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6. AUTHORS

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7. PERFORMING ORGANIZATION NAMES AND ADDRESSES
University of New Hampshire
51 College Road
UNH
Durham, NH 03824 -2585

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U.S. Army Research Office
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a. REPORT	b. ABSTRACT	c. THIS PAGE			Erik Berda
UU	UU	UU	UU		19b. TELEPHONE NUMBER 603-862-1762

RPPR Final Report
as of 19-Apr-2018

Agency Code:

Proposal Number: 64881CHYIP **Agreement Number: W911NF-14-1-0177**
INVESTIGATOR(S):

Name: Erik Berda
Email: erik.berda@unh.edu
Phone Number: 6038621762
Principal: Y

Organization: **University of New Hampshire**
Address: 51 College Road, Durham, NH 038242585
Country: USA
DUNS Number: 111089470 EIN: 026000937

Report Date: 13-Nov-2017 Date Received: 13-Nov-2017
Final Report for Period Beginning 14-Apr-2014 and Ending 13-Aug-2017
Title: YIP: Origami of single polymer chains via sequentially activated permanent and reversible intra-chain interactions
Begin Performance Period: 14-Apr-2014 **End Performance Period:** 13-Aug-2017
Report Term: 0-Other

Submitted By: Erik Berda Email: erik.berda@unh.edu
Phone: (603) 862-1762

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STEM Degrees: 22 **STEM Participants:** 38

Major Goals: The research goal of this YIP proposal was to fold discreet synthetic polymer chains into architecturally defined 3-dimensional nanostructures. We aimed to accomplish this goal using a combination of permanent and reversible or dynamic intra-chain cross-linking interactions applied sequentially in dilute solution. In order to establish a structure-property relationship for these foldable polymer chains we had 3 research objectives:

1) evaluate different types of intra-chain cross-linking chemistry
2) study the effect of polymer architecture on single-chain folding behavior
3) investigate improved structural characterization methods for these folded structures.

Rationale behind this design:

Fundamentally controlling nanoscale structure and the ability to modulate behavior (e.g. structural, optical, thermal, sensing, etc.) is at the core of next generation DOD systems. Our approach offers foundational work for applying polymer-based systems to these advanced applications by mimicking the elegance of naturally occurring folded biopolymers. In analogy to Nature, we combined multiple intra-chain interactions to effect this single-chain folding process:

Permanent intra-chain covalent linkages provide thermal and chemical stability to these structures, resulting in robust nanoscale colloidal particles in the sub 20 nm size regime.

Dynamic or reversible intra-chain linkages impart adaptability to these structures, resulting in nanomaterials that can respond to external stimuli or environmental conditions.

Applying these interactions sequentially allowed us to mimic the protein folding energy landscape more closely than previously reported systems, resulting in more precisely folded and structurally compact nano objects.

Accomplishments: See attached PDF file.

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Training Opportunities: This work has been an outstanding educational platform. It combines small molecule and polymer synthesis and characterization with techniques from materials science and nanotechnology. Students are interested in and excited by this work, particularly the bioinspired and biomimetic design features. It is easy to recruit talented students for this project; generally, we have more applicants to join our group than we can accommodate. a total of 38 students directly received laboratory training from this award. When outreach activities are included, the number of students benefiting from this support is well over 100.

Several students, particularly graduate students, also benefited from professional development activities such as workshops and conferences, including ACS national meetings.

Additionally, for 3 years we were awarded HSAP and URAP summer internship support. This allowed us to train 3 undergraduates and 3 high school students beyond what we originally planned.

Results Dissemination: In total we published 17 Peer Reviewed Journal articles and Books Chapters that acknowledge support from this ARO award.

Additionally, the PI and members of his group gave several invited talks and contributed talks and posters that acknowledge support from this award (more than 40 in total).

In addition to these methods, the PI conducted outreach presentations and activities to "Project Smart," a summer science camp for high school students. These consisted of lecture and lab activities to introduce polymer science and DoD research and scholarship opportunities to these stuents.

