Development and Testing of Aerogel Sorbents for CO_2 Capture

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Project Overview

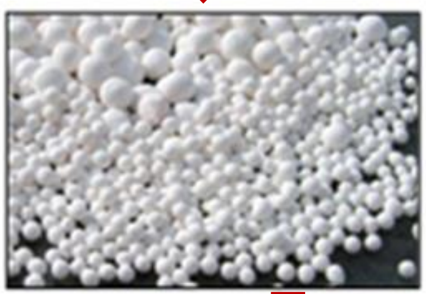
Develop and bench-scale test an advanced aerogel sorbent for post-combustion CO_2 capture from coal-fired power plants

"AFA"

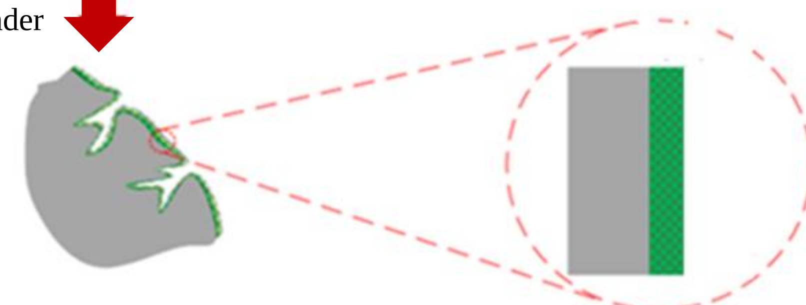
Amine Functionalized Aerogel Sorbent

<u>Develop Aerogel Sorbent at Bench Scale for CO_2 Capture</u>

- Improve Amine Functionalized Aerogels (AFA)
- Convert optimized sorbent into bead form
- Develop pellet binder formulations, and forming process
- Develop SO_x diffusion barrier for AFA sorbents
- Test & evaluate sorbent technology at bench scale



Form Pellets with Binder



Develop Compatible SO_x Resistant Binder

Bench Scale Evaluation

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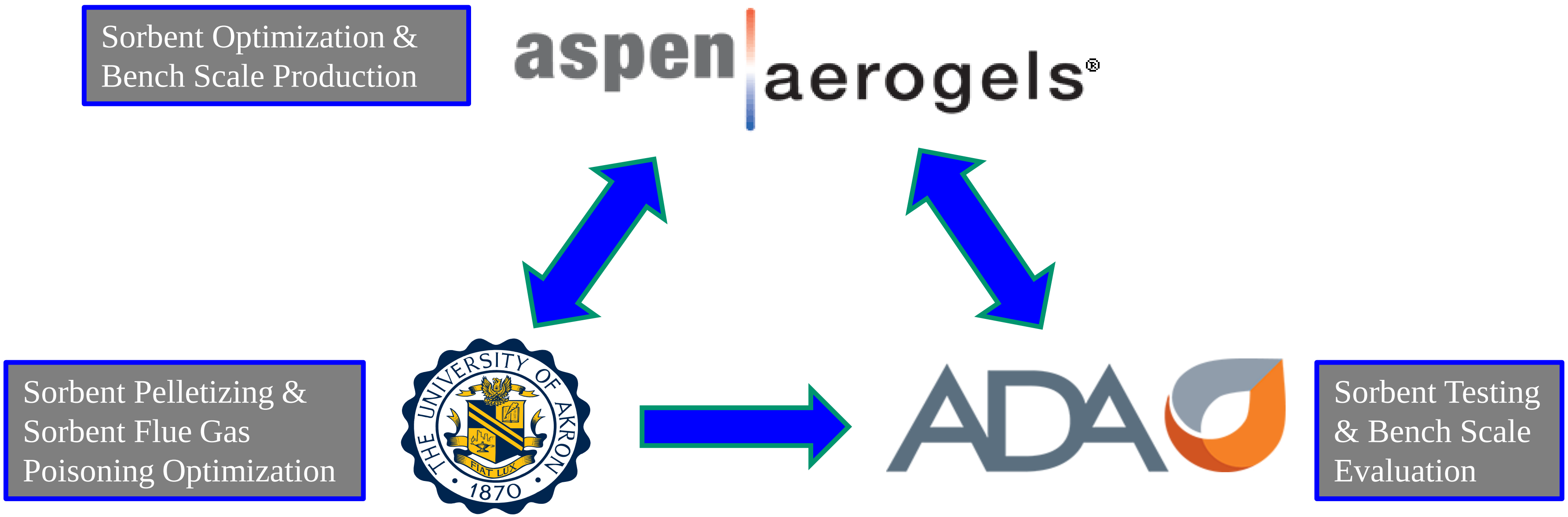
Project Objectives

1. Optimize sorbents for improved CO_2 capacity and SO_X poisoning resistance.
2. Convert optimized sorbent into durable pellet and bead form for analysis.
3. Produce the best candidate sorbent form (bead or pellet) in larger quantities for fluidized bed testing.
4. Assess the sorbent in fluidized bed bench-scale testing.
5. Conduct a technical and economic assessment of the sorbent technology and process.

