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Achieving Climate Goals by Capturing Atmospheric Carbon and Storing it Safely

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Carbon Dioxide and Global Climate Change

Over the last 300 years there has been an increase of 120 ppm of atmospheric CO₂

10 gigatones of atmospheric CO₂ must be removed annually by 2050 to stay below a temperature rise of 1.5 °C

Negative emission technologies focus on taking CO₂ out of the atmosphere and putting it into geological reservoirs or terrestrial ecosystems

Less disruptive, less expensive
Question

Where is the absorption profile of CO\textsubscript{2} gas at a maximum?

a) 3000 cm\textsuperscript{-1}

b) 1000 cm\textsuperscript{-1}

c) 1700 cm\textsuperscript{-1}

d) 2300 cm\textsuperscript{-1}
Adsorption of CO$_2$: Porous Materials

Adsorption of CO$_2$ onto porous structure: zeolites, metal–organic frameworks (MOFs), mesoporous silica, clay, porous carbons, porous organic polymers (POPs), etc.

Properties to Consider:
• Capacity under adsorption conditions
• Selectivity
• Regenerability

RSC Advances, 2021, 11, 12658-12681

Adsorption of CO$_2$: Mixed Matrix Membranes (MMMs)

Combine the mechanical properties and processability of polymers with permeability and selectivity of the filler

Core-shell MOF-ZIF blended with polysulfone
Tailored composition showed higher CO$_2$ permeability and better selectivity

Poly(ether-block-amide) with silver nanoparticles and ionic liquid (IL); ternary blend increased permeability and selectivity

Nano Lett, 2017, 17, 6752

ACS Appl. Mater. Interfaces, 2017, 9, 10094
**CO₂ Absorbing Liquids: Amines**

**Aqueous Amines (30 wt%)**
Energy intensive process, need to recover solvent vapor, corrosive

![Aqueous Amines](image)

Applied Energy, 2017, 185, 1433-1449

**Nanoparticle Organic Hybrid Materials (NOHMs)**
Integrate amine or PEG functionalities in polymer canopy of nanoparticles

![Nanoparticle Organic Hybrid Materials](image)


**CO₂ Absorbing Ionic Liquids**

**Ionic Liquids**
Water lean solvent with chemical flexibility allows tuning properties

![Ionic Liquids](image)

J. Phys. Chem. Lett. 2010, 1, 3459-3464


Green Chem., 2010, 12, 2019-2023

ACS Sustainable Chem. Eng. 2021, 1090-1098
What is the price of the Ionic Liquids [Bmim][PF6] when buying 5g. in USD?

<table>
<thead>
<tr>
<th>Weight (g)</th>
<th>Price (USD)</th>
</tr>
</thead>
<tbody>
<tr>
<td>50</td>
<td>$278.00</td>
</tr>
<tr>
<td>250</td>
<td>$929.00</td>
</tr>
</tbody>
</table>

- $0.50/g
- $5/g

Encapsulation of CO₂ Sorbent Liquids

Encapsulation addresses issues with high viscosities or changes in physical properties of liquids. Can greatly increase surface area and CO₂ absorption rate provided shell is permeable (selective).
**Encapsulation of CO$_2$ Sorbent Liquids**

**Encapsulated Ionic Liquids (ENILs)**
Impregnate carbon with ionic liquid in acetone

![Diagram of IL solvent and ENIL material](image)

**Growth of polymer by CVD around droplets of IL with PTFE particles**

Chem. Commun. 2012, 48, 10046-10048

Langmuir, 2012, 28, 10276

**Encapsulation of CO$_2$ Sorbent Liquids**

Microfluidic device used to encapsulate ionic liquids or CO$_2$-binding organic liquids (CO$_2$-BOLs)

![Diagram of microfluidic device and photopolymerization](image)

Faraday Discuss. 2016, 192, 271-281

**Encapsulation of NOHMs**

Add NOHMs to a UV-curable resin, emulsify, and cure.

**Solvent Impregnated Polymer Films (SIPs)**

Accelerated CO$_2$ sorption kinetics due to increased interfacial area and enhanced oxidative thermal stability.

Electrospinning NOHMs with polyimides gives coaxial fibers.


---

**Soft Template Approach to Encapsulating ILs**

Graphene oxide (GO) nanosheets stabilize droplets of IL in water.

Alkylated graphene oxide (GO) nanosheets stabilize droplets of IL in oil.

