

Characterizing Hydrated Blended Cementitious Systems for Performance Assessment

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Outline

- Project Objectives
- Research Program
- Recent Results

Problems/Challenges

- Materials performance depends upon mineral phases present
- Materials performance depends upon pore solution composition
- Anticipate broad range of mixture proportions
- Anticipate future use of low-calcium cement systems
- Need to validate performance prediction tools
- Identify missing knowledge gaps
- Lack of standards for characterizing hydrated phases

Objectives

- Characterize the pore solution of blended systems
- Identify/Quantify hydrated phases
- Develop characterization methods, as needed
- Publish validation data
- Fill knowledge gaps

Research Program

PHASE I:

- Identify representative mixture proportions
- Develop phase identification/quantification techniques

PHASE II:

- Use representative waste solutions
- Pore solution analysis

PHASE III:

- Data after one year of hydration

Phase I

- Identify representative mixtures
- Constant material source and water:cement ratio
- Characterize starting materials
- Identify/Quantify phases after 3 months hydration
 - XRD: crystalline phases
 - TGA: portlandite content

Experimental Details

Mixture Proportions: (mass fractions)

Mix #	Cement	Fly Ash	Slag	Silica Fume
1	1.00			
2	0.70	0.30		
3	0.70		0.30	
4	0.70	0.20		0.10
5	0.50	0.50		
6	0.50		0.50	
7	0.30	0.35	0.35	
8	0.10	0.45	0.45	

Chemical Analyses

	Cement	Fly Ash Class C	GGBFS	Silica Fume
CaO	0.605	0.246	0.371	0.005
SiO ₂	0.191	0.370	0.373	0.962
Al ₂ O ₃	0.050	0.200	0.104	0.003
Fe ₂ O ₃	0.033	0.053	0.005	0.000
MgO	0.041	0.048	0.116	0.001
SO ₃	0.031	0.014	0.022	0.000
Na ₂ O	0.001	0.015	0.002	0.001
K ₂ O	0.007	0.006	0.003	0.005
TiO ₂	0.003	0.016	0.005	0.000
CaCO ₃	0.034			
Sulfide			0.010	

Experimental Details

Program:

- Cast 25 mm x 25 mm cylinders, into sealed bags
- De-mold at 24 hours (48 hours for Mixes 7 & 8)
- Store at 25 C in air-tight LDPE jars
- Cut 1 mm slices on diamond saw w/ ethanol

- TGA: 50 mg sample composed of a few 'chunks'
 - Estimate portlandite content

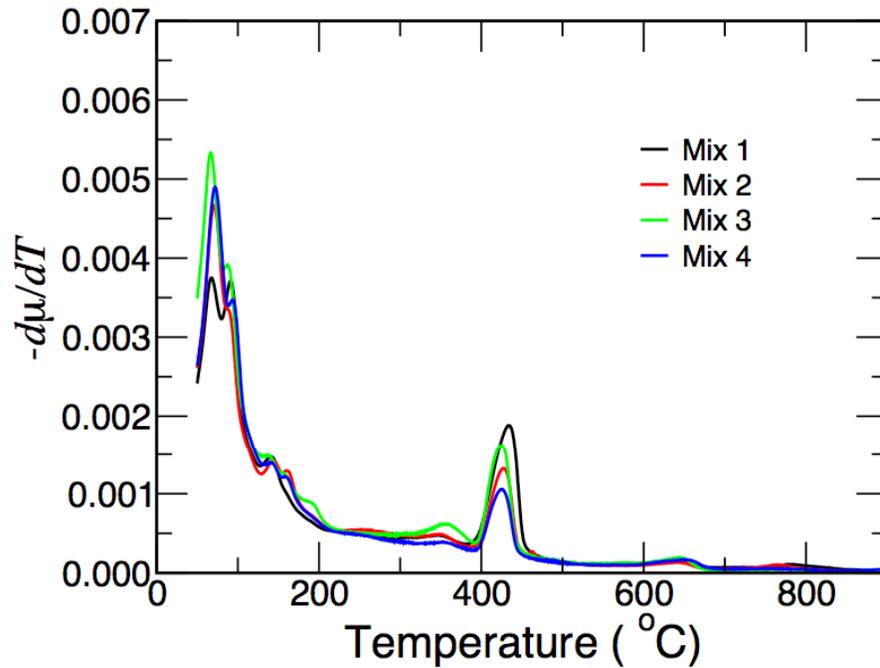
- XRD: briefly polish with 600 grit sandpaper
 - Identify/Quantify hydrated crystalline phases
 - Use TGA data to estimate amorphous content