Honors and Awards: 2017:
Gloria G. and Robert E. Lyle Professorship (UNH)
Pioneering Investigator (RSC Polymer Chemistry)
2015:
James D. Morrison Early Career Award (UNH)
Emerging Investigator (RSC Polymer Chemistry)

Protocol Activity Status:

Technology Transfer: Nothing to Report

PARTICIPANTS:

Participant Type: Graduate Student (research assistant)
Participant: Ruiwen Chen
Person Months Worked: 1.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Graduate Student (research assistant)
Participant: Elizabeth Bright
Person Months Worked: 1.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Graduate Student (research assistant)
Participant: Erinn Reville

Person Months Worked: 1.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Graduate Student (research assistant)
Participant: Christian Tooley
Person Months Worked: 3.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Graduate Student (research assistant)
Participant: Justin Cole
Person Months Worked: 3.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Graduate Student (research assistant)
Participant: Ashley Hanlon
Person Months Worked: 9.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Graduate Student (research assistant)
Participant: Kyle Rodriguez
Person Months Worked: 3.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Graduate Student (research assistant)
Participant: Peter Frank
Person Months Worked: 6.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Graduate Student (research assistant)

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as of 19-Apr-2018

Participant: Bryan Tuten
Person Months Worked: 9.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Graduate Student (research assistant)
Participant: Christopher Lyon
Person Months Worked: 9.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Graduate Student (research assistant)
Participant: Alka Prasher
Person Months Worked: 6.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Graduate Student (research assistant)
Participant: Jessica Dickinson
Person Months Worked: 3.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Graduate Student (research assistant)
Participant: Andrea Duke
Person Months Worked: 1.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Undergraduate Student
Participant: Odin Achorn
Person Months Worked: 3.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

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Participant Type: Undergraduate Student
Participant: Annika Taylor
Person Months Worked: 3.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Undergraduate Student
Participant: David Waste
Person Months Worked: 3.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Undergraduate Student
Participant: Ellen Hill
Person Months Worked: 2.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Undergraduate Student
Participant: Karen Richards
Person Months Worked: 6.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Undergraduate Student
Participant: Conor Loynd
Person Months Worked: 3.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Undergraduate Student
Participant: Rachel Olsen
Person Months Worked: 1.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

RPPR Final Report
as of 19-Apr-2018

Participant Type: Undergraduate Student
Participant: Robert Biro
Person Months Worked: 3.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Undergraduate Student
Participant: Ian Martin
Person Months Worked: 3.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Undergraduate Student
Participant: Jacob Lessard
Person Months Worked: 6.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Undergraduate Student
Participant: Mark Cashman
Person Months Worked: 3.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Undergraduate Student
Participant: Ashley Ellis
Person Months Worked: 2.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Undergraduate Student
Participant: Jen Chouinard
Person Months Worked: 3.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Undergraduate Student
Participant: Courtney Leo
Person Months Worked: 3.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Undergraduate Student
Participant: Katherine Buckley
Person Months Worked: 1.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Undergraduate Student
Participant: Sarah Benware
Person Months Worked: 3.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Undergraduate Student
Participant: Claudia Willis
Person Months Worked: 3.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Undergraduate Student
Participant: Joey Mancinelli
Person Months Worked: 3.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Undergraduate Student
Participant: Christoper Lasalle
Person Months Worked: 1.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N

Other Collaborators:

Participant Type: Undergraduate Student
Participant: Cynthia Gerber
Person Months Worked: 2.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Undergraduate Student
Participant: Nathan Shipely
Person Months Worked: 1.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Undergraduate Student
Participant: Erinn Reville
Person Months Worked: 3.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: High School Student
Participant: Isabelle Crawford-Eng
Person Months Worked: 2.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: High School Student
Participant: Gabriel Patenotte
Person Months Worked: 4.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: High School Student
Participant: Katie DeLong
Person Months Worked: 2.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:

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National Academy Member: N
Other Collaborators:

Participant Type: Undergraduate Student
Participant: Daniel Darcey
Person Months Worked: 1.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

