Gradient Amine Sorbents for Low Vacuum Swing \( \text{CO}_2 \) Capture at Ambient Temperature

DE-FE0031958

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The University of Akron

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National Energy Technology Laboratory
Carbon Management Project Review Meeting
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Program Overview

Funding

- Total project funding
  - DoE share: $752,002
  - Cost share: $200,236

Overall Project Performance Dates
1/1/2021 - 12/31/2022

Project Participants

- PI: Steven. S. C. Chuang, Akron
- Students: J. King, Huhe, S. Billy, S. Starkey, and P. Hollopeter
- Co-PI: Redouane Begag, Aspen
  Nicholas Leventis, Aspen

Overall Project Objectives

- To develop a novel VSA process by designing, fabricating, and refining the structure of amine sites which can accommodate various climate conditions, testing the low vacuum swing process, evaluating scalability, and cost and life cycle analysis.
- To determine the cost-effectiveness of the proposed technology.
Temperature Swing Adsorption (TSA)  Vacuum Swing Adsorption (VSA)

**Technical Background**

**Temperature Swing Adsorption (TSA)**

- **Feed:** Air with 400 ppm CO₂
- **Minimum work needed for separation of mixture with composition 1 and 2:**
  \[
  W_{\text{min}} = \Delta G_{\text{mix}} = -T \Delta S_{\text{mix}} = RT \left( x_1 \ln x_1 + x_2 \ln x_2 \right)
  \]
- **Lost work:** \( LW_{\text{TSA}} = Q_{\text{ Humans}} \left( 1 - \frac{T_0}{20 + T_0} \right) \)
- **Separation efficiency:** \( \eta_{\text{TSA}} = \frac{W_{\text{min}}}{LW_{\text{TSA}}} \)
- **Technical challenges:**
  - Identification of amine sites for weakly adsorbed CO₂.
  - Production of high purity CO₂ (>99%)
  - Fabrication of hierarchical sorbents with a high density of weakly adsorbed CO₂ sites.
  - Construction of a low leakage vacuum swing unit.

**Vacuum Swing Adsorption (VSA)**

- **Feed:** Air with 400 ppm CO₂
- **Lost work:** \( LW_{\text{VSA}} = RT \ln \left( \frac{1 \text{ atm}}{P_{\text{VAC}}} \right) \)
- **Separation efficiency:** \( \eta_{\text{VSA}} = \frac{W_{\text{min}}}{LW_{\text{VSA}}} \)
- **Technical advantages:**
  - Operation at ambient temperature without the input and removal of thermal energy of the sorbent bed.
  - Scalable and modular design

**Technical challenges:**

- Sorbent degradation
- Thermal energy

**Technical advantages:**

- Ambient temperature
- Low energy
IR absorbance of adsorbed CO\textsubscript{2} as a function of time during Ar purge at 150 cm\textsuperscript{3}/min at 25 °C. The slope of the IR decay curve corresponds the rate of desorption of weakly adsorbed CO\textsubscript{2}.

20 wt\% TEPA, 20 wt\% PEG/SiO\textsubscript{2}

10 wt\% TEPA, 10 wt\% PEG/SiO\textsubscript{2}
In situ infrared (IR) spectroscopy coupled with mass spectroscopy (MS) allows simultaneous monitoring the dynamics of adsorption and desorption of strongly and weakly adsorbed CO$_2$ on amine sorbents.

- The project goal is to populate the porous and stable structure with high density of weakly adsorbed CO$_2$ sites for the vacuum swing adsorption.

