Pyrrhotite Oxidation - Insights into Laboratory Testing of Concrete Expansion and Deterioration, and the Acceleration of Reaction Rates

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Outline

- ❑Part 1: Factors Affecting Iron-sulfide Oxidation in Concrete
- ❑Conclusion (Part 1)
- ❑ Part 2: Electrochemical Acceleration Method
- ❑Conclusion (Part 2)

Factors affecting iron-sulfide oxidation in concrete

Table 1: Properties of sulfide bearing aggregates

Aggregate Type	ID	Total Sulfur S_T (%)	Minerology (Petrography)	
			main mineral constituents	Iron-sulfide
Connecticut	CT1	1.06	Quartz &Mica	Pyrrhotite
	CT2	0.77		
Canada	CA1	1.18	Plagioclase feldspar, pyroxene, Mica	Pyrrhotite, Pyrite
	CA2	1.25		
Controlled	R	0.04		
Pyrrhotite Rock	Po	25.5		Pyrrhotite
Pyrite	Py	54.1		Pyrite

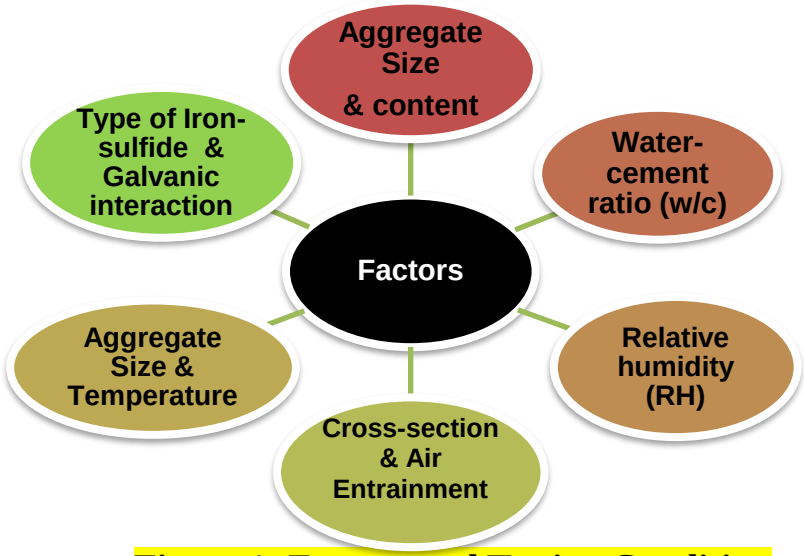
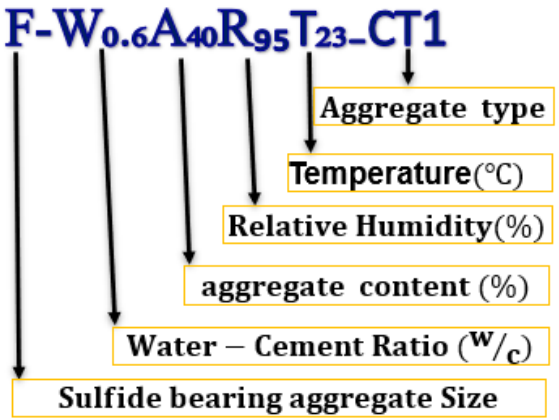


Figure 1: Factors and Testing Condition

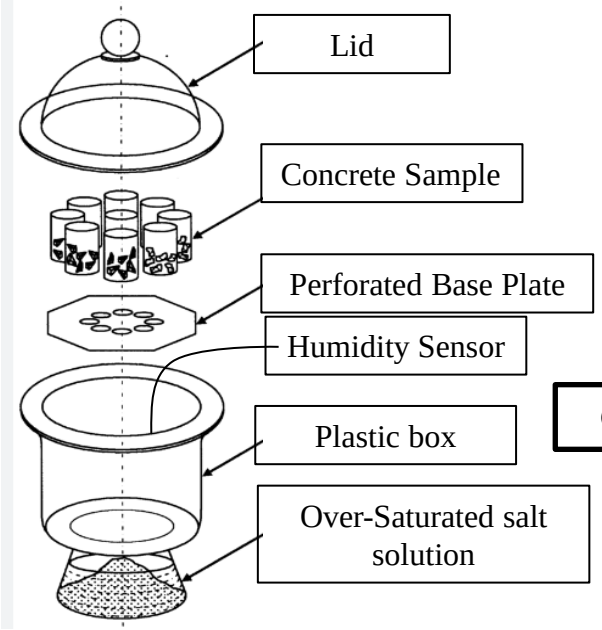
MIX ID:



