
Development and Testing of Aerogel Sorbents for CO₂ Capture

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July 29 – August 1, 2014

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Aspen Aerogels, Inc.

Aspen Aerogels, Inc.

- Founded in 2001
- 220 Employees
- Locations:
 - Northborough, MA
 - (*headquarters, R&D laboratories*)
 - East Providence, RI
 - (*manufacturing facility*)
- World's leading manufacturer of flexible aerogel blanket insulation
- “Large Scale Nanomaterial Manufacturing”
- ISO 9001-2000 (BVQi certified)

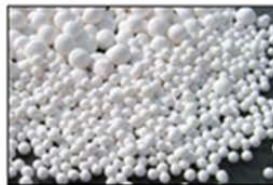


Project Overview

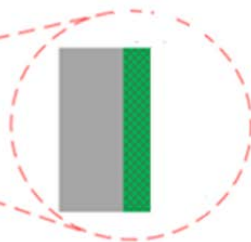
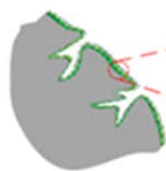
Develop and bench-scale test an advanced aerogel sorbent for post-combustion CO₂ capture from coal-fired power plants



Amine Functionalized
Aerogel Sorbent



Form Pellets with Binder



Develop Compatible SO_x resistant barrier



Bench Scale Evaluation

Develop Aerogel Sorbent at Bench Scale for CO₂ Capture

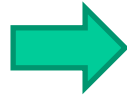
- Improve Amine Functionalized Aerogels
- Develop Pellet Binder Formulations
- Develop Pellet forming process
- Develop SO_x diffusion barrier
- Test & Evaluate Sorbent Technology at Bench Scale

Project Periods

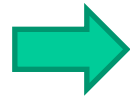
BP#	Description
BP1 (2013 – 2014)	AFA Sorbent Development Pellet Development and Optimization Sorbent Evaluation
BP2 (2014 – 2015)	Aerogel Bead Fabrication Coating Development Coated Pellet (and Beads) Evaluation
BP3 (2015 – 2016)	Pellet (or beads) Production Fluidized Bed Evaluation Techno-Economic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

Project Partners

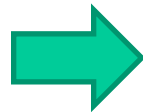
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Sorbent optimization – bench scale production



Sorbent testing – bench scale evaluation



Sorbent pelletizing – Sorbent flue gas poisoning optimization

- ❑ 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

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Amine Functionalized Aerogel (AFA) Development



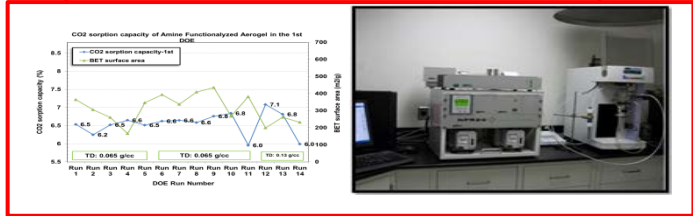
SBIR Phase I



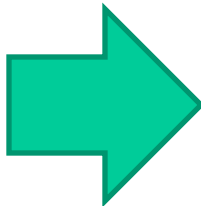
Cooperative Agreement Project



SBIR Phase II



AFA benefits

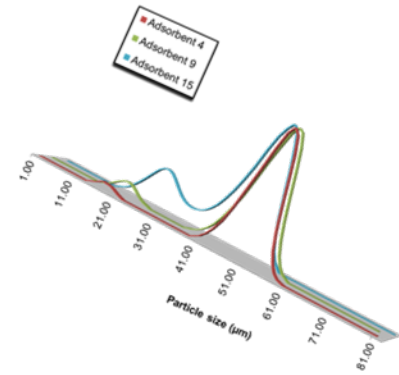
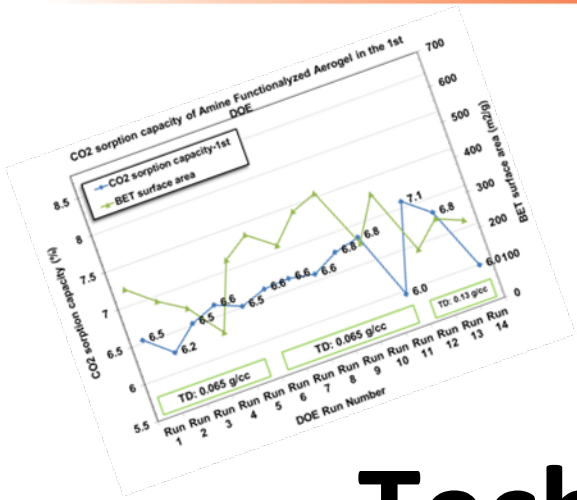


