U.S. DEPARTMENT OF ENERGY | Energy Efficiency & Renewable Energy

The Engineering of Catalyst Scale Up

WBS # 3.2.1.1

U.S. Department of Energy (DOE)

Bioenergy Technologies Office (BETO)

2019 Project Peer Review

Frederick Baddour – NREL

Kris Pupek – ANL

Goal Statement and Outcomes

Project Goal – Develop *a flexible, engineering-scale catalyst synthesis capability* to produce ***scalable and cost effective*** next-generation biomass conversion catalysts and mitigate commercialization risk by ***enabling large-scale performance evaluation***

Project Outputs and Outcomes

– An ***industrially guided*** catalyst scale-up capability

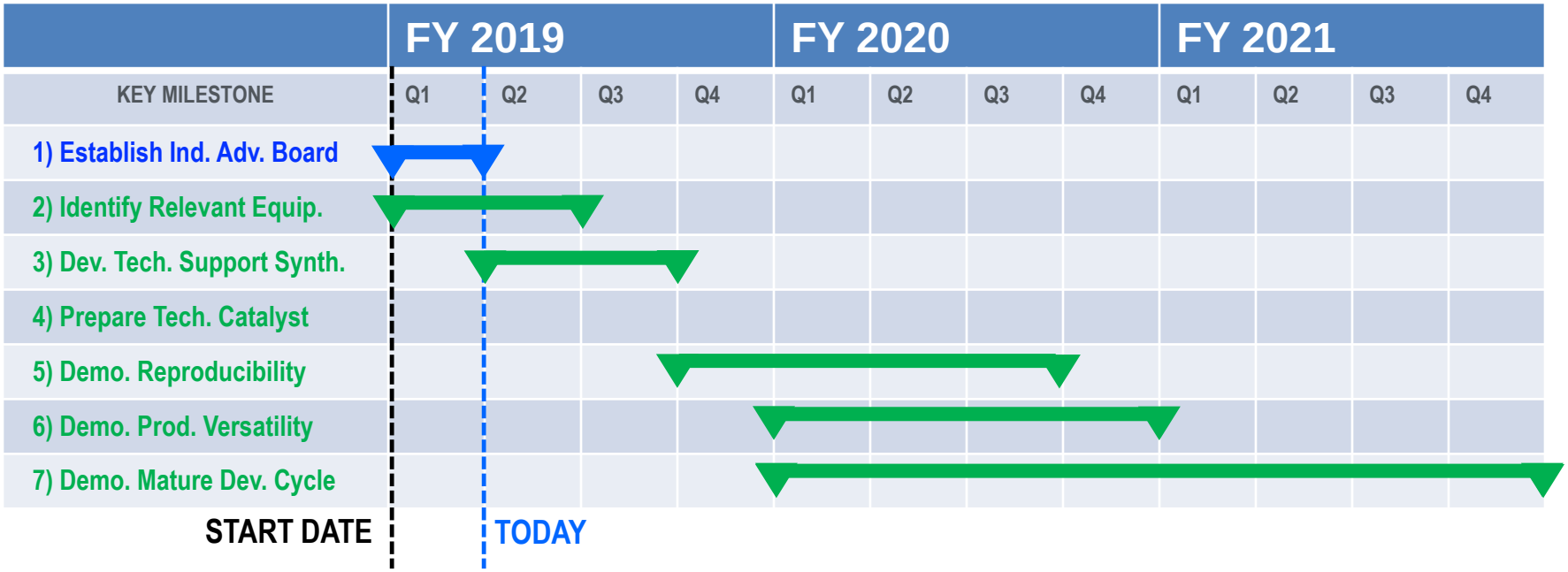
– The ***infrastructure and expertise*** required to translate emerging biomass conversion materials from the laboratory to commercial relevance

– *A focused* ***technical catalyst development effort*** *supporting engineering-scale performance evaluation* of novel catalytic materials.

Relevance to Biofuels

- **Significant number of biomass conversion processes rely on catalysis**
  - Catalytic technology development is leveraged by a major portion of conversion pathways across BETO's portfolio
  - Design and optimization of novel catalysts to improve selectivity, efficiency, and durability to enhance yields spans multiple R&D areas.
- **No dedicated effort to translate breakthrough biomass conversion catalysts from laboratory to pilot**
  - Evaluation of promising research catalyst at the pilot scale has been identified as a key research challenge.

Key Milestones



1) Establish industrial advisory board and determine baseline technical catalyst physical properties
2) Identify smallest industrially relevant process equipment
3) Develop synthetic platform for technical supports
4) De-risk catalytic technology verification through scale-up methodology development for technical catalysts to inform fundamental research
5) Demonstrate production versatility
6) Demonstrate a mature technical catalyst development Cycle

Project Budget Table

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	Original Project Cost (Estimated)			Project Spending and Balance		Final Project Costs
Budget Periods	**DOE Funding**	**Project Team Cost Shared Funding**	**Contingency**	**Spending to Date**	**Remaining Balance**	**What funding is needed to complete the project.**
FY19	$550k	-	-			
Scale-up Eng. R&D	$400k	-	-	$33k	$367k	$367k
Materials Eng. R&D	$150k	-	-	-	-	-
FY20	$550k	-	-	-	-	$550k
Scale-up Eng. R&D	$400k	-	-	-	-	$400k
Materials Eng. R&D	$150k	-	-	-	-	$150k
FY21	$550k	-	-	-	-	$550k
Scale-up Eng. R&D	$400k	-	-	-	-	$400k
Materials Eng. R&D	$150k	-	-	-	-	$150k

Quad Chart Overview

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Timeline

- Project Start: October 1, 2018
- Project End: September 30, 2021
- Percent Complete: 6%

	Total Costs Pre FY17	FY17 Costs	FY18 Costs	Total Planned Funding (FY19–FY21)
DOE Funded	$0	$0	$0	$1.6M
Project Cost Share	N/A	N/A	N/A	N/A

Partners (FY19–FY21):

NREL: $1.2M (73%)
ANL: $450k (27%)

Barriers addressed

Ct-G. *Decreasing the Time and cost to developing novel industrially relevant catalysts*
ADO-D. *Technical Risk of Scaling: Operations must be scaled-up and verified at the pilot-scale*

Objective

The goal of this project is to create *a flexible, engineering-scale catalyst synthesis capability within BETO* to develop the critical scientific basis of catalyst scale-up required to translate emerging biomass conversion materials from the laboratory to commercial relevance by *supporting engineering-scale performance evaluation* of novel catalytic materials.

