NC STATE UNIVERSITY
Wilson College of Textiles

Textile Engineering, Chemistry and Science Department

Engineering the Molecular Ladder: Designing Stronger, Sustainable Carbon Fiber for Global Innovation

Xiaorui Guo and Januka Budhathoki-Uprety*

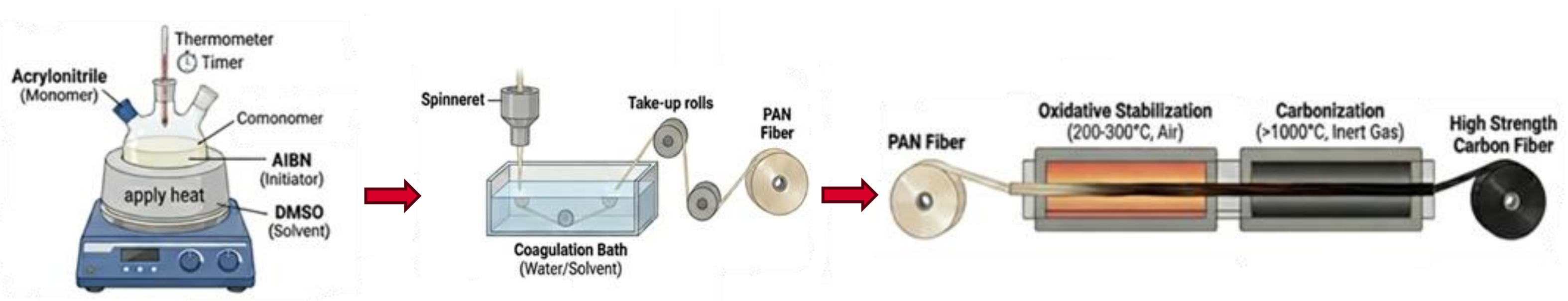
Background

- **Context:** Carbon fiber (CF) is a high-performance material with low density, ultra-high tensile strength and high modulus, critical for advanced materials in the aerospace, automobiles and defense sectors.
- **Challenge:** Reducing dependency on petroleum feedstocks; Improving properties of CFs.
- **Objective:** Making stronger, sustainable CFs through polymer precursors design and synthesis from renewable resources

Introduction

High-Performance Applications

Manufacturing Process

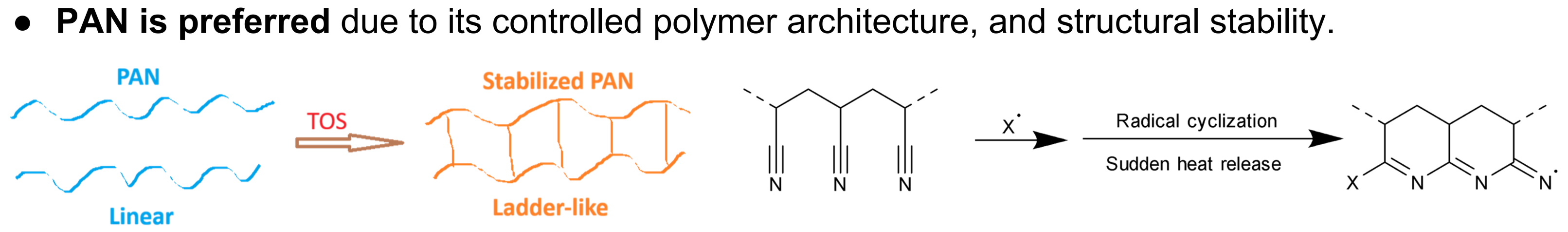


Polymer | Precursor Fiber | Carbon Fiber

Precursor production represents **the largest financial bottleneck** in carbon fiber manufacturing, accounting for ~51% of total costs and driving critical interest in precursor chemistry.

Polyacrylonitrile (PAN) as a Primary Precursor

- **Over 90%** of commercial carbon fibers are derived PAN-based precursors. The other precursors are pitch, lignin and cellulosic materials.
- **PAN is preferred** due to its controlled polymer architecture, and structural stability.