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Article Title: Electroactive Polyurea Bearing Oligoaniline Pendants: A Viable Candidate for Corrosion Inhibiting Coatings.
Authors:
Keywords: oligoaniline, electroactive, anticorrosion, electrochromic
Abstract: In this study, a novel polyurea with oligoaniline pendants was prepared in a single step from readily accessible monomers. Fourier-transform infrared spectra (FTIR), nuclear magnetic resonance (NMR) and gel permeation chromatography (GPC) were employed to characterize the molecular structure of the obtained electroactive polyurea (EPU). Its spectroscopic properties and thermal stability were also detected and evaluated. The electrochemical activity of EPU was explored by cyclic voltammetry in 0.5 M H2SO4 confirming a surface controlled process. The spectrochronoamperometry was applied to investigate the electrochromic performance of EPU/ITO electrode, which exhibited good electrochromic properties with high contrast value, moderate switching times and satisfactory coloration efficiency. Furthermore, corrosive protection of the EPU coatings on the cold rolled steel (CRS) in 5 wt% NaCl solution were studied by tafel plots analysis and electrochemical impedance spectroscopy. The results i
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Volume: 0 Issue: 0 First Page #: 0
Date Submitted: Date Published:
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Article Title: A Brief User's Guide to Single-chain Nanoparticles
Authors:
Keywords: SCNP nanoparticles synthesis
Abstract: In this review we outline the various methods that have been explored to synthesize architecturally defined nanoparticles from dicrete polymer chains, summarized the methods of characterization that are required to prove their formation and probe their morphology, and introduced a number of potential applications that are being explored currently. Given the small size of the nanostructures produced by these methods and the relative ease with which they can be tailored to specific end use applications it is likely such efforts will intensify in the coming years. So far, simple chemistry has been utilized and high-level characterization and modeling studies have been applied to understand the process by which these particles form and how they behave, both in the bulk and in solution. Although impossible to predict where this work will lead, we hope this "user's guide" will prove useful to the community as research on single-chain nanoparticles continues to evolve.

Acknowledged Federal Support:

Erik Berda, University of New Hampshire, Durham NH 03824
Agreement Number: W1911 NF-14-1-0177

Research accomplishments and scholarly productivity

Our laboratory is well poised to make a contribution in this area. Propelled by an ARO YIP award, we pioneered the synthesis of soft nanomaterials fabricated through the controlled collapse or folding of single polymer chains under appropriate conditions. These materials, termed single-chain nanoparticles (SCNP), can be envisioned as rudimentary synthetic mimics of protein tertiary structure. Our group, now regarded among the world leaders in this emergent branch of polymer research, ***coauthored 17 publications with acknowledging support under this award***, several of which are high profile and are receiving more than 10 citations per year, and 2 of which have been featured on the cover of leading journals.

Background Information

Proteins epitomize the structure/function relationship that underlies our understanding of polymeric materials. The polymer community well understands that protein primary structure dictates secondary and tertiary folded structures, which in turn dictate the complex functions and behaviors exhibited by these materials. Protein-inspired polymeric materials, as part of a larger category of bioinspired materials, represent a frontier area in polymer chemistry because of their potential to contribute to a number of current technological grand challenges.

Our research group has been highly active in this area over the past several years. Our primary research focus remains centered on so-called **s**ingle-**c**hain **n**ano**p**articles (SCNP), a class of architecturally defined 3 dimensional nanomaterials that approach the protein size regime (sub 20 nm). SCNP are generally synthesized through the intramolecular cross-linking of single, linear polymer chains in ultra-dilute solution. Our group and others demonstrated that this general process, akin to protein folding in a rudimentary way, is applicable to myriad polymer backbones, functionalities, and intra-chain linking motifs. Figure 1 depicts the basic SCNP fabrication protocol schematically.