Project Team

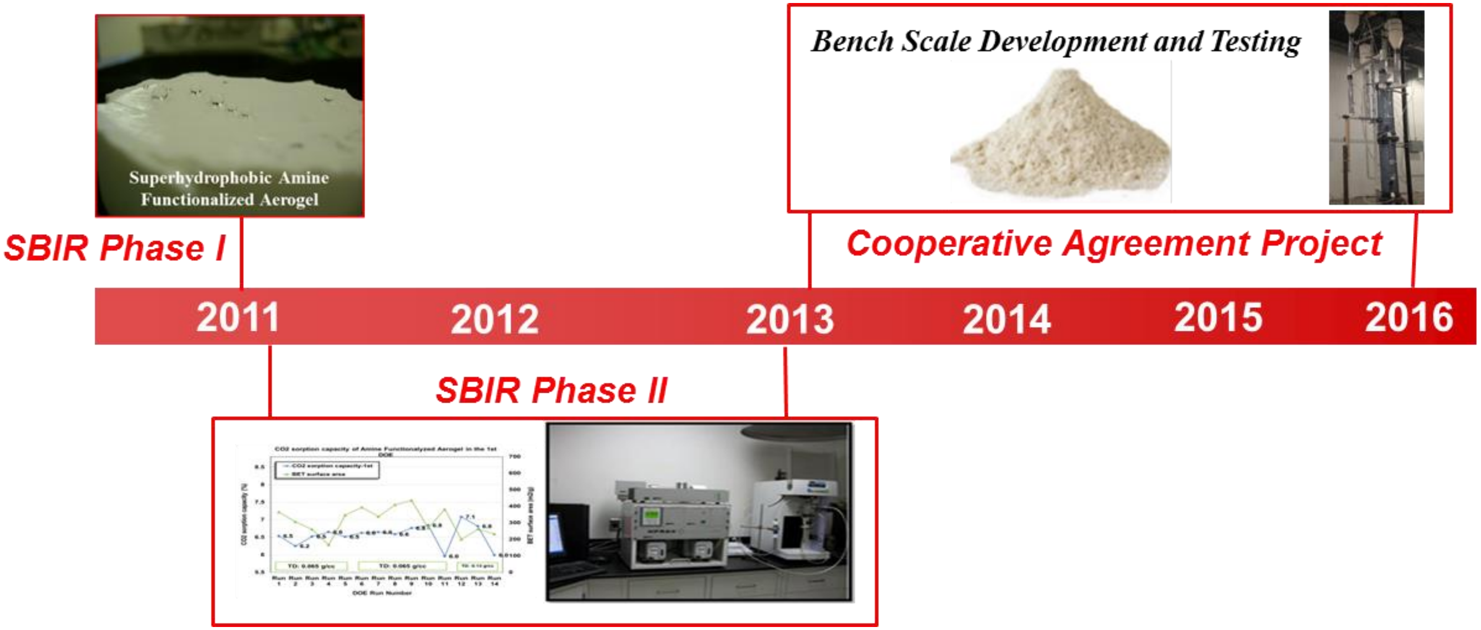


- Period of Performance:
  - 10-1-2013 through 09-30-2016
- Funding:
  - U.S.: Department of Energy: $2.99M
  - Cost share: $ 0.77 million
  - Total: $3.76 million

BP2 Project Tasks

BP#	Description
BP1 (2013 – 2014)	AFA Sorbent Development
	Pellet Development and Optimization
	Sorbent Evaluation
BP2 (2014 – 2015)	Aerogel Bead Fabrication
	Coating Development
	Coated Pellet and Bead Evaluation
BP3 (2015 – 2016)	Pellet (or Bead) Production
	Fluidized Bed Evaluation
	Techno-Economic Evaluation
	Environmental Health and Safety Evaluation

Amine Functionalized Aerogel (AFA) Development



AFA benefits

- High surface/high porosity material
- Hydrophobic to enhance CO_2 adsorption selectivity and stability
- Low specific heat, thus low energy regeneration
- High temperature stability
- Good routes for manufacture at reasonable cost and at high volume

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Accomplishments to Date

Sorbent CO_2 Capture Performance

- High total and working CO_2 adsorption capacities (~ 20 wt.%, ~ 8 wt.%)*
- Fast CO_2 adsorption kinetics (<15 min. to reach 80% of total CO_2 capacity)**
- Stable for at least 250 adsorption/desorption cycles

Regeneration Temperature and Delta Temperature (ΔT)

- Reduced the required regeneration temperature below 130 °C and kept the CO_2 working capacity above 6 wt.% target.

Moisture Uptake

- AFA moisture uptake > 1 wt.%
- However, AFA has high preferential adsorption of CO_2 vs. H_2O
- Maintaining acceptable CO_2 loading performance by reducing cycling time.

***** BP1 targets: > 12 wt.%, and > 6 wt.% (@ 40 – 100 °C, adsorption/desorption cycle)

****** *40 °C and 0.15 CO_2 bar*

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Accomplishments to Date

Pellet Sorbent Development

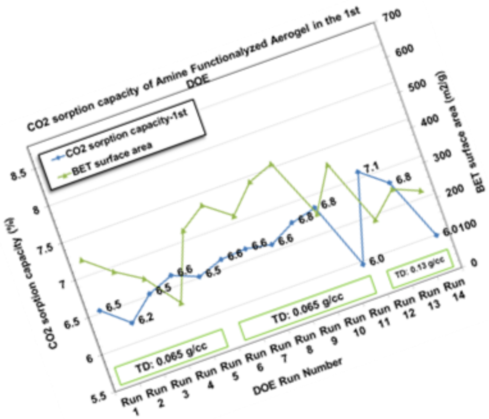
- 300 – 350 micron size pellets prepared.
- 85% capacity retention of the corresponding powder.

