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Questions
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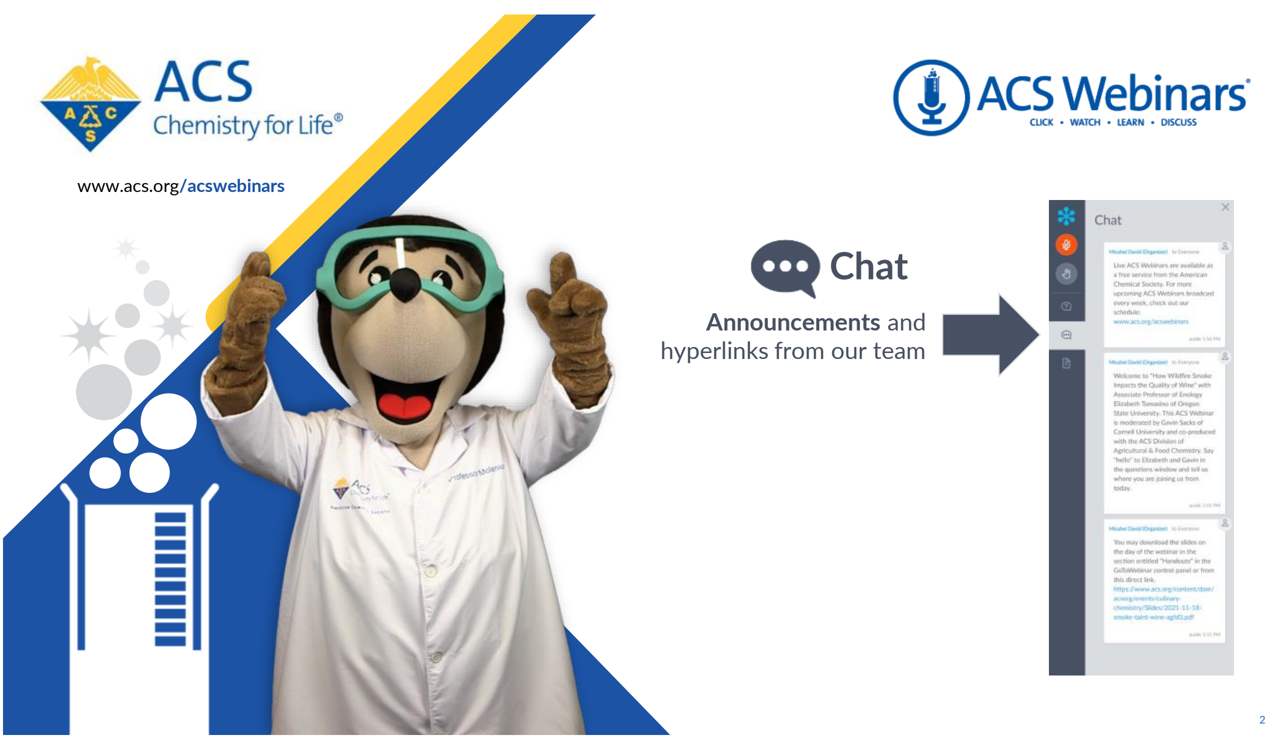
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https://chemidp.acs.org 5

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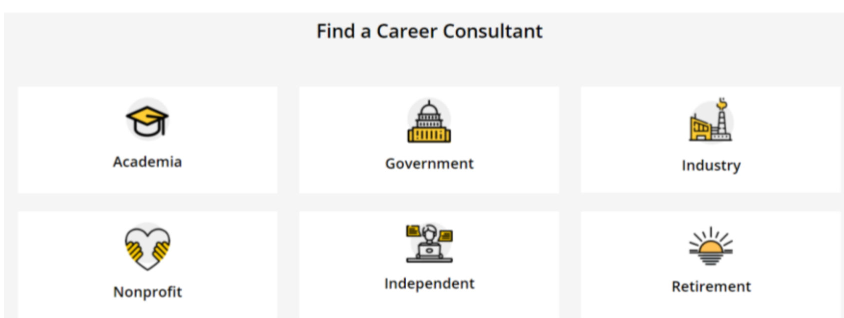

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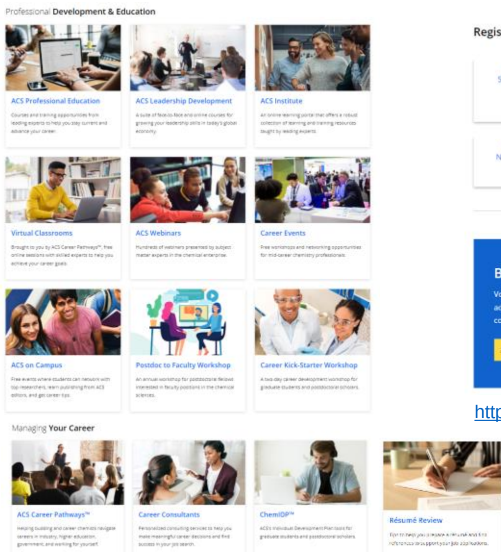
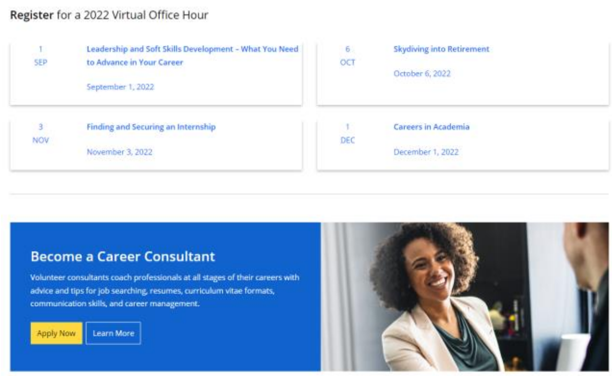
ACS Career Resources





https://www.acs.org/content/acs/en/careers/personal-career-consulting.html

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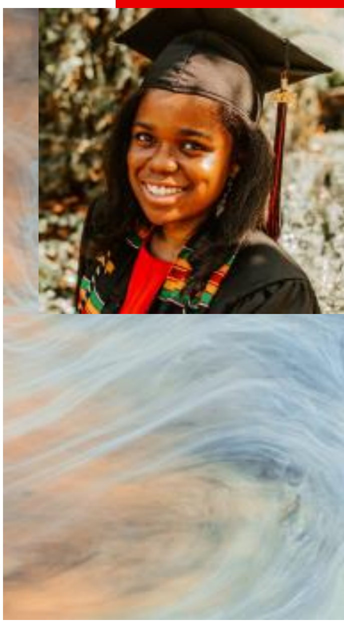

ACS Scholar Adunoluwa Obisesan

BS, Massachusetts Institute of Technology, June 2021
(Chemical-biological Engineering, Computer Science & Molecular Biology)

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Advancing ACS' Core Value of Diversity, Equity, Inclusion and Respect

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Quick Guide: Inclusion Moments
Learn more about what Inclusion Moments are and see ideas to host them during your meetings.

Quick Guide: How to host inclusive in-person events
Recommendations and best practices to ensure that your events can accommodate everyone.

Diversity, Equity, Inclusion, and Respect

**Adapted from definitions from the Ford Foundation Center for Social Justice:

Equity**
Seeks to ensure fair treatment, equality of opportunity, and fairness in access to information and resources for all. We believe this is only possible in an environment built on respect and dignity. Equity requires the identification and elimination of barriers that have prevented the full participation of some groups.

Diversity**
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Inclusion**
Builds a culture of belonging by actively inviting the contribution and participation of all people. Every person's voice adds value, and ACS strives to create balance in the face of power differences. In addition, no one person can or should be called upon to represent an entire community.

Respect
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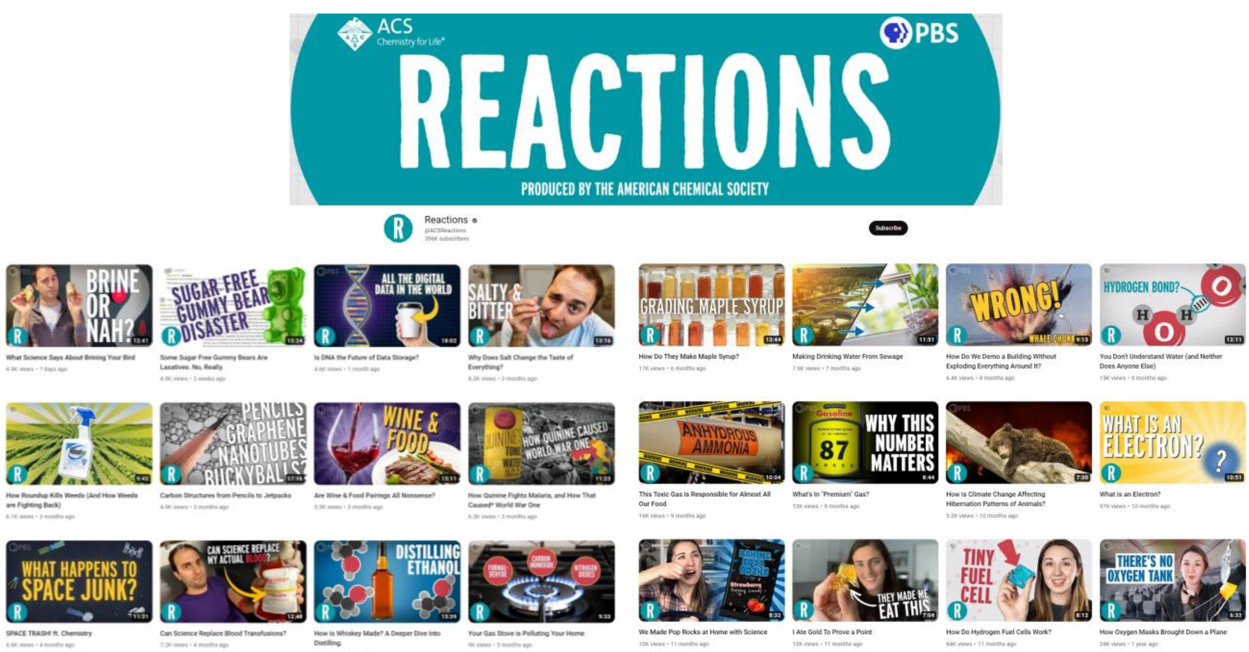

https://www.youtube.com/c/ACSReactions/videos

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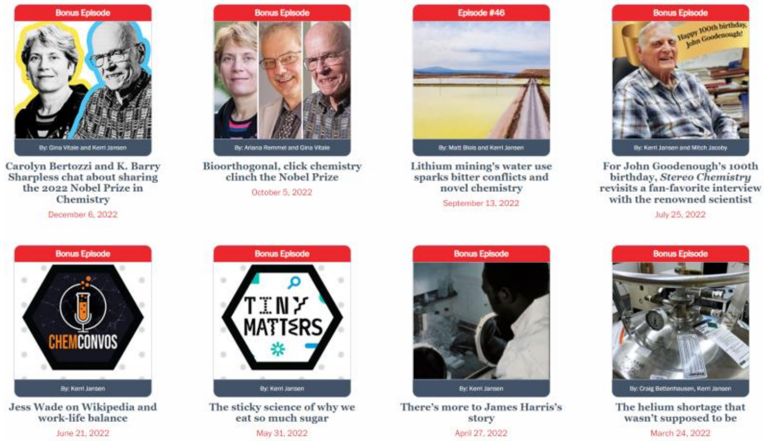

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Joanna Aizenberg
Harvard University

ACS Award in Polymer Chemistry
Karen I. Winey
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ACS Award in Pure Chemistry
Julia A. Kalow
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Arthur C. Cope Late Career Scholars Award
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Arthur C. Cope Mid-Career Scholars Award
Javier Read de Alaniz
University of California, Santa Barbara

E. V. Murphree Award in Industrial and Engineering Chemistry
Qinghuang Lin
Lam Research Corp.

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Wyatt Technology Corp.

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Cato T. Laurencin
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Ronald Breslow Award for Achievement in Biomimetic Chemistry
Laura L. Kiessling
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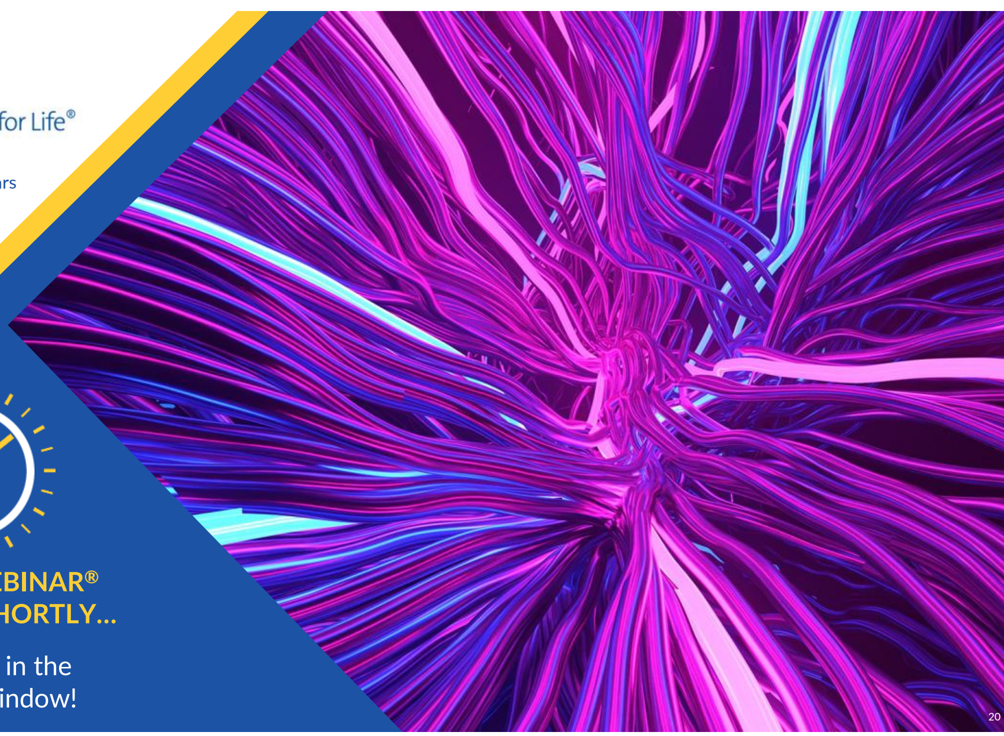

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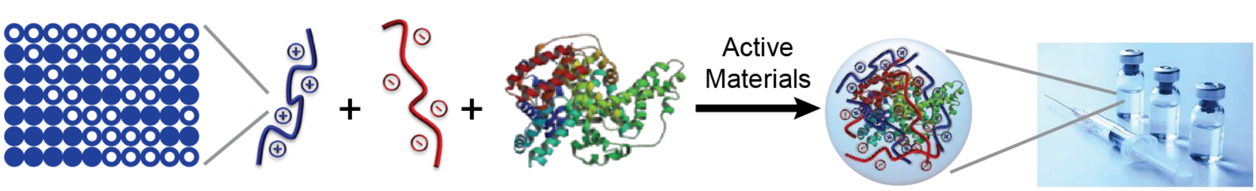
Molecular Engineering of Polyelectrolyte Complex Materials



Sarah L. Perry
University of Massachusetts Amherst

UMassAmherst

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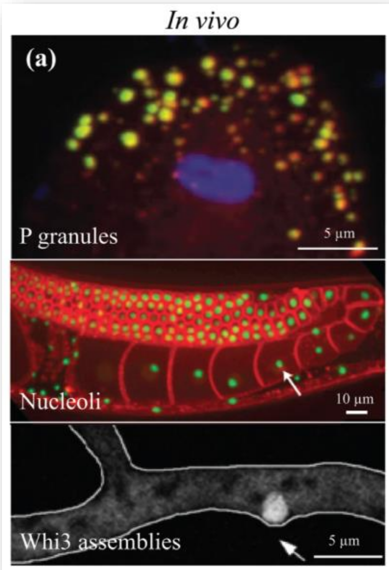
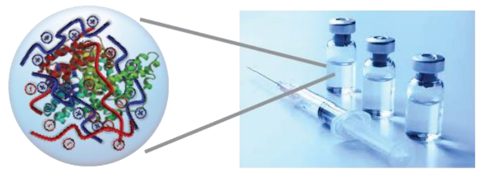
Inspiration: Phase Separation in Biology



Taylor *et al.*, *Soft Matter*, **2016**, *12*, 9142.

