BETO 2021 Peer Review
Renewable Carbon Fiber Consortium

WBS 2.3.4.102

March 10, 2021

Performance-Advantaged Bioproducts, Bioprocessing Separations, and Plastics

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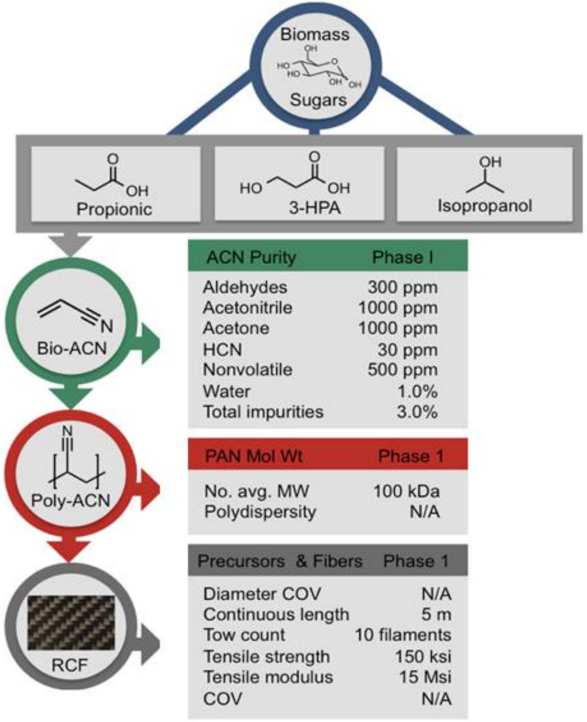
Project Background – Prior DOE Funded Work

Prior Project: 3-year, multi-institution competitive award to develop pathway to produce renewable carbon fiber

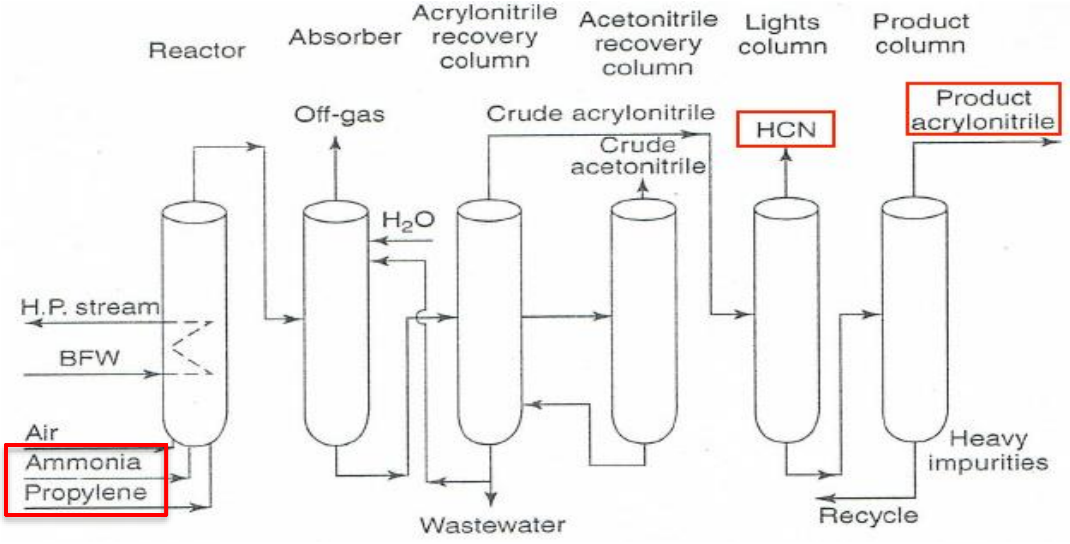
Goal: Develop novel bio-route to acrylonitrile (ACN) and leverage conventional carbon fiber production

Hypothesis: Starting with sugars will enable cheaper/simpler route to ACN relative to conventional propylene ammoxidation