TGA Experiment

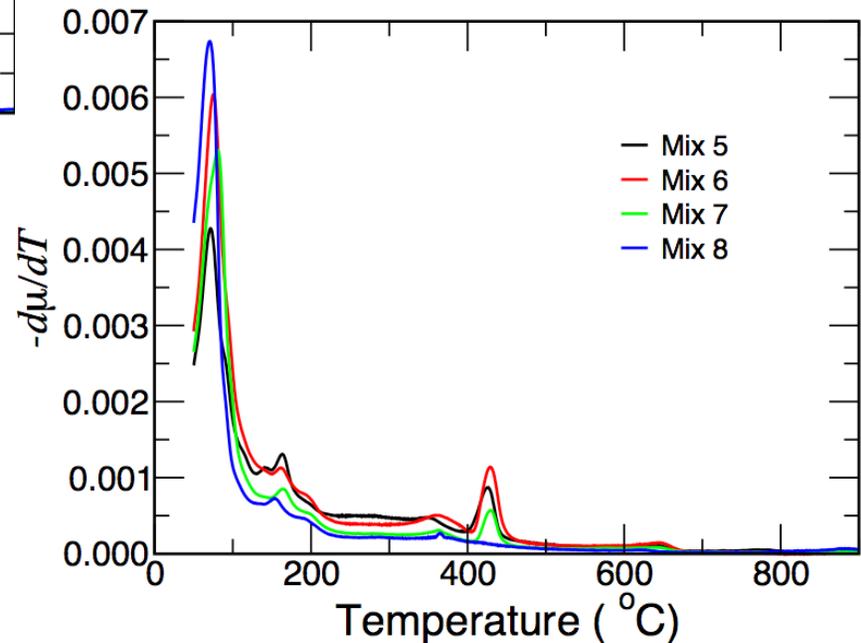
Thermogravimetric Analysis:

- 50 mg samples
- High Purity Nitrogen: 40 mL/min
- Procedure:
 - Equilibrate at 30 C
 - Mass loss: 30 C to 950 C
 - Scan Rate: 5 C / min
 - Mass Fraction wrt initial mass

TGA Data: Portlandite Peak Identification



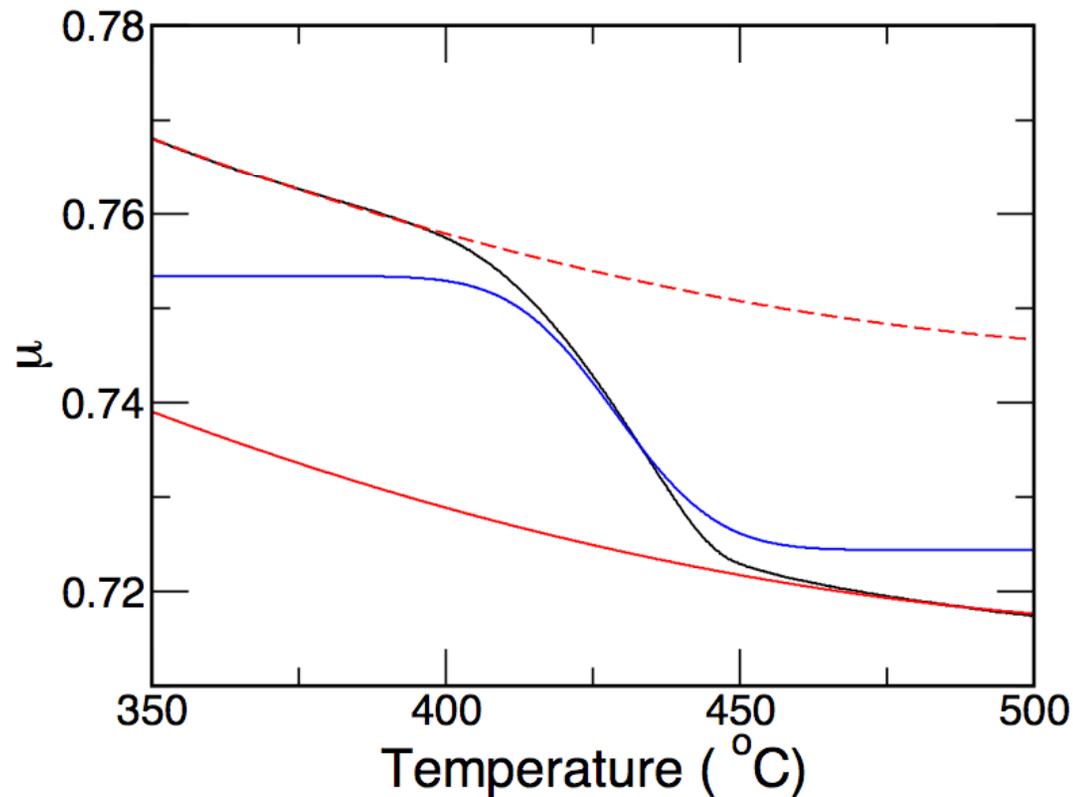
$$\mu = \frac{m(T)}{m_0}$$



TGA Analysis of Portlandite Peak

$$\mu = A_0 + A_1 (T - T_0) + A_2 (T - T_0)^2 + B_0 \operatorname{erfc} [B_1 (T - T_0)]$$

