Metal Monolithic Amine-grafted Zeolites for CO_2 Capture

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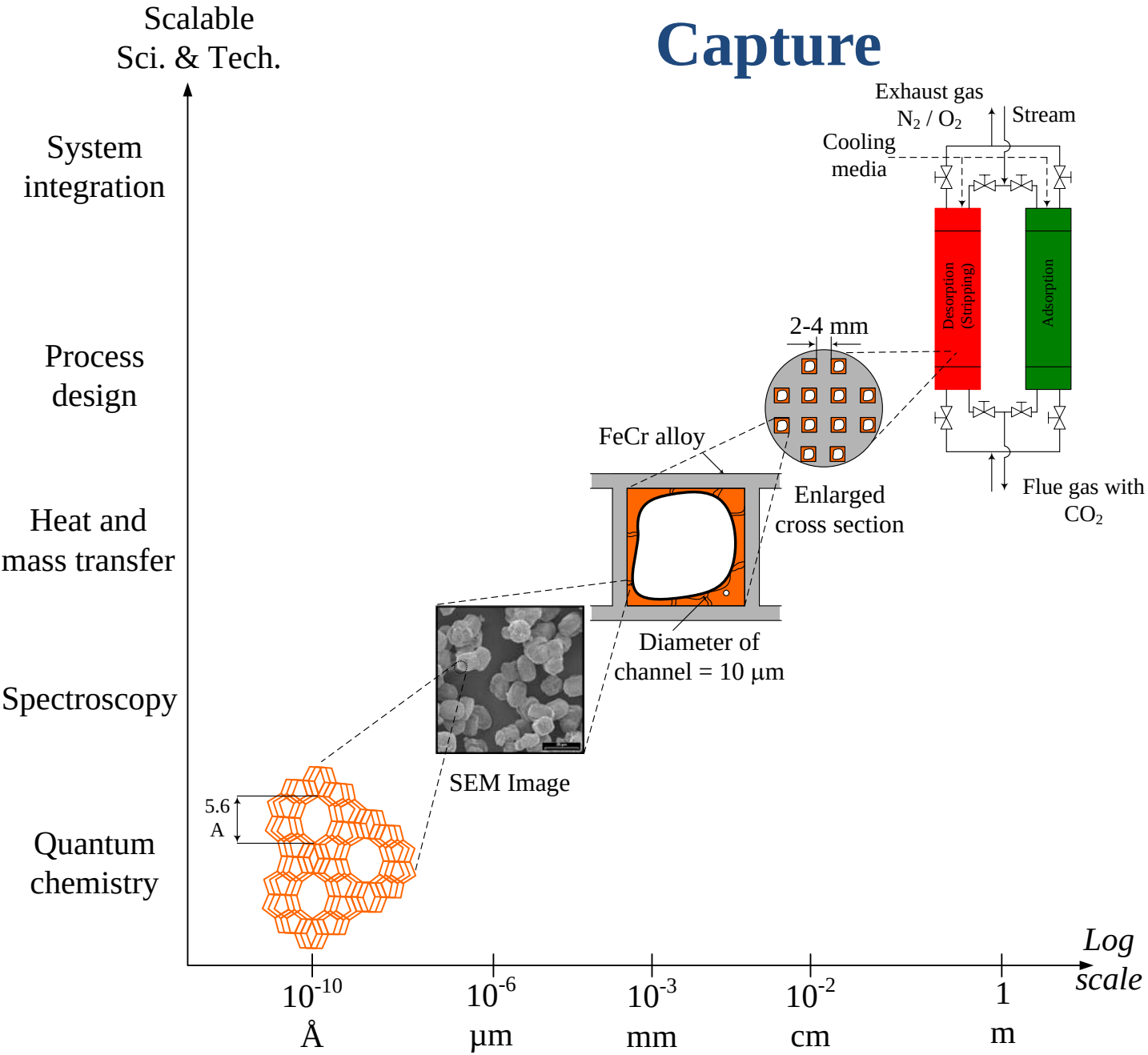
Acknowledgment:
DOE Project Manager: I. Andrew Aurelio and Tim Fout
U.S. Department of Energy, DE-FC26-07NT43086
FirstEnergy, Ohio Coal Development Office, Ohio Board of Regents; Babcock&Wilcox,

Project Overview

- Funding:
  - DOE: $764,964
  - The University of Akron: $192,838
- Date: 2/21/2007 – 12/31/2010
- Project participant: FirstEnergy and Babcock&Wilcox
- Overall Project Objectives:
  - The objective of this project is to develop a low cost CO_2 capture technology by integration of metal monoliths with grafted amine sorbent.
  - Phase I objective: Develop highly efficient CO_2 absorbents.
    - The performance of the amine-grafted zeolite should be above 1,500 µmol/g for CO_2 capture and above 1,000 µmol/g for SO_2 capture.
  - Phase II objective: Develop a pilot scale CO_2 absorption system.

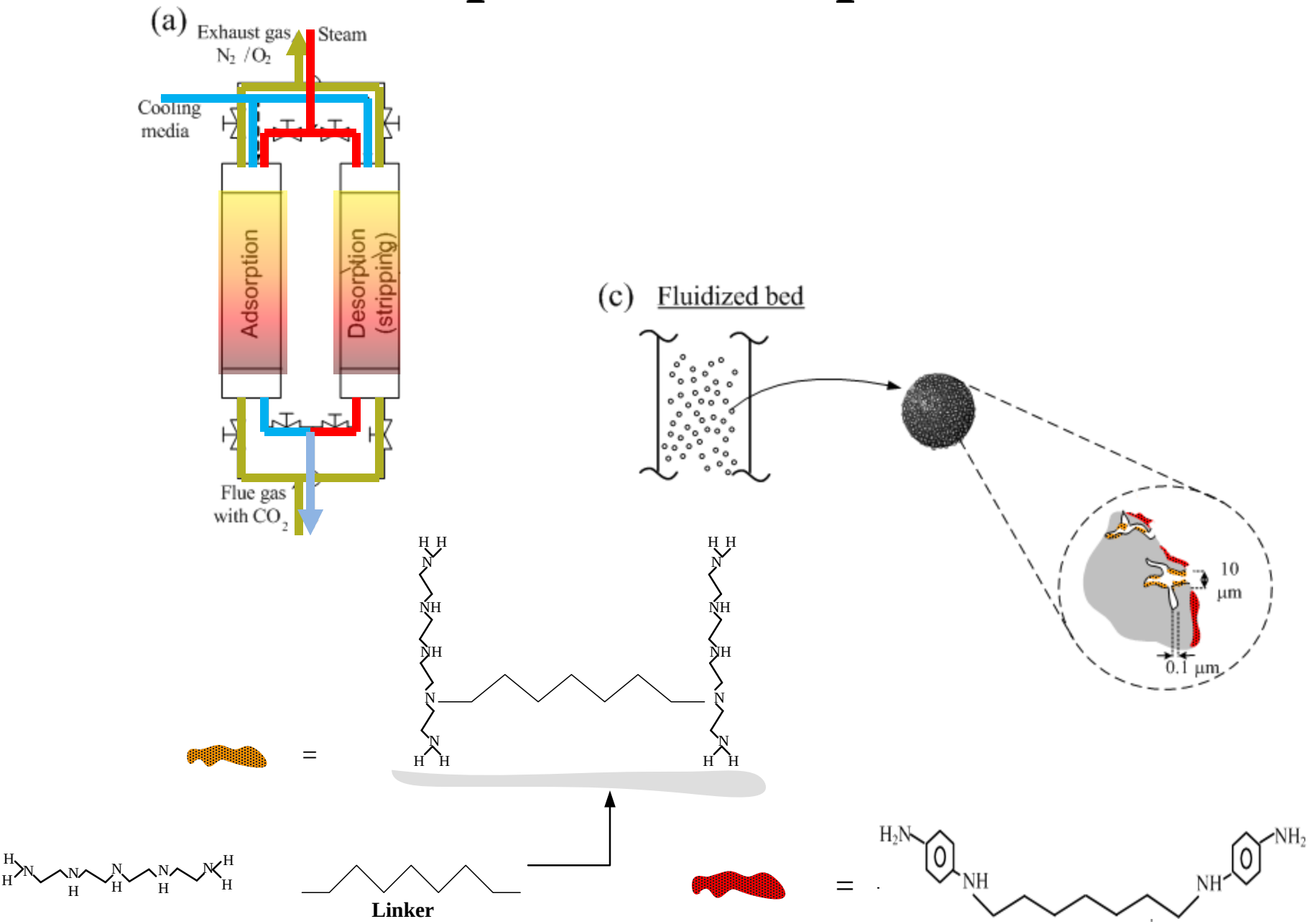
Metal Monolithic Amine-Grafted Zeolites for CO_2 Capture



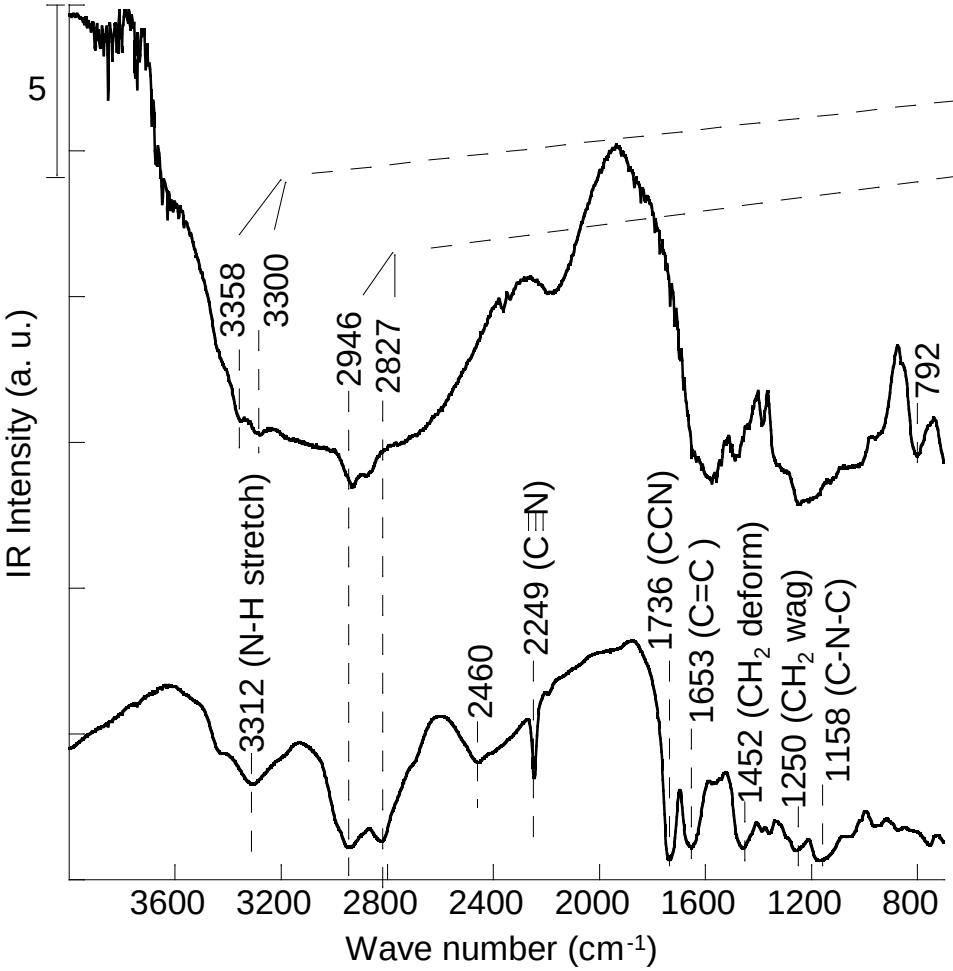
Unique features:

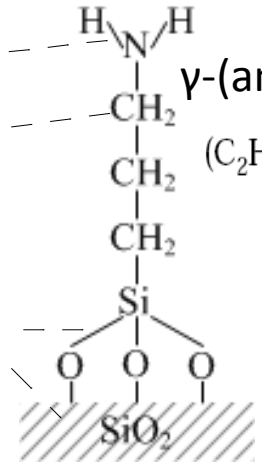
- High CO_2 adsorption capacity.
- Acceleration of the removal of adsorption heat.
- Low regeneration heat duty (i.e., low heat capacity of immobilized amine, zeolite, and metal monolith).
- Low cost immobilized amine sorbent.

Proposed Concept



DRIFTS single beam spectra of amine-grafted APTS-SBA-15 sample and commercial SA9-T sample





APTS-SBA-15

γ-(aminopropyl)triethoxysilane

$$(C_2H_5O)_3SiCH_2CH_2CH_2NH_2 + 3Si{-}OH \rightarrow (Si{-}O)_3{-}SiCH_2CH_2CH_2NH_2 + 3C_2H_5OH$$

Table 3. CO_2 Desorption Characteristics

	TPD in He flow			TPD in He/H_2O flow		
	amount of CO_2 des. (μmol/g)	peak temp. (K)	heat of CO_2 desorption (kJ/mol)	amount of CO_2 des. (μmol/g)	peak temp. (K)	Heat of CO_2 desorption (kJ/mol)
Fresh	227	393	6.67	308	375	4.54
1st Reg.	331	393	10.96	357	368	3.90
2nd Reg.	247	370	7.04	406	356	3.22

"In-Situ Infrared Study of CO_2 Adsorption on SBA-15 Grafted with -(Aminopropyl)- triethoxysilane", A. C. C. Chang, S. S. C. Chuang, M. Gray, and Y. Soong, Energy&Fuels, 17 (2), 468 -473, 2003.

Immobilized Amine CO_2 Capture Processess II

Where

C_{A0} = initial concentration

v_0 = volumetric flow rate

C_{PA} = heat capacity of gas

V = volume of fluidized bed

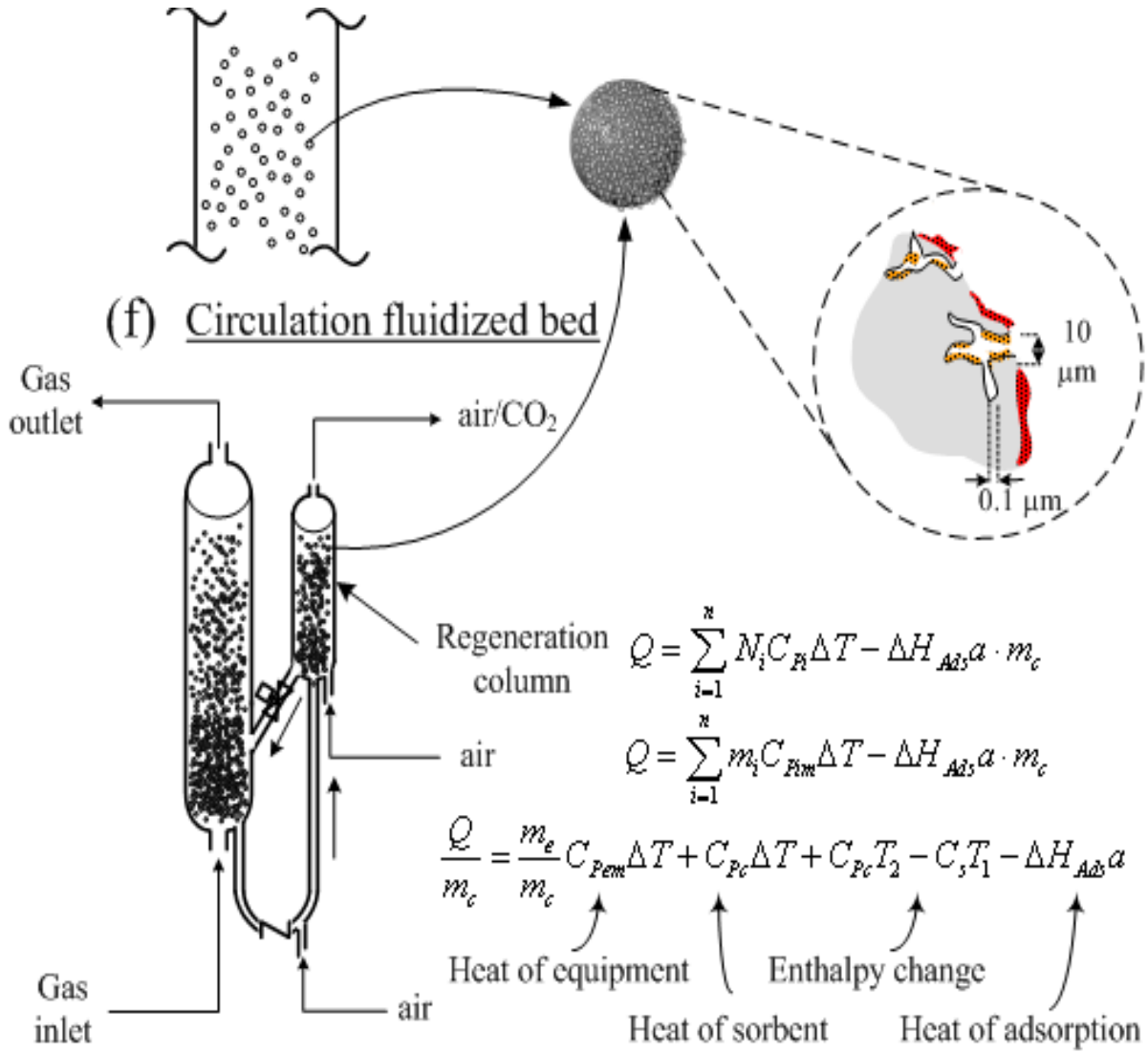
T_e = equipment surface temperature

$$\frac{\partial C_A}{\partial t} = \frac{v_0}{\varphi V}(C_{A0} - C_A) - R_A$$

$$\frac{\partial T}{\partial t} = \frac{\dot{Q} - v_0 C_{A0} C_{PA}(T - T_0) - \Delta H_{Ads} R_A V}{\sum_{i=1}^{n} N_i C_{Pi}}$$