Langmuir, 2018, 34, 10114.
Encapsulation of IL by Interfacial Polymerization

GO as particle surfactant provides access to IL-in-water (or oil) emulsion

Interfacial polymerization produces capsules with core of IL and shell of polymer/GO

Powders of IL Capsules

Powders of distinct, individual capsules are isolated by gravity filtration

IL remains inside and powders can easily be transferred without leakage

Cutting capsule reveals hollow core
IL-Filled Capsules with Shell of Polymer/GO

Compression of capsules with top glass slide or Omniprobe in SEM leads to leakage of the encapsulated IL.

Composition of IL Capsules

- **FTIR**: presence of IL and polyurea
- **Raman**: presence of GO
- **DSC**: thermal transitions of IL core
- **TGA**: slight decrease in thermal stability of IL

Extraction with acetone-\textsubscript{d}_6 and internal standard shows particles are 80 wt% IL.
Application of Encapsulated IL: CO$_2$ Uptake

Increased surface area can overcome kinetic limitations of CO$_2$ uptake.

Compare unagitated capsules to stirred IL.

Contribution of IL to CO$_2$ uptake is consisted with agitated IL and superior to still IL.

Encapsulated IL requires less time to reach equilibrium compared to agitated IL.


Capsules of Task Specific IL

Emulsion platform at alkylated GO used to encapsulate ILs in which the anions reversible react with CO$_2$.

Capsules are:
- Stable to multiple CO$_2$ uptake-release cycles
- Outperform zeolites at low pressures
- Stable under humid conditions

Lee, Edgehouse, Pentzer, Gurkan, ACS Appl. Mat. & Int., 2020, 19184.
Fusing Capsule Shells

Unlike ureas prepared from primary amines, when a secondary amine is used urea is reversible.

Dynamic covalent bonds enable reactive shells.

Hindered urea bonds enable fusion of capsules into monoliths or destruction of capsule shells.


Current Group Members:

PhD Students: Maria Escamilla, Katelynn Edgehouse, Sarah Lak, Ciera Cipriani, Yifei Wang, Randi Pulukkody, Huaixuan Cao, Cameron Taylor, Evan van Pelt, Krista Schoonover, Evan Fox, Nicholas Starvaggi, Gianni Spencer, Chia-Min Hseiu

MS Student: Greeshma Chathamkandath

Post Docs: Dr. Peiran Wei, Dr. Niradha Sachinethani

Undergrads: Kortney Tooker, Jordan Price, Joseph Duran, Ethan Hammond

Collaborators: Burcu Gurkan (CWRU), Alissa Park (Columbia), Michelle Kidder (ORNL), Rachel Getman (Clemson), Micah Green (TAMU), Jodie Lutkenhaus (TAMU), Mark Shiflett (Kansas), Ed Maginn (Notre Dame), Patrick Shamberger (TAMU), Dave Bergbreiter (TAMU), Alp Sehirlioglu (CWRU), Stuart Thickett (Tasmania)
 ✓ **Introduction into the CO₂ problem**

- Geological storage of CO₂
- Sealing systems
  1. Portland-based API Class well cements
  2. Calcium aluminate phosphate cement
  3. Epoxy resins & mechanical barriers
- **Summary and conclusion**

**CO₂ and Greenhouse Gas Effect**

- **Atmospheric CO₂ content**
  - Pre-industrial: 278 ppm
  - 50% increase: 417 ppm

- **Major contributors for CO₂ emission**
  - Coal
  - Oil
  - Gas
  - Cement

Cement represents ~ 8% of global CO₂ emission

The Industrial Revolution has triggered global warming
Greenhouse Gase Effect of CO₂

1895 Arrhenius for the first time describes the greenhouse effect of CO₂ gas

1903 Arrhenius received nobel prize in physical chemistry

CO₂ Released into Atmosphere - 2020

Total anthropogenic emission: ~ 41 billion tons/yr

- CO₂ adsorbed by biosphere: ~ 13 billion tons/yr
- CO₂ adsorbed by oceans: ~ 10 billion tons/yr

Total CO₂ remaining in atmosphere: ~ 18 billion tons/yr

Greenhouse gases and their Global Warming Potential (GWP):

<table>
<thead>
<tr>
<th>Gas</th>
<th>GWP</th>
</tr>
</thead>
<tbody>
<tr>
<td>CO₂</td>
<td>1</td>
</tr>
<tr>
<td>CH₄</td>
<td>21</td>
</tr>
<tr>
<td>H₂O</td>
<td>310</td>
</tr>
<tr>
<td>N₂O</td>
<td>265</td>
</tr>
<tr>
<td>SF₆</td>
<td>22,000</td>
</tr>
</tbody>
</table>

Audience Survey Question
ANSWER THE QUESTION ON BLUE SCREEN IN ONE MOMENT

Which sector contributes most to CO₂ emission?