- High surface/high porosity material
- Hydrophobic to enhance CO₂ adsorption selectivity and stability
- Low specific heat, thus low energy regeneration
- High temperature stability
- Good routes for manufacture at reasonable cost and at high volume

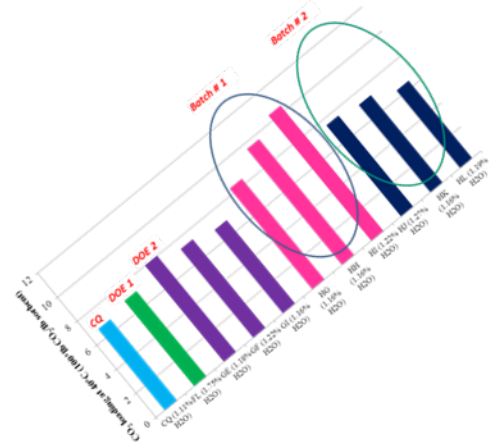
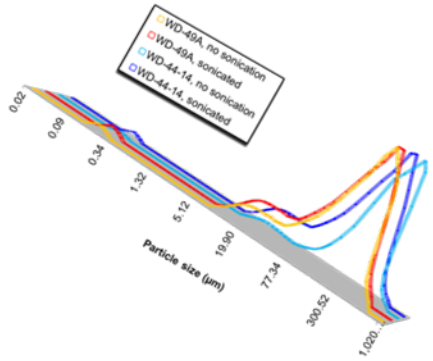
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Project Objectives

1. Optimize sorbents for improved CO₂ capacity and poisoning resistance.
2. Convert optimized sorbent into durable pellet and bead form.
3. Produce the best candidate sorbent form 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.



