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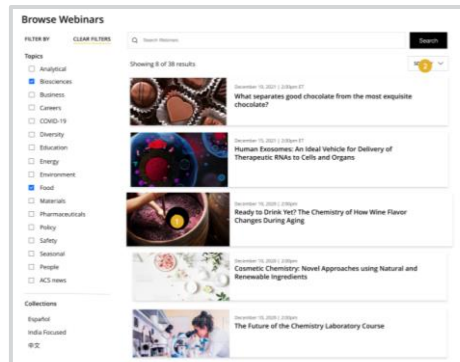


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https://chemidp.acs.org

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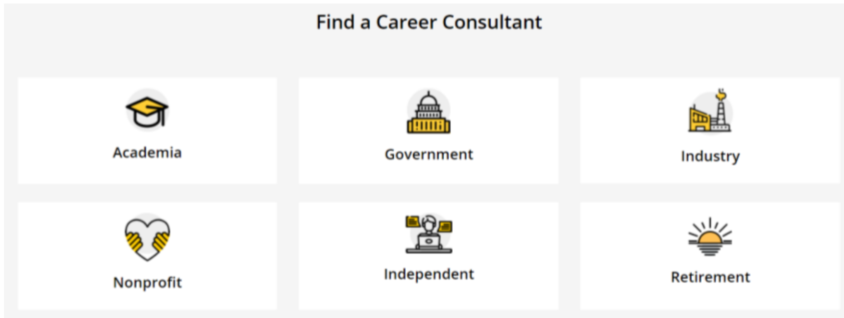
## Career Consultant Directory




- ACS Member-exclusive program that allows you to arrange a one-on-one appointment with a certified ACS Career Consultant.
- Consultants provide personalized career advice to ACS Members.
- Browse our Career Consultant roster and request your one-on-one appointment today!

www.acs.org/careerconsulting

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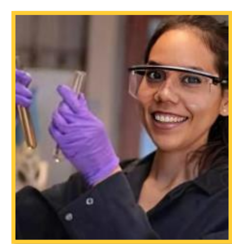

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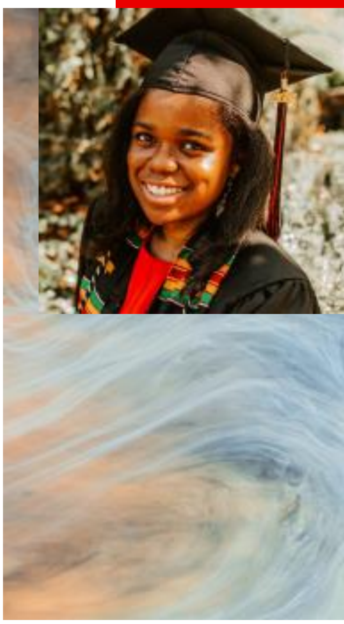

## ACS Scholar Adunoluwa Obisesan

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

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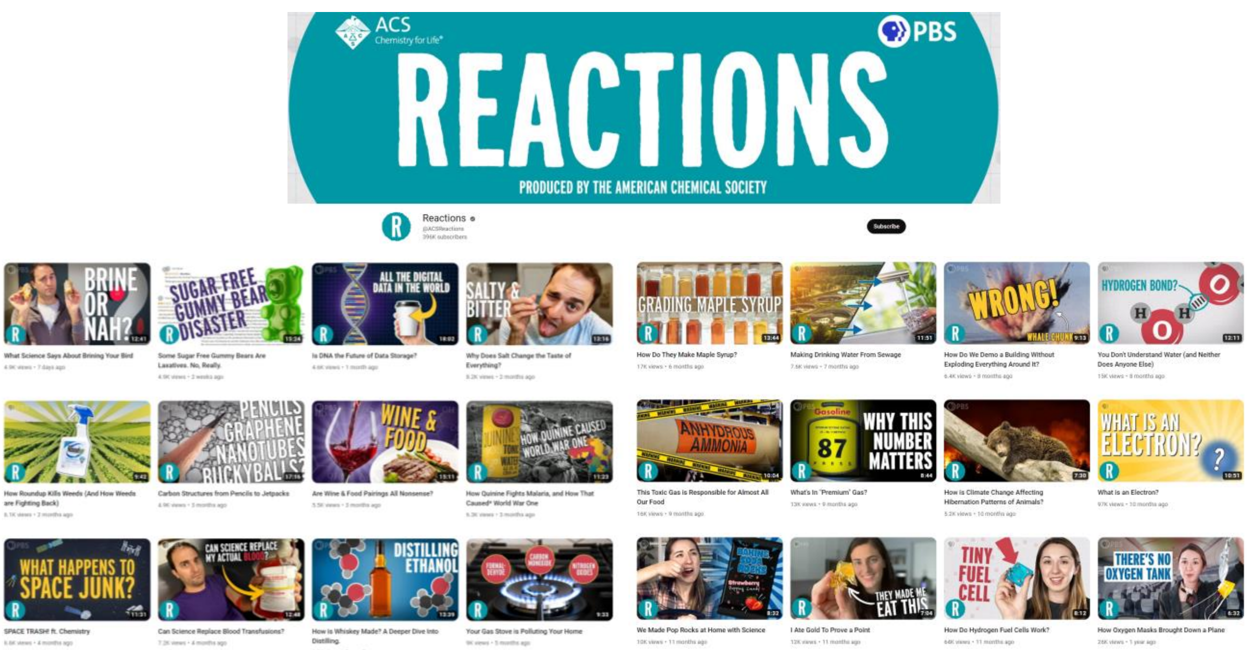


https://www.youtube.com/c/ACSReactions/videos

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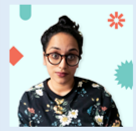

Deboki Chakravarti, PhD
*Science Writer & Co-Host*

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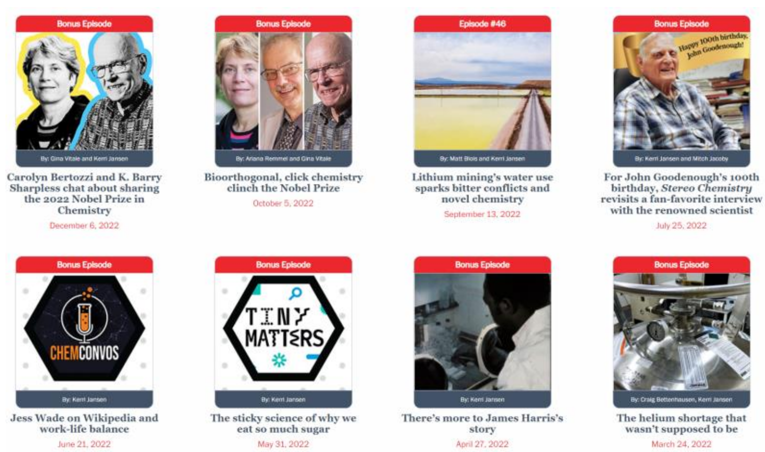


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**Preview Content:** **acs.org/indnl**

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ACS ON CAMPUS


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# ACS Career Resources

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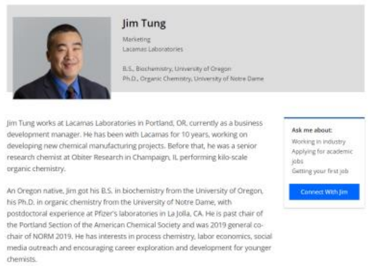
https://www.acs.org/careerconsulting.html

## Personal Career Consultations




https://www.acs.org/careerconsulting.html

## LinkedIn Learning


https://www.acs.org/linkedInlearning

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| | | | |
|---|---|---|---|
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| Get Involved | Enroll in a workshop | Become a Fellow | Take Action |

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ACS Technical Division
Chemical Health & Safety (CHAS)

ACS Committee
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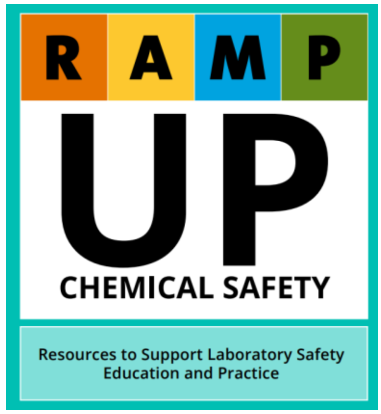


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# ACS OFFICE OF DEIR

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

## Resources

**Inclusivity Style Guide**
Designed to help staff and members use language and images that respect diversity in all its forms.

**ACS Webinars on Diversity**
Covering diversity and inclusion at the workplace

**ACS Publications DEIR Hub**
See what ACS Publications is doing for fostering inclusivity in scholarly publishing

**ACS Volunteer and ACS Meetings Code of Conduct**
Fostering a positive and welcoming environment for attendees, volunteers and staff.

**C&EN Trailblazers**
C&EN highlights scientists from different backgrounds who are making an impact in chemistry.

**NEW! Download DEIR Educational Resources**
Download this educational guide for additional recommendations on videos, articles, books, podcasts, and more on diversity, inclusion, and related topics.

**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****
The representation of varied identities and differences (race, ethnicity, gender, disability, sexual orientation, gender identity, national origin, tribe, caste, socio-economic status, thinking and communication styles, etc.), collectively and as individuals. ACS seeks to proactively engage, understand, and draw on a variety of perspectives.

**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**
Ensures that each person is treated with professionalism, integrity, and ethics underpinning all interpersonal interactions.

https://www.acs.org/diversity

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https://pubs.acs.org

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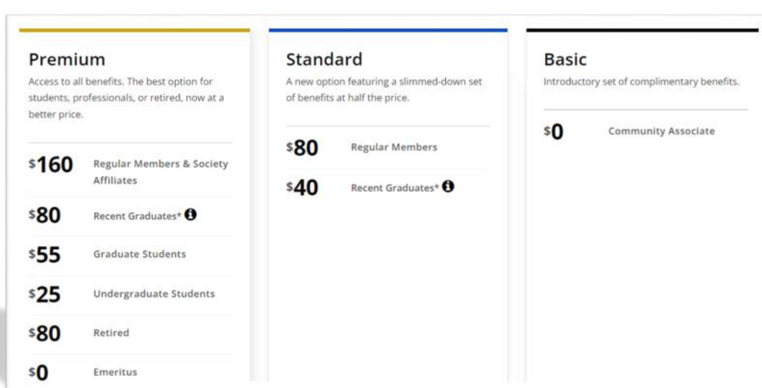


**Have a Different Question?**
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- ✓ Networking and professional development events at local/national ACS meetings and local POLY/PMSE chapters.
- ✓ Industrial scientist support and networking through IAB (Industrial Advisory Board)
- ✓ Polymer science-related conferences and workshops advertised through the POLY list serve
- ✓ Online educational webinar and webshop series covering cutting-edge polymer research
- ✓ Opportunity to vote for the executive committee (annually)
- ✓ Recognition for membership (5th, 10th, 20th, and 30th anniversaries)
- ✓ Student support – student awards, student symposia, career panels at ACS meetings, support for student chapters.
- ✓ An excellent support group for building strong networks in the polymer community!

TM

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https://polyacs.org 22

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**How Nanoscale Materials in Biosensors are Innovating Health from Concept to Care**

Co-produced with CAS, a division of the ACS


Thursday, May 9, 2024| 2pm-3pm ET

**Tools to Make Chemistry Education Accessible for Persons with Visual Impairments**

Co-produced with ACS Division of Professional Relations


Wednesday, May 15, 2024| 2pm-3pm ET

**Your Career Story: Crafting CVs and Resumes**

Co-produced with ACS Careers

**Register for Free**

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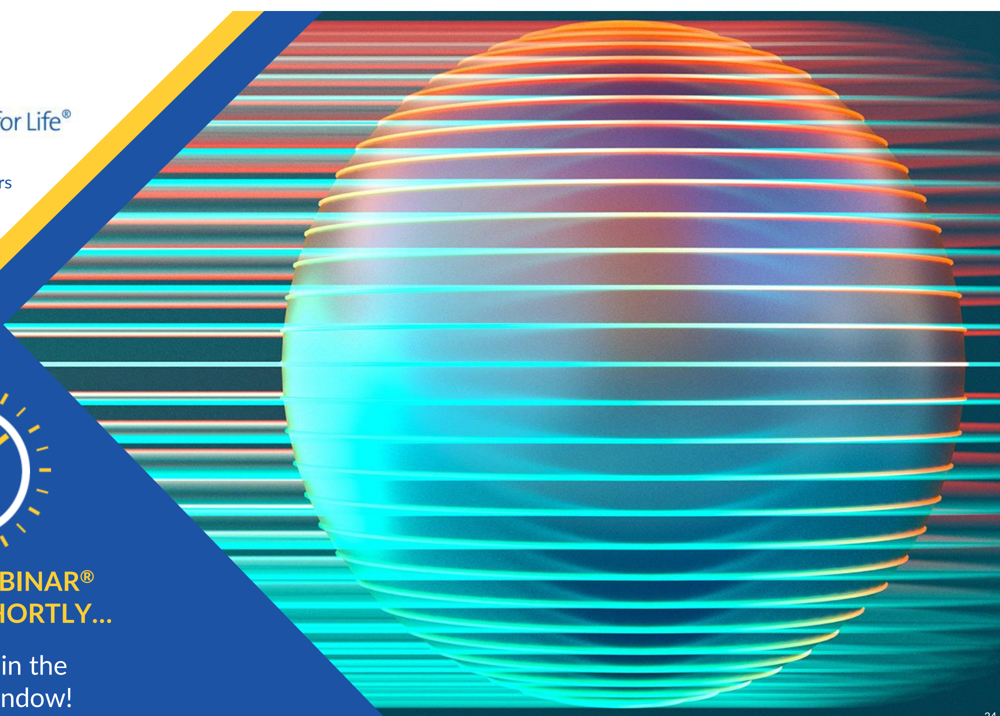

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## Better Biodegradable Vinyl Polymer Materials
by Improving Radical Ring-Opening Polymerization (rROP)


**JULIEN NICOLAS, PhD**

Director of Research at CNRS, Institut Galien Paris-Saclay, University Paris-Saclay/CNRS & Associate Editor, *Chemistry of Materials*



**JIA NIU, PhD**

Associate Professor of Chemistry, Boston College



**JOSH WORCH, PhD**

Assistant Professor of Chemistry, Virginia Tech


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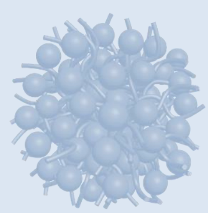
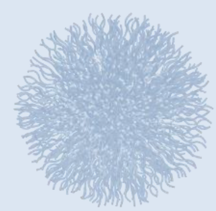
# DESIGN OF (BIO)DEGRADABLE MATERIALS BY RADICAL-RING OPENING COPOLYMERIZATION