Identification of weakly adsorbed CO$_2$ sites allows design and preparation of a hierarchically structure of amine sorbent for low vacuum swing adsorption.
Technical Approach/Project Scope

a. Experimental design and work plan
   - Preparation, characterization, and test of sorbents with weakly adsorbed CO₂ sites
   - Fabrication and test of a Kg scale vacuum swing adsorption unit for capture of CO₂ from air.

b. Project schedule

<table>
<thead>
<tr>
<th>Task/Subtask</th>
<th>Milestone Title &amp; Description</th>
<th>Planned completion date</th>
<th>Actual Completion Date</th>
<th>Verification method</th>
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<tr>
<td>4.0</td>
<td>Determination of CO₂ capture capacity and stability (4-15)</td>
<td>11/01/2021</td>
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<td>Quarterly report</td>
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<td>5.0</td>
<td>Fabrication of a Kg scale unit. (12-18)</td>
<td>11/01/2021</td>
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<td>Quarterly report</td>
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<td>02/01/2022</td>
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<td>Cost Analysis and Life Cycle Analysis.</td>
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<td>12/31/2022</td>
<td>Quarterly report</td>
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C. Project success criteria

<table>
<thead>
<tr>
<th>Decision Point</th>
<th>Date</th>
<th>Success Criteria</th>
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<tr>
<td>1</td>
<td>12/1/2021</td>
<td>Both sorbent plates and sorbent particles exhibit the same level in vacuum swing adsorption CO₂ capture capacity. Reaching the target listed in State-Point Data</td>
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<tr>
<td>2</td>
<td>2/1/2022</td>
<td>Completing the construction of the Vacuum Swing Adsorption unit</td>
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# Risk Management

<table>
<thead>
<tr>
<th>Description of Risk</th>
<th>Probability</th>
<th>Impact</th>
<th>Overall</th>
<th>Risk Management (Mitigation and Response Strategies)</th>
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<tbody>
<tr>
<td>Financial Risks:</td>
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<tr>
<td>Commitment to cost-sharing</td>
<td>Low</td>
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<td>Cost-sharing has been allocated and committed by the U. Akron.</td>
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<td>Cost/Schedule Risks:</td>
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<tr>
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<td>Medium</td>
<td>High</td>
<td>Moderate</td>
<td>Alternative suppliers</td>
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<tr>
<td>Technical/Scope Risks:</td>
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<tr>
<td>Equipment maintenance</td>
<td>Moderate</td>
<td>High</td>
<td>Moderate</td>
<td>Regular inspection and calibration</td>
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<td>Moderate</td>
<td>Moderate</td>
<td>Optimizing the sorbent preparation methods.</td>
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<td>Human Resource Risks:</td>
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<td>Availability of manpower to execute project</td>
<td>Moderate</td>
<td>Moderate</td>
<td>Moderate</td>
<td>Recruit domestic undergraduate students as assistants</td>
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<td>High</td>
<td>Moderate</td>
<td>Incentives for retaining the talents</td>
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<tr>
<td>Capability to coordinate</td>
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<td>High</td>
<td>Moderate</td>
<td>Regular and informal meetings and communications</td>
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<td>Environmental</td>
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<td>Low</td>
<td>Use of chemicals with low toxicity</td>
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<tr>
<td>Pandemics</td>
<td>High</td>
<td>High</td>
<td>High</td>
<td>Minimize interpersonal contacts</td>
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Progress and Current Status of Project

CO₂ capture capacity for thermal swing adsorption

Additional Photos of Samples and Lids

<table>
<thead>
<tr>
<th>Sample</th>
<th>CO₂ Capture 1</th>
<th>CO₂ Capture 2</th>
<th>CO₂ Capture 3</th>
<th>Avg</th>
<th>Stdev</th>
<th>Name</th>
<th>Ranking</th>
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<tbody>
<tr>
<td>1</td>
<td>1.06</td>
<td>1.12</td>
<td>0.98</td>
<td>1.05</td>
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<td>0.90</td>
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<tr>
<td>14</td>
<td>1.06</td>
<td>0.99</td>
<td>0.72</td>
<td>0.92</td>
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<td>0.87</td>
<td>0.50</td>
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</table>

KD100BMSPA: contains 4.92 mmol amine/g
KD75BMSPA: contains 5.96 mmol amine/g
(a) In situ infrared (IR) spectroscopy coupled with mass spectroscopy (MS) allows simultaneous monitoring the dynamics of adsorption and desorption.