Results: Novel “nitrilation” of 3-HPA resulted in cheaper, simpler, higher yield and more environmentally friendly route to ACN than conventional and resulting polymer and carbon fiber had properties completely consistent with conventional



Project Background – Conventional ACN Process

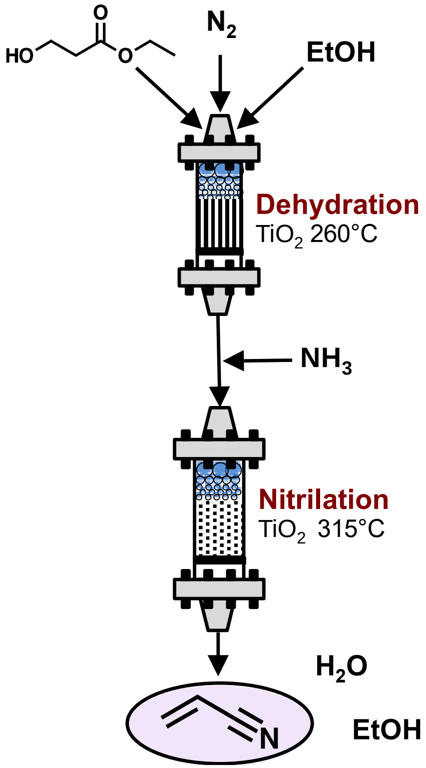


- Acrylonitrile (ACN) best precursor for high quality carbon fiber
- But ACN price is too high and too volatile leading to cost challenges for carbon fiber
- Because propylene ammoxidation is a complex, exothermic reaction with expensive catalysts, toxic by-products (hydrogen cyanide) and relatively low (~70%) yields

Project Background – Novel Nitrilation Process

Ester Nitrilation:

- Inspired by earlier reports on acid and ester conversion to nitriles
- 3-HPA () selected as substrate given ease of production from sugars and facile dehydration to acrylate product
- Esterification () used as a separations technique and reaction facilitator



Advantages relative to propylene ammoxidation:

- Simpler catalyst (TiO2) & higher yields (~95%)
- Endothermic reaction (easier to control)
- Non-toxic byproducts (water and recyclable alcohol)
- Not only biobased, but lower overall cost (~$0.50/lb)

Karp et al., *Science,* Dec 2017, Renewable Acrylonitrile Production
R&D 100 Award Winner - 2018