Goal

- Determining the factors that speed up iron sulfide oxidation
- Understanding and quantifying deterioration

Constant temperature and humidity Setup



Constant Humidity Chamber

Environmental Chamber

Curing room (23°C/95% RH)

Effect of Sulfur Content and water-cement ratio

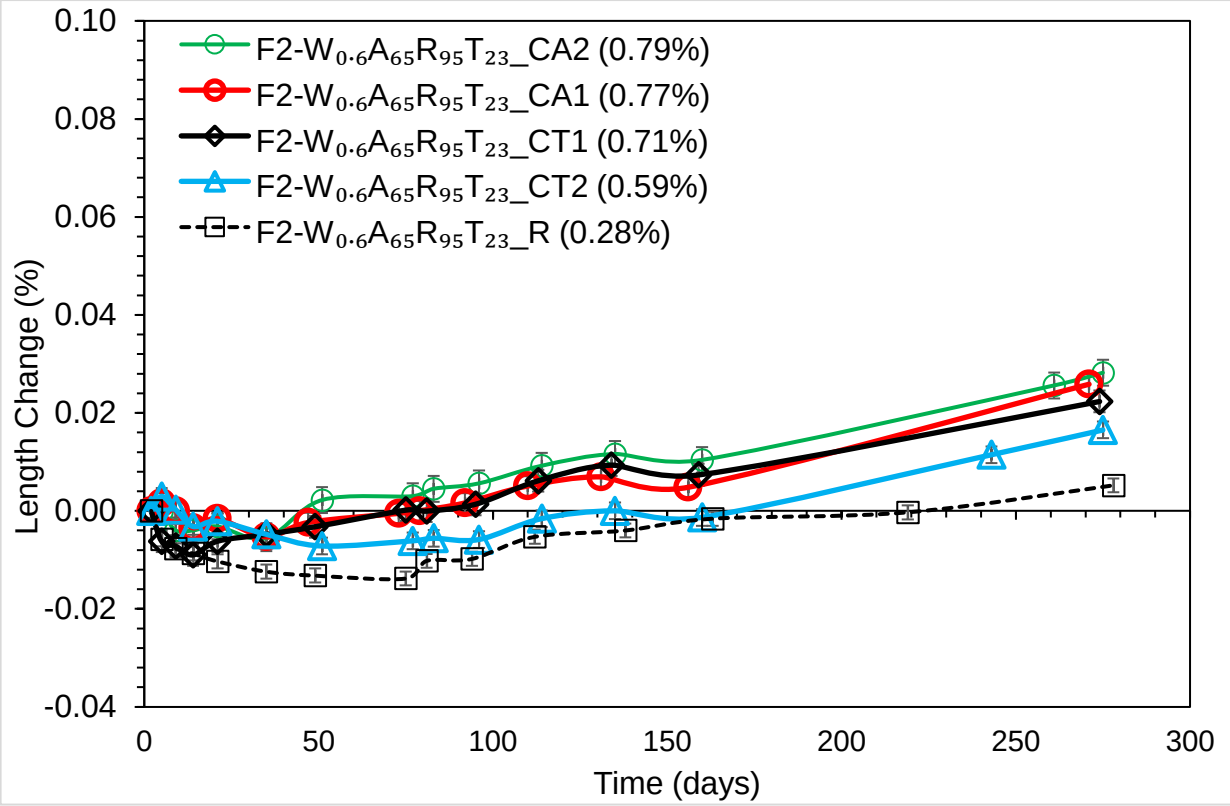


Figure 2: water-cement ratio (w/c) of 0.6

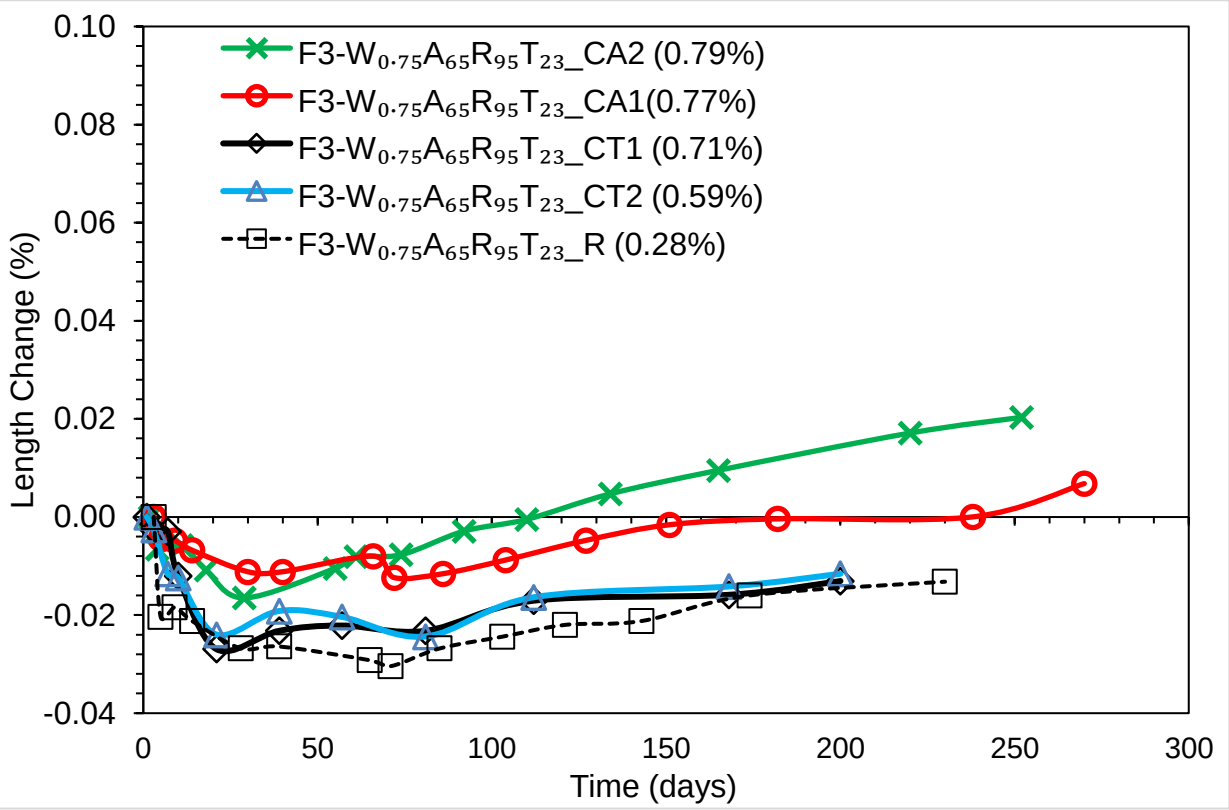


Figure 3: water-cement ratio (w/c) of 0.75

**Each data points corresponds to the AVG of three points*

Effect of Relative Humidity (95 and 100%)

Pyrrhotite rock (Po)