(b) MS profiles of the gaseous effluent from the IR cell during a CO$_2$ capture cycle: (i) pretreatment, (ii) CO$_2$ adsorption, (iii) Ar purge to remove weakly adsorbed CO$_2$, (iv) TPD (temperature-programmed desorption) to remove strongly adsorbed CO$_2$.

(c) CO$_2$ capture from air and CO$_2$ capture from 0.2% CO$_2$. 

Technology Approach
Progress and Current Status of Project

Amine-based sorbents
- CO₂ adsorption prior to H₂O adsorption
- CO₂ desorption prior to H₂O desorption

Ammonium carbonate (1560 cm⁻¹)
H₂O (3400 cm⁻¹)
Progress and Current Status of Project

Control the density of amine sites: Additives, Amino acids, and polyamines

Amine

4wt% A-Amine

20wt% B-Amine

25wt% amino acid/
25wt% C/ 50wt% silica
**Progress and Current Status of Project**

- Search for formulations of amine sorbents which provide a high density of weakly adsorbed CO₂
  - Additives
  - Amino acids
  - Polyamines

**Wavenumber (cm⁻¹)**

- 3400
- 2827
- 1650
- 1560
- 1496
- 1409

**Difference (a.u.)**

- Amine
- 4wt% A-Amine
- 20wt% B-Amine
- 25wt% amino acid / 25wt% PEG550 / 50wt% silica

**Formulations**

- 25wt% amino acid / 25wt% PEG550 / 50wt% silica
Progress and Current Status of Project

CO₂ Capacity: 0.46 mmol/g

PEG / amino acid / silica (molar ratio: 1 : 1 : 1)

Strongly-adsorbed CO₂
Weakly-adsorbed CO₂
Total adsorbed CO₂

CO₂ Capacity: 0.41 mmol/g

- PEG/amino acid provides both strongly and weakly adsorbed sites for CO₂ and water.
- CO₂ capacity and rate from 15% > CO₂ capacity and rate from air
Cross-link amine
- The rate of CO$_2$ adsorption and desorption
- The density of weakly adsorbed CO$_2$ sites

Cross-linked amine film
Approx. 1 mm thick

Progress and Current Status of Project
Progress and Current Status of Project

VSA Scheme
- Adsorption by flowing a 0.04% CO₂ stream over a sorbent bed
- Desorption of weakly adsorbed species by vacuum

Data collection
- Obtaining the concentration profiles of the effluent form the sorbent bed with adsorbed CO₂ under 8 psi vacuum determined by (a) MS before vacuum pump, (b) IR gas cell after vacuum pump.

A laboratory scale (5-10 cm³) VSA unit with a CO₂ sensor
Sample: 40cc TPESNa (E + P)  

22_02_28 TPESNa (E + P) VSA/D with Mass Spec

Progress and Current Status of Project

CO$_2$, < 100% was produced.
04_03_22 VSA and TSA experiments on carbon heater

Chemicals: Adhesive, Amine, fiber veil.

Progress and Current Status of Project

Pretreatment at 130 °C

- Concentrated CO$_2$, > 90%, was produced from VSA at 3 psia.
Future testing/development
- Improve the sorbent formulation and monolithic structure
- Integration with automatic control
Summary

Key findings:
- The density of weakly adsorbed CO₂ sites on amine sorbents can be controlled by the types of additives and polyamines.
- Some amine sorbents are able to adsorb CO₂ prior to H₂O; to desorb CO₂ prior to H₂O under flowing inert gas and vacuum.
- Concentrated CO₂, > 90%, was produced from VSA at 3 psia.
- Gradient amine layer can be achieved by sequential layer-by-layer deposition of amine-PEG (polyethylene glycol) onto the porous polymer pellet

Lesson learned:
- Fabrication of a laboratory scale VSA (vacuum swing adsorption) unit is highly challenging because of the lack of reliable fittings and valves for holding vacuum.