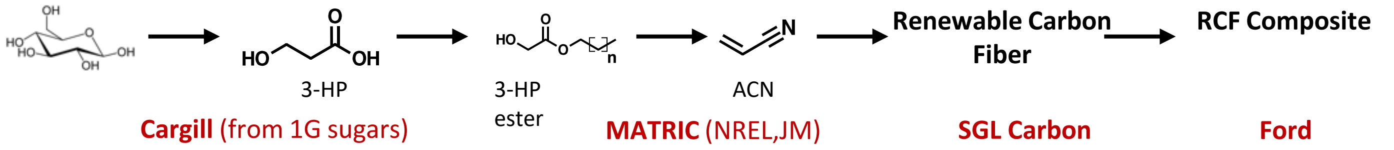
Current Project Overview

Lead: NREL

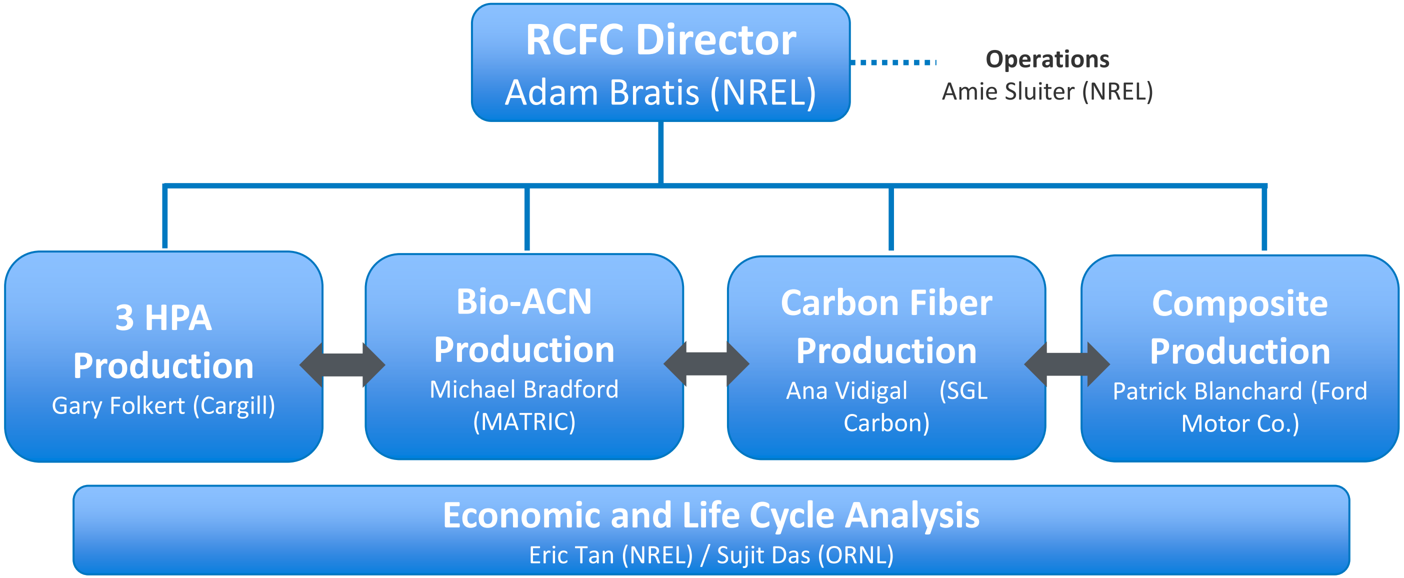
Partners: Cargill, Johnson Matthey, MATRIC, SGL Carbon, Ford, ORNL

Objective: Demonstrate viability of scale-up of nitrilation technology and renewable carbon fiber production and application

- Production of commercially relevant 3-HPA fermentation broth
- Demonstration of scalability of esterification/nitrilation technology and production of ~50kg of bio-acrylonitrile that meets commercial specs
- Production of carbon fiber from bio-ACN that meets commercial specs
- Production of composite that meets conventional carbon fiber application properties (vehicle structural material)
- Demonstrate economic (>50%) and environmental (>60% GHG) improvements relative to conventional process



Project Management



Keys to Project Management Success:

- Strong and relevant company expertise, capability and history for each step
- Managing the interfaces (e.g. expectations of outputs/inputs and roles)
- Good multi-institutional collaboration (e.g. open knowledge sharing)
- Working with partners to schedule within their business operational windows
- Monthly reporting and feedback loop with DOE

Project Approach

General Approach: 2 Stages (pre-go/no-go decision and post-go/no-go decision)

- 1st stage is for MATRIC to verify and reproduce NREL's results on esterification, nitrilation and purification and show a feasible pathway to pilot scale production of 50kg bio-ACN using 3-HPA from Cargill's commercially relevant process.
  – ($1.5M; ~20% cost share)

- 2nd stage is for larger scale production (50 kg bio-ACN) meeting a series of specs and yield targets at each step from 3-HPA (Cargill) through bio-ACN (MATRIC), Carbon Fiber (SGL Carbon) and Composite (Ford). – ($3.8M; ~20% cost share)

GNG Milestone: Deliver a report describing bench scale work performed on esterification, nitrilation, and purification, details on the success or failure of each process step, and critical parameters and roadblocks to pilot scale production of bio-ACN along with a recommendation to continue or terminate pilot scale work. (MATRIC)

