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Workshop Chair: Marc Mihaylo (mihaylo@chem.ucr.edu) or
Contact: Great Plains Polymer Group

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Advancing Polymer Science with Organic Catalysts

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Audience Survey Question

ANSWER THE QUESTION ON BLUE SCREEN IN ONE MOMENT

Do you have experience in using organic catalysis?

• Yes, I’m expert level!
• Yes, I have used them a lot
• Yes, but only a little
• No, I have never tried
Advancing Polymer Science with Organic Catalysts

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New Organocatalysts and Processes for the Synthesis of Functional Materials

Advancing Polymer Science with Organic Catalysts
Sept. 15, 2021
Robert Waymouth, Stanford University
New Organocatalysts and Processes for the Synthesis of Functional Materials

Advances in Catalyst Design Continue to Drive Innovation in Polymer Science

Ring-Opening Polymerization of Lactones: Metal Catalysts

\[ \text{ROH} + \text{O} \text{O} \xrightarrow{\text{catalyst}} \text{R'O O O H} \]

MOR, \( M = \text{Li, Na, K} \)
- \( \text{Al(OR)}_3 \)
- \( \text{Sn(O}_2\text{CR)}_2 \)

Kricheldorf, Jerome Pencek, Duda

Spassky, Feijen

Coates

Tolman, Hillmyer

Proposed Mechanism
Organocatalytic Polymerization

- Phosphines (2002)
- DMAP
- N-Heterocyclic Carbenes (NHCs) (2002)
- Thiourea-Amines (2005)
- Phosphazenes (2007)
- Amidines and Guanidines (2006)
- Breslow (1958)

Zhang, Hedrick Waymouth, Nat. Chem. 2016, 1047

Synthesis of Cyclic Polyesters via NHC Catalysts

1. THF, 25 °C, 5-120 s
2. CS₂

[laclide]ₙ = 0.6 M

- Highly Active
- Controlled
- Mn tracks % conversion
- Low PDIs
- Forms large ring cyclics

Mₙ (Da) | time (s) | M/Cat | conv (%) | PDI
---|---|---|---|---
28574 | 120 | 200 | 92 | 1.22
33957 | 120 | 100 | 94 | 1.24
28648 | 15 | 30 | 92 | 1.24
11742 | 12 | 200 | 30 | 1.14
12044 | 5 | 100 | 29 | 1.16
13566 | 5 | 30 | 29 | 1.11
5855 | 5 | 200 | 7 | 1.20

Mₙ tracks conversion, but non-zero intercept

Culkin, Szhony, Hedrick, Waymouth ACIEE, 2007, 2627
Jeong, Waymouth, et al, JACS, 2009, 4884
Mechanistic Anomalies: Zwitterionic Polymerization

(1) chain-growth with chain transfer
  Mn constant with conv., PDI = 2.0

(2) "living"
  Mn increase with conv., PDI = 1.0
  first-order kinetics

\[ k_p = 48.7 \text{ (M}^{-1}\text{s}^{-1}) \]
\[ k_i = 0.274 \text{ (M}^2\text{s}^{-1}) \]
\[ k_c = 0.0575 \text{ (s}^{-1}) \]
\[ k_d = 0.208 \text{ (s}^{-1}) \]


Kinetic Model of Zwitterionic Polymerization

- initiation: slow, second order in \([M]\)
- propagation fast
- cyclization slow

\[
\begin{align*}
\frac{d[Z_1]}{dt} &= \frac{k_1 k_2 [I] [M]^2}{k_1 + k_2 [M]} \\
\frac{d[I]}{dt} &= k_i [I] [M]^2 - k_2 [I] [M]^2 - k_1 ([I]_0 [Z_n]) \\
\frac{d[M]}{dt} &= 2k_i [I] [M]^2 + (k_p [M] + k_d ([Z_n])
\end{align*}
\]

where

\[ [Z_n] = ([I]_0 - [I]) \]