Technical Progress

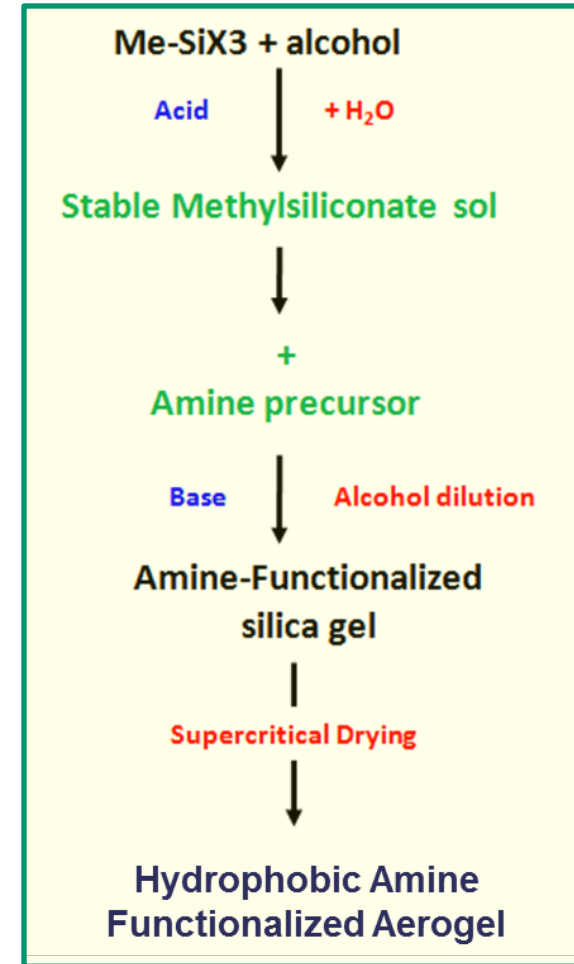


Amine Functionalized Aerogel Optimization

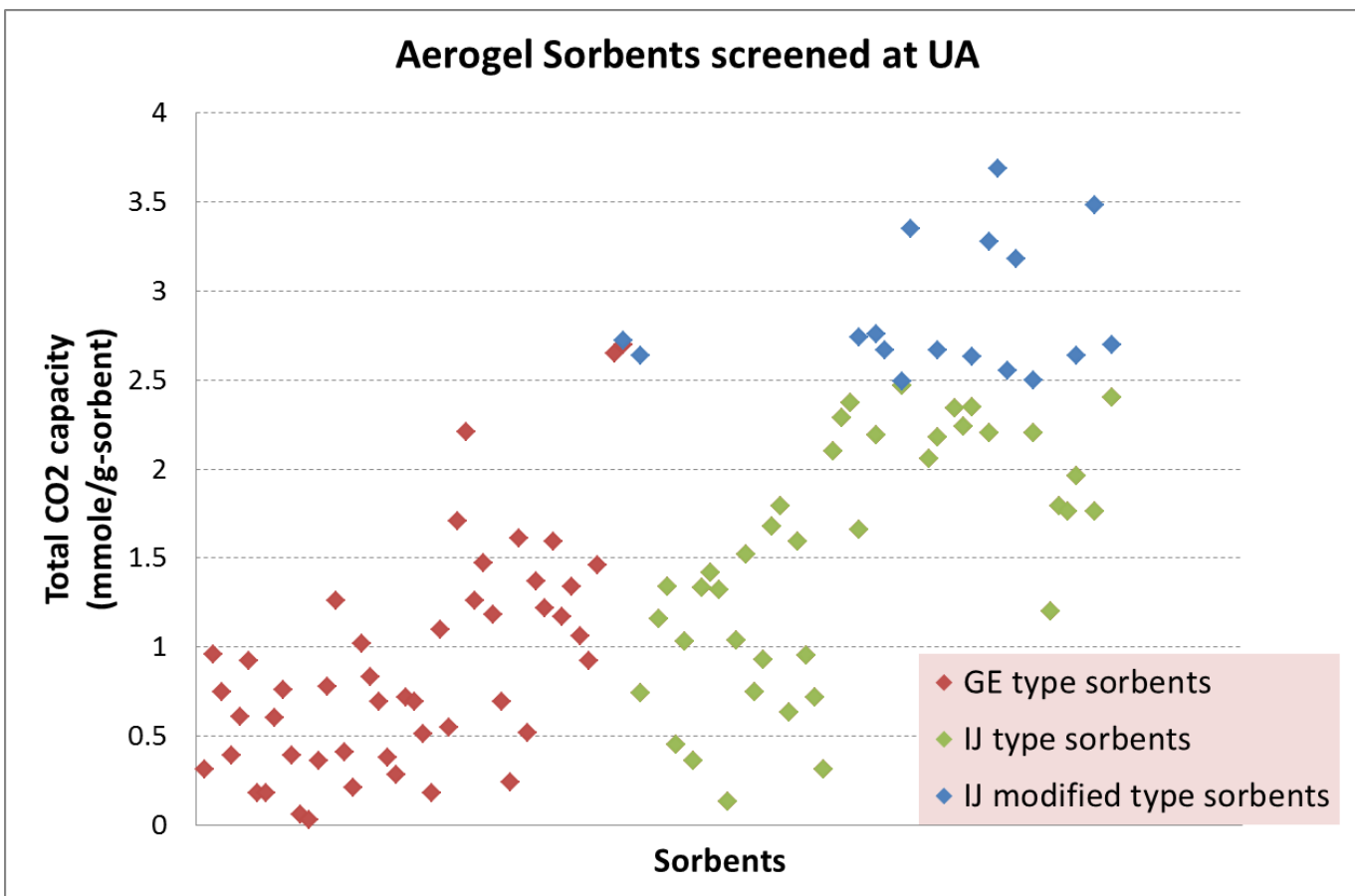
Aspen focused on improving the total CO₂ capacity of its sorbents while trying to maintain their lifetime/cycling stability, by:

- Adjust % amine loading