Project Progress

Cargill

Stage 1 (pre GNG):

- Produce and deliver 40kg of 3-HPA fermentation broth to MATRIC (completed)

Stage 2 (post GNG):

- Produce and deliver >400kg of 3-HPA fermentation broth to MATRIC (05/2021 pending GNG decision approval)

Performance and Quality Specifications

- >80g/L titer at all production scales (completed)
- Concentrated to 20% 3HP monomer with turbidity of <25 NTU (completed)

Project Progress

Stage 1 (pre GNG):

- Esterification – >90% purity and 75% yield of methyl acrylate and/or methyl 2-hydroxypropionate; scalable pathway to 200kg ester production (85% purity, 68% yield; close)
- Nitrilation - >85% yield of bio-ACN and scalable pathway to 75kg production (86% yield, completed)
- Purification - >80% bio-ACN purity with scalable process to 75kg production (>95% purity; completed)

Stage 2 (post GNG):

- Produce and deliver >75kg of bio-ACN to SGL that meets commercial ACN specs

Performance and Quality Specifications

- >50% sugar to purified ACN yield at all production scales (completed)

Project Progress

SGL GROUP
THE CARBON COMPANY

Stage 1 (pre GNG):

- n/a

Stage 2 (post GNG):

- Polymerize bio-ACN to polyacrylonitrile (PAN), spin to PAN fiber and carbonize to carbon fiber and deliver to Ford for composite production

Performance and Quality Specifications

- PAN: MWw of >200,000 Da and polydispersity index less than 5.0
- PAN fiber: >100m continuous length and <10% COV fiber diameter
- Carbon fiber: 1.7 GPa tensile strength, 170 GPa tensile modulus, and maximum 10% COV

Project Progress

Stage 1 (pre GNG):

- n/a

Stage 2 (post GNG):

- Manufacture composite panels and perform mechanical and performance testing

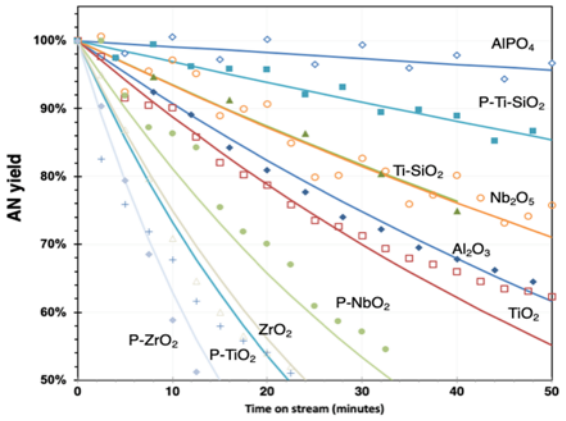
Performance and Quality Specifications

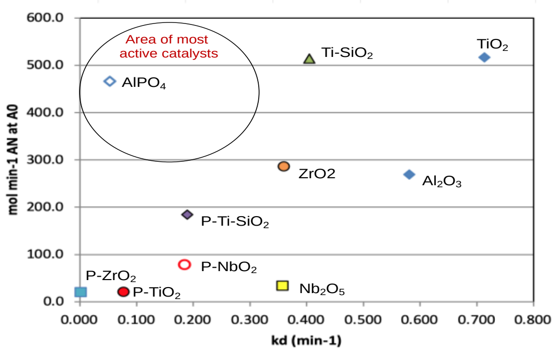
- Demonstrate performance of flat panel tests that meet mechanical metrics of 300 mPA tensile strength and 30 GPa tensile modulus

