ChemCatBio
Chemical Catalysis for Bioenergy

Advancing Catalytic Fast Pyrolysis through Integrated Experimentation and Multi-Scale Computational Modeling

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U.S. DEPARTMENT OF ENERGY | Office of ENERGY EFFICIENCY & RENEWABLE ENERGY
BIOENERGY TECHNOLOGIES OFFICE

Catalytic Fast Pyrolysis (CFP) Overview

Biomass Harvesting → Feedstock Pre-Processing → Fast Pyrolysis → Vapor Phase Catalytic Upgrading → Hydrotreating and Fractionation

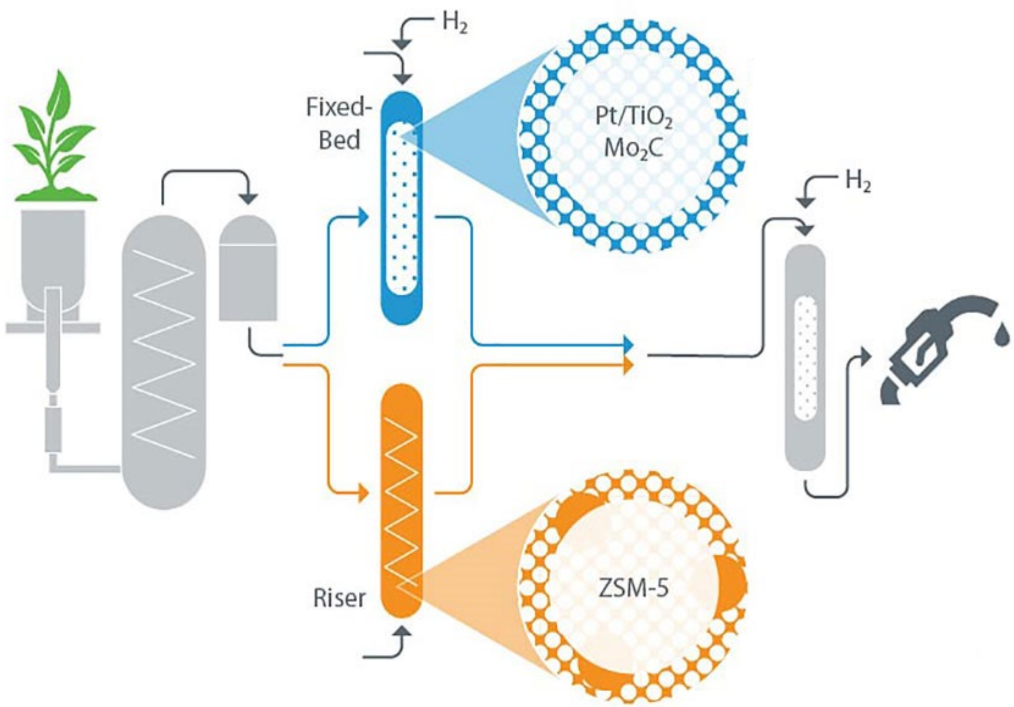
CFP is an adaptable pathway for the conversion of woody biomass and waste carbon sources into fuel blendstocks and chemical co-products

Ruddy, D. et al. *Green Chem.*, **2014**, 16, 454
Langholtz, M. H., et al. 2016 Billion Ton Report, US DOE, ORNL-TM2016-160

Approach to Catalytic Fast Pyrolysis

In-Situ CFP

Ex-Situ CFP



Technical approaches include different catalysts and reactor configurations

Fluidized Bed Zeolite CFP

Pilot and demonstration scale data demonstrate the technical feasibility of the approach

Challenge: Rapid coking lowers yields, necessitates frequent regeneration, and drives up fuel costs

Fixed Bed Hydrodeoxygenation

Fundamental research highlights opportunities for enhanced performance

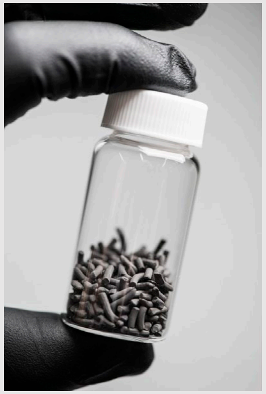
Gap: Lack of realistic reaction testing data increases risk and uncertainty

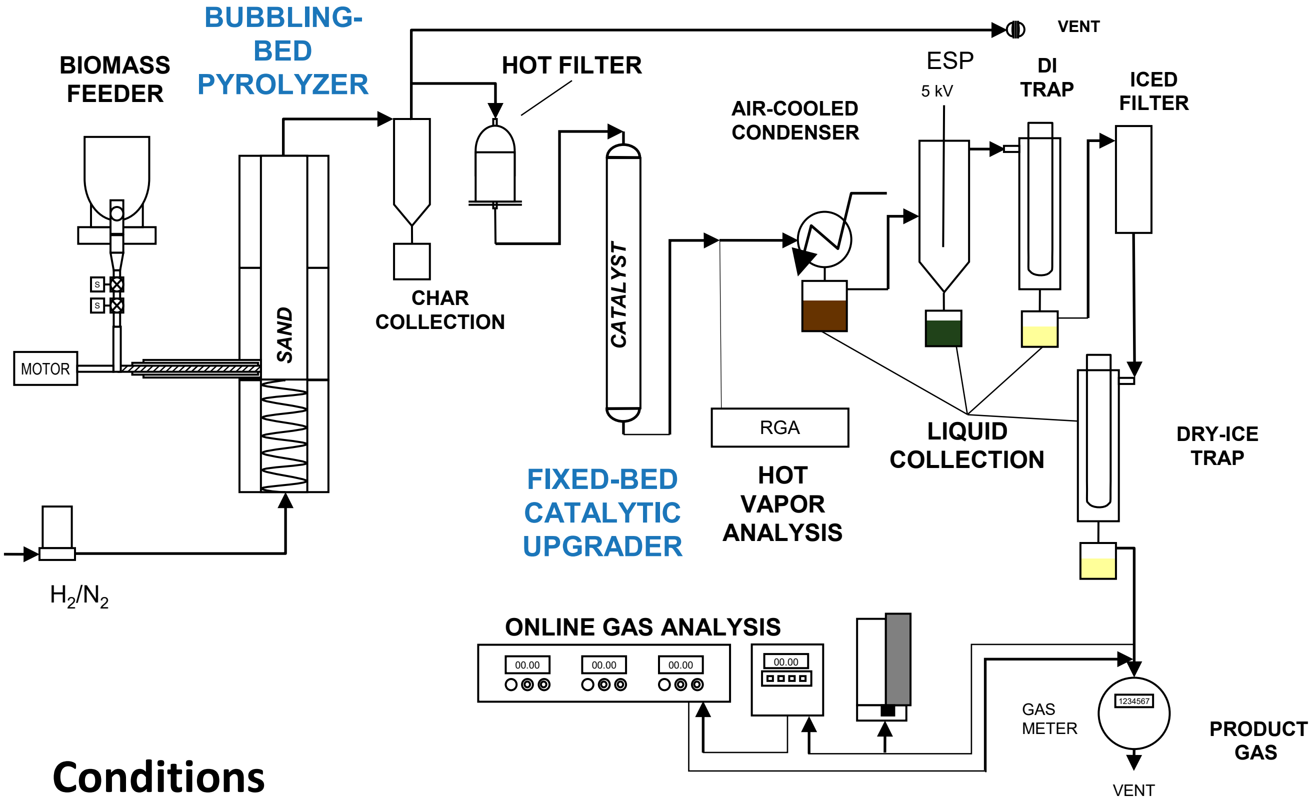
Integrated Reaction Testing With Biomass

Feedstock
Debarked Loblolly Pine and Forest Residues

Idaho National Lab

Catalyst
0.5-2.0 wt% Pt/TiO_2 on Technical Supports





Conditions

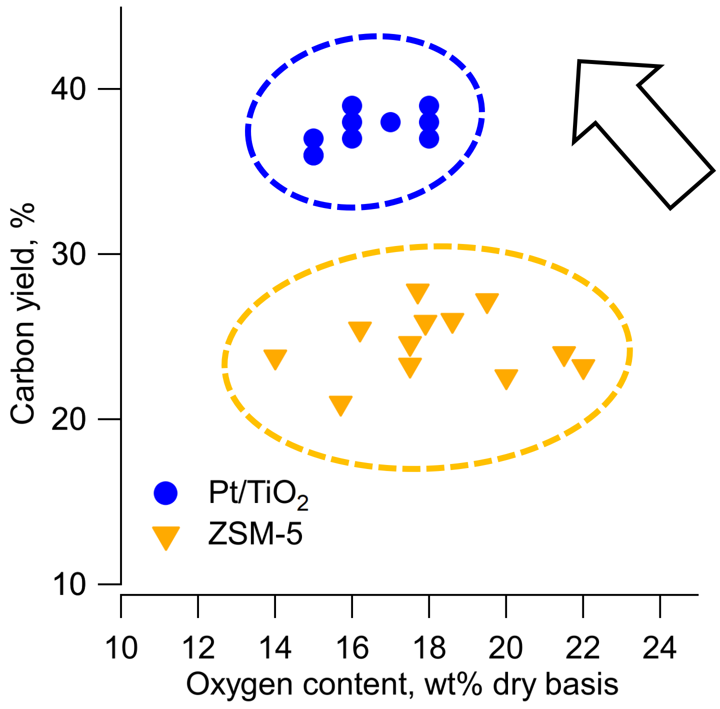
Pyrolysis Temperature: 500 °C
Upgrading Temperature: 435-450 °C
Catalyst Mass: 100 g

WHSV: 1.4 g biomass/gcat*h
Biomass:Catalyst Ratio: 3-13.2
Hydrogen Concentration: 83%

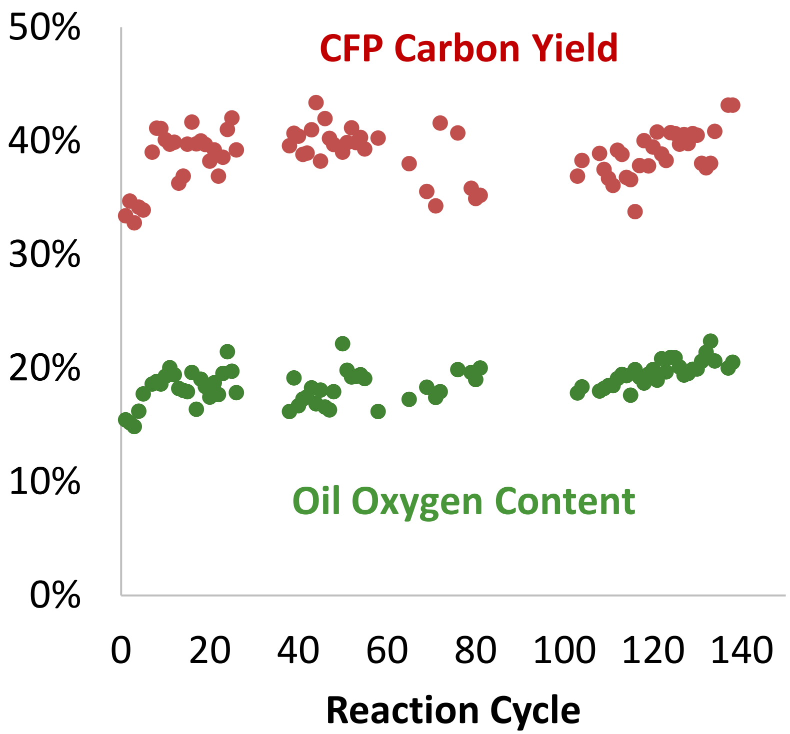
> 10 L of CFP-oil produced over 100+ reaction cycles

Reaction Testing Highlights Improved Performance

Pt/TiO_2 exhibited **improved carbon yields** at similar oxygen content compared to ZSM-5



Pt/TiO_2 exhibited **stable performance** over 100+ reaction/regeneration cycles

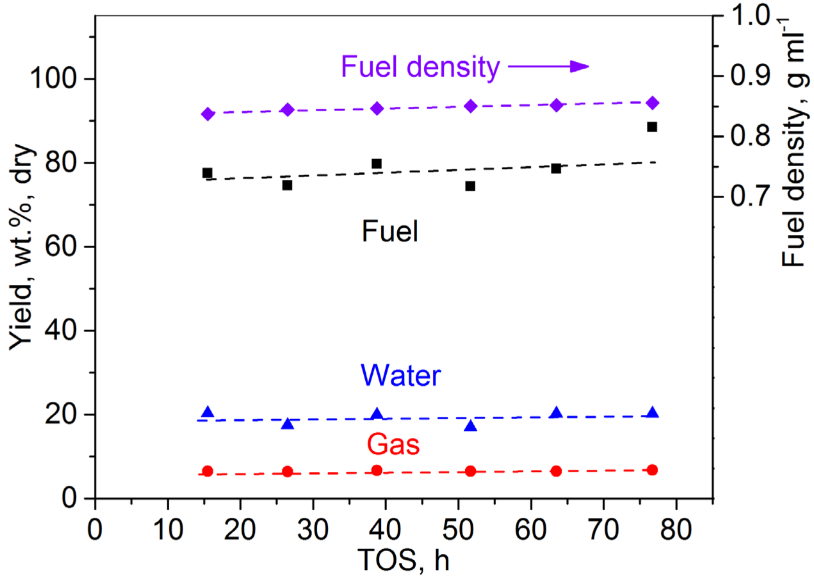


V. Paasikallio, et al. *Energy Technol* 5, **2017,** 94
V. Paasikallio, et al. *Green Chem* 16, **2014,** 3549

Griffin, M. et al., Energy Environ Sci, **2018,** 2904
K. Iisa, et al. *Energy Fuels* 30, **2016,** 2144
K. Iisa, et al. *Top Catal* 59, **2016,** 94

Stable Single Stage Hydrotreating

The Pt/TiO_2 CFP-oil was hydrotreated using a **single stage** system for 80+ hours without fouling or plugging

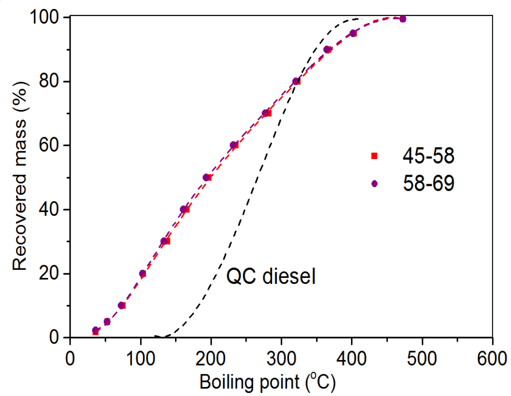


Carbon yield %	H/C mol/mol	O wt.% dry	Density g ml^{-1}
89	1.71	0.19	0.851

NiMo Sulfide, LHSV: 0.2-0.3, 13 MPa

Pacific Northwest NATIONAL LABORATORY

Fractionation indicates high selectivity to the distillate range



45 wt% in gasoline range

39 wt% in diesel range

Fuel testing reveals need for continued R&D

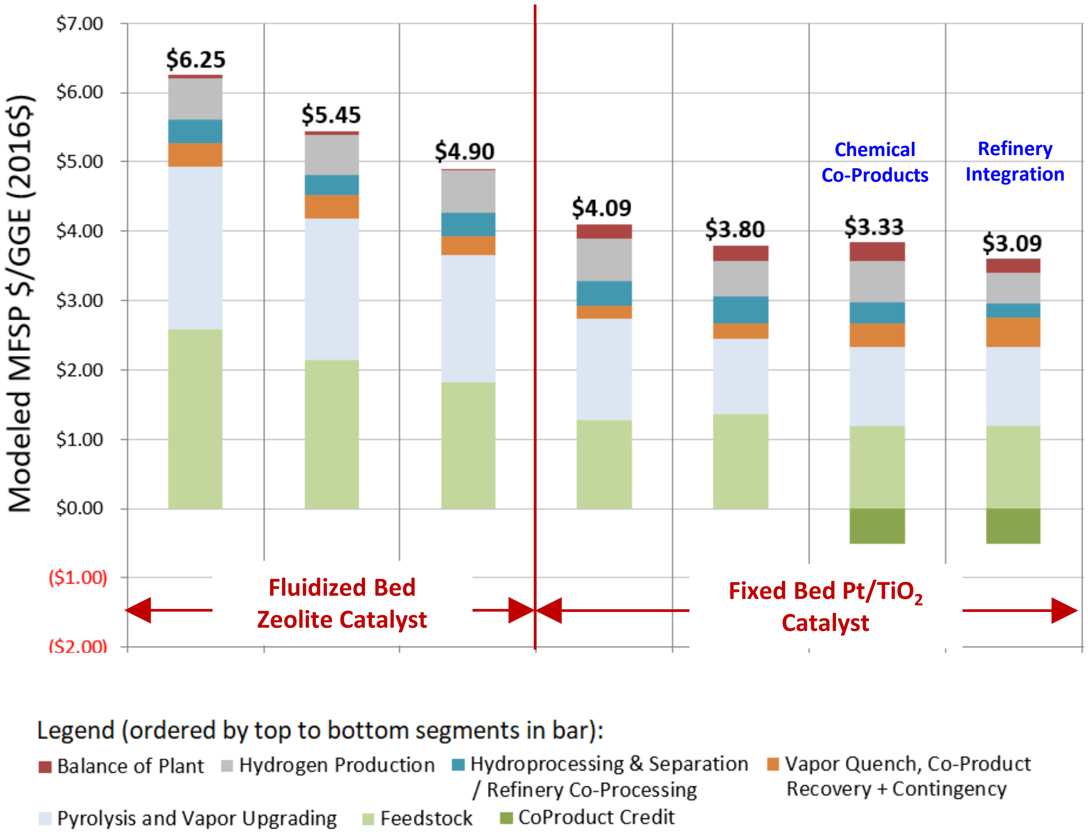
	Measured	Target
Gasoline AKI	65	85
Diesel DCN	24	40