Membraneless Organelles/Biomolecular Condensates

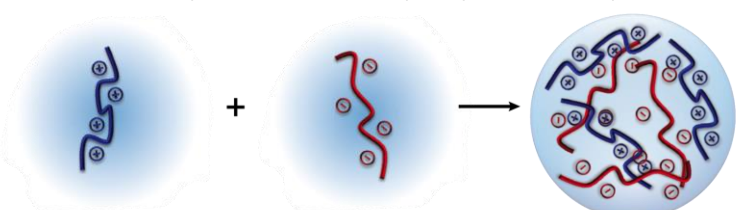
- Phase separation of intrinsically disordered proteins and RNA
- Can selectively sequester specific enzymes during periods of stress



- Need to understand the basic physics controlling these systems
- Can we design analogous materials?

Polyelectrolyte Complexation

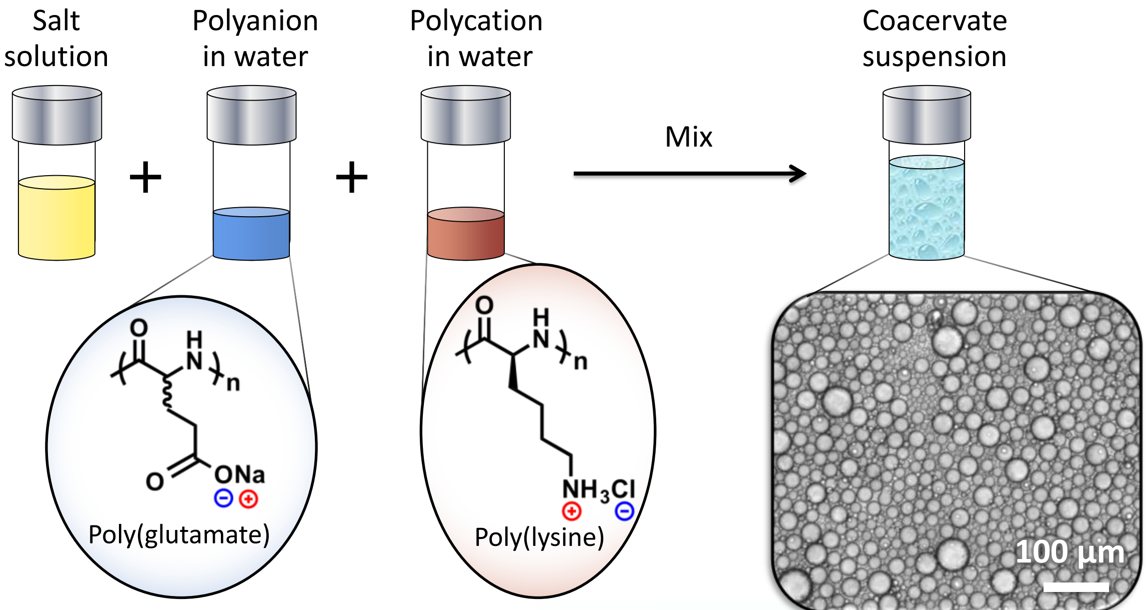
An associative phase separation resulting from electrostatic complexation of polyelectrolytes



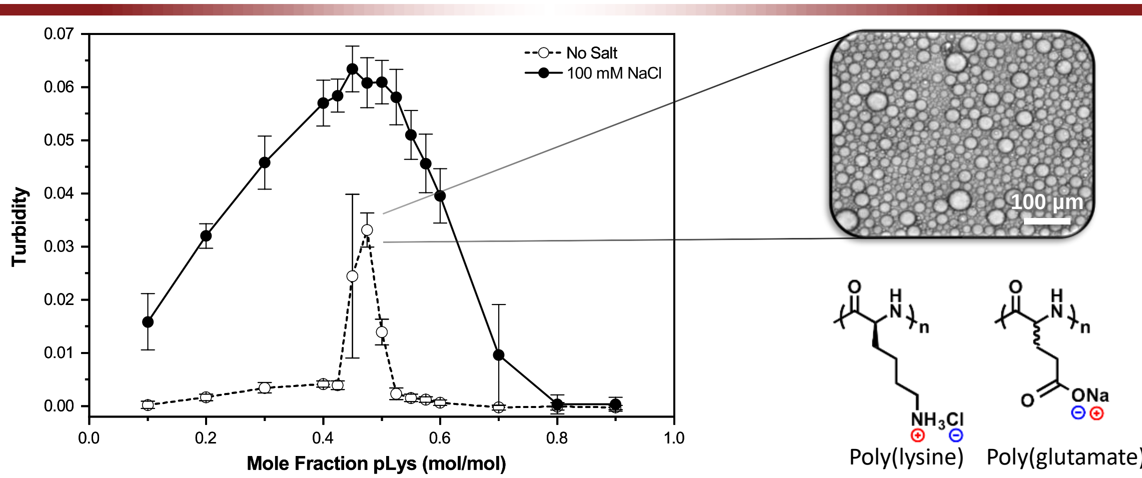
- Soluble complexes
- Complex coacervation
  - Drug delivery
  - Food and personal care
  - Adhesives
- Precipitated solid complexes

Hoffmann, Perry *et al.*, *Soft Matter*, **2015**, *11*, 1525.
Meng, Schiffman, Perry, *Macromolecules* **2018**, *51*, 8821. Patel *et al.*, Cell, **2015**, *162*(5), 1066.

Preparing Coacervates

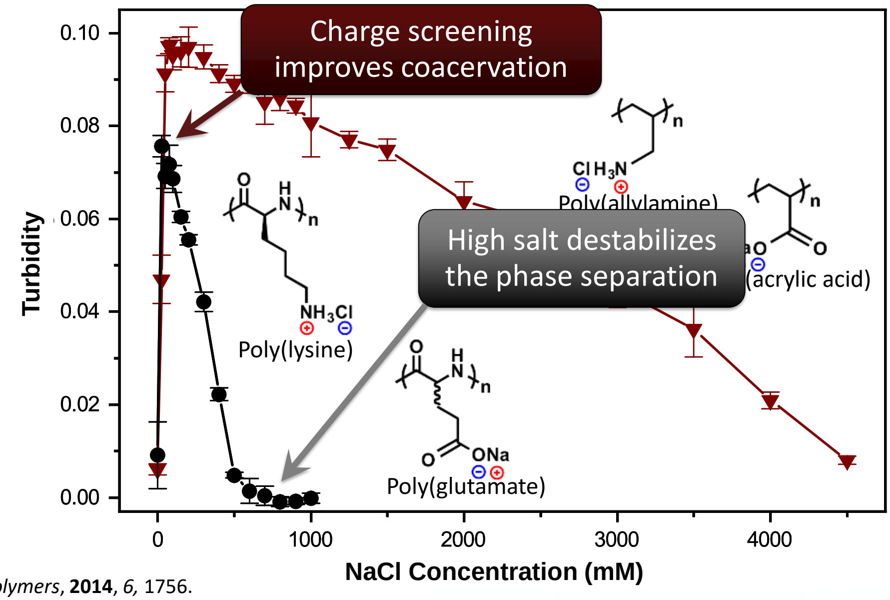


Controlling Coacervation: Charge Stoichiometry



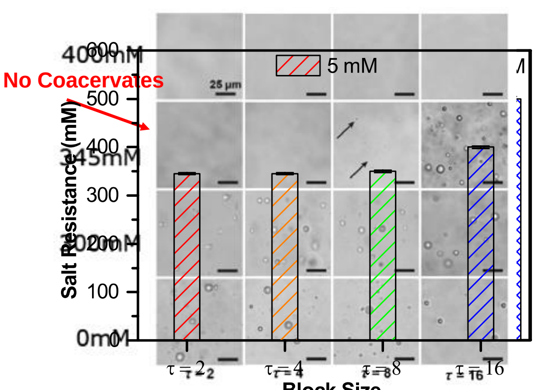
- Maximum complexation → charge stoichiometry
- Salt screening facilitates coacervate formation

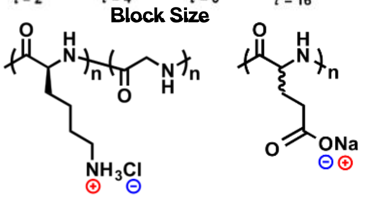
Coacervate Stability: Salt-Polymer Interactions

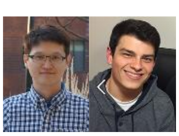


Perry, *et al.*, *Polymers*, **2014**, *6,* 1756.

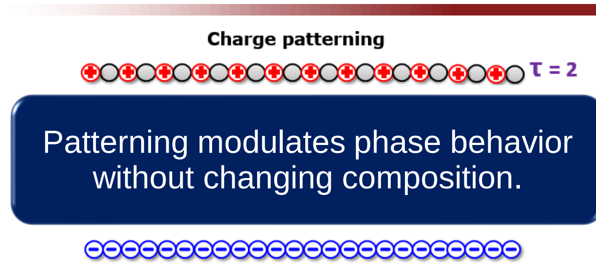
Charge Patterning and Phase Behavior

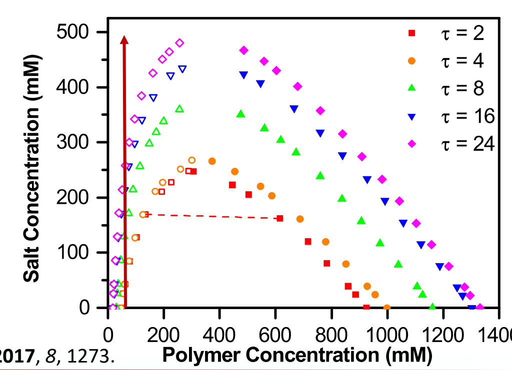






Dr. Li-Wei Chang, Jon Vélez



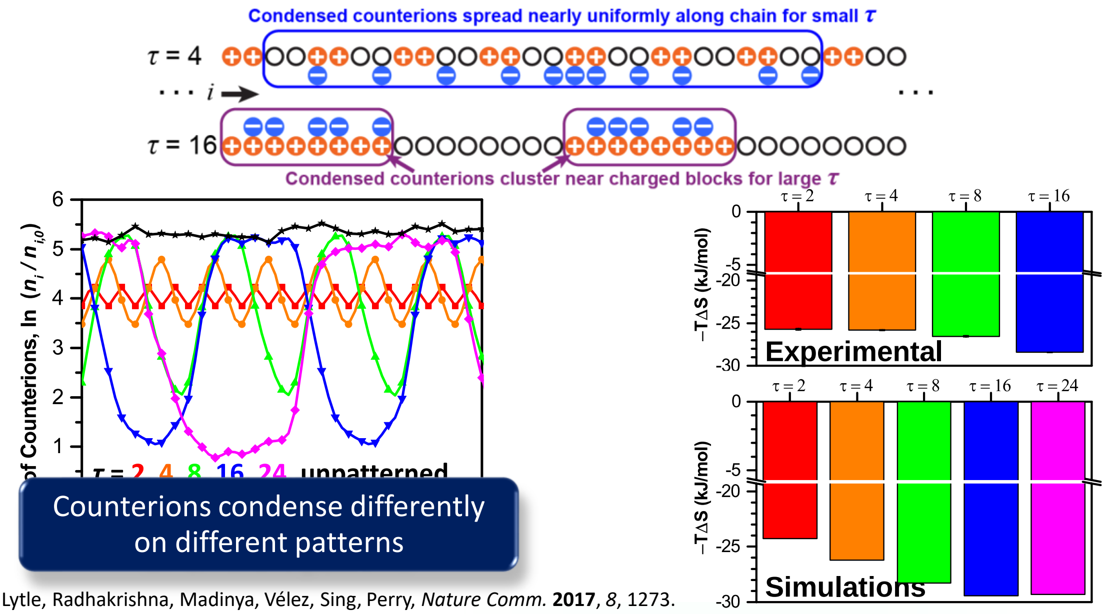


Prof. Charles Sing (UIUC)

Dr. Tyler Lytle

Chang, Lytle, Radhakrishna, Madinya, Vélez, Sing, Perry, *Nature Comm.* **2017**, *8*, 1273.

