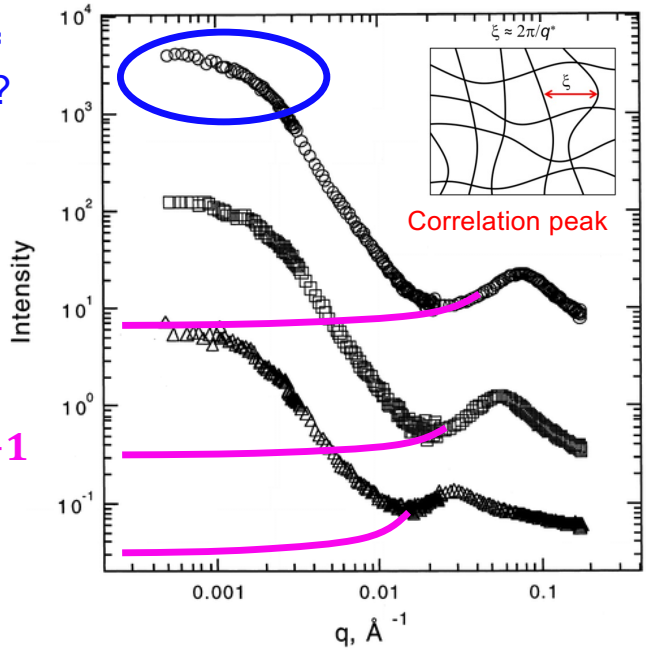
SANS/SLS

Low q upturn ≈ large domains?



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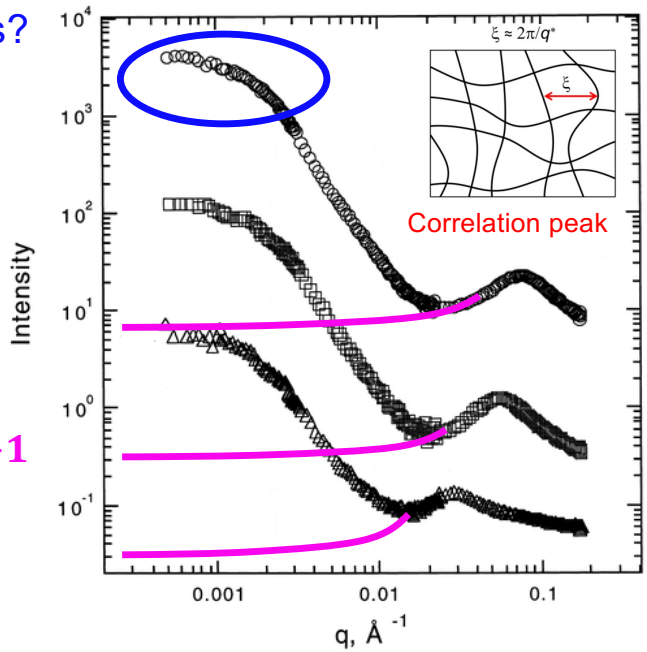

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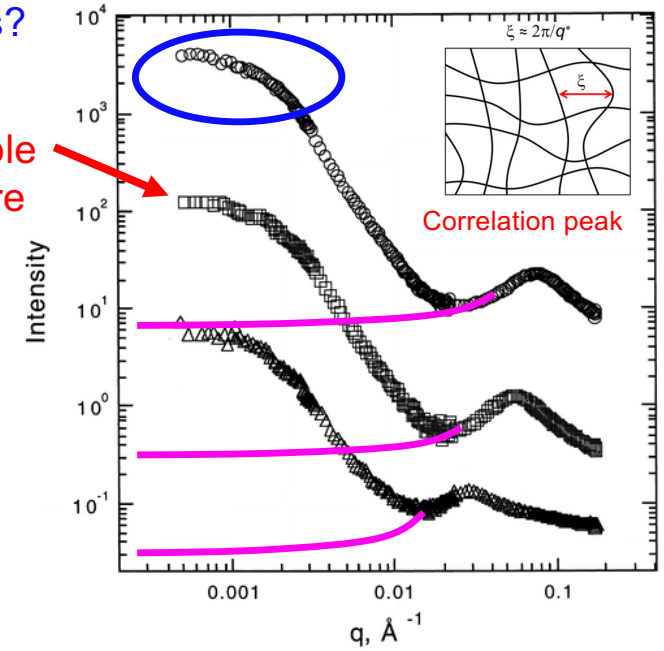
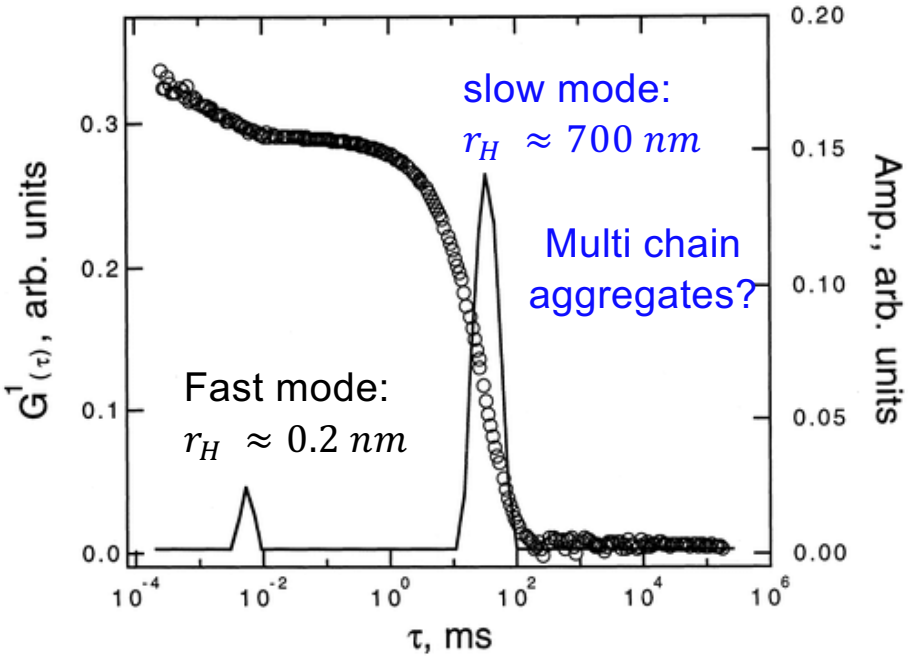
Low q upturn ≈ large domains?

This does not give a reasonable osmotic pressure

SANS/SLS



DLS



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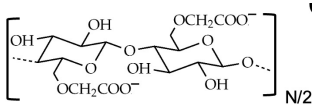
FILTRATION (MOSTLY) REMOVES LOW-*Q* UPTURN



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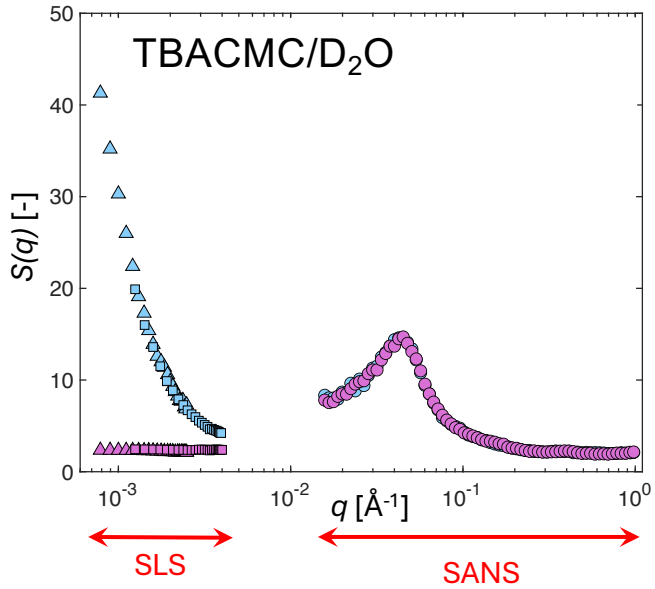

Filter
0.1 μm pore size
0.8 μm pore size

- **Filtration through sufficiently small pores suppresses upturn & slow mode.**
- **Mesh size is not affected by filtration → no polymer removed (density measurements further support this)**
- **Solutions are stable for at least 1 month**

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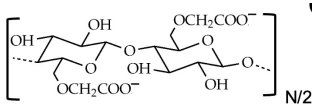

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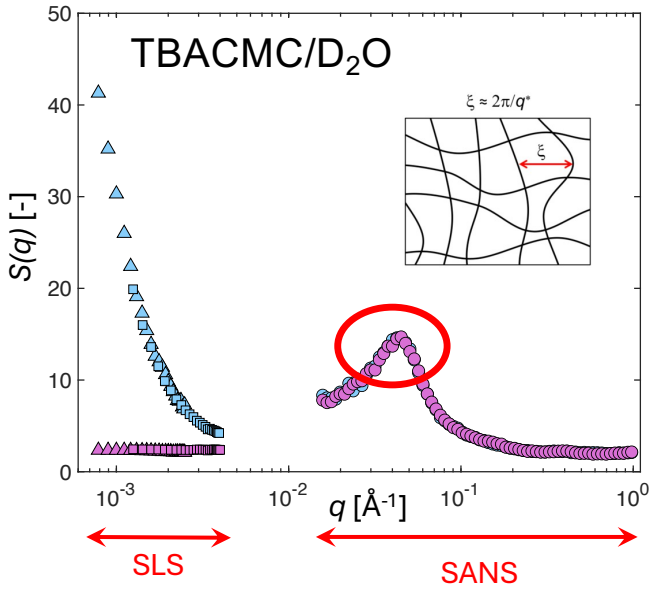

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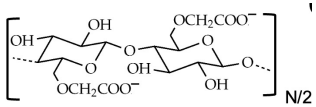