Future Plan:
- Search for reliable components for building a one-liter VSA unit.
- Fine-tune the formulation of amine sorbents and monolithic structure

Take-away:
- VSA for CO₂ capture is technically feasible.
Teams and Facilities

PI
Steven Chuang

UA leader
Steven Chuang

PhD student
Jaelynne King

PhD student
Hu He

Redouane Begag
Scientist

Nicholas Leventis
Director

Aspen Aerogels, Inc.
George Gould
Chief Technology Officer

Undergraduate Assistant
Sean Billy

Undergraduate Assistant
Samantha Starkey

Undergraduate Assistant
Preston Hollopeter

Teams and Facilities

Undergraduate Assistant
Samantha Starkey

Undergraduate Assistant
Preston Hollopeter

Diffuse Reflectance (DRIFTs)

Transmission
Appendix

- These slides will not be discussed during the presentation but are mandatory.
## Project Timeline

<table>
<thead>
<tr>
<th>Task name</th>
<th>Assigned resources</th>
<th>Year 1</th>
<th>Year 2</th>
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<tbody>
<tr>
<td>Task 1.0 – (UA/Aspen) Program requirements and Project Management</td>
<td>All of team members</td>
<td>Q1</td>
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<td>Task 2.1 – (UA) Fabrication of hybrid sorbents</td>
<td>UA PhD. Student A</td>
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<td>Task 2.2 – (Aspen) – Preparation of amine-functionalized aerogel</td>
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<td>Q3</td>
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<tr>
<td>2.2.1 – Amine functionalized aerogel (AFA) sorbent further optimization</td>
<td>Aspen Team member</td>
<td>Q4</td>
<td>Q1</td>
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<td>2.2.2 – AFA Bead Fabrication</td>
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<td>Q2</td>
<td>Q3</td>
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<tr>
<td>2.2.3 – Small scale AFA bead production (&lt;1kg)</td>
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<td>Task 3.0 – (UA) Physical characterization of sorbents</td>
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<tr>
<td>Task 4.0 – (UA) Determination of CO₂ capture capacity and stability (4 - 15)</td>
<td>UA PhD. Student B Team member</td>
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<td>Task 5.0 – (UA) Fabrication of a Kg scale unit. (12-18)</td>
<td>UA PhD. Student B Team member</td>
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<td>Task 6.0 – (UA) Vacuum swing test (6-18)</td>
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<td>Task 7.0 – (UA/Aspen) Cost Analysis and Life Cycle Analysis.</td>
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<td>Task 8.0 – Report preparation Final TMP, Draft of Final Report</td>
<td>UA PhD. Student B Aspen Team member</td>
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</table>
The Nature of Adsorbed Carbon Dioxide on Immobilized Amines during Carbon Dioxide Capture from Air and Simulated Flue Gas

Absorbance

Wavenumber / cm⁻¹

Weakly ads.
Strongly ads.
Total ads.

3367 3297 2999 2933 2809 2456 2142 1685 1639 1556 1411

15 %
1 %
0.2 %
0.04 %

Weakly adsorbed CO₂
Strongly adsorbed CO₂
Scope of Work

Task 2.0 – Sorbent Preparation and fine-tuning
Subtask 2.1 – Fabrication of hybrid sorbents

Focus on the development of the hierarchical polymer structure to immobilize the sorbent particles.

Subtask 2.2 – Preparation of amine-functionalized aerogel
8 amine-aerogel samples have been prepared.

Task 4.0 – Determination of CO₂ capture capacity and stability
CO₂ capture capacity for thermal swing adsorption have been determined. Investigate the approaches to accelerate desorption of weakly adsorbed CO₂.

Task 5.0 – Fabrication of a kg scale vacuum swing adsorption (VSA) unit
A tubular absorber with vacuum seals have been tested.
Hydrophilic Crosslinked Porous Poly(vinyl alcohol)

**Objective:** Prepare a porous PVA (Poly(vinyl alcohol)) film impregnated with PEI.

- 1.5g PVA
- 3ml H$_2$O
- 80°C
- 30 mins

- 3ml GA
- 80°C
- 3ml H$_2$O
- 25°C
- 5 mins

- 0.09g GA
- 80°C
- 3g Aq. PVA
- 100°C
- 2 hrs

- HCL
- 55°C
- 800 ml Acetone
- 30 mins

- PVA + GA
- Drying @ 25°C
- 30 mins

*Base = PVA
*CL = Crosslinker = Glutaraldehyde

1.5g Base
3ml CL
0.04g PEI
1g Acetone
(pH = 1)

1.5g Base
3ml CL
0.02g PEI
800ml Acetone
(pH = 1)
What are Aerogels?