$$\dot{Q} = U_a (T_e - T)$$

$$U_a = \frac{1}{A_0 / A_i h_i + (A_0 \ln(r_0 / r_i) / 2\pi k L + 1 / h_0}$$



$$Q = \sum_{i=1}^{n} N_i C_{Pi} \Delta T - \Delta H_{Ads} a \cdot m_c$$

$$Q = \sum_{i=1}^{n} m_i C_{Pim} \Delta T - \Delta H_{Ads} a \cdot m_c$$

$$\frac{Q}{m_c} = \frac{m_e}{m_c} C_{Pem} \Delta T + C_{Pc} \Delta T + C_{Pc} T_2 - C_s T_1 - \Delta H_{Ads} a$$

Project development: Phase 1 Results

Successful criteria:

• Develop sorbent capable of at least 1,500 μmol/g-sorb. CO_2 capacity.
• Achieve sorbent capable of greater than 500 times regeneration with less than 10% degradation in CO_2 capacity.

Accomplishments:

• Preparation, characterization, and determination of CO_2 capture capacity over 200 sorbents.
• Our sorbent achieved the target CO_2 capture capacity (i.e., 1,500 μmol-CO_2/g-sorb.) with an overall 13% degradation of its original activity after 500 cycles of CO_2 adsorption. T_{ad} = 40 ^oC T_{des} = 95 -100 ^oC.
•The issue of sorbent degradation was investigated by infrared spectroscopy (IR) studies, which revealed that the sorbent degraded via (i) evaporation of TEPA (tetraethylenepetamine) molecules, (ii) variation in configuration of TEPA and PEG (polyethylene glycol), and (iii) oxidation of TEPA.
• An inorganic species was found to be effective in slowing down oxidation of TEPA.
•Addition of more than 4% water vapor in the CO_2/air stream enhanced CO_2 capture capacity by more than 80%.

Project development: Phase 2 criteria and milestones

Successful criteria:

- Demonstrate a sorbent that has performance characteristics that translate to a process capable of achieving the DOE Carbon Sequestration Program goals for cost of electricity increase.

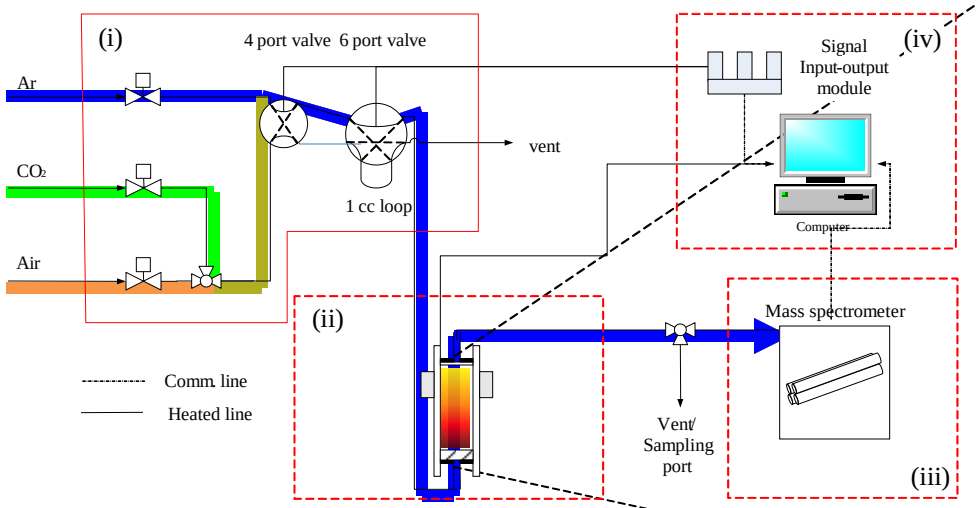
Milestones:

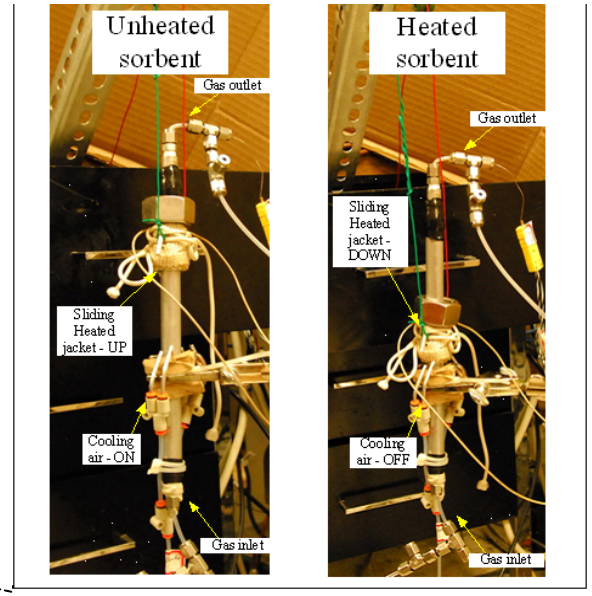
Task	Title	Verification Method	Planned Completion Date
A	- Binding the solid amine sorbents onto the metal foil. - Fabrication of metal foil structure and CO_2 adsorber unit.	- Submit a provisional patent on the approach for adhering amine sorbents onto metal foils.	09/2010
B	Determination of sorbent and adsorber performance.	- CO_2 capture capacity and thermal stability will be determined by 100 cycles of CO_2 capture with simulated flue gas containing 5%O_2, 3%H_2O, 10-15 %CO_2, 50 ppm SO_2, and balanced N_2.	08/2010
C	-Develop an engineering model and conduct an economic analysis of a large scale CO2 capture unit. -Determine the overall cost of the CO2 capture process including the capital and operating costs.	- Compare the cost of the metal monolithic CO_2 capture process with those of liquid amine and NH_3 processes.	12/2010

Summary of literatures on immobilized amines

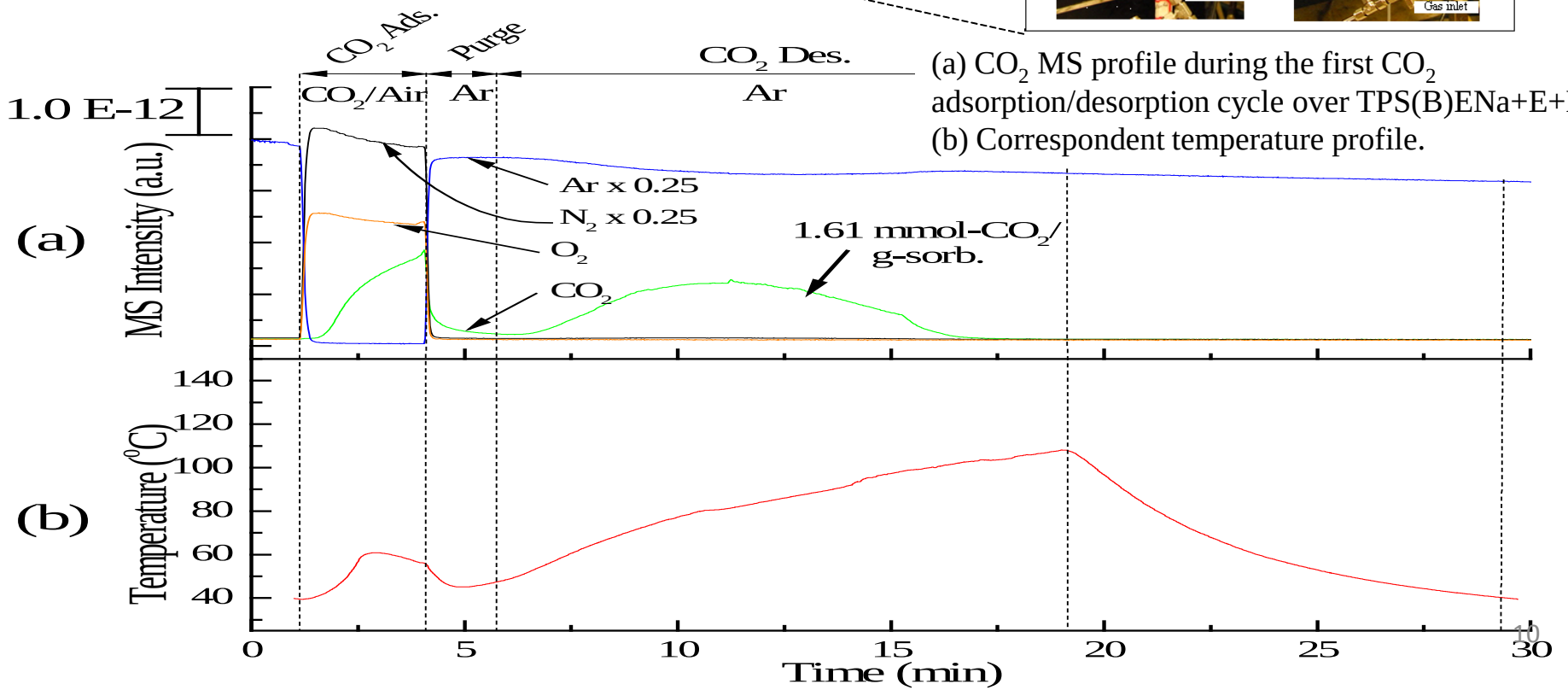
- The presence of H_2O enhanced the CO_2 capture capacity. The specific enhancement was not quantified.
- The presence of H_2O affects the accuracy in measuring the CO_2 capture capacity by TGA method
- Long term deactivation characteristics of the immobilized amines have seldom been reported in the literatures.

Fundamental study on solid amine sorbent

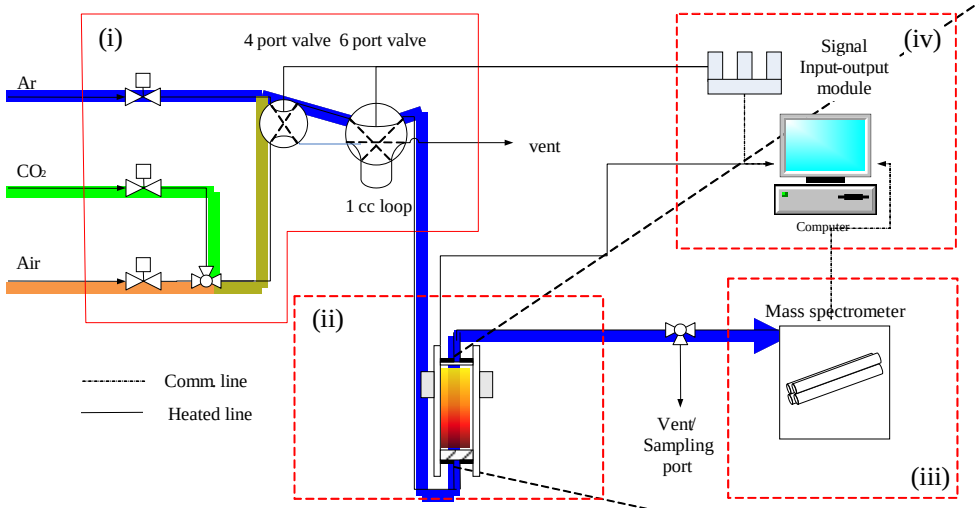


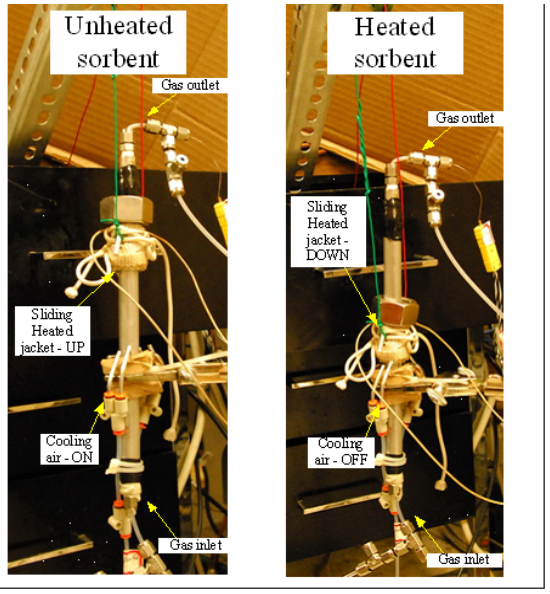


(a) CO_2 MS profile during the first CO_2 adsorption/desorption cycle over TPS(B)ENa+E+Na.
(b) Correspondent temperature profile.