Bifunctional Thiourea Catalysis for Lactide Polymerization

<table>
<thead>
<tr>
<th>[M]/[I]</th>
<th>Time</th>
<th>Conv %</th>
<th>DP</th>
<th>Mn, GPC</th>
<th>PDI</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>24 h</td>
<td>97</td>
<td>21</td>
<td>5200</td>
<td>1.08</td>
</tr>
<tr>
<td>50</td>
<td>32 h</td>
<td>98</td>
<td>53</td>
<td>12300</td>
<td>1.05</td>
</tr>
<tr>
<td>100</td>
<td>48 h</td>
<td>97</td>
<td>103</td>
<td>23000</td>
<td>1.05</td>
</tr>
<tr>
<td>200</td>
<td>105 h</td>
<td>98</td>
<td>215</td>
<td>42000</td>
<td>1.05</td>
</tr>
<tr>
<td>500</td>
<td>144</td>
<td>95</td>
<td>-^e</td>
<td>-^e</td>
<td>-^e</td>
</tr>
</tbody>
</table>

* a 5 mol% 1; [LA] = 1 M in CH₂Cl₂; ^b determined by 1H NMR; ^c degree of polymerization * not soluble in THF.

• Solvent Effect: Polymerization Observed in CH₂Cl₂, CHCl₃, toluene
  - No polymerization observed in THF, DMF


### Chain Extension: No Transesterification

**Minimal Transesterification**

\[
\begin{align*}
\text{RO} \left[ \begin{array}{c} O \end{array} \right]_n \rightarrow & \text{cat (5\%)} \\
1 \text{ week, RT} & \rightarrow \\
\text{RO} \left[ \begin{array}{c} O \end{array} \right]_n &
\end{align*}
\]

Mn = 21,300; PDI of 1.06

**Chain extension**

\[
\begin{align*}
\text{RO} \left[ \begin{array}{c} O \end{array} \right]_n & \rightarrow \text{cat (5\%)} \\
48 \text{ h, RT} & \rightarrow \\
\text{RO} \left[ \begin{array}{c} O \end{array} \right]_n &
\end{align*}
\]

DP of 103 (PDI of 1.05) additional 100 equiv of lactide

DP of 215 (PDI of 1.05)
Origin of High Selectivity?

$K = 39$  
$C_6D_6$

can’t measure by $^1H$ NMR

$K$ probably less than 5

DFT (Hans Horn, Julia Rice (IBM) Anthony De Crisci (Stanford)

Development of Fast AND Selective Catalysts

- Selective OR Fast Catalysts (2005)

- Selective AND Fast Catalysts


Origin of High Selectivity?

measured by $^{13}$C NMR

$K = 35$

$\text{CH}_2\text{Cl}_2$

$K = 24$

$\text{CH}_2\text{Cl}_2$

$K = 5$

$\text{CH}_2\text{Cl}_2$

• Higher Binding constant for s-trans lactones vs. s-cis esters:
  
  • catalyst highly selective for opening lactone vs. transesterification


Urea Anions: Efficient Catalysts for Polymer Synthesis

Lohmeier, Hedrick Waymouth, Macro. 2006, 8574

100 equiv.

CH$_2$Cl$_2$

120 hours, 78% conv

(M$_n$/M$_\theta$ = 1.04 @ 78% conv.)

3 equiv.

1 equiv. KOMe

THF

10 min, 94% conv

A New Catalyst Platform Tailored for Continuous Flow Processes


A New In-Flow Catalyst Switch for Rapid Generation of Multiblock Copolymers

poly(CL)_{25}-block-poly(TMC)_{25}-block-poly(L-LA)_{25}

r = 3.5 seconds, $M_n$GPC = 12 kDa, D = 1.08.
Programmed Library Generation in Flow Reactor

Automated, programmed generation of 100 separate VL-b-PLA diblock copolymers generated in 10 minutes

Length of each block ranging from 10 to 46 in increments of four monomer repeat units


Polycarbonates: Synthetic Multifunctional Polymers

Degradable Polycations: Synthesis and Mechanism


Degradation: Selectivity for DKP

Model S1: random at any point in chain; 
\( \frac{k_0}{k_6} = 10^6 \) 
\( k_6 \gg k_5 = 0.0412 \text{ min}^{-1} \)

Model S2: degradation from chain-end; 
\( \frac{k_0}{k_6} = 10^6 \) 
\( k_6 \gg k_5 = 1.44 \text{ min}^{-1} \)

Model S3: random at any point in chain; 
\( k_5 = k_6 = 0.483 \text{ min}^{-1} \)

Degradation: Influence of Structure

\[ \text{Degradable Polycations: A Mechanism of mRNA Release} \]

mRNA delivery: cell culture

Using new amphipathic materials we were able to transfect mRNA and elicit the expression of Green Fluorescent Protein (HeLa cells)