CFP provide opportunity to improve fuel quality by controlling hydrogenation and promoting ring opening reactions

Griffin, M. et al., Energy Environ. Sci., 2018, 11, 2904

Technoeconomic and Lifecycle Analysis

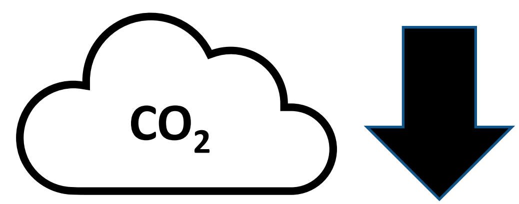
Conceptual process models indicate a minimum fuel selling price of \$3.80, with an opportunity for further reduction through refinery integration and the generation of chemical co-products



Overall Process Carbon Yield:

36% for Pt/TiO_2

≤ 22% for ZSM-5



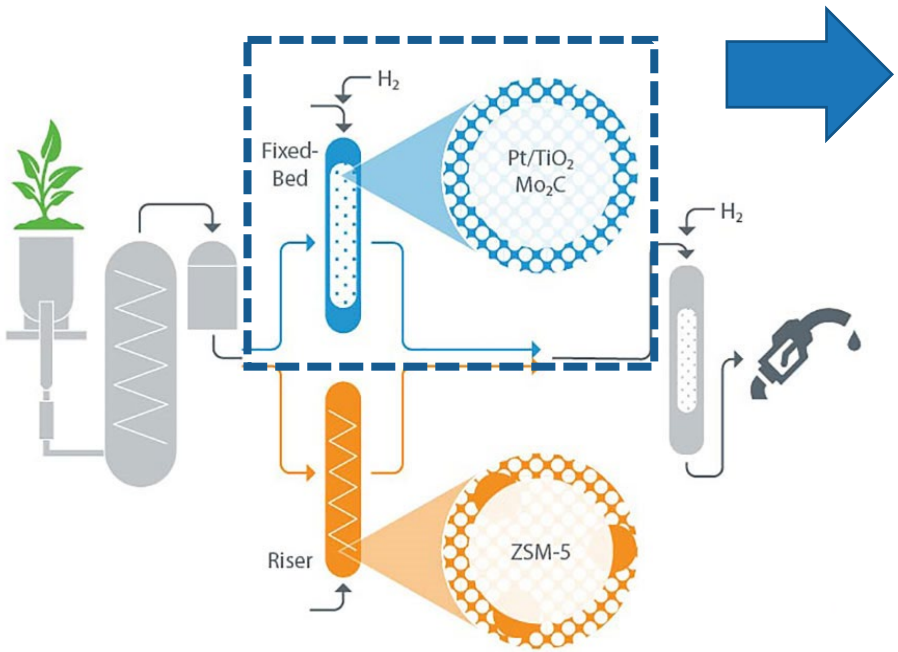
> 50%

Considerable reduction in carbon intensity

Reference: https://www.nrel.gov/docs/fy20osti/76269.pdf; **SOT:** State of Technology; **MFSP:** Minimum Fuel Selling Price

Summary and Research Needs

Integrated reaction testing confirmed potential for improved performance from fixed bed hydrodeoxygenation and motivates investigation of process scale up



- **High Yields** ✓
- **Improved Economics** ✓
- **Low Emissions** ✓
- **Process Stability** ✓
- **Scalability** ?

Leverage partnerships to perform particle and reactor scale computational modeling to directly address open questions about reaction kinetics and process scale-up

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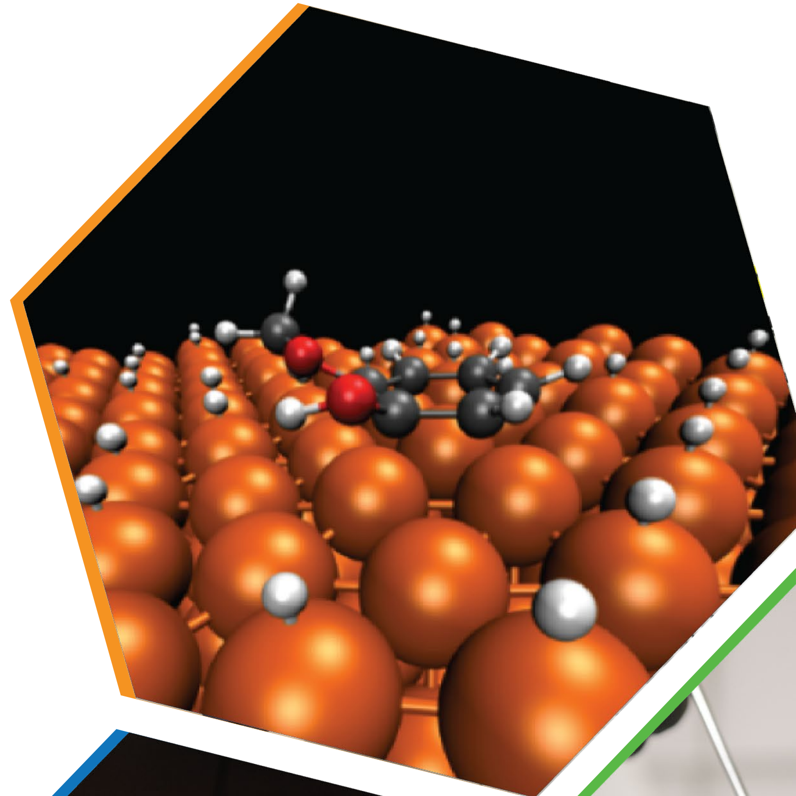
Teasing out fundamental information from bench top packed bed reactor experiments with multiscale modeling



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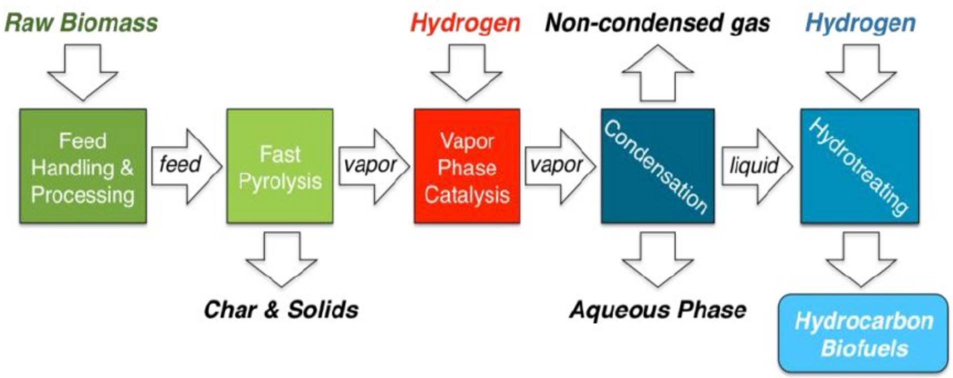
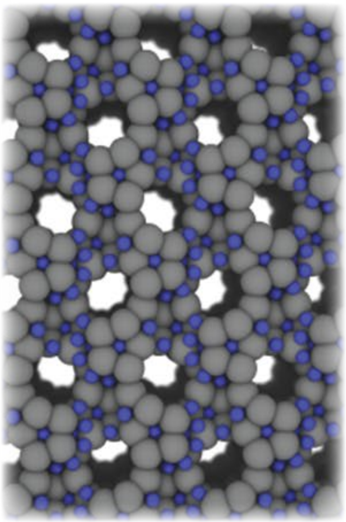
Introduction

- Promising bioenergy technologies often *fail at scale-up*
- Modeling can guide engineers moving from bench to pilot
  - Simultaneous transport phenomena at multiple scales
- Multiscale frameworks enable the use of DOE's high-performance computing (HPC) capacity
- In this work, we apply multi-scale modeling to catalytic fast pyrolysis vapor phase upgrading over platinum on titania

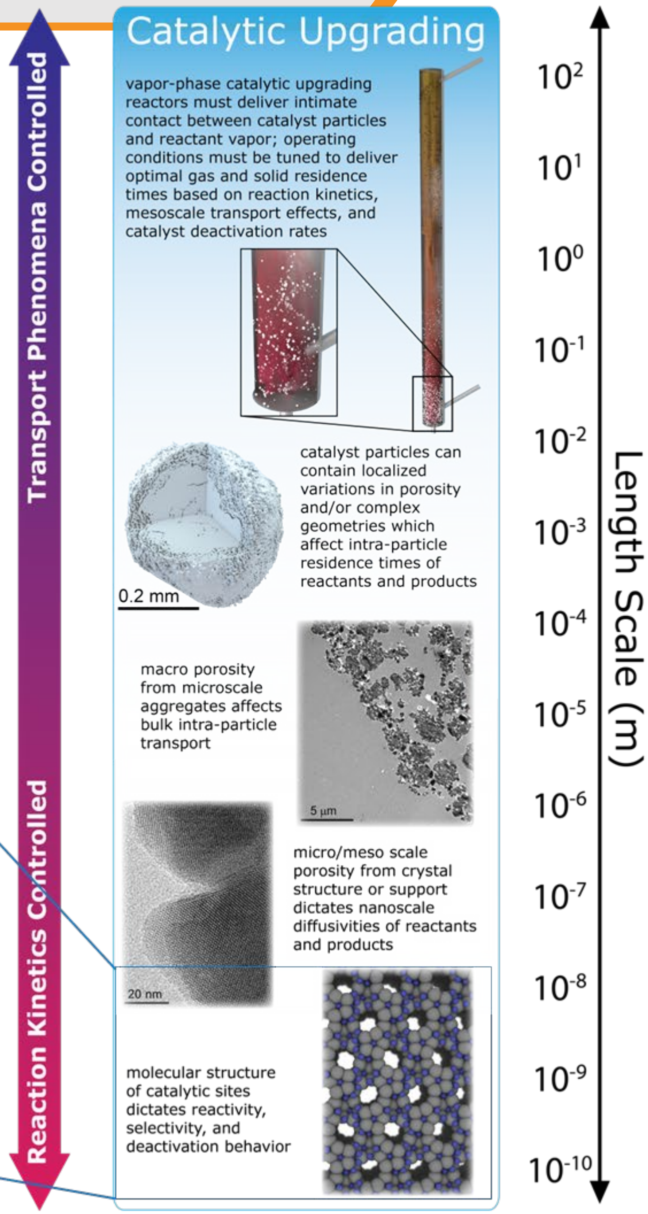


Multiscale phenomena in catalysis

molecular structure of catalytic sites dictates reactivity, selectivity, and deactivation behavior

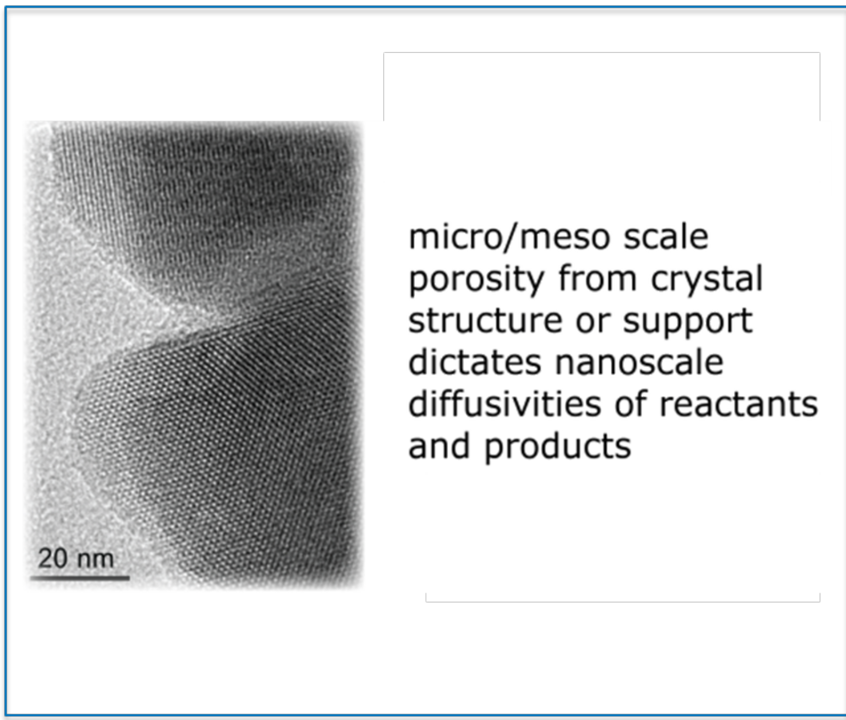


atomic scale

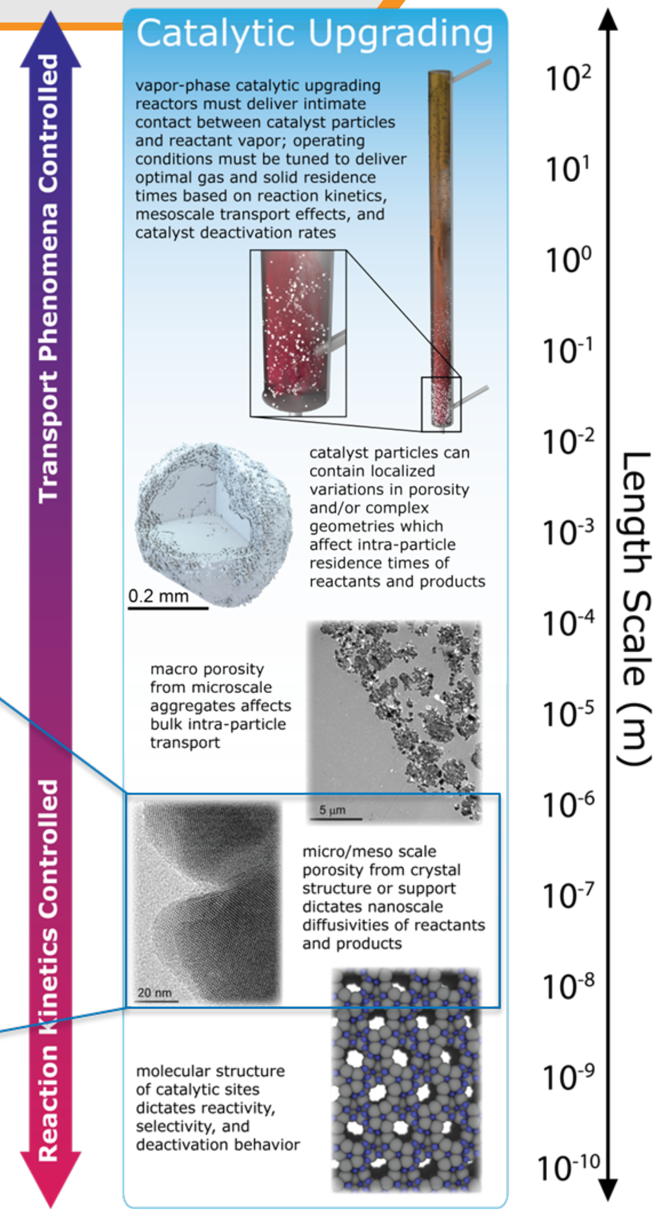


Ciesielski, Pecha, Bharadwaj, et al., Advancing catalytic fast pyrolysis through integrated multiscale modeling and experimentation: Challenges, progress, and perspectives. *Wiley Interdisciplinary Reviews: Energy and Environment* **2018,** *7,* 297.