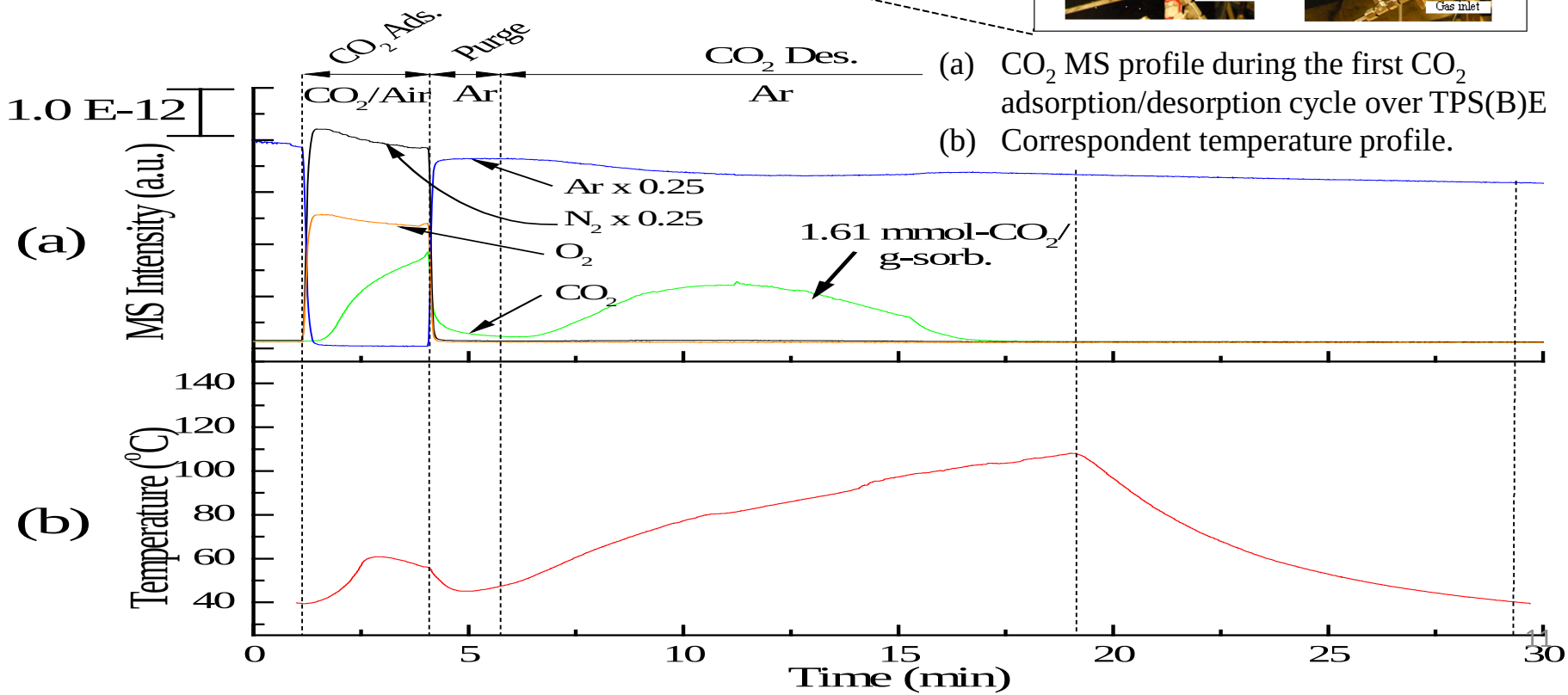
Fundamental study on solid amine sorbent



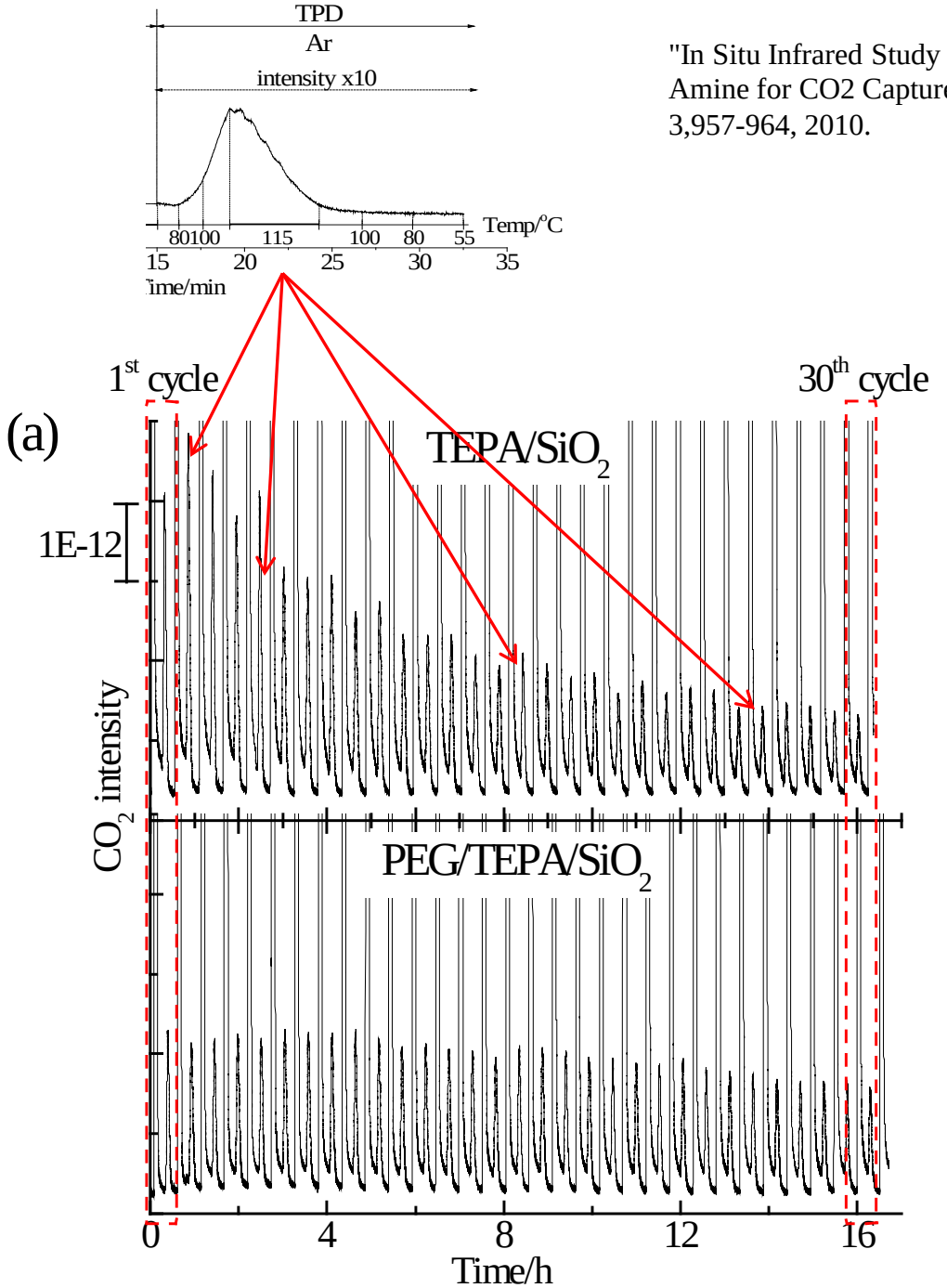


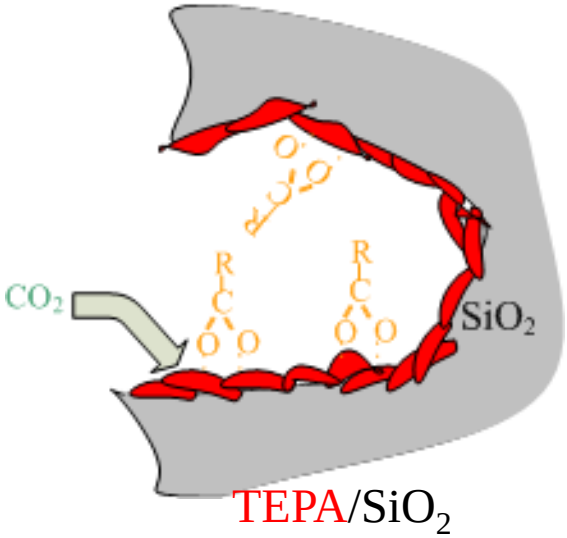
(a) CO_2 MS profile during the first CO_2 adsorption/desorption cycle over TPS(B)E

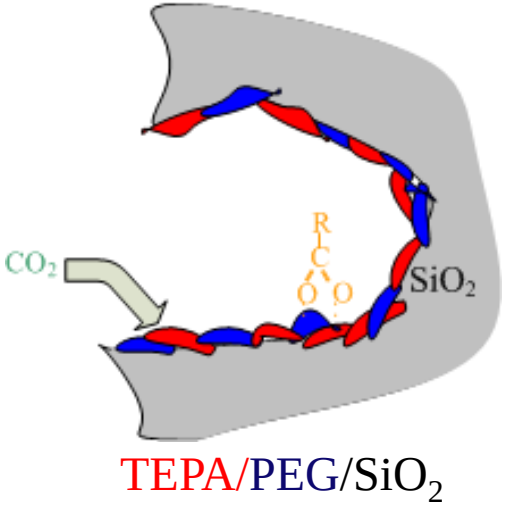
(b) Correspondent temperature profile.



"In Situ Infrared Study of the Role of PEG in Stabilizing Silica-supported Amine for CO2 Capture," J. Tanthana and S.S. C. Chuang, ChemSusChem, 3,957-964, 2010.







Sorbent Evolution

U.S. Provisional Patent Application

TEPA/Silica

- TS 9.1/90 – 0.77 mmol CO_2/g_{Sorb}
- TS 20/80 – 1.89 mmol CO_2/g_{Sorb}
- TS 33/66 – 3.20 mmol CO_2/g_{Sorb}
- TS 50/50 – 3.74 mmol CO_2/g_{Sorb}
- TS 60/40 – 2.38 mmol CO_2/g_{Sorb}

U.S. Provisional Patent Application

Sorbent Evolution

TEPA/Silica — TS 50/50, 3.74 mmol CO_2/g_{sorb} → Short Cycle Life

Add Dispersant (PEG) →

TEPA/PEG/Silica

- 10% PEG w/TEPA – 2.36 mmol CO_2/g_{Sorb}
- 20% PEG w/TEPA – 0.90 mmol CO_2/g_{Sorb}
- 30% PEG w/TEPA – 0.71 mmol CO_2/g_{Sorb}
- 10% PEG after TEPA – 1.80 mmol CO_2/g_{Sorb}
- 20% PEG after TEPA – 0.81 mmol CO_2/g_{Sorb}
- 30% PEG after TEPA – 0.61 mmol CO_2/g_{Sorb}

U.S. Provisional Patent Application

Sorbent Evolution

TEPA/Silica	TS 50/50 3.74 mmol CO_2/g_{sorb}	→ Short Cycle Life

Add Dispersant (PEG) →

TEPA/PEG/Silica	10% PEG w/TEPA 2.36 mmol CO_2/g_{sorb}	→ Short Cycle Life At Low Loading %

Adjust Weight Ratios →

TEPA/PEG/Silica

- TPS 12/48/40 – 1.18 mmol CO_2/g_{Sorb}
- TPS 24/36/40 – 2.56 mmol CO_2/g_{Sorb}
- TPS 30/30/40 – 2.58 mmol CO_2/g_{Sorb}
- TPS 36/24/40 – 2.54 mmol CO_2/g_{Sorb}

U.S. Provisional Patent Application

Sorbent Evolution

TEPA/Silica	TS 50/50 3.74 mmol CO_2/g_{sorb}	→ Short Cycle Life
↓ Add Dispersant (PEG) →		
TEPA/PEG/Silica	10% PEG w/TEPA 2.36 mmol CO_2/g_{sorb}	→ Short Cycle Life At Low Loading %
↓ Adjust Weight Ratios →		
TEPA/PEG/Silica	TPS 30/30/40 2.58 mmol CO_2/g_{sorb}	→ Degrades After 3 Cycles Due To TEPA Agglomeration
↓ Immobilization of Amine Groups →		
TEPA/PEG/Silica-Linker (L_A: Linker molecule)		

- 0.012 L_A: 5 TPS 24/36/40 – 2.34 mmol CO_2/g_{Sorb}
- 0.012 L_B: 5 TPS 24/36/40 – 2.31 mmol CO_2/g_{Sorb}

U.S. Provisional Patent Application

Sorbent Evolution

TEPA/Silica	TS 50/50 3.74 mmol CO_2/g_{sorb}	→ Short Cycle Life

↓ Add Dispersant (PEG) →

TEPA/PEG/Silica	10% PEG w/TEPA 2.36 mmol CO_2/g_{sorb}	→ Short Cycle Life At Low Loading %

↓ Adjust Weight Ratios →

TEPA/PEG/Silica	TPS 30/30/40 2.58 mmol CO_2/g_{sorb}	→ Degrades After 3 Cycles Due To TEPA Agglomeration

↓ Immobilization of Amine Groups →

TEPA/PEG/Silica-Linker (L_A: Linker molecule)	0.012 L_A:5 TPS 24/36/40 2.34 mmol CO_2/g_{sorb}	→ Maintained Higher Capture After 2 Days of Cycles

↓ L_A Ratio Optimization →

TEPA/PEG/Silica-L_A

- TPS-0.09L_A 24/36/40-100 – 2.22 mmol CO_2/g_{Sorb}
- TPS-0.13L_A 24/36/40-100 – 2.20 mmol CO_2/g_{Sorb}
- TPS-0.18L_A 24/36/40-100 – 1.77 mmol CO_2/g_{Sorb}
- TPS-0.13L_A 36/24/40-100 – 1.88 mmol CO_2/g_{Sorb}
- TPS-0.20L_A 36/24/40-100 – 1.91 mmol CO_2/g_{Sorb}
- TPS-0.27L_A 36/24/40-100 – 1.62 mmol CO_2/g_{Sorb}

U.S. Provisional Patent Application

Sorbent Evolution

TEPA/Silica	TS 50/50 3.74 mmol CO_2/g_{sorb}	→ Short Cycle Life
↓ Add Dispersant (PEG) →		
TEPA/PEG/Silica	10% PEG w/TEPA 2.36 mmol CO_2/g_{sorb}	→ Short Cycle Life At Low Loading %
↓ Adjust Weight Ratios →		
TEPA/PEG/Silica	TPS 30/30/40 2.58 mmol CO_2/g_{sorb}	→ Degrades After 3 Cycles Due To TEPA Agglomeration
↓ Immobilization of Amine Groups →		
TEPA/PEG/Silica-Linker (L_A: Linker molecule)	0.012 L_A:5 TPS 24/36/40 2.34 mmol CO_2/g_{sorb}	→ Maintained Higher Capture After 2 Days of Cycles
↓ L_A Ratio Optimization →		
TEPA/PEG/Silica-L_A	TPS-0.09L_A 24/36/40-100 2.22 mmol CO_2/g_{sorb}	→ Maintained High Capture (>2.00) Over 2 Days of Testing, Degraded On The Third Day Of Testing Due To Oxidation
↓ Inorganic Species To Help Resist Degradation →		
TEPA/PEG/Silica-L_A-Inorganic Species (I_A,I_B,I_C)		

- TPS-0.22L_A 24/36/40-100-0.05 I_A – 2.00 mmol CO_2/g_{Sorb}
- TPS-0.22L_A 24/36/40-100-0.25 I_A – 1.85 mmol CO_2/g_{Sorb}
- TPS-0.22L_A 24/36/40-100-0.05 I_B – 1.90 mmol CO_2/g_{Sorb}
- TPS-0.22L_A 24/36/40-100-0.05 I_C – 2.14 mmol CO_2/g_{Sorb}
- TPS-0.22L_A 24/36/40-100-0.25 I_C – 1.78 mmol CO_2/g_{Sorb}