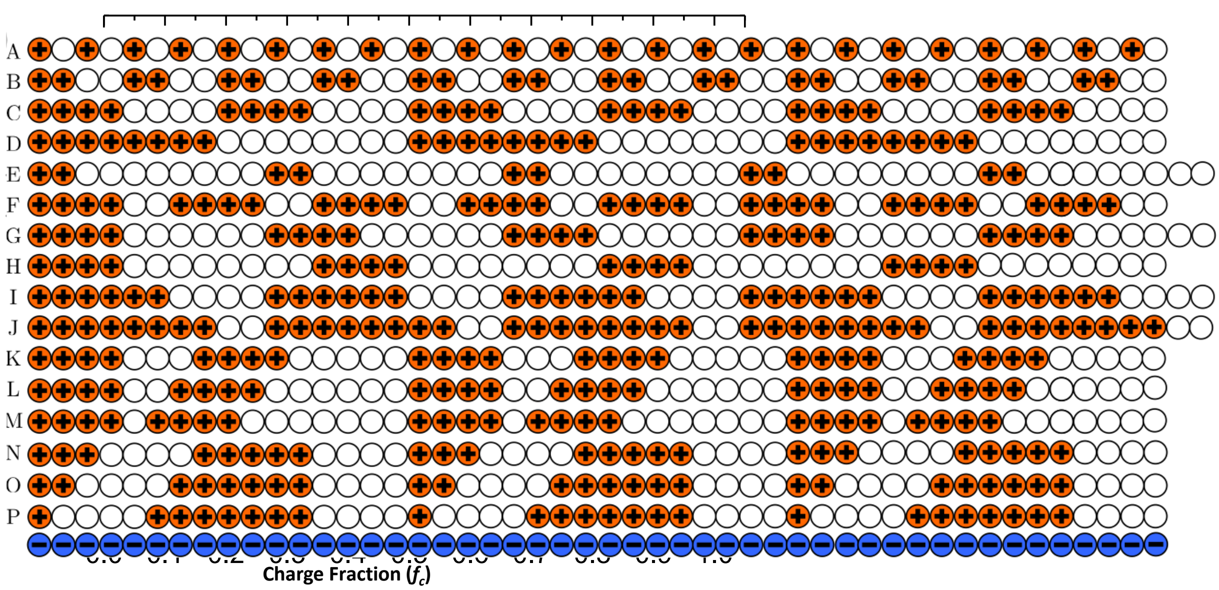
Charge Patterning - Thermodynamics



Counterions condense differently on different patterns

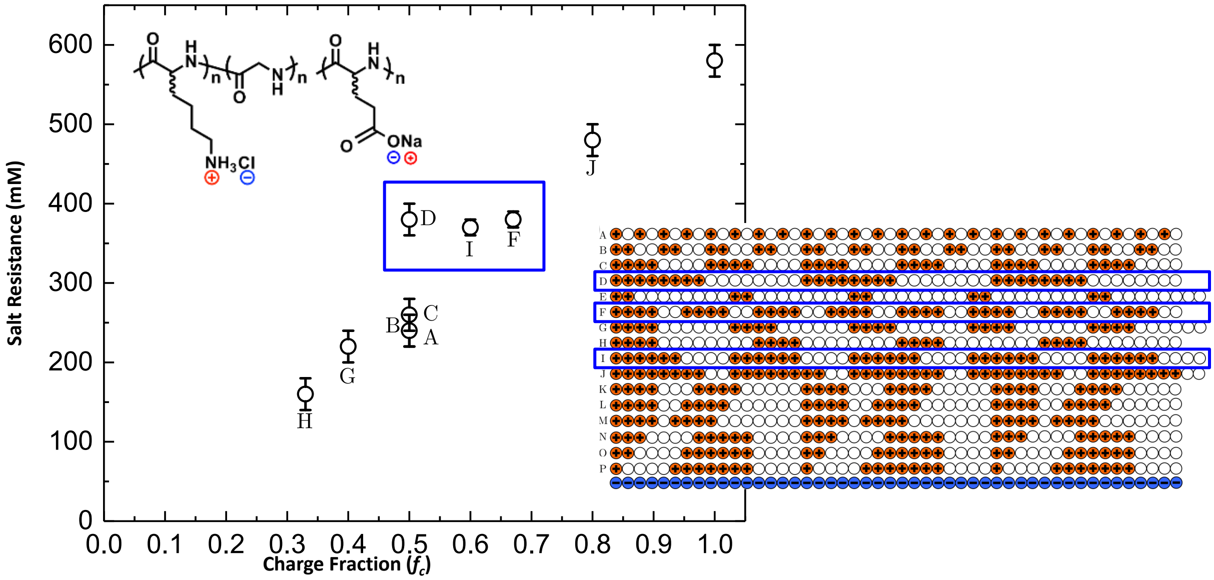
Chang, Lytle, Radhakrishna, Madinya, Vélez, Sing, Perry, *Nature Comm.* **2017**, *8*, 1273.

More Complex Patterns



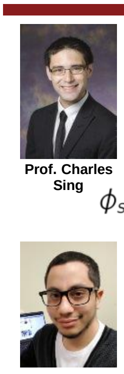
Lytle, Chang, Markiewicz, Perry, Sing, *ACS Central Science*, **2019**, *5*, 709.

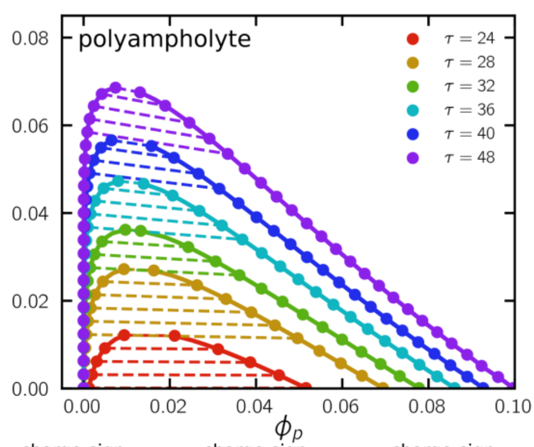
More Complex Patterns

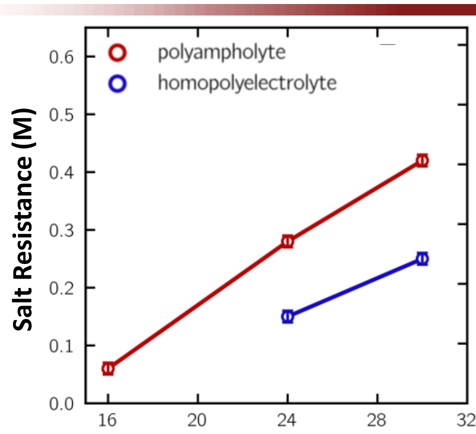


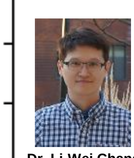
Lytle, Chang, Markiewicz, Perry, Sing, *ACS Central Science*, **2019**, *5*, 709.

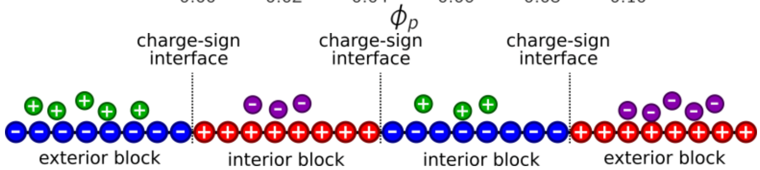
Self Coacervation of Polyampholytes







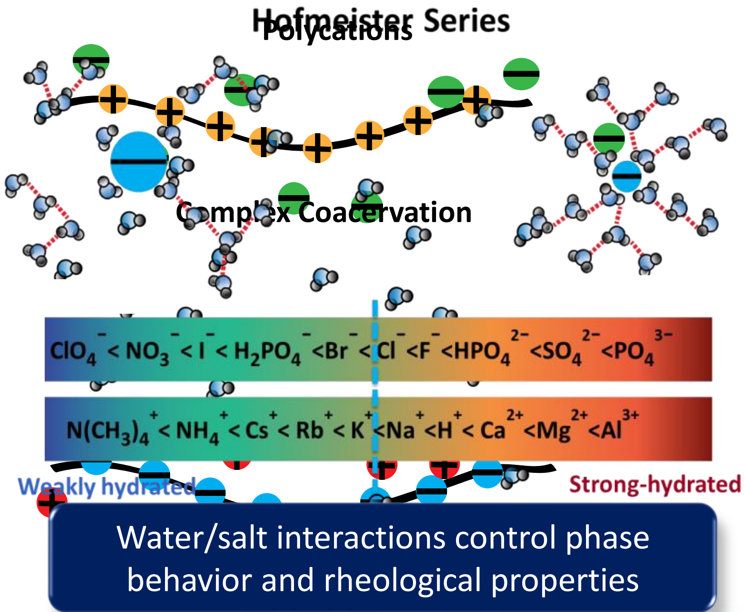


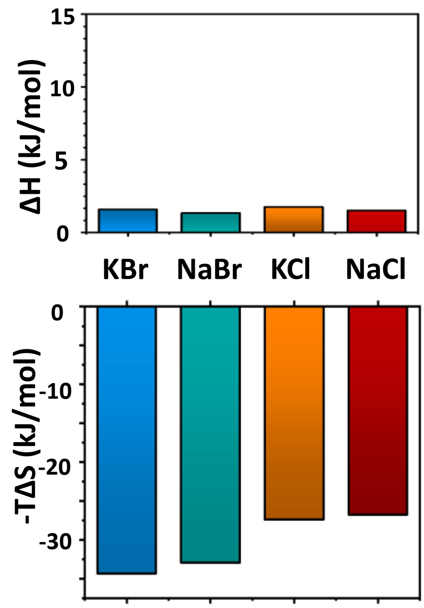


Blockiness is key for self-coacervation. Phase separation occurs for $\tau < 16$.

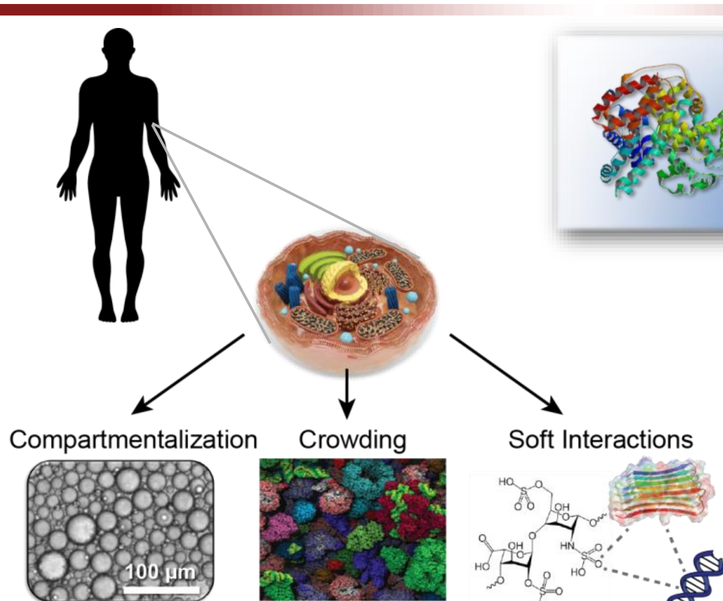
Madinya, Chang, Perry, Sing, *Mol. Syst. Des. Eng.* **2020,** *5,* 632-644.

Effects of Salt on Complex Coacervation

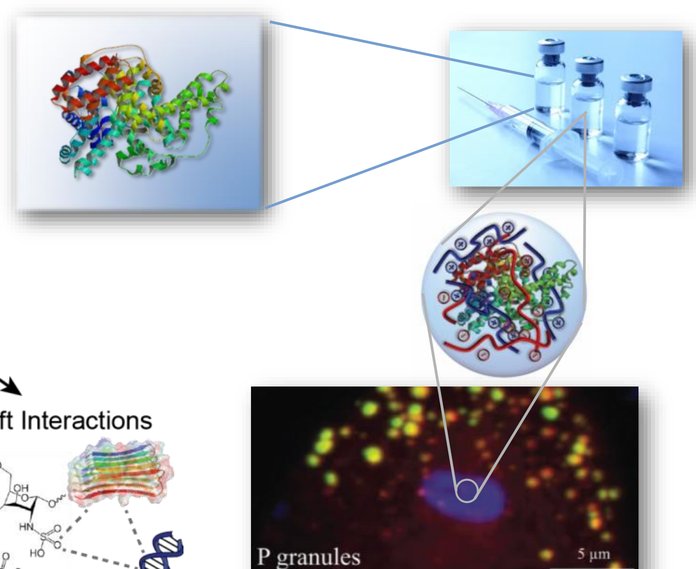




The Need for Protein/Biomolecule Encapsulation

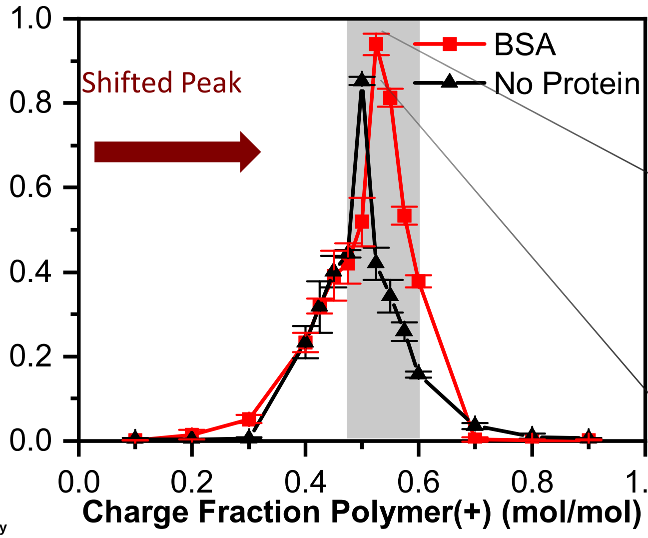


McGuffee and Elcock, *Plos Comp. Biol*, **2010**, *6*, e1000694.

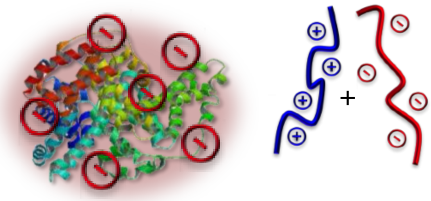


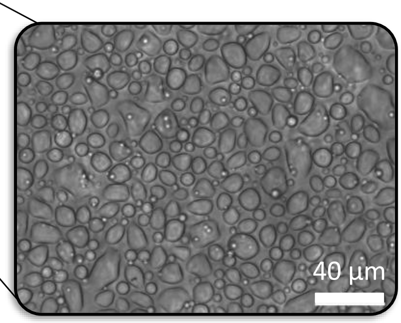
Taylor *et al.*, *Soft Matter*, **2016**, *12*, 9142.