End of Project Goal

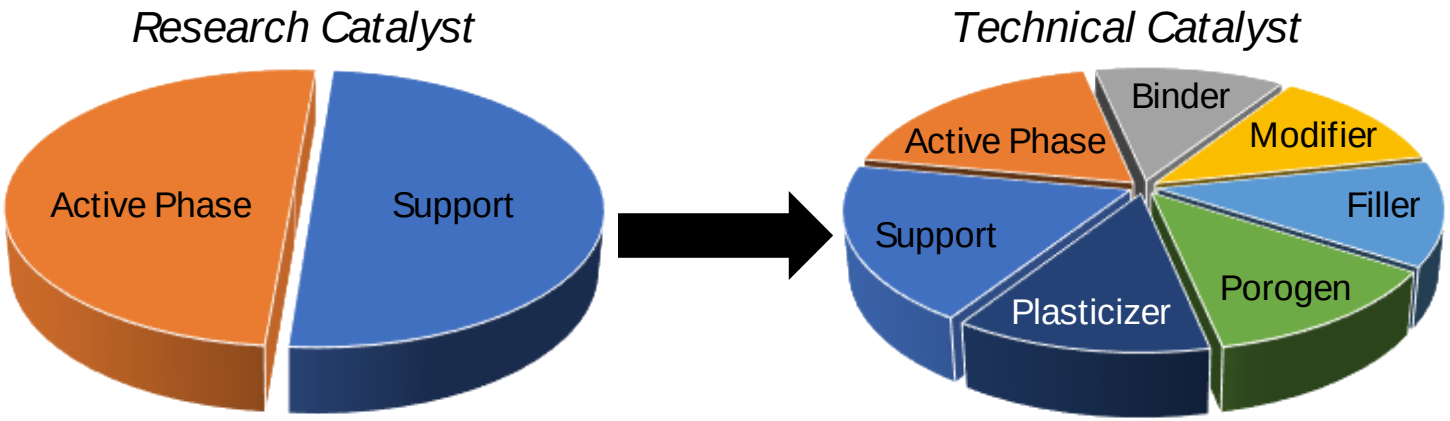
The 3-year goal of this project is to *demonstrate a mature technical catalyst development cycle* by preparing the necessary quantity (ca. 100 kg) of catalyst for the FY22 Catalytic Fast Pyrolysis technology verification that meet the physical specifications required for operation in NREL's Thermochemical Process Development Unit (TCPDU).

1 – Project Overview: Scale Up

The Challenge: A technical catalyst must faithfully ***reproduce the performance*** of laboratory preparations and possess the required physical properties ***for large scale operation***

Developing a technical catalyst from benchtop candidates requires ***at a minimum:***

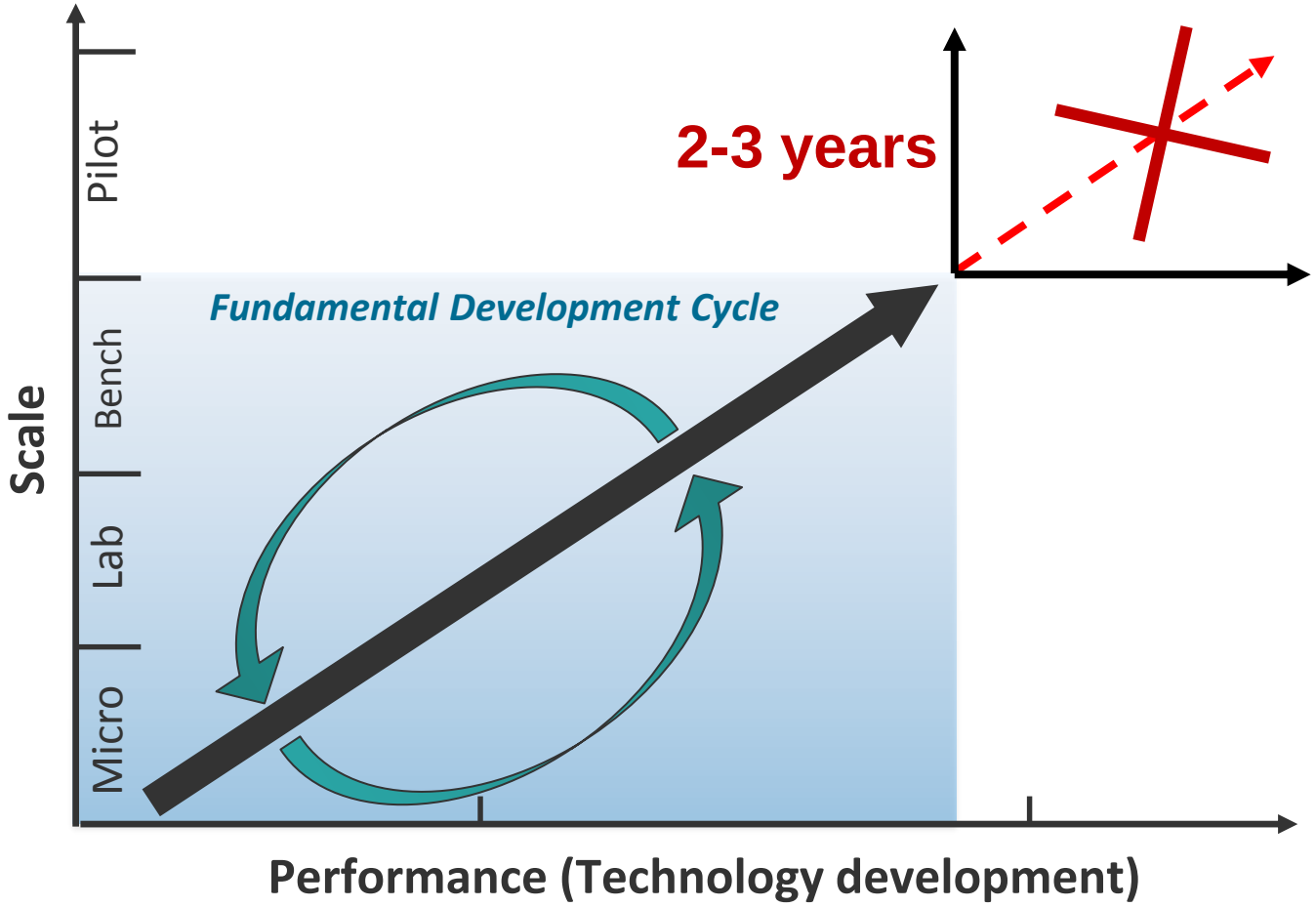
- Gram-to-kilo ***protocol adaptation***
- Determination of ***multi-component formulation***
- ***Shaping powders*** into reactor specific macroscopic forms