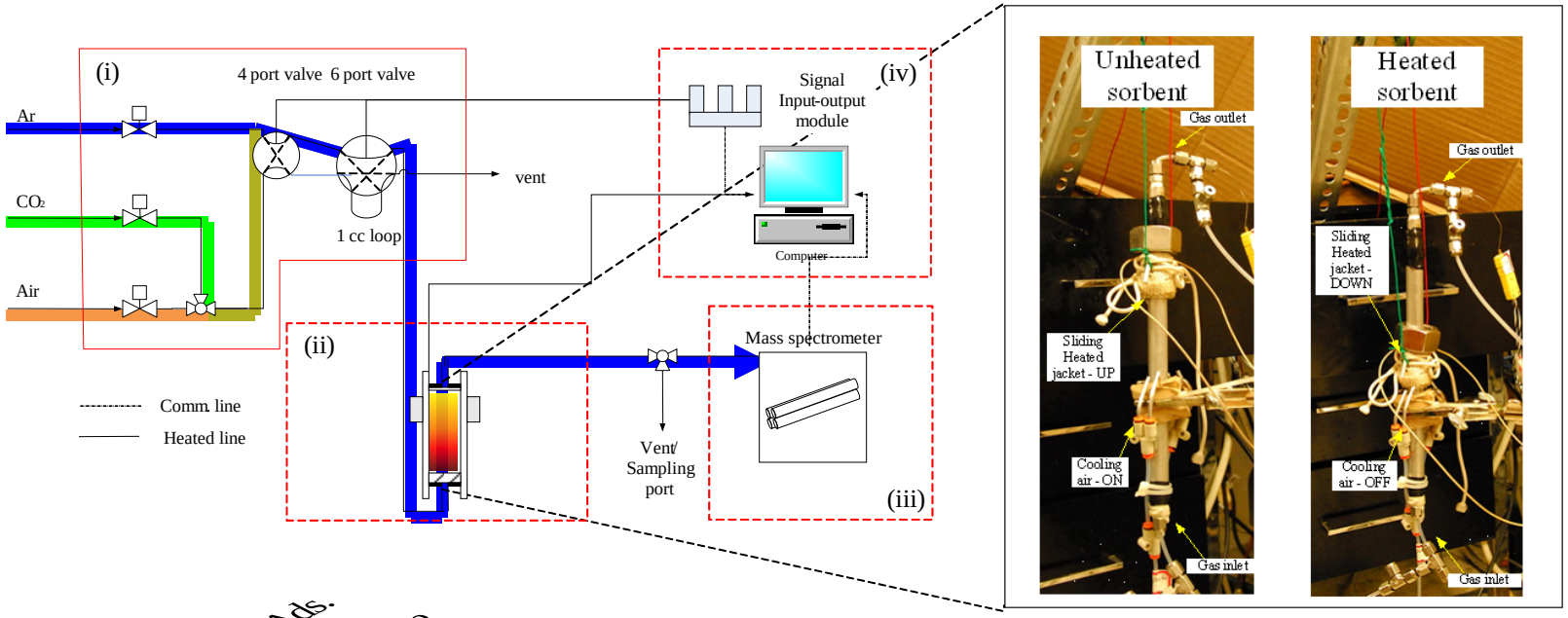
U.S. Provisional Patent Application

Sorbent Evolution

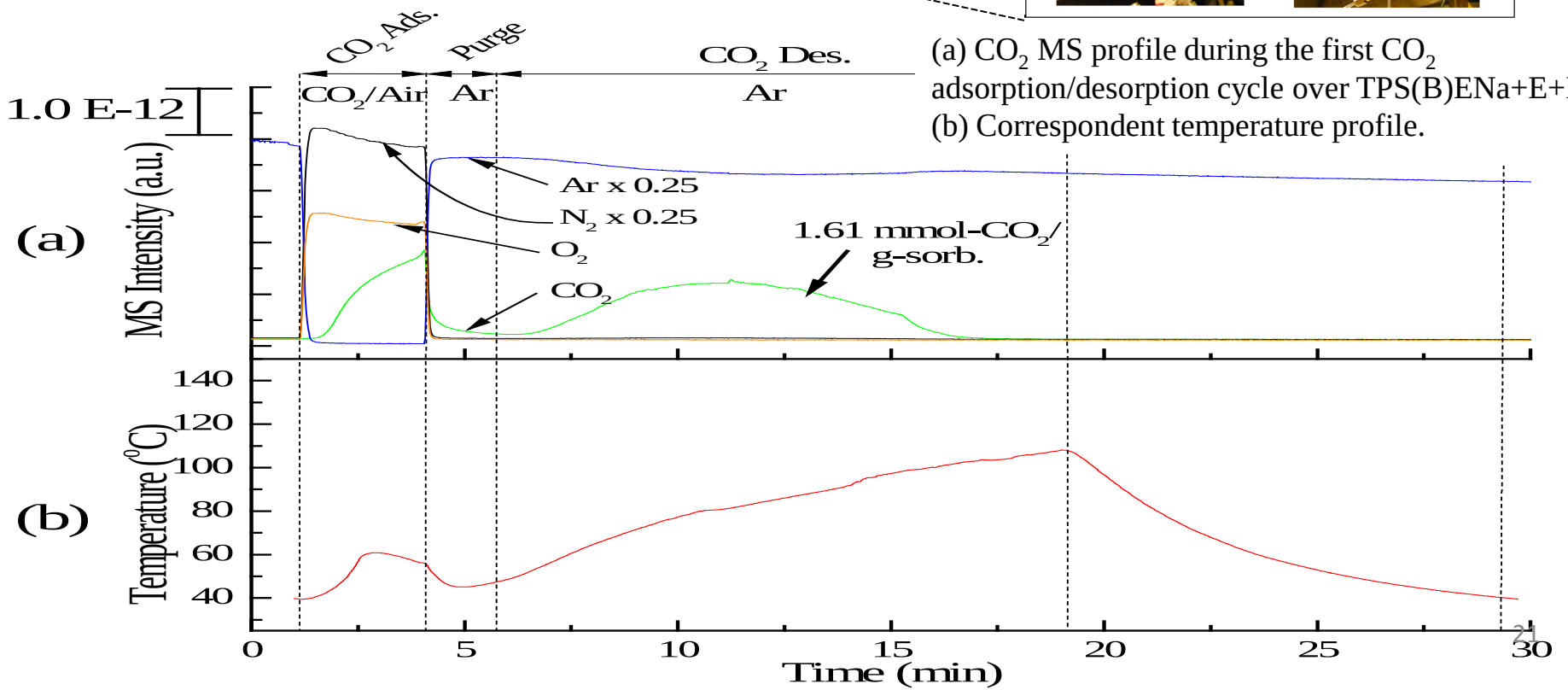
TEPA/Silica	TS 50/50 3.74 mmol CO_2/g_{sorb}	→ Short Cycle Life
↓ Add Dispersant (PEG) →		
TEPA/PEG/Silica	10% PEG w/TEPA 2.36 mmol CO_2/g_{sorb}	→ Short Cycle Life At Low Loading %
↓ Adjust Weight Ratios →		
TEPA/PEG/Silica	TPS 30/30/40 2.58 mmol CO_2/g_{sorb}	→ Degrades After 3 Cycles Due To TEPA Agglomeration
↓ Immobilization of Amine Groups →		
TEPA/PEG/Silica-Linker (L_A: Linker molecule)	0.012 L_A:5 TPS 24/36/40 2.34 mmol CO_2/g_{sorb}	→ Maintained Higher Capture After 2 Days of Cycles
↓ L_A Ratio Optimization →		
TEPA/PEG/Silica-L_A	TPS-0.09L_A 24/36/40-100 2.22 mmol CO_2/g_{sorb}	→ Maintained High Capture (>2.00) Over 2 Days of Testing, Degraded On The Third Day Of Testing Due To Oxidation
↓ Inorganic Species To Help Resist Degradation →		
TEPA/PEG/Silica-L_A-Inorganic Species (I_A,I_B,I_C)	TPS-0.22L_A 24/36/40-100-0.05 I_C 2.14 mmol CO_2/g_{sorb}	
Maintained Stability After Three Days Of Testing		

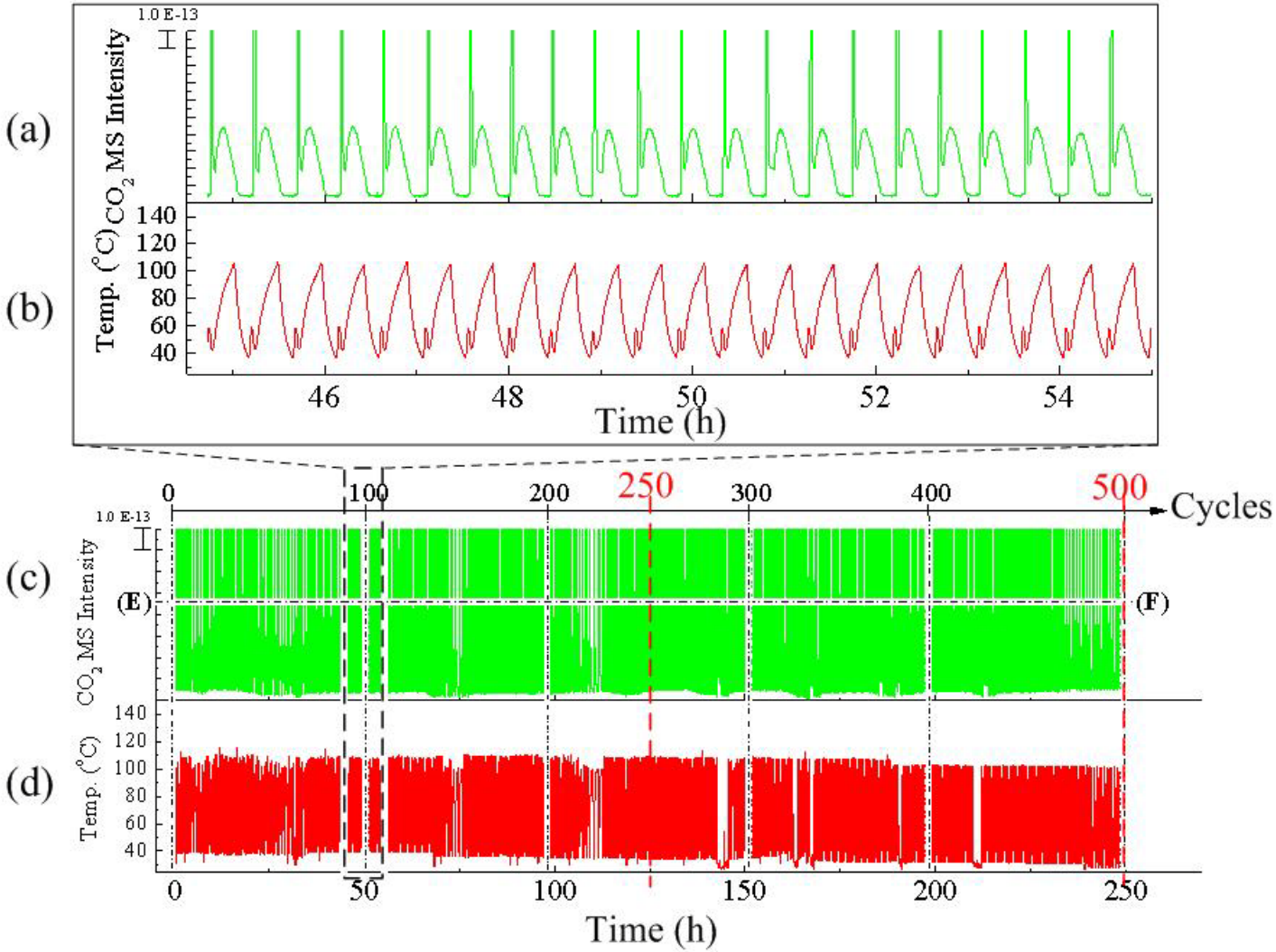
Physical properties of the sorbent

- Bulk Density: 0.43 g/cm^3
- Average particle size
  - before attrition test:270 µm
  - after 12 hours of attrition test: 81 µm for 2.08 wt% of the initial particles.
- Heat of Adsorption: 52 kJ/mol
- Heat Capacity: 1.49 kJ/kg K
- Adsorption halftime in 100 cm^3/min flow:0.13 min (7.8 s)



(a) CO_2 MS profile during the first CO_2 adsorption/desorption cycle over TPS(B)ENa+E+Na.
(b) Correspondent temperature profile.





(a). CO_2 MS profile during the 90-110 adsorption/desorption cycles over TPS(B)ENa+E+Na. (b) corresponded temperature profile. (c) overall MS CO_2 profile over 500 cycles. and (d) overall temperature profile.

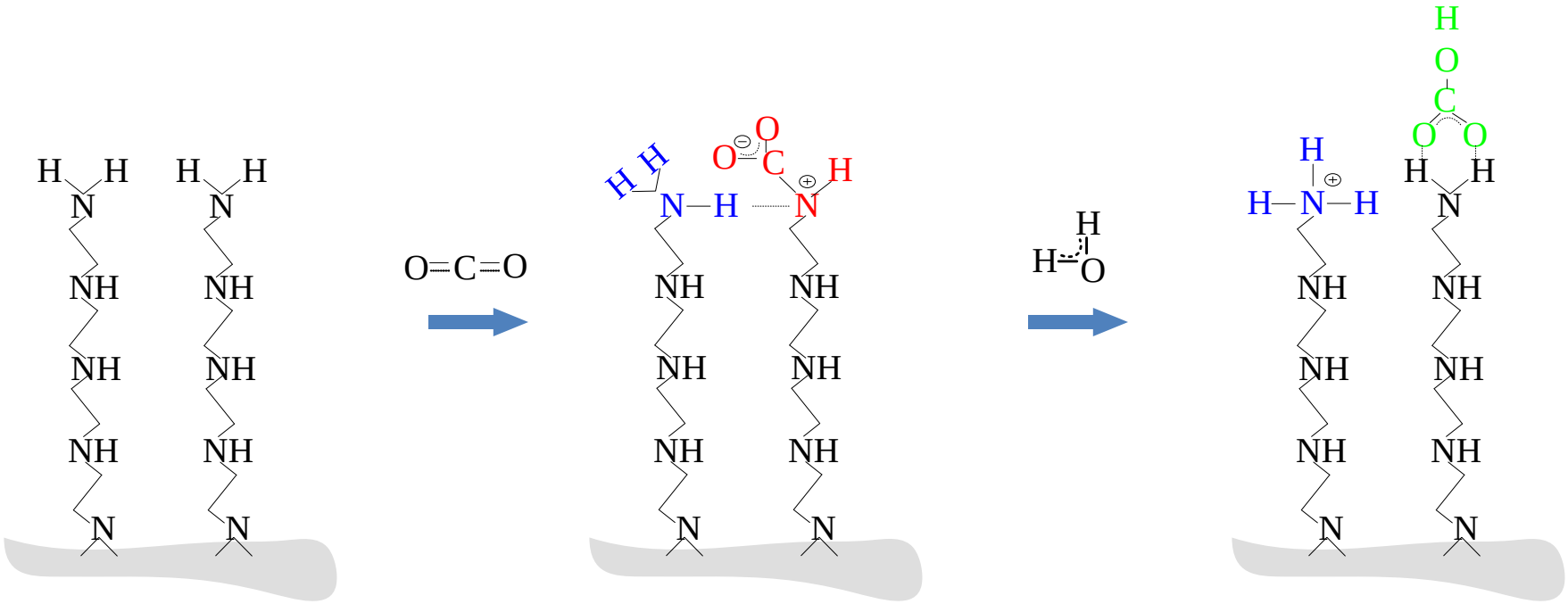
Amine-CO_2 reaction

Absence of H_2O:

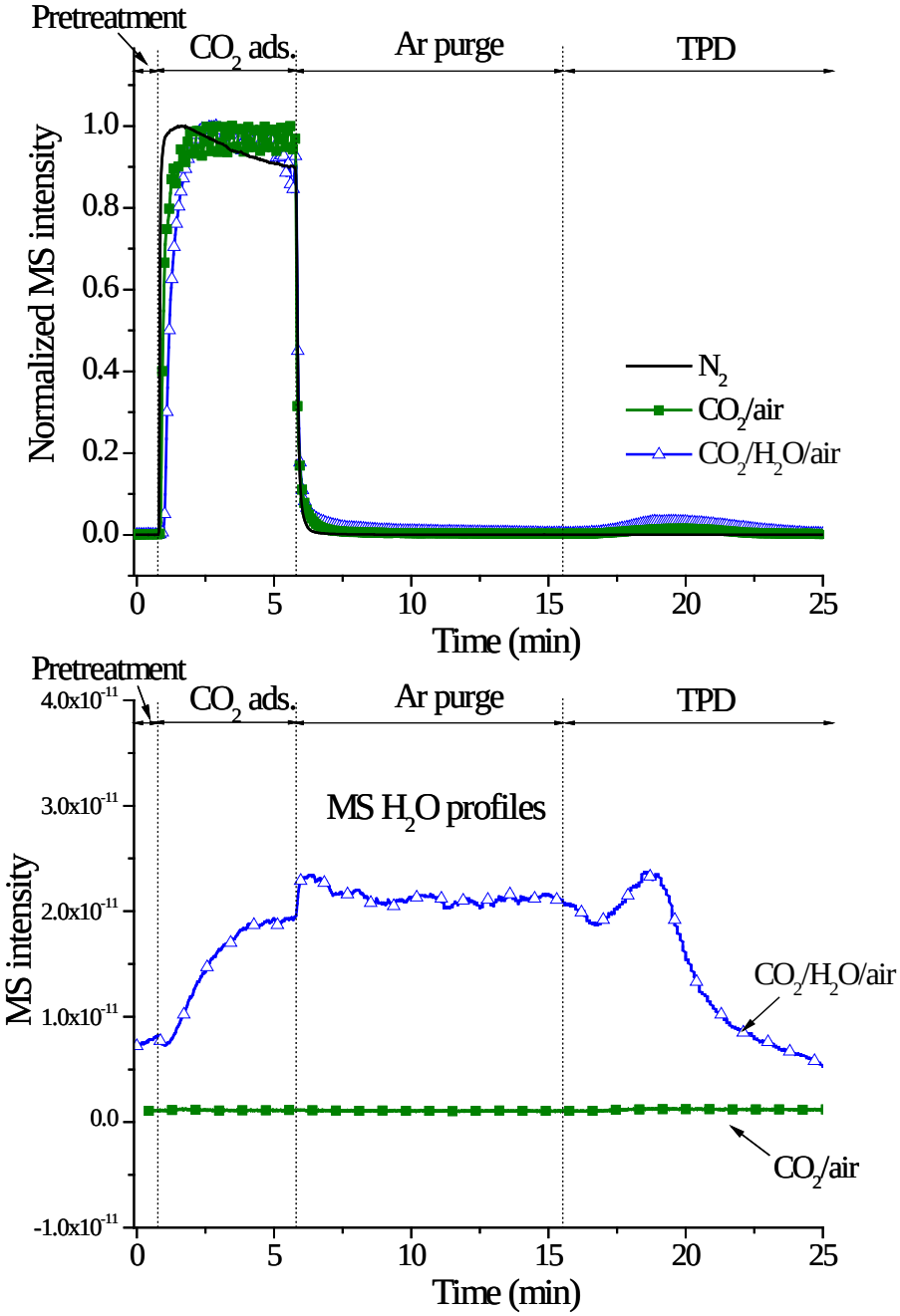
$$CO_2 + 2RNH_2 \leftrightharpoons \underset{\text{(ammonium ion)}}{RNH_3^+} + \underset{\text{(carbamate)}}{RNHCOO^-}$$

Presence of H_2O:

$$\underset{\text{(carbamate)}}{RNHCOO^-} + H_2O \leftrightharpoons RNH_2 + \underset{\text{(bicarbonate)}}{HCO_3^-}$$



Effect of H_2O on CO_2 capture on an immobilized amine sorbent



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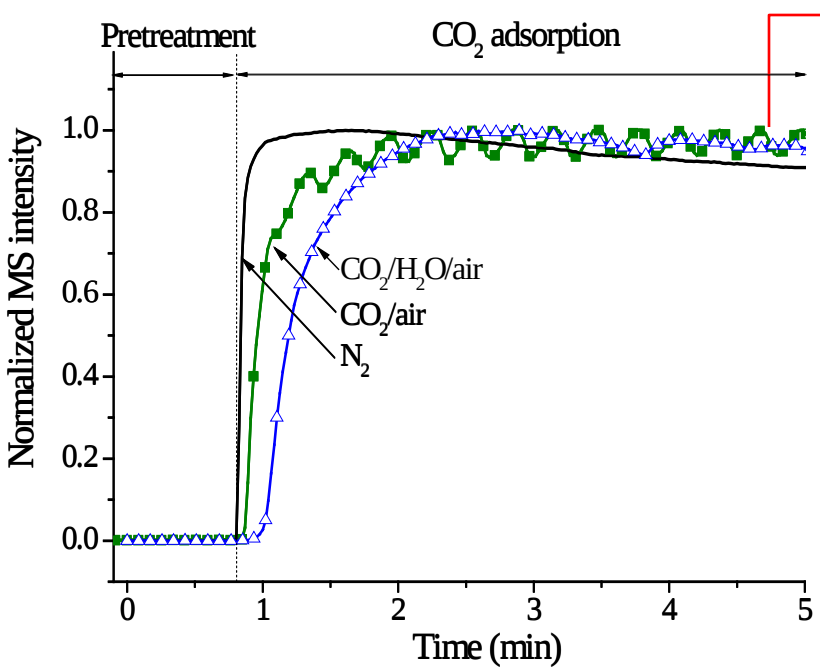

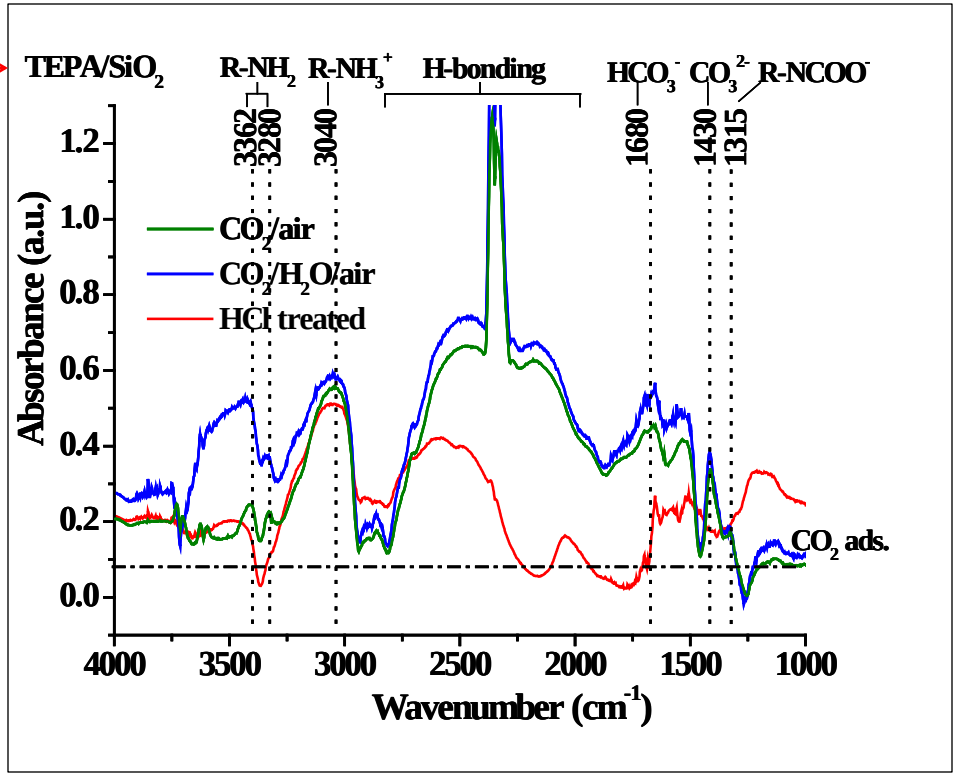

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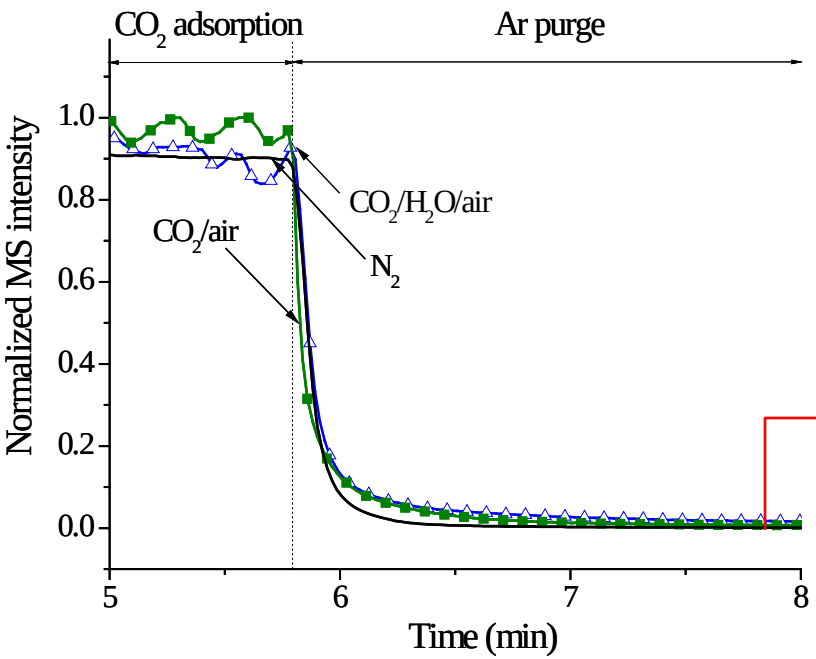

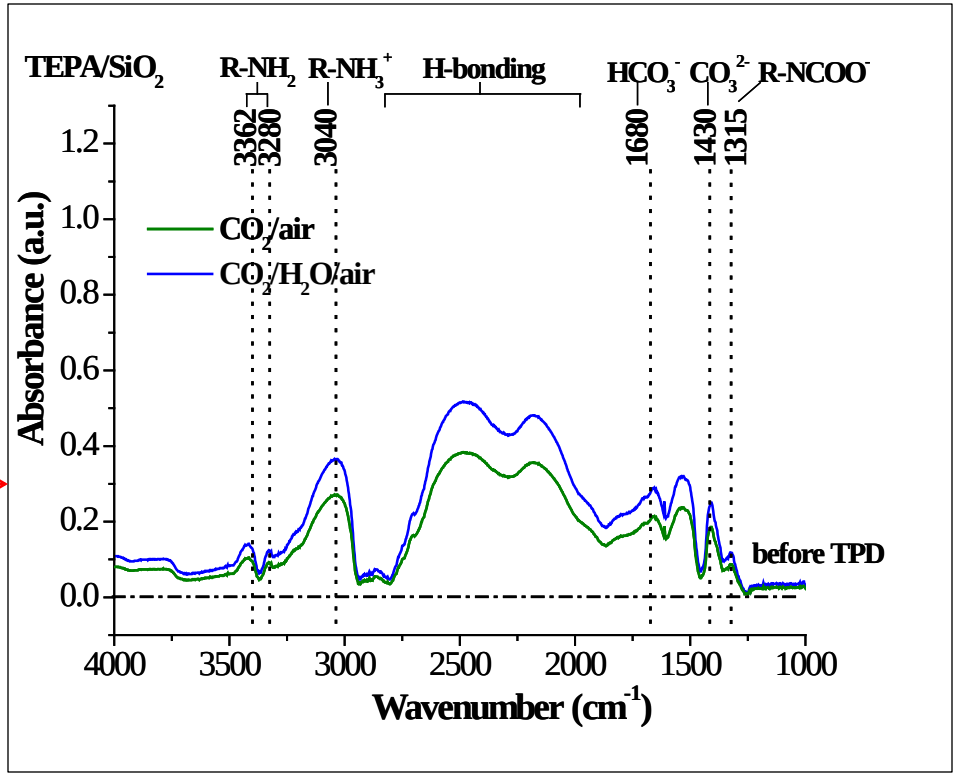

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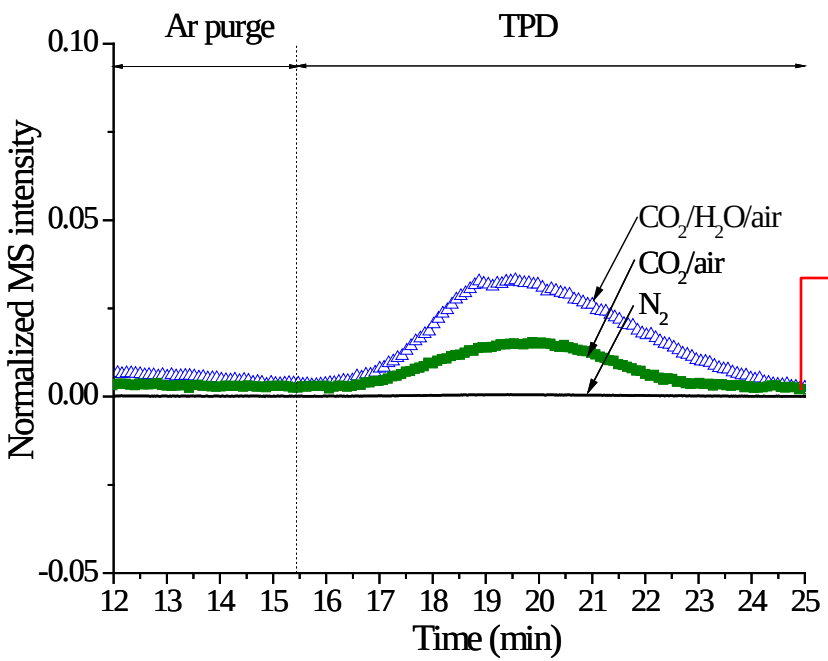
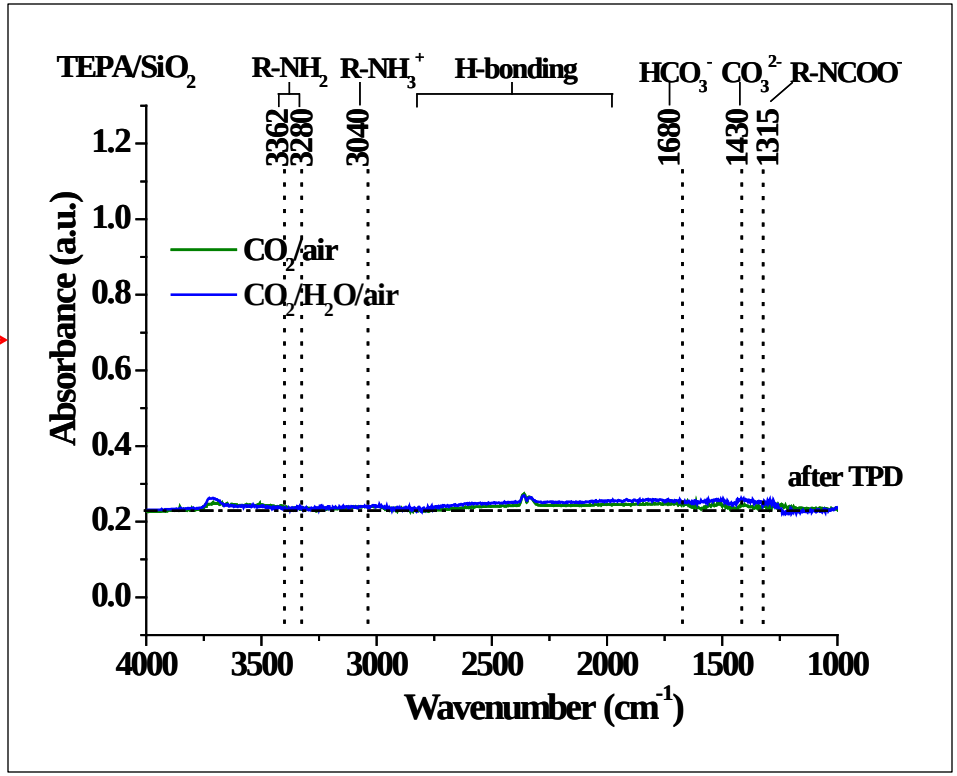
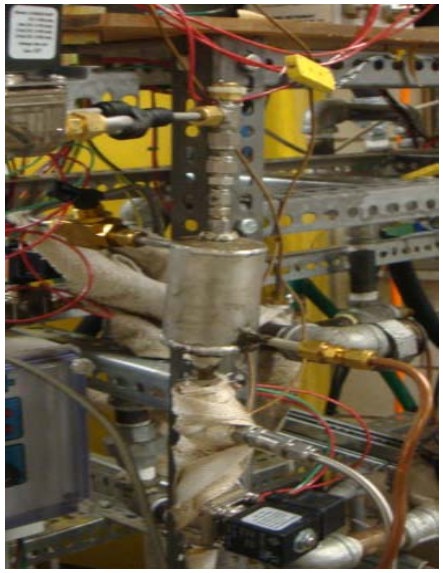