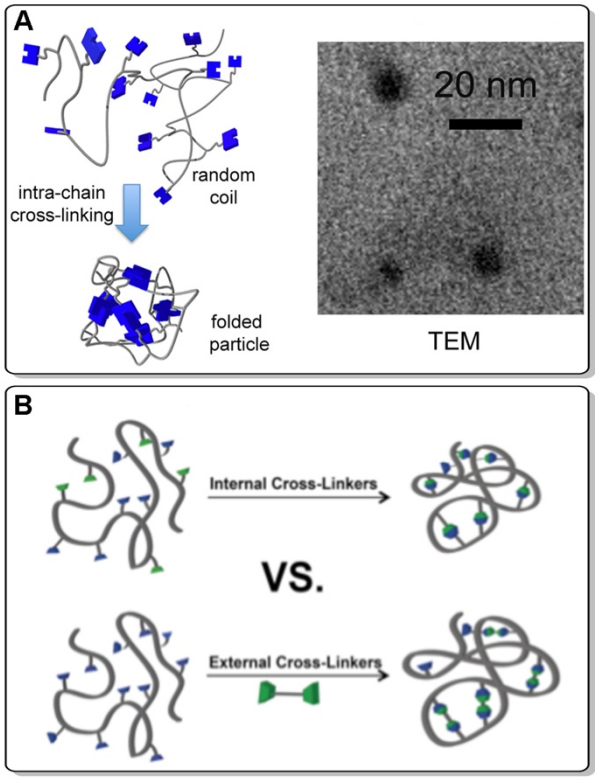

Figure 1: Single-chain nanoparticle (SCNP) synthesis. (A) basic SCNP fabrication scheme and TEM image of SCNP. (B) the two main strategies for making SCNP involve building all necessary chemistry into the parent polymer (top) or using an externally added cross-linking agent (bottom).

Our major contributions to this area relate to the basic design principles required to affect this process as well and improved methods to characterize SCNP formation. This section provides a brief summary of the important discoveries and advancements our group has made in SCNP recently.

We pioneered better methods for characterizing SCNP. our group was the first to show that combining SEC-MALS and SEC-Viscometry can provide quantitative data about SCNP fabrication by monitoring changes in size, conformation, and absolute molecular weight simultaneously. We have verified this method with other techniques such as DOSY, NMR, and DLS, and overall, these methods are well-received by the community and have been adopted by others groups across the globe.

Erik Berda, University of New Hampshire, Durham NH 03824
Agreement Number: W1911 NF-14-1-0177

We significantly broadened the scope of chemical reactions and polymer structures that can be used to prepare SCNP. Our publications in this area are distinguished by the rich diversity and sophisticated chemistry that we have investigated. First and foremost, we are synthetic chemists. Our willingness to attempt challenging and sophisticated chemistry has enabled us to build a vast palette of polymerization and cross-linking strategies for synthesizing SCNP.

We delineated the basic structure-property relationships for several SCNP design elements. Our results demonstrate the influence of varying polymer architectures, functional group placements, and functional comonomer incorporations. For example, one of the most interesting outcomes of this work was our recent finding that methods using internally and externally added cross-linkers, as illustrated in Figure 1b, result in distinct SCNP behaviors.

We learned to circumvent the ultra-high dilution problem. One of the drawbacks in traditional SCNP syntheses is the requirement for ultra-high dilution to prevent chain-chain coupling and multi-chain nanoparticle formation. We discovered that it is possible to overcome this problem with a continuous addition strategy by carefully choosing the intra-chain cross-linking chemistry. This process works for both reaction types shown in Figure 1b. We are in the process of extending this strategy to continuous flow reactors.

We demonstrated that combining several intra-chain cross-linking motifs is feasible. In fact, in our work we have seen interesting results when combining multiple intra-chain linking strategies. Our most encouraging results show that combining covalent and non-covalent intramolecular interactions, just as Nature does, is a very effective chain folding strategy. This work is highlighted further below.

Key Discoveries

Step-growth like cross-linking to install structural and functional segments.

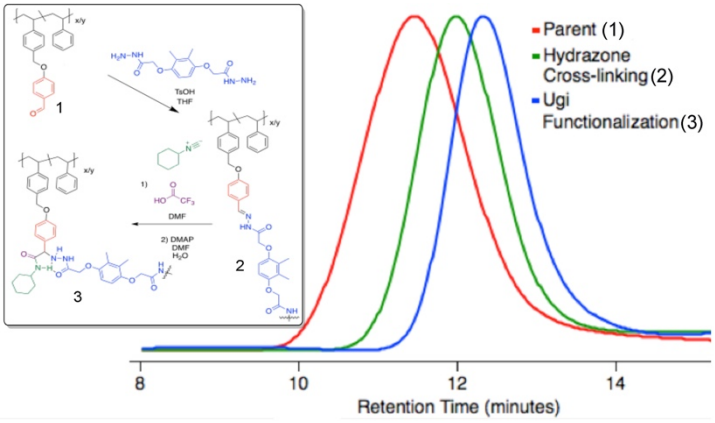

Figure 2: SCNP featuring protein-like secondary structure are accessible via intra-chain multicomponent reactions.