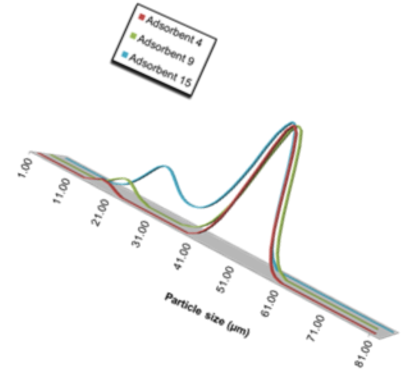
SO_2 Resistant Coating Development (on-going)

- Different coatings have been tested on sorbents in the presence of SO_2.
- Continuing SO_2 resistant coating optimization with goal of minimizing degradation of CO_2 capture performance.

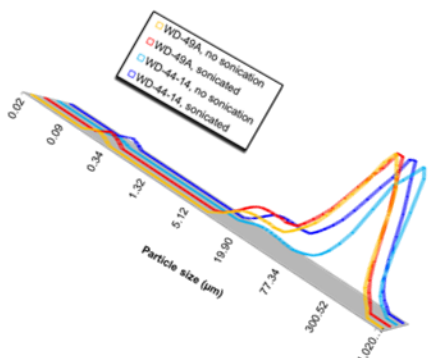
Sorbent Bead Development (on-going)

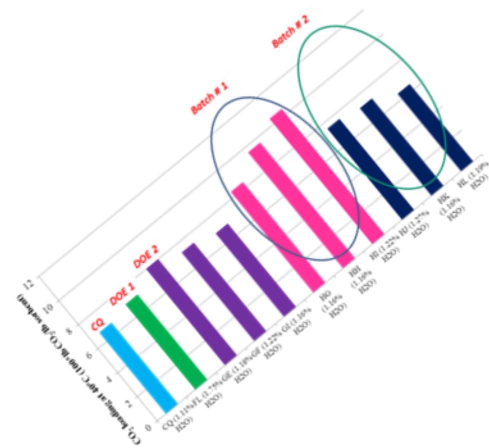
- Optimum sorbent formulation used to produce aerogel beads.
- Bead sizes 0.3 – 1.5 mm have been fabricated.





Technical Progress





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AFA Formulation Optimization

Top two AFA formulations from BP1

- **AFA Sorbent Type #1**
  - Direct amine grafting process, using amino-silane precursors/sol-gel process.
  - High thermal stability (~ 190 °C), CO_2 capacity (~ 14.3 wt. %).
- **AFA Sorbent Type # 2**
  - “Double functionalization” process by amine-grafting and impregnation methods.
  - Thermally stable up to 100 – 110 °C, high CO_2 capacity (> 15.4 – 20 wt.%).

Performance Trade?

Enhanced Capacity vs. Thermal Stability

AFA CO_2 Capture Performance

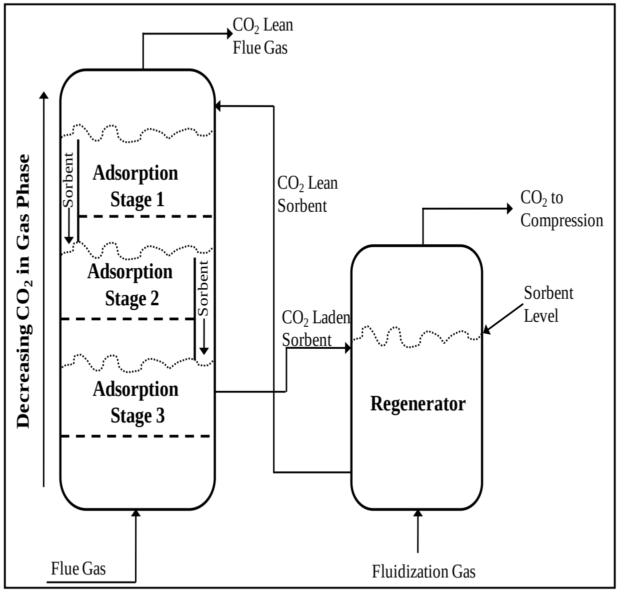
Top AFA sorbent performance under 100% CO_2 (TGA):

Sorbent Type #1

	Temp. swing adsorption/desorption cycle		
	@ 40°C - 100°C	*@ 40°C – 120 °C*	*@ 70°C - 120°C*
Total CO_2 capacity (wt.%)	13.8	13.3	8.9
Working CO_2 capacity (wt.%)	6.2	9.8	6.4

Sorbent Type #2

	Temp. swing adsorption/desorption cycle		
	@ 40°C - 100°C	*@ 40°C – 120 °C*	*@ 70°C - 120°C*
Total CO_2 capacity (wt.%)	17.3	17.2	10.8
Working CO_2 capacity (wt.%)	5.8	10.3	6.5
Heat of reaction (kJ/mole CO_2)	**50 – 60 (MEA ~ 84 kJ/mole CO_2)**		



ADAsorb™ Process Overview

- *High working capacity*
- *Low ΔT (thus low energy regeneration)*
- *Sorbent is thermally stable*