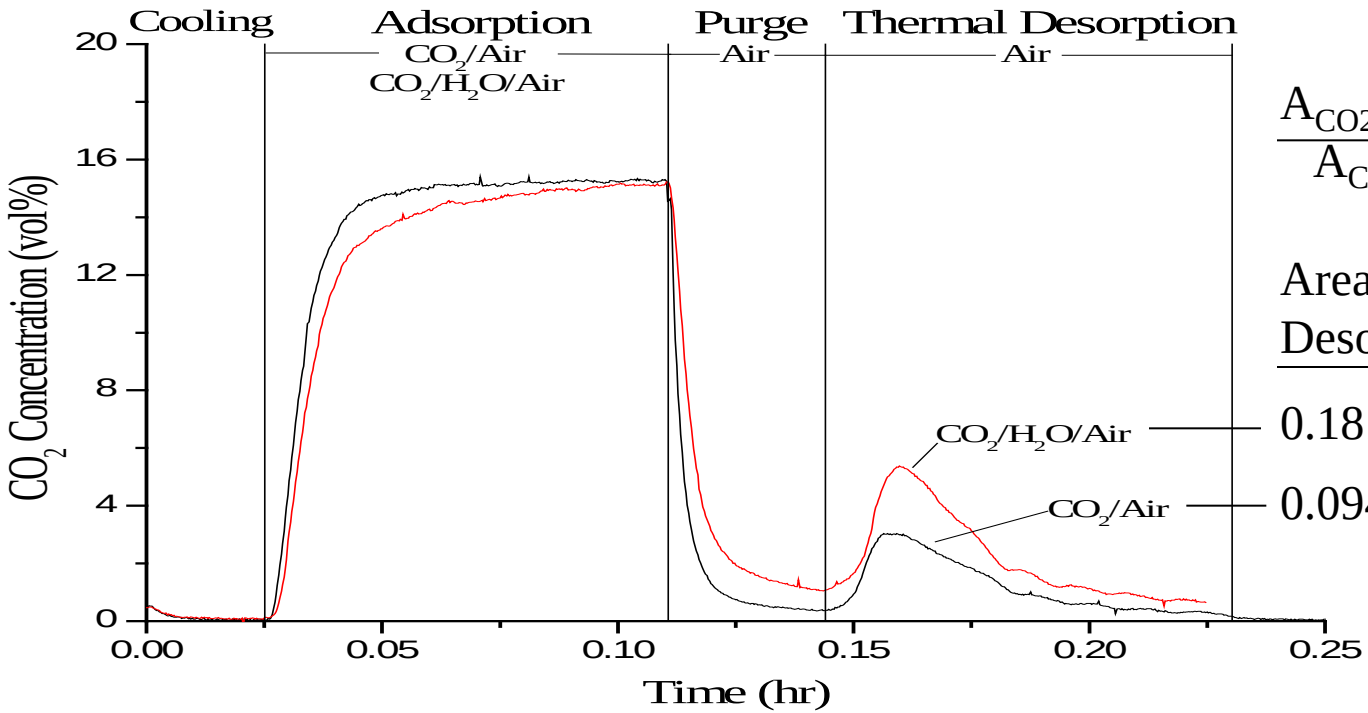


Effect of water on CO_2 capture (5 gram unit)



Experimental Conditions:

- Cooling – Reduce temperature from 100°C to 40°C
- Adsorption – 15% CO_2 @ 3L/min for 5 minutes
- Purge – Air @ 3L/min for 2 minutes
- Thermal Desorption – Increase temperature to 100°C with air @ 3L/min for 5 minutes



$$\frac{A_{CO2/H2O}}{A_{CO2}} = 1.92$$

Area of Desorption	
$CO_2/H_2O/Air$	0.1812
CO_2/Air	0.0942

Pellet Preparation

Formulation

TPSEN sorbent (Powder)

- Water
- Grafting agents
- TEPA
- Binders I
  - 1%
  - 2%
  - 10%
  - High cost
  - Low cost
- Binder II
  - 10% - Low cost
  - With inorg additives
  - With isocyanate
  - With grafting agents
- Binder III
  - 1% Polymer
  - 10% Polymer
  - Extra PEG

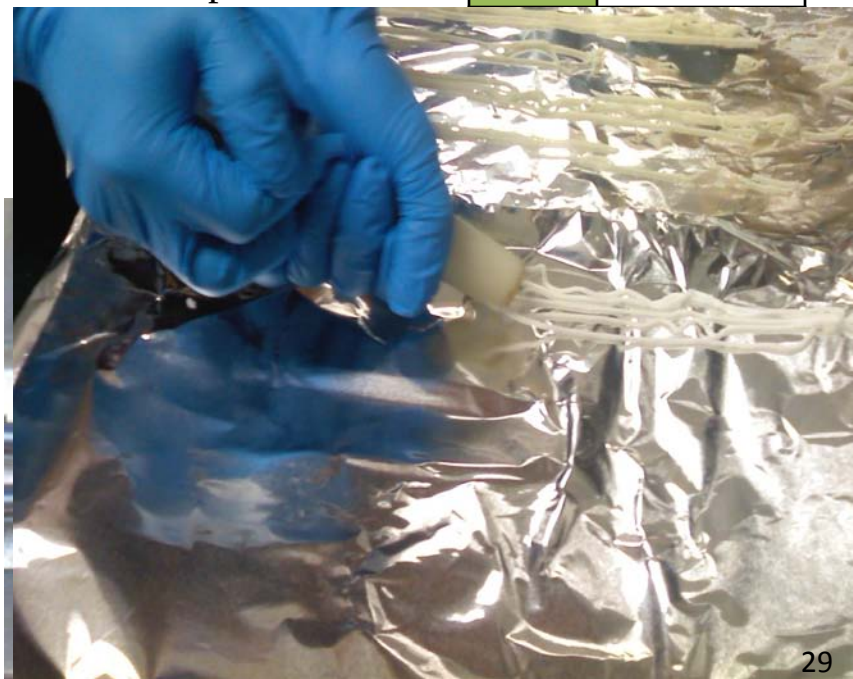
CO_2 capture mmol/g	
Initial	Degraded for 8 hr
1.40	1.20
1.96	0.90
1.68	0.61
1.43	0.60
2.14	0.74
2.36	0.73
1.60	0.74
1.60	0.74
1.88	0.96
1.86	1.30
1.10	0.74
1.33	1.10
1.78	1.38
1.48	1.08
2.29	1.20

Method of preparation

Process type

- Fluidization
- Droplets (lentil shape)
- Syringe extrusion
  - Pushing by hand
  - Syringe pump
  - Multiple holes

Strength	Size/ Shape
Weak	Uneven
Weak	3 mm / lentil
Strong	1 mm / rod
Strong	1 mm / rod
Strong	1 mm / rod



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1.78	1.38
1.48	1.08
2.29	1.20

Method of preparation

Process type	Strength	Size/ Shape
- Fluidization	Weak	Uneven
- Droplets (lentil shape)	Weak	3 mm / lentil
- Syringe extrusion		
• Pushing by hand	Strong	1 mm / rod
• Syringe pump	Strong	1 mm / rod
• Multiple holes	Strong	1 mm / rod
- Twin screw extruder		
• Low Temperature	Strong	1 mm / rod
• High Temperature	Weak	1 mm / rod

Pellet Preparation

Formulation

TPSEN sorbent (Powder)

- Water
- Grafting agents
- TEPA
- Binders I
  - 1%
  - 2%
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Method of preparation

Process type	Strength	Size/ Shape
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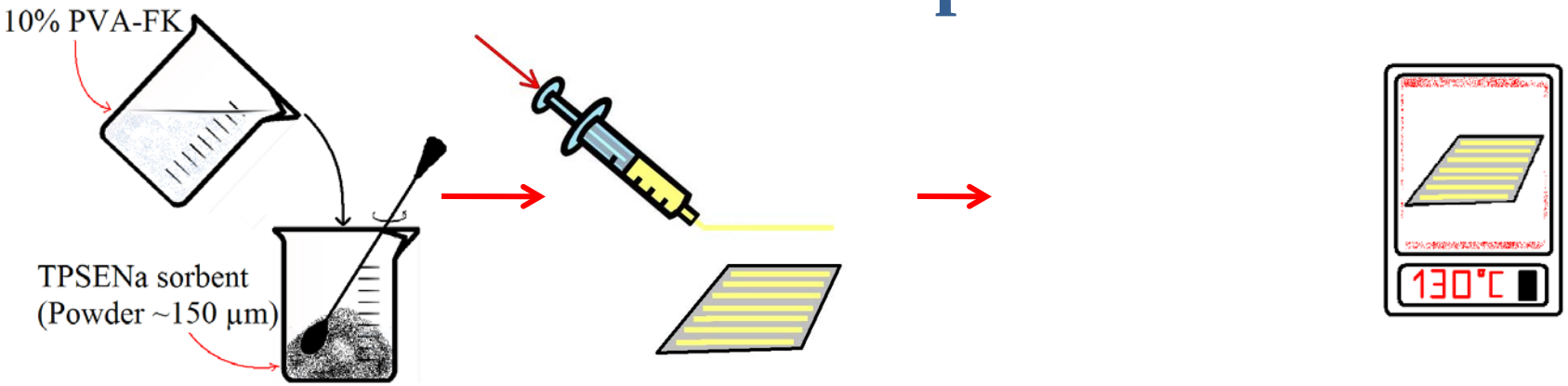
• Multiple holes	Strong	1 mm / rod
- Twin screw extruder		
• Low Temperature	Strong	1 mm / rod
• High Temperature	Weak	1 mm / rod
Slurry properties		
- Viscosity		
• Low viscosity	Weak	Uneven
•High Viscosity	Strong	Defined shape
- Mixing		
• Homogeneous	Strong	Defined shape
• Non homogeneous	Weak	Uneven

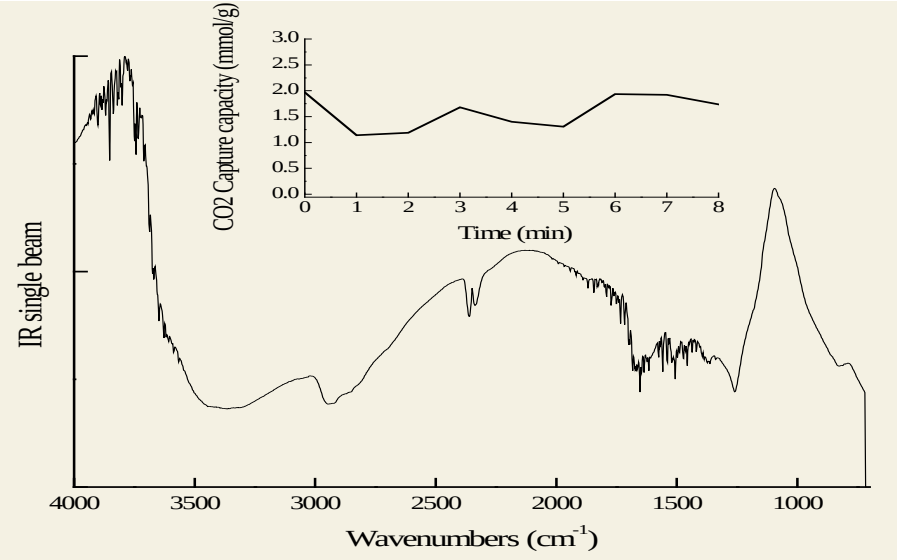
Pellet Preparation

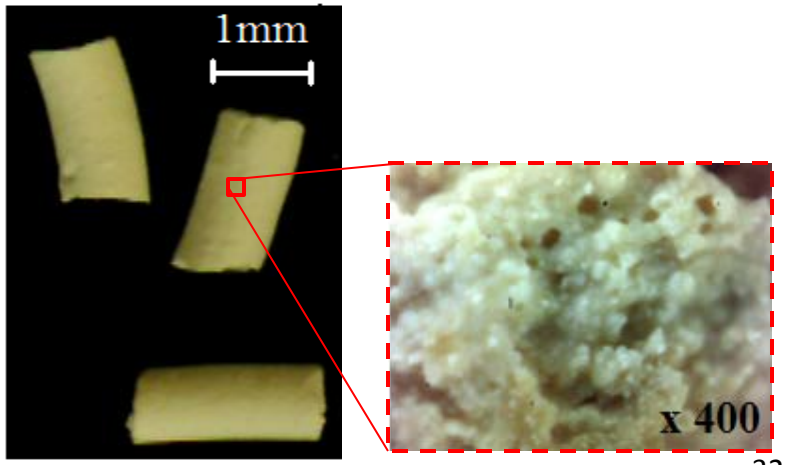


Mix well until getting a viscous paste → Load in a syringe and extrude 1mm rods → Crosslink for 15 min → Impregnate for 10 min → Drain and dry → Pellets (~1mm)