Figure 2 shows the synthetic scheme (left) and the GPC data (right) for SCNP fabrication using an isocyanide-based multicomponent reaction (IMCR). The intra-chain IMCR introduces both covalent and noncovalent interactions iteratively, evident in the two successive shifts to longer retention time in the GPC traces. These shifts are due to two reductions in hydrodynamic volume induced by each compaction. In this instance, the final compaction step introduces a protein-like β-turn motif. The highlight of this work is the synergistic interplay between both covalent and noncovalent linkages, confirmed through a variety of experiments and characterization strategies. The behavior of this system is strongly dependent on solvent, temperature, and chemical stimulus, paving the way toward truly adaptive single-chain nanomaterials. Although synthetically challenging, this data represents a major advancement toward true protein-like structure in SCNP.

Erik Berda, University of New Hampshire, Durham NH 03824
Agreement Number: W1911 NF-14-1-0177

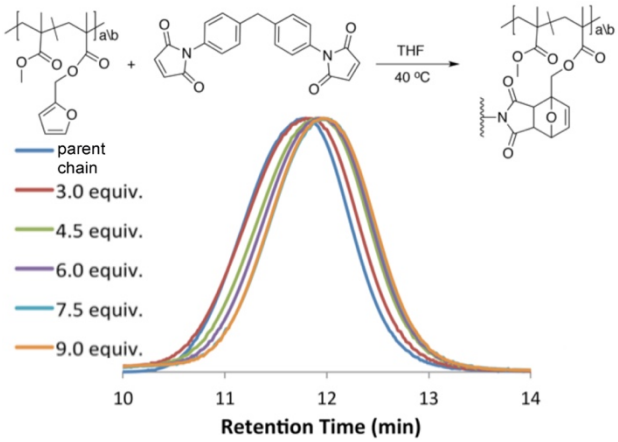

Figure 3: Effect of external cross-linker concentration on SCNP formation.

While simple in concept, the process conditions for intra-chain couplings that follow a step-growth like reaction with externally added cross-linkers are rather complicated. Figure 3 shows the effect of cross-linker concentration on an SCNP synthesis. We have found that it is important to use an excess of externally added cross-linker relative to the stoichiometric requirements. Although this seems somewhat counterintuitive at first, the excess ensures that the local concentration of cross-linker matches the local concentration of reactive units on the parent polymer chain.

The structure of the externally added cross-linker itself is very important. Figure 4 highlights the differences between two types of bifunctional cross-linkers and a trifunctional cross-linker. The data clearly reveal that more flexibility and higher cross-linking density are important for efficient compaction. Our results, as well as results from others, confirm that long range intramolecular cross-links improve the degree of compaction, this is exactly what is provided with highly functional, more flexible cross-linkers.

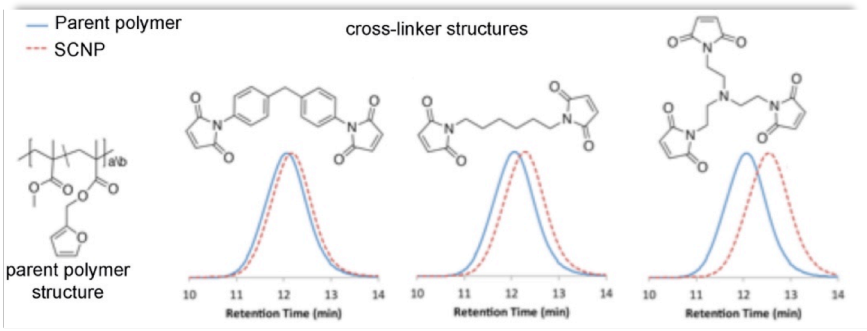

Figure 4: Effect of cross-linker structure on SCNP formation.