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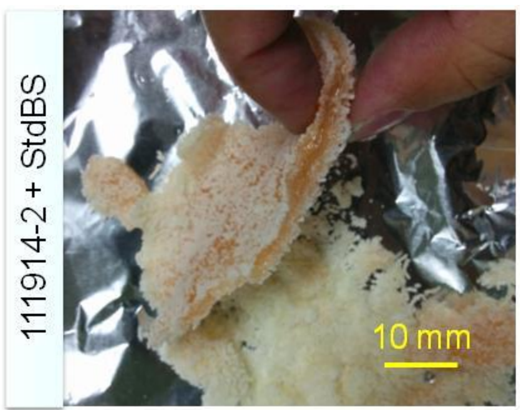
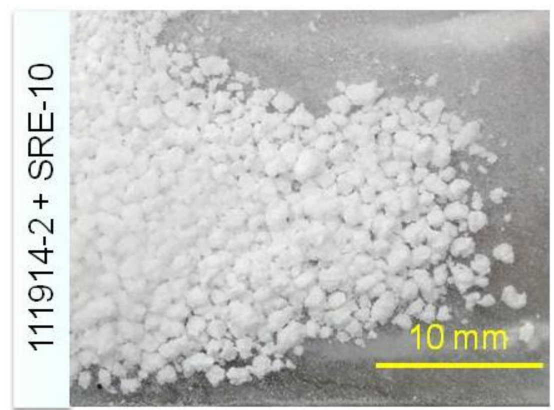
Pelletization of Powder Aerogel (AFA) Sorbent

Issues	Progress	Plan of Action
• Degradation of CO_2 performance by 50% when AFA pelletized with Standard Binder Solution (StdBS). • Sorbent Type #1 (1N) not compatible (dissolved) with StdBS.	• Applied SRE* coating. • Sorbent Type #1 pellets (with SRE): **12.5 wt.%** (~ 13.7% loss) • Attrition test using ASTM D5757. **Attrited weight < 0.1%**	• Optimize pelletization process (mixing, extrusion and drying) to reduce performance degradation



Before: "pellets" with StdBS



Now: pellets with SRE

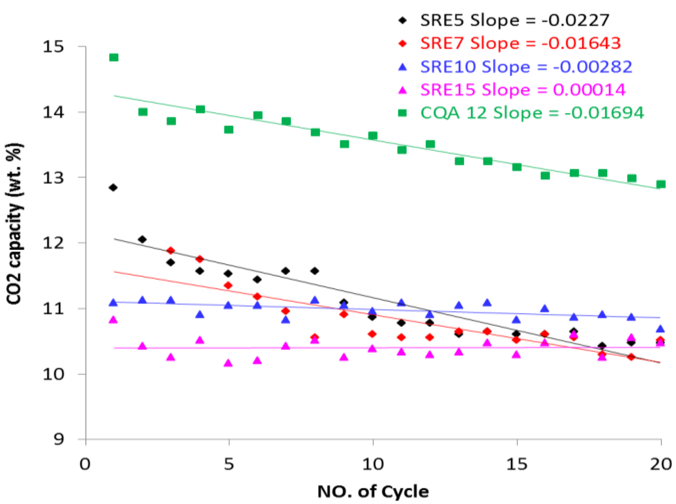
*SRE is a coating designed by UA for SO_2 poisoning resistance. Also used for pelletization.

SO_2 Removal Strategy and Process

- Amine-based sorbents suffer from SO_2 poisoning.
- There is currently **<u>not</u>** a sorbent which only adsorbs CO_2 without adsorbing SO_2.
- SO_2 not only poisons the sorbents but also decreases the purity of desorbed CO_2.
- The degree of SO_2 removal depends on important factors such as the sorbent tolerance to SO_2 and cost of the sorbent replacement and/or regeneration
- Current design of flue gas desulphurization (FGD) units can achieve more than 95 % removal of SO_2
- Effectiveness of the SO_2-resistant coating (develop by UA) is verified to reduce the SO_2 poisoning on the aerogel sorbents.
- Recent results exhibited only 4% degradation in the CO_2 capture capacity after a 20-cycle exposure to 40 ppm SO_2 in the simulated flue gas.
- UA has also proposed a desorption process to achieve both high-purity CO_2 with an insignificant energy penalty.

SO_2 Resistant Coating Development for AFA Pellets

Issues	Progress	Plan of Action
• SO_2 poisons Sorbent Type #2 • **~ 13%** degradation in CO_2 capture capacity after 20 cycles in presence of 40 ppm SO_2	• Developed SRE series SO_2 resistant coating • SRE-10 & SRE-15: **< 4%** degradation with cycling **< 25%** capture capacity loss on cycle 1 due to coating	• Reduce 1st-cycle capture capacity drop (compensate amines) • Study effect of moisture on the SRE coating • Higher SO_2 conc. testing



Sample	Polymer Linker	Cycle 1 CO2 capacity (Wt. %)	Cycle 20 CO2 capacity (Wt. %)	Degradation
SRE-5	5%sln.	12.80	10.50	18.48%
SRE-7	7%sln.	11.88	10.52	11.48%
SRE-10	10%sln.	11.09	10.65	3.97%
SRE-15	15%sln.	10.82	10.47	3.18%
CQA 12	/	14.83	12.89	13.00%