Project Progress

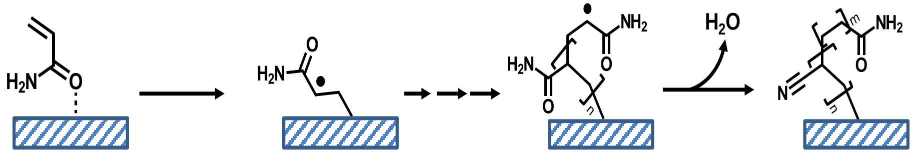
Recent work has improved catalysts

- $AlPO_4$ is active ~10x longer before a regeneration cycle is needed compared to TiO_2
- Hypothesis: Increased distance between nearest neighbor acid sites prevents acrylates from radical polymerization and carbon laydown
- Increased partial pressure of NH_3 decreases deactivation rate.
- Hypothesis: Increased NH_3 on surface decreases probability of acrylate polymerization of nearest neighbor adsorbates





Hypothesized deactivation mechanism



Project Progress - Summary

Cargill

- Has shown it can meet performance and quality specs for 3-HPA production
- Has delivered Stage I (~40kg) of 3-HPA in the form of concentrated broth to MATRIC
- Has equipment at the ready for Stage 2 production run in May

MATRIC

- Has already achieved nitrilation and purification targets, close to achieving esterification targets
- Has identified achievable pathway to pilot scale production of all steps

JM

- Has delivered industrially relevant supported, high performing catalyst to MATRIC for Stage I experiments and is at the ready to deliver more for Stage 2 if we pass GNG

SGL GROUP / Ford

- Has verbally committed to Stage 2 timeline and has relevant equipment identified

Scale Up Approach for Stage II

Cargill
- Generate 3-HP from 1st generation sugars with existing strain
- Demonstrate 3-HP productivity
- Produce and deliver bio-based 3-HP

MATRIC (Mid-Atlantic Technology, Research & Innovation Center; Market-Driven Innovation)
- Receive 3-HP from Cargill and produce bio-ACN
- Demonstrate total conversion to bio-ACN of >50% relative to theoretical
- Purify, stabilize and ship bio-ACN to FISIPE for carbon fiber production