Intra-chain polymerization of pendant monomer units.

Figure 5 (next page) shows a schematic representation of two routes we have successfully used to make SCNP out of polymer chains decorated with polymerizable pendant groups. This method follows a chain growth-like process and has proven useful over a fairly broad scope. Here, pendant monomer units “knit” a linear chain into an architecturally defined nanostructure after external initiation. Both direct polymerization of the pendant groups, as well as copolymerization with an externally added monomer are feasible. We have shown that well-defined sequences are possible using an alternating intramolecular polymerization strategy and that functional units can be introduced to SCNP in this fashion.

Erik Berda, University of New Hampshire, Durham NH 03824
Agreement Number: W1911 NF-14-1-0177

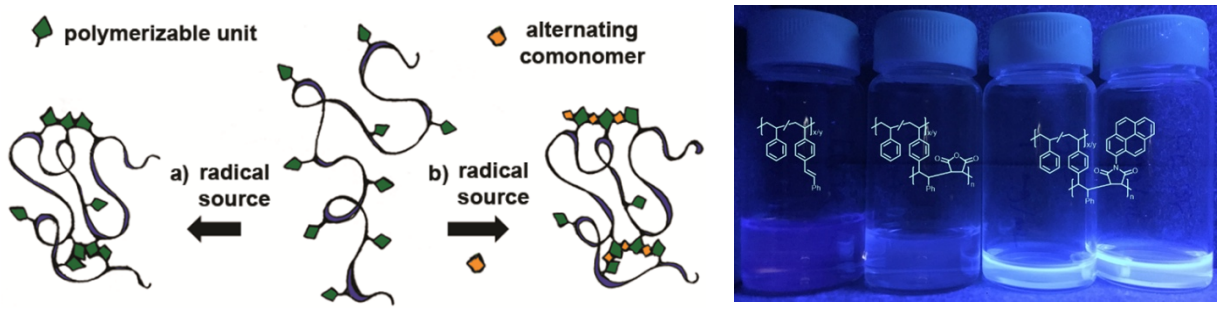

Figure 5: Schematic representation of SCNP synthesis via intra-chain polymerization (left), and an example of fluorescent nanoparticles made using this chemistry (right).

Intra-chain atom transfer radical coupling

One of the current pressing challenges in SCNP is scale up to access useful quantities of material while at the same time producing nanostructures that have functional capabilities. We have discovered a very convenient route to such SCNP using intra-chain atom transfer radical coupling (ATRC) as a cross-linking strategy. Here, we synthesize polymers that are functionalized with ATRP type initiators. In the presence of an ATRP catalyst but in the absence of monomer, only termination events are possible. By correctly tuning the ATRC active site structure, we have learned to promote bimolecular radical coupling as the primary termination event and used this as a way to make well-defined SCNP. These initial results are summarized in Figure 13. TEM images confirm spherical particles in the sub 10 nm size regime (which approaches the protein size regime). We also demonstrated that producing gram quantities of SCNP is feasible by this method.

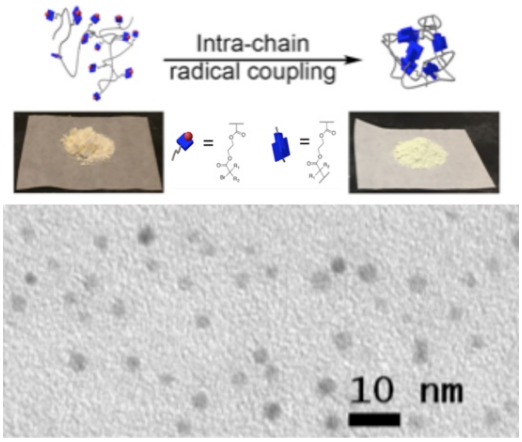

Figure 6: (Top) schematic representation of SCNP made by intra-chain ATRC and photograph of isolated, gram quantities of SCNP; (bottom) TEM image of SCNP made by this chemistry.

In summary, we successfully accomplished all of the major goals of this research. These results have been disseminated in top polymer journals, and 38 students benefited from this award, including 8 newly minted PhDs.