Translation of promising research catalysts to viable technical bodies is a non-trivial research challenge

1– Overview: Decoupling Technology Development and Scaling

Simultaneous technology development and scaling can hinder progress towards hitting performance targets



1– Overview: Decoupling Technology Development and Scaling

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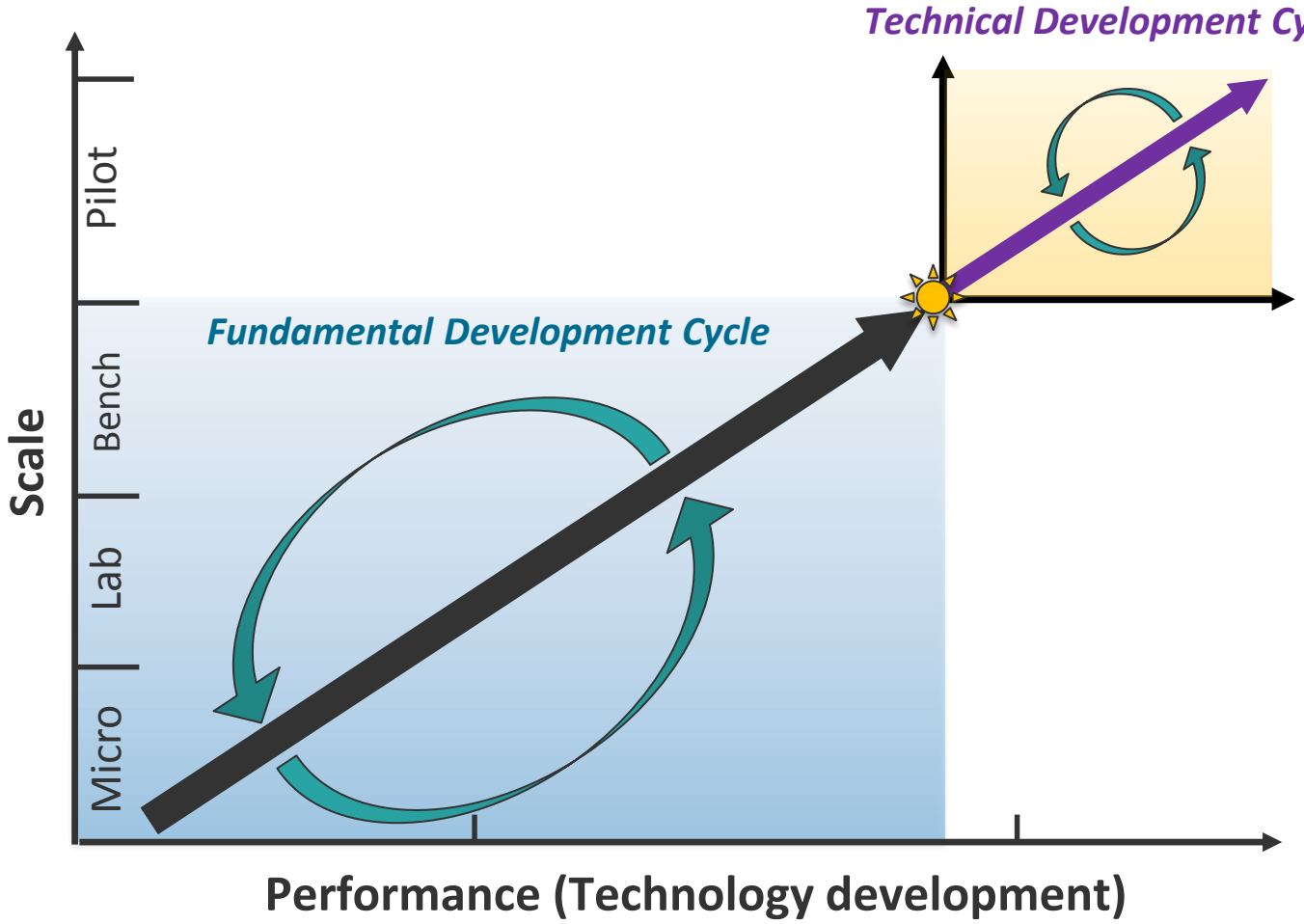

The EOS project seeks to develop a mature technical catalyst development cycle

1– Overview: Decoupling Technology Development and Scaling

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Challenges in Technology Verification Cycle: Cu/zeolite

High-performance C
identified for FY18 tech

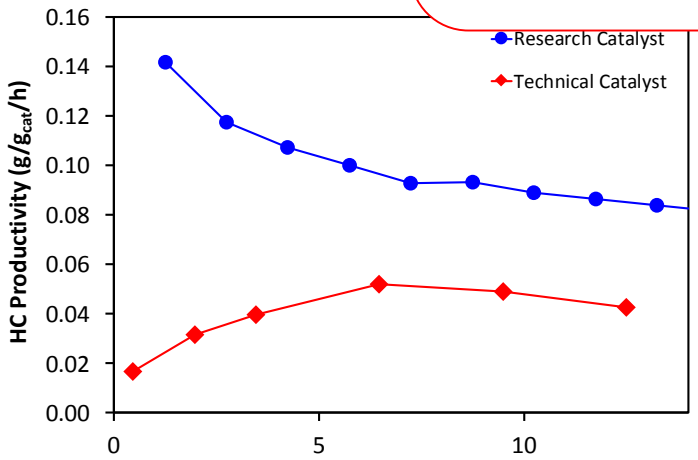
New Prep.