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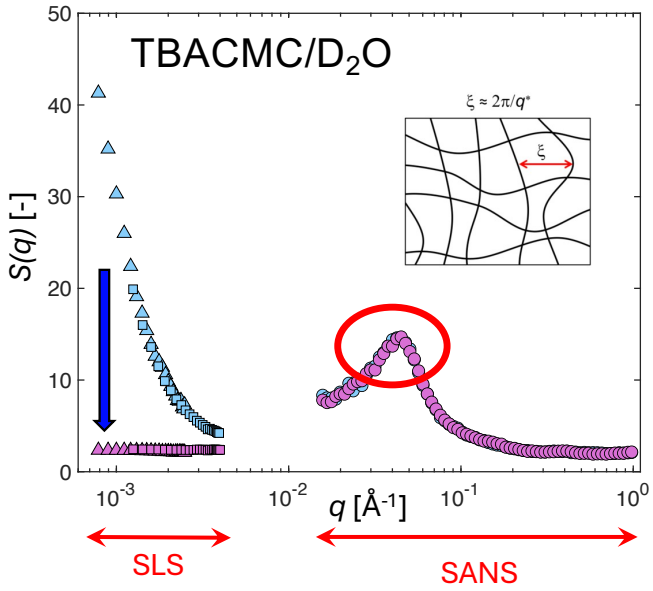

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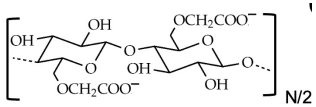

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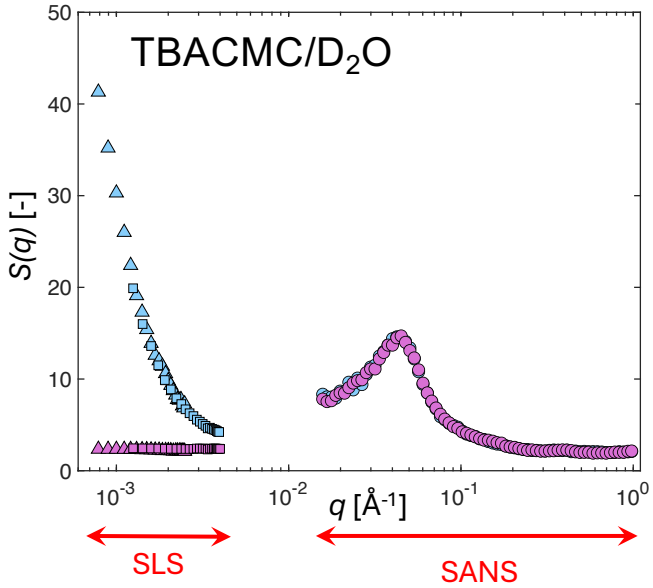

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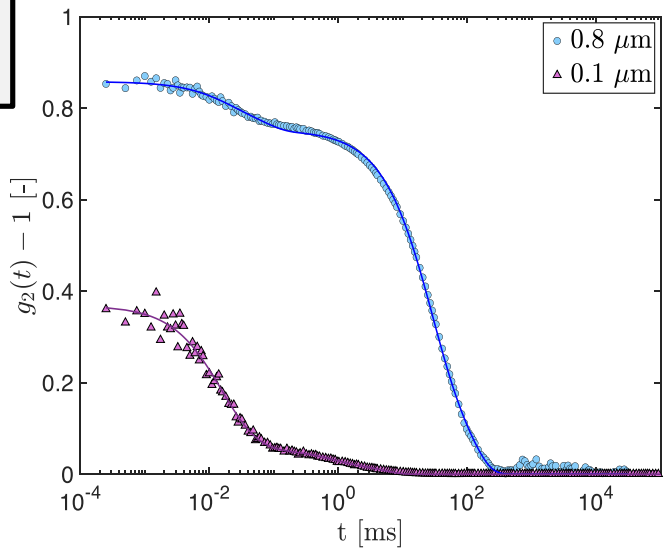

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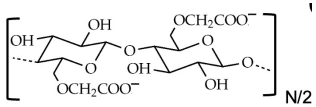
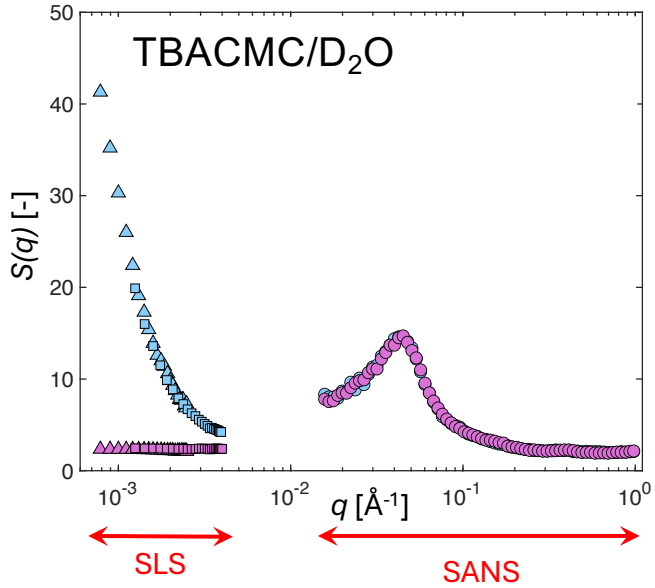
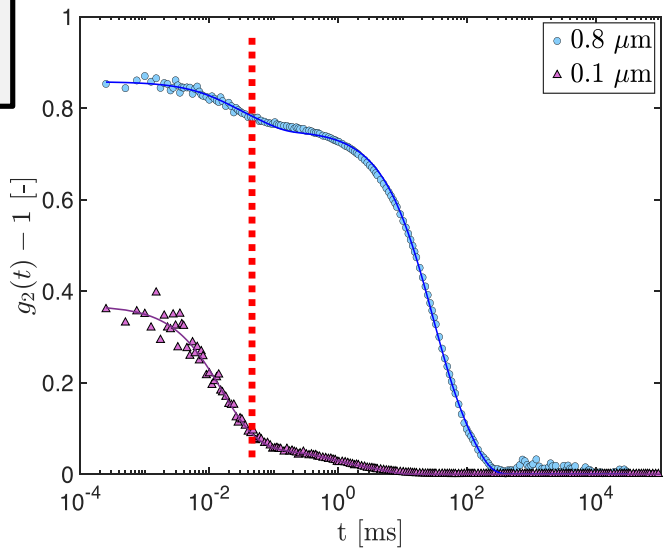


SANS/SLS



Filter
0.1 μm pore size
0.8 μm pore size

DLS

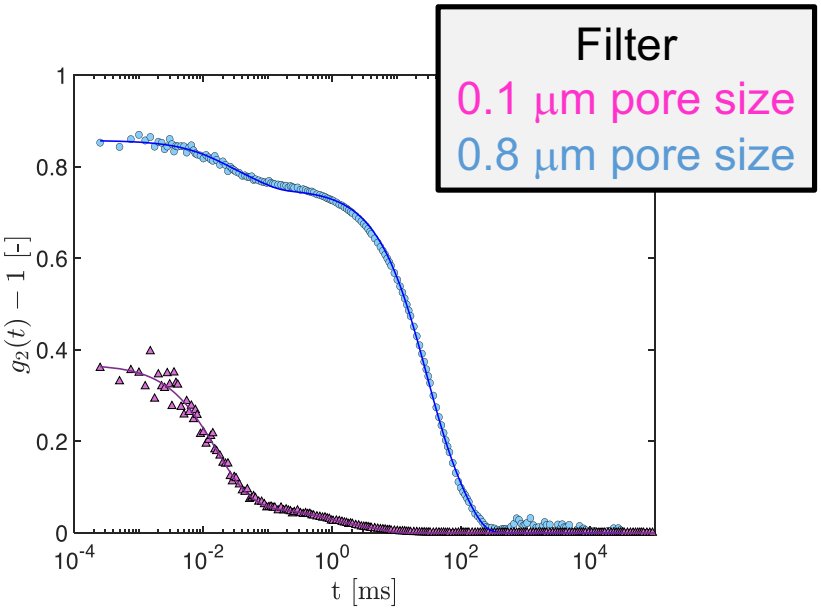


- **Filtration through sufficiently small pores suppresses upturn & slow mode.**
- **Mesh size is not affected by filtration → no polymer removed (density measurements further support this)**
- **Solutions are stable for at least 1 month**

CAN WE COMPLETELY REMOVE THE LOW-*Q* UPTURN?

