Pilot-scale CFP Commissioning: Creative Problem Solving and Lessons Learned

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Staged Multi-Scale Evaluation Improves Research Efficiency

**Microscale Reactor**

- Catalyst: 1g
- Biomass: 25 mg/run
- **Uses/Purpose:**
  - Rapid catalyst screening
  - Preliminary product analysis (no condensed oil)
  - Batch experiments
  - Mechanistic insight

**Laboratory Scale Fluid/Fixed Bed Reactor**

- Catalyst: 500g
- Biomass: 0.5 kg/h
- **Uses/Purpose:**
  - Catalyst evaluation with continuous biomass feed
  - Assess operating conditions
  - Full product/yield analysis
  - Extended time on stream
  - Fixed/Fluidized bed (not representative of riser)

**DCR (Small Pilot Scale)**

- Catalyst: 2kg
- Biomass: 3 kg/h
- **Uses/Purpose:**
  - Process evaluation/integration with industrially-relevant riser
  - Assess operating conditions compared to lab-scale
  - Co-processing of biomass and petroleum feeds (liq. and gas)

**R-Cubed (Pilot Scale)**

- Catalyst: 100kg
- Biomass: 20 kg/h
- **Uses/Purpose:**
  - Evaluation of process operability/uptime
  - Identification & assessment of scale-up challenges & impacts
  - Generate significant product quantities

**Process and catalyst evaluation at multiple scales:**

- Improves research efficiency, thus reducing cost
- Provides data that is directly transferrable to industry partners
- Allows for a tiered catalyst and process development approach
Challenges / Creative Problem Solving

• Design Constraints
• Measuring & controlling catalyst flow rate
• Pressure & level control
• Plugging in exit lines
• Air regeneration – complete coke combustion for catalyst efficiency
Design Constraints

- Must have built-in flexibility
- Ceiling height limit
  - Must account for thermal expansion
- Floor loading limit (Techlok flanges)
- BPVC: limited to 6-in. diameter pipe
- Highly fluidizable catalyst (Zeolite)
R³ Process Flow Diagram

Gas Exits

Air
Regen. (FBR)

4-in
Riser

Catalyst
Flow Path

Steam
Stripper

3-in
Transfer
Riser
R³ Process Flow Diagram

Catalyst Level measured by DP

Regen. DP

Stripper DP

Riser DP
How to Measure Catalyst Mass Flow

- Loaded 5 kg shots of catalyst
- 1 kg = 0.64 kPa at 60 kPa
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- 1 kg = 0.64 kPa at 60 kPa

\[ y = 0.64x + 13.42 \]
\[ R^2 = 1.00 \]
Catalyst mass flow required for kinetics

- Timed the transfer of catalyst from one FBR to other
- Simulated high/mid/low process gas flows with \( \text{N}_2 \)

\[
\dot{M} = (\Delta P_{\text{start}} - \Delta P_{\text{end}}) \times \frac{1 \text{ kg}}{0.64 \text{ kPa}} \times \frac{1}{\Delta t}
\]
Controlling catalyst flow via slide valves

• First generation: temperature at Riser exit (TE709B) controlled catalyst flow
  – Difficult due to thermal mass of riser & external heaters (thermocouple not sensitive to catalyst flow)
• Next generation: keeps catalyst flowrate constant, as measured by DP across riser (PDIT700)
Pressure Control in Fluidized Beds (Regen/Stripper)

REGENERATOR

CYCLONE OUTLET 60.3 kPa
MUST BE GREATER THAN

REGEN. LEVEL 7.2 kPa

FCV742 63.95% Open

PDIT742 8.61

DP across slide valve must be positive

\[ y = 0.97x - 3.81 \]

\[ R^2 = 0.99 \]

\[ 0 \quad 5 \quad 10 \quad 15 \]

CYCLONE DP, kPa

\[ 0 \quad 1 \quad 2 \quad 3 \quad 4 \quad 5 \quad 6 \quad 7 \quad 8 \]

LEVEL, kPa

\[ 3.3 \text{ kPa} \]

\[ 7.2 \text{ kPa} \]
Level Control & Maximum Level in FBRs

- Regen. Cyclone Outlet
- Regen. Headspace
- Regen. Level increases as Stripper Level decreases
- Max level in Regenerator plugs Sidearm
- ~13 kg of catalyst out cyclone gas exit
- DP across cyclone inverts
Hot Gas Filtration

• Catalyst + water = Mud = Plugged Sidearms = Catalyst out

• Hot Catalyst + water vapor = ok

• Required on ALL exit streams

CRITICAL TO SUCCESS OF OPERATION
Upgraded exit lines to remove catalyst particles while hot

Added heat exchangers in parallel

Cyclone moved upstream

Added filters

Added blowdown

Upsized diameter of entire line
Insufficient Regen Air

- Initial Design: Coke loading estimated from *preliminary* results on Bench-scale FBR
- No $O_2$ measured on regen exit
- Limited by reactor geometry:
  - Exit line too small diameter
  - Too much carryover (elutriation) at higher air flow

![Graph showing CO, CO2, and O2 concentrations over time with air flow into reactor and time in minutes. Early experiments, max air 50 slm.]
Elutriation of Catalyst at Increased Air Flow

Credit:
Bruce Adkins (ORNL) (using PSRI models)
Add Cyclone Return into Regenerator

- Increased air flow carries over MUCH more catalyst
- Effectively increased Regen. diameter
- Installed cyclone to return catalyst into reactor
- Tricky design: must keep horizontal section of return pipe fluidized
Air Regeneration Results

<table>
<thead>
<tr>
<th>Coke on Catalyst (%C by wt)</th>
<th>Insufficient Regen</th>
<th>Complete Regen</th>
</tr>
</thead>
<tbody>
<tr>
<td>Post-stripper, after ~2 hours</td>
<td>0.94%</td>
<td>0.58%</td>
</tr>
<tr>
<td>Post-regen, after ~2 hours</td>
<td>0.66%</td>
<td>0.01%</td>
</tr>
</tbody>
</table>
Catalyst mass flow rate, which is critical for VPU kinetics,
– can be empirically determined by change in level in fluidized bed reactors,
– then correlated to differential pressure across Riser,
– as long as gas flow rate stays constant
• Pressure in top of fluidized beds varies linearly with level of catalyst in bed

• D Pressure across Regen. cyclone must be positive
  - *Flowrate out sidearm must be greater than flow in, or pressure flips and catalyst empties*
Lessons Learned

DON’T PLUG SIDEARMS

- Don’t overflow fluidized beds
- Filter catalyst particles out while hot
  (mud plugs lines & is difficult to clean out)
Lessons Learned

NEED PLENTY OF $O_2$ FOR REGENERATION

- High-risk to scale up using bench-scale data from dissimilar reactor system
- We mitigated catalyst elutriation out of Regen. by adding a cyclone
- Ideally, disengagement zone (freeboard) keeps catalyst in reactor
- Pure oxygen is dangerous & expensive, but plausible
Acknowledgements

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DOE Advanced Development & Optimization program (ADO)

10/9 @ 4pm Jessica Olstad (NREL)
Co-Processing Catalytic Fast Pyrolysis Oils with Vacuum Gas Oil in a Davison Circulating Riser – Upgrading Track
Questions?

www.nrel.gov

NREL/PR-5100-75187