from lab scale

High Cost

Equipment too large for intermediate scale

Time, Cost, Performance, and <u>Value Proposition</u> Negatively Impacted



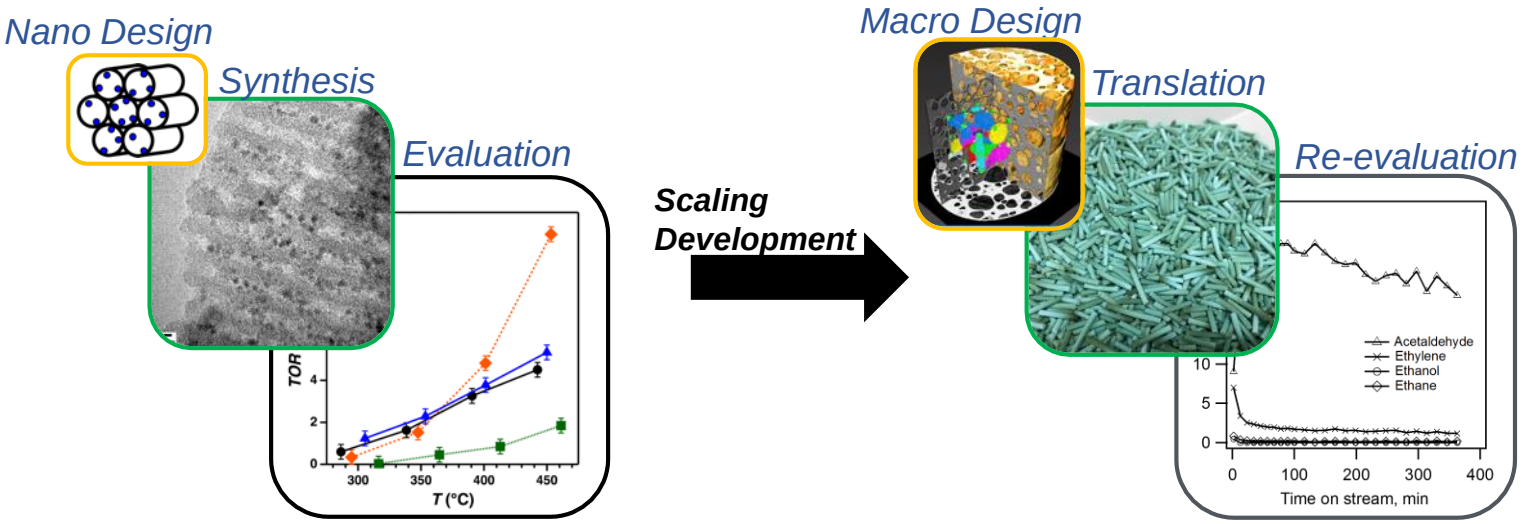
<u>Requirements for Verification</u>

- **≥10^5** increase in production from gram-scale to 100 kg
- Powder zeolite to formed extrudate

Performance impacted by available commercial extrudate formulation

What does an integrated catalyst scale-up capability offer?

- ***A flexible synthesis platform*** to support fundamental catalyst development
- ***A strengthened value proposition*** of novel catalysts developed in the DOE complex
- ***A fully leveraged*** PDU enabled by pilot-scale evaluation of novel materials
- ***A point of contact*** for industrial engagement
- ***A effective path*** to technology verification



2 – Technical Approach: Context

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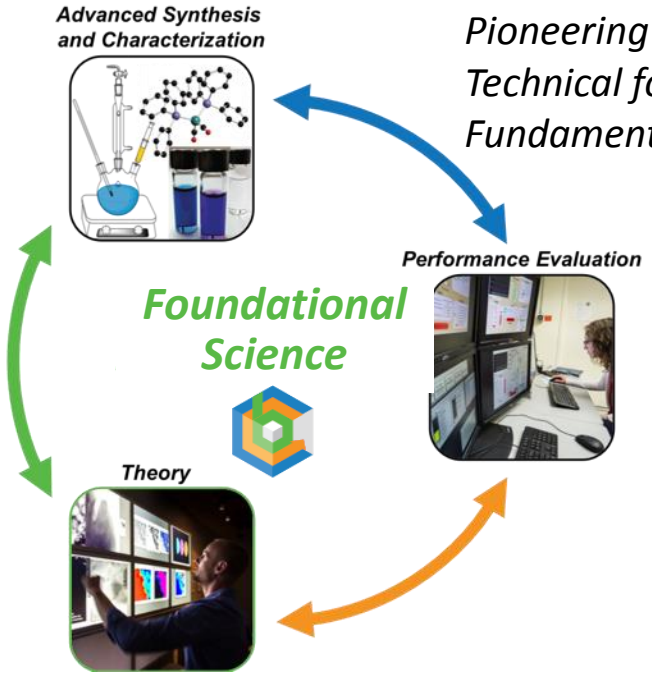
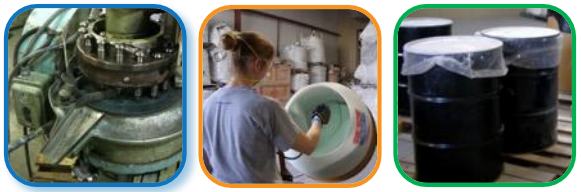
ChemCatBio/Conversion



Pioneering scale-up methodologies
Technical forms of CCB research catalysts
Fundamental scaling-performance relationships

ADO

Catalyst Scale-up



Industry responsive catalyst scale-up
Production of engineering-scale quantities
Enable PDU evaluation of next-gen catalysts
Facilitate effective verification efforts

Accelerating Market Deployment of Biomass Conversion Technologies

2 – Technical Approach: Context

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The EOS project is fully integrated with fundamental catalyst development and process scale-up efforts

Performance Evaluation (Core Catalysis Projects) (IDL, CFP, CUBI)

Foundational Science

Advanced Synthesis and Characterization (ACSC)

Computational Modeling (CCRC)