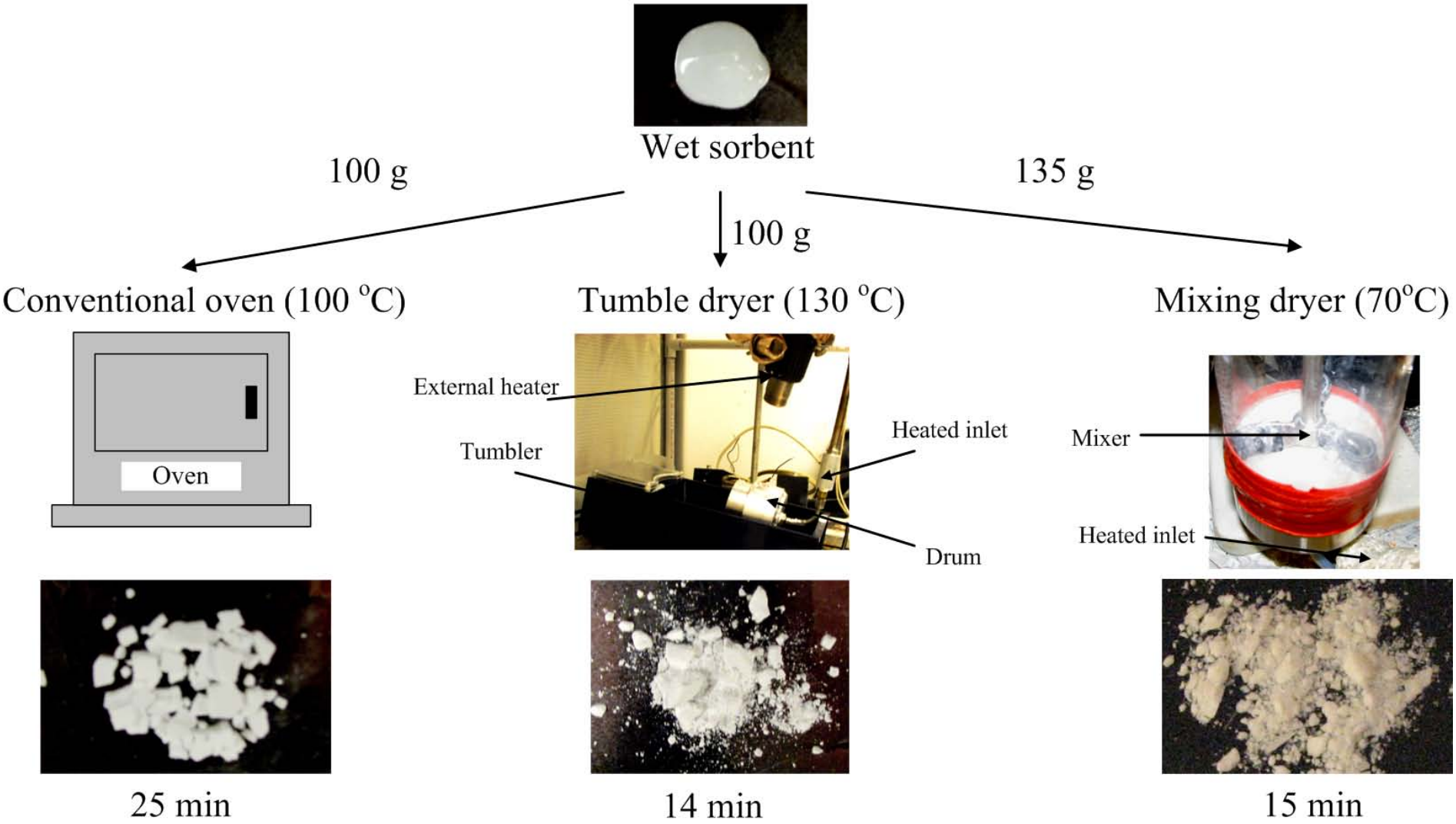
TPSENa	3 g
10% Binder II	1 g

Pellets	3 g
Inorganic (5%)	3 g





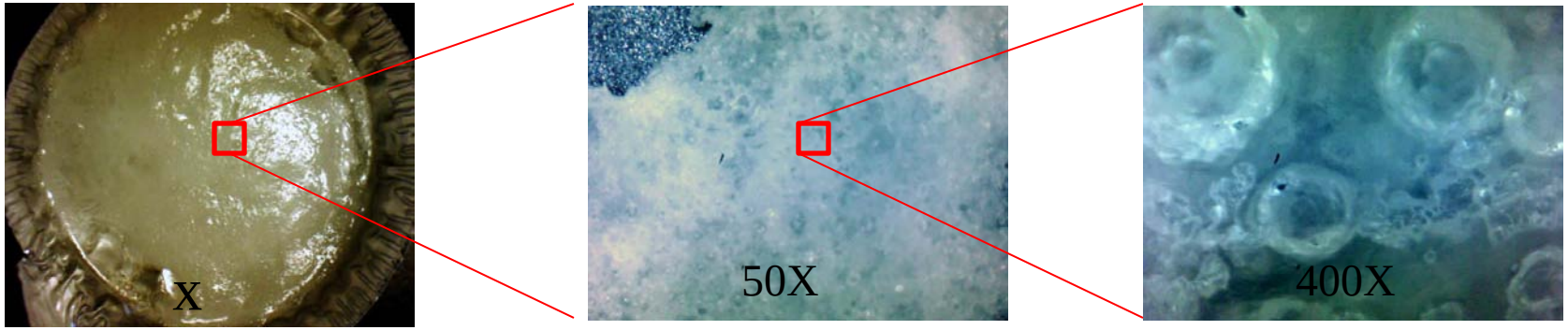
Methods of Drying

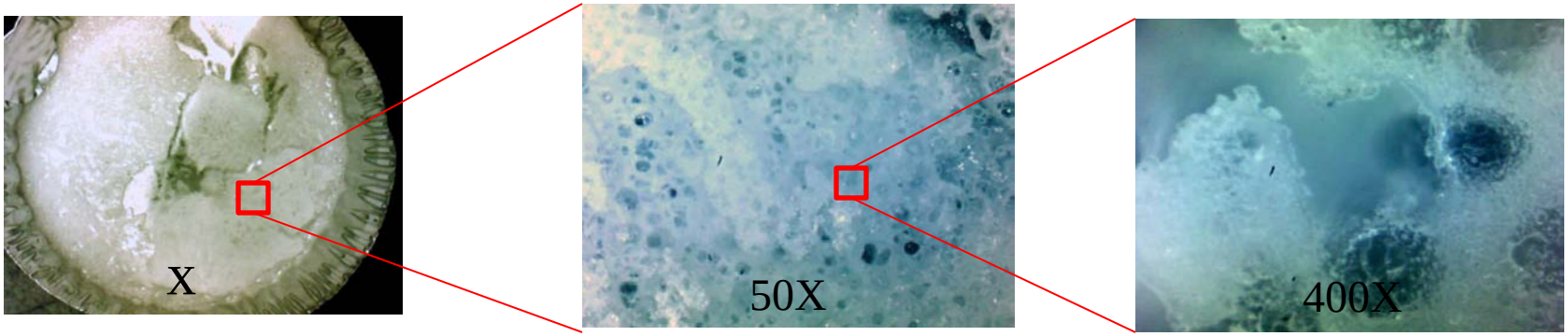


Metal Monolith

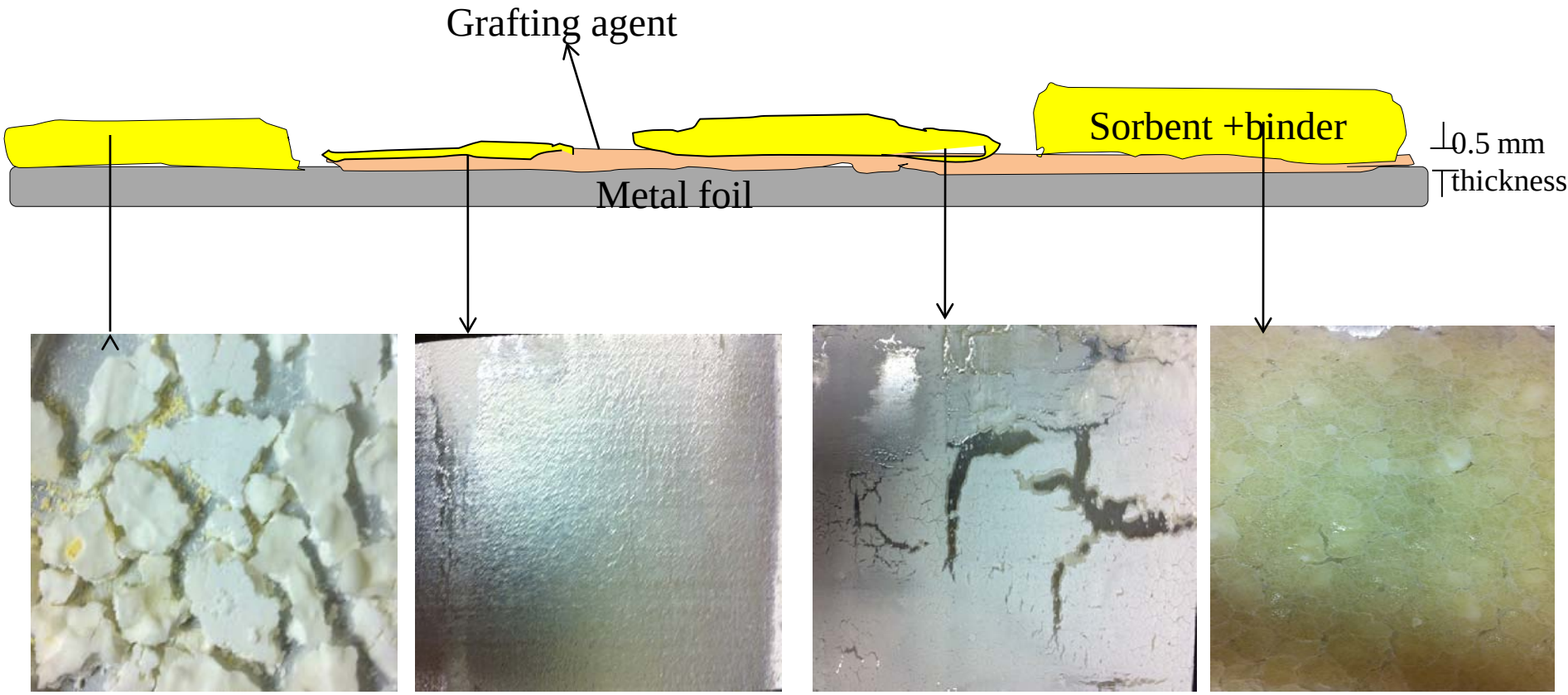
Preparation procedure

TEPA	0.25 g	Sonication 5 min		Sonication 5min		
Grafting agent	0.75 g	→	10-20 drops of water	→		Porous metal monolith
Solvent	1.00 g	60 ^{0}C		Dried in oven at 100 ^{o}C, 10 min		





Metal monolith sorbent



Thickness	2 mm	0.5 mm	1.5 mm	3 mm
CO_2 capture (mmol/g)	1.65	0.32	0.67	1.45

Carbon Dioxide Capture from Flue Gas

#Task	Description	Quarters											
		1	2	3	4	5	6	7	8	9	10	11	12
1	Preparation of amine-grafted zeolite												
2	Determination of CO_2 capture capacity and long term stability												
3	Determination of SO_2 capture capacity and long term stability												
4	Fine-tuning of zeolite composition												
5	Fabrication of metal foil structure and CO_2 absorber												
6	Performance test of CO_2 system												
7	Model development economic analysis												

Summary

- Fundamental research
  - Identification of the species leading to thermal degradation of the sorbent: carboxylate species (R-COO^-).
  - Enhancement of CO_2 capture in the presence of H_2O vapor.
  - Future work:
    - Mechanisms of degradation in steam environment, SO_2, and NO_2.
- Development
  - Successful fabrication of the pellet and metal monolith sorbent in the laboratory scale.
  - Determination of the rate-determining step for the sorbent preparation: drying of the sorbents.
  - Development of rapid drying approaches: rotary dryier and fluidized bed.

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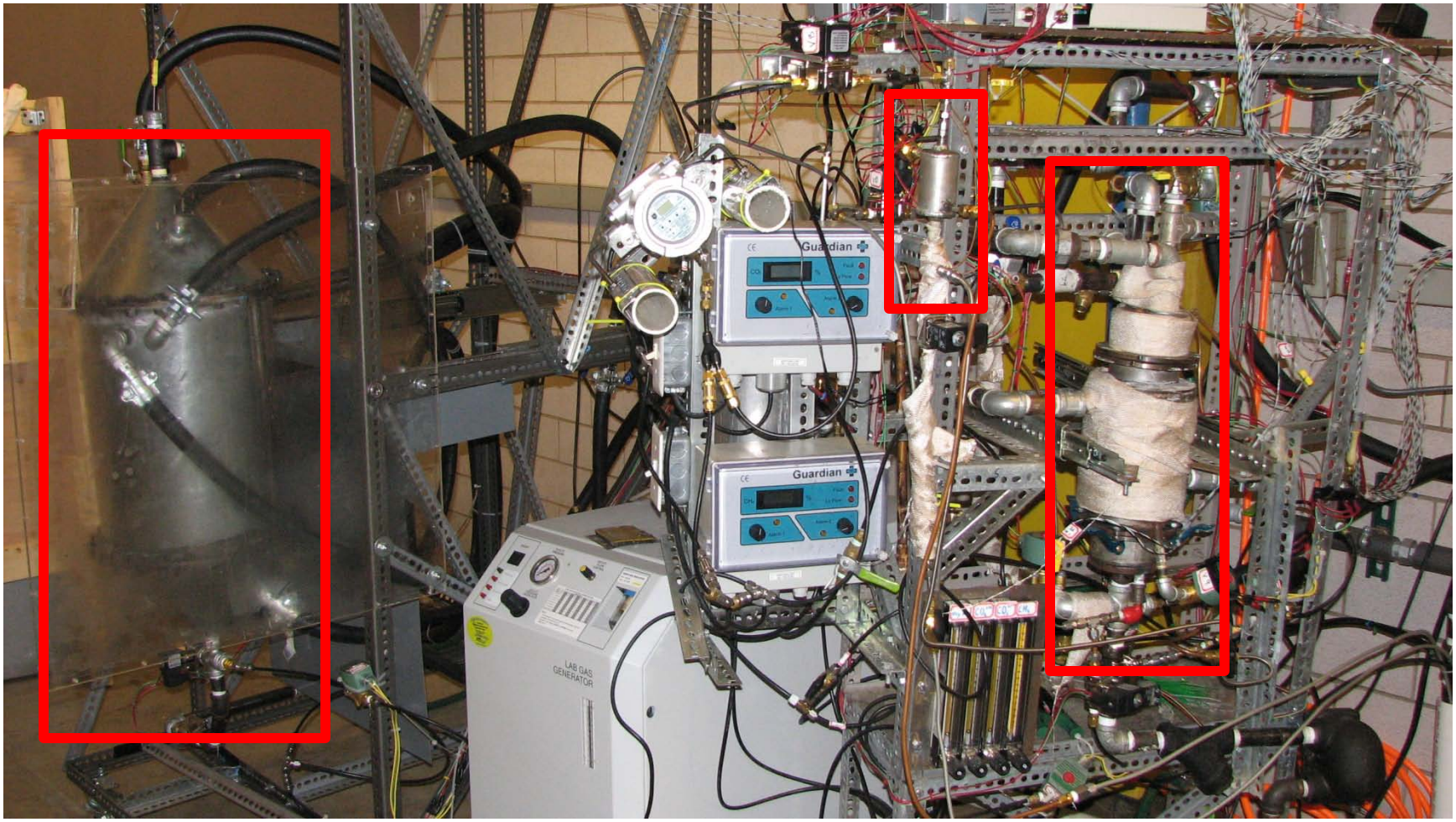
Plan for future testing/development/commercialization

- Testing:
  - Fine-tuning the composition of the amines on the oxide surface.
  - Characterization: elemental analysis, benzene adsorption/desorption.
  - Completion of testing of a 8 kW unit.
- Preliminary economic analysis of the 2 ton/day sorbent manufacturing process and a 0.5 MW CO_2 capture unit.
- Commercialization considerations after successful demonstration of a 10 kg scale unit.

25 Kg (8 kW) Adsorber Unit



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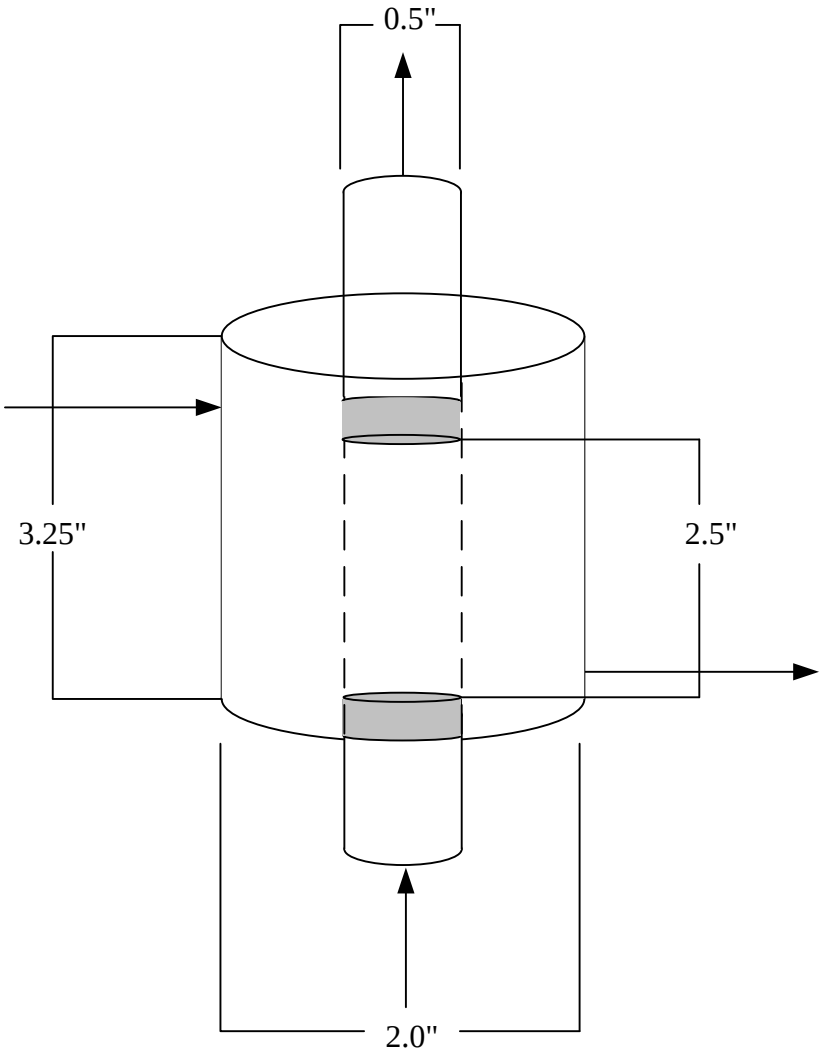