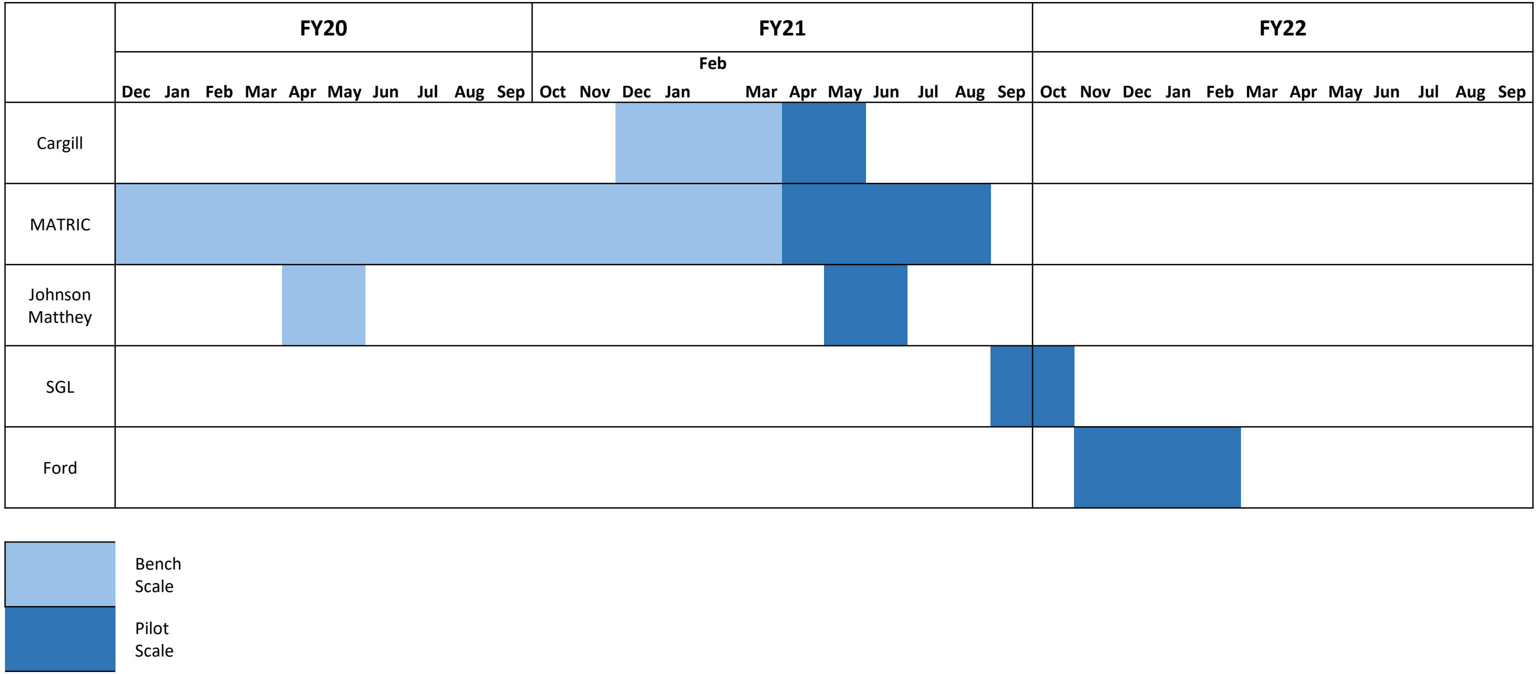
JM
- Provide catalyst to MATRIC

SGL GROUP (THE CARBON COMPANY)
- Receive bio-ACN from MATRIC
- Produce polyacrylonitrile
- Produce renewable carbon fibers (RCF)
- Ship carbon fiber spools to Ford

Ford
- Manufacture RCF composites and test relative to conventional CF composites
- Manufacture an RCF automotive component
- Demonstrate tensile strength and modulus from RCF panel

NREL (Transforming ENERGY)
- Gather data and validate assumptions across each process.
- Produce final report detailing the process design for meeting economic and sustainability metrics

Current Schedule



Original vs Updated Schedule

- GNG shifted 3 months (Dec 31 to Mar 31) due to workplace impacts of COVID-19
- Stage 2 plans in place with all 4 partners to accommodate 3-month shift

Project Impact

Carbon Fiber Properties:

- 5x stronger and 2x stiffer than steel
- 70% lighter weight (vs steel)
- 40% lighter weight (vs aluminum)
- 15% lighter weight (vs fiberglass)
- High cost limiting its widespread use

Implications:

- Existing markets limited to very high performance/high-cost applications (e.g. sporting goods, spacecraft, etc)
- Cost reduction could expand market significantly within everyday transportation sector leading to massive fuel economy implications from lightweighting

Market Trends

Product
- Gasoline/ethanol demand decreasing, diesel demand steady
- Increasing demand for aviation and marine fuel
- Demand for higher-performance products
- Increasing demand for renewable/recyclable materials

Feedstock
- Sustained low oil prices
- Decreasing cost of renewable electricity
- Sustainable waste management
- Expanding availability of green H_2
- Closing the carbon cycle

Capital
- Risk of greenfield investments
- Challenges and costs of biorefinery start-up
- Availability of depreciated and underutilized capital equipment

Social Responsibility
- Carbon intensity reduction
- Access to clean air and water
- Environmental equity

NREL's Bioenergy Program Is Enabling a Sustainable Energy Future by Responding to Key Market Needs

Value Proposition

- Provides proof of concept data for interested investors around scalability and applicability of nitrilation technology under market relevant conditions

Key Differentiators

- Cheaper, higher yielding, simpler and more environmentally friendly route to commercially equivalent acrylonitrile
- Fundamental reason for success is ease of biology/chemistry from sugars relative to petroleum
- Novelty was deploying hybrid biology/ chemistry approach to capitalize on that

Quad Chart Overview (2.3.4.102)