Requirements
Scaling Insight

Applied Engineering

Engineering of Catalyst Scale Up

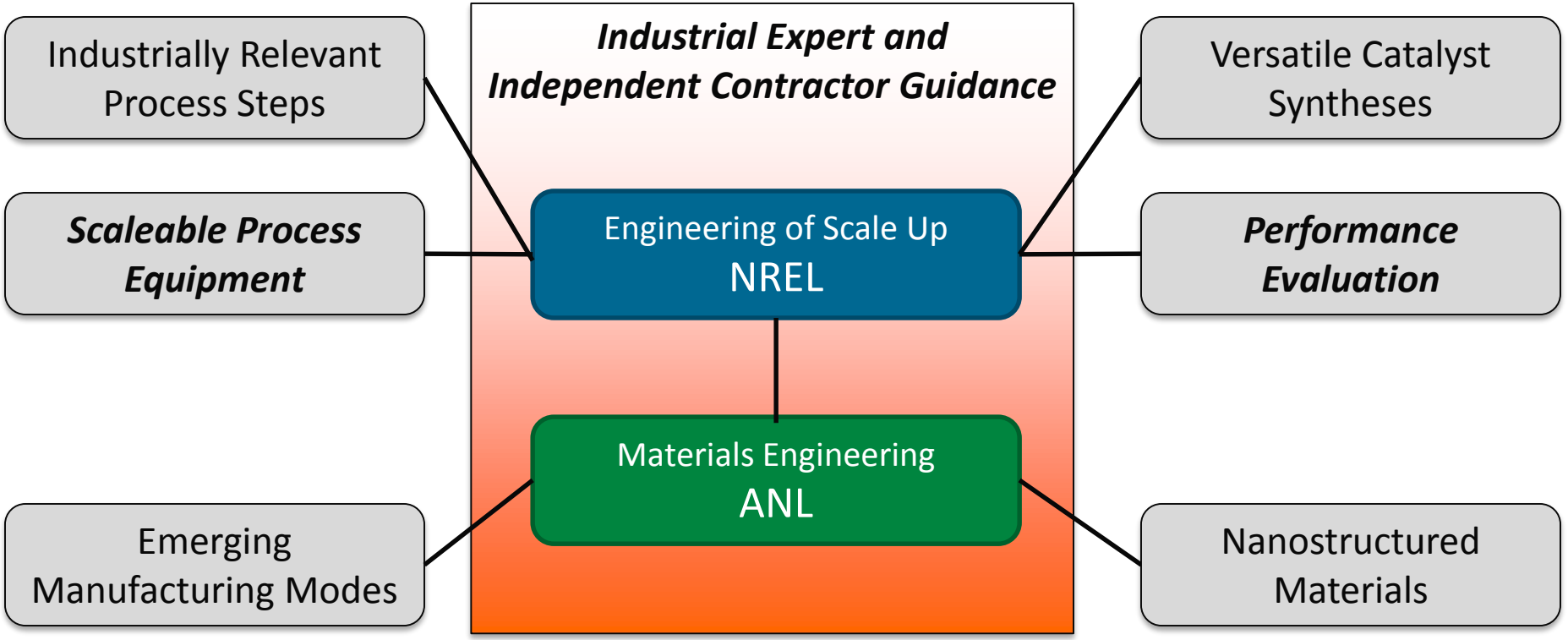
Process Scale Up to Production Environments

Co-development of catalysts, reactors, and processes

Catalyst targets and performance requirements informed by ***core catalysis projects***

Physical properties dictated by ***reactor design and process scale-up projects***

2 – Technical Approach



Success Factors

- ***Development*** of an engineering-scale catalyst synthesis capability
- ***Flexibility*** to handle multiple catalyst scale-up technologies
- ***Production*** of quantities sufficient for evaluation at DOE pilot plants
- ***Integration*** with emerging manufacturing modes (MERF)
- ***Feedback*** to fundamental catalyst development efforts

2 – Management Approach

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Closely integrated with industry to guide development and verify utility of capabilities

NREL
Frederick Baddour

- Catalyst Development
- Technical Body Form.
- Physical Forming
- Performance Eval.

ANL
Kris Pupek

- Advanced Materials
- Nanostructuring
- AP and support integ.

Assessment of SOT / Data Gathering

Implementation of Best Practices

Industrial Expert Review

Refinement

Industrial Expert Review

1st Generation Scaled Catalyst

Industrial Advisory Board

Biannual best practice review
(e.g.,methods, equip., char.)

Annual capability review

Approach gaps

Immediate changes to handling, verification, eval.

ID of Critical Components

Incorporation into AOP, go/no-go, planned milestone efforts

FY20 Go/No-go decision based on reproducibility and implementation

FY21 Target: Verification Catalyst

3 – Research Progress: Establishing Industrial Advisory Board

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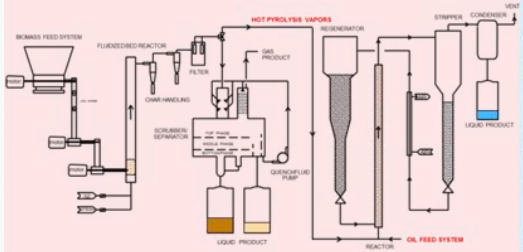
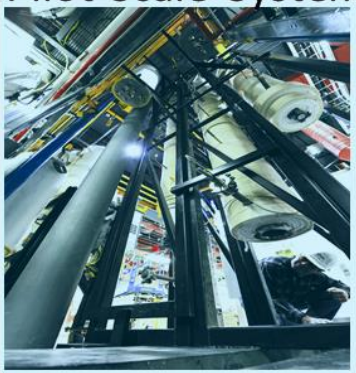
EOS Team has extensive track record for industrial and academic engagement

An initial team of industry advisory board members has been established consisting of members from

National Laboratories
Oil and Gas Producers
Catalyst Manufacturers
Chemical Producers
and Independent Contractors

Strong industrial connections to be leveraged to guide scale-up efforts to maintain ***relevant targets, methodologies, and performance metrics***

4 – Relevance: Enabling Large Scale Evaluation

	Microscale Reactor	Bench-Scale Fluid Bed Reactor	Large Bench-Scale DCR System	Pilot-Scale System
				