Fit autocorrelation function to bimodal decay:

$$g_1(\tau,q) = A_1(q)e^{-\Gamma_1(q)\tau}\left(1+\frac{\mu_{2,1}t^2}{2}\right) + A_2(q)e^{-\Gamma_2(q)\tau}\left(1+\frac{\mu_{2,2}t^2}{2}\right)$$



The relative amplitude of the fast decay in DLS is used to isolate the structure factor without the slow mode:

$$S(q)_{fast} = \frac{A_1(q)}{A_1(q)+A_2(q)}S(q)$$

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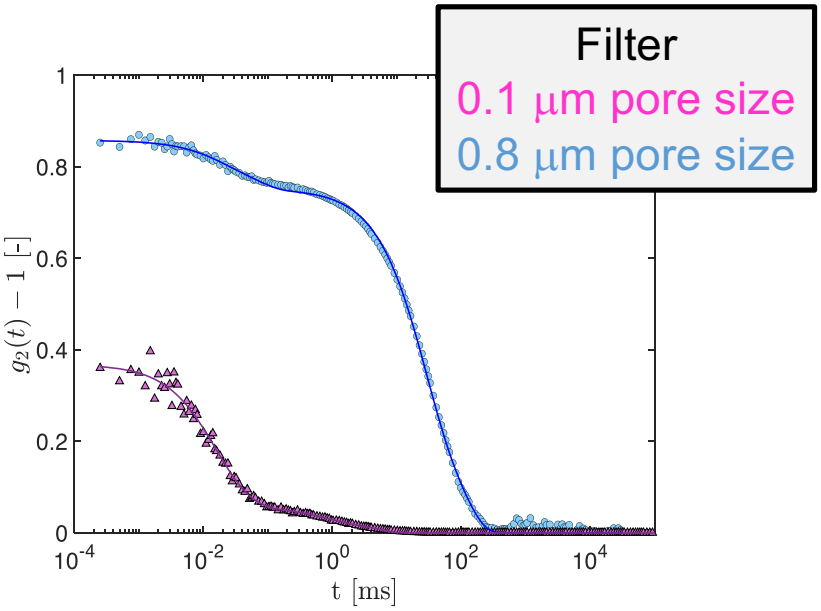

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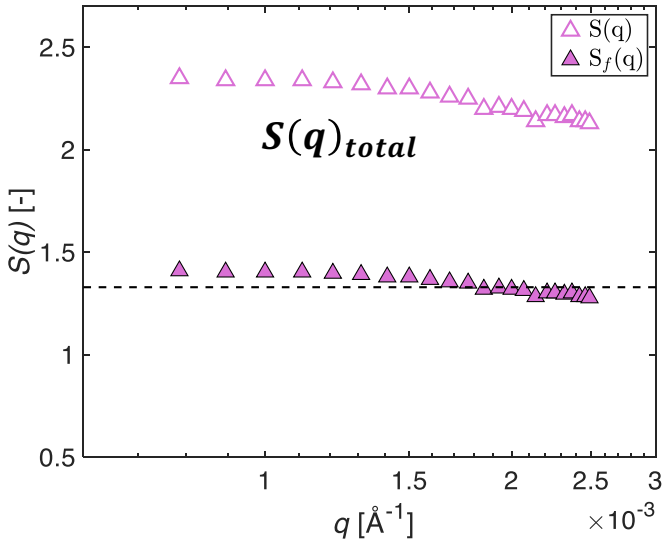

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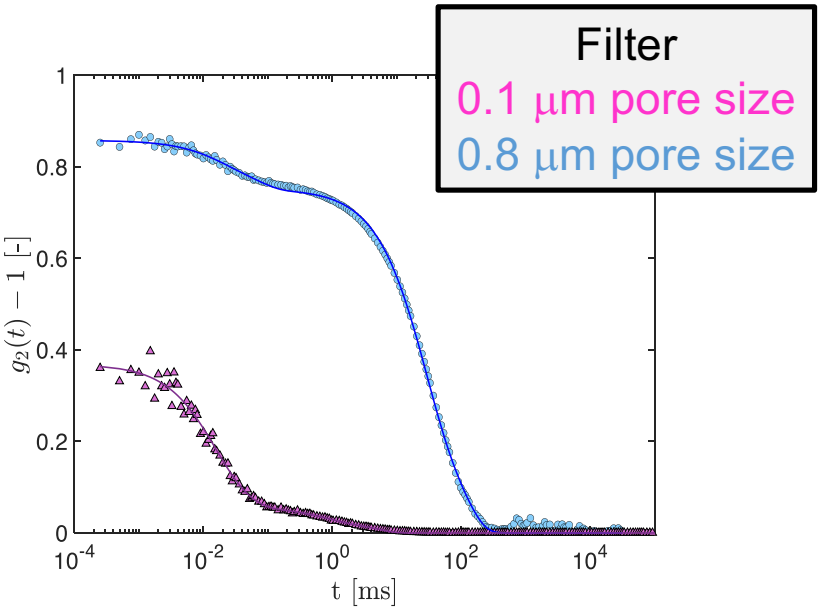

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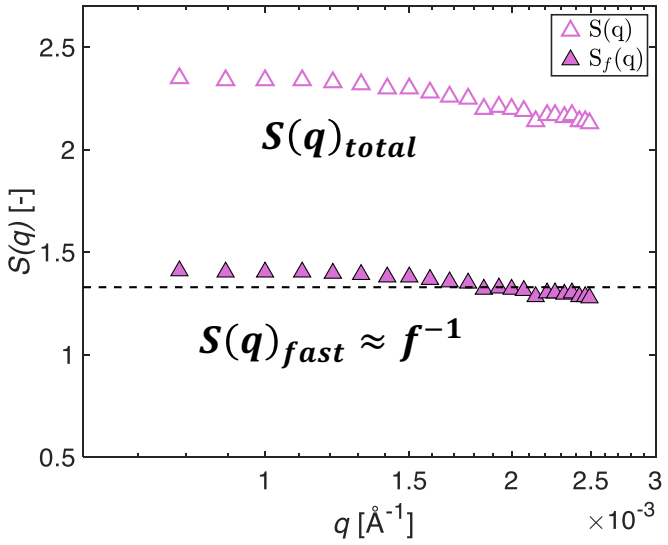

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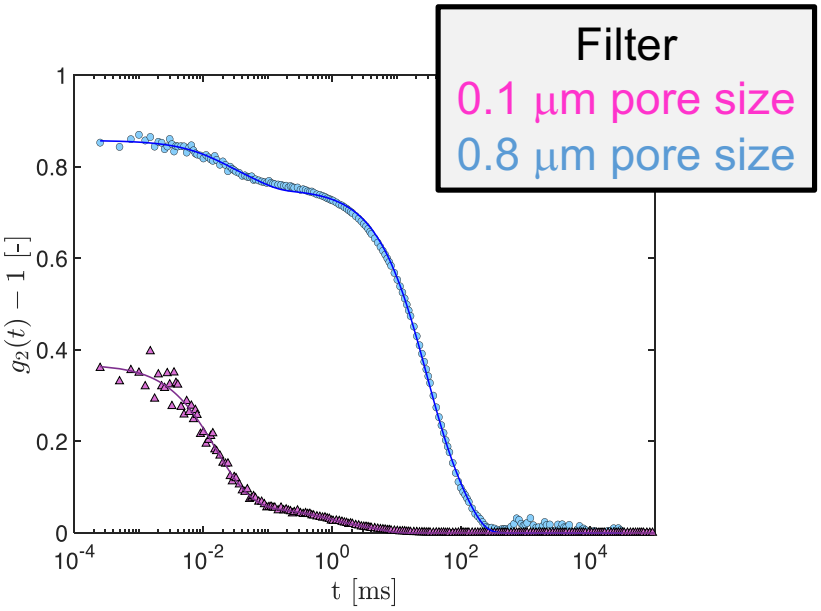

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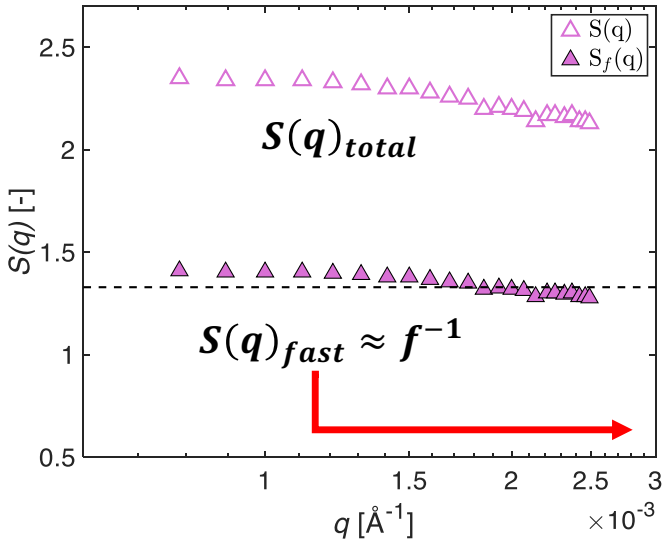

$f \approx 0.7$

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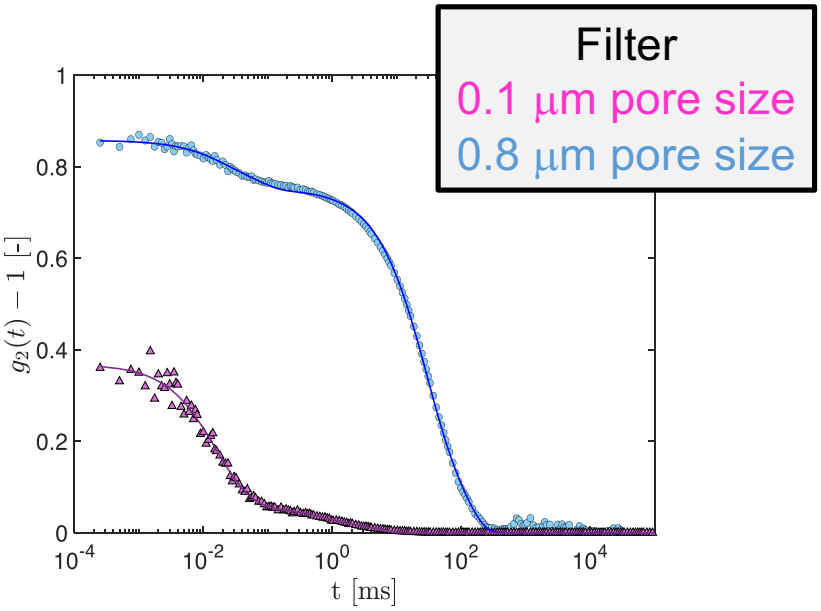