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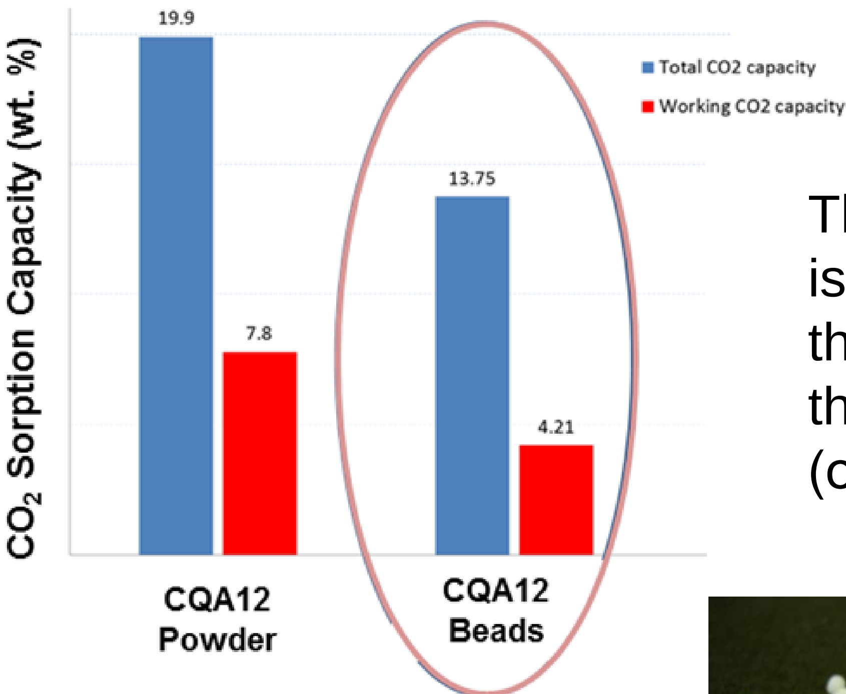
AFA Sorbent Fabrication in Bead Form



The objective of making sorbent beads is to skip the pelletization process; thus reducing the cost of production if the AFA sorbent beads perform better (or “as good as”) the pellets.

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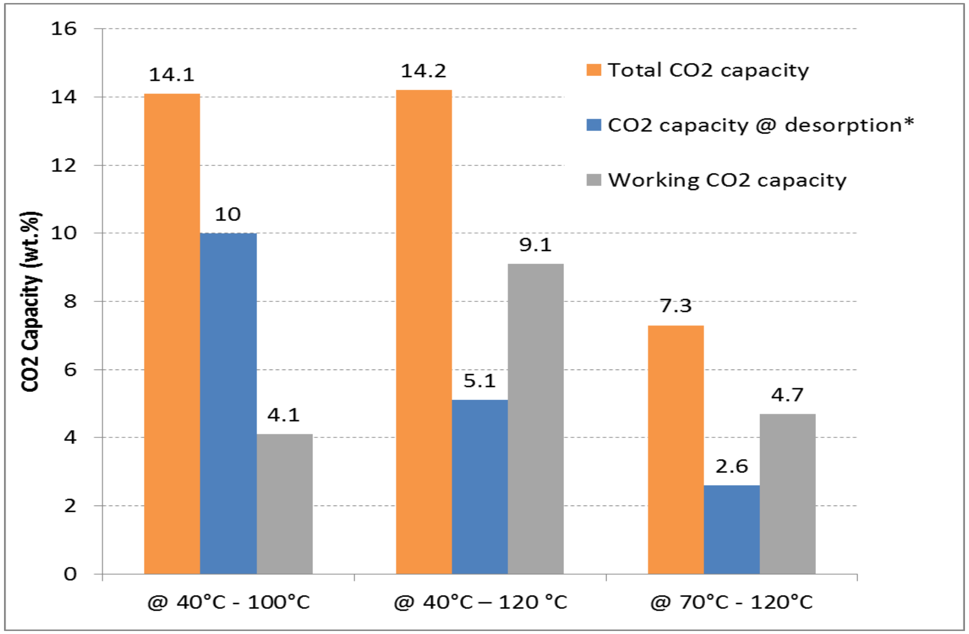
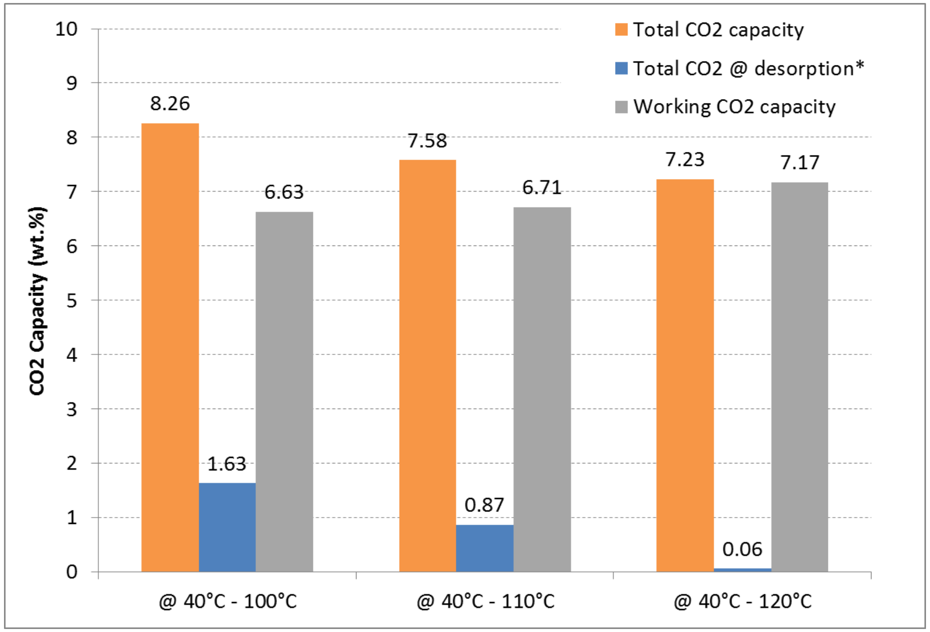
Bead AFA Sorbent CO_2 Capture Performance