Catalyst	1g	500g	2.0kg	100kg
Biomass	25 mg/run	0.5kg/h	3kg/h	30kg/h
	• *In-, ex-situ* CFP • Batch experiments • Catalyst screening	• *In-, ex-situ* CFP • Continuous experiments • Catalyst screening	• *In-, ex-situ* CFP • Continuous experiments • Catalyst screening • Catalyst regeneration • Hot gas filtration • Co-processing	• *In-, ex-situ* CFP • Continuous experiments • Catalyst regeneration • Catalyst screening

A catalyst scale up effort

Limited to commercial catalysts

- ***Enables*** emerging catalysts to be proven at industrially relevant scales
- ***Provides*** an opportunity for small businesses to evaluate the scalability and performance of new materials
- ***Minimizes risk*** and cost to evaluate economics of production and performance

4 – Relevance: Single POC Scale Up and Evaluation

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Process Scale-up

Gram-scale testing

Rxn kinetics

1 kg-scale evaluation

Transfer fx

≥10 kg verification

Small Start-up
Cat. Scale-up and verification

Catalyst Manufactures
Scaling relationships

Industrial Process Co.
Process intensification

Catalyst Scale-up

Bench Scale Synthesis

Fund. Char.

Technical preparation

Tech. Char.

kg-scale production

*Catalysts and processes with **proven scalability** and **demonstrated performance** at the engineering scale through a single, integrated point of contact*

4 – Relevance

Pre-commercial catalyst development and usage is heavily-leveraged within BETOs conversion portfolio

The EOS project enables engineering-scale evaluation of advanced catalyst materials

Uncertain catalyst performance at the pilot scale contributes significantly to commercialization risk

Industrial catalysis is extremely risk averse and performance uncertainty at relevant scales significantly impacts the value proposition of advanced catalyst materials

Catalyst scale up can pose an economic barrier to small biomass conversion companies

A national laboratory led scale-up effort can ***support domestic business*** through
(1) enabling large-scale performance evaluation to ***mitigate risk***
(2) ***serving as a pipeline*** for proven laboratory-developed catalysts and technologies to the commercial sector

5 – Future Work: Roadmap

FY 2019

Quarter 1
Establish guiding industrial advisory board

Advisory board will guide efforts to ensure Process, equipment, and targets remain ***relevant and produce value*** for the biomass community

Quarter 2
Identify smallest Industrially relevant equipment

Equipment will be selected to produce quantities of catalyst suitable for operation in NREL's TCPDU based on:

- Active deployment of ***large-scale analogs***
- ***Transferability*** of developed process knowledge
- Operational ***flexibility***

Quarter 3
Develop synthetic platform for technical supports

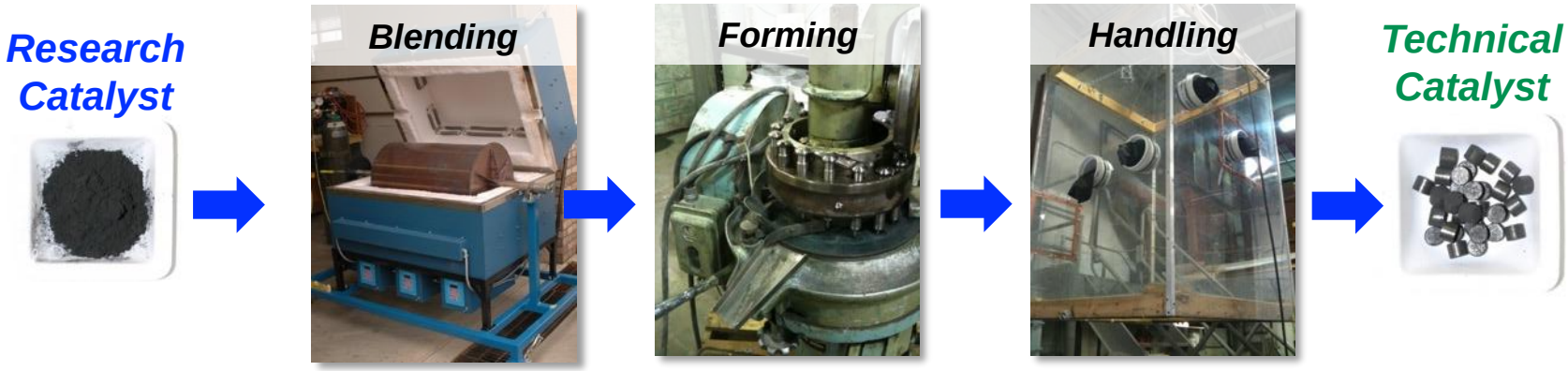
A baseline technical body will be prepared based on FY22 CFP verification targets that ***meets physical requirements*** for TCPDU operation

Quarter 4
Prepare Phase I scaled catalyst based on CCB verification target

A Phase I scaled catalyst informed by FY22 verification targets will be ***prepared in kilogram quantities*** for performance evaluation