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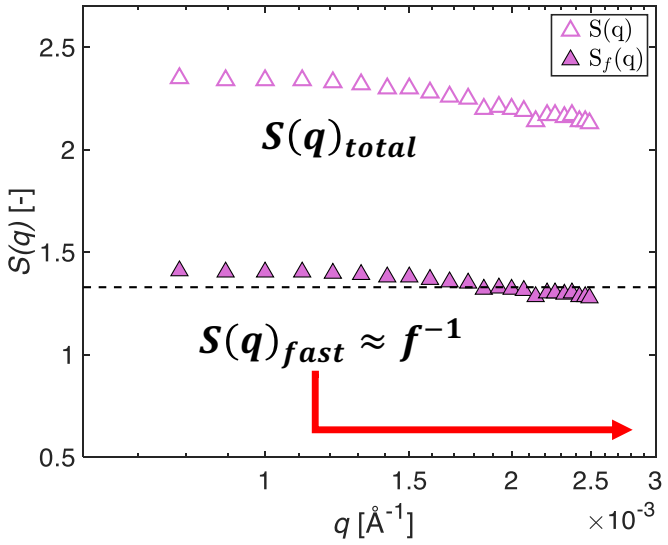
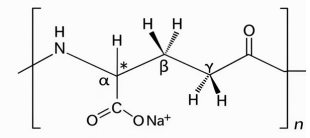
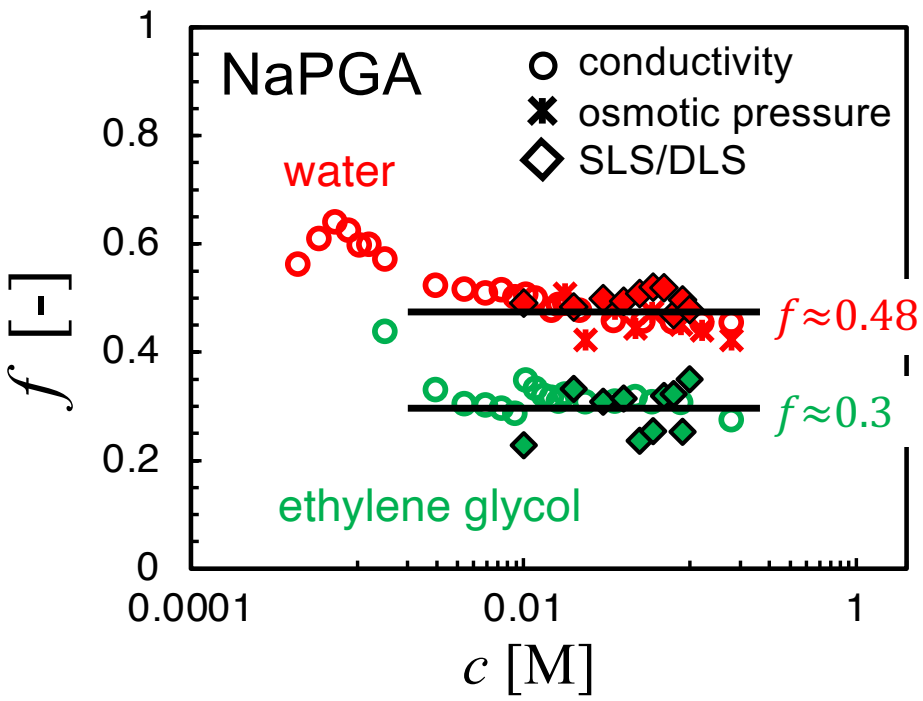
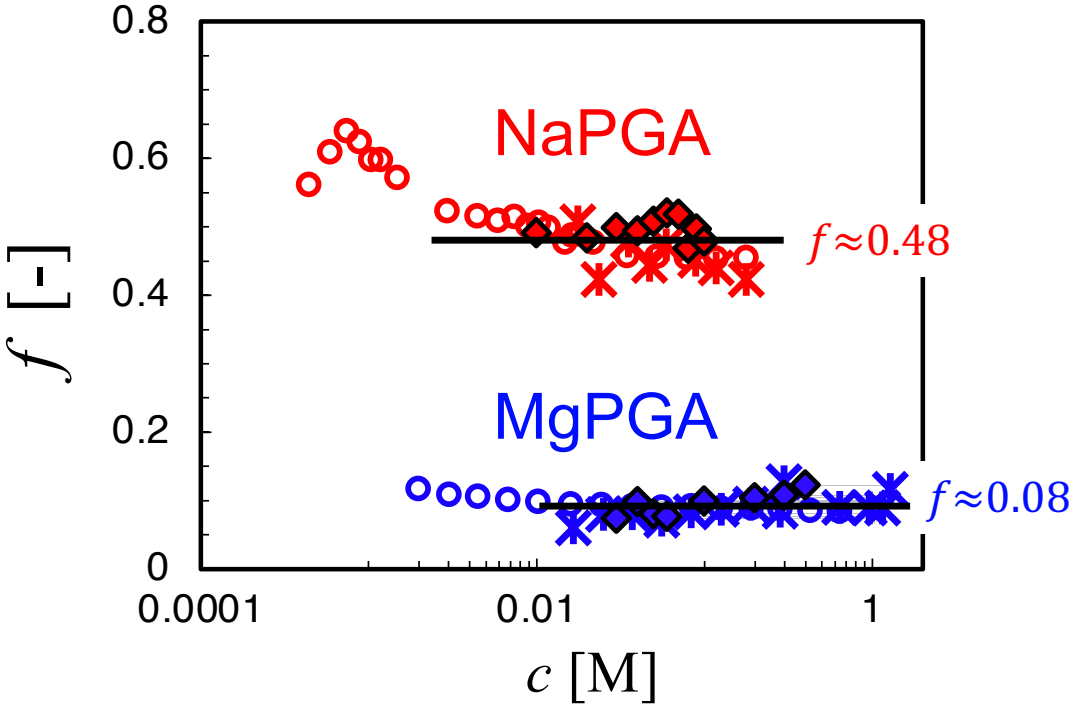


$f \approx 0.7$
$f_{cond} \approx 0.6$
not bad!

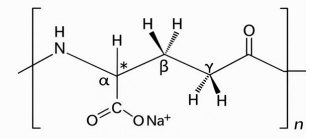


SLS/DLS METHOD: POLYGLUTAMIC ACID





Osmotic pressure measured by SLS after removing the slow mode contribution with DLS splitting gives the correct osmotic pressure



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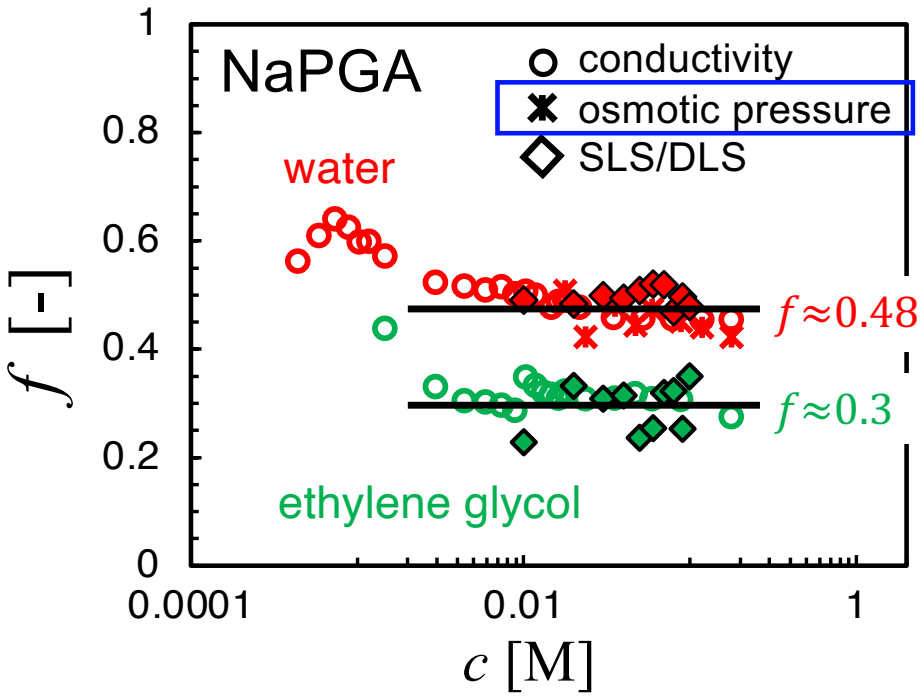

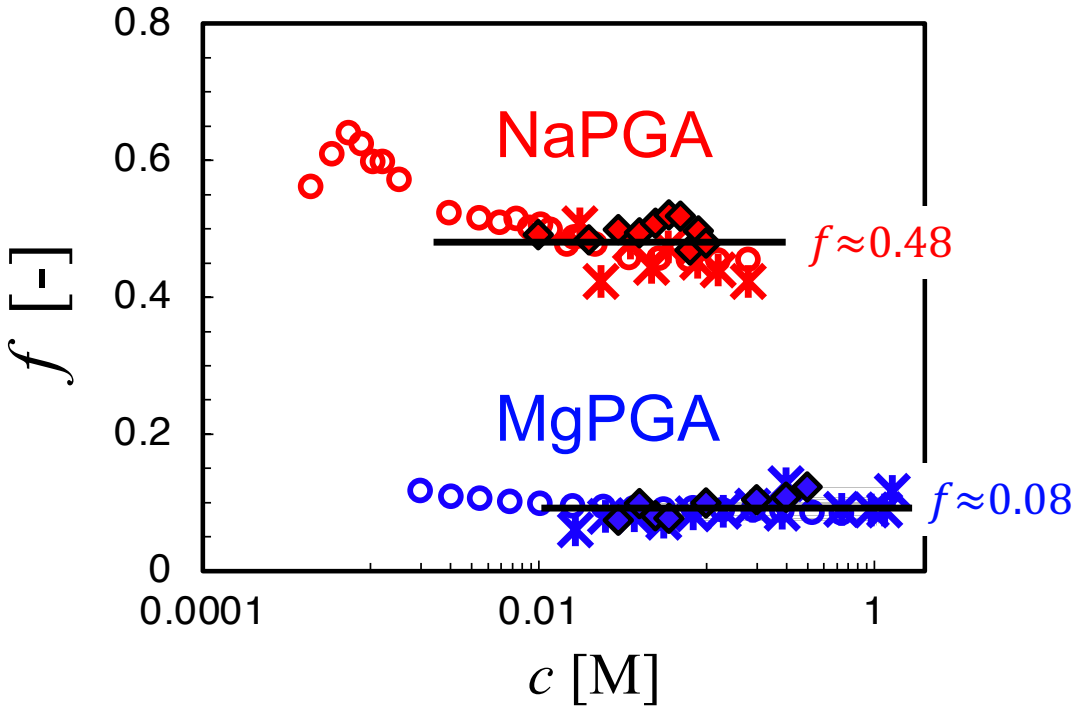

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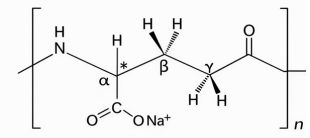