Nanoporous solid with a specific structural morphology......

• Open structure - up to 99% open porosity
• Pore diameters = ~ 10-40 nm on average (< 1/30,000th the width of a human hair)
• Nanoporosity slows heat and mass transport, providing record-low thermal conductivity values.

Si(OR)$_4$ + 2 H$_2$O $\rightarrow$ (SiO$_2$)$_x$ + 4 ROH

......and the method of production

1. Sol-gel Processing
2. Aging
3. Extraction (solvent exchange)
4. Drying (with supercritical fluids)
Task 2.0 – Sorbent Preparation and fine-tuning

Subtask 2.2 – Preparation of amine-functionalized aerogel

Subtask 2.2.1 – Amine Functionalized Aerogel (AFA) Sorbent Further Optimization

The Recipient will design AFA formulation to maximize hydrophobicity with different primary, secondary and tertiary amines for CO2 capture. The diamine linker with a secondary amine for weakly adsorbed CO2 will be incorporated into AFA formulation.

Subtask 2.2.2 – AFA Bead Fabrication

Optimum bead sorbent will be selected and produced for scale-up production to an amount up to 1 kg.
AFA Bead Fabrication

- The optimum AFA formulation will be used to fabricate AFA sorbents in the form of beads.

- The bead size and bead quality will depend on the:
  - Gel time of the AFA sol; and, the
  - Mixing speed and temperature of the “dispersing medium”

**Type #1 Sorbent Beads**
- Translucent
- 300 – 900 µm
- ~ 0.7 g/cc- packed beads
- $S_{BET} = 389 \text{ m}^2/\text{g}$
- Ave. Pore diameter = 10 nm

**Type #2 Sorbent Beads**
- Opaque
- 300 – 1,000 µm
- ~ 0.8 g/cc- packed beads
- $S_{BET} = 292 \text{ m}^2/\text{g}$
- Ave. Pore diameter = 22 nm
## Our results

<table>
<thead>
<tr>
<th>Amine</th>
<th>Support</th>
<th>Ads. $t_{1/2}$/s</th>
<th>Conc. CO$_2$%</th>
<th>$T_{ads}$°C</th>
<th>CO$_2$ capacity (mmol/g)</th>
<th>Amine efficiency (mol CO$_2$/mol N)</th>
<th>Fraction of weakly-adsorbed CO$_2$</th>
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<tbody>
<tr>
<td>PEG/lysine (0.3:1)</td>
<td>SiO$_2$</td>
<td>10.5</td>
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<td>15% in Ar</td>
<td>0.61</td>
<td>0.18</td>
<td>39%</td>
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<td>40</td>
<td>0.46</td>
<td>0.21</td>
<td>87%</td>
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<tr>
<td>Glycerol/lysine (1.8:1)</td>
<td></td>
<td>2.7</td>
<td></td>
<td></td>
<td>0.94</td>
<td>0.28</td>
<td>60%</td>
<td></td>
</tr>
<tr>
<td>PEG/lysine (0.3:1)</td>
<td></td>
<td>16.5</td>
<td></td>
<td></td>
<td>0.58</td>
<td>0.17</td>
<td>24.5%</td>
<td></td>
</tr>
<tr>
<td>PEG/lysine (1:1)</td>
<td></td>
<td>-</td>
<td></td>
<td></td>
<td>0.41</td>
<td>0.19</td>
<td>87%</td>
<td></td>
</tr>
<tr>
<td>PEG/lysine (2:1)</td>
<td></td>
<td>-</td>
<td></td>
<td></td>
<td>0.15</td>
<td>0.10</td>
<td>88%</td>
<td></td>
</tr>
</tbody>
</table>

### Footnotes:

- Link: [Link to more details on the Glycerol/lysine (1.8:1) system]