- OROP and functionalized monomers allows for rapid screening for function
- Expression levels are higher than commercial Lipofectamine
- Non-toxic

McKinlay, Vargas, Blake, Hardy, Kanada, Contag, Wender, Waymouth, "Charge-altering Releasable Transporters (CARTs) for the delivery and release of messenger RNA in living animals" Proc. Natl. Acad. Sci., 2017, E448

To Express mRNA, mRNA Must Escape Endosome

Confocal Microscopy
- Allows for independent imaging of transporter and cargo on a cell-by-cell basis

Fluorophores
- Dansyl: attached to transporter
- GFP: indicates expression has occurred
- Dextran: stains endosomes
- Cy5: attached to mRNA

Conditions: HeLa cells, 10:1 +/- charge ratio, 4 hours following treatment
Broad Chemical Space

- Functional initiators
- New lipids
- New charge altering polycations
- Fluorophores / Dyes

mRNA delivery with CARTs into T lymphocytes

- Combinatorial library: CART mixtures (2:1) High throughput screen
- >99% transfection efficiencies in many cultured cell types
- Low lymphocyte transfection

**Background**

**Jurkat Cells**

- 80% transfection using hybrid and mixed lipid CARTs
- 8-fold increase over D13:A11 and Lipo

Lipid-mixed CARTs enhance mRNA delivery into:

- T cells and B cells (primary and immortalized)

McKinlay, C.; Vargas, J. et al. PNAS 2017, 114, E448

McKinlay, C.; Benner, N. et al. PNAS 2018, 115, E5859
mRNA expression is effective via multiple routes of administration in vivo

Intramuscular

Intravenous (tail-vein)

Topical

Biodistribution of mRNA expression influenced by mode of administration

The Plague Year

See: “the Plague Year”. Lawrence Wright, The New Yorker, Jan 4&11, 2021
The CART-mRNA Approach to COVID 19 Vaccination:
Deliver the message, cells make the vaccine
Levy, Waymouth and Wender labs

mRNA vaccines are quick and inexpensive to make, produce RBD and elicit immune response

RNA encoding RBD Protein Fragment

CART-mRNA-RBD plus adjuvant

CART-mRNA-RBD plus adjuvant

In vivo cell machinery

RBDs only

Immune system

RBD Antibodies & Effector cells

CV exposure

CV clearance


The CART-mRNA Approach to COVID 19 Vaccination:
Levy-Waymouth-Wender labs

CART-mRNA-RDB elicits a protective immune response

Sera from immunized mice (IM in red, IV in blue, n=5) was harvested on Day 28. Serum from blood donors (n=13) who were vaccinated with the Pfizer/BioNTech mRNA vaccine was collected either within 7 days before (pre boost, black) or 15±4 days after the boost (post boost, green) was tested for the ability to inhibit RBD/ACE-2 binding using a commercially available surrogate Virus Neutralization Test.

• Neutralizing antibody levels of immunized mice are comparable to those achieved in vaccinated humans

New Organocatalysts and Processes for the Synthesis of Functional Materials

Advances in Catalyst Design Continue to Drive Innovation in Polymer Science

Dr. Tim Blake, Rebecca McClellan, Keith Armstrong, Conor Galvin, Summer Ramsay-Burroughs, Vince Pane, Caleb Jadrich, Dan Marron, Dr. Trevor Del Castillo, Dr. Blaine McCarthy Jim Zhang, Yuan Jia, Isaac Appelbaum

**Collaborators**

Dr. James Hedrick (IBM)
Dr. Nathaniel Park (IBM)

Prof. Paul Wender (Stanford)
Prof. Ron Levy (Stanford)
Prof. Grant Rotskoff (Stanford)
Prof. Catherine Blish (Stanford)
Prof. Eric Kool (Stanford)
Dr. Ole Haalbeth (Stanford)

Prof. Jeff Glenn (Stanford)
Prof. V. Sebastiani (Stanford)

Prof. Dick Zare (Stanford)
Prof. Craig Criddle (Stanford)

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The Leukemia and Lymphoma Society
NASA
IBM
EVONIK
Cancer TNT Program (Stanford)
SPARK Program (Stanford)
ChEM-H (Stanford)
Center For Molecular Analysis and Design (Stanford Chemistry)
Team Expertise and Background

Polymers synthesis
Gene delivery

Prof. Robert M. Waymouth
Bob has pioneered the metal-free synthesis of biocompatible polymers, which are now used in many therapeutic indications, including antimicrobials, gene and drug delivery agents.