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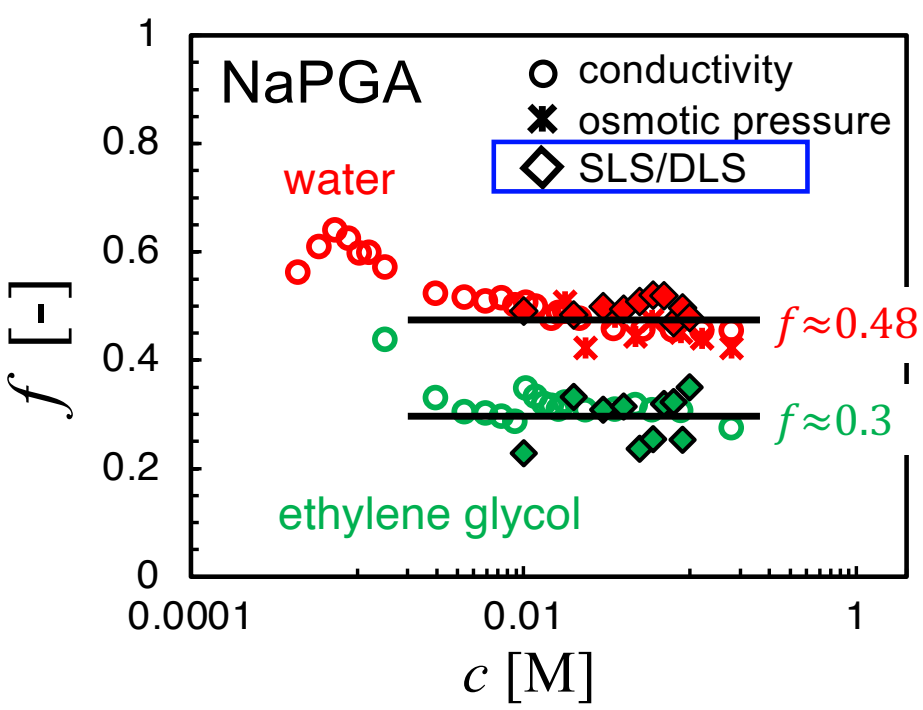
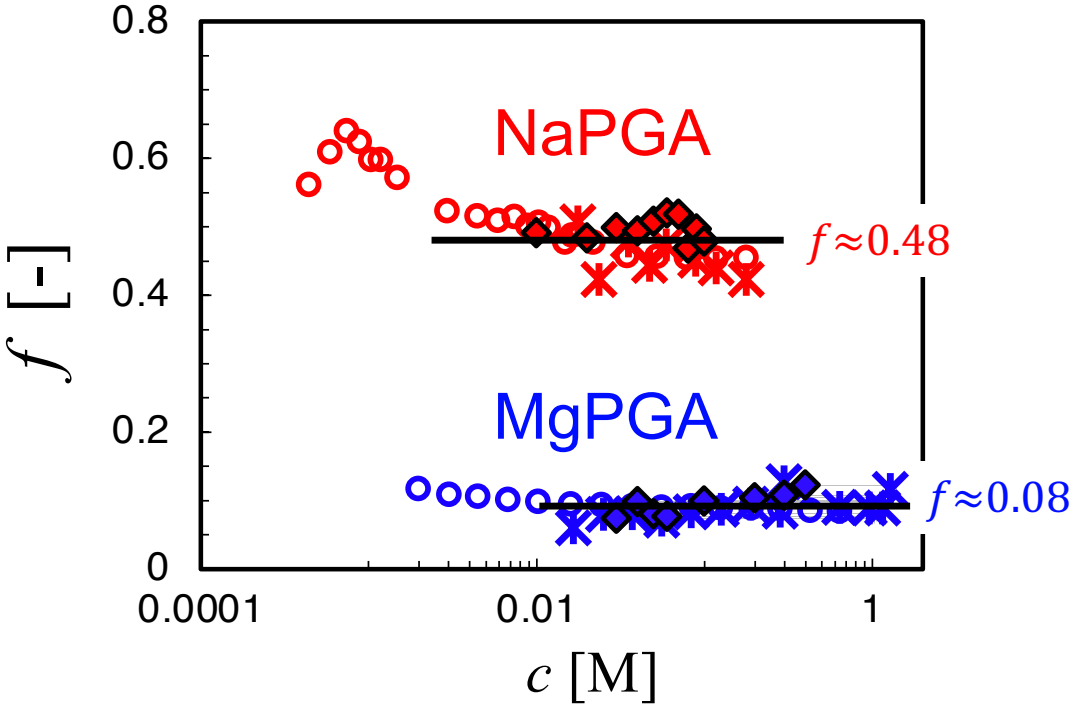
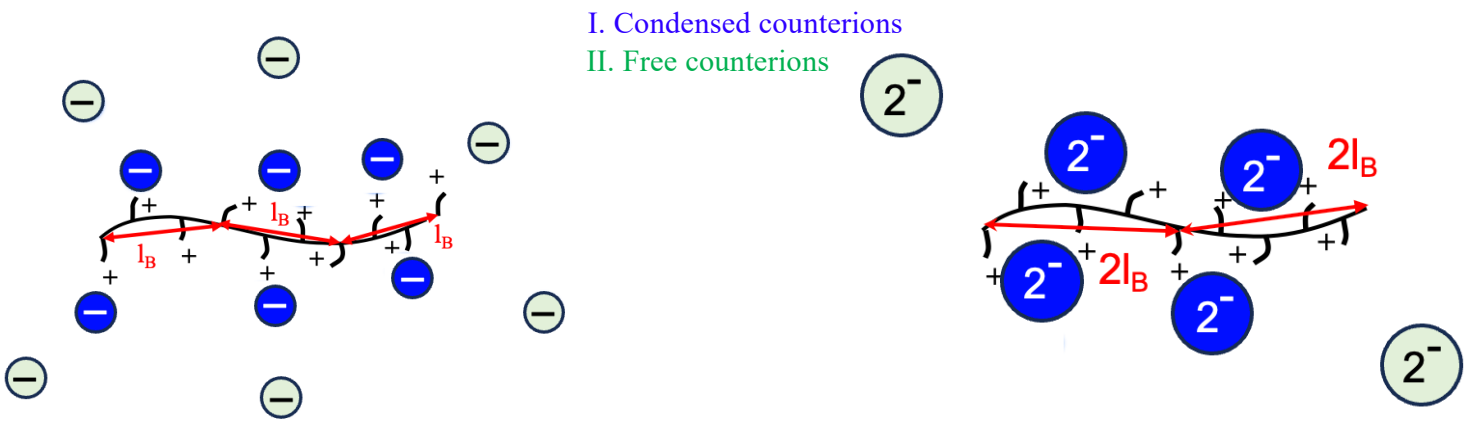




Osmotic pressure measured by SLS after removing the slow mode contribution with DLS splitting gives the correct osmotic pressure

Oosawa-Manning theory of condensation

Counterions will condense onto the chain until the charge density decreases to a value of e/l_B.



Effective charge:

$$b > l_B \rightarrow f \approx 1$$

$$b < l_B \rightarrow f \approx \frac{b}{Zl_B}$$

b – distance between charges
Z – counterion valence
l_B – Bjerrum length

$$l_B = \frac{e^2}{4\pi k_B T \epsilon}$$

TEST OF OOSAWA-MANNING THEORY

Effective charge: $f \approx \frac{b}{Zl_B}$

b – distance between charges
Z – counterion valence
l_B – Bjerrum length

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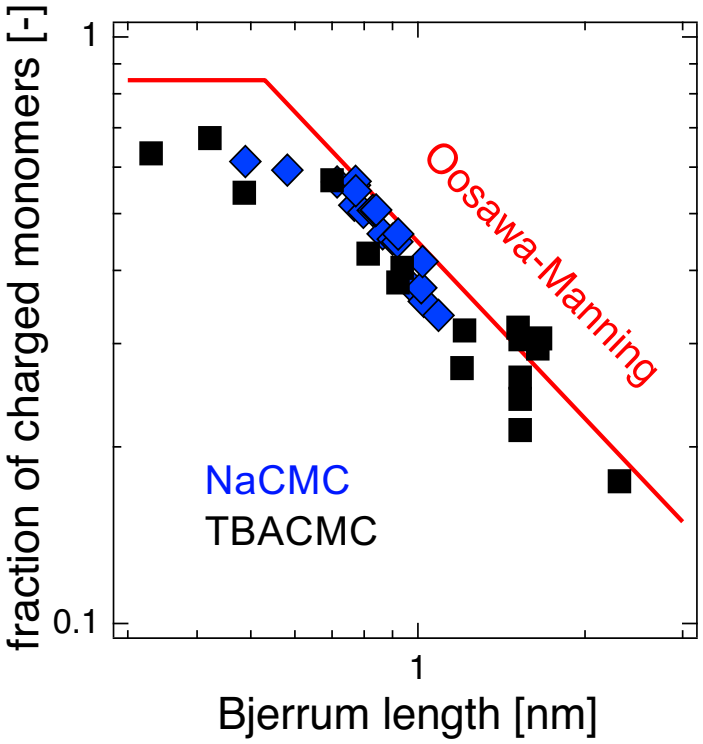
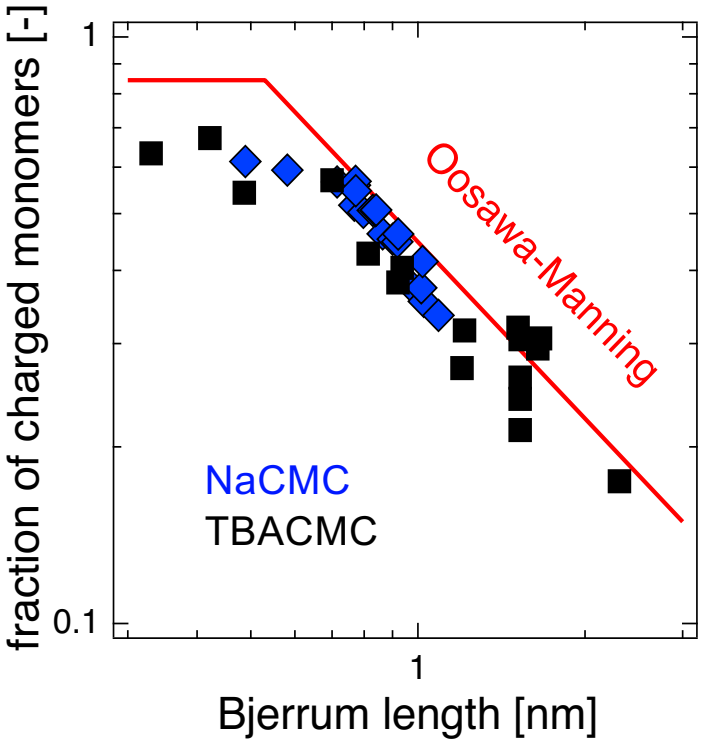