Multiscale phenomena in catalysis



micro/mesopore scale



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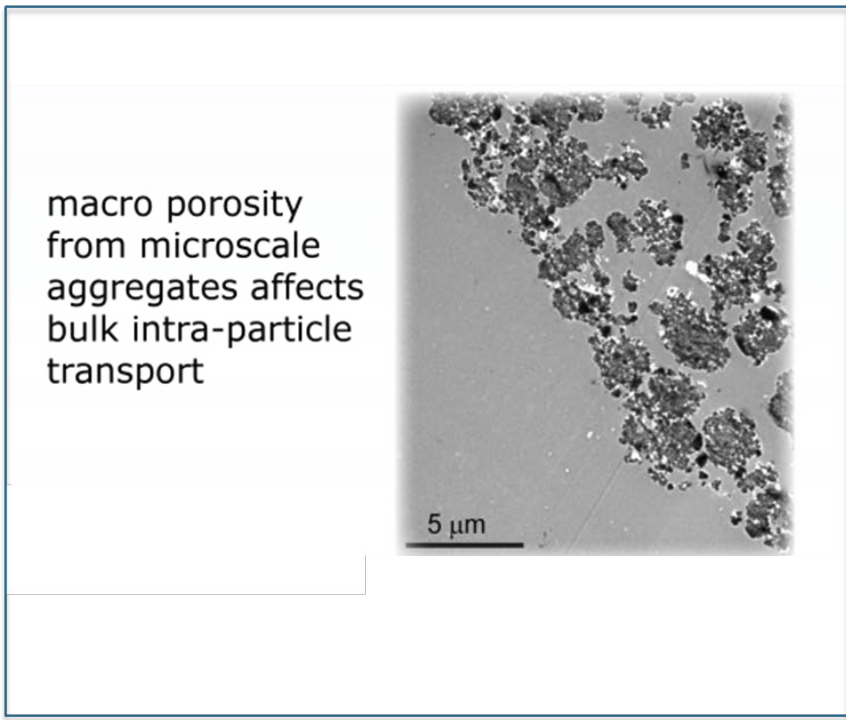
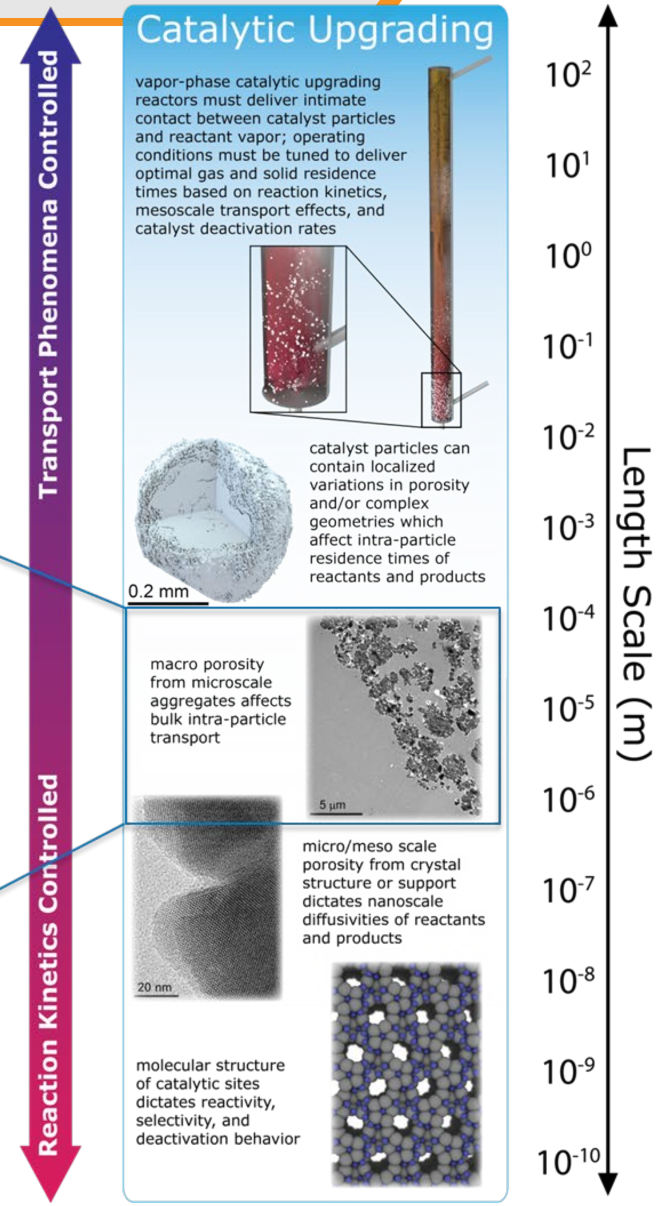
Multiscale phenomena in catalysis



macropore scale



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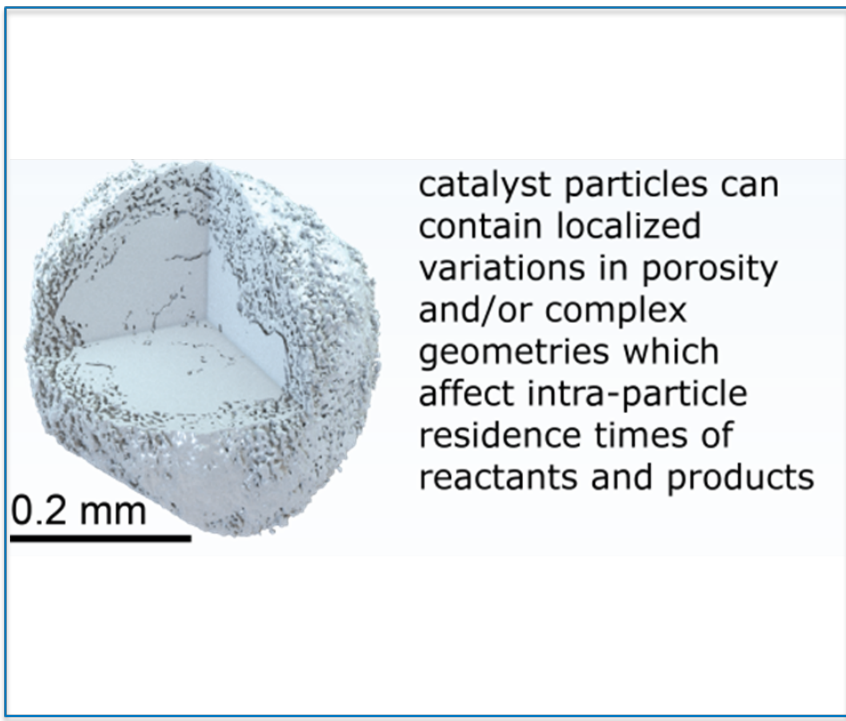
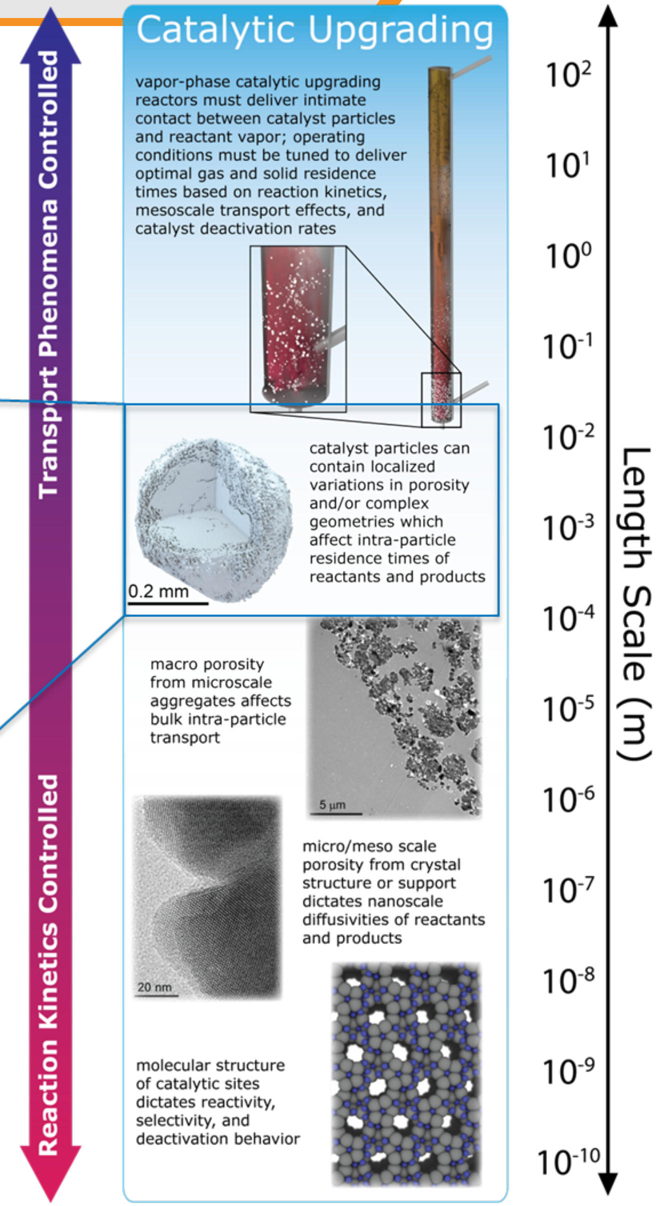
Multiscale phenomena in catalysis

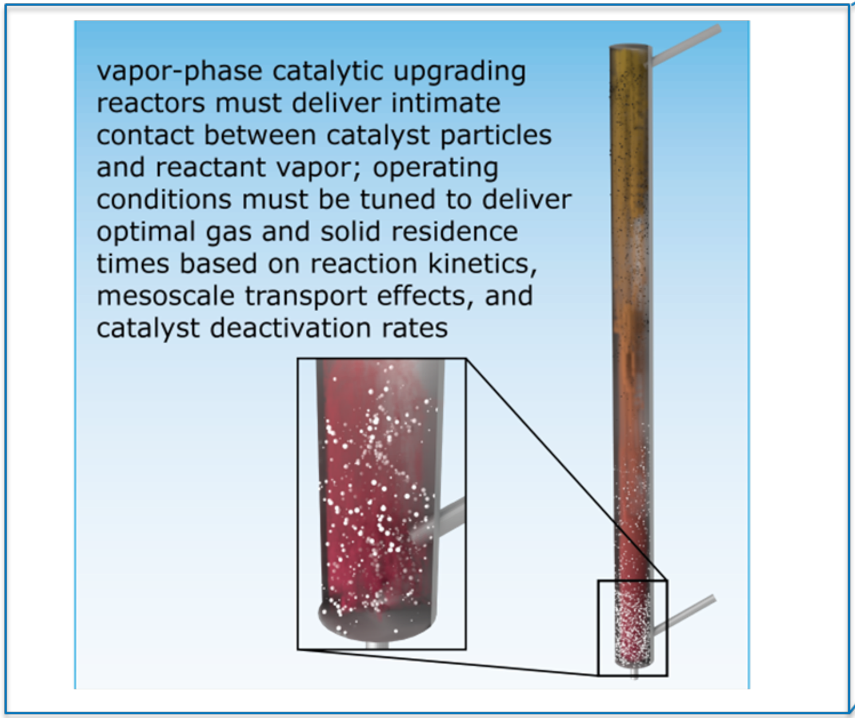


particle scale

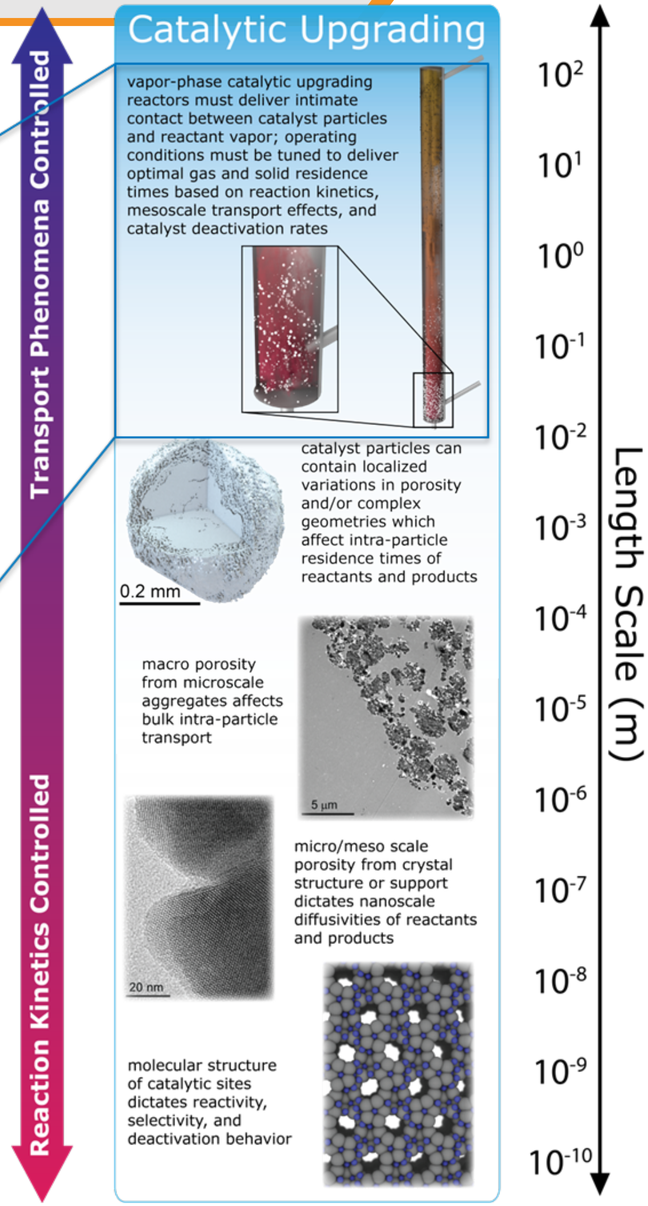


Ciesielski, Pecha, Bharadwaj, et al., Advancing catalytic fast pyrolysis through integrated multiscale modeling and experimentation: Challenges, progress, and perspectives. *Wiley Interdisciplinary Reviews: Energy and Environment* **2018,** *7,* 297.

Multiscale phenomena in catalysis

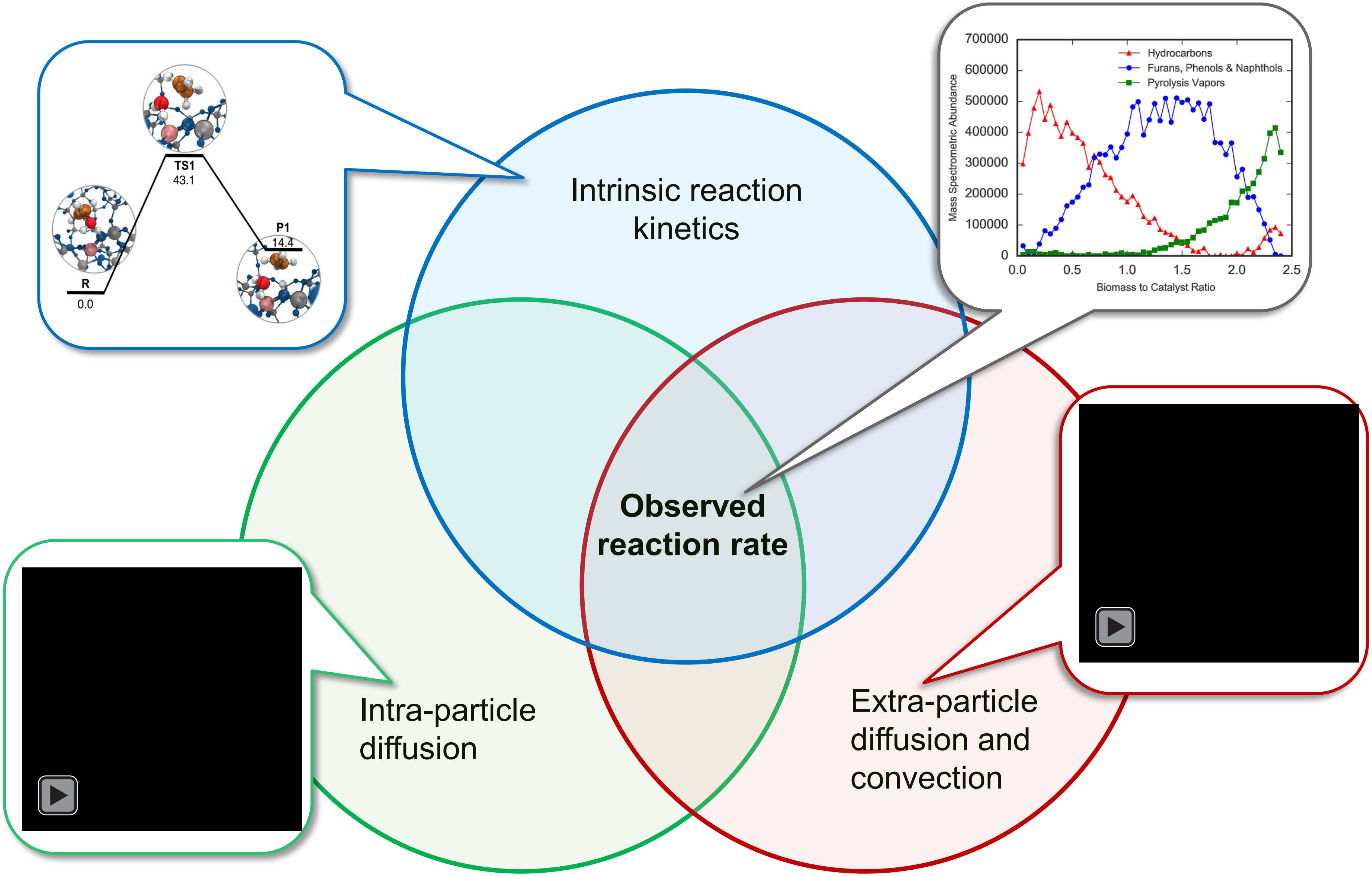


reactor scale



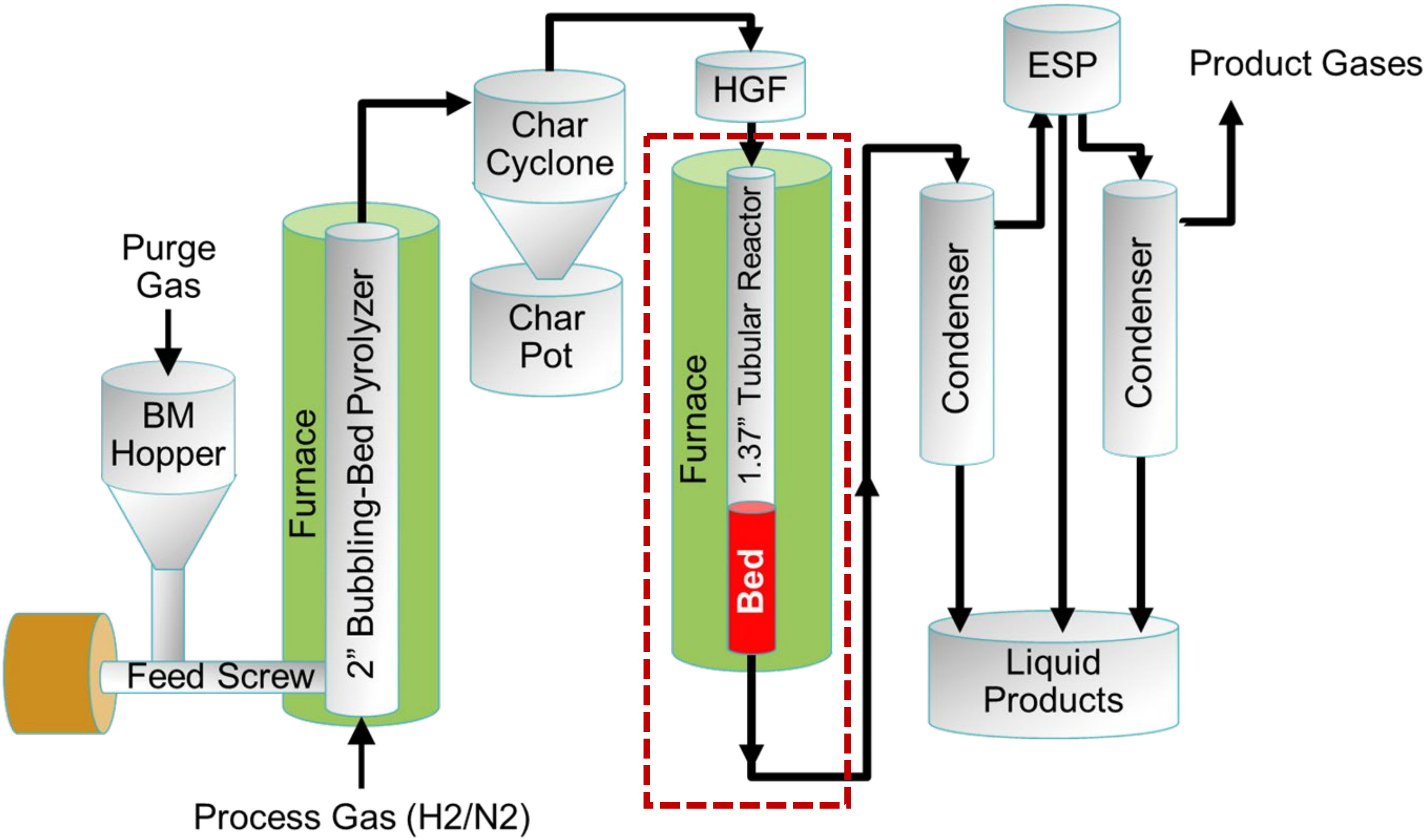
Ciesielski, Pecha, Bharadwaj, et al., Advancing catalytic fast pyrolysis through integrated multiscale modeling and experimentation: Challenges, progress, and perspectives. *Wiley Interdisciplinary Reviews: Energy and Environment* **2018,** *7,* 297.