- Increase the density of the aerogel sorbent (> 0.25 g/cc).
- Optimize chemically grafted amine functional aerogel formula.

Sol-Gel/Aerogel process



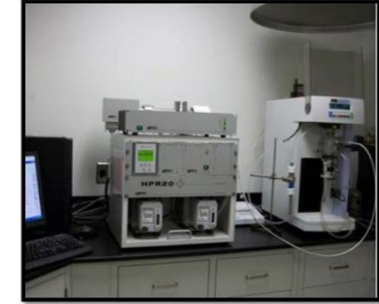
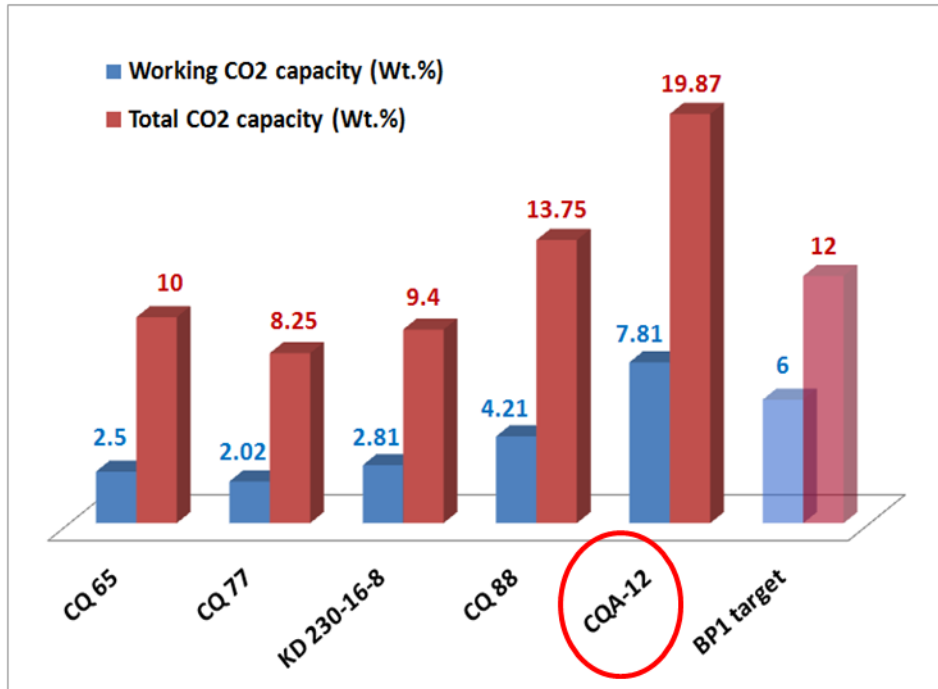
Total CO₂ Capacity - The University of Akron (UA)