Charge Neutrality and Ternary Coacervation



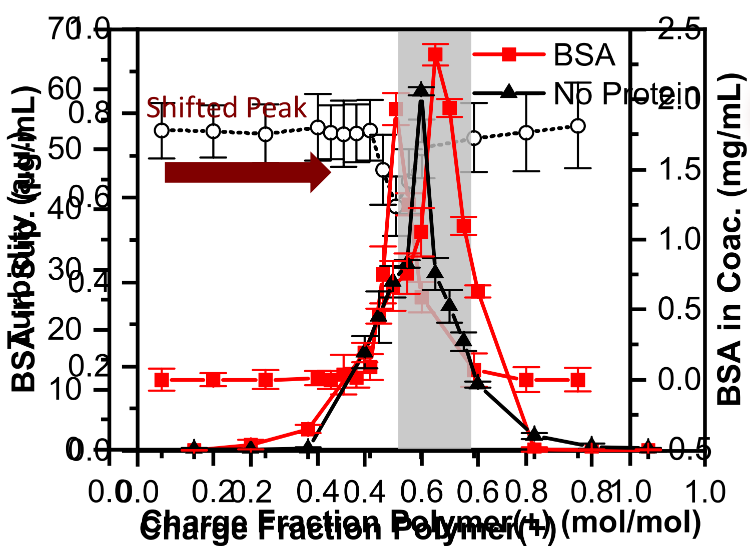
Prof. Whitney Blocher McTigue

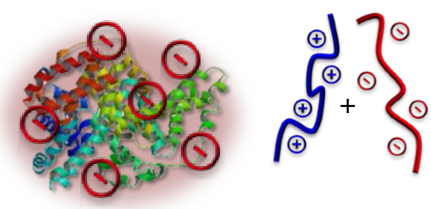




Blocher McTigue and Perry, *Soft Matter*, **2019**, *15*, 3089. Samples prepared in 10 mM HEPES, pH 7.0

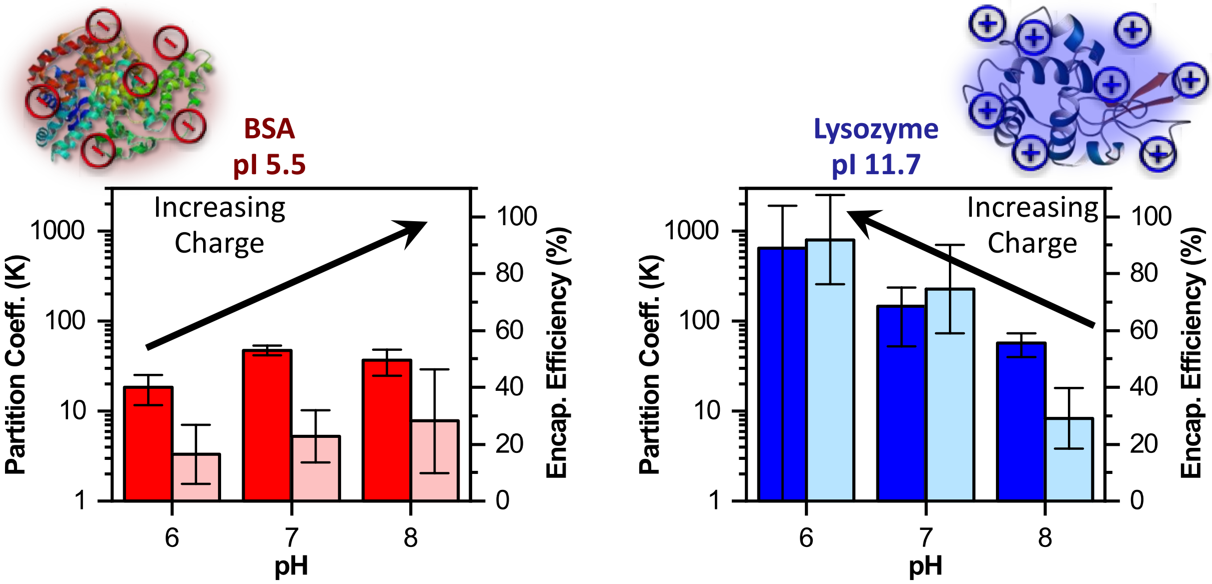
Charge Neutrality and Ternary Coacervation





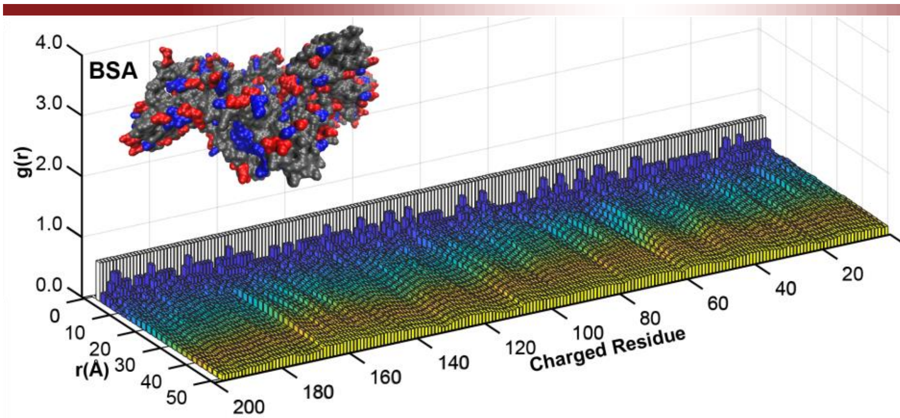
Blocher McTigue and Perry, *Soft Matter*, **2019**, *15*, 3089. Samples prepared in 10 mM HEPES, pH 7.0

Encapsulation Scales with Protein Charge

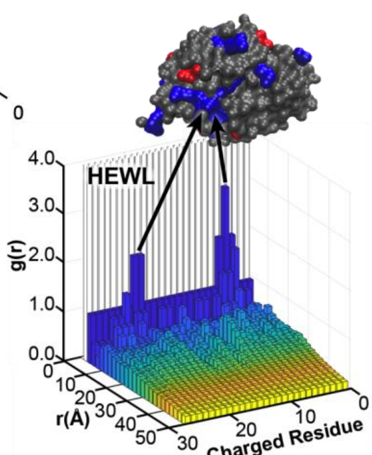


Blocher McTigue and Perry, *Soft Matter*, **2019**, *15*, 3089.

Charge Patchiness and Uptake

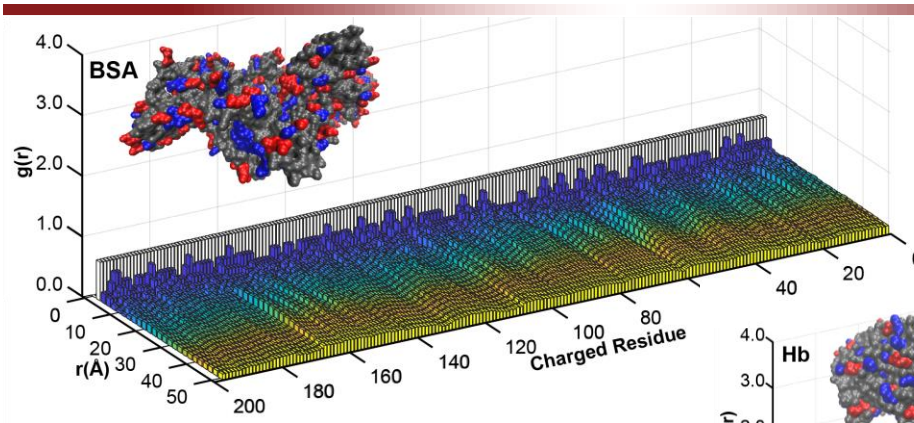


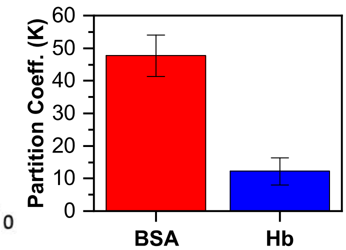
- Uptake is facilitated by charge clustering
- Diffuse surface charge enables uptake, but is less sensitive to design parameters



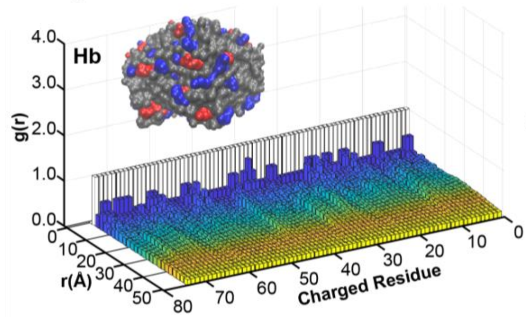
Blocher McTigue and Perry, *Soft Matter*, **2019**, *15*, 3089.
g(r) calculations performed using PDB: 3V03 for BSA and 1DPX for lysozyme, 2Å resolution for integration.

Charge Patchiness and Charge – Applying these Design Rules





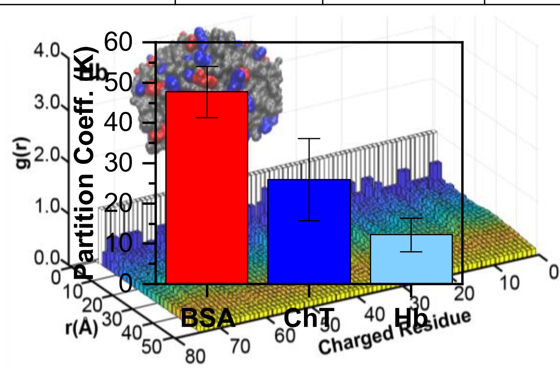
	pI	Net Charge pH 7.0	MW (kDa)
BSA	5.5	-15.3	66.4
Hemoglobin	9.0	2.8	64.5

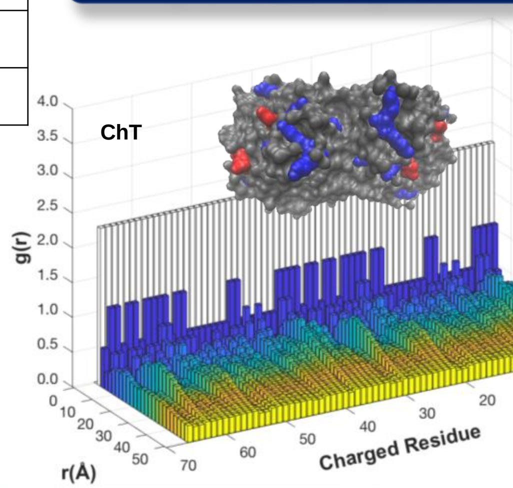


Charge Patchiness and Charge – Applying these Design Rules

	pI	Net Charge pH 7.0	MW (kDa)
BSA	5.5	-15.3	66.4
Hemoglobin	9.0	2.8	64.5
Chymotrypsin	9.7	3.0	51.0

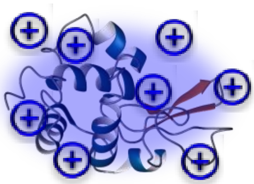
Net charge and charge patchiness promote encapsulation



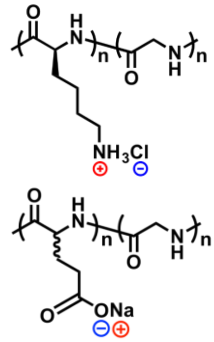


Yimin Sun

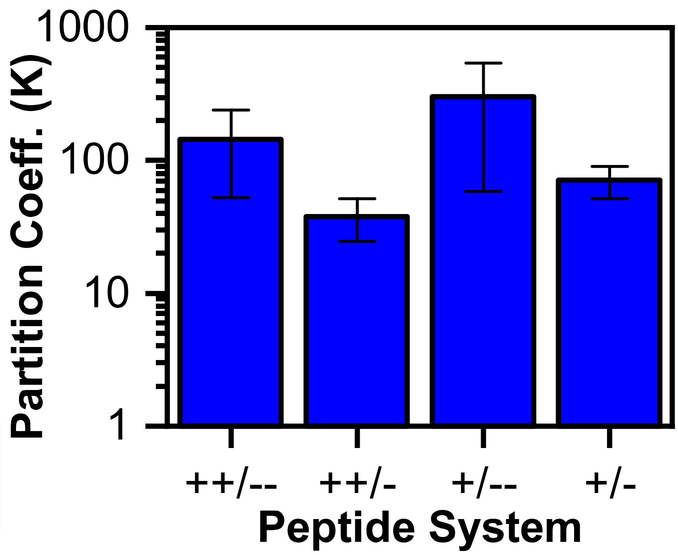
Protein Encapsulation vs. Peptide Charge



Lysozyme
pI 11.7

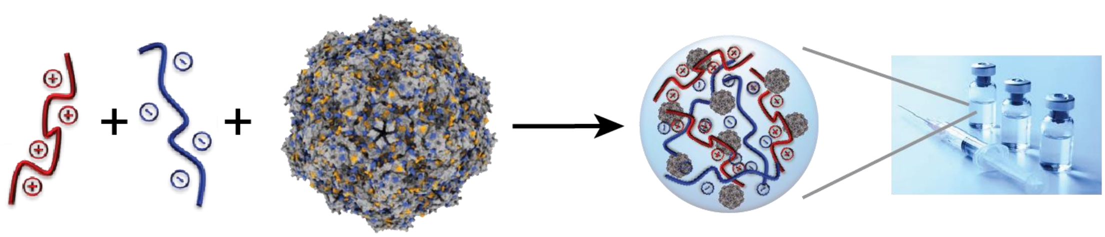


Encapsulation is dominated by competition between protein and peptide charge



Blocher McTigue and Perry, *Soft Matter*, **2019**, *15*, 3089. Samples prepared in 10 mM HEPES, pH 7.0

Encapsulating Viruses



Virus	Capsid	Family	Nucleic Acid	Size (nm)	pI	Related Human Viruses
Porcine parvovirus (PPV)	Non-enveloped	Parvoviridae	ssDNA	18-26	~5.0	B-19 human parvovirus
Bovine Viral Diarrhea Virus (BVDV)	Enveloped	Flaviviridae	ssRNA	40-60	–	Hepatitis C

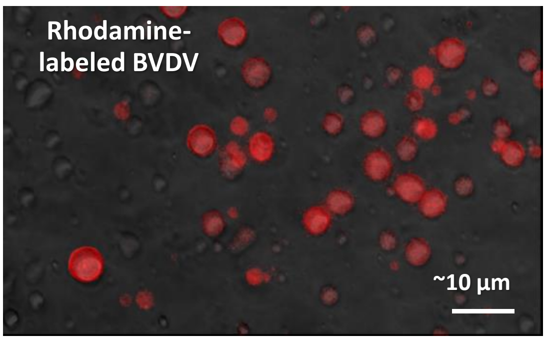
Prof. Caryn Heldt (MTU)

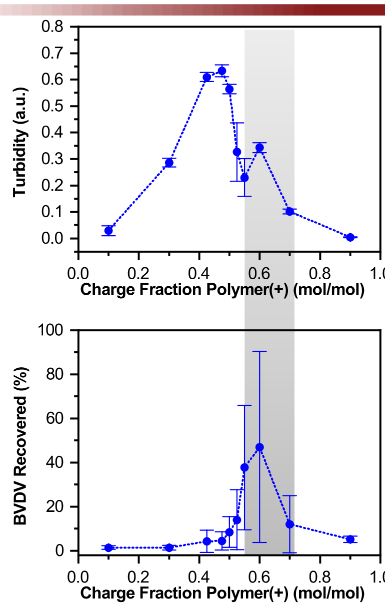
Dr. Xue Mi

Mi, Blocher McTigue, Joshi, Bunker, Heldt, Perry, *Biomaterials Science*, **2020**, *8*, 7082.

Selective Virus Encapsulation

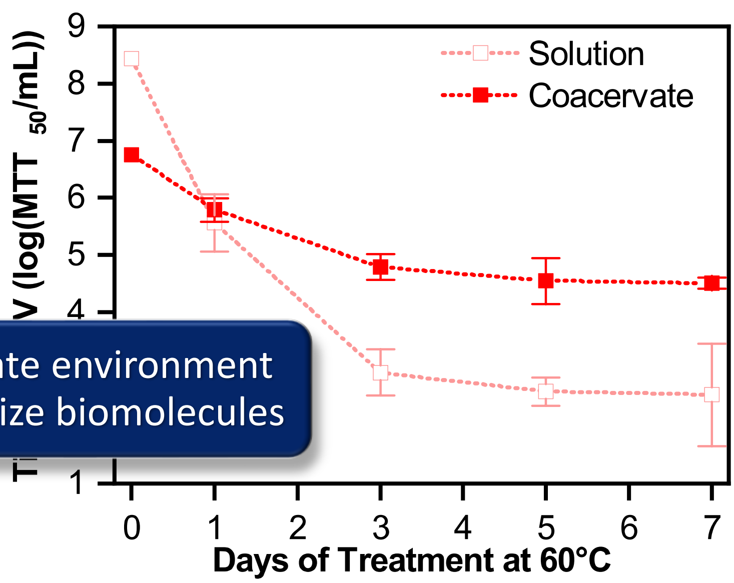
- Turbidity shows evidence of two distinct populations
- Quantification methods only measure infectious virus
- Possible purification strategy





Mi, Blocher McTigue, Joshi, Bunker, Heldt, Perry, *Biomaterials Science*, **2020**, *8*, 7082.