Observed reaction rate: Physics at all scales



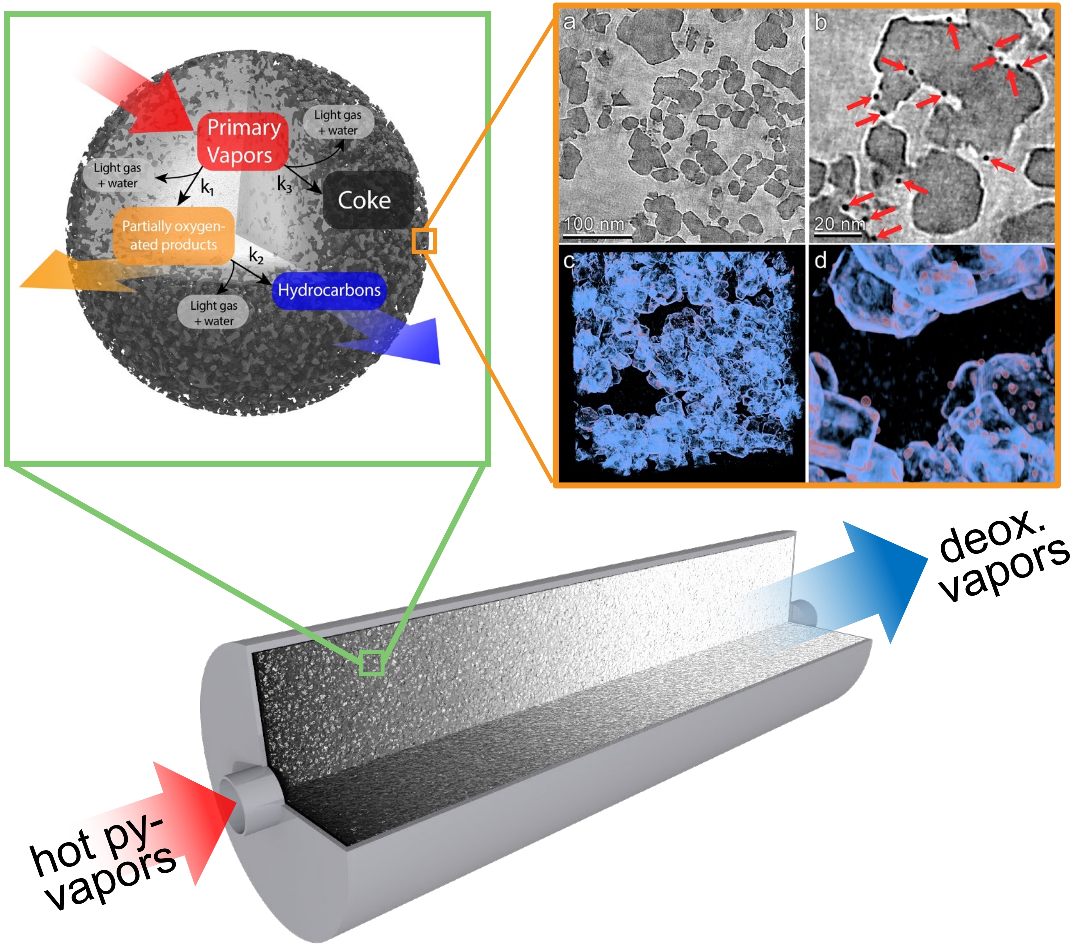
Experimental setup for CFP

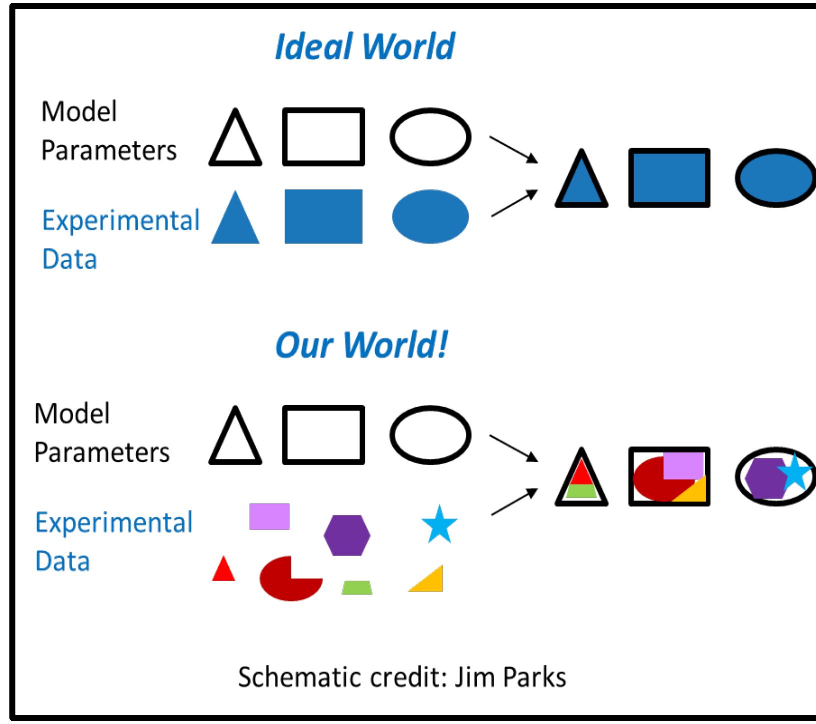
Bench scale *ex-situ* catalytic fast pyrolysis system utilized in this work with a packed bed (fixed bed) of catalyst



Problem description

Packed bed vapor phase upgrading reactor



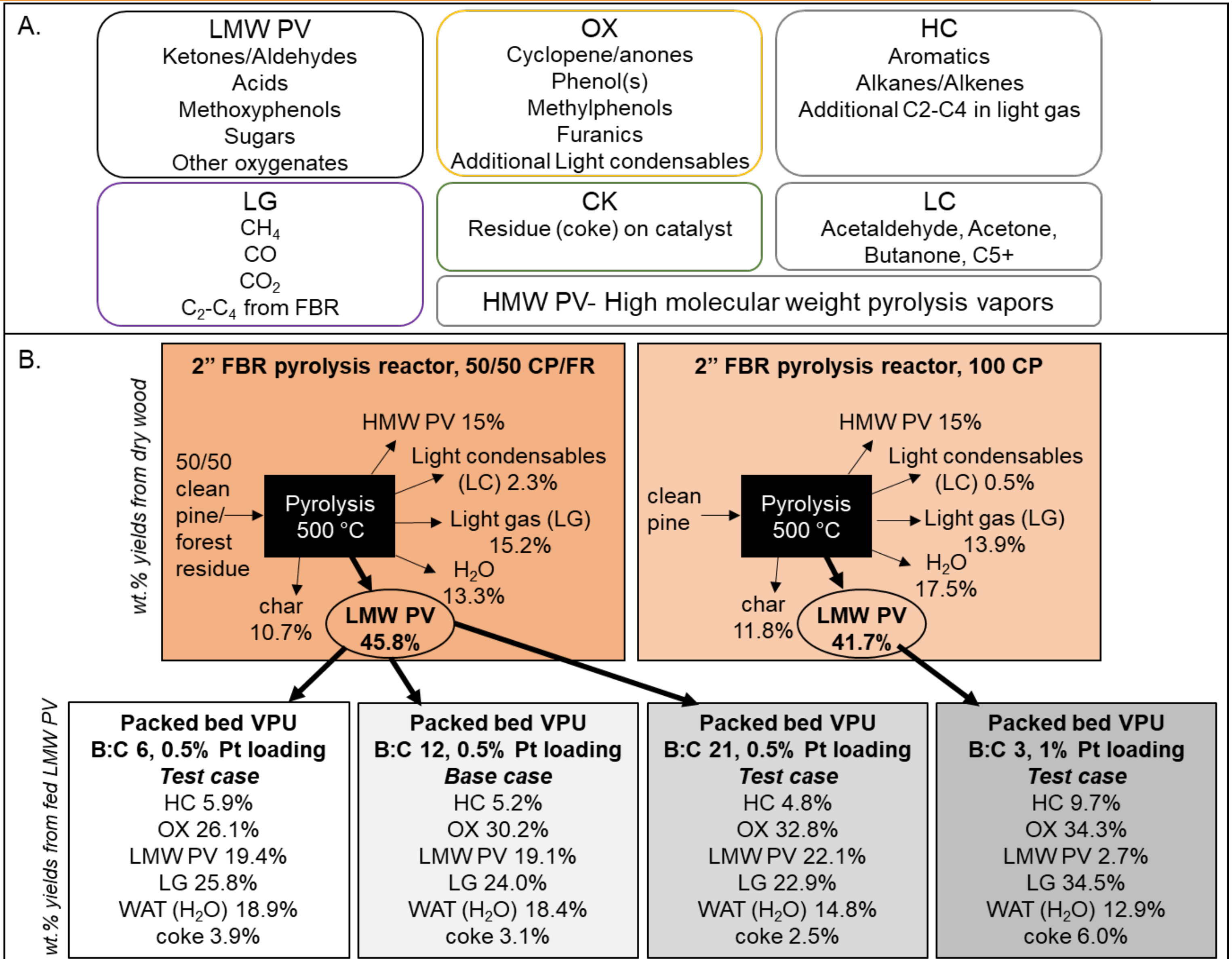


***Relevance*:**

- **Coke profiles predicted by the simulation enable detailed simulation of regeneration cycles.**
- **Transport-independent kinetic parameters enable computational scaling studies and in-silico reactor optimization.**

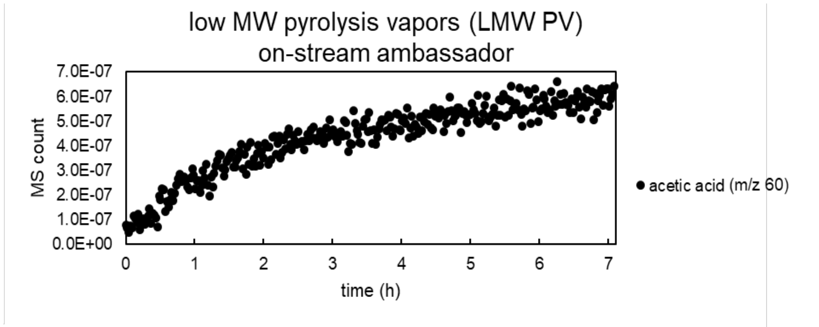
Pecha, Iisa, Griffin, Mukarakate, French, Adkins, Bharadwaj, Crowley, Foust, Schaidle, and Ciesielski. "Ex situ upgrading of pyrolysis vapors over PtTiO 2: extraction of apparent kinetics via hierarchical transport modeling." *Reaction Chemistry and Engineering*, **2020**

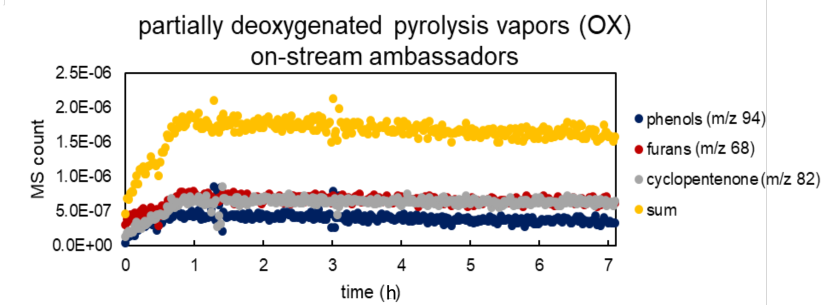
Yields can be broken down into lumps

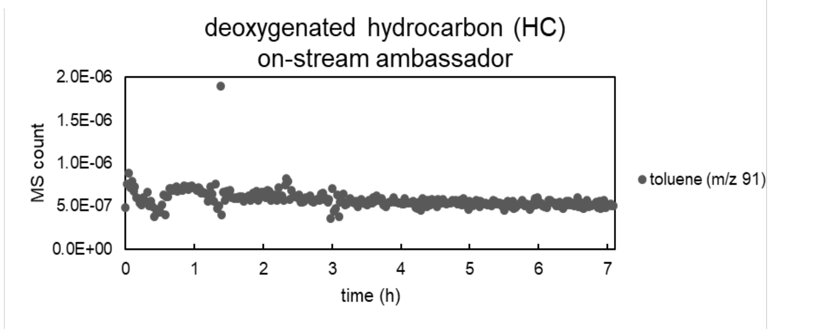


Deactivation and multiple active sites

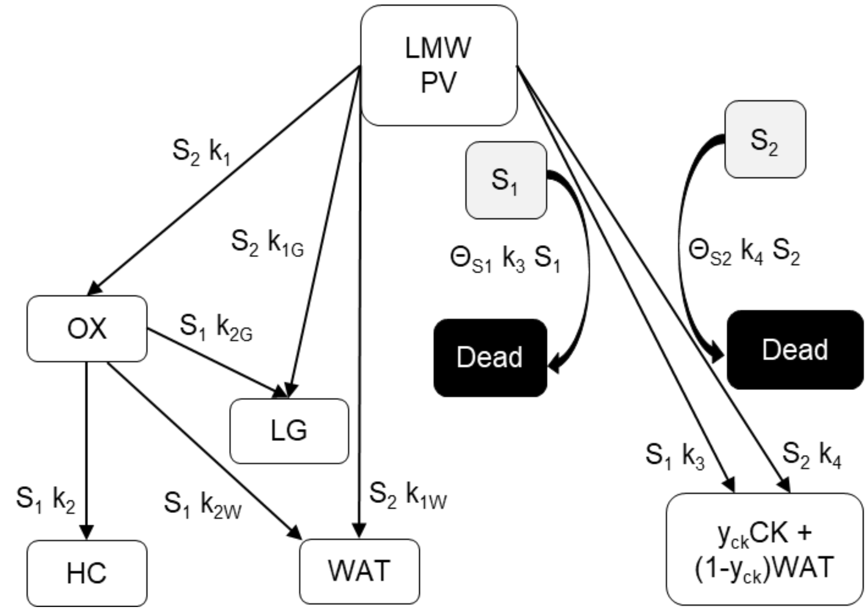
On-stream MS shows rapid deactivation







Lumped reaction scheme describes organic fraction of pyrolysis vapors over PtTiO2



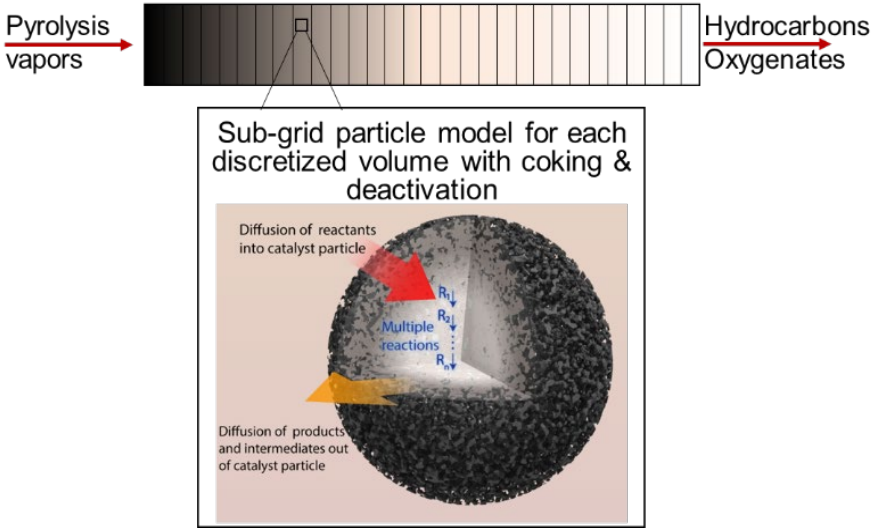
How do changes to catalyst properties and operating conditions impact process performance metrics (yield, composition, catalyst lifetime)?