May 2nd, 2024


**Julien NICOLAS**

Université Paris-Saclay, CNRS
Institut Galien Paris-Saclay
Orsay, France

CM CHEMISTRY OF MATERIALS *Associate Editor*

julien.nicolas@universite-paris-saclay.fr
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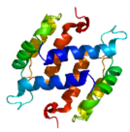
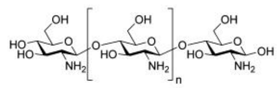
# (BIO)DEGRADABLE POLYMERS

## Natural and synthetic (bio)degradable polymers

### Natural polymers



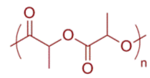


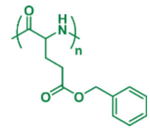
**Polyaminoacids** (peptides, proteins)

**Polysaccharides** (dextran, chitosan, etc.)

- ✓ Biodegradable, biocompatible
- ✓ Fragile and expensive
- ✓ No easy functionalization
- ✓ Particles from preformed polymers

### Synthetic polymers





**Polyesters** (PLA, PLGA, PCL, etc.)

**Polypeptides** (NCAs: PBLG, PBLA, etc.)

- ✓ Biodegradable, biocompatible (FDA-approved)
- ✓ No easy access to well-defined architectures
- ✓ No easy functionalization
- ✓ Particles from preformed polymers

**Vinyl polymers**

- ✓ Easy synthesis
- ✓ Well-defined architectures
- ✓ Easy functionalization
- ✓ In situ synthesis of particles
- ✓ Not biodegradable

- **Traditional biodegradable polymers**: cumbersome synthesis and functionalization, formulation of preformed polymers
- **Vinyl polymers**: easy synthesis and functionalization but not (bio)degradable

Sung, Y.K., Kim, S.W. *Biomater Res.* **2020**, *24*, 12

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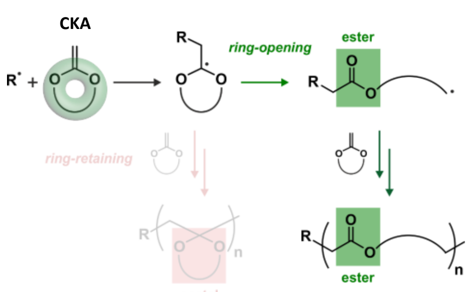
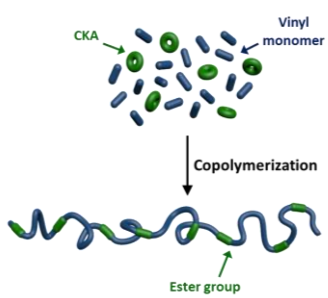
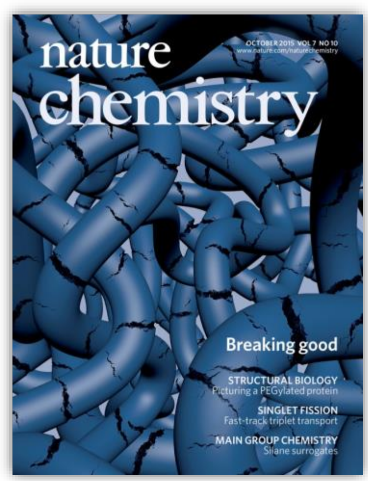
# IMPART DEGRADABILITY TO VINYL POLYMERS

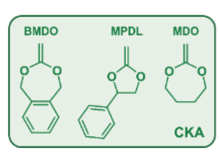
## Radical ring-opening polymerization (rROP)




- Ester bond precursor → labile group in the main chain
- Based on a radical polymerization mechanism
- CKA can be copolymerized with certain vinyl monomers
- Compatible with RDRP techniques
- Three main CKAs are used




*Agarwal, Albertson, Matyjaszewski, Hawker, Dove, O'Reilly, Guillaneuf, Niu, Jackson, Maynard, Kikuchi, Thoniyot, Roth, Harrisson, Destarac, Johnson, Sumerlin, Gaitzsch, Bates, Reineke, Paulusse, Gutekunst, D'Hooge, Tsarevsky, Frisch, Carter, Miyake…*

Pesenti, T.; Nicolas, J. *ACS Macro Letters* **2020**, *9*, 1812
Delplace, V.; Nicolas, J. *Nature Chem.* **2015**, *7*, 771
Tardy, A.; Nicolas, J.; Gigmes, D.; Lefay, C.; Guillaneuf, Y. *Chem. Rev.* **2017**, *117*, 1319

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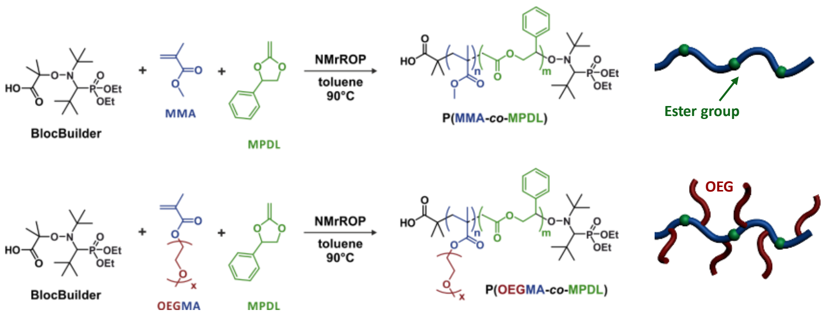
# DEGRADABLE POLYMETHACRYLATES

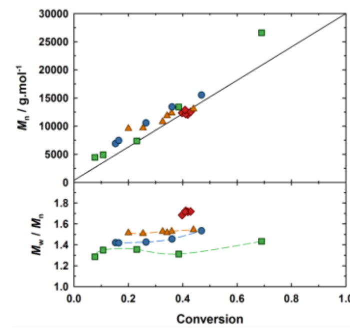
## Synthesis of hydrophobic and hydrophilic copolymers




mol %

| $f_{MPDL,0}$ | $F_{MPDL}$ |
|---|---|
| 0 | 0 |
| 20 | 4 |
| 40 | 11 |
| 70 | 25 |

$r_{MPDL}$ = 0.03 ; $r_{OEGMA}$ = 7




- NMP of MPDL with MMA (or OEGMA) with the SG1-based alkoxyamine
- Good control: linear evolution of $M_n$ vs. conv., $Đ$ ~1.3–1.4
- Tuneable insertion of ester groups (open MPDL, $F_{MPDL}$ = 4–25 mol %)

Delplace, V.; Guégain, E.; Harrisson, S.; Gigmes, D.; Guillaneuf, Y.; Nicolas, J. *Chem. Commun.* **2015**, *51*, 12847
Tran, J.; Guégain, E.; Ibrahim, N.; Harrisson, S.; Nicolas, J. *Polym. Chem.* **2016**, *7*, 4427

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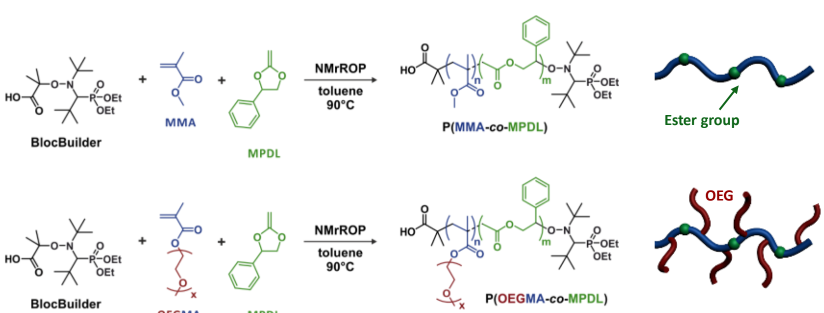
# DEGRADABLE POLYMETHACRYLATES

## Synthesis of hydrophobic and hydrophilic copolymers




mol %

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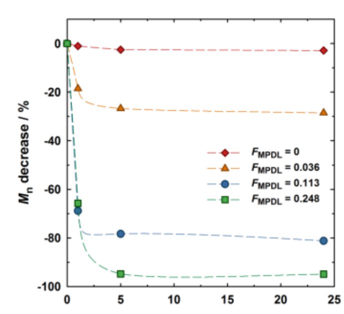


- NMP of MPDL with MMA (or OEGMA) with the SG1-based alkoxyamine
- Good control: linear evolution of $M_n$ vs. conv., $Đ$ ~1.3–1.4
- Tuneable insertion of ester groups (open MPDL, $F_{MPDL}$ = 4–25 mol %)
- Fast and up to complete hydrolysis under accelerated conditions (KOH 5 %)

Delplace, V.; Guégain, E.; Harrisson, S.; Gigmes, D.; Guillaneuf, Y.; Nicolas, J. *Chem. Commun.* **2015**, *51*, 12847
Tran, J.; Guégain, E.; Ibrahim, N.; Harrisson, S.; Nicolas, J. *Polym. Chem.* **2016**, *7*, 4427

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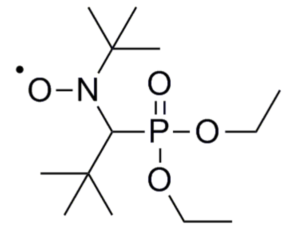
**One of the most potent nitroxides ever developed for NMP is called SG1.**

**But where does the name SG1 come from?**




1. **Super Great nitroxide #1**
2. **Stargate SG-1**
3. **Sandra Grimaldi**
4. ***N-tert*-butyl-*N*-[1-diethylphosphono-(2,2-dimethylpropyl)]**

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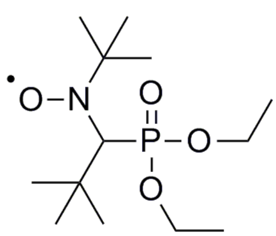
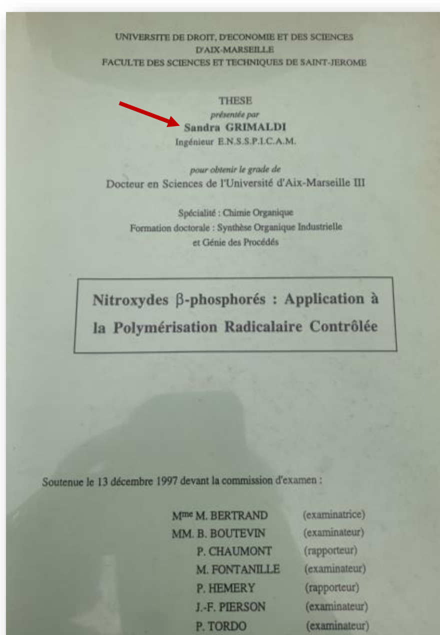



**SG1**

*N-tert*-butyl-*N*-[1-diethylphosphono-(2,2-dimethylpropyl)]



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D'AIX-MARSEILLE
FACULTE DES SCIENCES ET TECHNIQUES DE SAINT-JEROME

THESE
*présentée par*
**Sandra GRIMALDI**
Ingénieur E.N.S.S.P.I.C.A.M.

*pour obtenir le grade de*
Docteur en Sciences de l'Université d'Aix-Marseille III

Spécialité : Chimie Organique
Formation doctorale : Synthèse Organique Industrielle
et Génie des Procédés

**Nitroxydes β-phosphorés : Application à la Polymérisation Radicalaire Contrôlée**

Soutenue le 13 décembre 1997 devant la commission d'examen :

| | |
|---|---|
| Mme M. BERTRAND | (examinatrice) |
| MM. B. BOUTEVIN | (examinateur) |
| P. CHAUMONT | (rapporteur) |
| M. FONTANILLE | (examinateur) |
| P. HEMERY | (rapporteur) |
| J.-F. PIERSON | (examinateur) |
| P. TORDO | (examinateur) |

Photo courtesy of Yohann Guillaneuf

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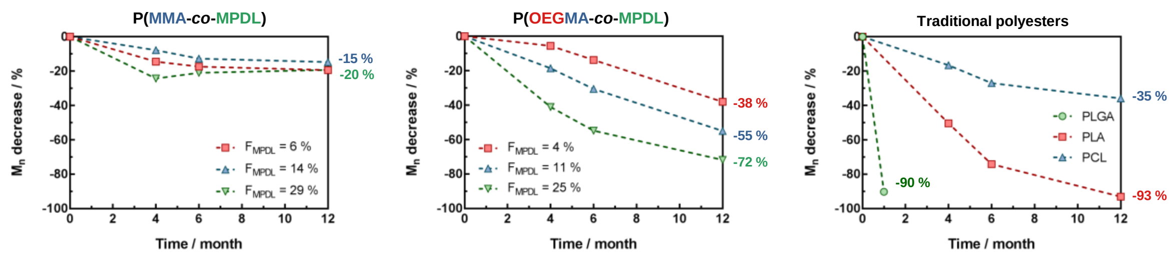
## DEGRADABLE POLYMETHACRYLATES

Degradation under physiological conditions

37 °C, PBS, pH 7.4




- Copolymers hydrolytically degrade in physiological conditions
- Faster and tunable hydrolytic degradation with hydrophilic copolymers
- Degradation of P(MMA-*co*-MPDL) ~ **PCL**
- Degradation of **PCL** ≤ P(OEGMA-*co*-MPDL) ≤ **PLA**

Guégain, E.; Michel, J.-P.; Boissenot, T.; Nicolas, J. *Macromolecules* **2018**, *51*, 724

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## LIMITATIONS OF rROP WITH CKAs

- **Unfavorable reactivity ratios** → **Can we increase CKA contents?**
  - $r_{CKA}$ ~ 0; $r_{(meth)acrylate}$ ~ 4-10
- **Slow hydrolytic degradations of CKA-containing copolymers** → **Can we improve the degradation?**
  - ~Several months in water/PBS
  - Like PCL or PLA
- **Hydrophobic CKAs** → **Can we use more hydrophilic CKAs?**
  - MDO, BMDO, MPDL
  - New CKAs are very difficult to synthesize
- **Poor hydrolytic stability of CKAs** → **How to obtain aqueous suspensions of degradable particles?**
  - Polymerization in aqueous dispersed media (emulsion, PISA) is challenging
- **Rather limited range of degradable vinyl polymers** → **Can we extend the range of vinyl monomers?**
  - Mainly (meth)acrylates, styrenics and vinyl acetates

Pesenti, T.; Nicolas, J. *ACS Macro Letters* **2020**, *9*, 1812

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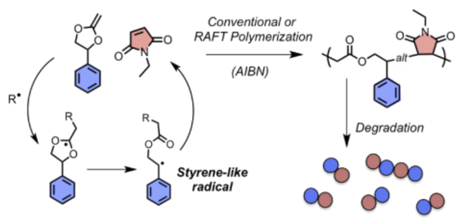
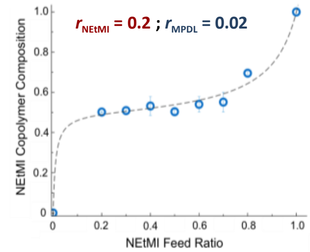
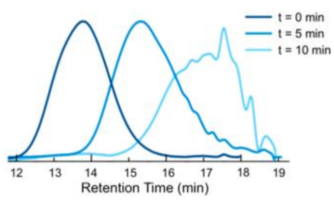
6