Uptake and Stabilization of PPV



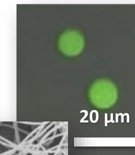
Coacervate environment can stabilize biomolecules

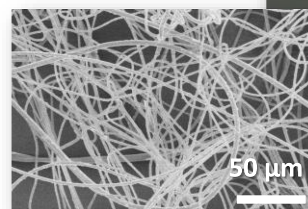
Mi, Blocher McTigue, Joshi, Bunker, Heldt, Perry, *Biomaterials Science*, **2020**, *8*, 7082.

Summary: Molecular Design of Material Environments

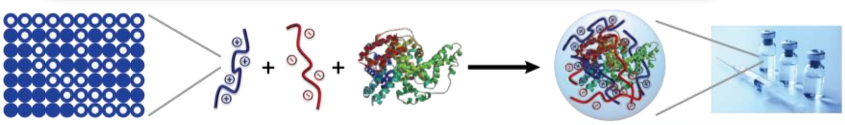
Understanding the self-assembly and phase behavior of coacervation is critical for its utility and translation

- Charge patterning dictates phase behavior
- Design of materials affects protein/cargo uptake
- Interplay between phase behavior and rheology
- Effective functional materials





- Control interactions through chemistry
- Design materials for specific applications



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- Shannon McIntosh
- Emily Ng
- Jon Vélez
- Timothy Wheeler
- Henry Xu

Soft Materials Life Sciences
NSF
CMMI: 1727660
CBET: 1804177
DMR: 1905559
DMR: 2118788
BASF We create chemistry
National Institutes of Health
CBI
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POLYELECTROLYTE-BASED COATINGS FOR FIRE AND FOOD PROTECTION

Jaime C. Grunlan

DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING,
DEPARTMENT OF MECHANICAL ENGINEERING,
& DEPARTMENT OF CHEMISTRY
TEXAS A&M UNIVERSITY

ACS Webinar – 26 January 2023

Polymer NanoComposites (PNC) Lab (http://nanocomposites.tamu.edu)

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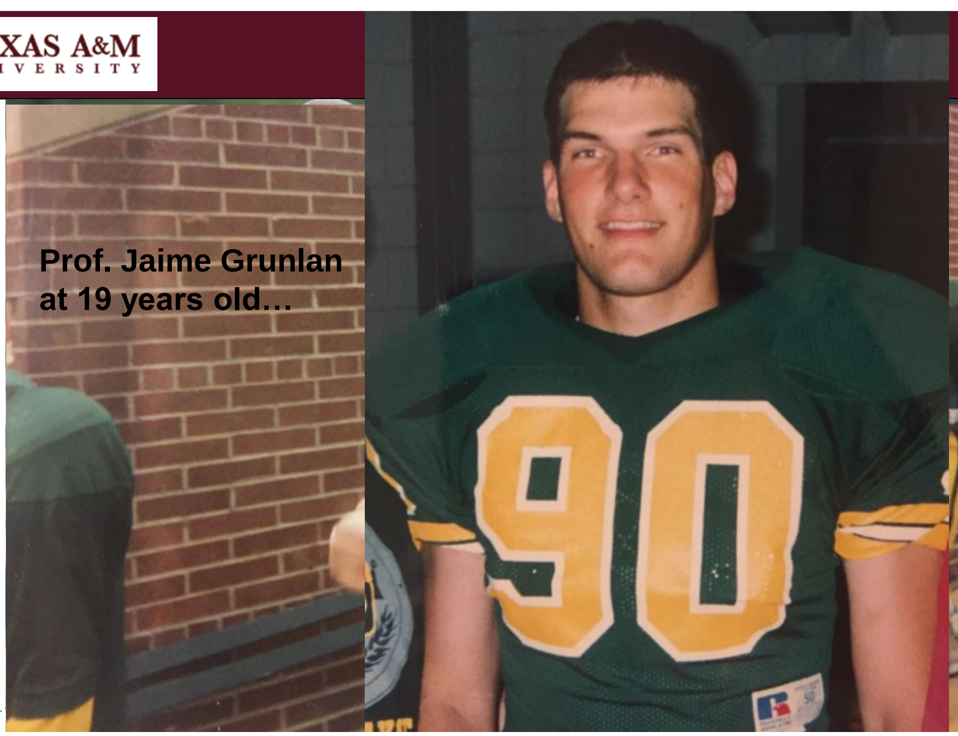

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Texas A&M University

Jaime Grunlan's Research Thrusts

Scientific Pillars:

- ❖**Polyelectrolyte complexation / assembly**
- ❖**Polymer-nanoparticle interactions**
- ❖**Water-based processing**
- ❖**Renewable chemistry**

Nature Rev. Mater. **2020**	*Adv. Mater. Interf.* **2019**	*Adv. Electronic Mater.* **2019**
ACS Mater. Lett. **2020**	*Macro. Rapid Comm.* **2017**	*Advanced Materials* **2018**
ACS AMI **2018**	*Green Materials* **2016**	*Nano Energy* **2016**
J. Mater. Sci. **2017**	*Macromolecules* **2015**	*Adv. Energy Mater.* **2016**
Adv. Mater. Interf. **2015**	*Langmuir* **2015**	*Advanced Materials* **2015**
Advanced Materials **2011**	*Macro. Rapid Comm.* **2015**	*ACS Nano* **2010**
ACS Nano **2009**	*ACS Macro Lett.* **2014**	*Nano Letters* **2008**

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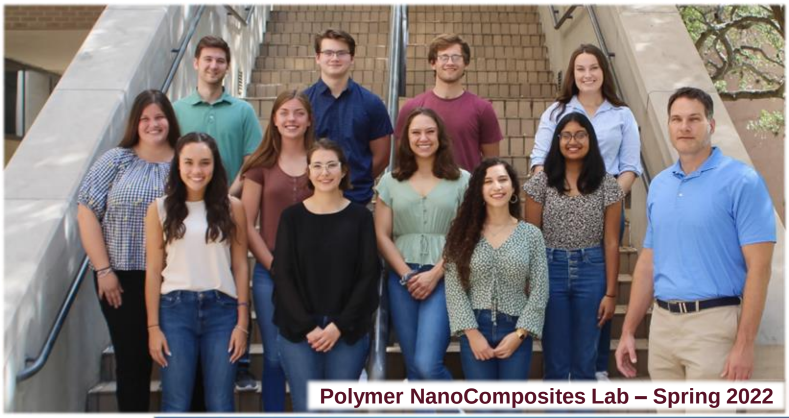
Acknowledgements



Polymer NanoComposites Lab – Spring 2022

NATO OTAN This activity is supported by: The NATO Science for Peace and Security Programme

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Collaborators:

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Xin Wang (USTC)
Guan Yang (UNSW)

Psalm 19:1-6

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Presentation Outline

- Overview of polyelectrolyte complexes of fire problem
- Flame retardant treatments
- Gas barrier from polyelectrolyte-based coatings

PNC

Polymer NanoComposites (PNC) Lab (http://nanocomposites.tamu.edu)

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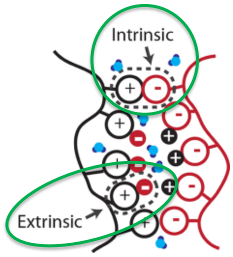
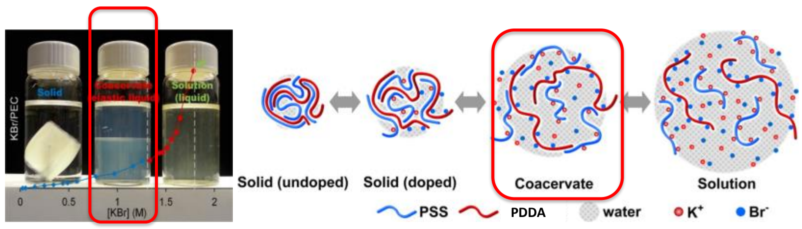
Polyelectrolyte Complexes



Zhang, Y., et al. *ACS Cent. Sci.* **2018**, *4*, 638.

Coulombic interactions cause polyelectrolyte complexation (PEC).

- Entropic driving force through expulsion of small counter ions and water.
- PEC form along a spectrum from insoluble complex to soluble solution.

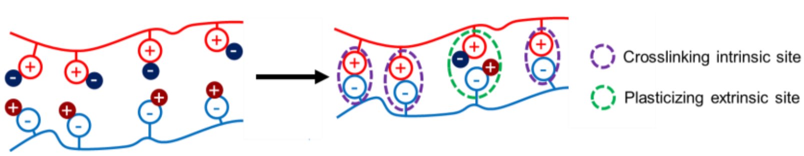


Chiang, H.-C.; Grunlan, J. C.; et al. *Macromol. Rapid Comm.* **2021**, *42*, 2000540.

Wang, Q., et al. *Macromolecules*, **2014**, *47*, 3108.

Polyelectrolyte Complexes

- Entropy-driven association of oppositely charged polyelectrolytes
  - DS > 0 due to expulsion of bound counterions

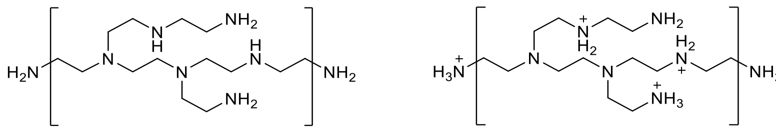


  - Complex can be plasticized with salt or water
- Complexation requires high charge density

J. Fu, J. B. Schlenoff, *Journal of the American Chemical Society* **2016**, *138*, 980.
J. Fu, H. M. Fares, J. B. Schlenoff, *Macromolecules* **2017**, *50*, 1066.

Weak Polyelectrolytes

- Polyelectrolyte with pH-dependent charge
  - Example: polyethylenimine



High pK_a
High pH = Low Charge

High pK_a
Low pH = High Charge

- Solution pH can serve as a stimulus to form a PEC on demand

Fire is a Problem…

- **More than 1.3 million fires reported in the U.S. in 2017**
- **Resulted in about 3400 civilian deaths, 15000 civilian injuries, and $23 billion in property damage**
- **Increased to $25.6 billion in property damage in 2018 as a result of a $12 billion loss due to wildfires in California**
- **Heat and flame protection of polymeric materials continues to be a major area of concern**

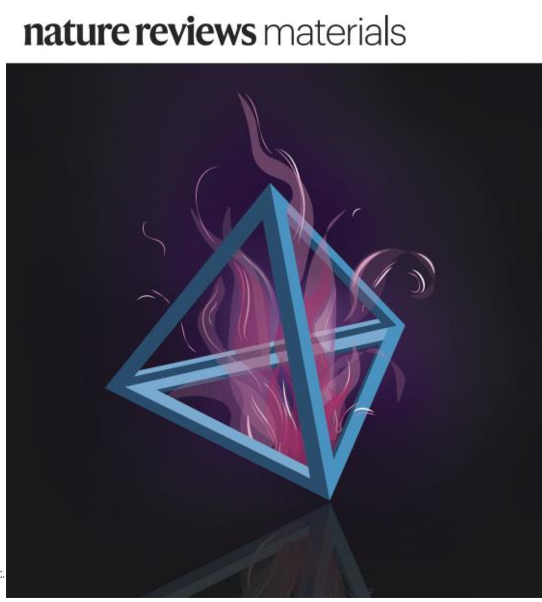
https://www.telegraph.co.uk/news/2017/07/19/grenfell-tower-cladding-would-have-burned-quickly-petrol-expert/
https://www.vox.com/world/2019/4/15/18311852/notre-dame-cathedral-fire-spire-collapse-photos-pictures-paris-france
https://www.niehs.nih.gov/health/materials/flame_retardants_508.pdf
http://www.latimes.com/local/lanow/la-me-ln-school-bus-fire-20150909-story.html
https://www.nfpa.org/News-and-Research/Publications/NFPA-Journal/2018/September-October-2018/Features/2017-US-Fire-Loss-Report

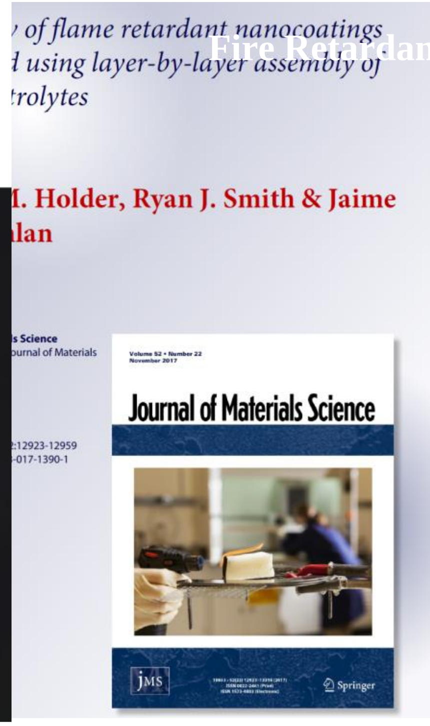
TE
UNI

Fire Retardant Facts

April 2020 volume 5 no. 4
www.nature.com/natrevmats

Review of flame retardant surface treatments…





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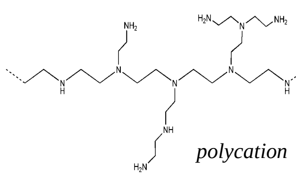
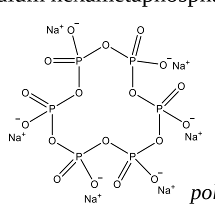
Flame retardant PEC

Polyethylenimine (PEI)



polycation

Sodium hexametaphosphate (PSP)



polyanion

pKa of 8.25

- Chemistry helps to form insulating char
- pH affects PEI degree of protonation
- Polyelectrolytes flocculate at pH ≤ 8, but mutually suspended above pH 9

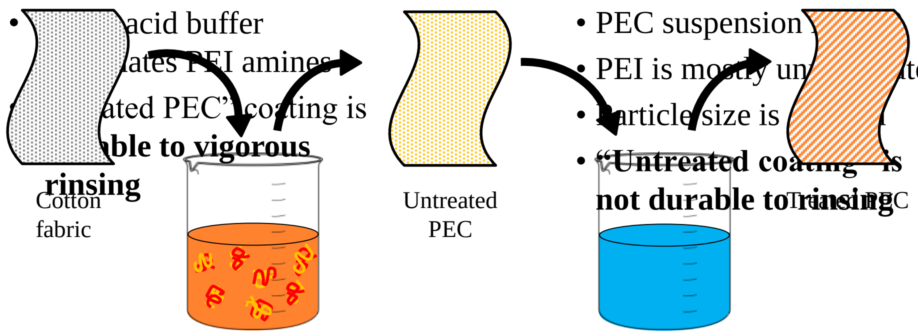
M. Haile, C. Fincher, S. Fomete, J. C. Grunlan, *Polym. Degrad. Stab*. **2015**, 114, 60–64.

J. C. Grunlan, "Aqueous Polyelectrolyte Complex as One Pot Nanocoating Solution to Impart Antiflammable Behavior to Various Substrates," **U.S. Patent 9,840,629**.