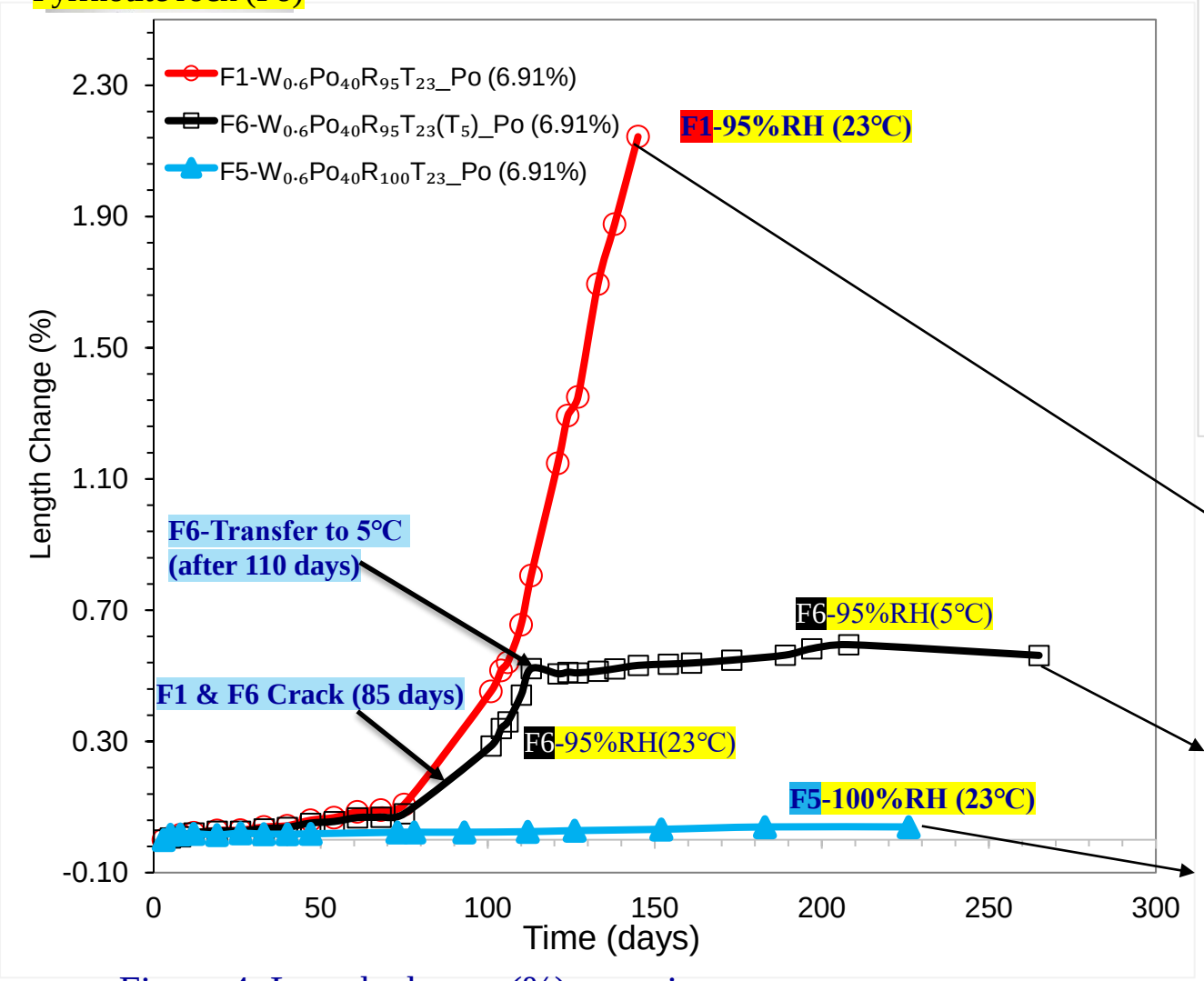


Figure 4: Length change (%) over time

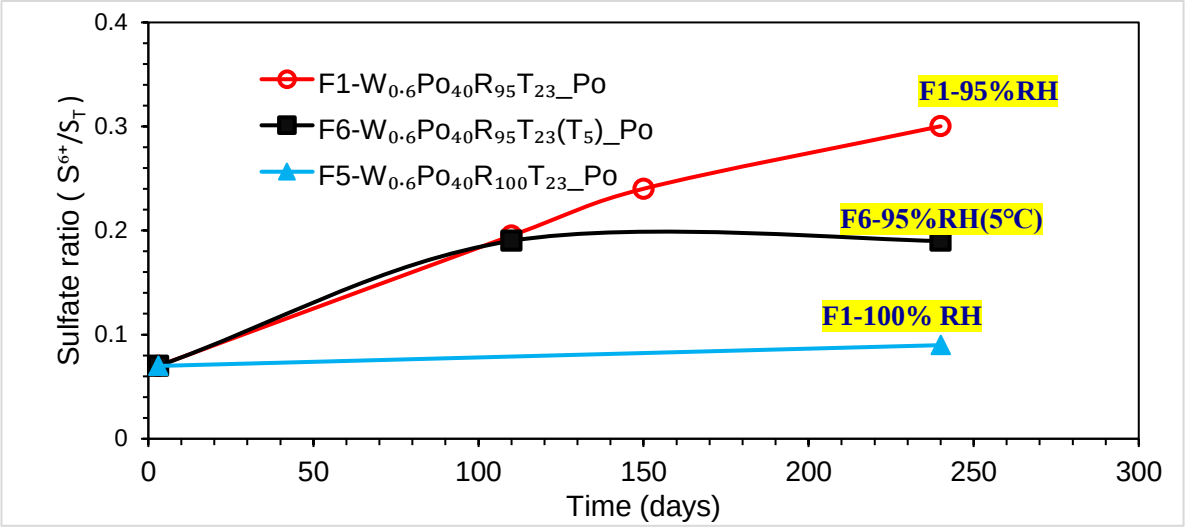
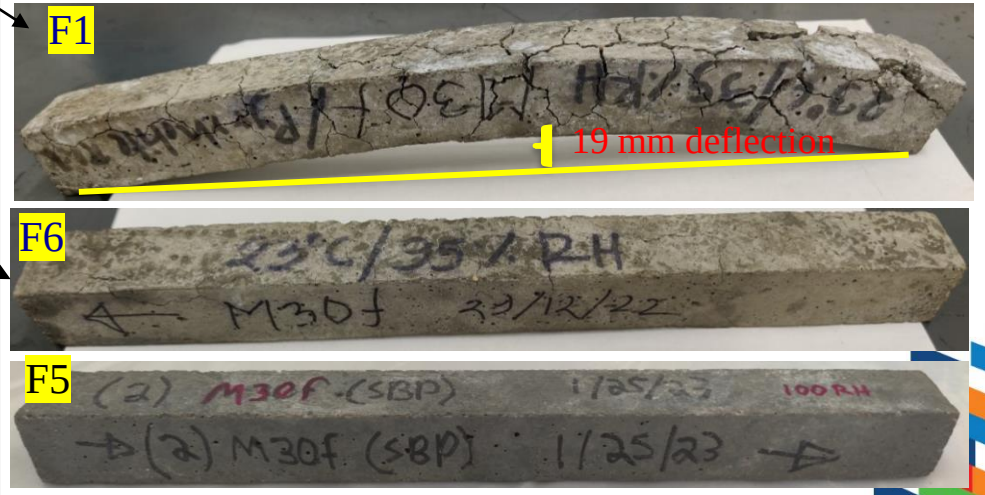
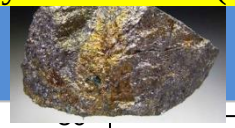