Hou C., Richtering W. & Lopez CG, Test of Oosawa–Manning condensation in a semiflexible polyelectrolyte across solvents of varying Bjerrum length *EPL* (accepted)

TEST OF OOSAWA-MANNING THEORY

$$f \approx \frac{b}{z l_B}$$



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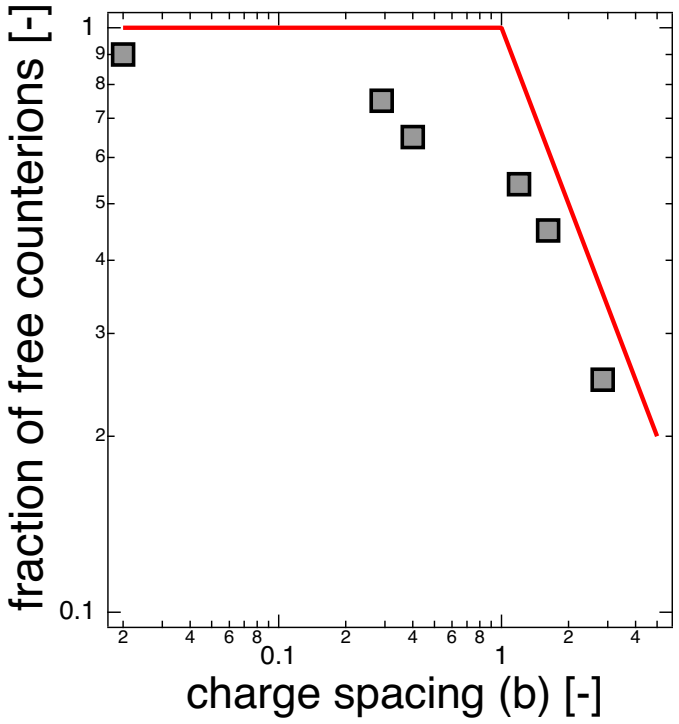

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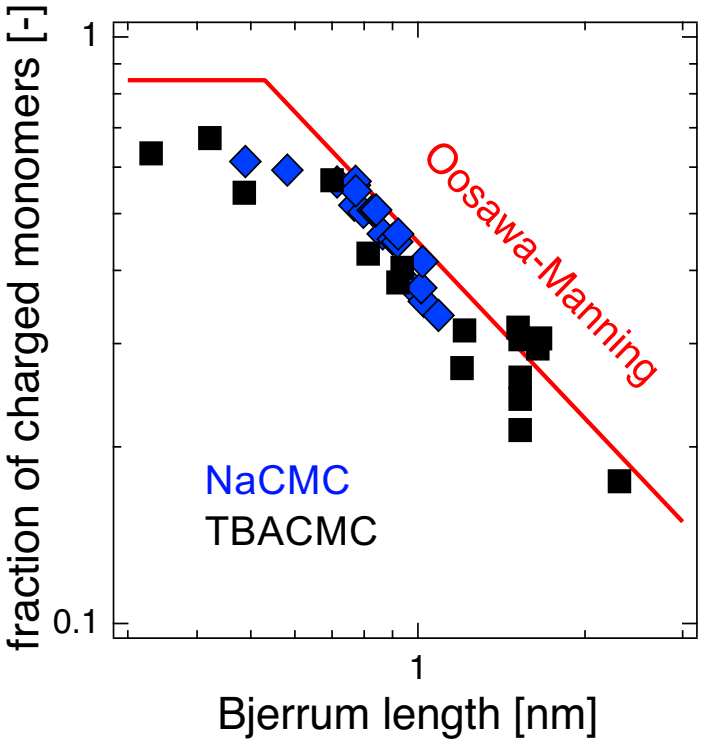

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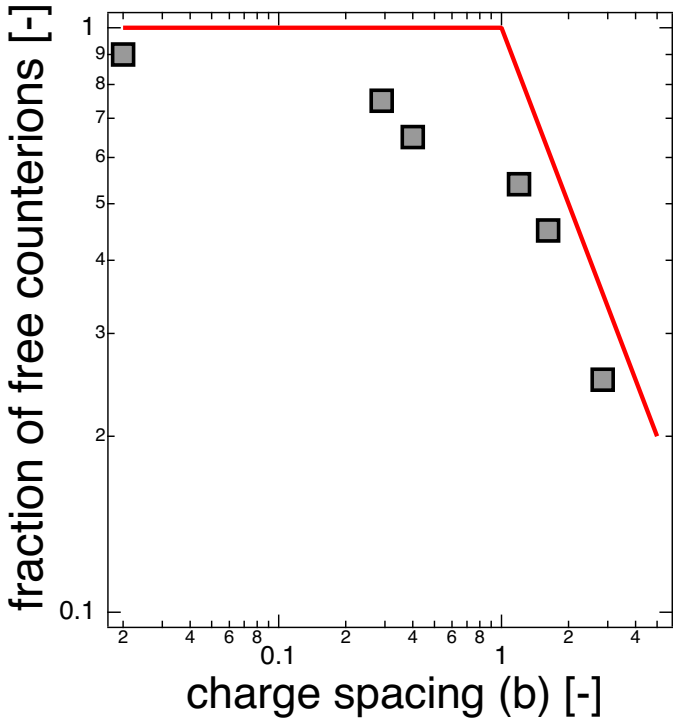

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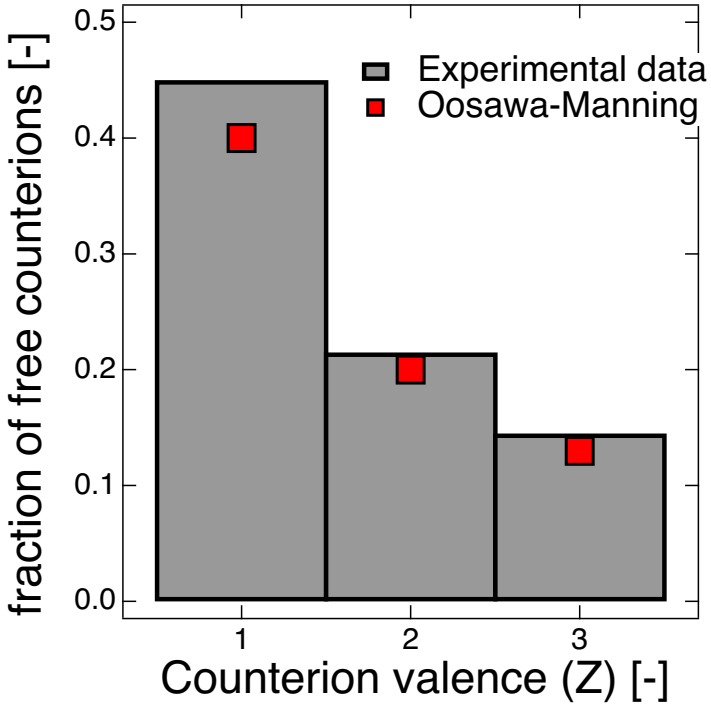

米勢政勝, 柘植英哉, and 岸本博. "種々の対イオンを有するコンドロイチン硫酸 A および C 塩の実用浸透圧係数." *日本化学会誌 (化学と工業化学)* 1978, no. 1 (1978): 108-112.