PEC coating of cotton fabric

- ... acid buffer ...ates PEI amines
- ...ated PEC" coating is ...ble to vigorous rinsing

- PEC suspension ...
- PEI is mostly un...ted
- Particle size is ...
- **"Untreated coating" is not durable to rinsing**



M. Haile, C. Fincher, S. Fomete, J. C. Grunlan, *Polym. Degrad. Stab*. **2015**, 114, 60–64.

J. C. Grunlan, "Aqueous Polyelectrolyte Complex as One Pot Nanocoating Solution to Impart Antiflammable Behavior to Various Substrates," **U.S. Patent 9,840,629**.

Flame testing of cotton

Uncoated control

Untreated PEC coating

PEC coating treated by pH 2 buffer

M. Haile, C. Fincher, S. Fomete, J. C. Grunlan, *Polym. Degrad. Stab*. **2015**, 114, 60–64.

J. C. Grunlan, "Aqueous Polyelectrolyte Complex as One Pot Nanocoating Solution to Impart Antiflammable Behavior to Various Substrates," **U.S. Patent 9,840,629**.

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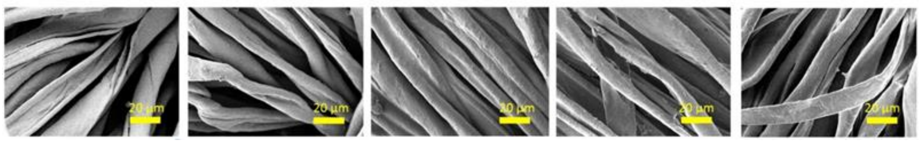
Microscopy of coated cotton

Uncoated control | Untreated coating | pH 6 treatment | pH 4 treatment | pH 2 treatment



M. Haile, C. Fincher, S. Fomete, J. C. Grunlan, *Polym. Degrad. Stab*. **2015**, 114, 60–64.

J. C. Grunlan, "Aqueous Polyelectrolyte Complex as One Pot Nanocoating Solution to Impart Antiflammable Behavior to Various Substrates," **U.S. Patent 9,840,629**.

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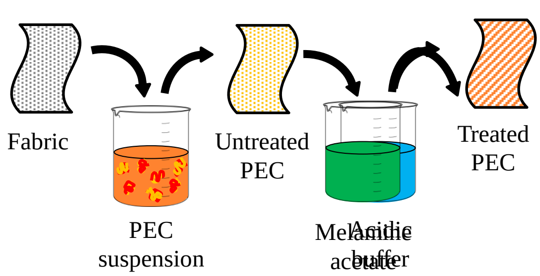
PEC for nylon-cotton

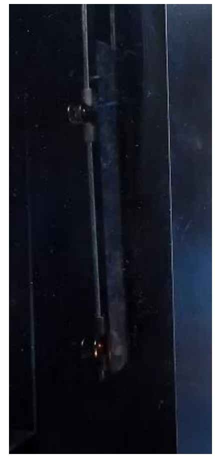
- Nylon-cotton (NYCO) fabric particularly challenging substrate
- Phosphate acts to catalyze the charring of cellulose
- Melamine polyphosphate can add further FR protection



M. Leistner, M. Haile, S. Rohmer, A. Abu-Odeh, J. Grunlan, *Polym Degrad Stab,* **2016,** *122,* 1-7.

Flammability of NYCO

no coating



19 wt% (PEC + Mel$^{2\%}$)

M. Leistner, M. Haile, S. Rohmer, A. Abu-Odeh, J. Grunlan, *Polym Degrad Stab,* **2016,** *122,* 1-7.

J. C. Grunlan, "Aqueous Polyelectrolyte Complex as One Pot Nanocoating Solution to Impart Antiflammable Behavior to Various Substrates," **U.S. Patent 9,840,629**.

Flammability of NYCO

control | PEC | PEC + Mel$^{2\%}$ | PEC + Mel$^{5\%}$

PEC: 7 wt% PEI + 14 wt% APP

M. Leistner, M. Haile, S. Rohmer, A. Abu-Odeh, J. Grunlan, *Polym Degrad Stab,* **2016,** *122,* 1-7.

J. C. Grunlan, "Aqueous Polyelectrolyte Complex as One Pot Nanocoating Solution to Impart Antiflammable Behavior to Various Substrates," **U.S. Patent 9,840,629.**

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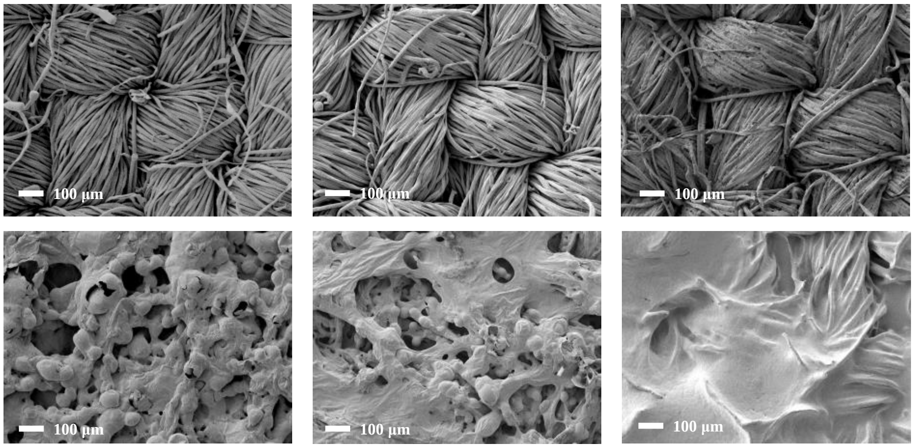
Melamine for char-improvement

no coating | PEC | PEC + Mel[2%]



Melamine addition creates strong, dense char that acts as heat shield and barrier to oxygen and volatiles.

M. Leistner, M. Haile, S. Rohmer, A. Abu-Odeh, J. Grunlan, *Polym Degrad Stab,* **2016,** *122,* 1-7.

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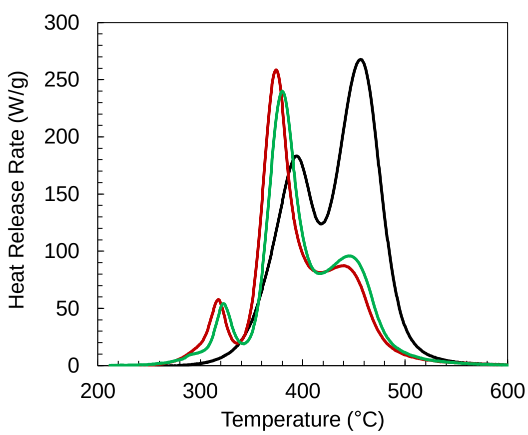
Heat release of NYCO



No coating
- 2 peaks
- THR: 19.1 kJ/g

PEC
- 3 peaks
- THR: 13.6 kJ/g

PEC + Mel[2%]
- 3 peaks
- THR: 13.7 kJ/g

PCFC (aka MCC) not able to detect char density or cooling effects.

M. Leistner, M. Haile, S. Rohmer, A. Abu-Odeh, J. Grunlan, *Polym Degrad Stab,* **2016,** *122,* 1-7.
Patent Pending

TEXAS A&M UNIVERSITY

Heat sink during pyrolysis

	VFT	THR (by PCFC)	Energy balance* (260 – 500°C)
no coating	burned off	19.1 kJ/g	+ 340 J/g
PEC	burned off	13.6 kJ/g	+ 70 J/g
PEC + Mel[2%]	self-extinguishing	13.7 kJ/g	– 60 J/g

* measured by DSC in N_2 at a heating rate of 10 K/min

DSC reveals a change in the energy balance during pyrolysis that reveals melamine addition making a more endothermic situation.

M. Leistner, M. Haile, S. Rohmer, A. Abu-Odeh, J. Grunlan, *Polym Degrad Stab,* **2016,** *122,* 1-7.

J. C. Grunlan, "Aqueous Polyelectrolyte Complex as One Pot Nanocoating Solution to Impart Antiflammable Behavior to Various Substrates," **U.S. Patent 9,840,629**.

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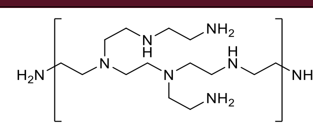
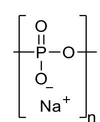
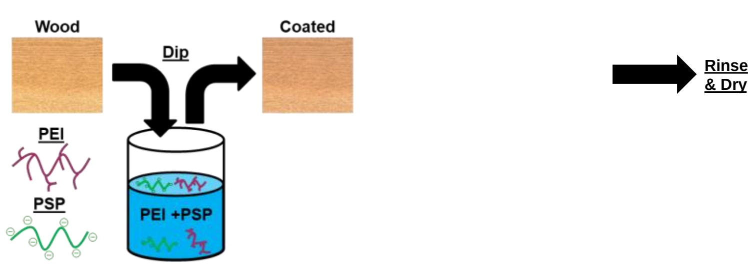
PEC Flame Retardant for Wood

- 7.5 wt% Polyethenylenimine (PEI)
  - M_n = 10 kDa, M_w = 25 kDa, pH 9
- 15 wt% Sodium hexametaphosphate (PSP)
  - M_n ~ 3 kDa (estimated)
- Citric Acid (100 mM, pH 3)
- Wood (Slices of untreated 2x4 stud)







T. J. Kolibaba, J. C. Grunlan, *Macromol. Mater. Eng.* **2019**, *304*, 1900179.

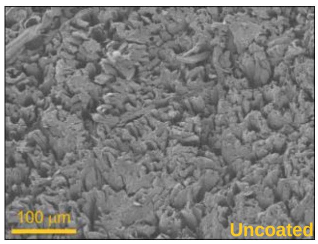
Properties of Coated Wood

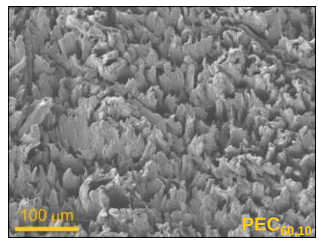
- Preserves aesthetic of wood
- Weight gain
  - Depends on dip time and cure time

$PEC_{Dip,Cure}$	$PEC_{1,1}$	$PEC_{1,10}$	$PEC_{60,1}$	$PEC_{60,10}$
Weight Gain (%)	1.3 ± 0.3	1.3 ± 0.5	4.2 ± 0.2	5.9 ± 1.0

$PEC_{x,y}$
x = Dip time
y = Cure time

- Conforms to wood microstructure



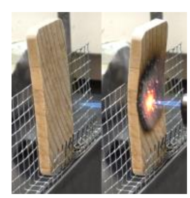


T. J. Kolibaba, J. C. Grunlan, *Macromol. Mater. Eng.* **2019**, *304*, 1900179.

Fire Testing of Wood

- Homebuild blowtorch test
  - Burn sample for 45 s with butane torch

Uncoated $PEC_{60,10}$ Uncoated $PEC_{60,10}$



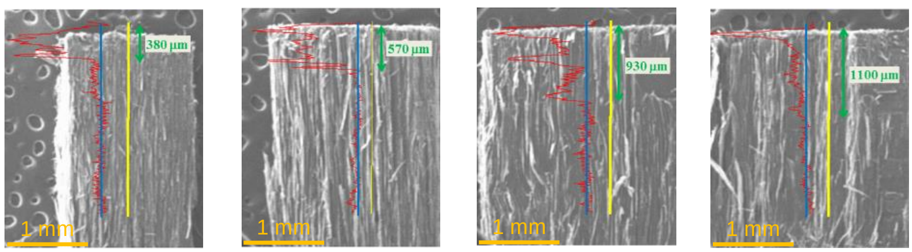
0 s 20 s

$PEC_{Dip,Cure}$	Control	$PEC_{1,1}$	$PEC_{1,10}$	$PEC_{60,1}$	$PEC_{60,10}$
Weight Gain (%)	-	1.3 ± 0.3	1.3 ± 0.5	4.2 ± 0.2	5.9 ± 1.0
Afterflame (s)	246 ± 18	171 ± 23	139 ± 92	53 ± 73	4.3 ± 0.6
Residue (%)	15.8 ± 0.6	52 ± 17	65 ± 18	78 ± 16	90.4 ± 0.6

T. J. Kolibaba, J. C. Grunlan, *Macromol. Mater. Eng.* **2019**, *304*, 1900179.

Depth of PEC Penetration

- Analyzed with EDS mapping of phosphorus
  - Natural wood contains almost no phosphorus
  - Phosphorus signal indicates penetration by PSP



$PEC_{1,1}$ (1.3% wt. gain) — $PEC_{1,10}$ (1.3% wt. gain) — $PEC_{60,1}$ (4.2% wt. gain) — $PEC_{60,10}$ (5.9% wt. gain)

T. J. Kolibaba, J. C. Grunlan, *Macromol. Mater. Eng.* **2019**, *304*, 1900179.

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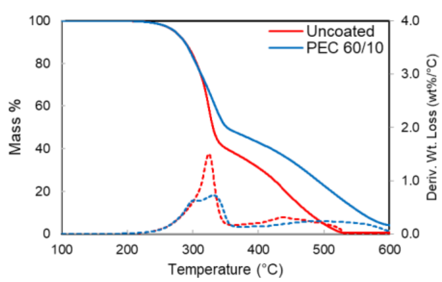
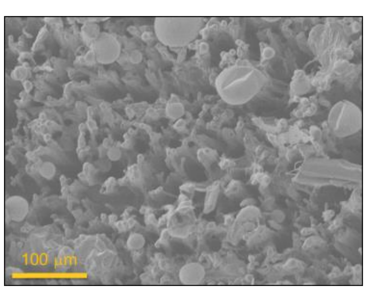
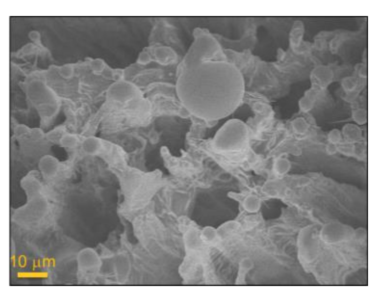
Fire Protection of Wood



	Uncoated	$PEC_{60,10}$	*Change*
TTI (s)	49 ± 9	62 ± 6	*+27%*
pkHRR(kW/m²)	379 ± 9	338 ± 18	*-11%*
THR (MJ/m²)	29.0 ± 0.8	19.0 ± 0.7	*-35%*
TSR (m²/m²)	209 ± 9	210 ± 12	*N/A*
Residue (%)	26 ± 3	42 ± 3	*+62%*



$PEC_{60,10}$ Post-burn



T. J. Kolibaba, J. C. Grunlan, *Macromol. Mater. Eng.* **2019**, *304*, 1900179.
M. Jimenez, T. Guin, S. Bellayer, R. Dupretz, S. Bourbigot, J. C. Grunlan, *Journal of Applied Polymer Science* **2016**, *133*, 43783.