Drug and Gene delivery

Prof. Paul A Wender
Paul’s work is directed at using chemistry and synthesis to address unsolved problems in medicine, including drug delivery, a cure for HIV/AIDS, cancer immunotherapy, Alzheimer’s disease and antibiotic resistance.

Clinical Oncology

Prof. Ronald Levy
Ron researches how the immune system can be harnessed to fight cancer. His work has led to personalized anticancer drugs, inventing an antibody-based drug, Rituxan, that is widely used to treat lymphoma.

Stanford Team

Dr. Timothy R. Blake
Dr. Rebecca McClellan
Dr. Blaine McCarthy
Dr. Trevor Del Castillo
Dr. Ralph Lange
Summer Ramsay-Burrough
Yuan Jia
Isaac Applebaum

Dr. Colín McKinlay
Dr. Jessica Vargas
Dr. Nancy Benner
Harry Rahn
Zhijian Li
Gillian Sun
Dr. Steven Stanton

Using organic catalysts for step growth polymerization and depolymerisation

Prof. Andrew P. Dove
Step-Growth vs Chain Growth Polymerisation

**Step-Growth**

- $n = 1$
- $n = 2$
- $n = 3$
- $n = 4$
- $n = 5$
- $n = 6$

**Chain-Growth**

- $n = 1$
- $n = 2$
- $n = 3$
- $n = 4$
- $n = 5$
- $n = 6$

*Polymer structure is distinct from polymerisation process*
**Audience Survey Question**

**ANSWER THE QUESTION ON BLUE SCREEN IN ONE MOMENT**

What do you think is the biggest remaining challenge to overcome for organic catalysis in polymer science?

- Reaction scope
- Increasing reactivity
- High temperature operation
- Better stereoselectivity
- Other (Tell us more in the chat!)

---

**Organocatalytic Polycondensation**

Basterretxea, Jehanno, Mercerreyes, Sardon. *ACS Macro Lett.* 2019, 8, 1055-1062
Thermal Stability is Commonly a Problem

1,5,7-Triazabicyclo[4.4.0]dec-5-ene (TBD)

+ methane sulfonic acid (MSA)

37.4 kcal.mol⁻¹ more stable than TBD and MSA

TBD:MSA

TBD:MSA isochemical degradation @180 °C

TBD isochemical degradation @100 °C


Thermally stable organocatalysts

Basterretxea, Jehanno, Mercerreyes, Sardon. ACS Macro Lett. 2019, 8, 1055-1062
Basterretxea, Gabirondo, Jehanno, Zhu, Flores, Muller, Etxeberria, Mecerreyes, Coulembier, Sardon, ACS Sustainable Chem. Eng. 2019, 7, 4103-4111
Lower Temperature Step-Growth


Thiol-ene Additions in Polymer Chemistry

Radical Alkene

Radical Alkyne

Nucleophilic Alkene

Nucleophilic Alkyne
Nucelophilic Thiol-yne Addition


Speed and Selectivity

The Importance of Stereochemistry in Polymers

**Optical Isomerism**

*Example: Polypropylene*

- *Isotactic*: ~80% crystallinity; $T_m = 176 ^\circ C$
- *Syndiotactic*: ~30% crystallinity; $T_m = 130 ^\circ C$
- *Atactic*: amorphous, no defined $T_m$

**Geometric Isomerism**

*Natural rubber (cis-1,4-polyisoprene)*

- Elastic, $E = 2$ MPa

*Gutta Percha (trans-1,4-polyisoprene)*

- Brittle, $E = 80$ MPa


---

**Step-Growth Thiol-yne Addition**

*cis: 20:80 trans: 71:29*

Stereochemistry Dependent Mechanical Properties

---

Stereochemistry in Naturally-Sourced Monomers

---


Organocatalytic Thiol-ene Step-Growth

**Organocatalytic Step-Growth Polymerisation**

**Mini-synopsis**

- Organocatalysis can be used for a wide range of step-growth polymerisations – the frequently used ones and many more!
- Using organic salts, the thermal stability can be significantly increased to allow higher temperature operation for longer
- Using organocatalyzed nucleophilic thiol-yne addition chemistry, high levels of stereoselectivity can be obtained with which to control polymer properties.