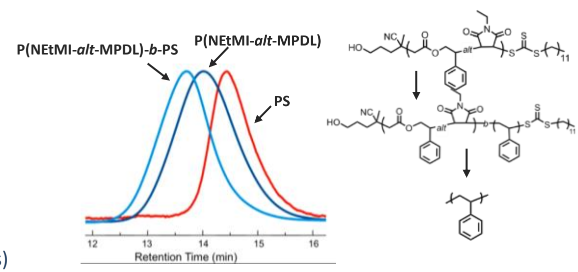
# INCREASING THE CKA CONTENT

## Copolymerization between CKA and maleimides (MI)




- **Alternating** P(NEtMI-*alt*-MPDL) copolymers → $F_{CKA}$ **~0.5**
- Conventional and RAFT polymerization (block copolymers)

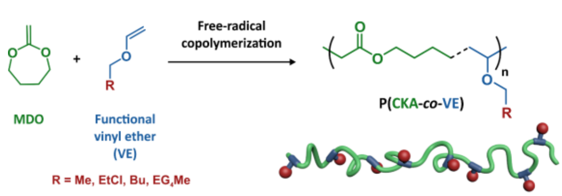
Hill, M. R.; Guégain, E.; Tran, J.; Figg, C. A.; Turner, A. C.; Nicolas, J., Sumerlin, B. S. *ACS Macro Lett.* **2017**, *6*, 1071

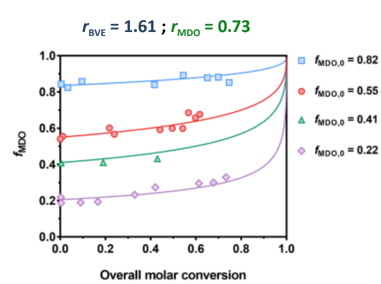
35

7

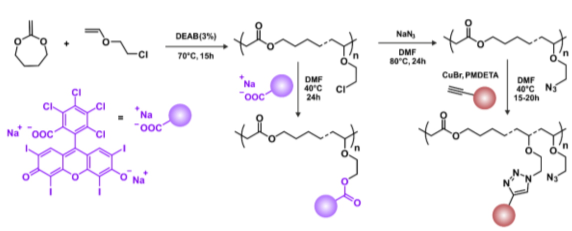
# INCREASING THE CKA CONTENT

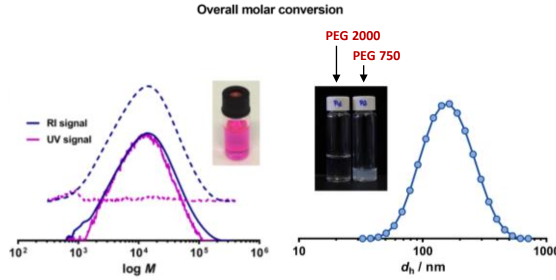
## Copolymerization between CKA and vinyl ethers (VE)




- Nearly ideal copolymerization → $F_{CKA}$ **~0.9** (PCL-like copolymers)




- Facile functionalization from functional VE derivatives

Tardy, A.; Honoré, J.-C.; Tran, J.; Siri, D.; Delplace, V.; Bataille, I.; Letourneur, D.; […] Lefay, C.; Gigmes, D.; Nicolas, J.; Guillaneuf, Y. *Angew. Chem., Int. Ed.* **2017**, *56*, 16515

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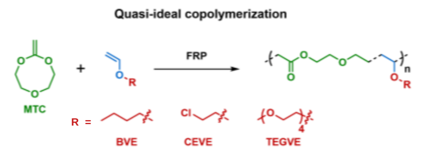
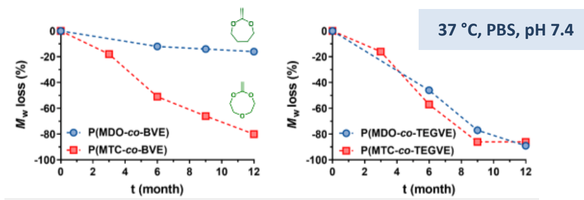
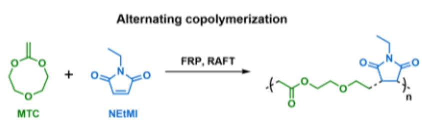
# IMPROVING THE HYDROLYTIC DEGRADATION

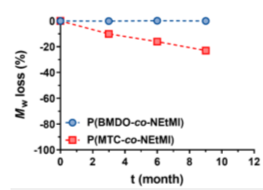
## Using more hydrophilic CKAs




- Free-radical copolymerization between VE derivatives and MTC
- $F_{MTC}$ ~90 mol % (polyester-like copolymers)
- Faster hydrolytic degradations under physiological conditions (PBS, pH 7.4, 37°C for hydrophobic copolymers)




37 °C, PBS, pH 7.4

- Alternating MTC/NEtMI copolymers
- Faster hydrolytic degradations under physiological conditions

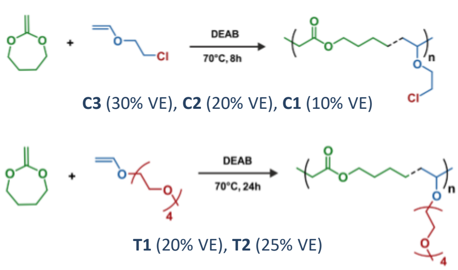
Pesenti, T.; Gillon, E.; Ishii, S.; Messaoudi, S.; Guillaneuf, Y.; Imberty, A.; Nicolas, J. *Biomacromolecules* **2023**, *24*, 991

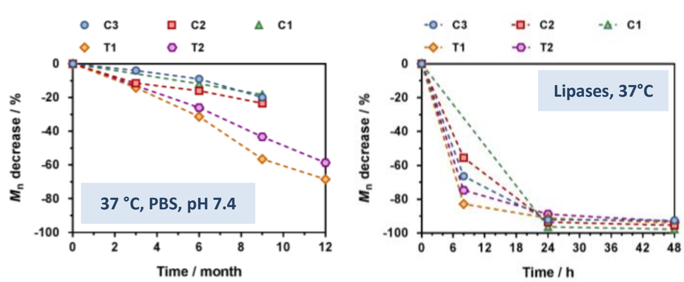


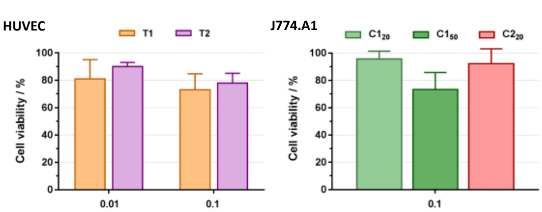


# IMPROVING THE HYDROLYTIC DEGRADATION

## Using more hydrophilic vinyl monomers




- Enhanced hydrolytic degradation with TEGVE
- Significant enzymatic degradation
- Hydrolytic degradation similar to PCL (**C1-C3**) and PLA (**T1-T2**)
- No acute cytotoxicity on 2 healthy cell lines

Tran, J.; Pesenti, T.; Cressonier, J.; Lefay, C.; Gigmes, D.; Guillaneuf, Y.; Nicolas, J. *Biomacromolecules* **2019**, *20*, 305

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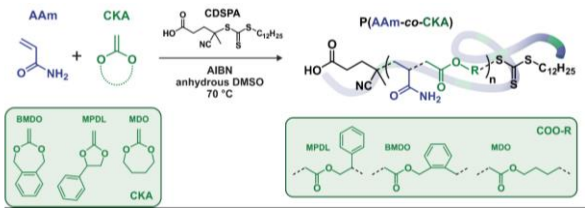
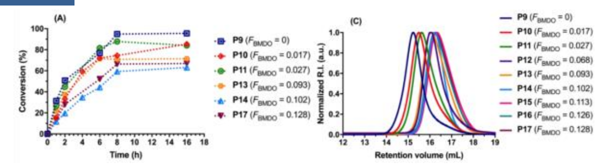
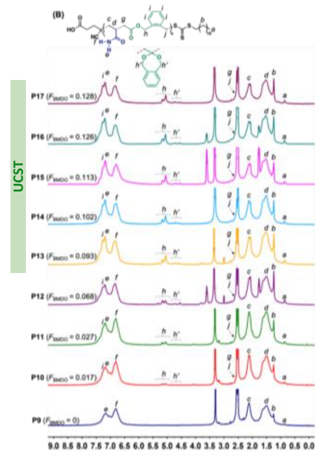
# IMPROVING THE HYDROLYTIC DEGRADATION

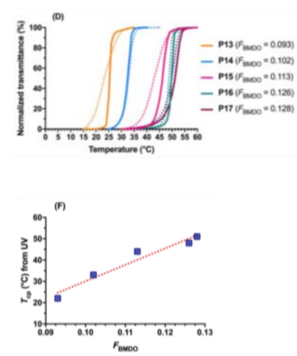
## Using more hydrophilic vinyl monomers




$M_n$ = 6.1–13.5 kg/mol; $Đ$ = 1.2–1.4




- Analogy with P(AAm-*co*-Sty) → non-degradable UCST copolymer
- Successful RAFT-mediated synthesis with MPDL/BMDO
- High conversions, low dispersities, tunable $F_{MPDL/BMDO}$
- **Sharp and tunable UCST transitions** ($T_{cp, BMDO}$ = 23–55°C)
- No thermosensitivity with P(AAm-*co*-MDO) copolymers
- **Hydrolytic degradation** in PBS (up to -75% $M_n$)
- Faster hydrolytic degradation than PLA and even PLGA!

Bossion, A.; Zhu, C.; Guerassimoff, L.; Mougin, J.; Nicolas, J. *Nature Commun.* **2022**, *13*, 2873

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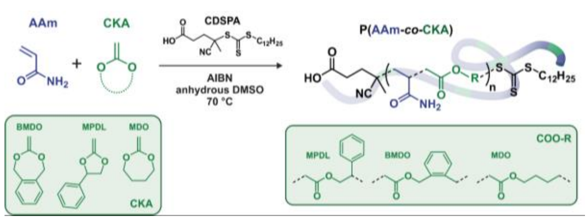
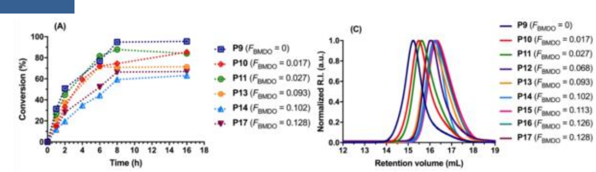
# IMPROVING THE HYDROLYTIC DEGRADATION

## Using more hydrophilic vinyl monomers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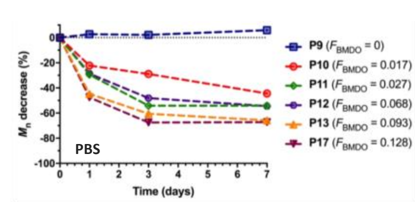


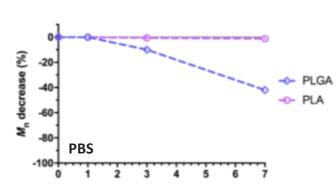



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Bossion, A.; Zhu, C.; Guerassimoff, L.; Mougin, J.; Nicolas, J. *Nature Commun.* **2022**, *13*, 2873


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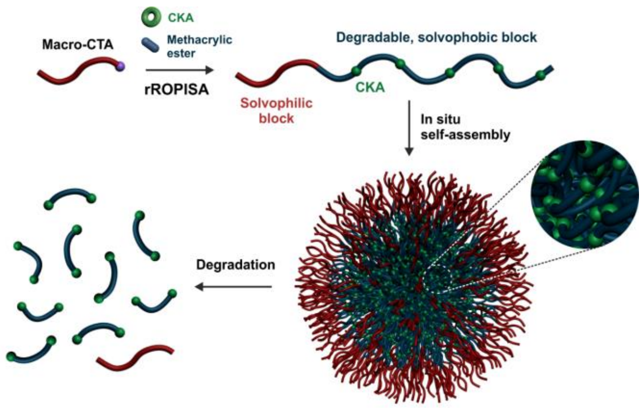
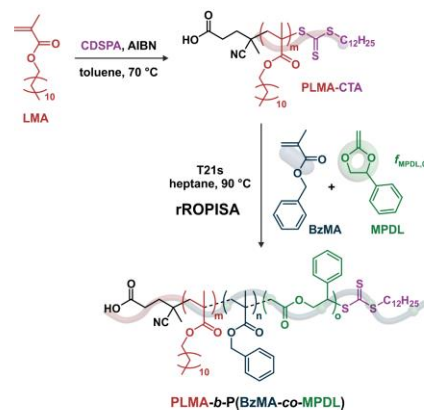
# DEGRADABLE PARTICLES IN SITU

## Radical ring-opening polymerization-induced self-assembly (rROPISA)




- Synthesis of core-degradable nanoparticles
- **rROPISA in heptane** to prevent early degradation of CKAs
- PLMA as the solvophilic block and P(BzMA-*co*-CKA) as the solvophobic one
- PISA in heptane (CKA highly sensitive to protic solvents)

Guégain, E.; Zhu, C.; Giovanardi, E.; Nicolas, J. *Macromolecules* **2019**, *52*, 3612

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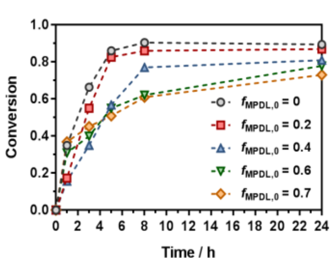
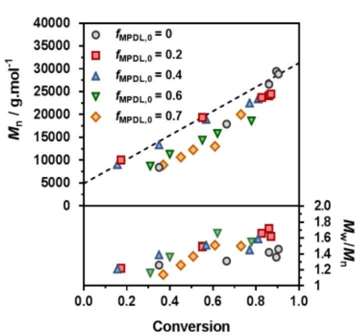
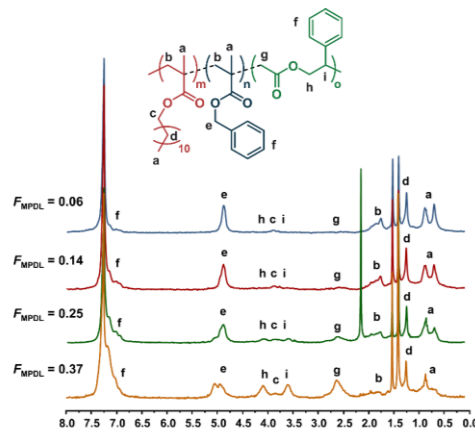
# rROPISA IN A NON-POLAR SOLVENT