Timeline
- October 2019 - Start
- September 2022 - Finish

	Total Project	Funded to date
DOE Funding	Stage 1 - $1.5M Stage 2 - $3.8M *(~20% additional industry cost share*	$2.7M from DOE, remainder to be funded (or recovered) after GNG

Project Partners (NREL lead)
- Cargill
- MATRIC
- Johnson Matthey
- SGL Carbon
- Ford Motor Co.
- ORNL

Barriers addressed
- Ct-K. Developing Methods for Co-product Production
- ADO-D. Technical Risk of Scaling

Project Goal
Demonstrate cost competitive, commercially relevant, integrated scale-up of renewable carbon fiber through a bio-acrylonitrile intermediate.

End of Project Milestone
Final report demonstrating >50kg production of bio-acrylonitrile at <$1/lb with 50% reduction in GHG emissions along with subsequent renewable carbon fiber production with performance properties identical to conventional.

Funding Mechanism
AOP Funded Project for integration and scale-up

Building on a 3-year competitively awarded project that developed bench scale technology

Project Summary

Progress/Outcomes:

- Novel pathway to bio-ACN demonstrated w/commercial partners
- Cost & Performance benefits vs conventional
- Partners & full pathway from sugars to CF composites identified and committed

Impact:

- Generation of "touch and feel" volumes of bio-ACN and RCF to peak interest of end users
- Demonstration at scale, by commercial entities, gives potential bio-ACN producers proof of concept
- Affordable Carbon Fiber could lightweight structural materials in many sectors w/huge GHG and efficiency benefits

Next Steps:

- Perform Stage II at pilot scale
- Identify commercial partner and transfer/license nitrilation technology for market impact

Q&A

www.nrel.gov

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NREL Transforming ENERGY

Additional Slides

Previous Reviewers' Comments

Comments from 2019 Peer Review as FOA (bench scale) project ended and AOP project (integration and scale-up) was being laid out

"The Renewable Carbon Fiber Consortium is a great example of science being translated to commercial use. It appears that the team has moved to precommercial scale and testing, and presumably has the economics to make it work. What is presented is an excellent example of good science and good application following a well-laid-out path to a potentially important product. The weaknesses are minimal, in that the route to bio-ACN has been demonstrated, and initial work in scale-up is also showing success. The project has a clearly defined goal and is therefore directly understandable to a wide audience (i.e., "we will make cheap carbon fiber"). A real strength of the project is its identification of the key members of the team needed to carry out each of the steps in production and evaluation. Overall, all the steps, partners, and planned work is exactly what is needed to move this program to the next phase. All important questions are being answered and all challenges have appropriate plans in place to make it work. The handoff from one partner to another is really well laid out. They have experts on each step in place. Bigger picture, this is a really nice organization addressing exactly the needs necessary to prove out the overall approach. Good science has led to good applications that attract industry and offer opportunities for commercial deployment.

As the effort proceeds, the program will need to continue their close organization of a number of partners to make sure that problems with one do not impact the overall plan and schedule. It would be helpful to have more detail on how the partners were vetted. For example, it is interesting (and surprising) that the PIs are using an external producer of their carbon fiber, given the availability of the carbon fiber manufacturing facility at ORNL. Finally, catalyst deactivation seems important, and the team has identified it as a key issue. Getting a sense of whether there might be multiple solutions to the issue would also be useful to know. This could be a showstopper, so some insight as to what's going on would be helpful."

- Using Fisipe/SGL Carbon for CF production (vs ORNL) because size, scale and continuous processing matched perfectly for amount of material being generated
- Starting to do work to extend lifetime of catalyst and address longevity issues (NREL/JM) - ~10x improvement with $AlPO_4$ vs TiO_2

Publications, Patents, Presentations, Awards, and Commercialization

Karp et al., *Science,* Dec 2017,
Renewable Acrylonitrile Production

R&D 100 Award Winner - 2018