Plastic Waste

Overview of Plastic Recycling Options


Organocatalytic Circular Economy Approaches


Organocatalytic Depolymerisation


Higher Temperature Stability


Depolymerise and Repolymerise

Not all polymers can be recycled...

\[
\begin{align*}
\text{BPA} + \text{Phosgene} &\rightarrow \text{Polycarbonate} \\
\text{BPA} + \text{Carbon Dioxide Gas} &\rightarrow \text{Waste Plastic}
\end{align*}
\]

Upcycling to Aliphatic Polycarbonates

\[
\begin{align*}
\text{BPA-PC} + \text{glycol} &\rightarrow 90 - 160 ^\circ \text{C} \rightarrow \text{BPA} + \text{cyclic carbonate}
\end{align*}
\]

Mixed Plastics

Selective Chemical Depolymerisation

Organocatalytic Depolymerisation

Mini-synopsis

- In the same way that organic catalysis offers excellent opportunities to create polymers, it offers excellent methods for depolymerisation of a wide range of polymers
- Thermally-stable catalysts offer a high activity alternative at high temperatures
- Leveraging kinetic differences in depolymerisation rate, different plastics can be selectively and sequentially depolymerised.
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Dr Josh Worch
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Emma Catterson
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Connor Stubbs
Recent Alumni
Dr Vinh Truong (PDRA)
Dr Craig Bell (PDRA)
Dr Ian Barker (PDRA)

Key Academic Collaborators
Prof. Matthew Becker (Duke University, USA)
Dr Haritz Sardon (POLYMAT, Spain)

http://www.dovegrouplab.com
@Dove_group
Further Reading

Reviews
Opportunities for organocatalysis in polymer synthesis via step-growth methods.

Dual Organocatalysts Based on Ionic Mixtures of Acids and Bases: A Step Toward High Temperature Polymerizations.
Basterretxea, Jehanno, Mercerreyes, Sardon. *ACS Macro Lett.* 2019, 8, 1055-1062

Organocatalytic ring-opening polymerization of l-lactide in bulk: A long standing challenge.

Click Nucleophilic Conjugate Additions to Activated Alkynes: Exploring Thiol-yne, Amino-yne, and Hydroxyl-yne Reactions from (Bio)Organic to Polymer Chemistry.

Stereochemical enhancement of polymer properties.

Original Research Articles
Switching from Controlled Ring-Opening Polymerization (cROP) to Controlled Ring-Closing Depolymerization (cRCDP) by Adjusting the Reaction Parameters That Determine the Ceiling Temperature
Olsen, Undin, Odelius, Keul, Albertsson, *Biomacromolecules* 2016, 17, 3995-4002
Further Reading

Independent Control of Elastomer Properties through Stereocontrolled Synthesis.

Organocatalytic, Regioselective Nucleophilic “Click” Addition of Thiols to Propiolic Acid Esters for Polymer–Polymer Coupling.

Organocatalysed depolymerisation of PET in a fully sustainable cycle using thermally stable protic ionic salt.

Unsaturated Poly(ester-urethanes) with Stereochemically Dependent Thermomechanical Properties.

Base-to-Base organocatalytic approach for one-pot construction of poly(ethylene oxide)-Based macromolecular structures.

Further Reading

Synthesis of Functionalized Cyclic Carbonates through Commodity Polymer Upcycling.

Elastomeric polyamide biomaterials with stereochemically tuneable mechanical properties and shape memory.

Concomitant Control of Mechanical Properties and Degradation in Resorbable Elastomer-Like Materials Using Stereochemistry and Stoichiometry for Soft Tissue Engineering.
Wandel, Bell, Yu, Arno, Dreger, Hsu, Pitto-Barry, Worch, Dove, Becker, *Nature Commun.*, 2021, 12, 446.


Further Reading

Completely Recyclable Monomers and Polycarbonate: Approach to Sustainable Polymers.

Metal-Free Synthesis of Novel Biobased Dihydroxyl-Terminated Aliphatic Polyesters as Building Blocks for Thermoplastic Polyurethanes.

Unique Base-Initiated Depolymerization of Limonene-Derived Polycarbonates.

Polyether Synthesis by Bulk Self-Condensation of Diols Catalyzed by Non-Eutectic Acid–Base Organocatalysts.

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15/09/2021

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San Diego, CA USA
Organizers: J. Fuchs, C. Hartmanns-Thompson, H. Gao, and B. Burdett

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