Limitation

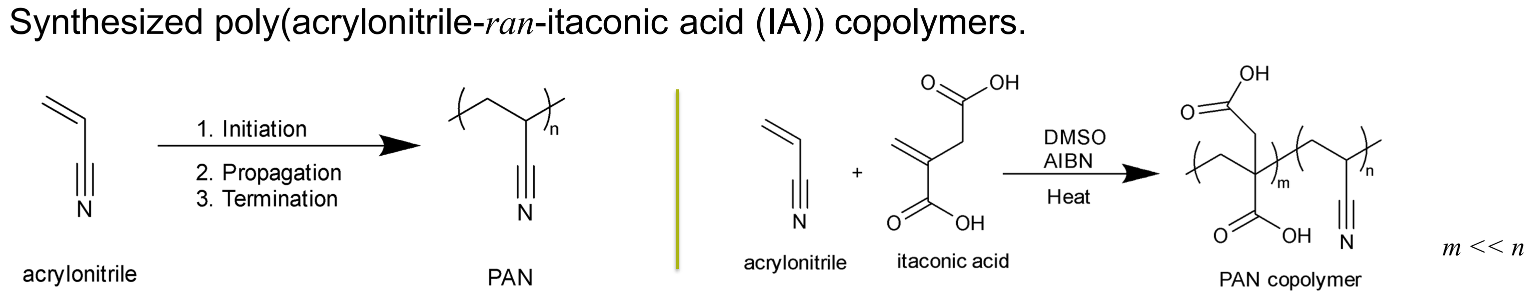
Thermal oxidative stabilization (TOS) in PAN homopolymer is a free radical cyclization process causing rapid heat release and structural defects.

Our Approach

- Synthesize copolymers, verify their structures and evaluate their thermal profiles to identify the optimal formulation for **sustainable**, high-performance CF manufacturing.
- Select comonomers to improve TOS.
- Comonomers shift the reaction to a controlled ionic pathway that ensures smoother cyclization and reduces defects.

Polymer synthesis

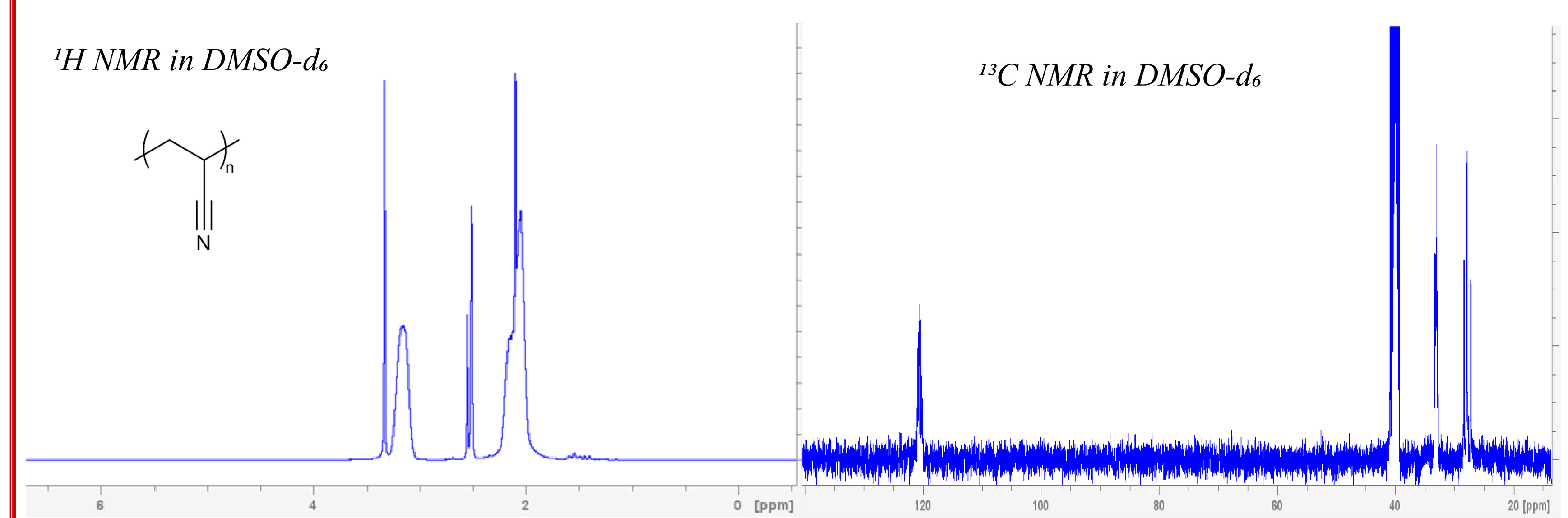
- Chain-growth polymerization of acrylonitrile (AN) and comonomers.
- Synthesized poly(acrylonitrile-*ran*-itaconic acid (IA)) copolymers.



Results

Nuclear Magnetic Resonance (NMR)

- Both ¹H and ¹³C NMR confirmed the polymer structures.
- ¹H NMR specifically quantified the actual comonomer concentration.

