Reactive Flow Through Experiments
- A Look at Foamed Cement and CO$_2$
Resistant Cement

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Presentation Outline

• Project Overview
• Background
• Previous Work
• Research Scope
• Results
• Future Work
**Goal**

- The objective of the effort is to investigate and evaluate the fracture opening or self-sealing of foamed cements and CO₂ resistant cements: Flow-through CO₂-saturated brine interactions at subsurface conditions typical in the Gulf of Mexico (GOM).

**Research Questions**

- Will foamed cements with a leak pathway (i.e. fracture) self-seal in a similar manner as ordinary Portland cement?
- When are CO₂-resistant cements needed?
  - Significantly more expensive to use than traditional Portland cements. In addition, they create problems for service companies because they are not compatible with the traditional Portland cements used in other sections of the well.
- When can we use Portland cements and when should we use a specialized cement?
- These answers will improve safety, well integrity, and have significant economic benefits.

**Approach**

- It is unfeasible to run experiments on every single variable that exists in the subsurface. Therefore, the team needs to understand the fundamental mechanisms to make predictions. Flow-through experiments are being conducted on various cement formulations.

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**This project is in its second year**
Previous studies have shown that the self-healing ability of cement is likely a combination of several factors:

- fracture geometry (aperture size and domain length)
- cement type
- time
- fluid flow rate
- fluid composition
- reservoir conditions

Research has shown that Ordinary Portland Cement (OPC) can self-heal under a wide range of conditions.

Huerta et al, 2013, 2015
Previous Research

Unexposed foamed cement

Foamed cement exposed to SCCO$_2$ under static conditions (56 days)

The bubbles in the alteration zone are filled with calcium carbonate crystals.

Illustrates how carbonation alters pore space by precipitation.

Stitched CT Core montage on the XZ direction for neat, 10%, 20% and 30% cores exposed for 6 months. Stitched from approximately 9,000 2D images associated with the full scan of the core.
Current Research Scope

Samples
1. Generate foamed cement using API RP 10 B-4 procedures
   a. Three different foam qualities (10%, 20%, and 30% gas volume)
2. Generate various CO₂-resistant cements
   a. Fly Ash-modified Calcium Aluminate Phosphate Cements (Na₂O-CaO- Al₂O₃-SiO₂-P₂O₅-H₂O system)

Experiments
1. Cement cores fractured using the Brazilian method
2. Uniaxial Hasler cells with a confining pressure to create flow through the cement core
   a. Predetermined flow rates for predetermined lengths of time.
   b. In consideration are variable flow and constant flow rates.
   c. Constant flow rate short core experiments
   d. Constant pressure differential composite core experiments
3. CT- flow-through experiments

Analysis
1. Multi-scale computed tomography (CT) scanning*
2. Scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS)
3. ATR-FT-IR (Attenuated Total Reflectance-Fourier Transform Infrared Spectroscopy)
4. Mechanical testing
   a. Porosity
   b. Permeability
   c. Strength measurements
RESULTS
Ordinary Portland Cement

Photos of foamed cement sample (20% foam quality) exposed to variable flow of saturated CO₂ in the medical CT scanner.
CT-Scanner - Reaction Zone Development

24 Hour 72 Hour 192 Hour 264 Hour
CT-Scanner - Reaction Zone Development

Light Reaction

Dark Reaction

Fracture
SEM image of a 20% foam quality foamed cement sample that was exposed to variable flow of saturated CO₂ in the medical CT scanner.

Inlet and moving towards the interior
The 3 polymorphs of calcium carbonate are frequently observed – often within microns of each other.
OPC samples analyzed to establish a proper methodology

Sample A4

Flow direction

5.6 cm,
0.03-0.2 mL/min,
60 mL fluid flowed,
0.18 md to 0.03 md

Most reaction happens within 5 cm
Flow is channelized

Sample A1

Sample A3

10.3 cm,
0.05 mL/min,
50 mL fluid flowed,
0.17 md to 0.10 md
OPC cross section, Sample A1

1 mm at 3 cm
OPC cross section, Sample A4

- Middle area carbonated
- Surface dissolution
- Pore filled with CaCO₃
- 3mm at inlet
Fourier Transform Infrared Spectroscopy

- IR spectroscopy was used to further investigate the SEM analysis that showed that:
  - The bubbles in the alteration zone are filled with calcium carbonate crystals
  - These crystals have varying shapes that are representative of three polymorphs of calcium carbonate
Class H cement shows pressure-dependent polymorphs of calcium carbonate.

CO₂ resistant cement did not show changes in the calcite polymorph structure.

Although, vaterite was seen in the SEM, no characteristic peaks were present in the spectra.
Future Work: CO$_2$-Resistant Cement
Questions?
CT-Scanning Flow-through Experiments

Samples
- 20% Quality samples made in Pittsburgh (Rick Spaulding)
- Fractured using traditional Brazilian technique

Experimental Setup
- Flow Through
  I. Confining pressure = 1200 PSI
  II. Pore pressure = 800 PSI
  III. DI water at equilibrium with CO₂ as injected fluid (room temperature)
  IV. Flow rate of 0.2 ml/min for a period of ~260 hours
- CT Scanning
  I. Resolutions of 17 μm
  II. Scans taken as time permitted (no weekend scans)

ImageJ Processing
- Images were scaled by 50% (reduction to 0.5 in X/Y/Z)
  - Size management critical to processing speed and efficiency
- Images underwent bright outlier removal at 2 pixel radius
- Images were then filtered using 2x2 mean (3-D)
  I. Processes facilitated easier segmentation & feature isolation
Current Research Scope: Fourier Transform Infrared Spectroscopy

– Samples: Class H cement and CO₂-Resistant cement
– Conditions:
  • CO₂-Fluid-Cement Interface
    – Samples prepared in Millipore water to create water film
    – 40°C and scanned at one pressure ranging from 0 to 1200 psig
OPC, cross section, sample A3

~50µm reaction depth at inlet

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CaCO₃
Mg-silicate
More Ca
Al and Fe
unhydrated
Less Ca