- Traffic & Transportation
- Industry
- Buildings & Construction
- Agriculture
- Other (Lets us know more in the chat!)

Sources of Greenhouse Gas Emissions - Europe

<table>
<thead>
<tr>
<th>Sector</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Construction, heating &amp; cooling of buildings</td>
<td>40 %</td>
</tr>
<tr>
<td>Industry</td>
<td>25 %</td>
</tr>
<tr>
<td>Agriculture</td>
<td>17 %</td>
</tr>
<tr>
<td>Transportation &amp; traffic</td>
<td>15 %</td>
</tr>
<tr>
<td>Miscellaneous</td>
<td>13 %</td>
</tr>
</tbody>
</table>
Global CO₂ Emission - 2019

<table>
<thead>
<tr>
<th>Country</th>
<th>CO₂ Emission</th>
</tr>
</thead>
<tbody>
<tr>
<td>China</td>
<td>27.9%</td>
</tr>
<tr>
<td>Japan</td>
<td>3.0%</td>
</tr>
<tr>
<td>Saudi-Arabia</td>
<td>1.6%</td>
</tr>
<tr>
<td>USA</td>
<td>14.5%</td>
</tr>
<tr>
<td>Iran</td>
<td>2.1%</td>
</tr>
<tr>
<td>Canada</td>
<td>1.6%</td>
</tr>
<tr>
<td>EU</td>
<td>10.0%</td>
</tr>
<tr>
<td>Germany</td>
<td>1.9%</td>
</tr>
<tr>
<td>South Africa</td>
<td>1.3%</td>
</tr>
<tr>
<td>India</td>
<td>7.2%</td>
</tr>
<tr>
<td>Indonesia</td>
<td>1.7%</td>
</tr>
<tr>
<td>Brasil</td>
<td>1.3%</td>
</tr>
<tr>
<td>Russia</td>
<td>4.6%</td>
</tr>
<tr>
<td>S. Korea</td>
<td>1.7%</td>
</tr>
<tr>
<td>Mexico</td>
<td>1.2%</td>
</tr>
</tbody>
</table>

https://de.statista.com/statistik/daten/studie/179260/umfrage/die-zehn-groessten-c02-emittenten-weltweit/

Main CO₂ Emission Sources in China:
- Coal power plants
- Steel and cement plants (less)

US Strategy:
- Replace coal with natural gas
- Increase use of solar and wind energy

EU Strategy:
- Wind, solar, geothermal energy
- Germany: shut down coal power plants by 2038

✓ Introduction into the CO₂ problem
✓ Geological storage of CO₂

• Sealing systems
  1. Portland-based API Class well cements
  2. Calcium aluminate phosphate cement
  3. Epoxy resins & mechanical barriers

• Summary and conclusion

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**CCS, CCUS and CCU**

- **CCS**: Carbon capture and storage of CO₂
- **CCUS**: Carbon capture and underground storage
- **CCU**: Carbon capture and utilization

Source: https://www.nature.org/en-us/what-we-do/our-insights/perspectives/carbon-capture-utilization-storage-albritton/

---

**CCS – Carbon Capture & Storage**

**Geological storage of CO₂** in depleted oil and gas reservoirs (first proposed by C. Marchetti in 1977*)

Why we need supercritical CO₂


* C. Marchetti “On geoengineering and the CO₂ problem”, *Climate change*, 1 (1977) 59 - 68.
**Carbon Capture and (Underground) Storage (CCS)**

- Capture of CO$_2$ with amines from the exhaust gas stream
- Compression of CO$_2$ to supercritical fluid
- Transport and injection into underground formations (e.g. depleted oil fields, saline aquifers, unmineable coal beds)

Source: https://www.greentechmedia.com/articles/read/how-to-make-money-in-carbon-capture#disqus_thread

**Global CCS Projects**

- Currently ~ 200 CCS projects in operation
- 40+ CCS projects under construction
- CO$_2$ captured from gas stream of coal power plants, cement & steel industry
- Most active countries: Norway, U.K., Canada
- Major oil companies involved: Shell, BP, Equinor

**Norway:**
- Re-injects 20 million tons CO$_2$/yr
- About 2/3 of its total CO$_2$ emission

Source: https://www.visitnorway.de/listings/northern-lights-tour-at-the-cable-car/208036/
Transport of CO₂ to CCS Wells

- Using existing pipelines
- Via railway or ship (LNG)
- **Safety aspects:**
  - leakages or accidents