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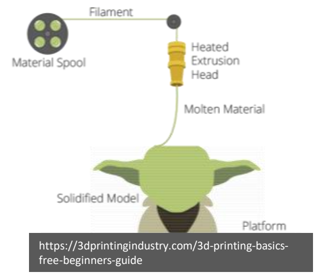
Additive Manufacturing

- Fused Filament Fabrication
  - Filaments are flammable thermoplastics
    - Causes fires, limits part applications



s counterproductive for filaments

retardant not localized to surface of part

3D printer blamed for fire inside Cain Building on Texas A&M campus

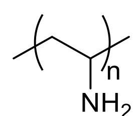
Firefighters say a 3D printer started a fire inside a classroom Wednesday afternoon inside the James J. Cain Building on the Texas A&M campus.

https://kbtx.com

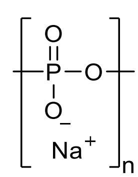
C. B. Sweeney, B. A. Lackey, M. J. Pospisil, T. C. Achee, V. K. Hicks, A. G. Moran, B. R. Teipel, M. A. Saed, M. J. Green, *Sci. Adv.* **2017**, *3*, e1700262.

Flame Retardant Filament

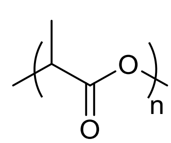
- Polyvinylamine (PVA)
  - BASF Lupamin 9095
  - Estimated M ~ 205 kDa



- Sodium hexametaphosphate (PSP)
  - M_n ~ 3 kDa (estimated)



- Polylactic acid (PLA)
  - 3D Solutech filament
  - Most common 3D printing filament



T. J. Kolibaba, C.-C. Shih, S. Lazar, B. L. Tai, J. C. Grunlan, *ACS Materials Letters* **2020**, *2*, 15.

PEC Production and Processing

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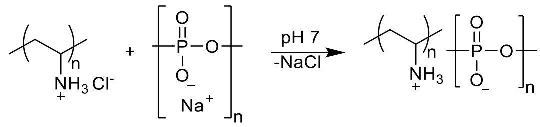
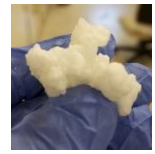
- Mix PVA & PSP
  - Separate solutions each pH 7, 0.25 M



  - Dry in overnight at 120 °C



- Resultant PEC can be extruded
  - Plasticize with DI water, extrude at 90 °C

- Intrinsically flame retardant

T. J. Kolibaba, C.-C. Shih, S. Lazar, B. L. Tai, J. C. Grunlan, *ACS Materials Letters* **2020**, *2*, 15.

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3D Printing

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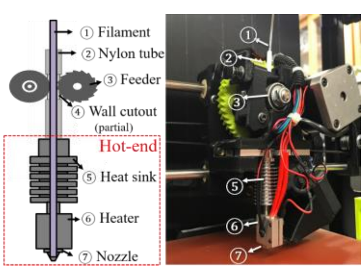
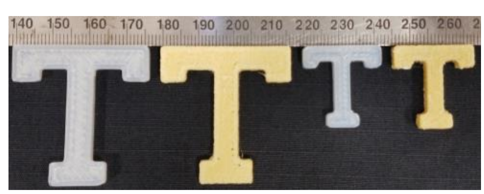
- Filament
  - 25% PEC, 75% PLA
  - Mixed in microcompounder/extruder
    - Plasticized with DI water prior to extrusion
  - Printed at 200 °C, 3000 mm/min
    - Identical to 'normal' parameters for PLA

MI-LAB
MANUFACTURING INNOVATION LABORATORY





PLA PLA-PEC

T. J. Kolibaba, C.-C. Shih, S. Lazar, B. L. Tai, J. C. Grunlan, *ACS Materials Letters* **2020**, *2*, 15.

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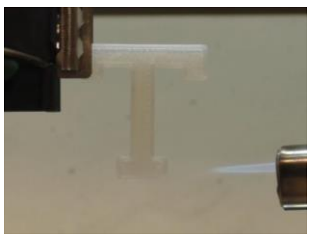
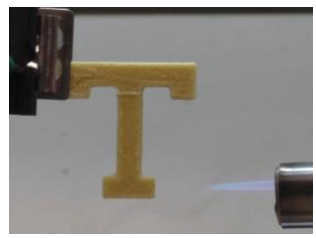
Printed Part Flame Retardancy

- Microscale Combustion Calorimetry

Sample	Char Yield (wt%)	pkHRR (W/g)	pkHRR Temp (°C)	THR (kJ/g)
PLA	0.8 ± 0.2	530 ± 40	392 ± 5	16.8 ± 0.1
PLA-PEC	13.6 ± 0.3	309 ± 3	391	13.6 ± 0.1
Change	***+1600%***	***-42%***	***-***	***-19%***

- Open flame test





T. J. Kolibaba, C.-C. Shih, S. Lazar, B. L. Tai, J. C. Grunlan, *ACS Materials Letters* **2020**, *2*, 15.

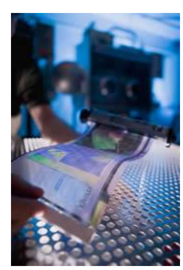
Applications for Gas Barrier Films

Food Packaging

http://www.cptplastics.com/Low%20O2trays_pouches_cr.html

Medical Packaging

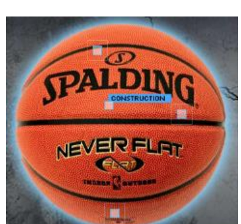
www.alcan-packaging.com



Flexible Displays

http://flexdisplay.asu.edu

www.bfgoodrichtires.com



www.spalding.com

www.sealmaster.com

Inflatable Objects

Tires

Sports equipment

Seals

Pumps

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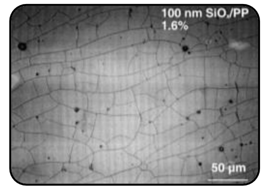
Established Transparent Barrier Technologies

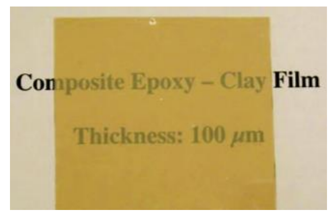
- <u>Silicon Oxide (SiO_x) Barrier Film</u>
  - Transparency, microwavability
  - Cracking, poor adhesion to polymer
  - Requires vacuum processing (PECVD)

Roberts et. al. *J. Membr. Sci.* **2002**, *208*, 75.
Leterrier, Y. *Prog. Mater. Sci.* **2003**, *48*, 1.



- <u>Bulk Composite</u>
  - Poor transparency
  - Relatively low barrier

Nazarenko et. al. *J. Polym. Sci. B: Polym. Phys.* **2007**, *45*, 1733.



Kirwan, M., *Food and Beverage Packaging Technology*, Wiley-Blackwell, **2011**, 157.

Polyelectrolyte complex coatings combines the best features of these systems without the drawbacks.

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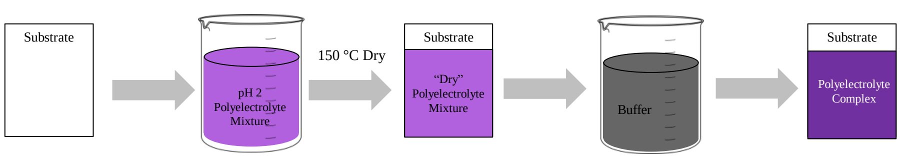
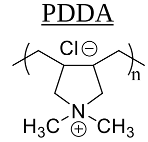
PEC Gas Barrier Deposition

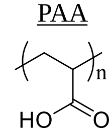


- PDDA mixed with PAA in a 1:3 mol:mol ratio.
  - 1.5, 3, 4.5, and 6 wt% in water
  - PDDA 400-500 kg/mol
  - PAA 250 kg/mol
  - pH 2
- Citric Acid (curing buffer) pH 3
  - 25, 100, 200, 300 mM
  - NaCl added to 25, 100, and 200 mM solutions to keep ionic strength ~150mM.
- Poly(ethylene terephthalate) (PET)
  - 178 µm thick
  - Purchased from Tekra.

<u>PDDA</u>



<u>PAA</u>



Smith, R. J.; Grunlan, J. C. *et al*, *Langmuir* **2018**, 34, 11086.

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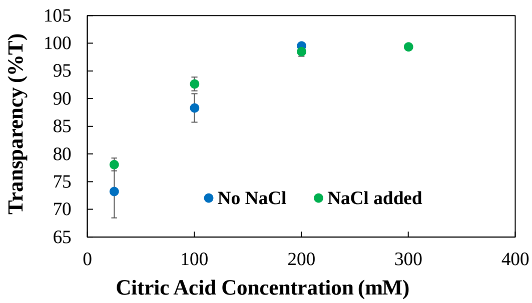
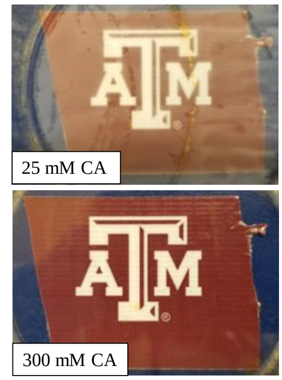
PEC Transparency





Roughness (R_q)	25 mM CA [nm]	100 mM CA [nm]	200 mM CA [nm]	300 mM CA [nm]
No NaCl	55.7 ± 3.0	23.9 ± 1.3	7.4 ± 0.7	3.3 ± 1.4
With NaCl	29.0 ± 5.2	11.5 ± 0.4	4.5 ± 0.2	N/A

Transparency of PEC thin films increases with buffer concentration and added salt due to decreasing surface roughness.

Smith, R. J.; Grunlan, J. C. *et al, Langmuir* **2018**, 34, 11086.

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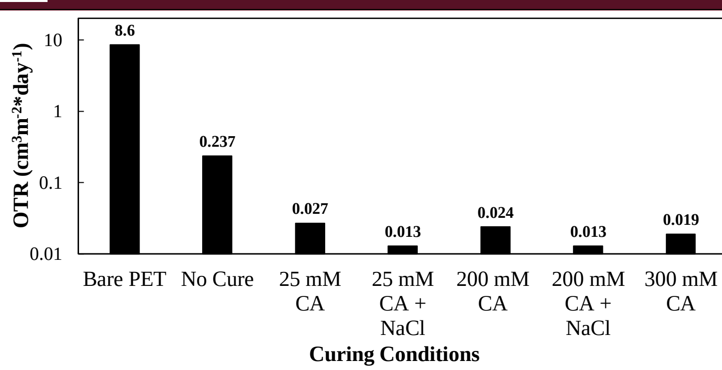
PEC Gas Barrier



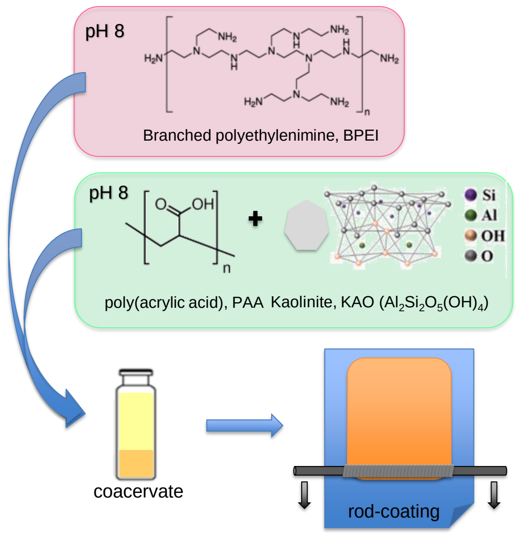
Reduced Modulus	No Cure [GPa]	25 mM CA [GPa]	100 mM CA [GPa]	200 mM CA [GPa]	300 mM CA [GPa]
No NaCl	6.95 ± 0.11	12.53 ± 3.69	13.01 ± 1.55	12.43 ± 0.96	11.37 ± 0.35
With NaCl	N/A	12.00 ± 2.11	12.86 ± 1.21	11.98 ± 0.61	N/A

- Uncured coatings reduce the OTR by a factor of 36.
- Curing the films leads to another order of magnitude reduction.
- Curing solutions with salt improves barrier (~2x) with an overall ~660x reduction in OTR.

Smith, R. J.; Grunlan, J. C. *et al, Langmuir* **2018**, 34, 11086.

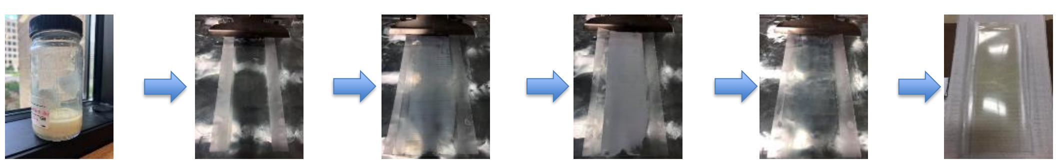
Clay-Filled Complex Gas Barrier

- **Branched Polyethylenimine (BPEI)**
  (Purchased from Sigma–Aldrich)
  Mw = 25,000 g/mol
  Positively charged in water
- **Poly(acrylic acid) (PAA)**
  (Purchased from Sigma–Aldrich)
  Mw = 250,000 g/mol
  Negatively charged in water
- **Kaolinite clay (KAO)**
  (Purchased from Sigma–Aldrich)
  Negatively charged in pH8
  Stabilized in PAA solution



Chiang, H.-C.; Grunlan, J. C.; et al. *Macromol. Rapid Comm.* **2021**, *42*, 2000540.

Clay Complex Coating



coacervate, annealed in 70°C for 2hr → 7mil PET, corona-treated → rod-coated → cured in pH4 200 mM CA buffer for 5min → humidity post-treatment → dried in 150 °C for 2h

	Total solid	KAO content	PAA_8	$BPEI_8$
Film A	10 wt%	0.1 wt%	4.95 wt%	4.95 wt%
Film B	10 wt%	0.5 wt%	4.75 wt%	4.75 wt%
Film C	10 wt%	1.0 wt%	4.5 wt%	4.5 wt%
Film D	10 wt%	2.0 wt%	4.0 wt%	4.0 wt%
Film E	10 wt%	4.0 wt%	3.0 wt%	3.0 wt%

Chiang, H.-C.; Grunlan, J. C.; et al. *Macromol. Rapid Comm.* **2021**, *42*, 2000540.

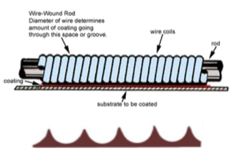
Rheology for Rod Coating PEC

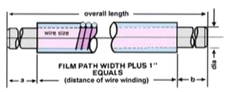
Rheometry
(40 mm cone plate, PEI_8 + PAA/KAO_8, 1 wt% clay)

NaCl conc. in mixture	η at 10^{-2} 1/s	η at 10^{3} 1/s
0.50 M	15262 cP	5624 cP
0.75 M	5377 cP	2778 cP
1.00 M	1684 cP	1088 cP
1.50 M	49 cP	23 cP

Ideal range of viscosity for Meyer-rod technique is around 1000 cP.

Film Thickness





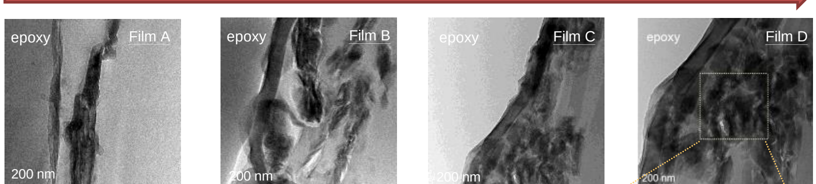
Gardco.com

Rod No.	Wire Dia. (Inches)	Wet Film Thickness (μm)	Thickness (μm)
2.5	.0025	6.4	1.74 ± 0.37
3	.003	7.7	2.12 ± 0.01
5	.005	12.8	2.85 ± 0.04
10	.010	25.6	3.78 ± 0.62

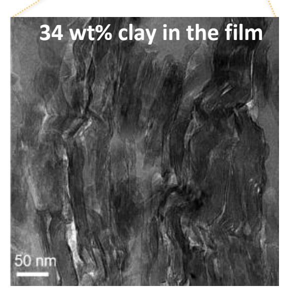
Chiang, H.-C.; Grunlan, J. C.; et al. *Macromol. Rapid Comm.* **2021**, *42*, 2000540.