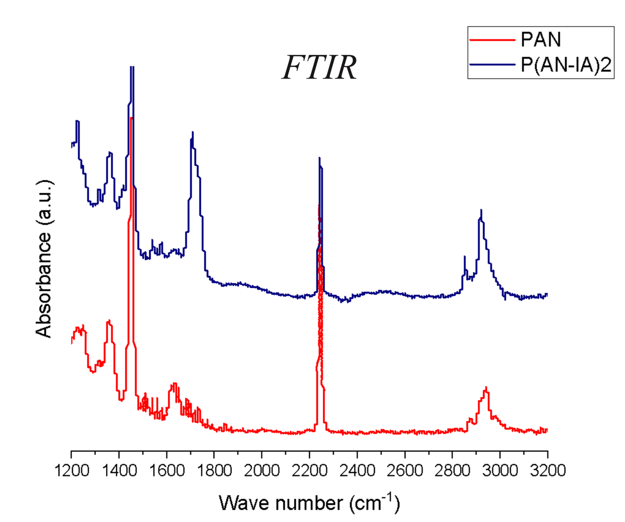


NMR analysis confirmed the expected structure of the PAN homopolymer, characterized by two distinct proton and three carbon environments. Additionally, ¹H NMR integration verified that the incorporated IA concentration closely matches the initial monomer feed ratio (See the table).

Entry	AN feed (mol%)	IA feed (mol%)	IA actual (mol%)	Conversion (%)
P(AN-IA)1	99	1	**1.06**	70.0
P(AN-IA)2	98	2	**2.00**	70.0

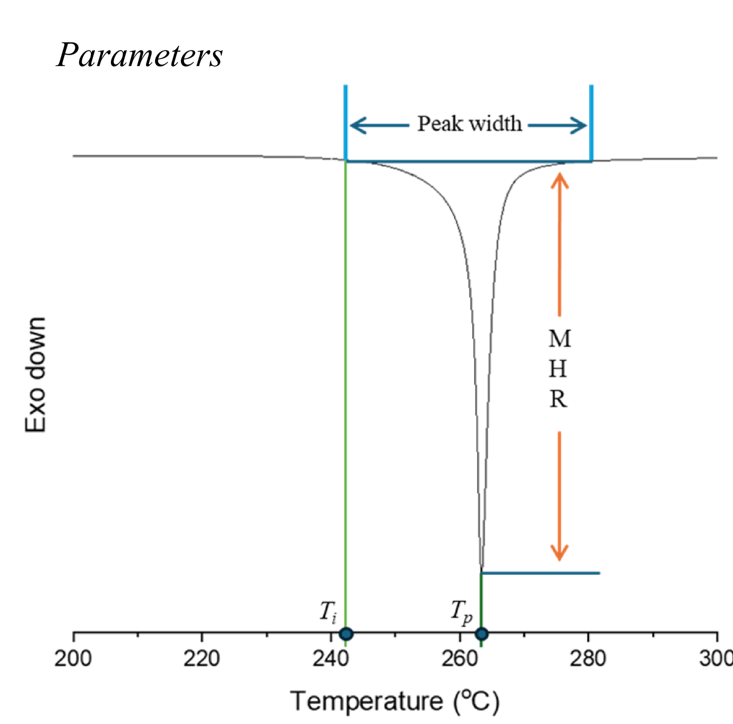
Infrared spectroscopy (FTIR)

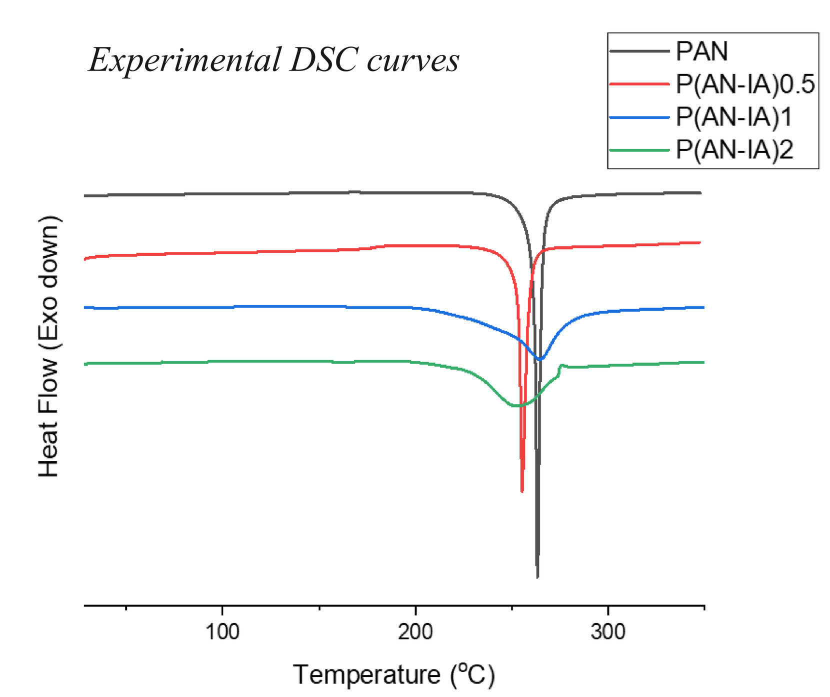
FTIR confirmed the functionalities in the polymers. The nitrile group exhibits a strong, sharp peak at 2240 cm^{-1}, while the carbonyl group in IA shows a peak near 1700 cm^{-1}.