Modeling approach: Extending the Thiele effectiveness factor

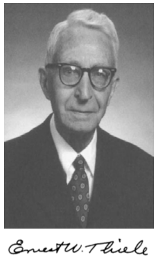
Problem: Accurately model multi-step reactions requires heavy computational resources, not suitable for iterative parameter extraction

Hypothesis: An analytical solution to diffusion-reaction-deactivation is mathematically feasible and will accurately represent multi-step reactions

Solution: Extend the effectiveness factor



State of the art for accounting for diffusion limitations in porous catalysts: Thiele (1930s) + Aris (1970s)



$$\phi = \sqrt{\frac{ka^2}{D_{\text{eff}}}}$$

$$\eta = \frac{3C_{Bi}}{\phi^2}\left(\phi \coth(\phi) - 1\right)$$

$$C_{Bi} = \frac{Bi}{(\phi \coth(\phi) - 1 + Bi)}$$

***No** coupling of intraparticle sequential reactions*

Extending the Thiele effectiveness factor: A bridge between scales

1) Unsteady advection-diffusion-reaction

$$\frac{\partial C_i}{\partial t} + \mathbf{u} \cdot \Delta(C_i) = \Delta \cdot \mathbf{J}_i - \sum_{j=1}^{N} \dot{r}_{ij} + \sum_{m=1}^{N} \dot{r}_{im}$$

2) Assume no advection, sphere

$$\frac{\partial C_i}{\partial t} = \frac{1}{r^2}\frac{\partial}{\partial r}\left(r^2 D_{i,\mathrm{eff}} \frac{\partial C_i}{\partial r}\right) - \sum_{j=1}^{N} \dot{r}_{ij} + \sum_{m=1}^{N} \dot{r}_{im}$$

3) Nondimensionalize, cons. & prod. TM

$$\hat{C}_i = \frac{C_i}{C_{1,\infty}} \qquad \hat{r} = \frac{r}{R_p} \qquad \hat{t}_i = t\frac{D_{i,\mathrm{eff}}}{R_p^2}$$

$$\phi_i = \sqrt{\frac{R_p^2 \psi^t \sum_{j=1}^{N} k_{ij}}{D_{i,\mathrm{eff}}}} \qquad \phi_{im} = \sqrt{\frac{R_p^2 \psi^t k_{im}}{D_{i,\mathrm{eff}}}}$$

4) Quasi-steady state + BCs in sphere (Ω)

$$\frac{d^2\hat{C}_i}{d\hat{r}^2} + \frac{2}{\hat{r}}\frac{d\hat{C}_i}{d\hat{r}} - \phi_i^2 \hat{C}_i = -\sum_{m}^{N} \phi_{im}^2 \hat{C}_m \qquad \text{in } \Omega$$

$$\frac{d\hat{C}_i}{d\hat{r}} = 0 \qquad \text{on } \partial\Omega_1$$

$$\frac{d\hat{C}_i}{d\hat{r}} = Bi\left(1 - \hat{C}_i\right) \qquad \text{on } \partial\Omega_2$$

5) Use matrix-vector form (matrix of Thiele moduli for consumption-production)

$$\frac{d^2\hat{\mathbf{C}}}{d\hat{r}^2} + \frac{2}{\hat{r}}\frac{d\hat{\mathbf{C}}}{d\hat{r}} - \bar{\phi}^2 \hat{\mathbf{C}} = 0$$

Lattanzi A, Pecha MB, Bharadwaj VS, Ciesielski PN, "Beyond the effectiveness factor: multi-step reactions with intraparticle diffusion limitations," *Chemical Engineering Journal* (2020) 380, 15, 122507.

Extending the Thiele effectiveness factor: A bridge between scales

6) When eigenvalues (λ) are real, solution is hyperbolic function

$$\hat{U}_i = A_1 \sinh(\sqrt{\lambda_i}\hat{r}) + A_2 \cosh(\sqrt{\lambda_i}\hat{r}) \qquad \lambda_i > 0$$

7) Converting back to concentration & BCs (P is eigenvector matrix)

$$\hat{\mathbf{C}} = \bar{\mathbf{P}}\bar{\mathbf{D}} \left(\frac{C_{Bi} \sinh\left(\sqrt{\lambda}\hat{r}\right)}{\sinh\left(\sqrt{\lambda}\right)\hat{r}} \right) \bar{\mathbf{P}}^{-1} \hat{\mathbf{C}}_{\mathrm{Rat},\infty}$$

8) Volume-averaging the rates

$$< \dot{r}_{ij} >\equiv \frac{4\pi R_p^3 \psi k_{ij} C_{1,\infty}}{4/3\pi R_p^3} \int_0^1 \hat{C}_i \hat{r}^2 \, d\hat{r} = \psi k_{ij} C_{1,\infty} \eta_i,$$

9) Multi-step effectiveness vector! (MEV)

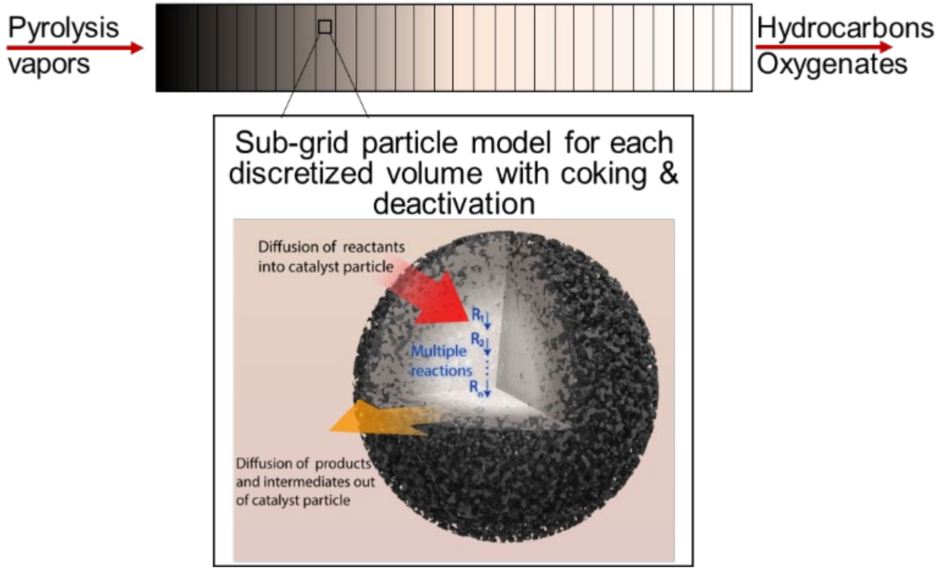
$$\boldsymbol{\eta} = \bar{\mathbf{P}}\bar{\mathbf{D}} \left(\frac{3C_{Bi}}{\lambda} \left(\sqrt{\lambda} \coth(\sqrt{\lambda}) - 1 \right) \right) \bar{\mathbf{P}}^{-1} \hat{\mathbf{C}}_{\mathrm{Rat},\infty}$$

10) Individual rates with MEV!

$$< \dot{r}_i >\equiv \sum_m < \dot{r}_{im} > - \sum_j < \dot{r}_{ij} >= \psi C_{1,\infty} \left(\sum_m k_{im}\eta_m - \sum_j k_{ij}\eta_i \right)$$

Lattanzi A, Pecha MB, Bharadwaj VS, Ciesielski PN, "Beyond the effectiveness factor: multi-step reactions with intraparticle diffusion limitations," *Chemical Engineering Journal* (2020) 380, 15, 122507.

Apply multistep effectiveness vector to PBR



Apparent rate equations inside MEV formulation

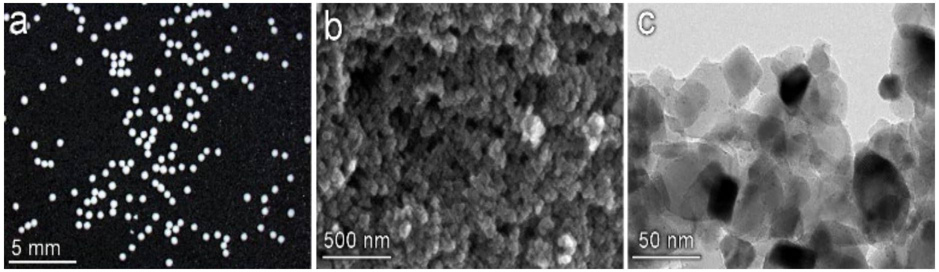
$$R_1 = PV_{LMW} k_1 S_2$$
$$R_{1G} = PV_{LMW} k_{1G} S_2$$
$$R_{1W} = PV_{LMW} k_{1W} S_2$$
$$R_2 = OX\ k_2 S_1$$
$$R_{2G} = OX\ k_{2G} S_1$$
$$R_{2W} = OX\ k_{2W} S_1$$
$$R_3 = PV_{LMW} k_3 S_1$$
$$R_4 = PV_{LMW} k_4 S_2$$

Packed bed transport equations

$$\frac{\partial PV_{LMW}}{\partial t} = -u \frac{\partial PV_{LMW}}{\partial x} + D_{PV} \frac{\partial^2 PV_{LMW}}{\partial x^2} - R_{PV_{LMW},eff}\left(1-\varepsilon_p\right)$$
$$\frac{\partial OX}{\partial t} = -u \frac{\partial OX}{\partial x} + D_{OX} \frac{\partial^2 OX}{\partial x^2} - R_{OX,eff}\left(1-\varepsilon_p\right)$$
$$\frac{\partial HC}{\partial t} = -u \frac{\partial HC}{\partial x} + D_{HC} \frac{\partial^2 HC}{\partial x^2} - R_{HC,eff}\left(1-\varepsilon_p\right)$$
$$\frac{\partial LG}{\partial t} = -u \frac{\partial LG}{\partial x} + D_{LG} \frac{\partial^2 LG}{\partial x^2} - R_{LG,eff}\left(1-\varepsilon_p\right)$$
$$\frac{\partial WAT}{\partial t} = -u \frac{\partial WAT}{\partial x} + D_{WAT} \frac{\partial^2 WAT}{\partial x^2} - R_{WAT,eff}\left(1-\varepsilon_p\right)$$
$$\frac{\partial S1}{\partial t} = R_{S1,eff}\left(1-\varepsilon_p\right)$$
$$\frac{\partial S2}{\partial t} = R_{S2,eff}\left(1-\varepsilon_p\right)$$
$$\frac{\partial CK}{\partial t} = R_{CK,eff}\left(1-\varepsilon_p\right)$$
$$PV_{LMW} = PV_{LMW,0},\ x = 0$$
$$HC = OX = LG = WAT = 0,\ x = 0$$
$$\frac{dPV}{dt} = \frac{dOX}{dt} = \frac{dHC}{dt} = \frac{dLG}{dt} = \frac{dWAT}{dt} = 0,\ x/L = 1$$

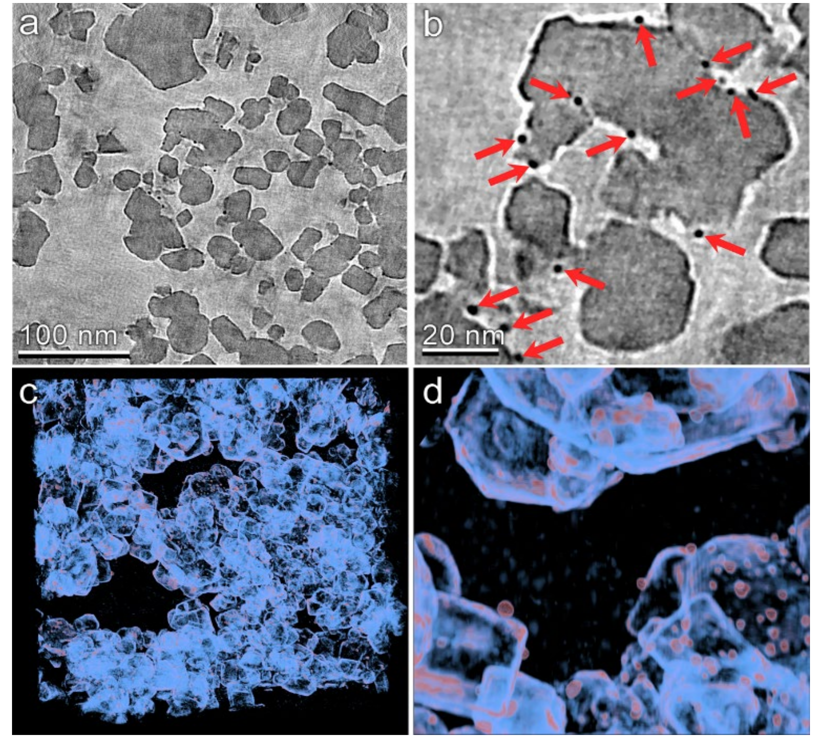
Catalyst characterization

Multiscale imaging of the Pt/TiO2 catalyst particles



(a) Light microscopy of catalyst particles showing the spherical bulk geometry with narrow size distribution. (b) Scanning electron microscopy (SEM) of the particle surface reveals a porous support structure formed by the agglomeration of TiO2 nanoparticles. (c) Transmission electron microscopy shows the presence of ~5 nm Pt particles visualized as dark spots on the surface of the larger TiO_2 support structure.

TEM Tomography of the TiO_2 catalyst particle mesostructure



(**a, b**) Slices through the tomographic volume are shown at two different magnifications. Pt particles are clearly identified by their higher electron density (indicated by red arrows in panel b). (**c, d**) 3D visualizations of the reconstructed volume are shown at two different magnifications.

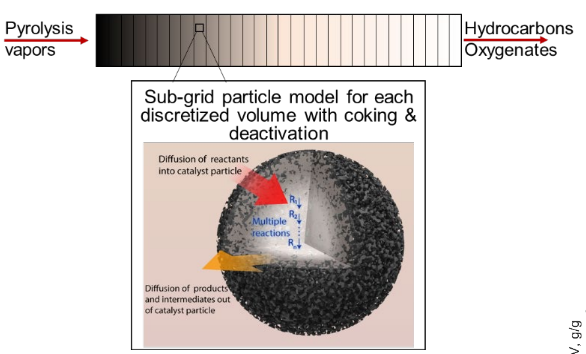
Apparent rate constants fit to real data

Initial guess for 10 apparent rate parameters + experimental data

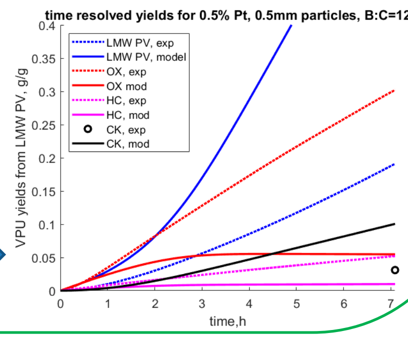
Simplex parameter optimizer + PBR model

New parameters from optimizer

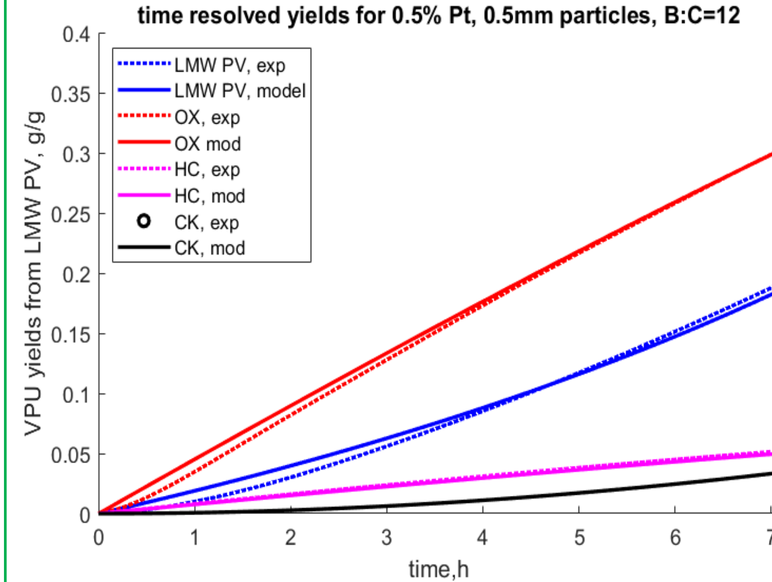
Multiscale packed bed model



Objective function (yield vs time)