CO₂ Leakage from CCS Wells

- CO₂ can penetrate cement and potentially destroy it
- Migration to the surface poses a safety risk for population
- Example: CO₂ eruption from lake Nyos, Cameroon killed 1,760 people in 1986

Source: J. Plank et al., Resistance of cementing systems under the conditions of permanent geological storage of CO₂ (CCS technology), ZKG 2013, 5, 28-35
✓ Introduction into the CO₂ problem
✓ Geological storage of CO₂
✓ Sealing systems

1. Portland-based API Class well cements
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Main Constituents of OPC and Its Hydrates

<table>
<thead>
<tr>
<th>Main Constituents</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>C₃S Ca₂O(SiO₄)</td>
<td>~ 55%</td>
</tr>
<tr>
<td>C₂S Ca₂(SiO₄)</td>
<td>~ 22%</td>
</tr>
<tr>
<td>C₄AF Ca₄Al₂Fe₂O₁₀</td>
<td>~ 20%</td>
</tr>
<tr>
<td>C₃A Ca₆(Al₂O₃)</td>
<td>~ 3%</td>
</tr>
</tbody>
</table>

Main Hydration Reactions:

- C₃A + H₂O + CaSO₄ → [Ca₃Al(OH)₁₆]₂ · (SO₄)₃ · 26 H₂O (ettringite)
- C₃S, C₂S + H₂O → C·S·H + Ca(OH)₂ (Portlandite)
Carbonation Reactions of Portland Cement

Reactions of cement hydrates with $H_2CO_3$ (wet $CO_2$):

$$\text{Ca(OH)}_2 + H_2CO_3 \rightarrow \text{CaCO}_3 (s) \ + \ 2 \ H_2O$$

$$\text{C-S-H} + H_2CO_3 \rightarrow \text{CaCO}_3 (s) \ + \ \text{SiO}_2 \cdot n \ H_2O$$

\[\text{silica gel}\]

$$[\text{Ca}_3\text{Al(OH)}_6 \cdot (\text{SO}_4)_3 \cdot 26 \ H_2O + 3 \ CO_2] \rightarrow 3 \ \text{CaCO}_3 \ + \ 3 \ \text{CaSO}_4 \cdot 2 \ H_2O \ + \ 2 \ \text{Al(OH)}_3 \ + \ 27 \ H_2O$$

Formation of **water soluble** calcium hydrogen carbonate:

$$\text{CaCO}_3 + H_2CO_3 \rightarrow \text{Ca(HCO}_3)_2$$

---

Early Studies on $CO_2$ Stability of Cement

References:


Conclusions:

- Portland cement thermodynamically unstable against $CO_2$
- Initial $CaCO_3$ formation densifies cement
- Subsequent leaching of $CaCO_3$ as $Ca(HCO_3)_2$ increases permeability
  
  Carbonation proceeds more rapidly
Lab Study on Modified Portland Cement Samples

1 Month Storage: at 90° C and 400 bar CO₂ pressure no major effect of scCO₂ on the specimens

6 Months Storage:
- System A: rough surface like sandstone
- System B: no effect of scCO₂
- System C and D show severe crack formation (direct pathway for CO₂ leakage!)

Cement Porosity & Crack Formation

According to Fabbri et al., low porosity (w/c ratio) generally leads to increased crack formation

- CaCO₃ crystals need expansion space for their growth

- If expansion space is not available, crystallization pressure will destroy the cementitious matrix
  
  crack formation

Low porosities of cementing systems C and D explain the crack formation after scCO₂ exposure

High porosity (w/c ratio) promotes the leaching of CaCO₃

Field Experience from CO₂ Injection Wells

Natural CO₂ producing well in Dakota sandstone formation (30 years):

• Sample from direct proximity to reservoir (~ 6 m) showed almost complete conversion of portlandite to calcium carbonate

• Increase in porosity and permeability

• Samples recovered at further distance from reservoir (~ 50 m) and at top of the caprock carbonated only slightly

CO₂ injection well in the Permian basin of West Texas (30 years):

• Only minor carbonation of cement sample collected ~ 3.5 m above the reservoir

• Minor increase in porosity and permeability

Contradicting reports from the field!