T adsorp. = 40 °C (100% dry CO₂), 10 min. adsorption

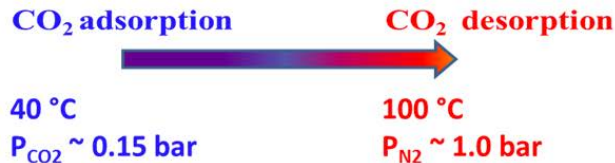
T desorp. = 100 °C (ambient), 10 min. desorption

Aerogel Sorbent Evaluation at ADA



TGA and Mass Spec

- Thermogravimetric analysis (TGA)
 - 100% CO₂ for regeneration
- ~ 1 vol.% moisture
- Various pressures of CO₂ (P_{CO₂})

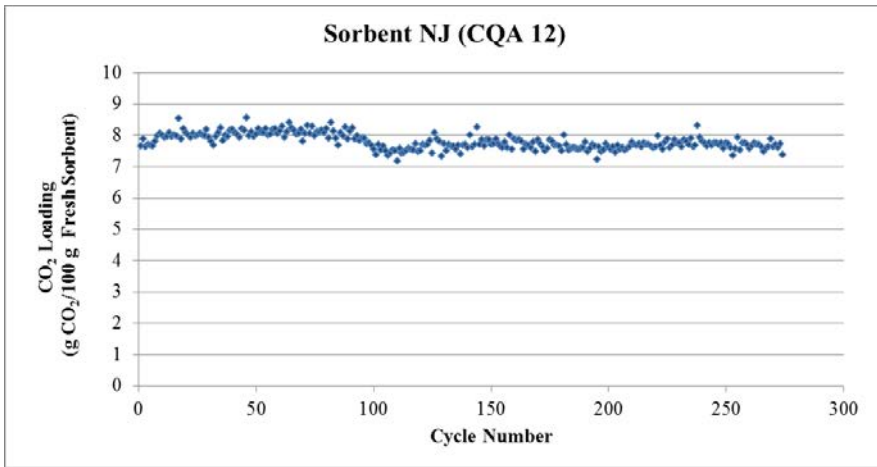


Total CO₂ capacity

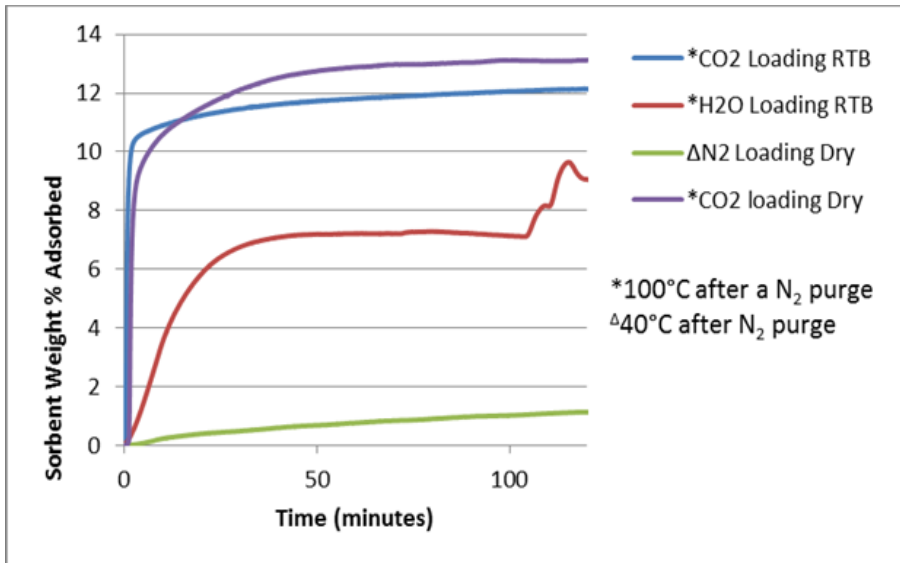


Total working CO₂ capacity

Sorbent Cyclic Stability & CO₂ / H₂O Adsorption Rate Comparison



Stable for up to 250 adsorption/desorption cycles under flue gas conditions of 18.5% CO₂, 4% H₂O, and 77.5% N₂ - **exceeding the 100 cycle BP1 target.**



After 3.9 minutes, CQA12 adsorbs:

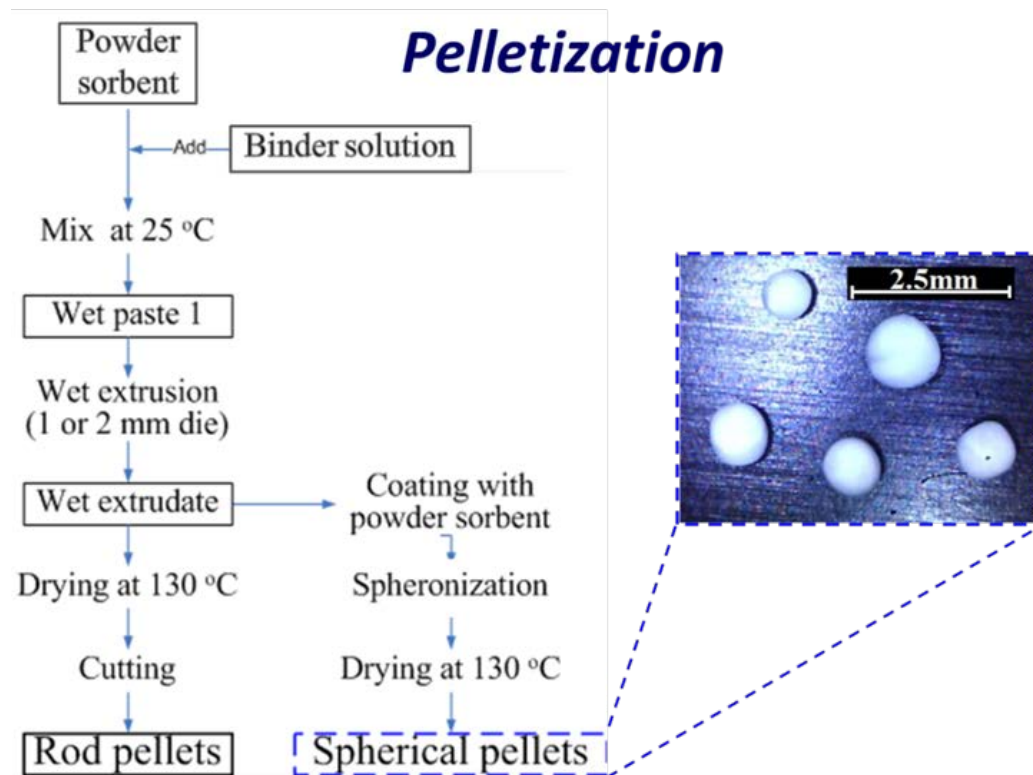
- 1 wt. % H₂O
- 10.54 wt. % CO₂



The sorbent cycling time may be reduced to control moisture loading and still maintain high CO₂ loading performance. → Smaller absorber system volume?