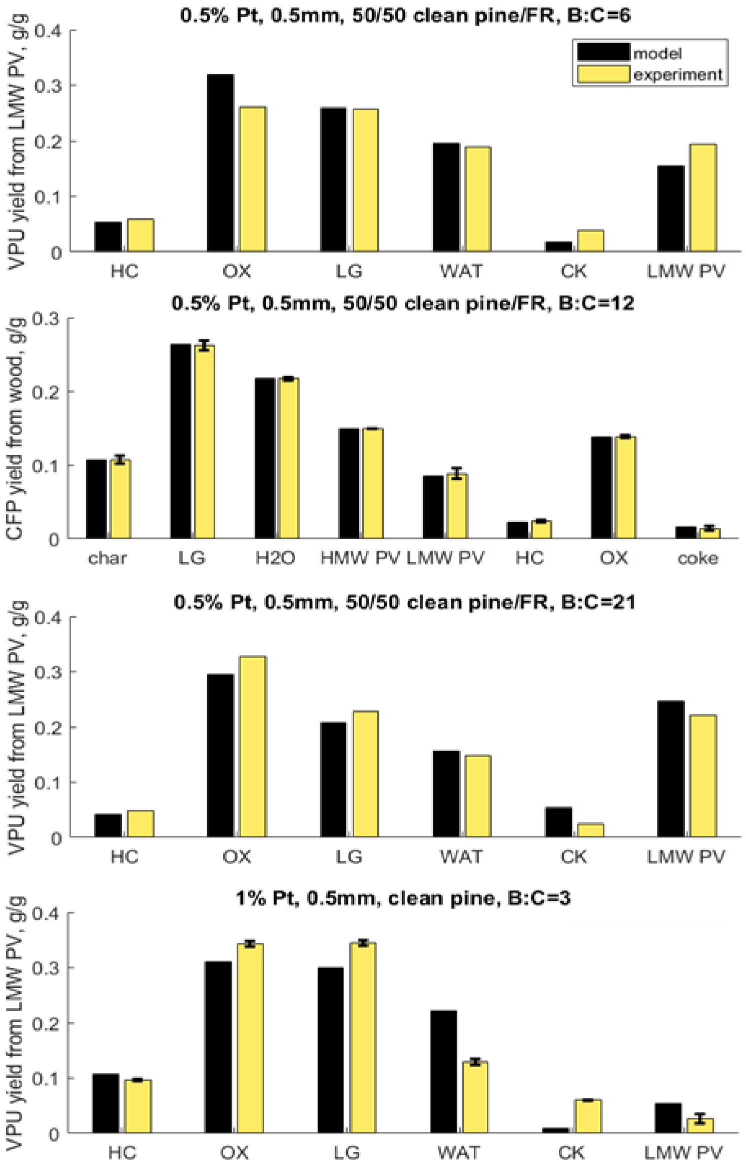
Best-fit lumped rate constants fit for base case



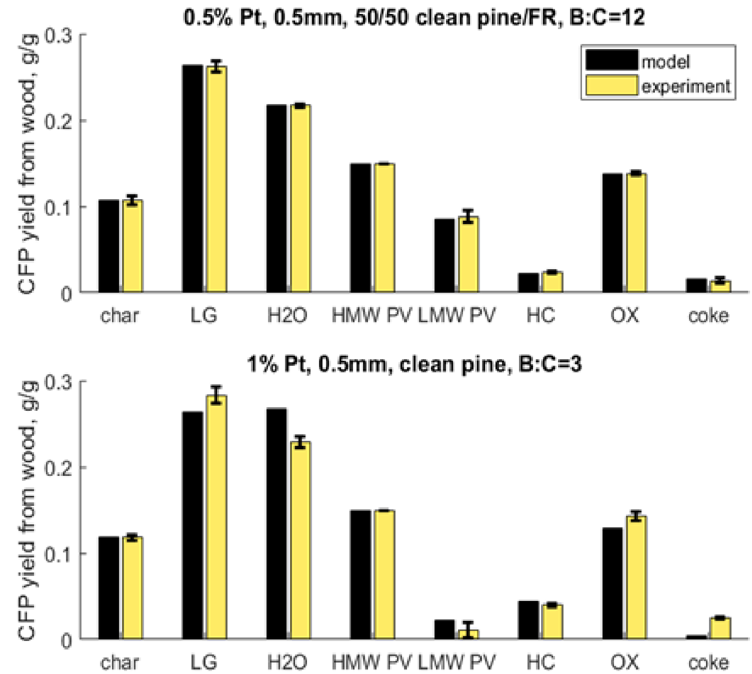
RATE CONSTANT	FITTED VALUE
k1 [s^{-1}]	76
k1g [s^{-1}]	50.5
k1w [s^{-1}]	39
k2 [s^{-1}]	5.4
k2g [s^{-1}]	0.7
k2w [s^{-1}]	7.9E-10
k3 [s^{-1}]	7E-14
k4 [s^{-1}]	3.7E-4
Θ_{S1}	1.2E-3
Θ_{S2}	15.2

Results: Model validation

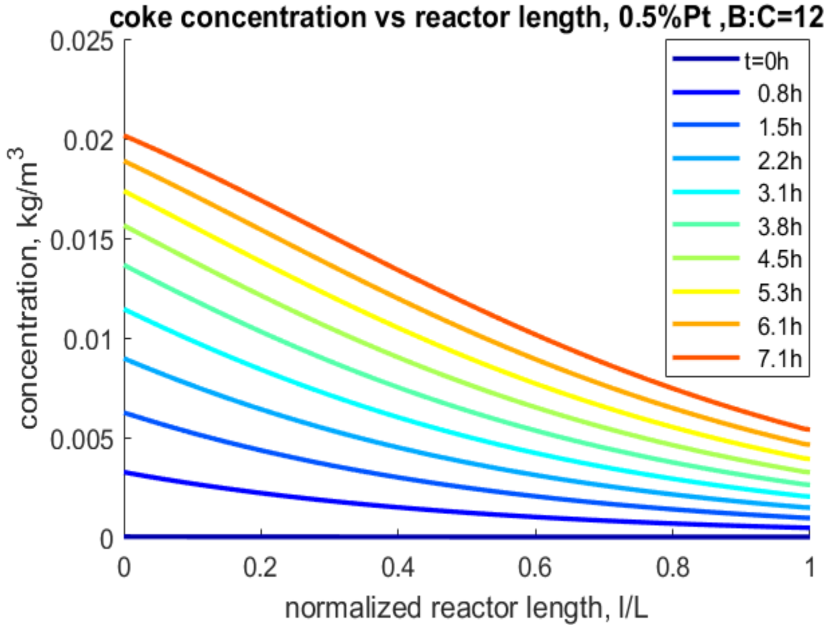
Yields from low MW pyrolysis vapors, VPU only

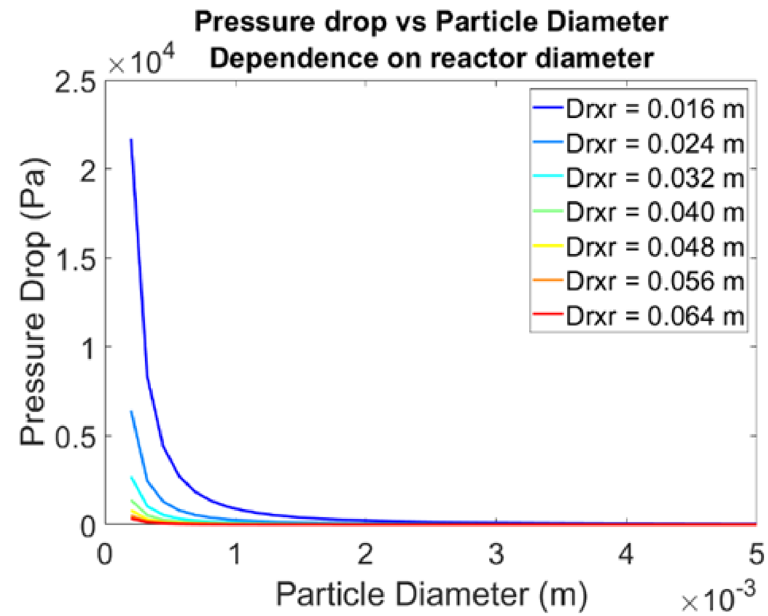


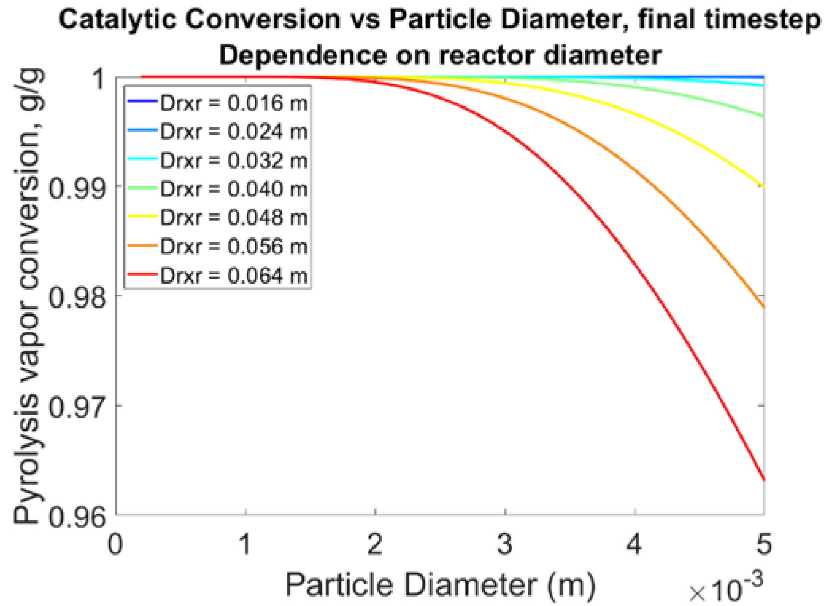
Yields from dry wood for pyrolysis + VPU



Results: Predictions and extrapolations





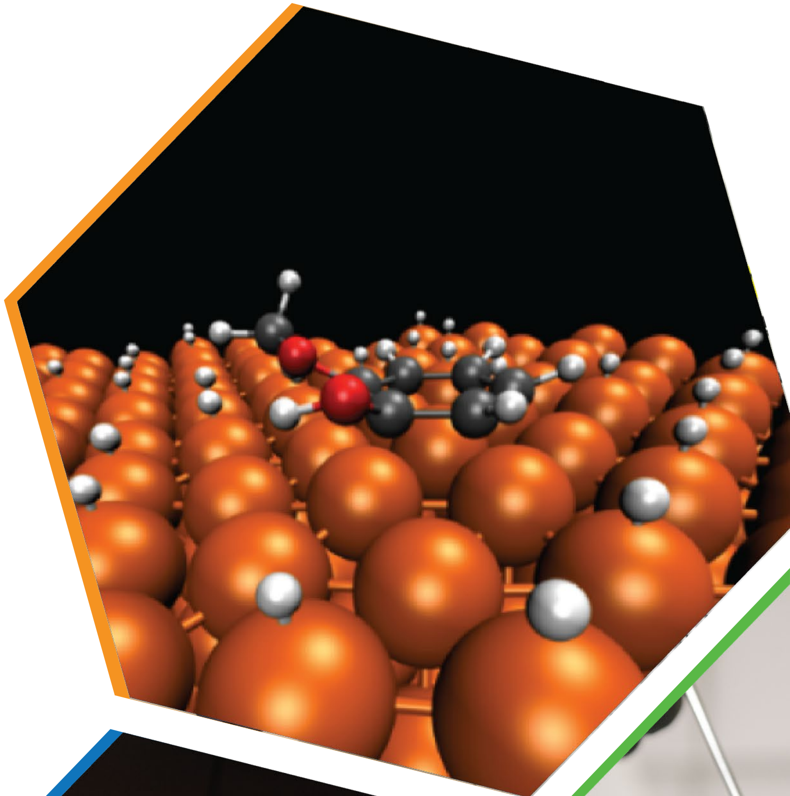


Conclusions

- New multiscale simulation framework was capable of capturing
  - multiple cascading reactions
  - multiple operating conditions
  - catalyst loadings
  - active site deactivation
- Fast, accurate, can be used to mine old *good* data
- Future work will extend the model to other catalyst shapes, other technologies
- In the next slides, you will see how results from this work were used to design a catalytic regeneration system at a much larger scale with a different set of modeling tools.

ChemCatBio

Chemical Catalysis for Bioenergy

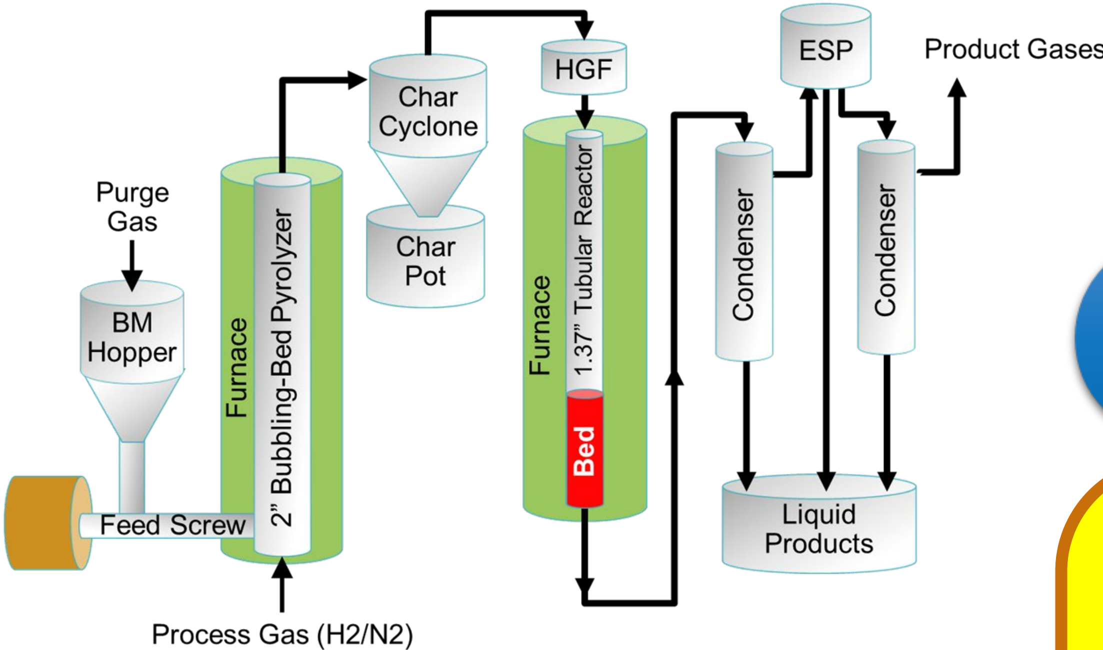


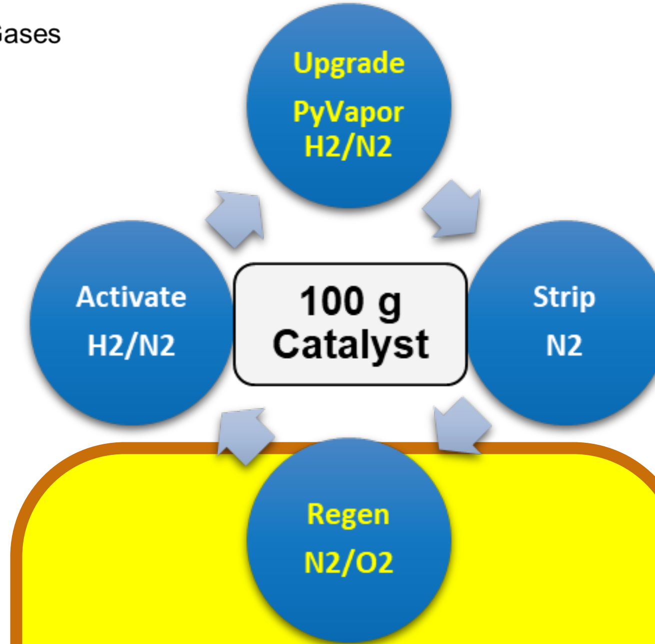
Packed Bed Reactor Scale-up Using High Fidelity Reactor Models

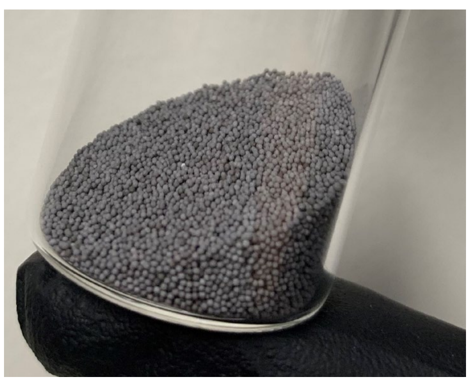
U.S. DEPARTMENT OF ENERGY | *Office of* ENERGY EFFICIENCY & RENEWABLE ENERGY

BIOENERGY TECHNOLOGIES OFFICE

Lab Scale Packed Bed Reactor (PBR) and Catalyst







0.5 mm Pt/TiO_2 Spheres

Upgrading	
Biomass Feed, g/hr	150
Inlet Pressure, kPa	110
Inlet Temperature, °C	410
H2 Flowrate, SLPM	13.5
N2 Flowrate, SLPM	2.4
WHSV, hr^{-1}	1.5
Duration, B/C (hr)	12 (8)

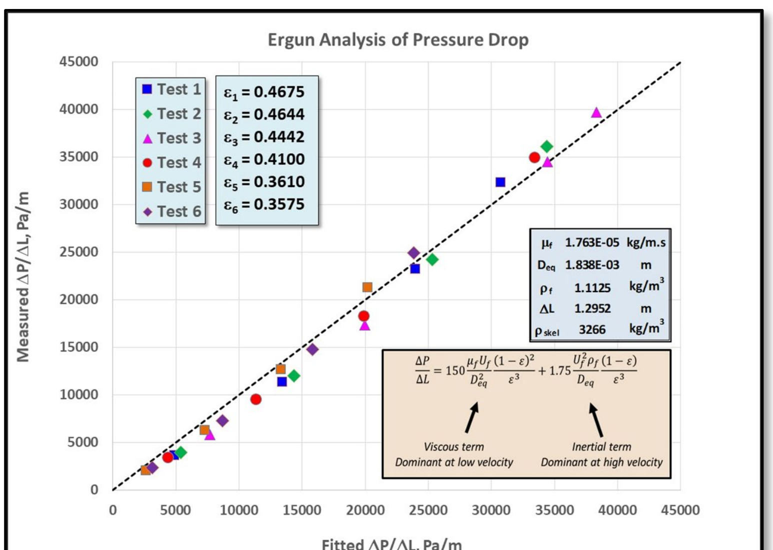
Oxidative Regeneration	
Inlet Temperature, °C	410
Outlet Temperature, °C	450
N2 Flowrate, SLPM	15.6
Air Flowrate, SLPM	0.42
Duration, hr	8