TEM Imaging of KAO Coatings

Increasing clay conc.



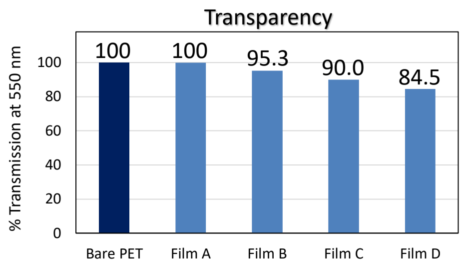
0.1 wt% clay in PEC | 0.5 wt% clay in PEC | 1.0 wt% clay in PEC | 2.0 wt% clay in PEC

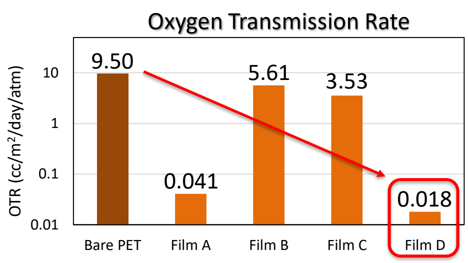


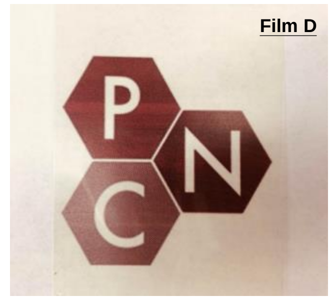
Cross-sectional TEM shows clay alignment.

Chiang, H.-C.; Grunlan, J. C.; et al. *Macromol. Rapid Comm.* **2021**, *42*, 2000540.

Transparency and Oxygen Barrier







34 wt% clay in the film

OTR is reduced by two orders of magnitude.

Chiang, H.-C.; Grunlan, J. C.; et al. *Macromol. Rapid Comm.* **2021**, *42*, 2000540.

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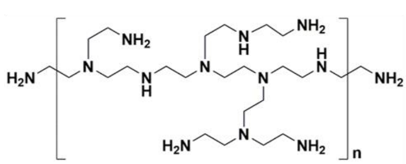
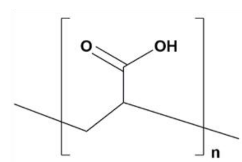
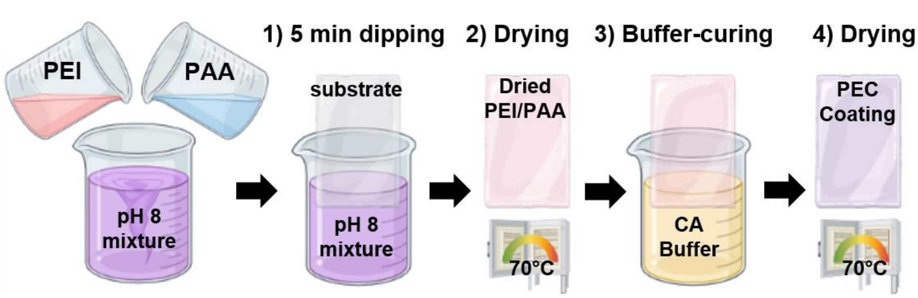
Moisture Resistant PEC Gas Barrier Coating



Branched Polyethyleneimine (PEI)



poly(acrylic acid) (PAA)



Chiang, H.-C.; Grunlan, J. C.; et al. *J. Appl. Polym. Sci.* **2023**, *140*, e53473.

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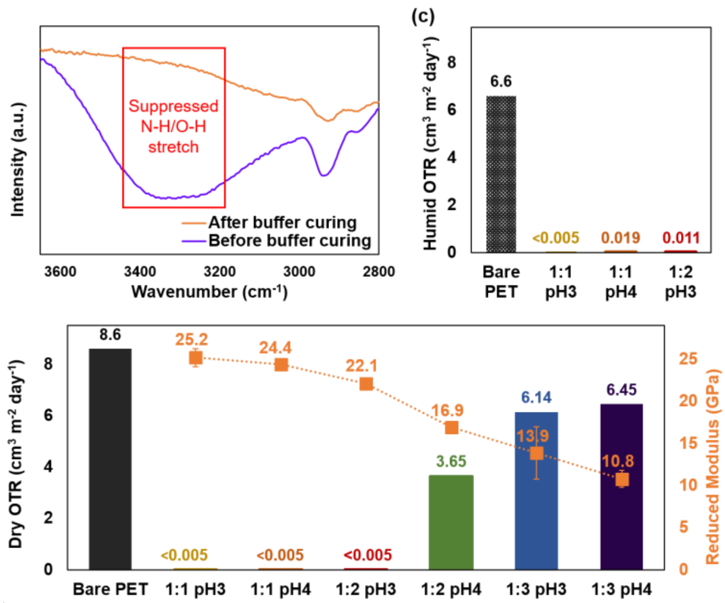
Correlation Between Density and Gas Barrier



Chiang, H.-C.; Grunlan, J. C.; et al. *J. Appl. Polym. Sci.* **2023**, *140*, e53473.

J. C. Grunlan, M. Haile, R. Smith, "Method for applying gas-impermeable coatings," **U.S. Patent 11,518,903** (issued December 6, 2022)

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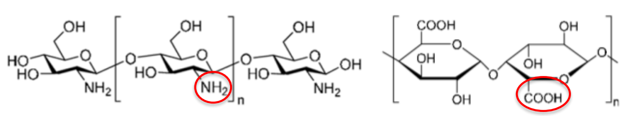
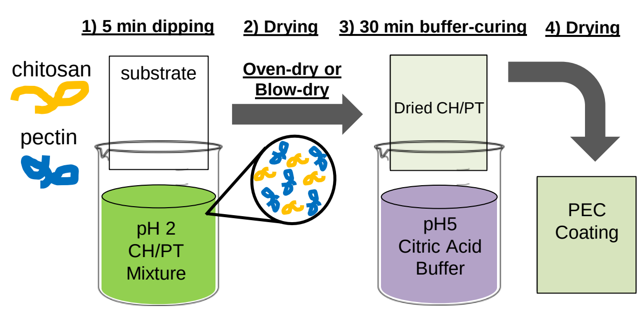
Edible Gas Barrier



Chitosan (CH) pKa ~6.5 **Pectin (PT)** pKa 2.8~4.1



Chiang, H.-C.; Grunlan, J. C.; et al. *ACS Food Sci. Technol.* **2021**, *1*, 495.

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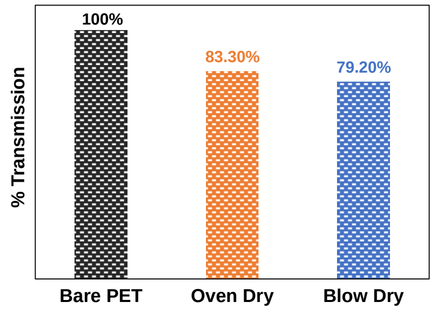
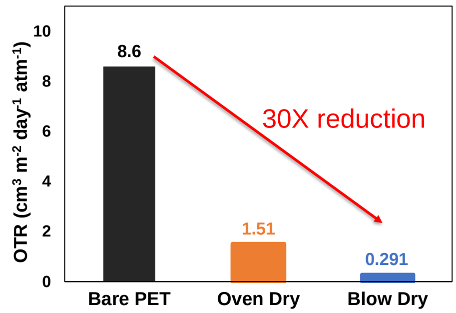
Transparency and Oxygen Barrier





- The as-deposited films has good optical transparency.
- The better gas barrier performance of blow-dried film is likely due to the thicker deposition.

Chiang, H.-C.; Grunlan, J. C.; et al. *ACS Food Sci. Technol.* **2021**, *1*, 495.

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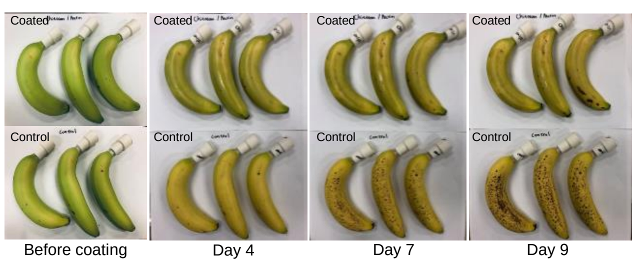
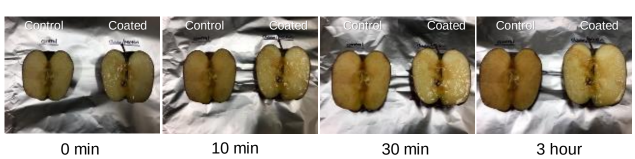
Preservation of Fresh Fruit





CH/PT edible coating can slow down aging and browning of fruits.

Chiang, H.-C.; Grunlan, J. C.; et al. *ACS Food Sci. Technol.* **2021**, *1*, 495.

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Other Properties from Polyelectrolyte Complexes…

Anti-Corrosion

ACS Appl. Nano Mater. **2018**, *1*, 5516 [**nanobrick wall protection of aluminum**].
ACS AMI **2018**, *10*, 21799 [**nanobrick wall protection of copper**].

Thermoelectric (Body Heat → Voltage)

Advanced Materials **2018**, *30*, 1704386 [**> 1000 S/cm and PF > 2000 μW/m·K^2**].

Antimicrobial / Antifouling

ACS Biomater. Sci. Eng. **2017**, *3*, 1845 [**prevention of bacterial adhesion to polyester**].
Langmuir **2009**, *25*, 10322 [**tailoring efficacy of quaternary ammonium-based system**].

UV-Resistance

RSC Advances **2020**, *24*, 8314 [**chitosan and lignin protection**].
ACS Macro Letters **2015**, *4*, 335 [**melanin-based multilayer nanocoatings**].

High Dielectric Breakdown Strength

Macromolecules **2022**, *in press* [**multilayer nanocoatings with high breakdown strength**].

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Conclusions

- **Polyelectrolyte complexes (PEC) provide an opportunity to quickly deposit functional films**
- **Effective flame retardant and gas barrier coatings deposited in 1 or 2 steps from water-based solution**

Polymer NanoComposites (PNC) Lab (http://nanocomposites.tamu.edu)

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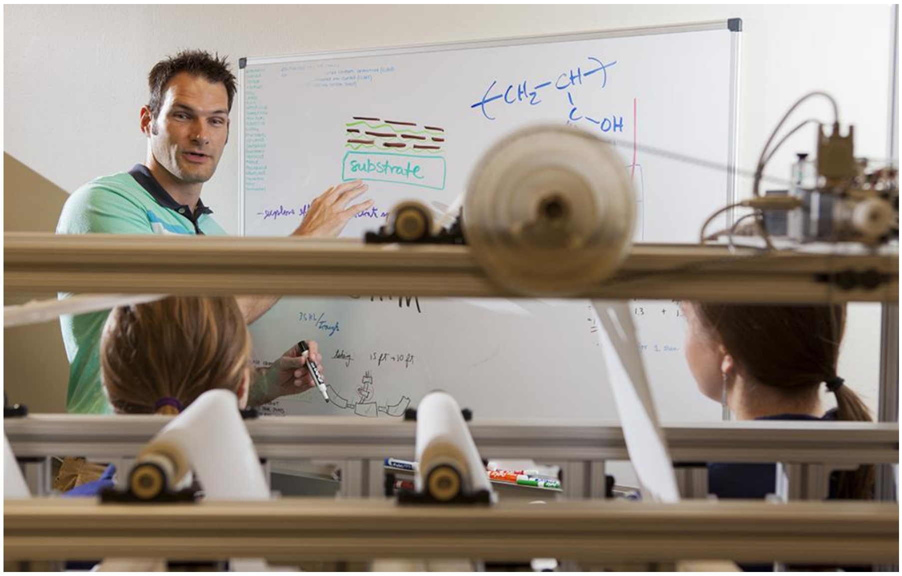
QUESTIONS???



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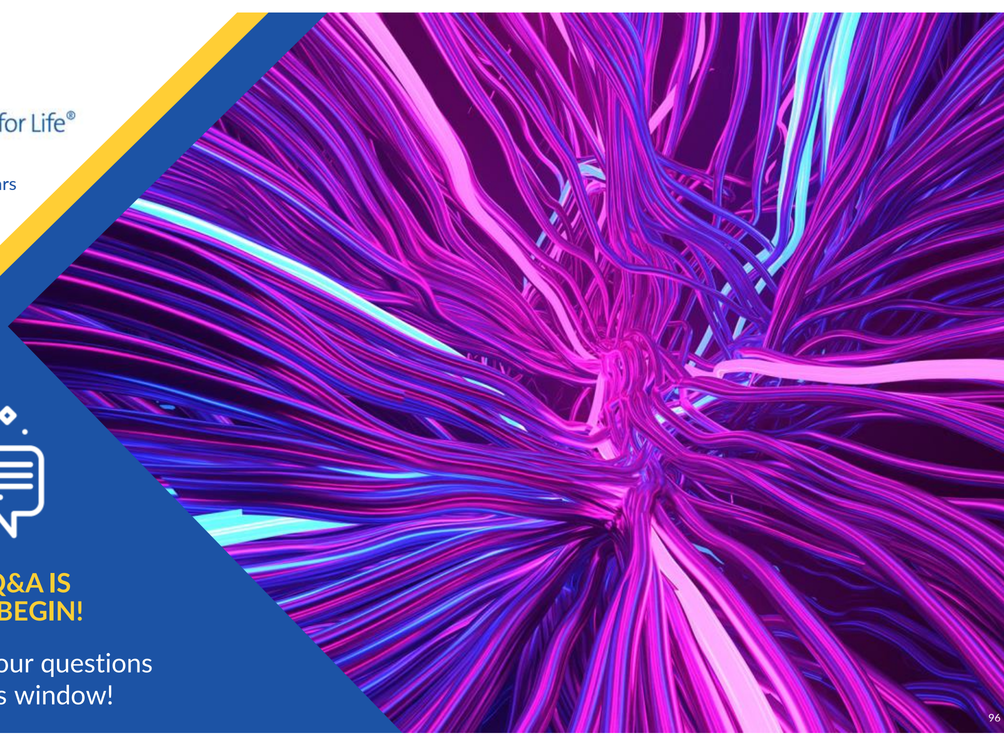

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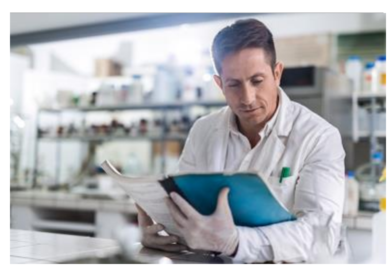

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