✓ Introduction into the CO₂ problem
✓ Geological storage of CO₂
✓ Sealing systems

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Prof. Dr. Johann Plank
Calcium Aluminate Phosphate Cement

- Invented by T. Sugama for geothermal wells
- Extremely corrosive environment (H$_2$SO$_4$, H$_2$S, CO$_2$)
- Temperatures up to 320 °C, pH = 2
- Massive corrosion problems

Main Hydration Reactions in CAPC

1. Hydrothermal Reactions:
   \[
   \text{CaHPO}_4 \cdot 2 \text{H}_2\text{O} + \text{Ca}_4\text{O}(\text{PO}_4)_2 \rightarrow \text{Ca}_5(\text{PO}_4)_3(\text{OH}) + 2 \text{H}_2\text{O}
   \]
   hydroxy apatite

2. \[
   5 \text{Ca} + \text{3NaPO}_3 + \text{7H}_2\text{O} \rightarrow \text{Ca}_5(\text{PO}_4)_3\text{OH} + \text{10AlO(OH)} + \text{3NaOH}
   \]
   hydroxy apatite boehmite

3. \[
   5 \text{Ca} + 3 \text{Al}_6\text{O}_9\cdot\text{Si}_2\text{O}_4 + 3 \text{NaPO}_3 + 11.5 \text{H}_2\text{O} \rightarrow 3 \text{NaAlSi}_2\text{O}_6 \cdot \text{H}_2\text{O} + \text{Ca}_5(\text{PO}_4)_3\text{OH} + 16 \text{AlO(OH)}
   \]
   analcim apatite boehmite

4. \[
   5 \text{Ca} + 5 \text{Al}_6\text{O}_9\cdot\text{Si}_2\text{O}_4 + 6 \text{NaPO}_3 + 24 \text{H}_2\text{O} \rightarrow 2 \text{Na}_2\text{Al}_3\text{Si}_6\text{O}_{16} \cdot 6 \text{H}_2\text{O} + 2 \text{Ca}_5(\text{PO}_4)_3\text{OH} + 34 \text{AlO(OH)}
   \]
   NaP zeolite apatite boehmite

no CaCO$_3$ is formed!
Laboratory Testing of CAPC

60 % calcium aluminate cement (CAC)  
40 % ASTM Class F Fly Ash  
20 % sodium polyphosphate solution

- Stored in 4 % Na$_2$CO$_3$ solution  
- Exposure over 1 month  
- Temperature 300 °C

T. Sugama,  
“Advanced Cements for Geothermal Wells”  
Brookhaven National Library, BNL-77901-2007-IR

Results – Carbonate Stability of CAPC

- excellent carbonate resistance  
- no cracks, no deterioration  
- low permeability  
- some strength reduction

Source: K. Agapiou, S. Charpion “Cement and Wellbore Integrity”  
International Cement Review, August 2013, p. 113 - 116

currently by far the best field tested cementing system for CO$_2$ wells
CAPC – Issues and Open Questions

• CAPC causes flash set when in contact with OPC! → use of dedicated equipment

• CAPC requires special additives (retarder, dispersant etc.)

• So far no lab results from storage in scCO₂

• CAPC is not alkaline → corrosion protection of casing?


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Prof. Dr. Johann Plank
Epoxy Resin Cement

- 2 component system: epoxy prepolymer + amine hardener

Chemical reactions occurring in epoxy resin formation:

\[ \text{epoxy prepolymer} \rightarrow \text{polymerization} \]

Epoxy well-known organic binder, extremely resistant against acids and solvents; widespread use in construction

Lab Results – CO₂ Exposure of Epoxy

- Samples stored up to 1 year in brine
- 500 bar CO₂ pressure, 100 °C
  - No visual deterioration
  - Stable weight
  - No change in permeability
  - Decrease of strength

Epoxy resin cement appears to exhibit high CO₂ tolerance
Mechanical Barrier Against CO₂

Expandable packers as mechanical seal for CO₂

- Introduction into the CO₂ problem
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• Summary and conclusion
Cementing Systems Suitable for CCS

Criteria: Borehole integrity over 500 – 1,000 years

At present only those potential candidates:
- Calcium aluminate phosphate cement
- Epoxy resin cement

Open question:
Corrosion protection for casing!


Critical Importance of Mud Displacement For Sealing Off CCS Wells

Use of best practice:
- Centralizers
- Scratchers
- Pipe rotation

Mud displacement by spacer fluid:
Summary and Conclusion

- Safe sealing of CCS presents a challenge for cementing technology
- Portland cement-based systems do not provide century-long resistance against CO₂, modification can improve their stability
- CAPC presents a much more stable alternative, however field application is more complicated
- Organic binder systems and mechanical barriers present an alternative
- Dual containment strategy appears to work best and guarantees maximum safety

Achieving Climate Goals by Capturing Atmospheric Carbon and Storing it Safely

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