- ➢ Optimum AFA formulations used in bead process optimization
- ➢ Bead size and quality depends on:
  - Mixing speed of the "inert medium"
  - Gel time of the AFA sol
  - Temperature of the "inert medium"



Type #2 sorbent beads



Type #1 sorbent beads

* Desorption @ 1 atm CO_2

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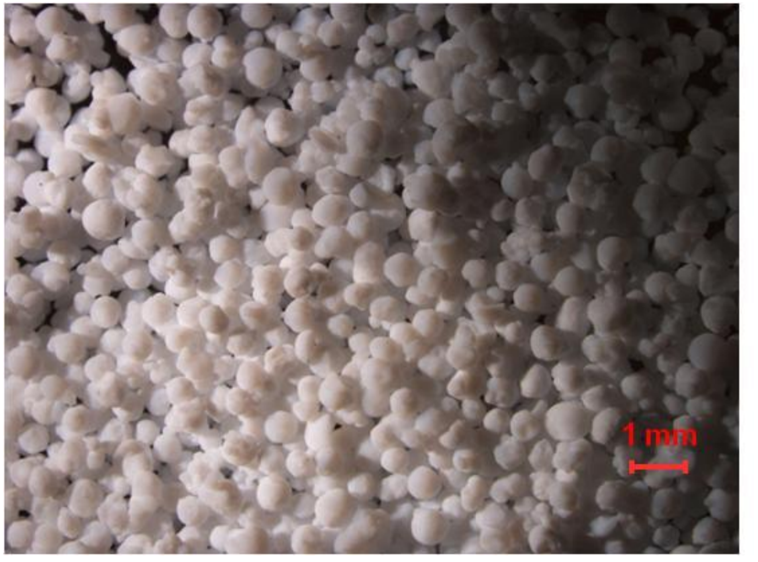
➢ Working CO_2 capacity ~ 80-99% of Total CO_2 capacity for Type #1

AFA Sorbent Fabrication in Bead Form

Different size beads (density ~0.25 g/cc) have been prepared and are being tested at ADA:

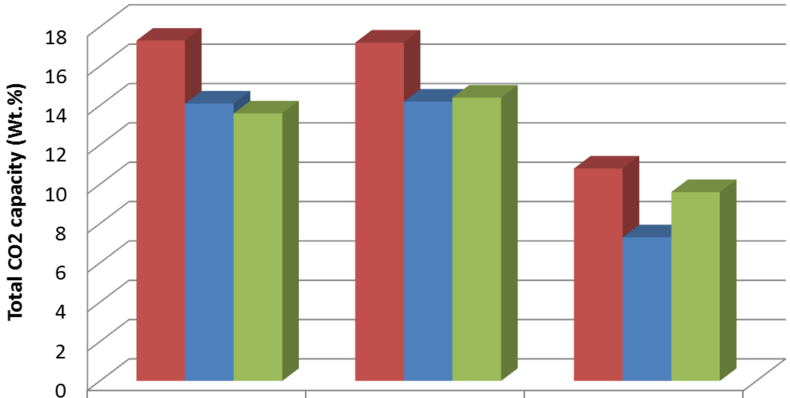
- 0.60 – 1.00 mm
- 0.35 – 0.60 mm
- < 0.35 mm



Issues	Plan of Action
• Amine leaching out during bead process fabrication. • Long gel time of AFA sol formulations. • “medium inert” might affect bead sorbent CO_2 capture performance.	• Improve the conditions of bead prep. • Increase working CO_2 capacity of the beads above 6 wt.% at reduced ΔT. • Apply SO_2 resistant coating on beads and assess performance.

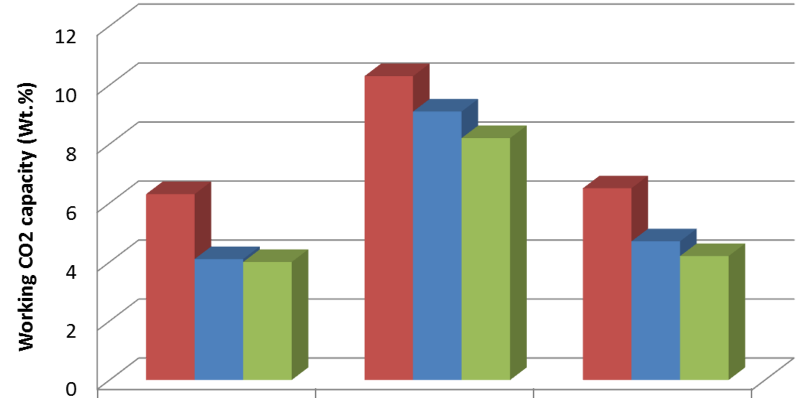
AFA Sorbent Performance Comparison (Type #2)

Total CO_2 Capacity



	@ 40°C - 100°C	@ 40°C – 120 °C	@ 70°C - 120°C
CQA-12 powder	17.3	17.2	10.8
CQA-12 beads	14.1	14.2	7.3
CQA-12 pellets	13.6	14.4	9.6