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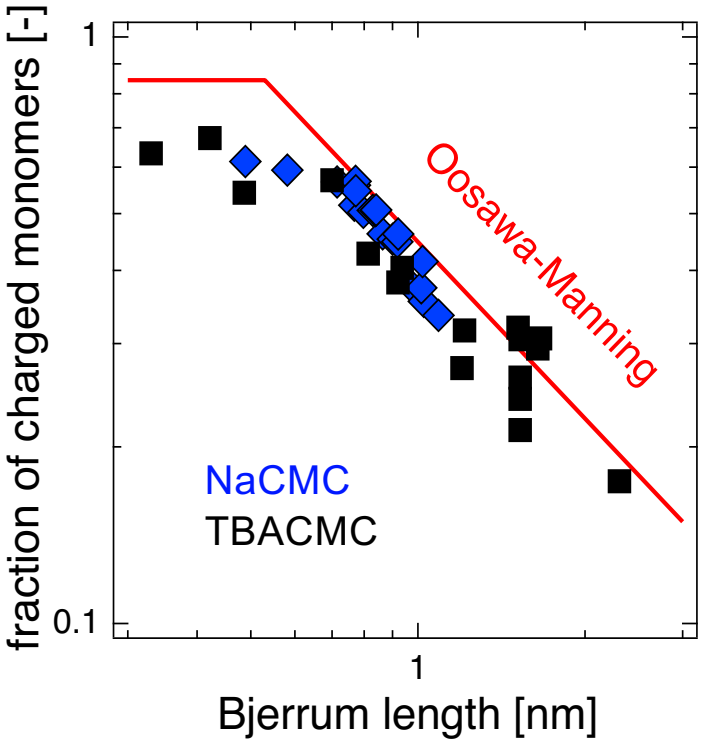

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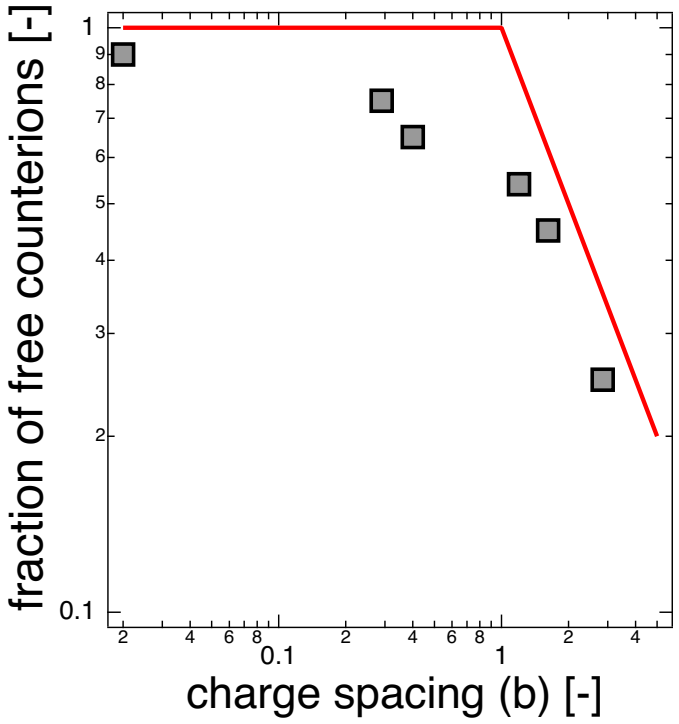

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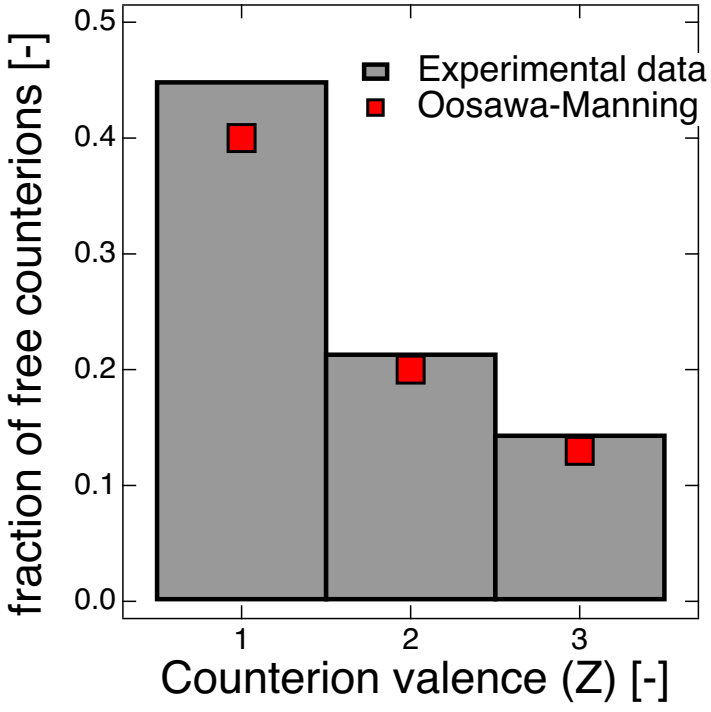
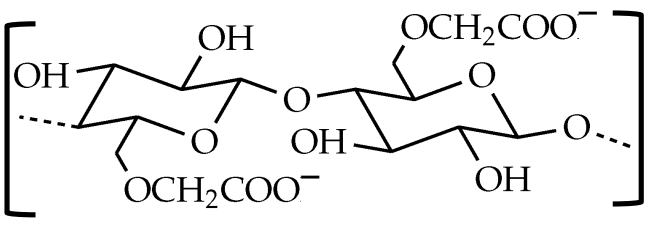


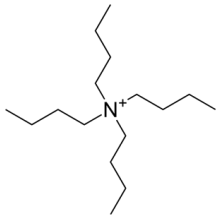
Red lines/symbols are parameter-free predictions!

Presentation Outline

- Counterion condensation: the central problem in polyelectrolyte physics
- Light scattering as a tool to study counterion condensation
- **Beyond the two-phase model: insight from SANS and SAXS into counterion distributions**

Delocalisation of hydrophobic counterions

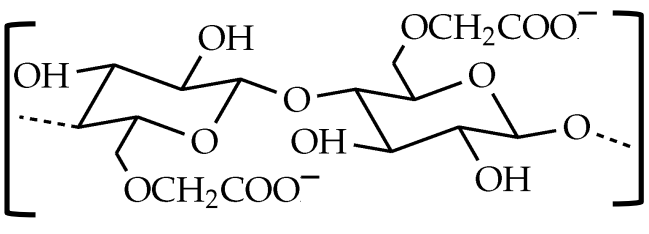


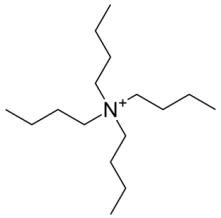


SANS: contrast from H-rich counterions

SAXS: contrast from polymer backbone

Delocalisation of hydrophobic counterions

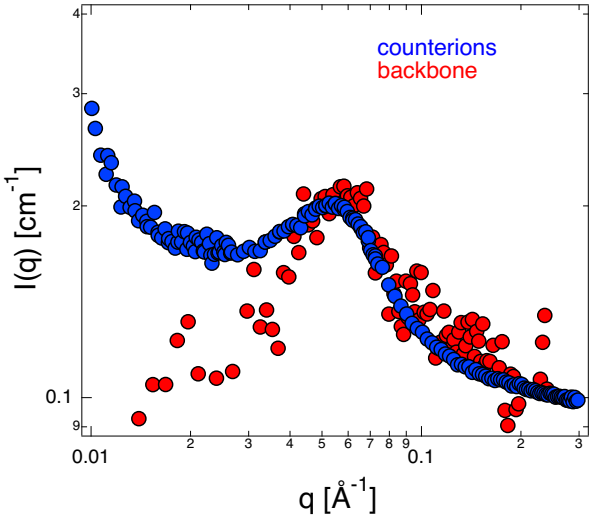




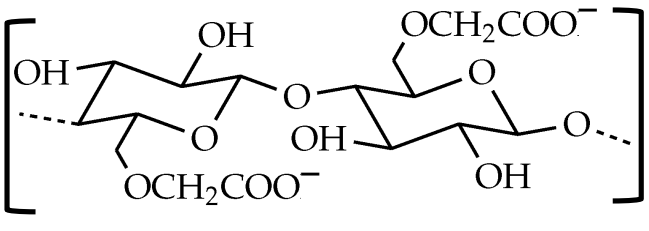
SANS: contrast from H-rich counterions

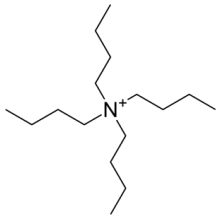
SAXS: contrast from polymer backbone

c ≈ 0.03 M



Delocalisation of hydrophobic counterions





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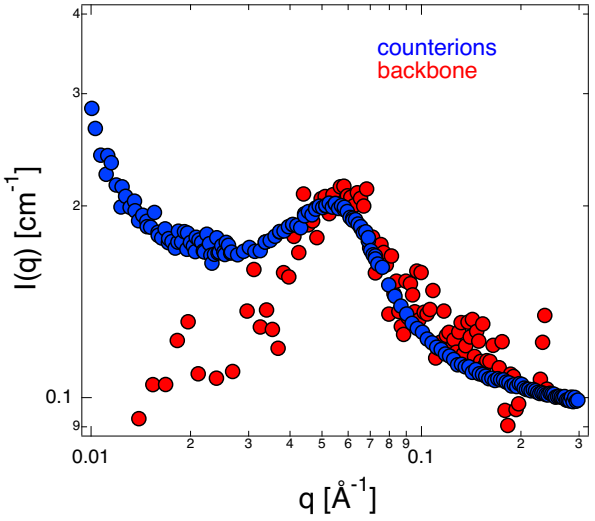
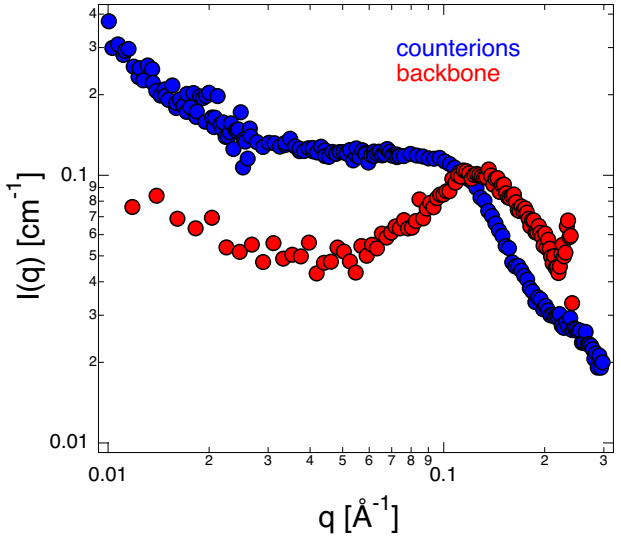
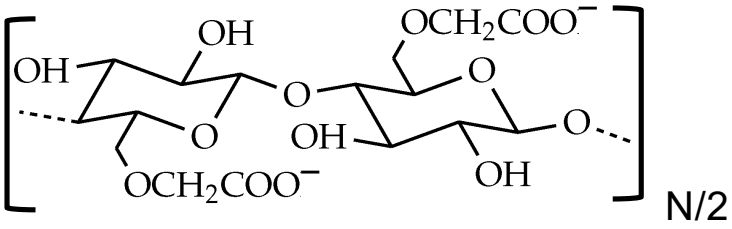


c ≈ 0.14 M



Hydrophobic counterions de-localise from the backbone at high concentrations

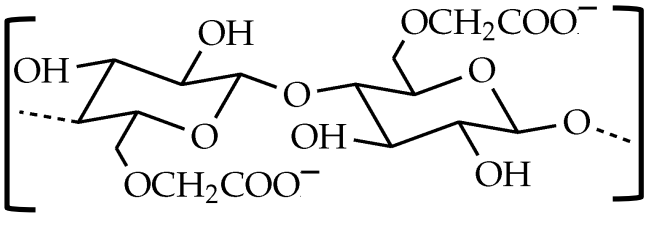
Effect not observed for hydrophilic ions