5 – Future Work: Near Term

Development of expertise in technical body preparation

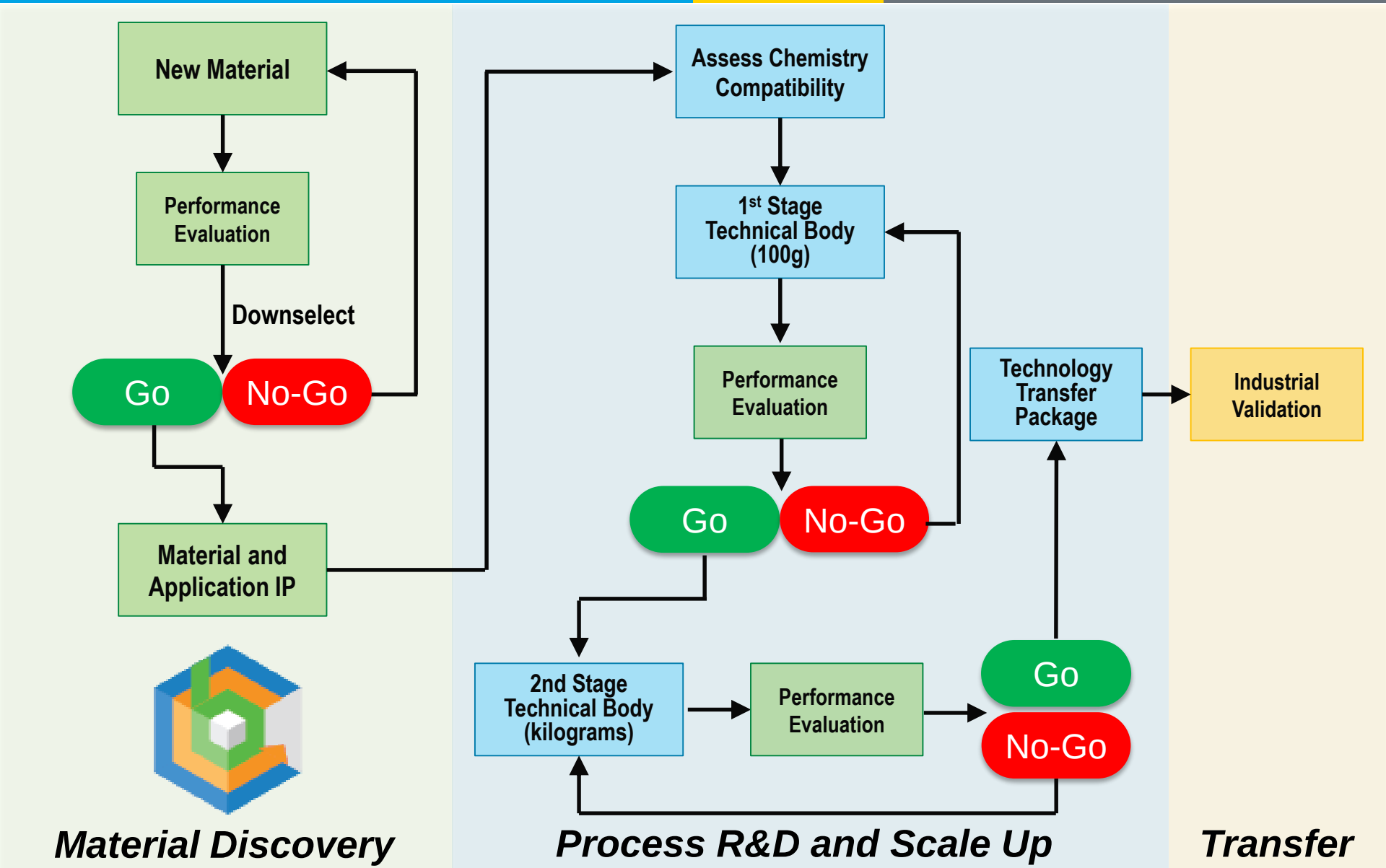


- ***Dedicated in-house equipment*** for inert processing, thermal treatment, separation, precipitation, physical forming
- ***Ability to optimize*** translation from research catalyst to technical body
- ***Transferable knowledge*** for more rapid and simplified contract manufacturing at relevant scales

Initial Target: Pt/TiO_2 for FY22

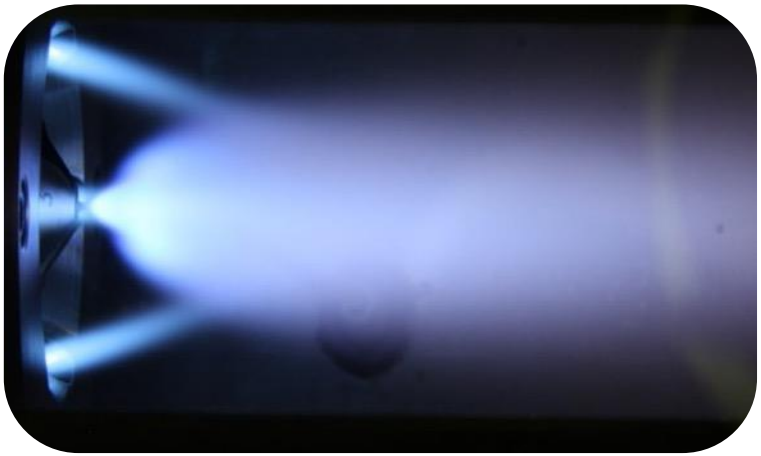
Catalytic Fast Pyrolysis Technology Verification

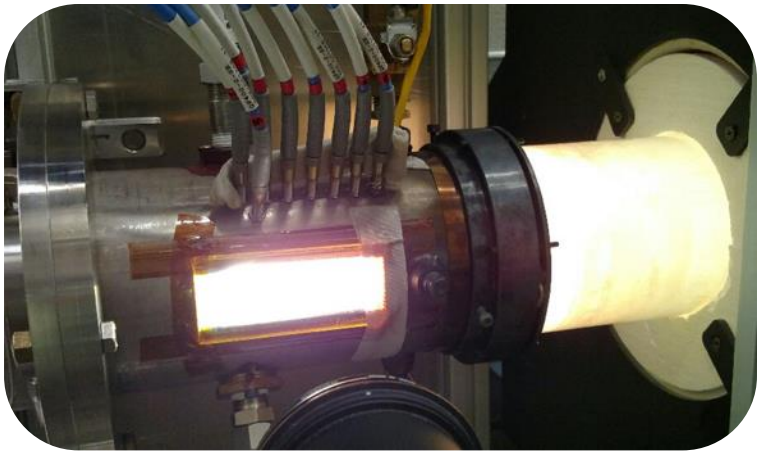
5 – Future Work: Staged Scale Up



5 – Future Work: Advanced Materials

Development of scalable advanced technologies for incorporation of nanostructured, non-traditional active phases into technical catalysts





- Utilize Aerosol Manufacturing processes for ***high-volume continuous manufacturing of catalyst nanopowders*** (100 g/hr)
- ***One-step synthesis*** of active phase and support (e.g, Pt/TiO_2)
- Development of ***incorporation strategies*** of nanopowder catalyst into extrudates
- Employ laser-based combustion zone diagnostic to ***control active phase*** formation