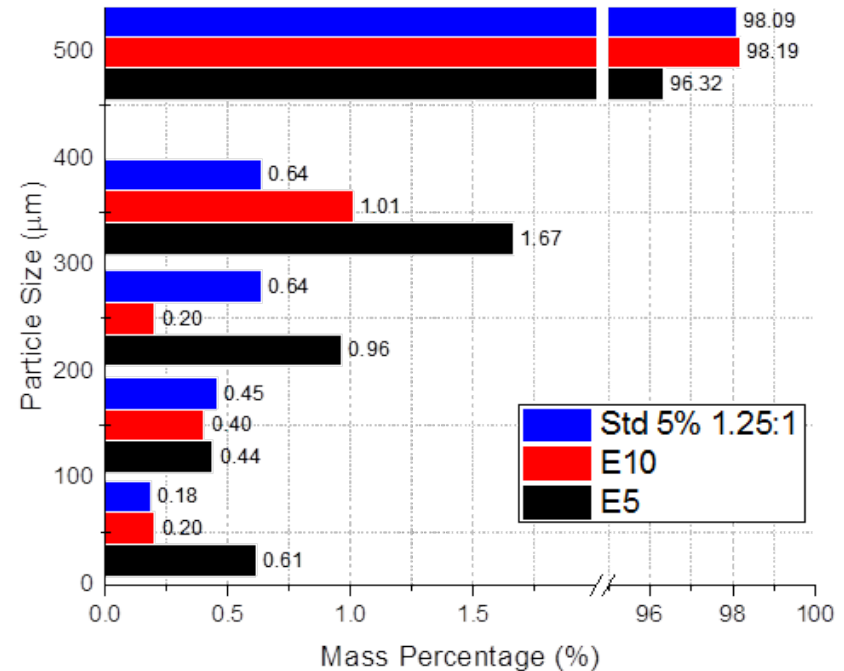
Pelletization Process Optimization - UA

- The CO₂ capture capacities of the pellets prepared with UA Standard binder solution can retain over **90%** of that of the powders.
- CO₂ capture retention for the pellets depends on binder/aerogel ratio and density of active amine sites.



Attrition Index Measurement - UA

- CQA12 pellets were subjected to ASTM D5757 (*Standard Test Method for Determination of Attrition of FCC Catalysts by Air Jets*).
- Initial size ~ 500 μm .
- % mass attrited measured after 3 hours
- Two of UA's binder recipes gave **<2% attrition**.

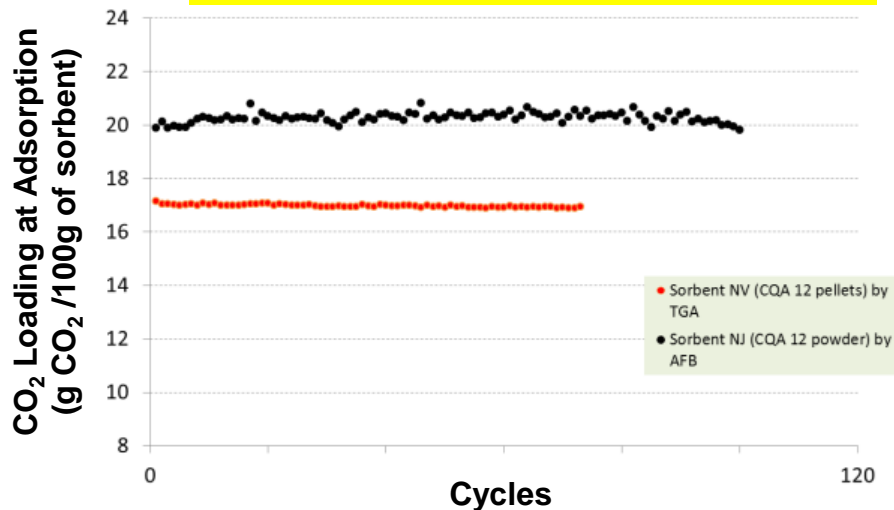


- Modified E series binding solution recipe will be applied as an SO_2 resistance coating.

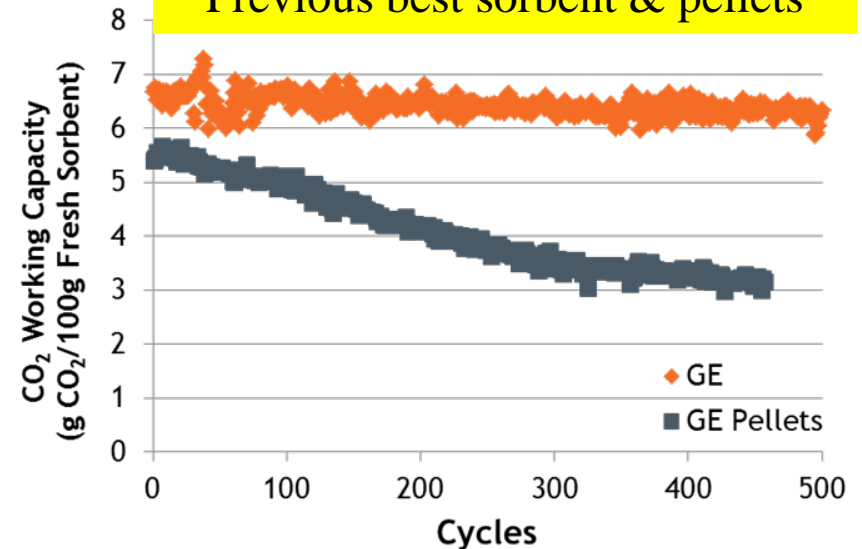
Cyclic Stability of CQA12 Pellets - ADA

- CQA12 Pellet was tested for cyclic stability using TGA.
 - Retained > 88% of the total CO₂ capacity of CQA12 powder.
 - Kinetics were **fast** during CO₂ adsorption for the first 3 cycles as well as the last 3 cycles (cycles 71 – 73).