## Synthesis and macromolecular characterization

**$PLMA_{18}$ and $DP_{n,BzMA}$ = 150**




- Rather high monomer conversions (75–90%)
- The higher $f_{MPDL,0}$, the lower the monomer conversion
- Linear evolution of $M_n$ vs. conversion with rather low dispersities
- Tunable MPDL insertion monitored by $^1$H NMR

Guégain, E.; Zhu, C.; Giovanardi, E.; Nicolas, J. *Macromolecules* **2019**, *52*, 3612

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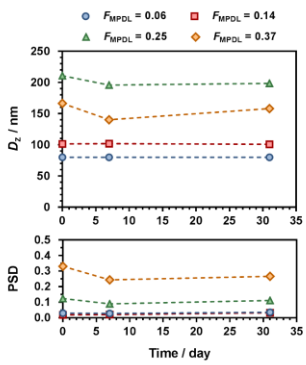
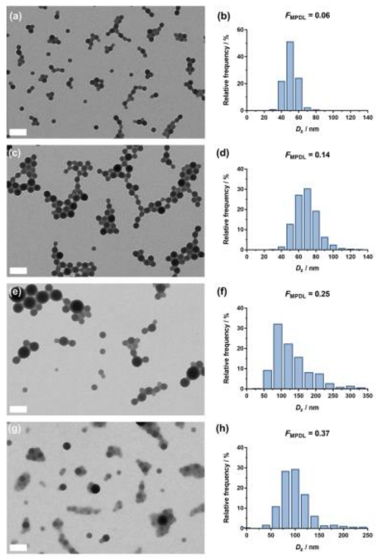
# rROPISA IN A NON-POLAR SOLVENT

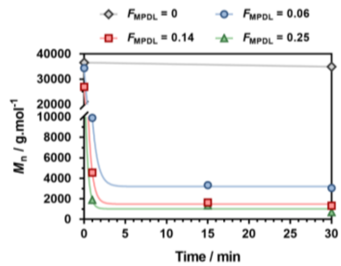
## Colloidal properties and degradation

**PLMA$_{18}$** and ***DP***$_{n,BzMA}$ **= 150**



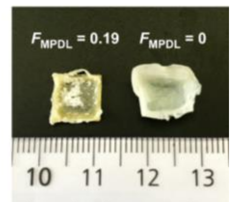




- Stable particles with narrow PSD
- The higher $f_{MPDL,0}$, the higher $D_z$




**31%** water uptake

**14%** weight loss




*(after 3 days)*

**11%** water uptake

**1.6%** weight loss

- Up to complete copolymer degradation (KOH 5%)
- MPDL-containing film: brittle, many cracks

Guégain, E.; Zhu, C.; Giovanardi, E.; Nicolas, J. *Macromolecules* **2019**, *52*, 3612

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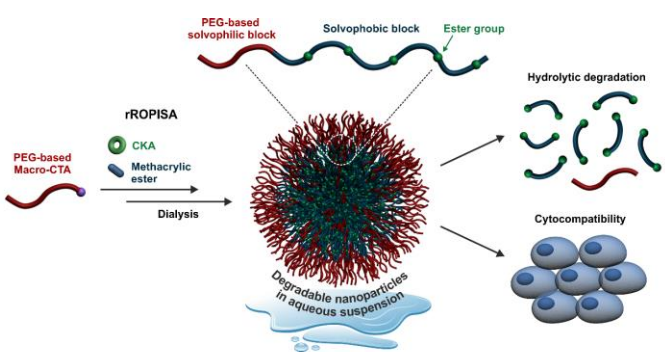
# rROPISA IN DMF AND TRANSFER TO WATER

## Synthetic strategy




- Aqueous suspensions of degradable CKA-containing nanoparticles

Zhu, C.; Denis, S.; Nicolas, J. *Chem. Mater.* **2022**, *34*, 1875

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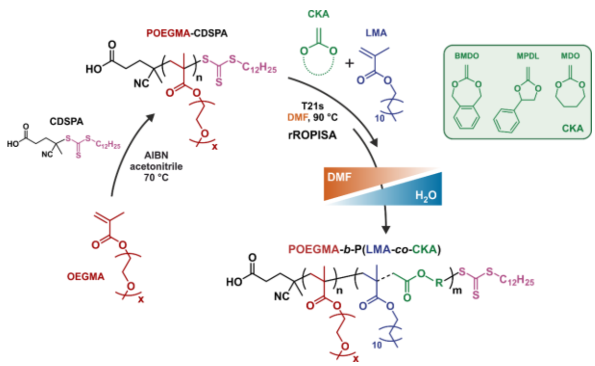
14

# rROPISA IN DMF AND TRANSFER TO WATER

## Synthetic strategy




- Aqueous suspensions of degradable CKA-containing nanoparticles

Zhu, C.; Denis, S.; Nicolas, J. *Chem. Mater.* **2022**, *34*, 1875



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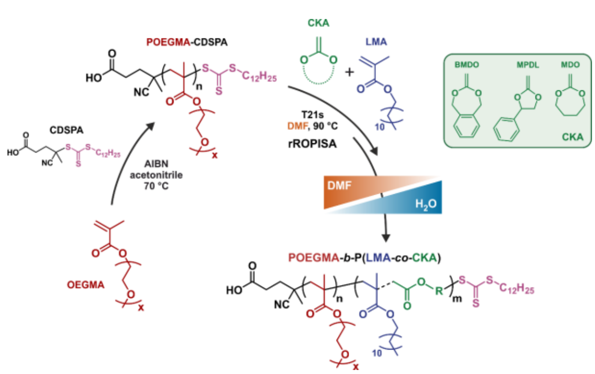
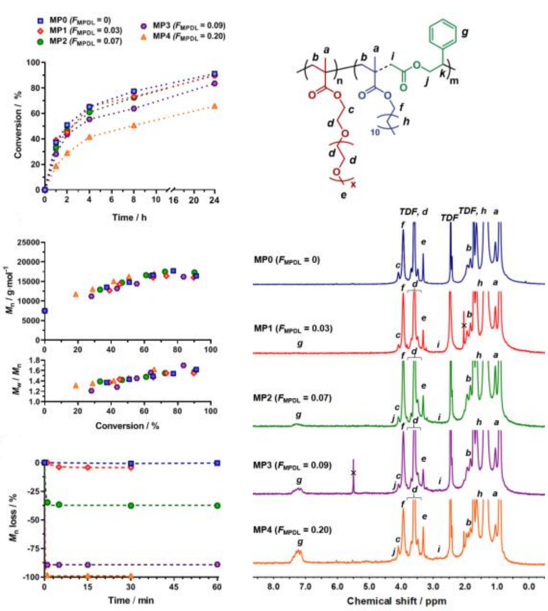
# rROPISA IN DMF AND TRANSFER TO WATER

## Synthesis and macromolecular characterization



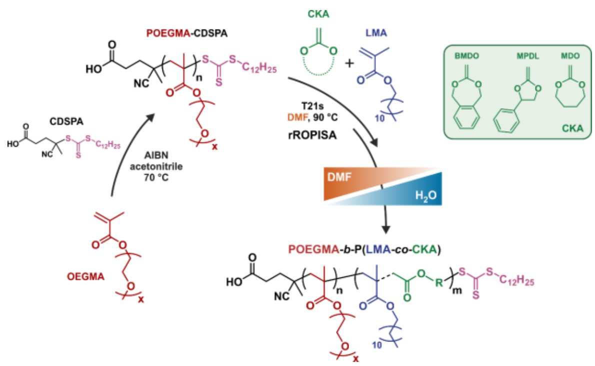



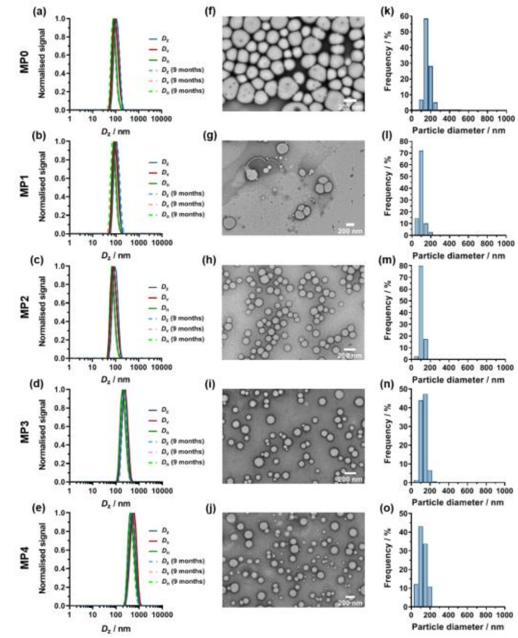

- Aqueous suspensions of degradable CKA-containing nanoparticles
- High conversions, good control, tunable $F_{MPDL}$ and degradation

Zhu, C.; Denis, S.; Nicolas, J. *Chem. Mater.* **2022**, *34*, 1875

# rROPISA IN DMF AND TRANSFER TO WATER

## Colloidal properties after transfer to water




- Aqueous suspensions of degradable CKA-containing nanoparticles
- High conversions, good control, tunable $F_{MPDL}$ and degradation
- Successful transfer to water, stable and narrowly dispersed nanoparticles
- Colloidal characteristics maintained $D_{z,DMF}$ ~ $D_{z,water}$

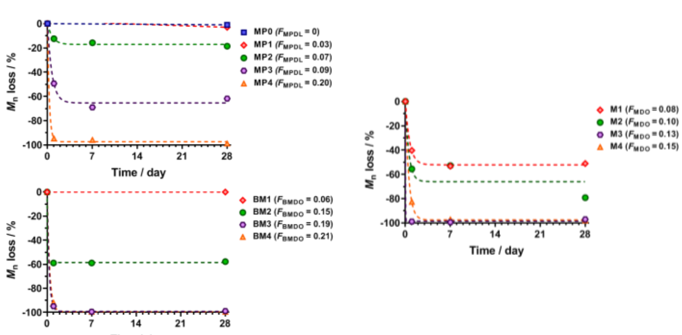
Zhu, C.; Denis, S.; Nicolas, J. *Chem. Mater.* **2022**, *34*, 1875

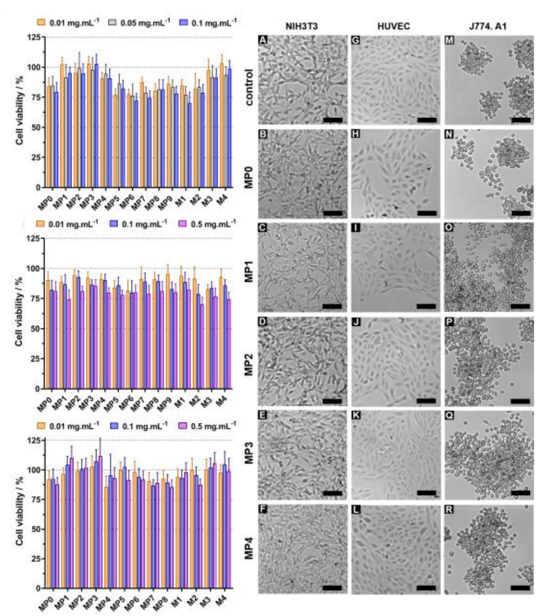




# rROPISA IN DMF AND TRANSFER TO WATER

## Degradation and cytocompatibility



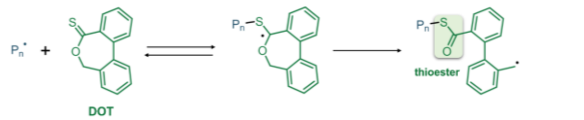




- Hydrolytic degradation of the nanoparticles (MPDL, BMDO and MDO) under accelerated conditions
- High cell viabilities up to 0.5 mg.mL$^{-1}$ on NIH/3T3, HUVEC and J774.A1 cells
- No cellular morphological changes on the three cell lines used
- Excellent cytocompatibility

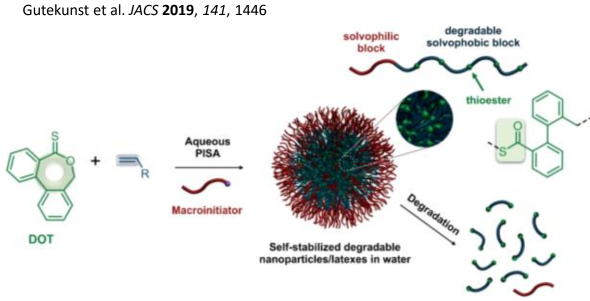
Zhu, C.; Denis, S.; Nicolas, J. *Chem. Mater.* **2022**, *34*, 1875

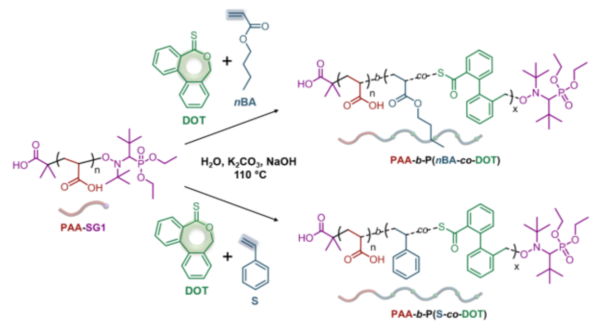
# AQUEOUS rROPISA USING A THIONOLACTONE

## Synthetic strategy using NMP




Roth et al. *Chem. Commun.* **2019**, *55*, 55
Gutekunst et al. *JACS* **2019**, *141*, 1446




- CKAs rapidly degrade in water
- Use of DOT (thionolactone) → thioester group upon rROP
- PAA-SG1 as macroinitiator, chain-extended by *n*BA (or Sty) + DOT

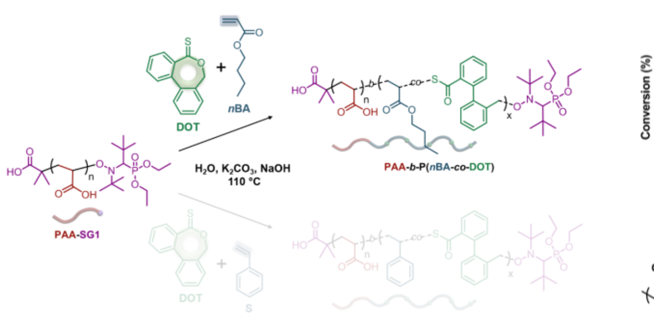
Lages, M.; Gil, N.; Galanopoulo, P.; Mougin, J.; Lefay, C.; Guillaneuf, Y.; Lansalot, M.; D'Agosto, F.; Nicolas, J. *Macromolecules* **2022**, *55*, 9790