5 – Future Work: Integrated Scale Up Vision

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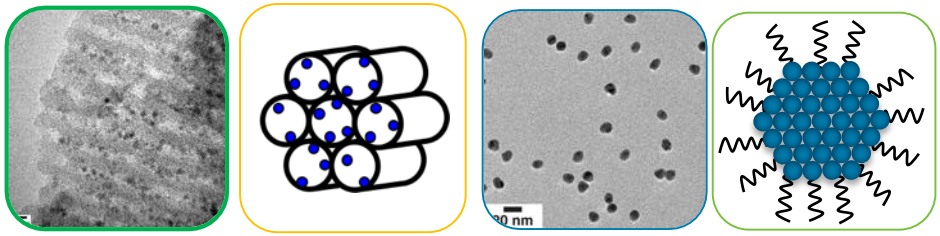
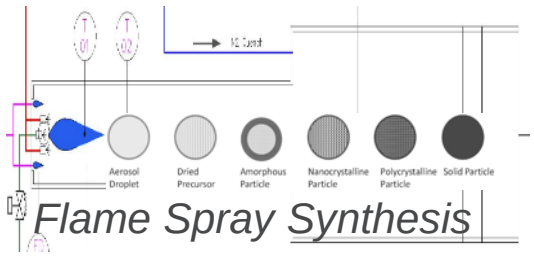
Advanced Catalyst Discovery Engine (CCB)



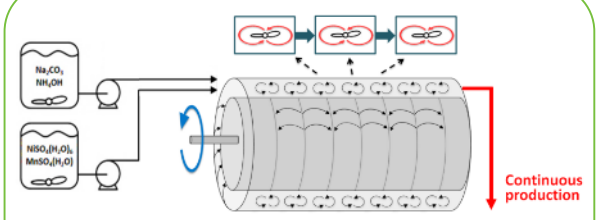
Scale-up targets, performance metrics

Pilot-scale evaluation

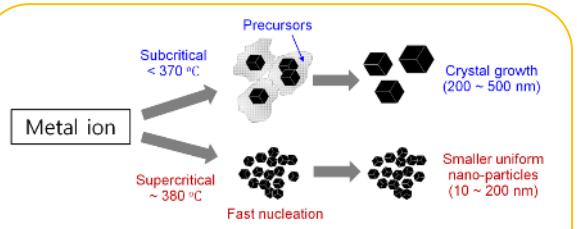
Pioneering Scale-Up Methodologies



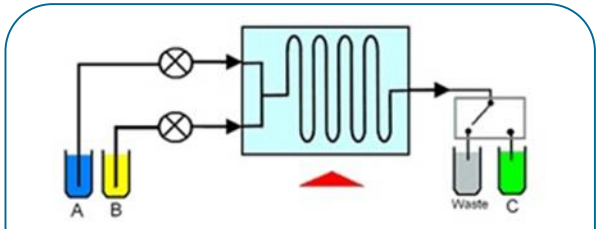
Flame Spray Synthesis



Taylor Vortex Synthesis

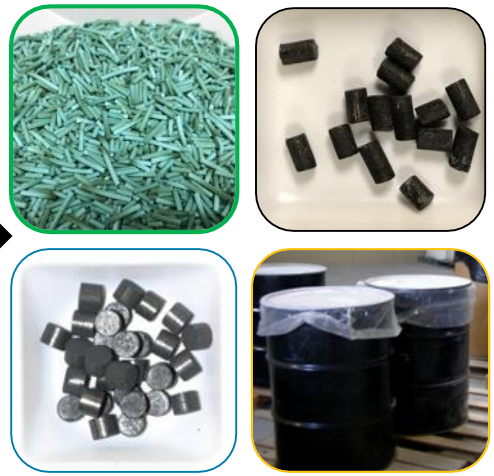


Hydrothermal Synthesis



Continuous Flow Synthesis

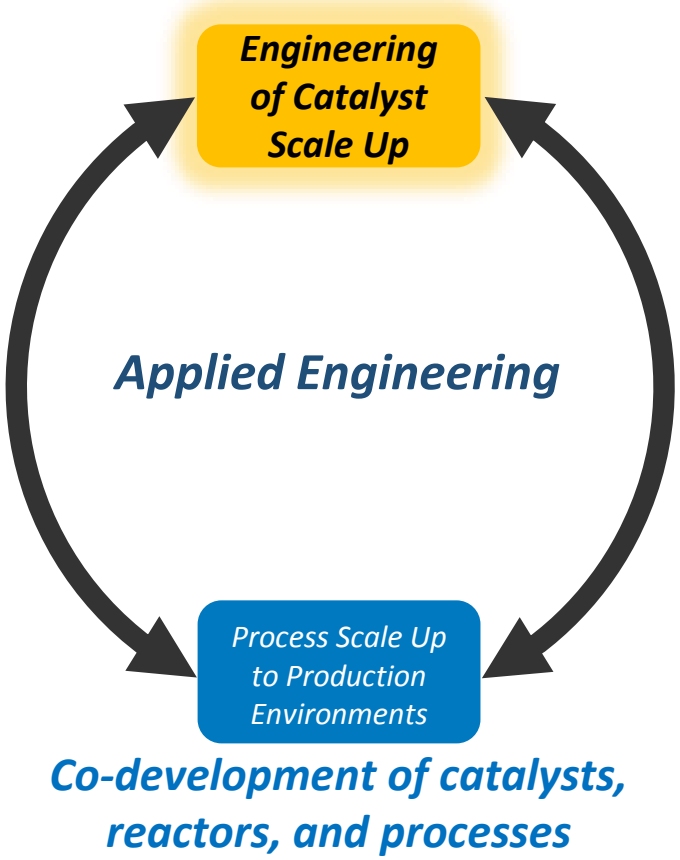
Technical Body Production



Summary

Project Goal – Develop *a flexible, engineering-scale catalyst synthesis capability* to produce ***scalable and cost effective*** next-generation biomass conversion catalysts and mitigate commercialization risk by ***enabling large-scale performance evaluation***

- An integrated catalyst scale-up effort is crucial to ***minimizing optimization time***
- In-house synthesis of engineering-scale quantities of catalyst can significantly ***reduce the economic investment*** required to verify large-scale performance
- Collaboration between emerging scale-up methodologies and traditional catalyst manufacture provides an opportunity for ***scalable performance enhancement***



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