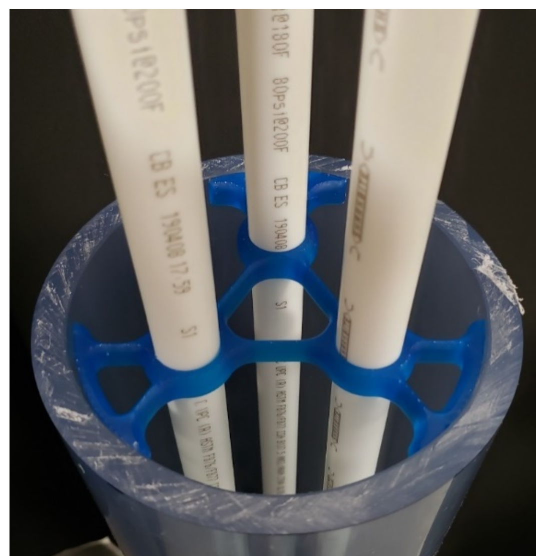
Scaling Up the PBR

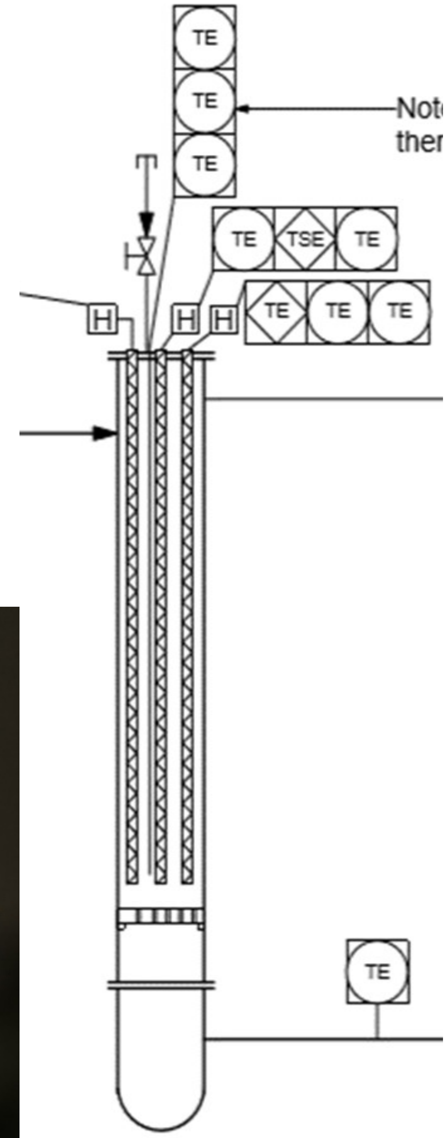
- TCPDU-PBR
  - 6 kg cat
  - 9 kg/hr biomass
  - WHSV 1.5 hr^{-1}
- Constraints
  - PBR ΔP 20 kPa or less
  - No wall heat removal (mimic industrial scale)
  - Gas temperature ≈ 400°C to minimize cycle time and ensure quick light-off
  - B/C = 12 corresponds to 25 wt% coke (g C / g fresh)

Scaling Up the PBR (2)

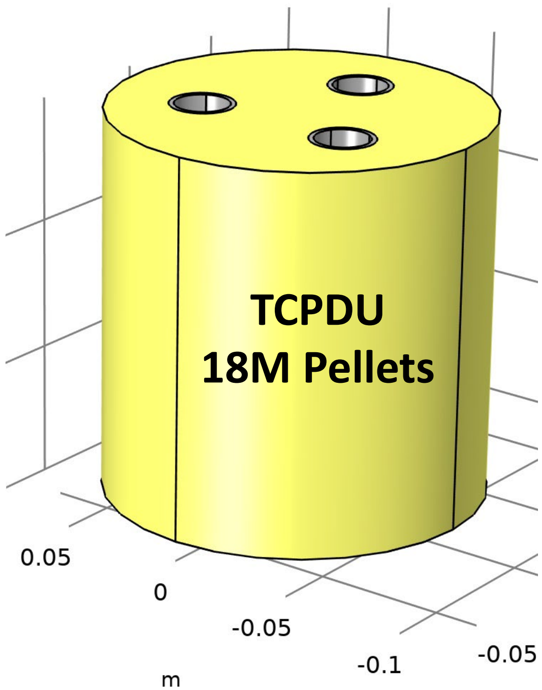
- Split the 6 kg bed between 3 existing reactors, 2 kg each
- Per-bed scale-up = 20 X
- N2 flow limit = 1200 SLPM, 400 kg per bed
- Each reactor has 3 heating rods which can be converted to cooling tubes
- Air flow limit = 1800 SLPM, 200 per tube

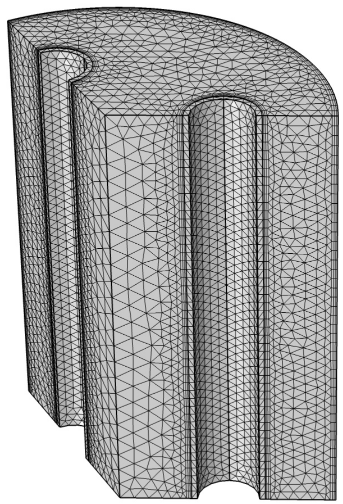






Model Details





n_{cell} = 152k $n_{pellet/cell}$ = 120

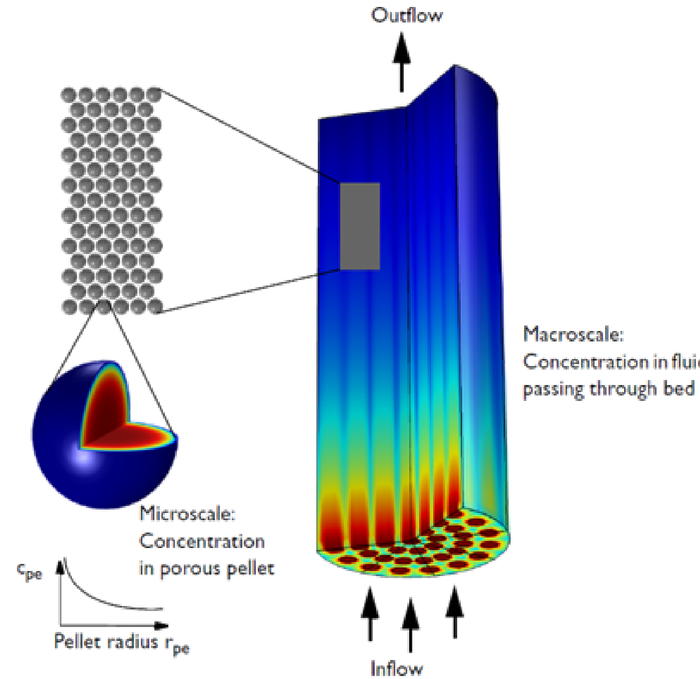
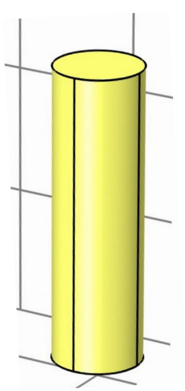


Image made using COMSOL Multiphysics® software and provided courtesy of COMSOL.[26]



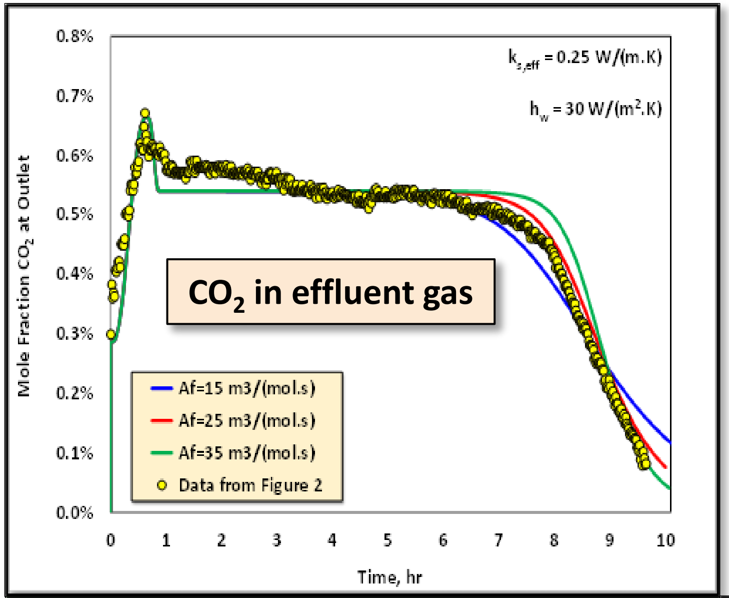
2FBR
900k Pellets

n_{cell} = 434k $n_{pellet/cell}$ = 42

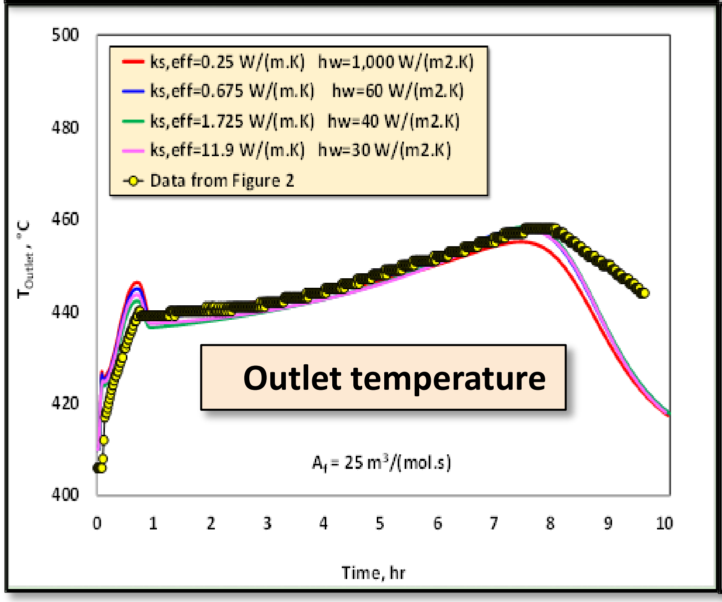
N2 Flow, SLPM (410°C)	Cooling Air Flow, SLPM (30°C)		
	600	300	No Flow
400	Case 1	Case 2	Case 3
300	Case 4	Case 5	Case 6
200	Case 7	Case 8	Case 9

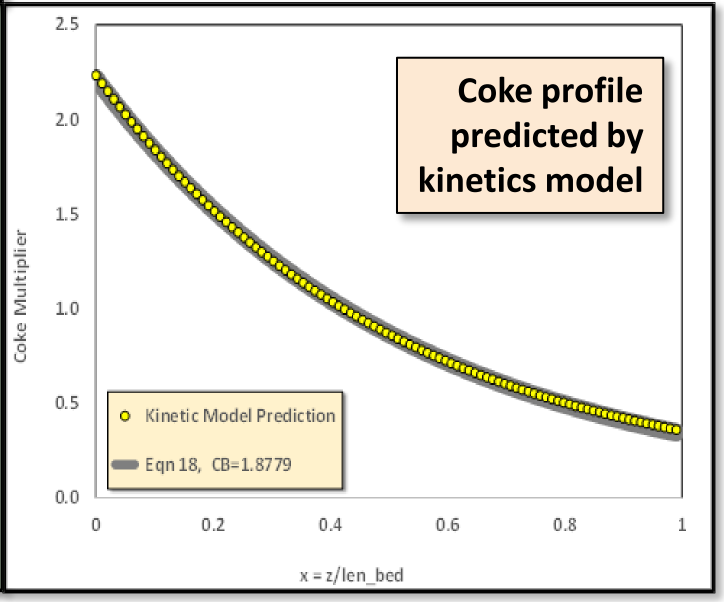
2FBR Data Used in Model Development

Catalyst / Bed Parameters	
A_f, m³/(mol.s)	25
E_a, J/mol	$5x10^4$
ABD, kg/m³	900
ρ_{skel}, kg/m³	3,900
ρ_{pe}, kg/m³	1,900
Pellet porosity	0.592
Bed voidage	0.437
Total voidage	0.770
BET, m²/g	54
Pore diam, nm	27

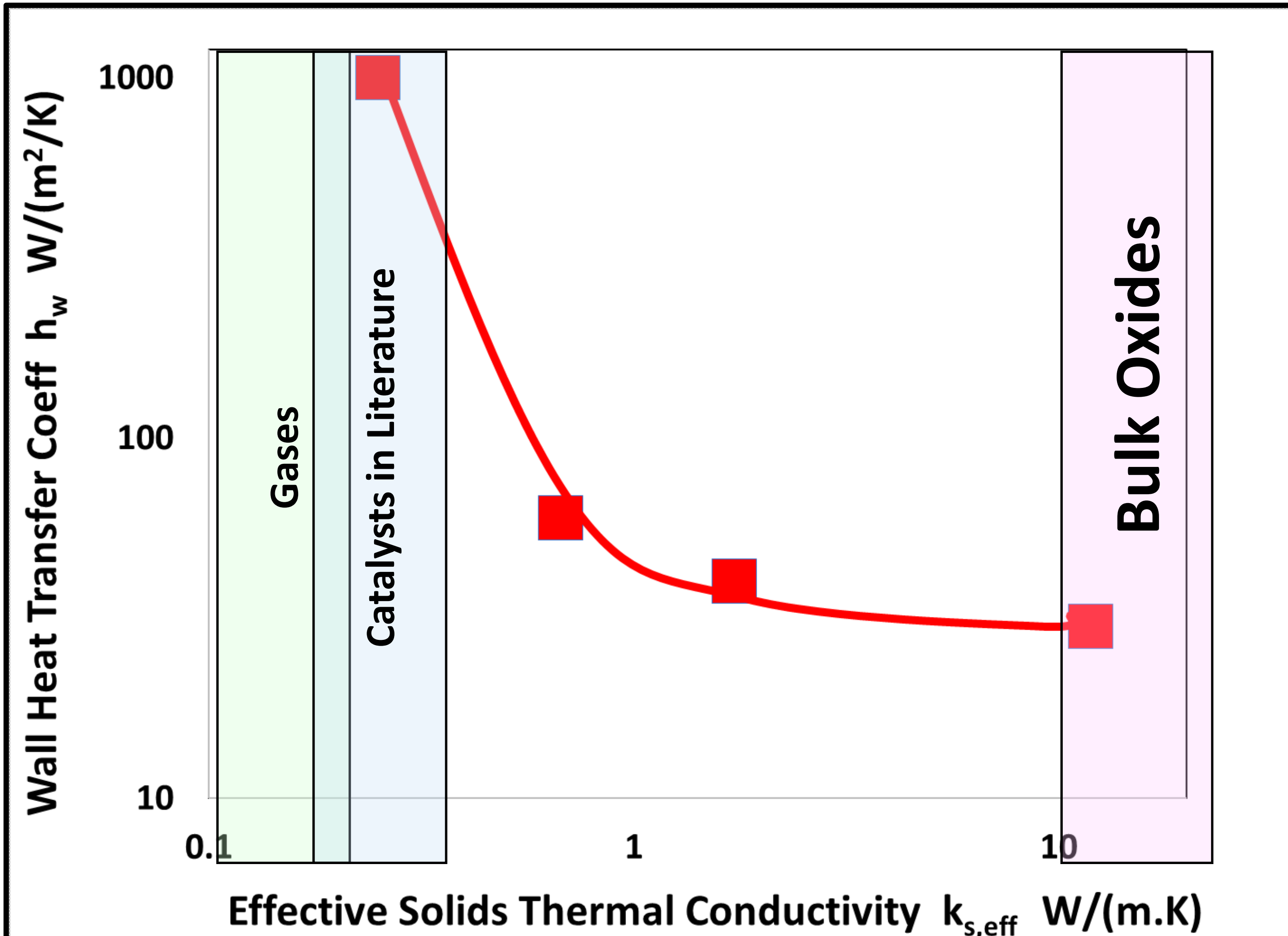


Thermal Parameters	
ΔH, J/mol	$3.94x10^5$
Thermal conductivity, W/(m.K)	$k_{s,eff}$
Wall heat transfer, W/(m².K)	h_w
Bulk heat capacity, J/(kg.K)	680