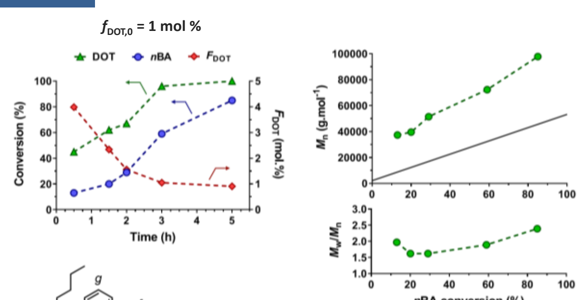


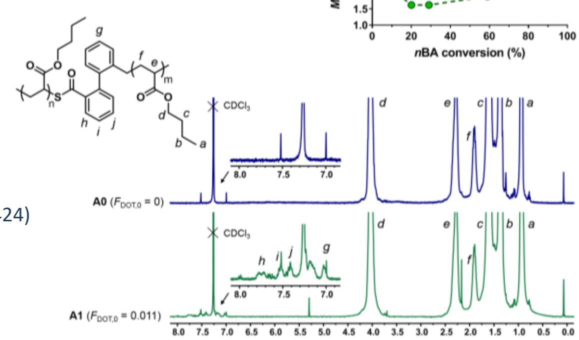


# AQUEOUS rROPISA USING A THIONOLACTONE

## Synthesis and characterization




- More favorable reactivity ratios than with CKAs ($r_{DOT}$ = 0.003; $r_{MA}$ = 0.424)
- Variation of $F_{DOT}$ (1–3 mol %), $DP_{n,PAA}$ (20–40), $DP_{n,PnBA}$ (200–600)

Lages, M.; Gil, N.; Galanopoulo, P.; Mougin, J.; Lefay, C.; Guillaneuf, Y.; Lansalot, M.; D'Agosto, F.; Nicolas, J. *Macromolecules* **2022**, *55*, 9790

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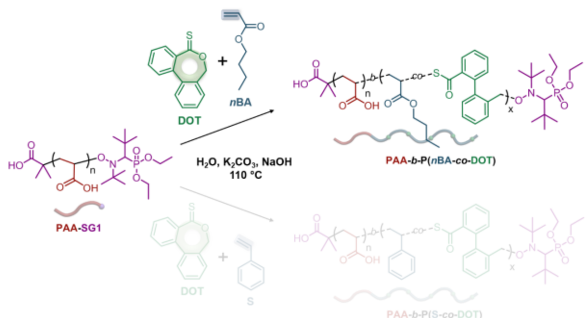
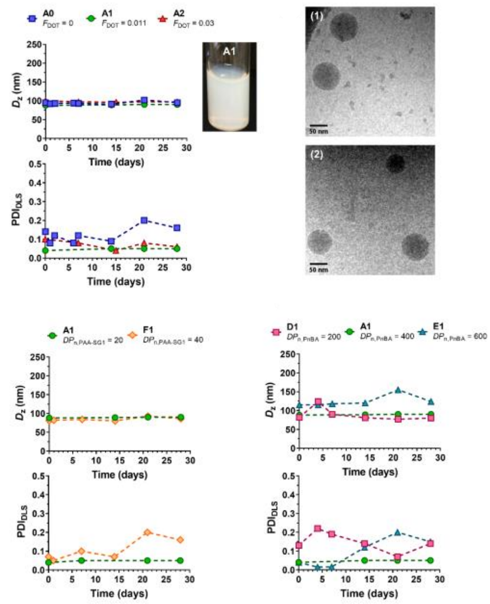
# AQUEOUS rROPISA USING A THIONOLACTONE

## Colloidal properties




- More favorable reactivity ratios than with CKAs ($r_{DOT}$ = 0.003; $r_{MA}$ = 0.424)
- Variation of $F_{DOT}$ (1–3 mol %), $DP_{n,PAA}$ (20–40), $DP_{n,PnBA}$ (200–600)
- Stable suspensions of nanoparticles/latexes, narrow PSD

Lages, M.; Gil, N.; Galanopoulo, P.; Mougin, J.; Lefay, C.; Guillaneuf, Y.; Lansalot, M.; D'Agosto, F.; Nicolas, J. *Macromolecules* **2022**, *55*, 9790

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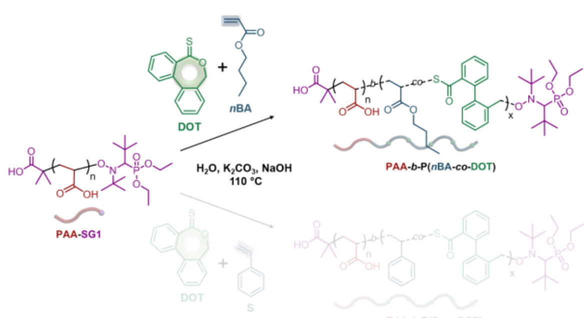
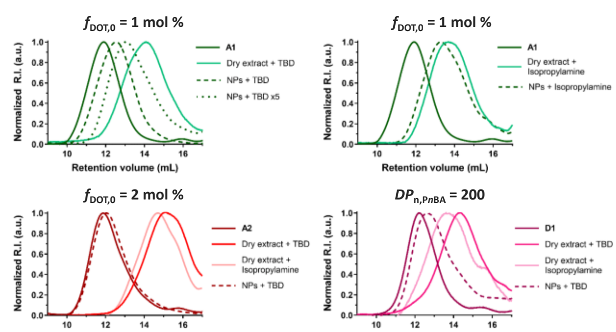
# AQUEOUS rROPISA USING A THIONOLACTONE

## Synthesis, characterization and degradation

TBD = triazabicyclodecene




- More favorable reactivity ratios than with CKAs ($r_{DOT}$ = 0.003; $r_{MA}$ = 0.424)
- Variation of $F_{DOT}$ (1–3 mol %), $DP_{n,PAA}$ (20–40), $DP_{n,PnBA}$ (200–600)
- Stable suspensions of nanoparticles/latexes, narrow PSD
- Significant **degradation** of the **dry extracts** and of the **latexes** by TBD and isopropylamine

Lages, M.; Gil, N.; Galanopoulo, P.; Mougin, J.; Lefay, C.; Guillaneuf, Y.; Lansalot, M.; D'Agosto, F.; Nicolas, J. *Macromolecules* **2022**, *55*, 9790

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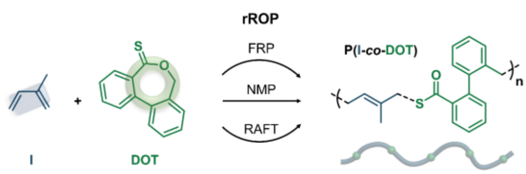
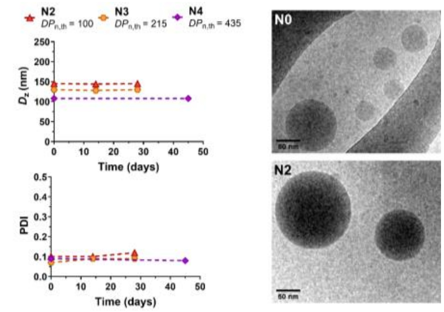
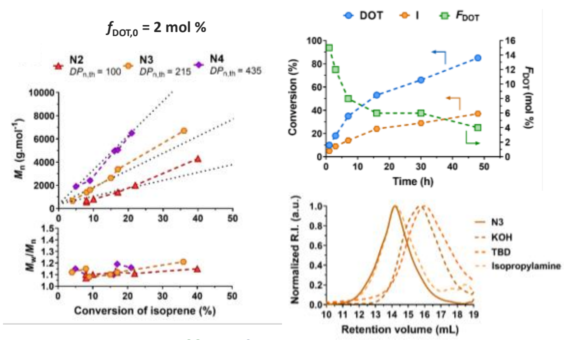
# EXTENDING THE RANGE OF VINYL MONOMERS

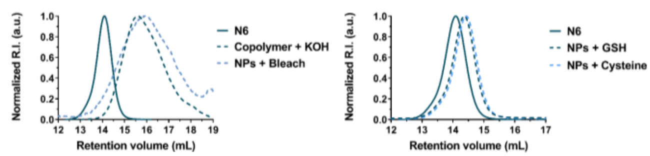
## Copolymerization of DOT with isoprene




$r_{DOT}$ = 4.28 ; $r_I$ = 0.14




- P(I-*co*-DOT) by FRP, NMP and RAFT
- P(I-*co*-DOT) nanoparticles by nanoprecipitation
- Successful degradation of the NPs by bleach, GSH and cysteine

Lages, M.; Pesenti, T.; Zhu, C.; Le, D.; Mougin, J.; Guillaneuf, Y.; Nicolas, J. *Chem. Sci.* **2023**, *14*, 3311

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# CONCLUSIONS

### RADICAL RING-OPENING POLYMERIZATION (rROP)

- Impart degradability to vinyl polymers
- Use of CKA as precursors of ester bonds in the backbone
- Applied to the synthesis of degradable poly(meth)acrylates and polystyenics
- Degradation under accelerated & physiological conditions

### NEW COPOLYMERIZATION SYSTEMS FOR rROP

- Alternating copolymers with maleimides
- Functional polyester-like copolymers with vinyl ethers
- Rapidly degradable polyacrylamides with UCST properties
- All-water formulation of degradable nanoparticles
- Degradable PI under biological conditions

### RADICAL RING-OPENING POLYMERIZATION-INDUCED SELF-ASSEMBLY (rROPISA)

- In situ synthesis of degradable nanoparticles
- rROPISA in a non-polar solvent
- rROPISA in DMF and transfer to water
- Direct aqueous rROPISA with a thionolactone
- Degradation of the nanoparticles

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## ACKNOWLEDGEMENTS


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Erika Giovanardi

Julie Mougin
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Didier Gigmes
Noémie Gil

CP2M

Franck D'Agosto
Muriel Lansalot
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IMRCP

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LA LIGUE CONTRE LE CANCER
HEALTHI
imagine
Fondation Bettencourt Schueller


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# Reactivity-Oriented Design of New Cyclic Monomers for Radical Ring-Opening Polymerization


Jia Niu
Email: jia.niu@bc.edu
X: @niu_group

Associate Professor
Department of Chemistry
Boston College


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May 2,2024

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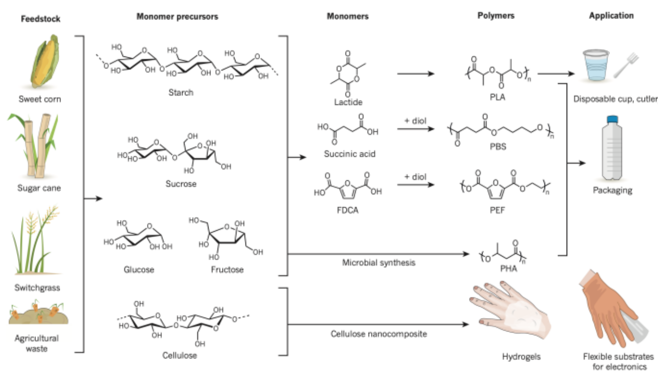
# Plastics Accumulation: Solution from Nature




Petroleum plastics: non-degradable

Bioplastics: from carbohydrate biomass

**Can we directly use carbohydrates as building blocks for polymers?**

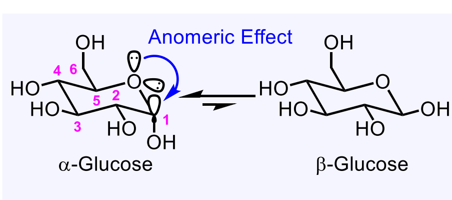
57

Image source: USA Today; Williams C. K. et al. *Nature* **2016**, *540*, 354-362.

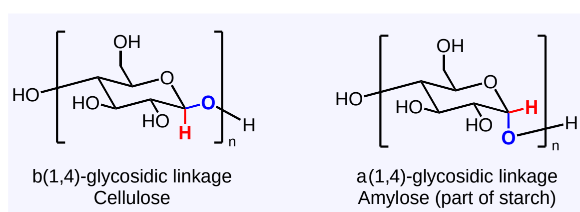


# Unique Reactivities of Carbohydrates

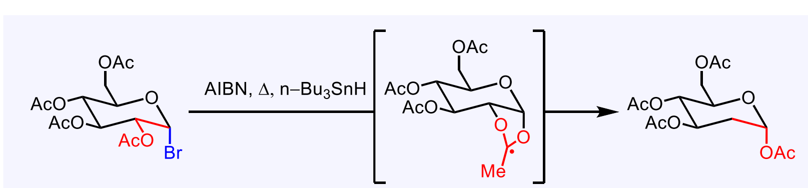
Anomeric Effect




Glycosyl Linkage




Radical 1,2-Rearrangement




(1) *Glycoscience: Chemistry and Chemical Biology*, 2nd ed.; Fraser-Reid, B. O. *et al.* Springer-Verlag, 2008. (2) Giese, B. *et al. Angew. Chem. Int. Ed.* **1987**, *26*, 233-234

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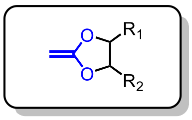
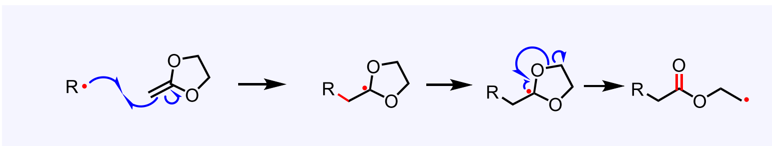
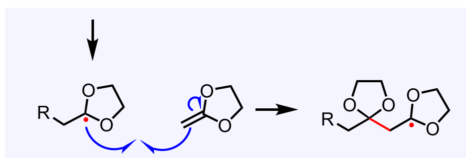
# Limitations of CKA: 1. Incomplete Ring Opening




Cyclic Ketene Acetals (CKA), reported by Bailey, W. J. and coworkers in 1982



**A. Ideal propagation**



**B. Undesired ring-retaining propagation**

For reviews on CKA polymerizations, see: (a) Agarwal, S. *Polym. Chem.* **2010**, *1*, 953. (b) Guilaneuf, Y. *et al. Chem. Rev.* **2017**, *117*, 1319.