Mix 1



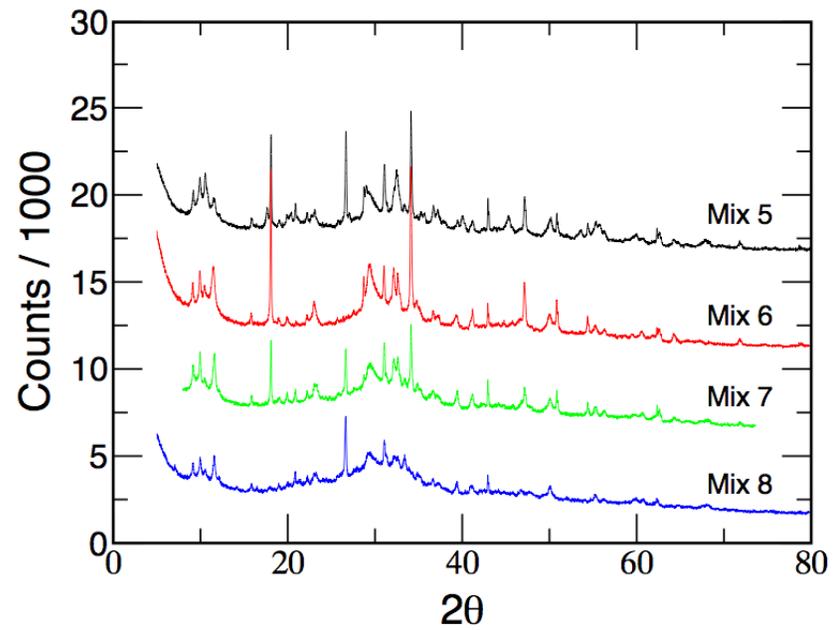
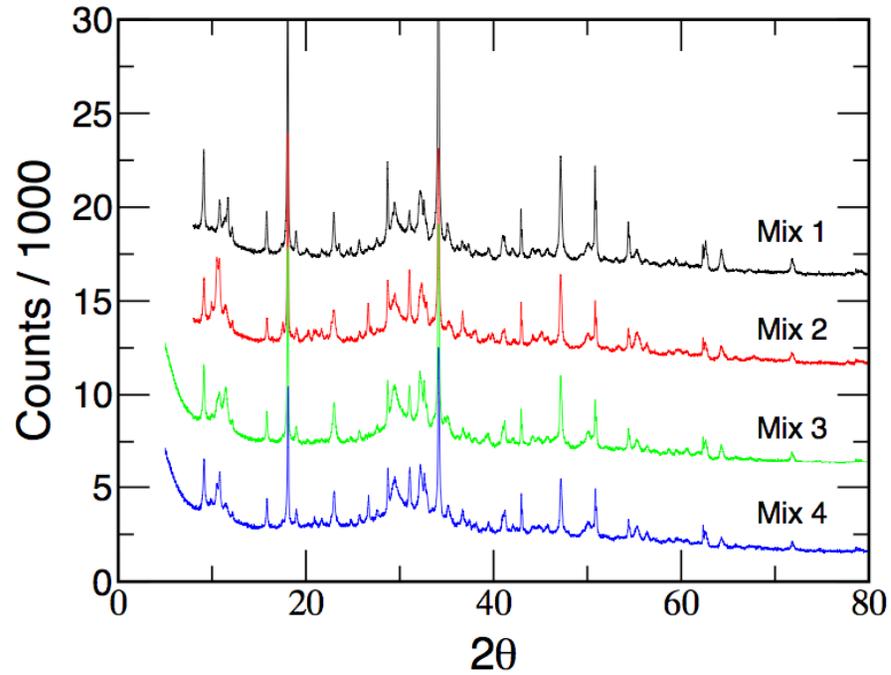
TGA Results

Mix #	Portlandite Mass Fraction
1	0.119
2	0.064
3	0.063
4	0.049
5	0.031
6	0.039
7	0.019
8	N/A

XRD Experiment

- 1 mm disk - no grinding
- Polish with 600 grit sandpaper
- Data range: $8 \leq 2\theta \leq 80$ Step Size: 0.016 degrees
- Rotate sample: increased sampling volume

XRD Data: Phase Identification



XRD Rietveld Analysis

- Structure Data:
 - Cement phases: published data
 - Hydrated Phases:
 - Public databases
 - Hemicarboaluminate: structural data from Taylor
- Refinement: 'Fix Structures'
 - calcium silicate cement phases: raw cement
 - aluminate/ferrite cement phases: SAM extraction
 - no preferred orientation
 - Mix 8: corundum (87% crystalline) spike

Measured vs. Calculated

Similarities:

- Hydrated phases
- Cement phases

Differences:

- Oxides and carbonates: quartz, periclase, calcite

Future Work

- X-ray microanalysis:
 - Multiphase corroboration
 - Volume fractions
- Pore Solution Analysis
 - Alkali binding
 - Rigorous calculation validation
- 'Waste Solution' mix water