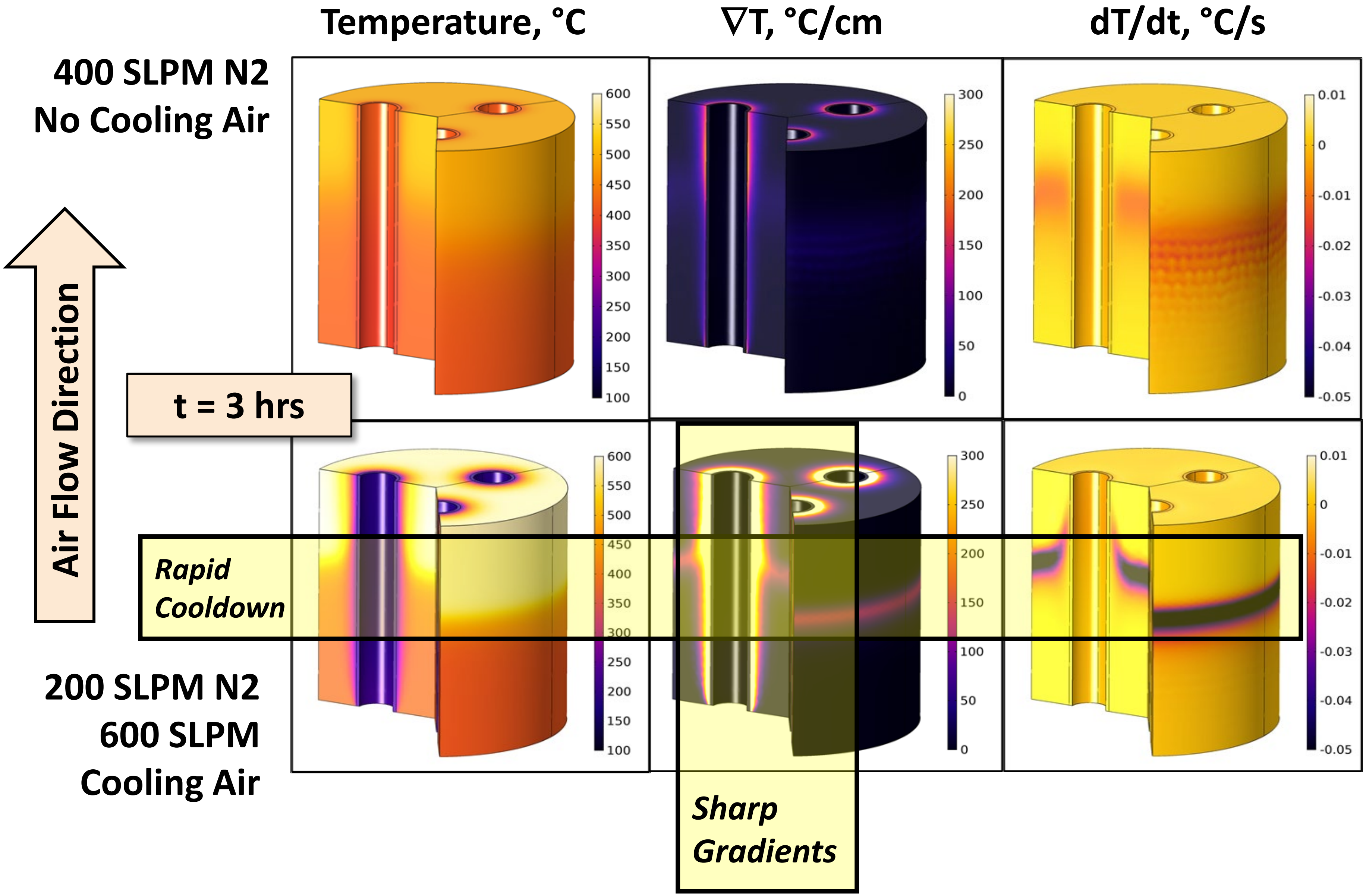




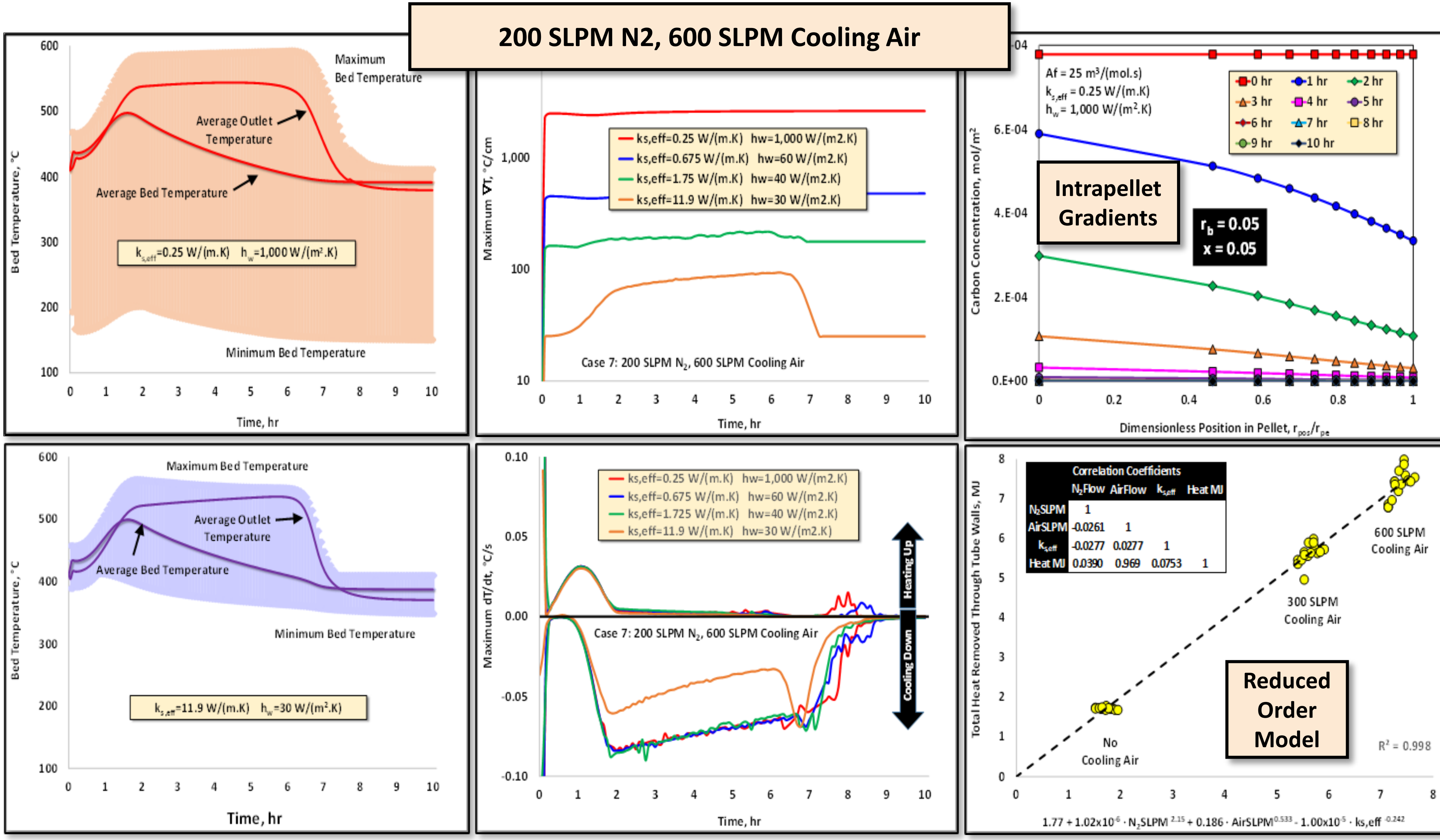
Biggest Unknown: Heat Transfer Parameters



TCPDU Model Predictions



TCPDU Model Predictions (2)



B.D. Adkins et.al, ***Predicting thermal excursions during in-situ oxidative regeneration of packed bed catalytic fast pyrolysis catalyst,*** **submitted to *Reaction Chemistry and Engineering***

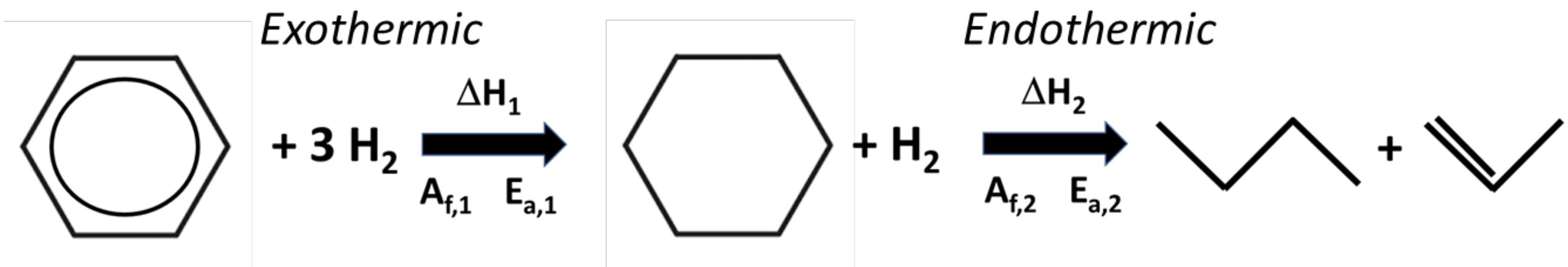
Conclusions

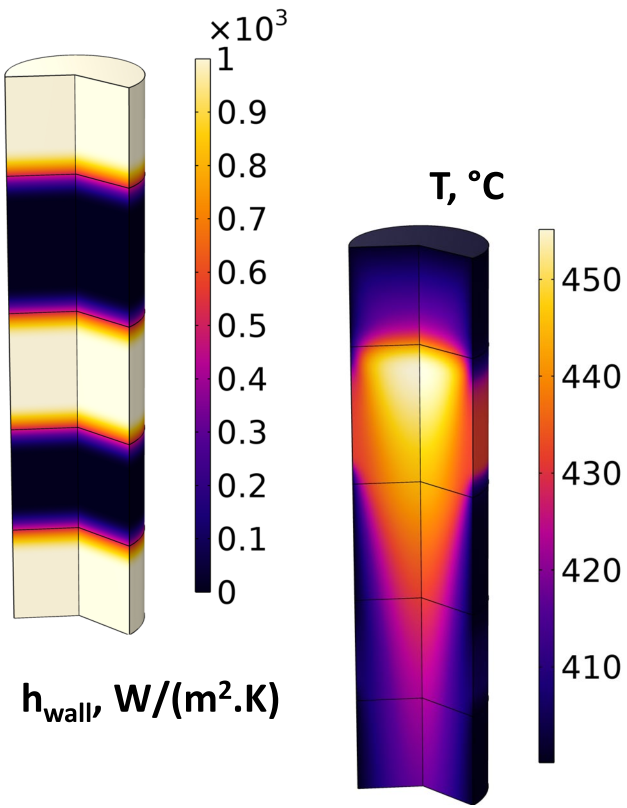
1. **Risk of catalyst damage and/or accelerated irreversible deactivation from thermal excursion is high in proposed TCPDU design**
   - **Pressure drop associated with small catalyst particle size (0.5 mm) constrains bed depth and process gas flow rate, both of which constrain heat removal**
2. **Potential design improvements**
   - **Construct reduced order models and throughly map catalyst / bed design space**
   - **Evaluate moving bed alternatives to packed bed. Not fluid bed: more like Continuous Catalytic Reformers (CCRs)**
3. **Although small by industry standards, a scale-up factor of 20 can be substantial, as demonstrated here**

Improvements in Reactor-Scale Models

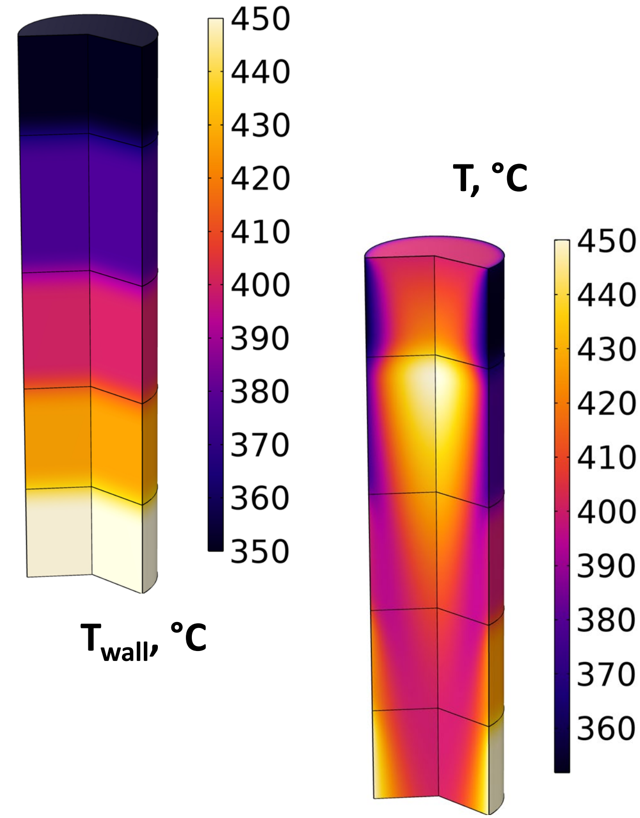
1. **Firm up conclusions from regen model by addressing key unknowns**
   - **Thermal conductivity of catalyst pellets**
     - **Experimental measurements**
     - **High resolution mesoscale modelling of heat transfer**
   - **Coke distribution**
     - **Bed dissection**
     - **Carbon distribution in pellet interiors**
2. **Expand model to include stacked beds with multiple catalysts**

Stacked Bed Model

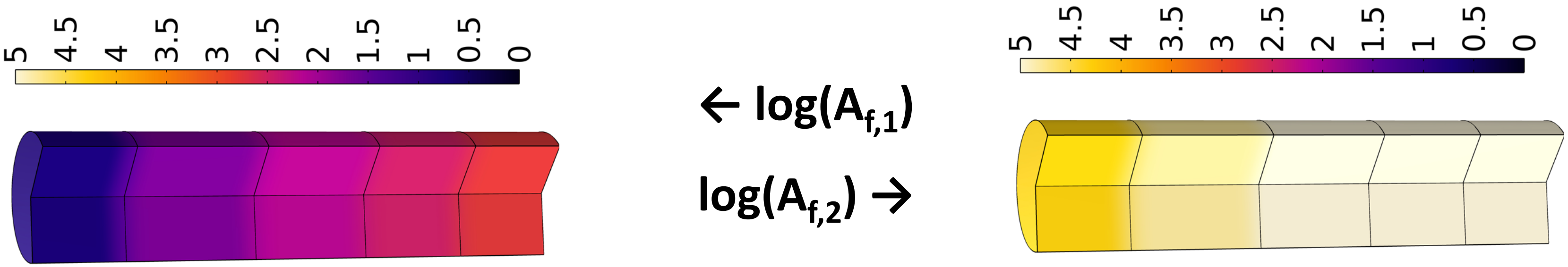


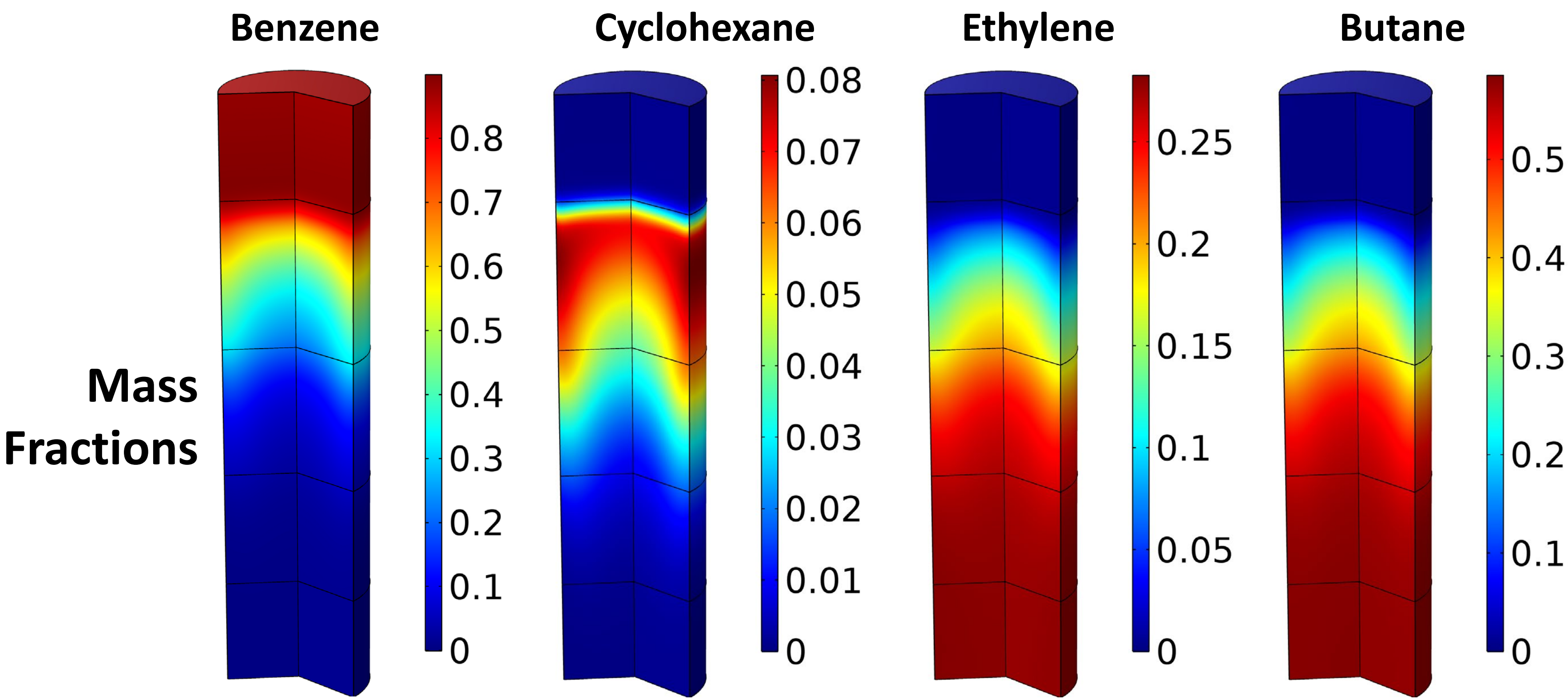


Bed-Specific Parameters
Mass of catalyst
ABD
Skeletal density
Particle size
Particle shape
Pore volume
Surface area
Pore diameter
Porosity
Void fraction
Thermal conductivity
Heat capacity
Wall heat transfer coeff.
Reaction rate constants
Reaction enthalpy
…



Stacked Bed Model





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Pacific Northwest NATIONAL LABORATORY
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Thank you. Let's Discuss.

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