Working CO_2 Capacity

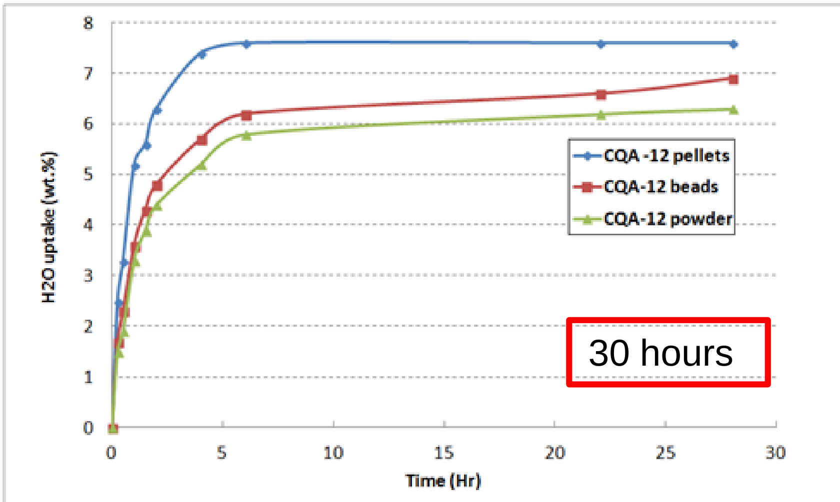


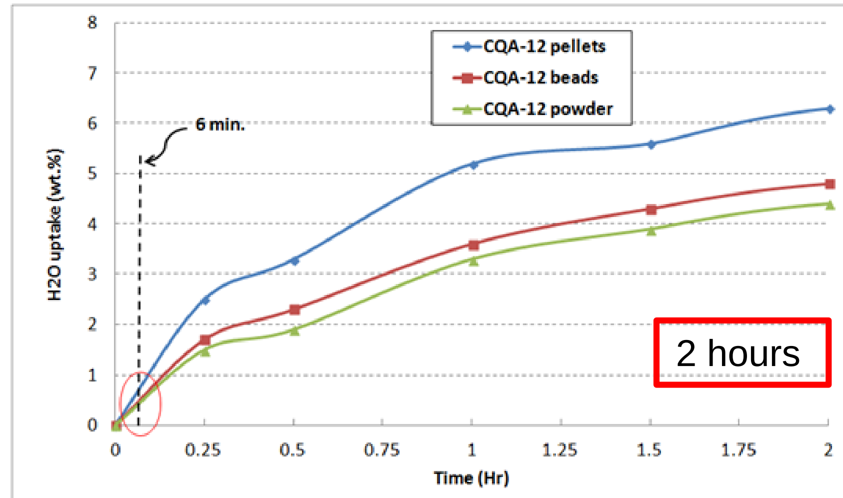
	@ 40°C - 100°C	@ 40°C – 120 °C	@ 70°C - 120°C
CQA-12 powder	6.3	10.3	6.5
CQA-12 beads	4.1	9.1	4.7
CQA-12 pellets	4	8.2	4.2

- CQA-12 sorbent powder exhibits definitively the optimum performance.
- CQA-12 beads have slightly better capacity than pellets.
- Binder/coating decreases CQA-12 pellet performance.
- Total and working CO_2 capacities are maximized when temperature of adsorption is 40 °C and temperature of desorption is 120 °C.

Performance Comparison for Water Uptake

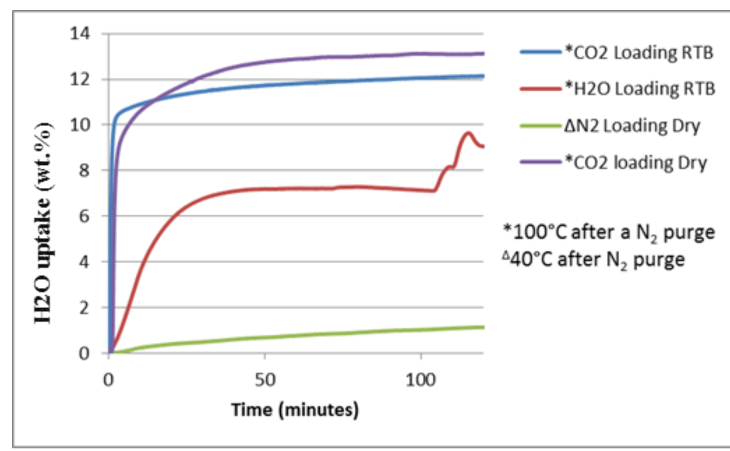
Water uptake @ 40 °C and 60% RH





< 1 wt.% water adsorption @ < 6 minutes

The sorbent cycling time may be reduced to control moisture loading and still maintain acceptable CO_2 loading performance.