CQA12 sorbent and pellets



Previous best sorbent & pellets



- Improved stability achieved in BP1 at 3x improvement of capacity vs SBIR program best results

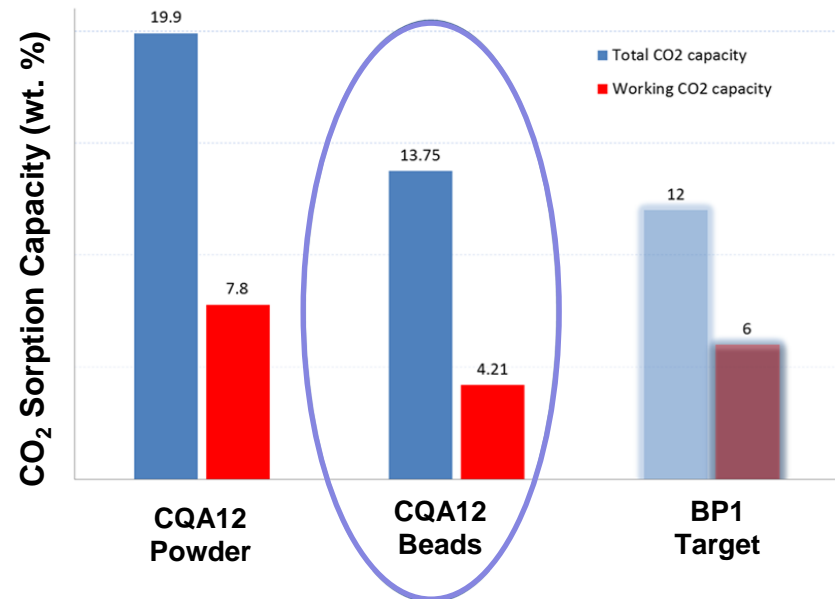
- Cyclic stability (40°C – 130°C)
- Lost ~40% of initial delta CO₂ working capacity after 450 cycles.

Aerogel Sorbent Beads - Study Initiation



- The optimum aerogel formulation (CQA12) was used to make small quantities of beads with different sizes (<500 microns – 3 mm)
- May offer alternative pathway to higher working capacity and faster kinetics without pelletization costs
- Further work on optimization in BP2.

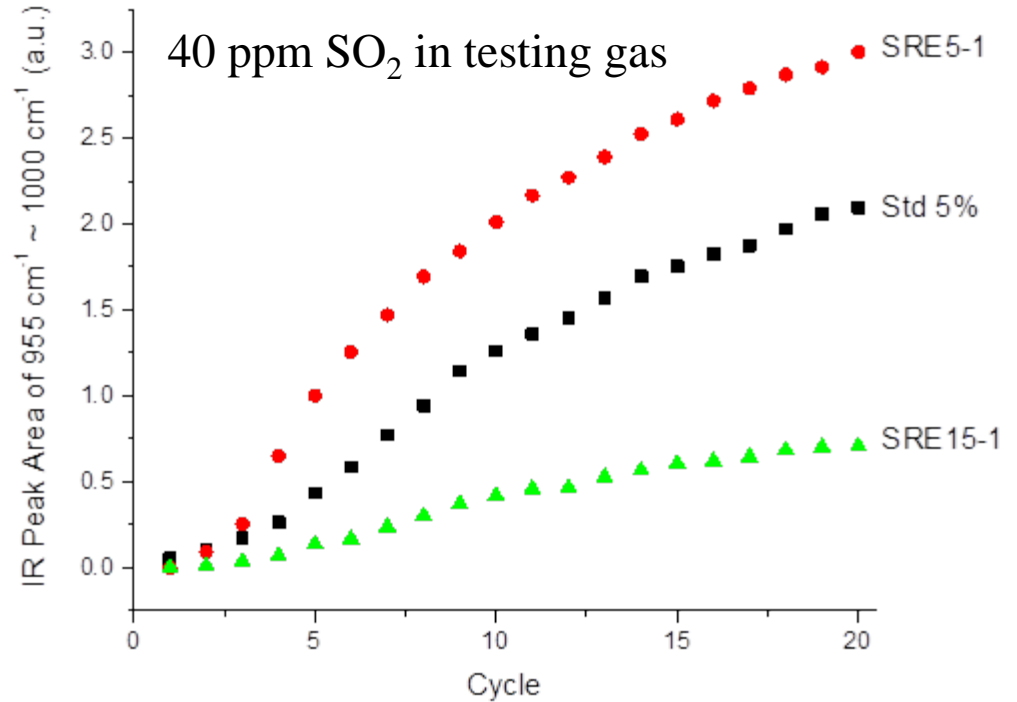
Promising CO₂ adsorption capacity.