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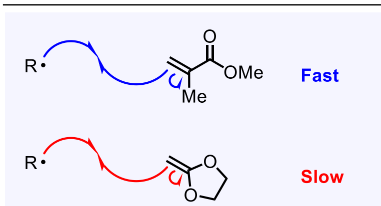
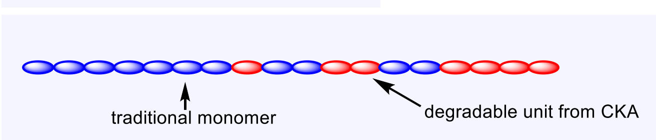
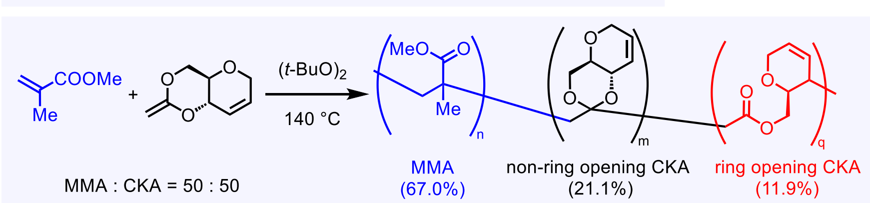
# Limitations of CKA: 2. Low Reactivity w/ Vinyl Monomers




CKA is less reactive than other vinyl monomers




Result: tapered sequence




Buchard and coworkers, 2023

(a) Buchard, A. et al. *ACS Macro Lett.* ***2023***, *12*, 1443−1449.
For reviews on CKA polymerizations, see: (b) Agarwal, S. *Polym. Chem.* **2010**, *1*, 953. (c) Guillaneuf, Y. et al. *Chem. Rev.* **2017**, *117*, 1319.

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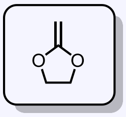
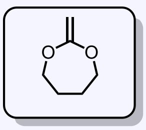
## Monomer Design Inspired by Carbohydrate Chemistry




**CKA1** 20-80% ring opening ratio



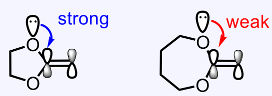

**CKA2** 100% ring opening ratio

Ring strain:


6.2 kcal/mol 6.2 kcal/mol

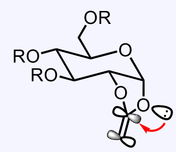
***Why reactivities are so different?***

***Weaker conjugation increases reactivity***




Geometry determined

***Our hypothesis: carbohydrate-fused ring to break conjugation***




twisted 5 member ring

⇓

weak conjugation

⇓

**higher reactivity**

(a) Taskinen, E.; Pentikainen, M.-L. *Tetrahedron* **1978**, *34*, 2365-2370. (b) Guillaneuf, Y. *et al. Chem. Rev.* **2017**, *117*, 1319; (c) Jiang, N.-C., Zhou, Z., Niu, J. *J. Am. Chem. Soc.* **2024**, *146*, 5056-5062. 61

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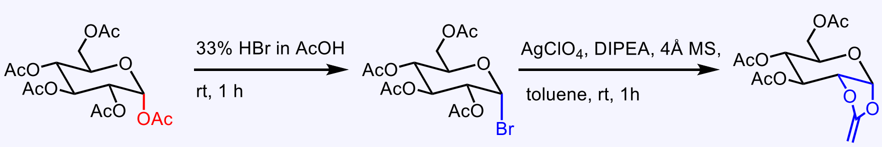
## Monomer Synthesis and Homopolymerization

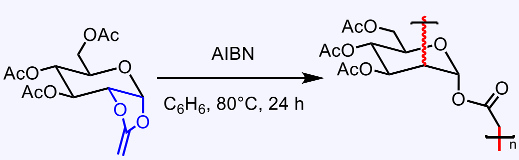



$Ac_5$-Glucose, 5g
52 USD/kg

***No column chromatography !***

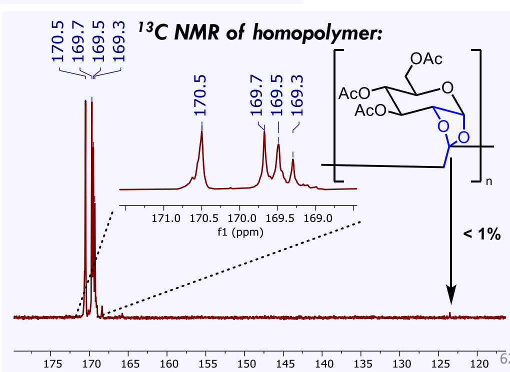
**Glucose CKA**
4.0 g obtained, 94% yield

Na-Chuan Jiang




**Glu-CKA**, 2 g

**P(Glu-CKA)**, 60% yield
$M_{n,\ MALS}$ = 21.0 kDa, $Đ$ = 1.58




Jiang, N.-C., Zhou, Z., Niu, J. *J. Am. Chem. Soc.* **2024**, *146*, 5056-5062.

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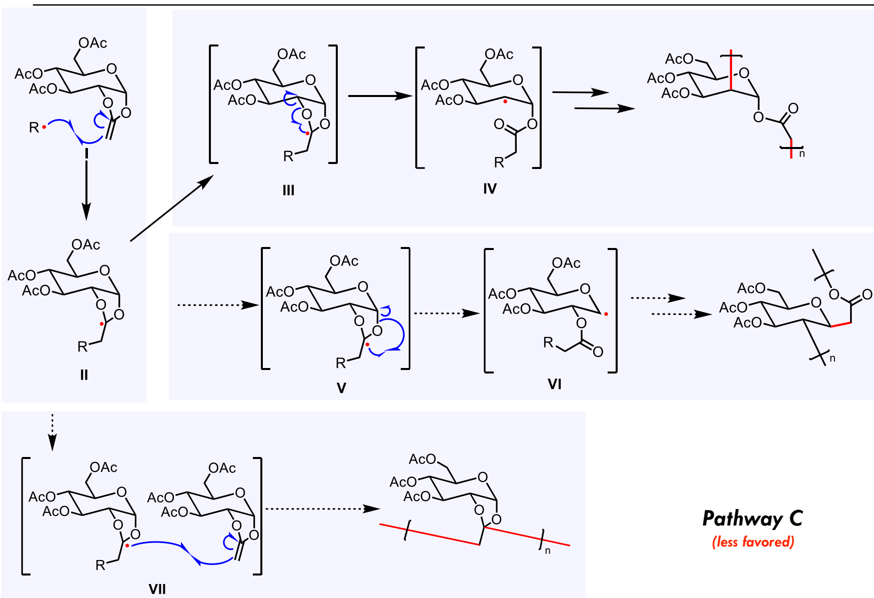
# Proposed Reaction Mechanism




***Pathway A***
*(favored)*

***Pathway B***
*(less favored)*

***Pathway C***
*(less favored)*

Jiang, N.-C., Zhou, Z., Niu, J. *J. Am. Chem. Soc.* **2024**, *146*, 5056-5062.

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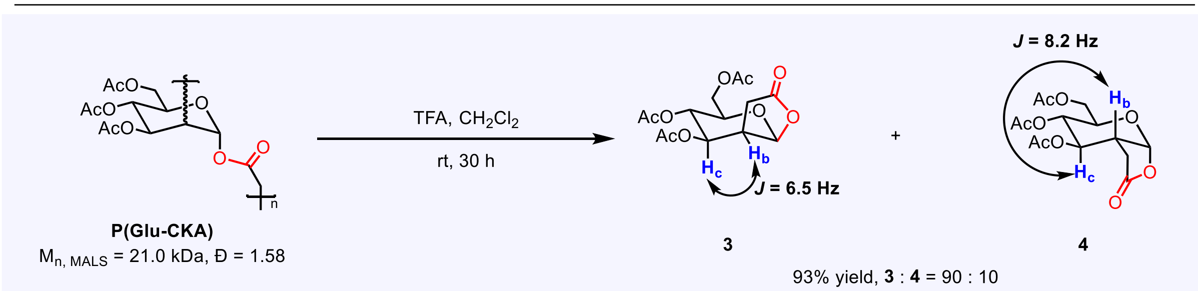
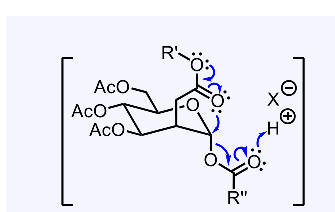
# Structural Confirmation via Degradation



Jiang, N.-C., Zhou, Z., Niu, J. *J. Am. Chem. Soc.* **2024**, *146*, 5056-5062.

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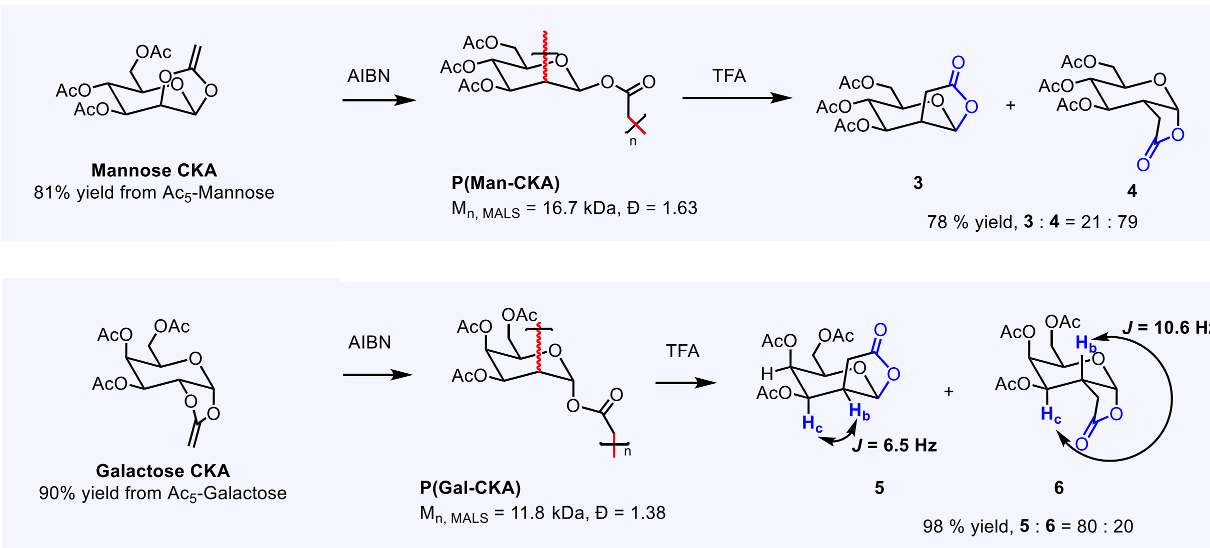
# What If We Change the Sugar?




Jiang, N.-C., Zhou, Z., Niu, J. *J. Am. Chem. Soc.* **2024**, *146*, 5056-5062.

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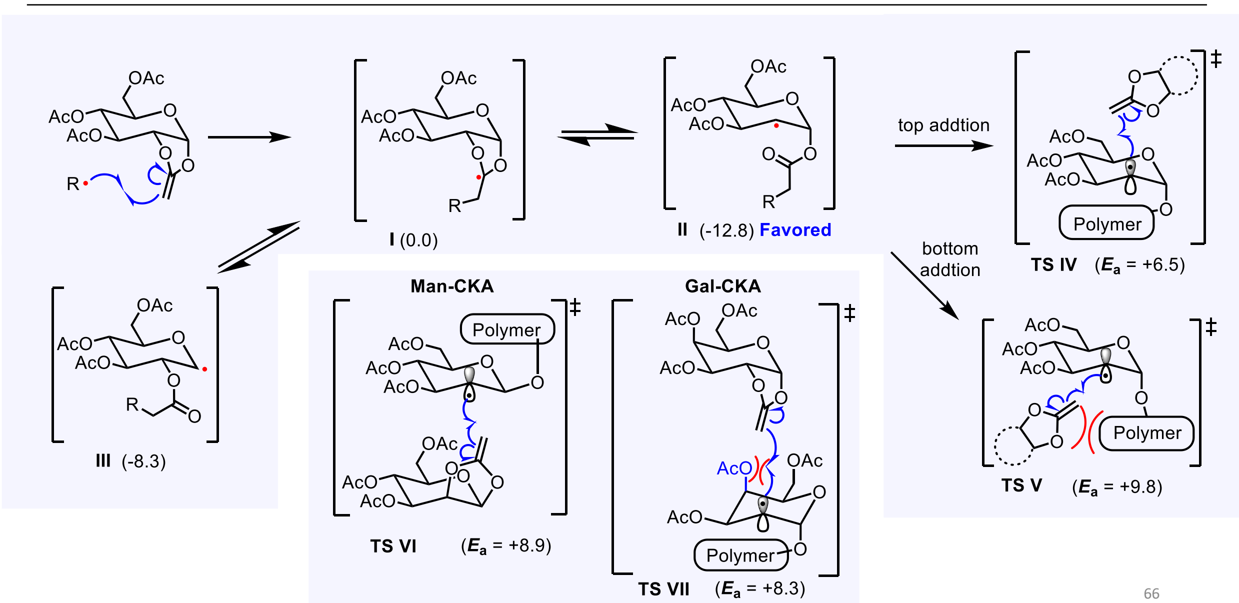
# Stereoselectivity of Monosaccharide CKAs

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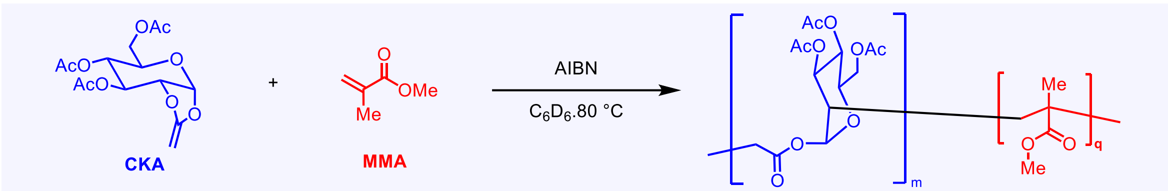
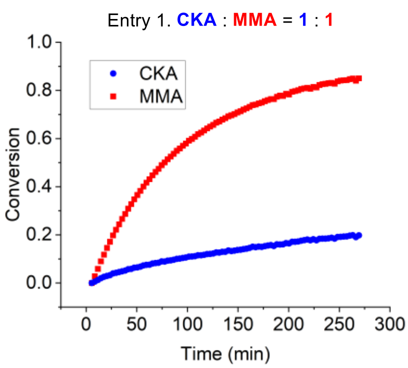
# Copolymerization w/ Vinyl Monomers




Jiang, N.-C., Zhou, Z., Niu, J. *J. Am. Chem. Soc.* **2024**, *146*, 5056-5062.