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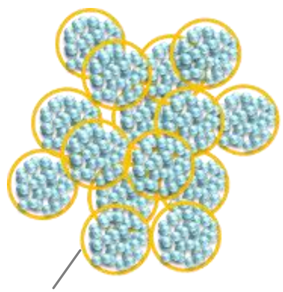
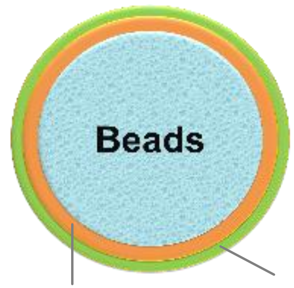
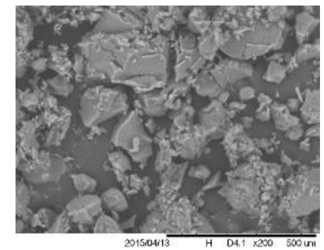
SO_2 Resistant Coating Development on AFA Beads



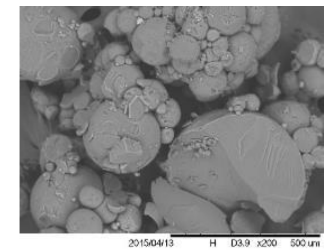
Coated / Pelletized Powder
Process #1



Coated Beads
Process #2



Process #1



Process #2

*Bead form is well retained

Sample	Process	CO_2 Capture Capacity (wt.%)	Difference
AFA Bead*, uncoated	/	11.18	/
AFA Bead, coated, SRE-10	1	6.38	- 42.9%
AFA Bead, coated, 1% XL	2	13.11	**+ 17.3%**
AFA Bead, coated, 3% XL	2	14.12	**+ 26.4%**
AFA Bead, coated, 5% XL	2	14.52	**+ 29.9%**

- The total CO_2 capture capacity **increased** using Process #2

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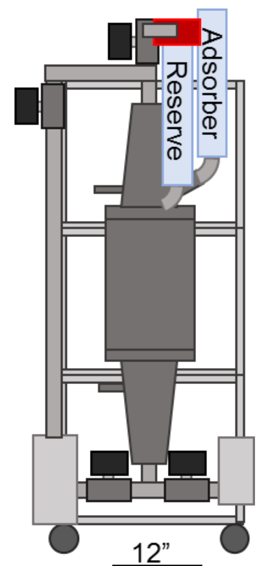
Future Plans

- Finalize bead fabrication process (Aspen).
- Finalize the optimization of SRE coating composition and process application (Akron).
- Crush strength (ADA).
- Perform the attrition tests according to the standard protocol based on the Jet Cup Attrition Standard Procedure (representative of the process to be used) (ADA).
- Test cyclic stability of the most promising sorbent over 1000 cycles (ADA).
- Investigate alternative regeneration process (low CO_2 partial pressure with steam as sweep gas) to increase CO_2 desorption of sorbent and improve working capacity (ADA).

Future Plans

- Determine the CO_2 vs. H_2O uptake in MSFB (mass spec fixed bed) for promising sorbents (ADA).
- Bench-Scale CO_2 Capture Unit (Akron):
  - Build and optimize 1-kW bench-scale fluidized bed CO_2 capture unit.



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Performance vs. Goals

	Verification Method	BP2 Performance Target	Planned completion date	Actual completion date
AFA (beads/pellets)	Total CO_2 adsorption capacity[(1)]	> 17 wt.%	06/30/2015	09/15/2015 *Close to target* (14 wt.%)
	Working CO_2 capacity[(2)]	> 6 wt.%	06/30/2015	04/01/2015 *Exceeded target* (9.1 wt.%) [(3)]
	Adsorption/desorption kinetics[(4)]	Fast	03/31/2015	03/31/2015 *Met target*
	Water adsorption[(5)]	< 1 % @ 40 °C	06/30/2015	06/01/2015 *Met target*
	Cycling stability (CO_2 adsorption/desorption)	Stable over 500 cycles.	06/30/2015	09/15/2015 *Testing scheduled*
	Size (micron)	300 - 350	04/30/2015	03/31/2015 *Met target*
	Attrition Index	<3%[(6)]	06/30/2015	03/31/2015 *Met target*
	Total CO_2 capacity in the presence of 40 – 60 ppm SO_2 and 80 ppm NO in flue gas.	< 10%	09/30/2015	04/30/2015 *Met target*[(7)]

((1): Adsorption @ 40 °C, Desorption @ 100 - 120°C and 0.15 CO_2 bar.
(2): Adsorption @ 40 °C, Desorption @ 100 - 120°C and 1.0 CO_2 bar
(3): Desorption @ 120 °C and 1.0 CO_2 bar
(4): < 15 min. to reach 80% of total CO_2 capacity at 40 °C and 0.15 CO_2 bar
(5): During adsorption/desorption cycle (i.e. water adsorption should be < 1% wt. during the first 6 min of adsorption)
(6): loss under fluidizing condition for 3 hours.
(7): Testing in presence of NO and SO2 in flue gas is scheduled during the remaining of BP2.

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Summary

- ➢ All BP2 milestones met and completed on schedule.
- ➢ Optimized process of AFA bead and pellet fabrication.
- ➢ High CO_2 capture performance of top AFA (beads) sorbent:
  - Total CO_2 capacity ~14 wt.%
  - Working CO_2 capacity ~ 6 - 9.1 wt.%
  - Fast adsorption kinetics
  - The rate of moisture uptake is < 1 wt.%
- ➢ SRE coating proven as efficient SO_2 resistant coating.
  - Uncoated AFA: ~ 13% CO_2 capacity degradation
  - AFA pellets: < 4 % CO_2 capacity degradation
  - AFA beads: Increased CO_2 capacity by 30%
- ➢ The degree of SO_2 removal depends on:
  - Sorbent tolerance to SO_2 /cost of the sorbent /replacement and/or regeneration

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Thank You
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