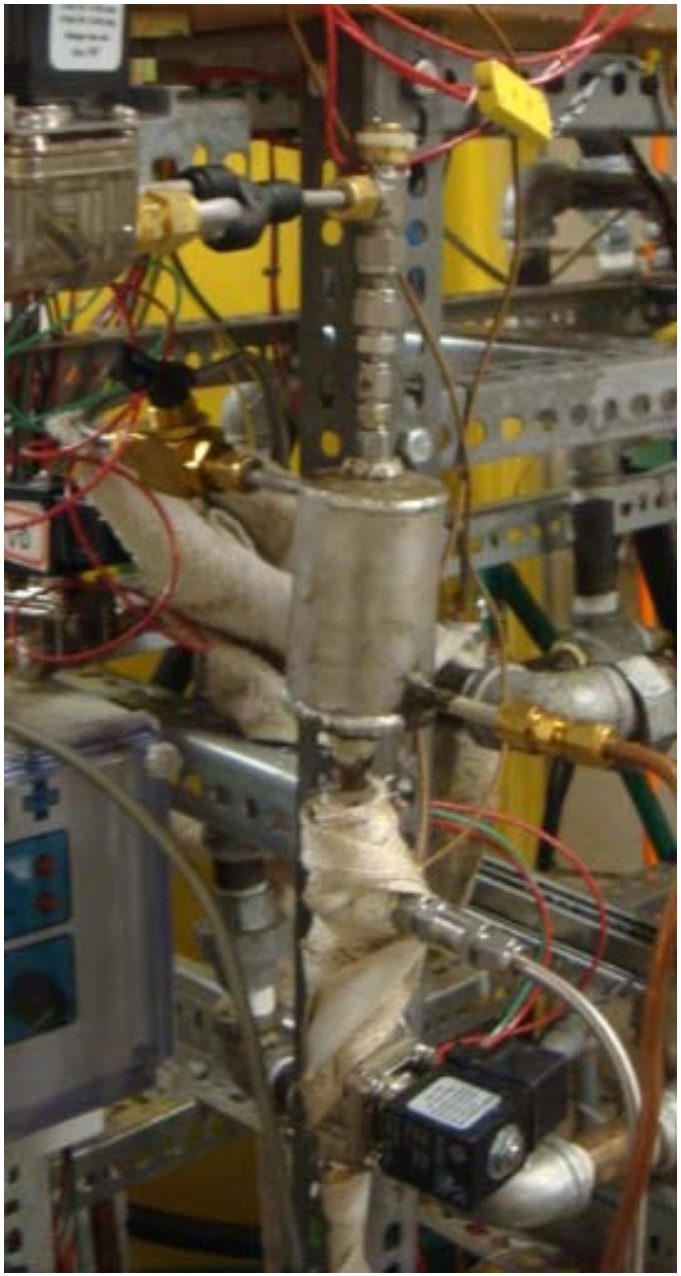

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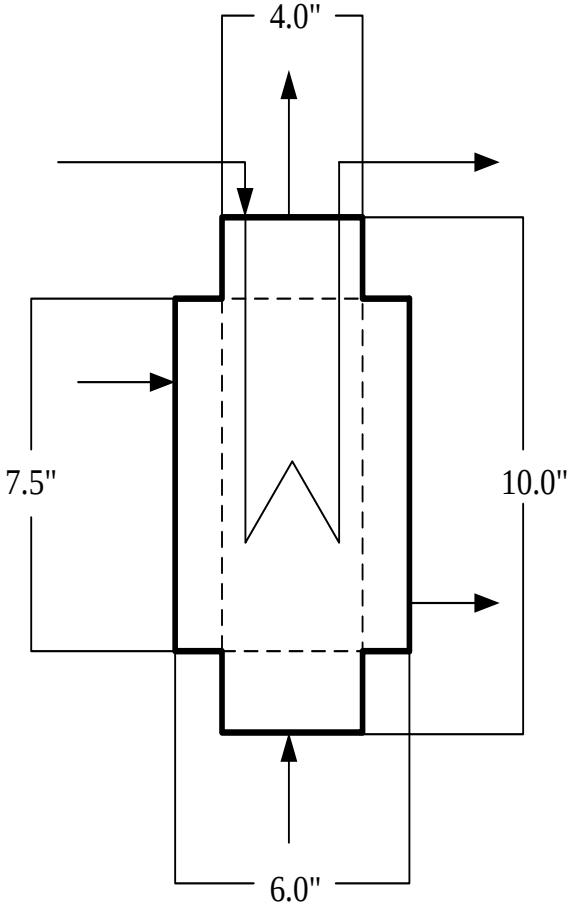

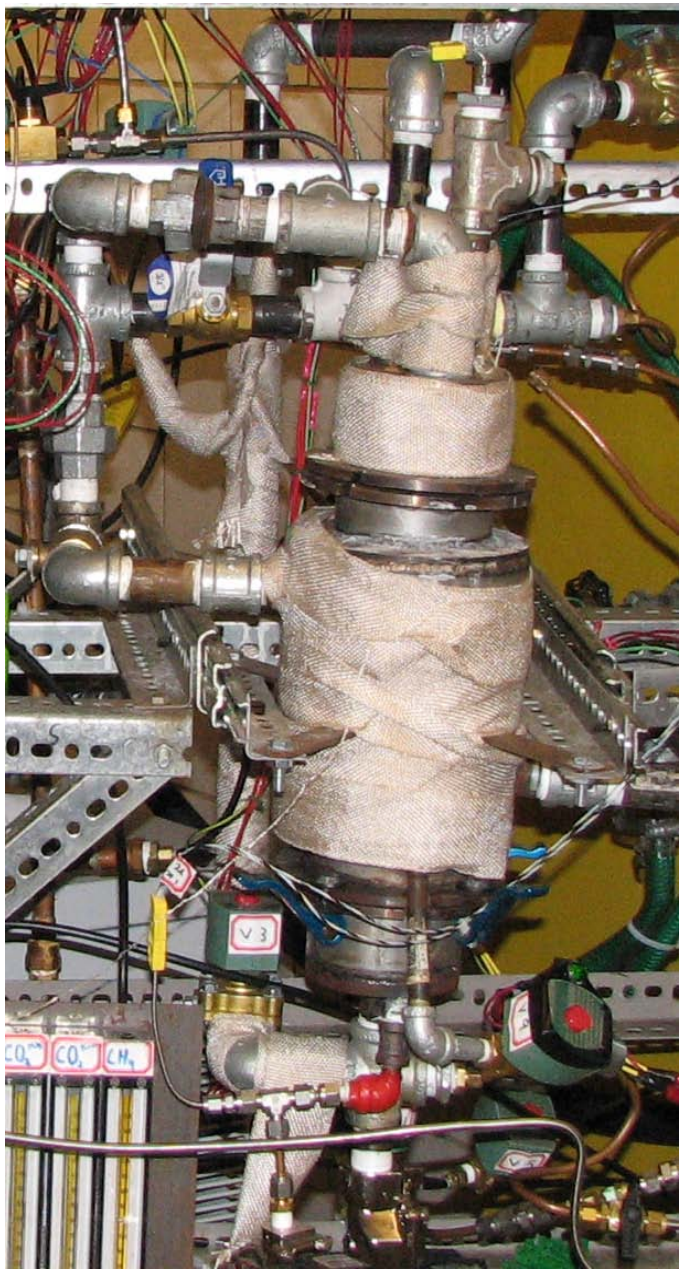

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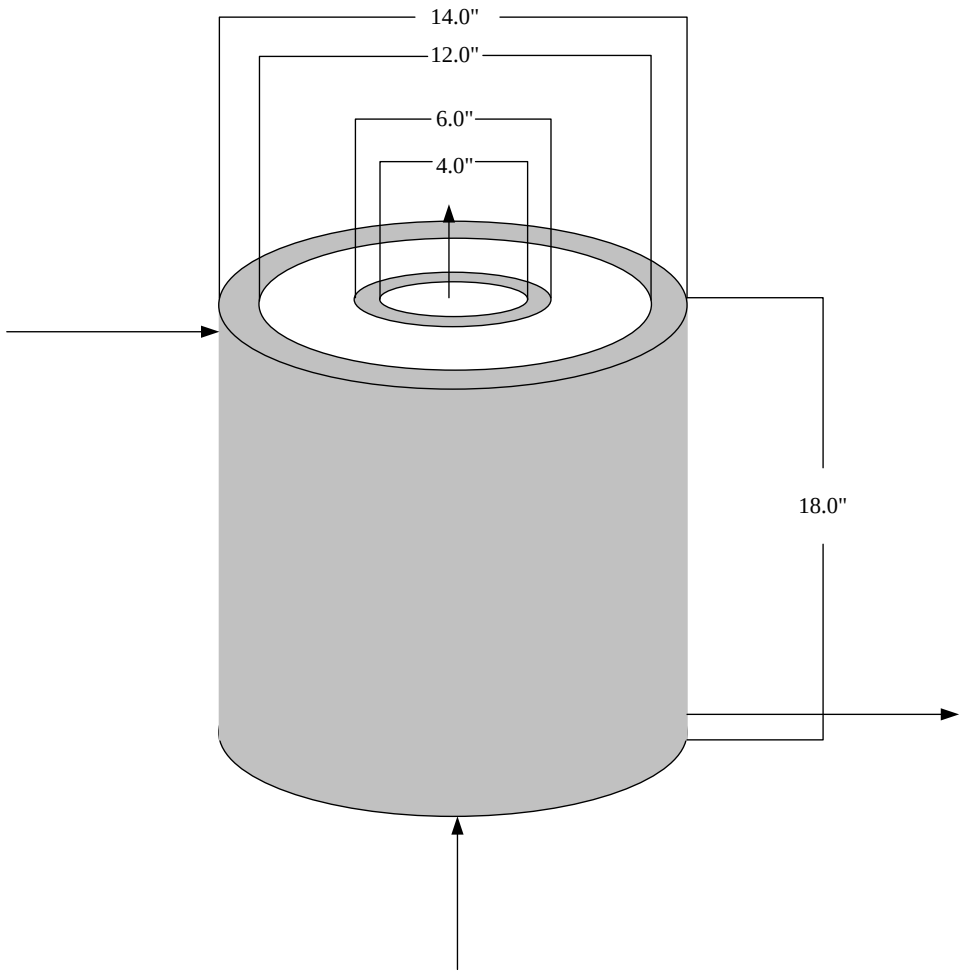

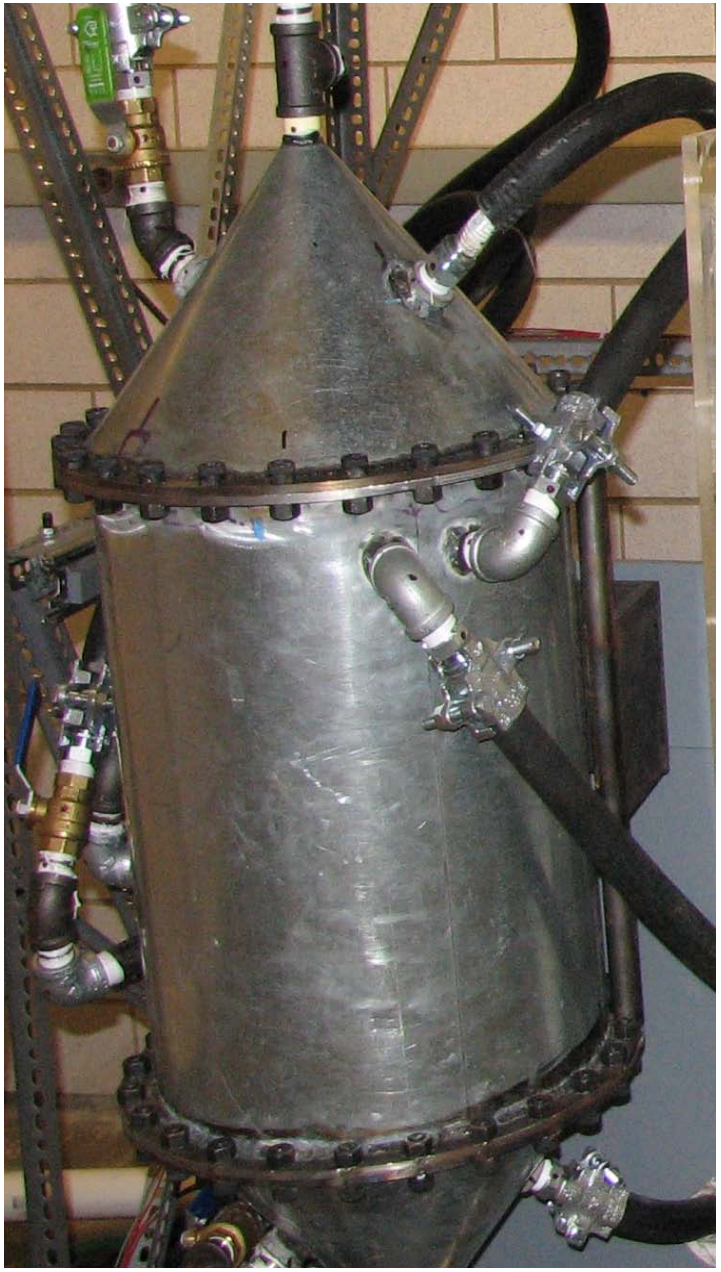

FirstEnergy Advanced Energy Research Center

First Energy Advance Research Center,
Steven Chuang PhD ’85 University of Pittsburgh
Mary Ann Breslin - Secretary

- CO_2 Capture
  - Lab Scale Adsorbent Testing
  - Scale up Adsorbent Testing
  - Automation/Control
  - Mechanic Fabrication
- Fuel Cell
  - Fuel Cell Fabrication
    - Tape Casting
    - Lamination
    - Screen Pringting
  - Fuel Cell Testing
  - Mechanic Fabrication
- SO_2 Capture and $DeNO_x$ Research

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Uma Tumuluri, 2nd year PhD (Chem E) Student

Support/Amine	ads./des. temp. (oC)	CO2 inlet (%)	dry/H2O inlet (%)	Quant. Method	CO2 (mmol/g-sorb.)	Ads. CO2 ratio (wet/dry)	no. of runs	Degradation (%)	ref
zeolite/TEPA	30/70	15	0/6	CO2 analyzer	2.56/4.32	1.69	20	15	[1]
Glass fiber/PEI	30/120	16	0/4	gas chromatogram	2.26/3.98	1.76	2	7	[2]
MCM-41/TRI	25/100	5	0/2	TGA	1.01/1.01	1.00	1	-	[3]
PE-MCM-41/TRI	25/100	5	0/2	TGA	1.01/1.01	1.00	1	-	[3]
MCM-48/APS	25/75	5	0/presented	TGA	1.14/2.28	2.00	2	0	[4]
MCM-41/MONO	105/105	100	0/4	TGA	1.14/0.95	0.83	40	40	[5]
MCM-41/TRI	50/120	100	0/4	TGA	2.39/2.55	1.07	40	2	[5]
MCM-41/PEI	105/105	100	0/4	TGA	1.80/1.82	1.01	20	35	[5]
SiO2/APS	25/50	100	0/-	-	-	-	1	-	[6]
AC/Na2CO3	60/150	1	0/1.3	TCD	2.39/-	-	1	yes	[7]
FA/MEA	30/30	100	-/1	weighing	-/1.56	-	1	-	[8]
MgO/K2CO3	50/350	1	-/11	TCD	-/2.73	-	5	90	[9]
SBA-15/PEI	25/25	100	0/2	IR	-	-	-	-	[10]
SBA-15/EDA	25/150	10	-/2	TGA	-/1.95	-	10	0	[11]
AC/PEI	25/25	N/A	N/A	weighing	-	-	3	10	[12]
SBA-15/TEPA	55/100	N/A	N/A	TGA	3.93/-	-	7	8	[13]
SBA-15/APS	25/120	4	4	IR/MS	0.25/0.41	1.84	3	0	[14]
FA/PEI	75/100	100	0/-	TGA	0.91/-	-	1	-	[15]
-/TEDAM	40/70	20	0/-	CO2 analyzer	2.59/-	-	1	-	[16]

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