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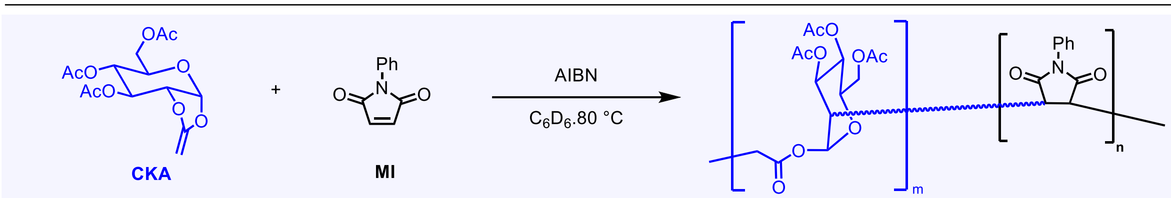
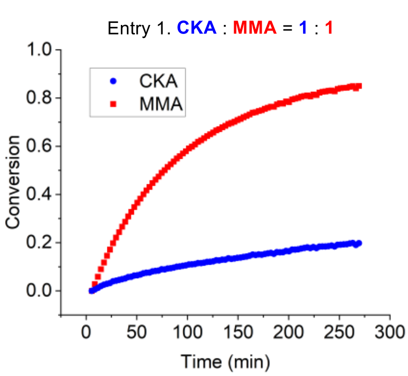
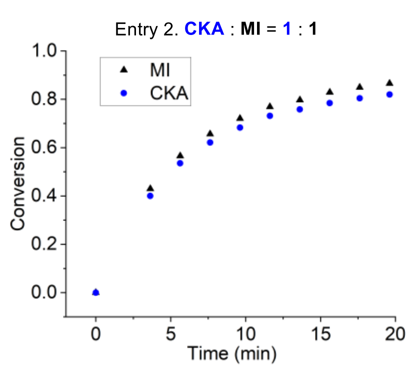
# Copolymerization w/ Vinyl Monomers




Jiang, N.-C., Zhou, Z., Niu, J. *J. Am. Chem. Soc.* **2024**, *146*, 5056-5062.

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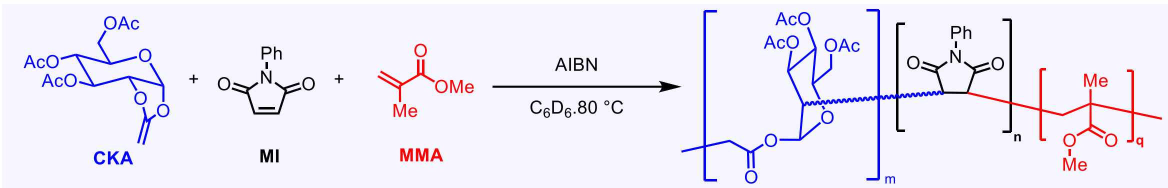
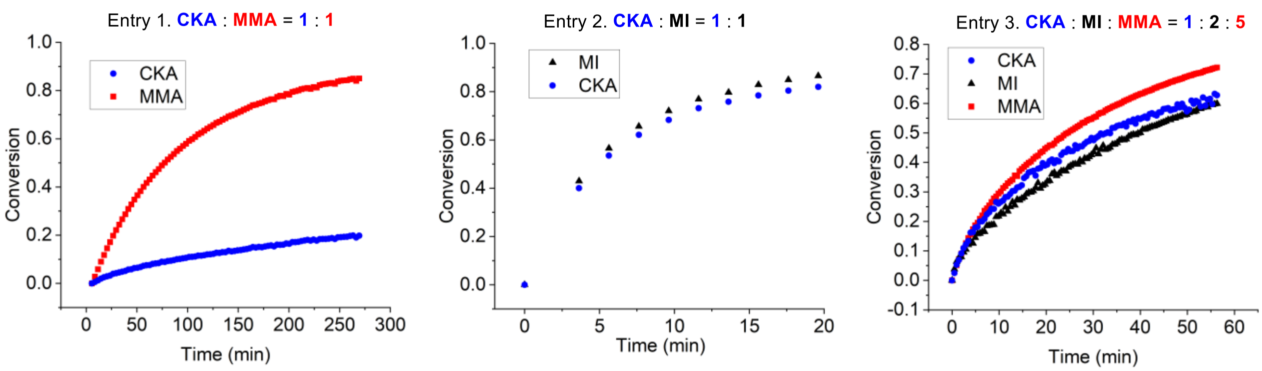
# Copolymerization w/ Vinyl Monomers




Jiang, N.-C., Zhou, Z., Niu, J. *J. Am. Chem. Soc.* **2024**, *146*, 5056-5062.

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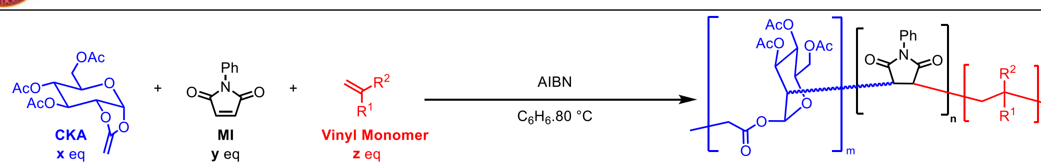
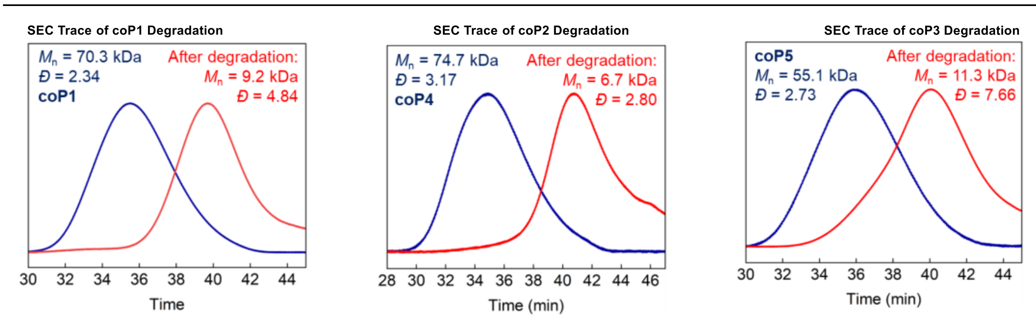
# Basic Degradation of the Copolymers




| Entry | Copolymer | Vinyl Monomer | x : y : z (feeding ratio) | m : n : q (ratio in copolymer) | Yield | $M_n$ (kDa) | $Đ$ |
|---|---|---|---|---|---|---|---|
| 1 | coP1 | MMA | 1 : 2 : 5 | 1 : 3.1 : 9.3 | 71% | 70.3 | 2.34 |
| 2 | coP2 | MA | 1 : 3 : 10 | 1 :3.3 : 10.7 | 92% | 74.7 | 3.17 |
| 3 | coP3 | DMA | 1 : 3 : 10 | 1 : 4.3 : 12.6 | 90% | 55.1 | 2.73 |

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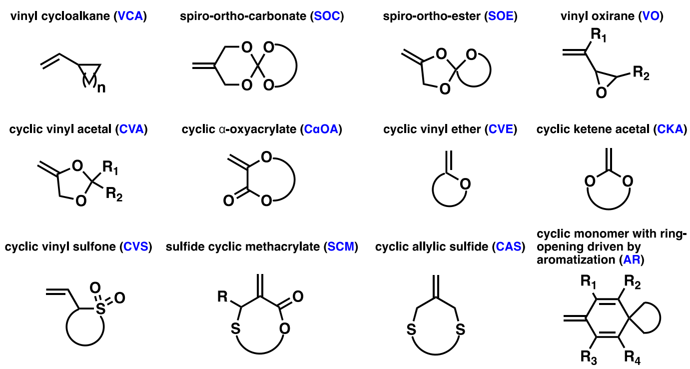
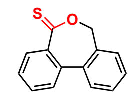
# Challenges to Traditional Cyclic Monomers

## Explored cyclic monomers

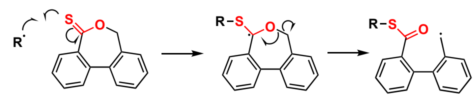



Guillaneuf, Y. et al. *Chem. Rev.* **2017**, *117*, 1319−1406.

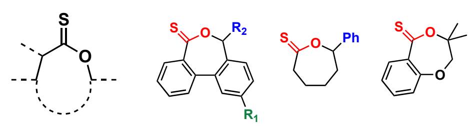
## Emerging thionolactone monomers



Dibenzooxepane-5-thione (**DOT**)



Roth, P. J. et al. *Chem. Commun.* **2019**, *55*, 55-58.
Gutekunst W. R. et al. *J. Am. Chem. Soc.* **2019**, *141*, 1446-1451.



Roth, P. J. et al. *Macromolecules* **2023**, *56*, 9787-9795.
Guillaneuf Y. et al. *J. Am. Chem. Soc.* **2023**, *145*, 27437-27449.
Johnson, J. A. et al. *J. Am. Chem. Soc.* **2024**, *146*, 9142-9154.

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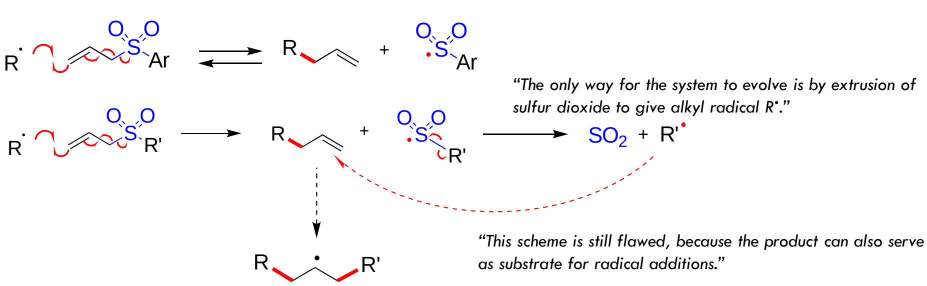
# Allylic Sulfone: Providing Driving Force to rROP

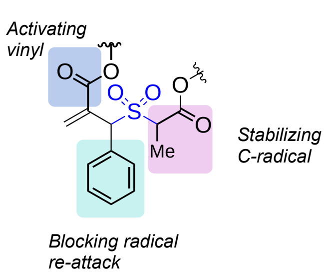


*"The only way for the system to evolve is by extrusion of sulfur dioxide to give alkyl radical R•."*

*"This scheme is still flawed, because the product can also serve as substrate for radical additions."*

- **Energetically favored**
- **Limited by side reaction**

Quiclet-Sire, B. and Zard, S. *J. Am. Chem. Soc.* **1996**, *118*, 1209-1210.




**the "ring-opening trigger"**

Hanchu Huang

Wenqi Wang

Zefeng Zhou

Brayan Rondon

Huang, H.;…Niu, J. *J. Am. Chem. Soc.* **2018**, *140*, 10402-10406.
Wang, W.;… Niu, J. *Angew. Chem. Int. Ed.* **2022**, e202113302.

- **Provides driving force for rROP**
- **Side reaction is blocked**

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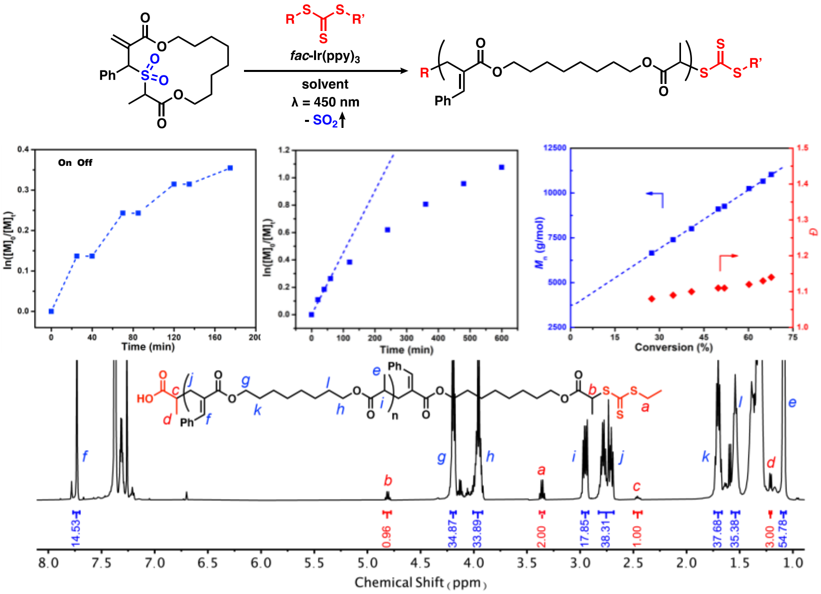
# Cyclic Allylic Sulfones: Cascade-Triggered rROP



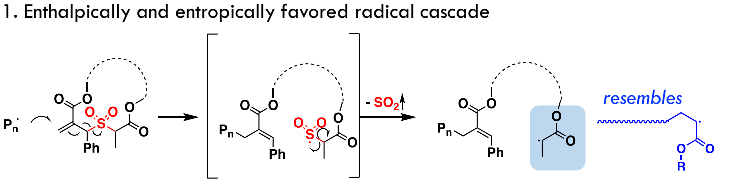

- **Light-mediated polymerization**
- **Deviation from 1st-order kinetics**
- **Living polymerization**





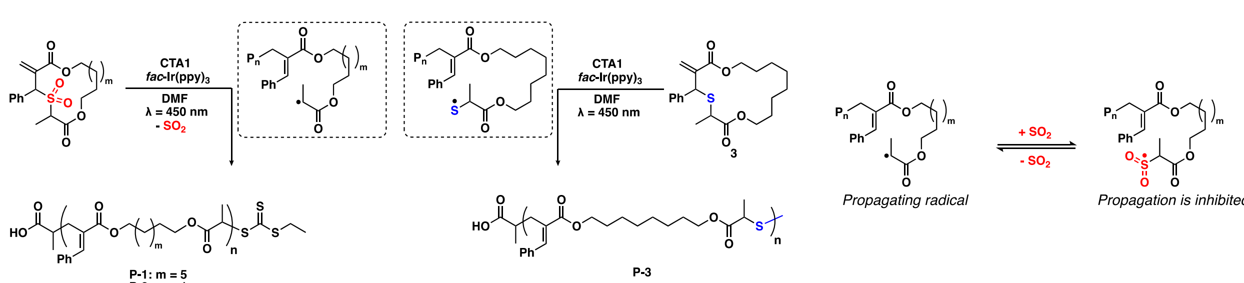
# Mechanistic Investigation

1. Enthalpically and entropically favored radical cascade



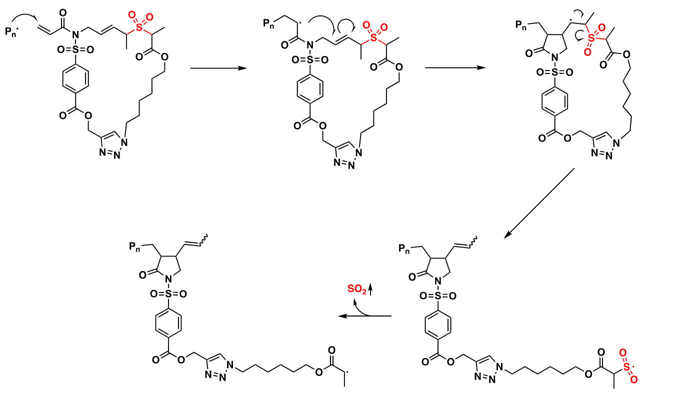

- **Complete ring opening**
- **Propagating like an acrylate radical**

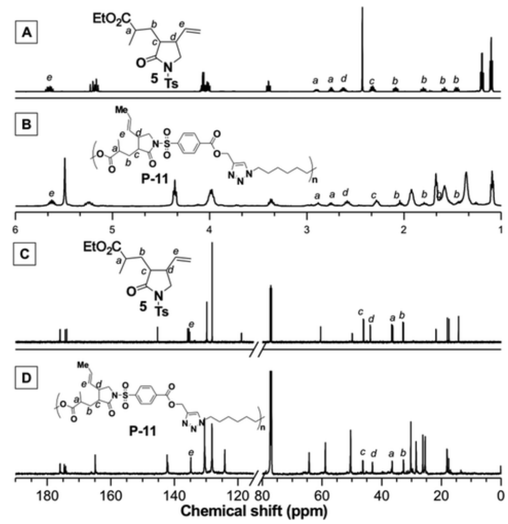
2. Reversible inhibition by $SO_2$




Wang, W.;... Niu, J. *Angew. Chem. Int. Ed.* **2022**, e202113302.