Differential Scanning Calorimetry (DSC)

We use DSC to track the thermal transitions of PAN. Heating PAN triggers a highly exothermic cyclization (forming a stable ladder structure), resulting in a distinct exothermic peak on the DSC curve.





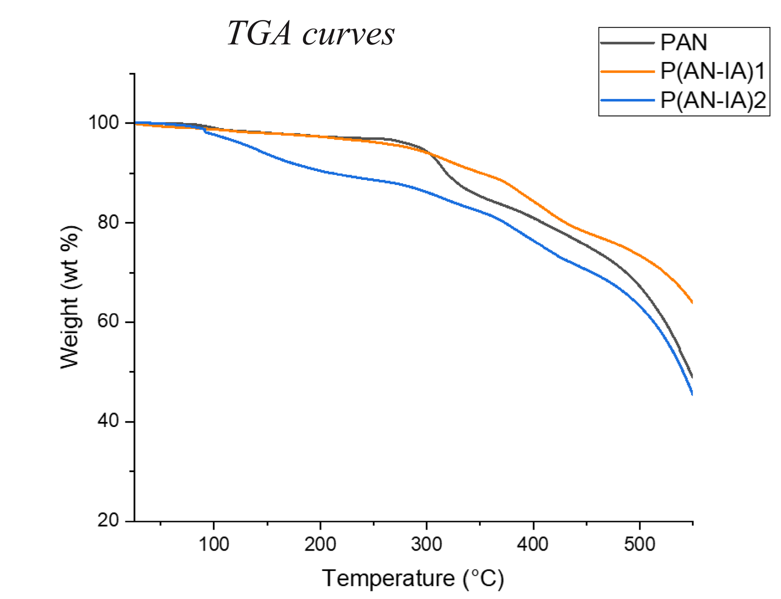
Polymer	T_i	T_p	ΔT	MHR*
PAN	238	265	36	-12.288
P(AN-IA)0.5	225	260	50	-21.869
P(AN-IA)1	188	265	152	-2.725
P(AN-IA)2	187	252	153	-2.325

MHR represents Maximum Heat Release (W/g).

Results

Thermogravimetric Analysis (TGA)

We use TGA to measure continuous weight loss during heating. For CF precursors, the remaining weight at **450°C**—just before carbon oxidizes—provides an estimate of the final carbon char yield.



Polymer	Char yield at 450 °C
PAN	75.45
P(AN-IA)1	78.10
P(AN-IA)2	70.56

Making Sustainable CF

- Synthesized copolymers using AN produced **from CO_2-negative monomers** (in collaboration with **Mars Materials Inc.**).
- Ongoing fiber spinning and characterizations (in collaboration with **Dr. Ericka Ford** and her team).
- Future direction: Conversion into high-performance and **sustainable carbon fibers**.

Conclusions

- **Optimized Thermal Control:** Incorporating itaconic acid (IA) effectively lowers the initiation temperature and moderates exothermic heat release during cyclization. This stabilizing effect scales positively from 0.5 to 2.0 mol% IA.
- **Maintained Efficiency:** The PAN copolymers achieve comparable char yield to literature.
- **Sustainability:** Demonstrated polymer synthesis from sustainable AN monomer

Acknowledgments

NSF
TEXTILE INNOVATION ENGINE

- TECS Analytical Services Lab
- METRIC, NC State
- Dr. Ericka Ford, Daniel Zhou
- Mars Materials Inc.
- Budhathoki Research Group

References

Mainka et al. "Lignin – an Alternative Precursor for Sustainable and Cost-Effective Automotive Carbon Fiber." *Journal of Materials Research and Technology* 4, no. 3 (2015): 283–96.

Ju et al "Effect of Comonomer Structure on the Stabilization and Spinnability of Polyacrylonitrile Copolymers." *Carbon* 54 (April 2013): 323–35.

Frank et al. "Carbon Fibers: Precursor Systems, Processing, Structure, and Properties." *Angewandte Chemie International Edition* 53, no. 21 (2014): 5262–98.

Joo et al. "Recent Advances in Activated Carbon Fibers for Pollutant Removal." *Carbon Letters* 35, no. 1 (2025): 21–44.