SANS: contrast from H-rich backbone

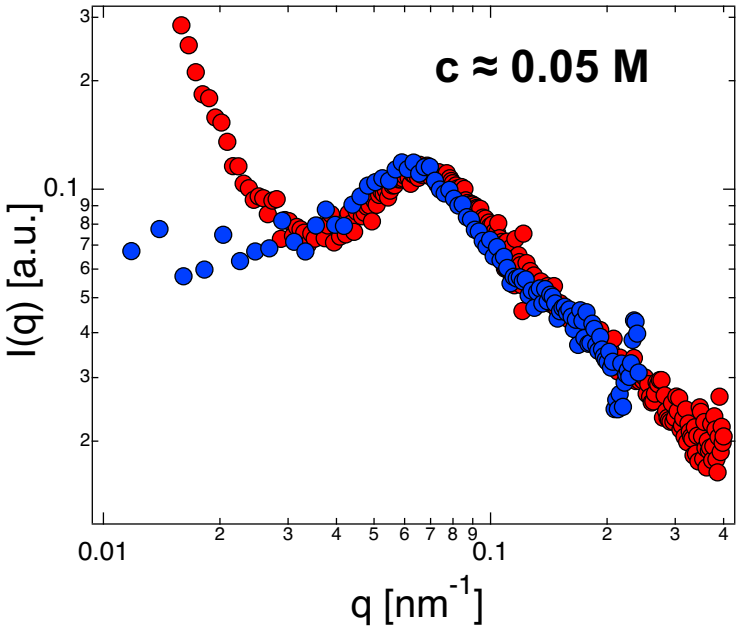
SAXS: contrast from electron-rich counterion

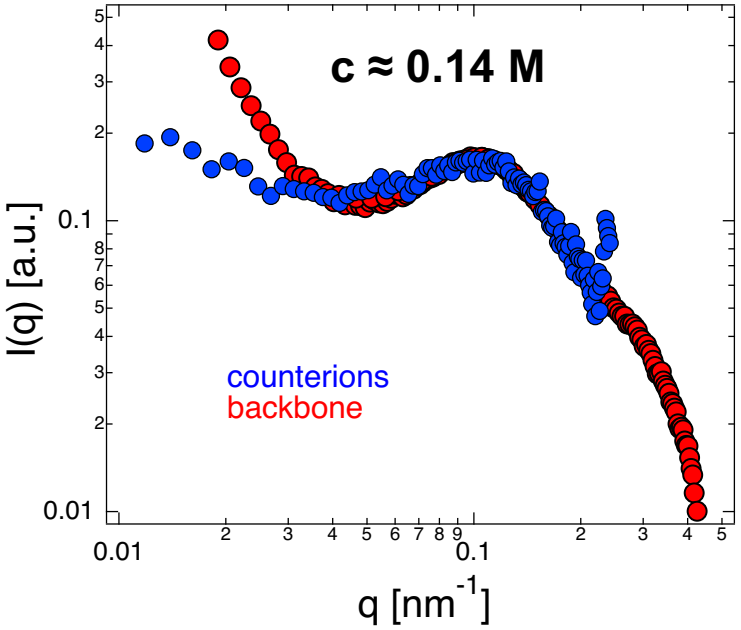
Effect not observed for hydrophilic ions



SANS: contrast from H-rich backbone

SAXS: contrast from electron-rich counterion

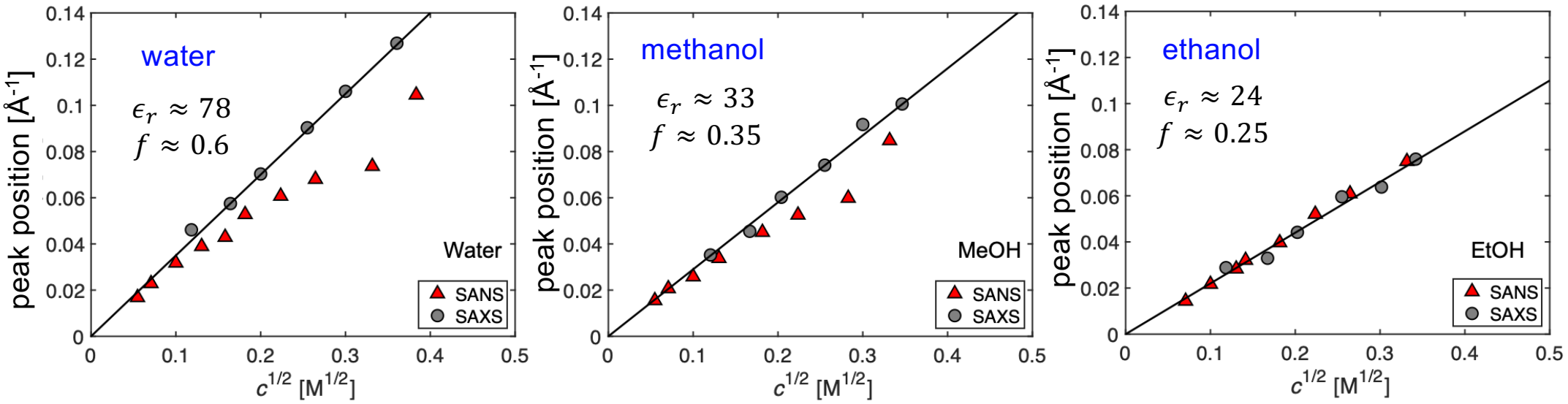




Hydrophilic counterions remain tightly correlated with the backbone at low and high concentrations

POLYMER-COUNTERION DECOUPLING

Contrast from polymer backbone
Contrast from counterions



Dielectric constant decreases

Polymer & counterion fluctuations de-couple

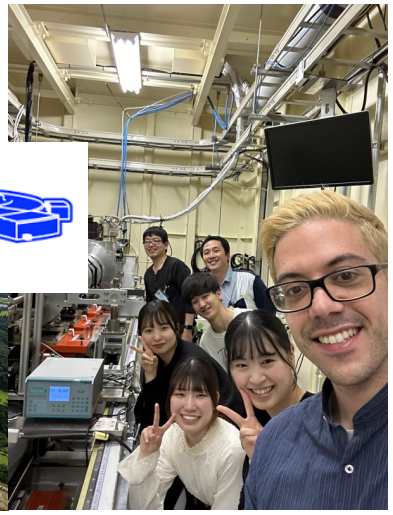
Gulati, A., Meng, L., Hou, C., Watanabe, T. and Lopez, C.G., 2026. Solution structure and counterion condensation of carboxymethyl cellulose in organic solvents. *ACS Applied Polymer Materials*. 8, 11, 8183–8197

CONCLUSIONS

- Combined static and dynamic **light scattering offers a way to measure the osmotic pressure of polyelectrolytes**.
- Oosawa-Manning correctly predicts the fraction of free counterions for semiflexible polyelectrolytes.
- SANS and SAXS can be used to probe the local concentration fluctuations of counterions and polyion backbone. **Hydrophobic counterions de-couple** from the backbone at high concentrations while hydrophilic ones do not.

ACKNOWLEDGEMENTS

Spring-8 synchrotron (Hyogo, Japan)



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CHRNS CENTER FOR HIGH RESOLUTION NEUTRON SCATTERING

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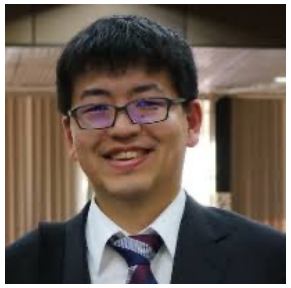

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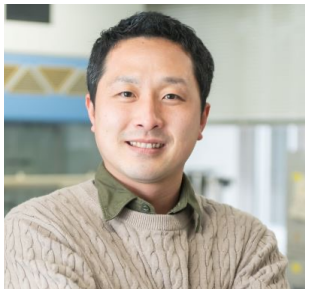

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ISIS Neutron Source (Didcot, UK)

SANS-U beamline at JRR-3 (Ibaraki, Japan

Japan Atomic Energy Agency JRR-3 Japan Research Reactor-3

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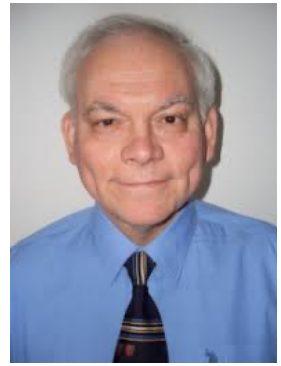

Preprints:

Effect of dielectric constant