# Radical Ring-Closing/Ring-Opening Cascade Polymerization




- Both ring-closing and ring-opening steps are energetically favored
- Enhanced driving force
- More complex main-chain structure

Huang, H.;… Niu, J. *J. Am. Chem. Soc.* **2019**, *141*, 12493-12497.

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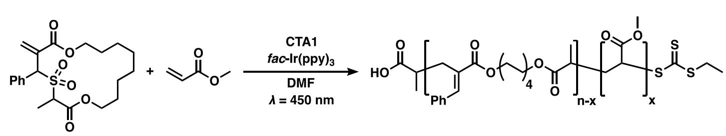
# Copolymerization w/ Vinyl Monomers Made Easy

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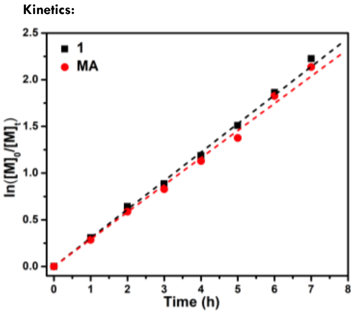
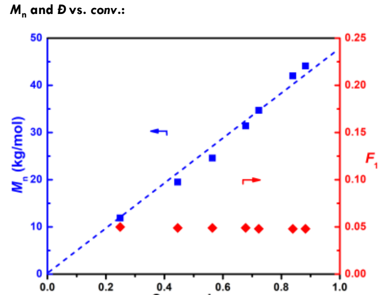
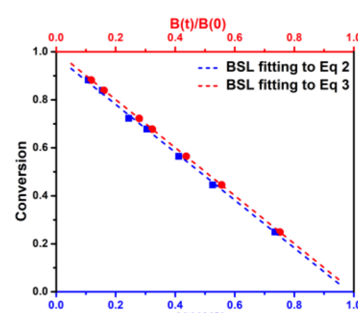
Kinetics:




$M_n$ and $Đ$ vs. conv.:




- First-order kinetics for both comonomers
- Copolymer composition remained identical.

Reactivity ratios: $r_1$ = 1.07 and $r_{MA}$ = 0.94

Wang, W.;… Niu, J. *Angew. Chem. Int. Ed.* **2022**, e202113302. Beckingham, B. S.; Sanoja, G. E., Lynd, N. A. *Macromolecules* **2015**, *48*, 6922-6930.

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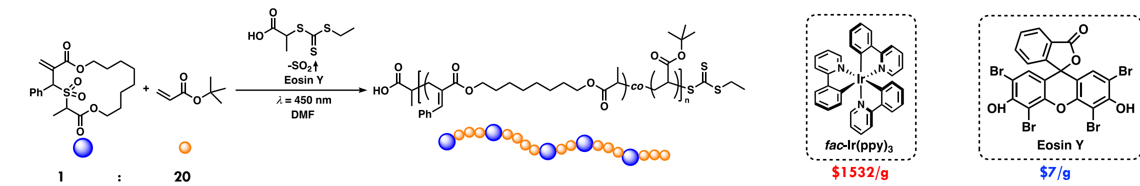
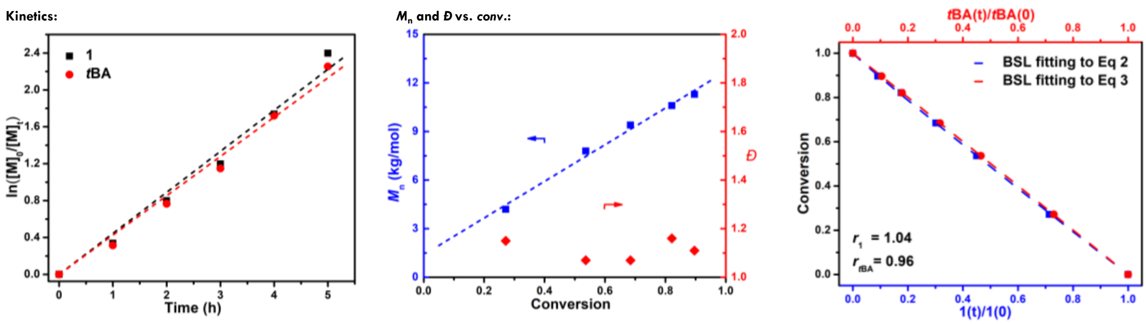
# Organocatalyst Eosin Y Reduced Cost of Reaction




Wang, W.; Rondon, B.; and Niu, J. *Macromolecules* **2023**, *56*, 2052-2061.

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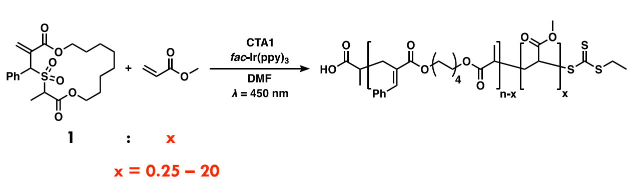
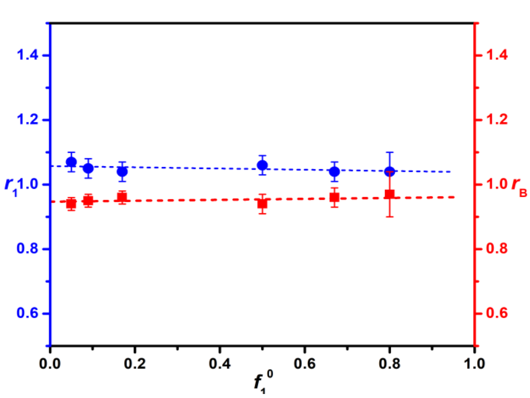
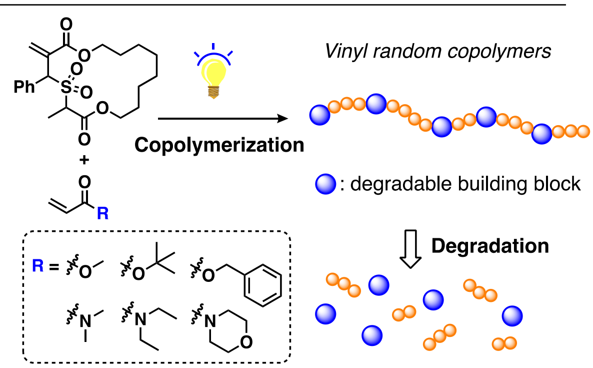
# Favorable Reactivity w/ Various Feed Ratios and Monomers




| No. | B | $f_1^0$ | Conv. | $M_n^{(SEC)}$ (g/mol) | Đ | $r_1$ | $r_B$ | $F_1$ |
|---|---|---|---|---|---|---|---|---|
| 1 | tBuA | 0.09 | 85% | 39000 | 1.29 | 1.04 | 0.96 | 0.09 |
| 2 | BnA | 0.09 | 89% | 22100 | 1.38 | 0.84 | 1.19 | 0.08 |
| 3 | DMA | 0.09 | 75% | 16100 | 1.21 | 1.03 | 0.97 | 0.11 |
| 4 | DEA | 0.09 | 79% | 20100 | 1.14 | 0.89 | 1.16 | 0.07 |
| 5 | NAM | 0.09 | 82% | 20900 | 1.34 | 0.94 | 1.07 | 0.09 |

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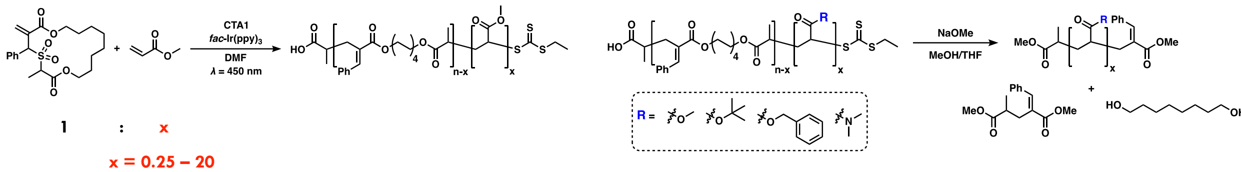
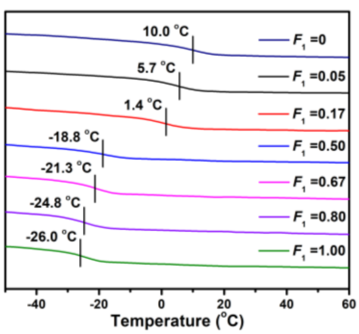
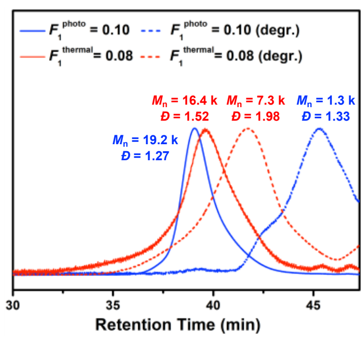
# Random Sequence Improved Degradability of Copolymer




- $T_g$ tunable by copolymer composition




- Degradability optimized by random sequence

Wang, W.;… Niu, J. *Angew. Chem. Int. Ed.* **2022**, e202113302.

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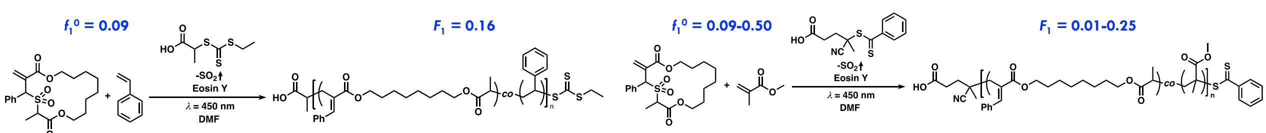
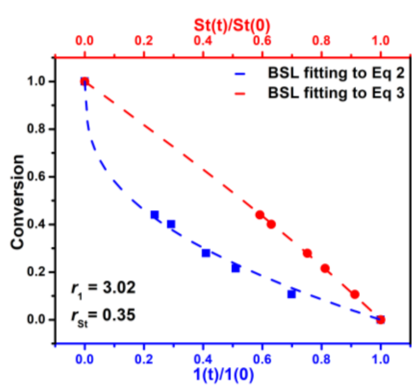
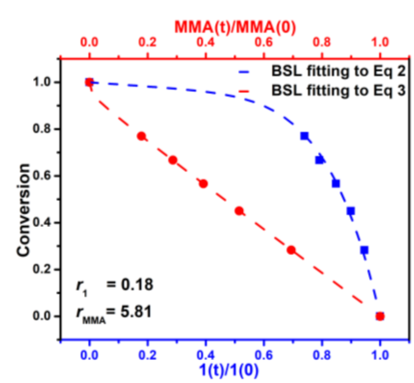
# Copolymerization w/ Methacrylate & Styrene




Wang, W.; Rondon, B.; and Niu, J. *Macromolecules* **2023**, *56*, 2052-2061.

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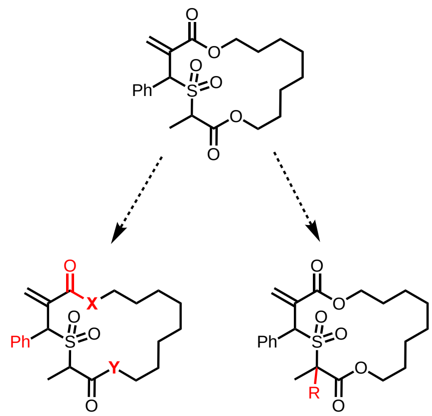
# Future Direction: Mechanism-Guided Structure Optimization

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# Conclusion

- Fusing CKA with a carbohydrate substrate enhanced reactivity in rROP.
- Adding maleimides improved CKA incorporation in copolymerization.
- Radical cascade reactions can be used to provide additional driving force for rROP.
- Cyclic allylic sulfones exhibited enhanced reactivities in homopolymerization and copolymerization.

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- Prof. Dunwei Wang
- Prof. Chenfeng Ke
- Prof. Natalia Shustova

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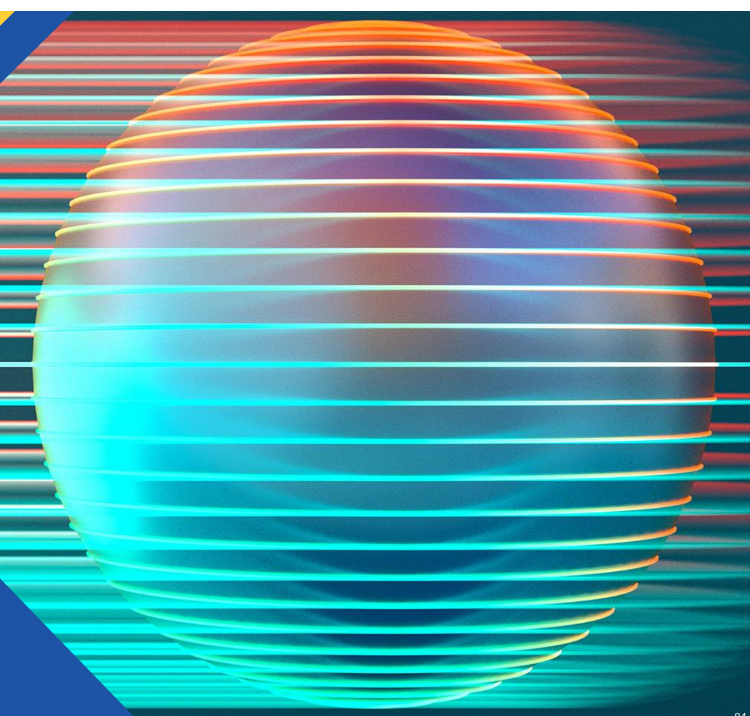

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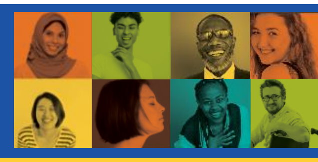

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