Beads will be optimized for better mass transport to increase working capacity

SO₂ Resistant Coating - Preliminary Study

- The SO₂ accumulation of adsorbed SO₂ is suppressed on SRE15-1 coated sorbent compared to the other two samples.
- The composition of the coating has a critical impact on the resistance to SO₂.
- Further studies on optimizing the composition of SO₂ resistant coatings in BP2.



Change of the IR peak area for the sulfate peak (peak center is 981 cm⁻¹)

BP1 Achievements and Planned/Actual Completion Dates

– Through Month 9

	Verification Method	BP1 Target	Planned completion date	Actual completion date
Optimized AFA Powder	Total CO ₂ adsorption capacity ⁽¹⁾	> 12 wt. % (2.72 mmolCO ₂ /g-sorb)	03/31/2014	2/17/2014 Exceeded Target (19.9 wt.%)
	Working CO ₂ capacity ⁽¹⁾	> 6 wt.% (1.36 mmolCO ₂ /g-sorb)	03/31/2014	2/17/2014 Exceeded Target (7.8 wt.%)
	Adsorption/desorption kinetics	Fast ⁽²⁾	03/31/2014	2/25/2014 Medium-Fast at 40 - 100 °C⁽³⁾ Fast at 40 - 110°C⁽³⁾
	Water adsorption	< 1 wt. % @ 40 °C	05/31/2014	05/26/2014 Met Target⁽⁴⁾
	Cycling stability (CO ₂ adsorption/desorption)	Stable over 100 cycles.	05/31/2014	3/21/2014 Exceeded Target (stable over 250 cycles)

1): Adsorption @ 40 °C, Desorption @ 100 °C

2): < 15 min. to reach 80% of total CO₂ capacity at adsorption temperature

3): **Medium-fast** (15.58 min.) , **Fast** (11.43 min.)

4): Water adsorption varies as a function of the hydrophobicity of the sorbent.

BP1 Achievement and Planned/Actual Completion Dates

– Through Month 9

	Verification Method	BP1 Target	Planned Completion Date	Actual Completion Date
Optimize AFA Pellets	CO ₂ adsorption capacity ⁽¹⁾	> 9% wt. (2.04 mmolCO ₂ /g-sorb)	06/30/2014	06/06/2014 Exceeded Target (17 wt.%)
	Density (g/cc)	> 1.2 g/cc	06/30/2014	06/06/2014 ~ 1.3 g/cc Achieved
	Size (micron)	300 - 350	06/30/2014	06/06/2014 Met Target (300 -350 mm)
	Attrition Index	3% ⁽²⁾	06/30/2014	06/18/2014 Exceeded Target (< 2%)
Develop SO₂ coating resistance	the preliminary results should demonstrate the poisoning resistance against SO ₂		06/30/2014	Initiated on 06/15/2014
Initiate AFA bead development	Assess aerogel beads CO ₂ capture performance.		06/30/2014	Initiated on 04/12/2014

1): After optimization of aerogel/binder ratio.

2): loss under fluidizing condition for 3 hours.

BP1 Conclusions

- All milestones have been met and completed on schedule or ahead of schedule.
- Three methods of amine incorporation were investigated.
- Optimized AFA sorbents with a reduced delta T for adsorption/desorption (40 – 100 °C)
- Best performing sorbent (CQA 12) has demonstrated:
 - ✓ High CO₂ total capacity (> 20 wt.%)
 - ✓ High CO₂ working capacity (8 wt.%)
 - ✓ Stable for up to 250 adsorption/desorption cycles
- ✓ High sorbent selectivity → cycling time can be reduced to control moisture loading.
 - ✓ **Several process advantages** (smaller quantities of sorbent, reduced size of the absorber units, reduced capital costs, less parasitic loss etc.).
- Optimized pellet fabrication – up to 90% CO₂ capture retention and low attrition index (< 2%).
- Project team initiated two important studies that will continue during BP2:
 - ✓ SO₂ resistant coating development
 - ✓ Amine Functionalized Aerogel bead development

Acknowledgements

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- **ADA-ES (M. Lindsay, W.J. Morris)**
- **University of Akron (S. Chuang, E.S. Mojica, L. Zhang and U. Tumuluri)**