Figure 5: Sulfate generated over time (WD-XRF)



Pyrrhotite rock (Po)



Effect of Relative Humidity (95% and 100%)

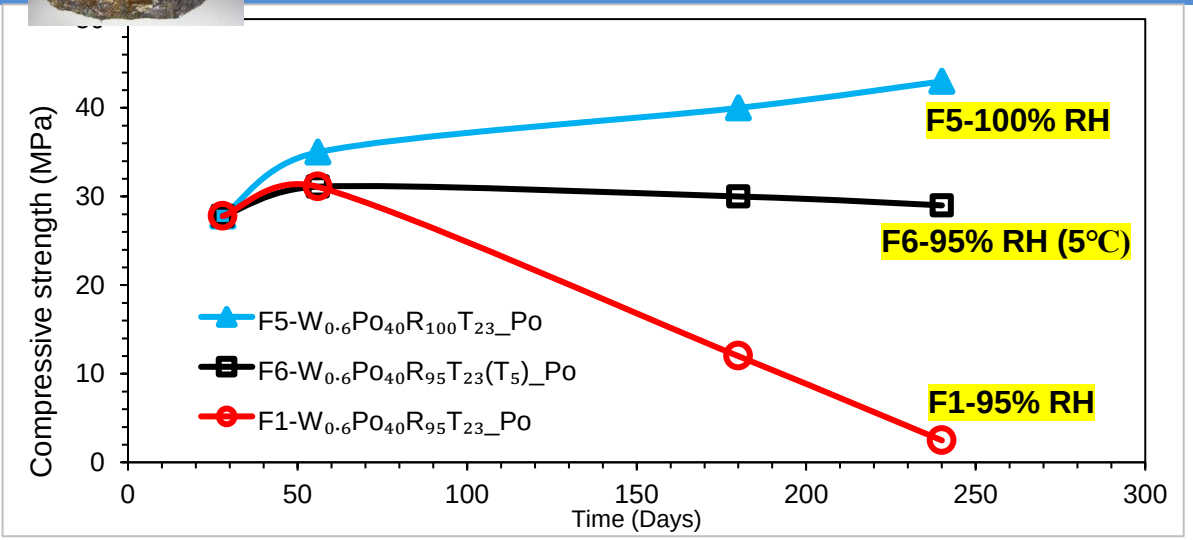


Figure 6: Degradation of concrete compressive strength

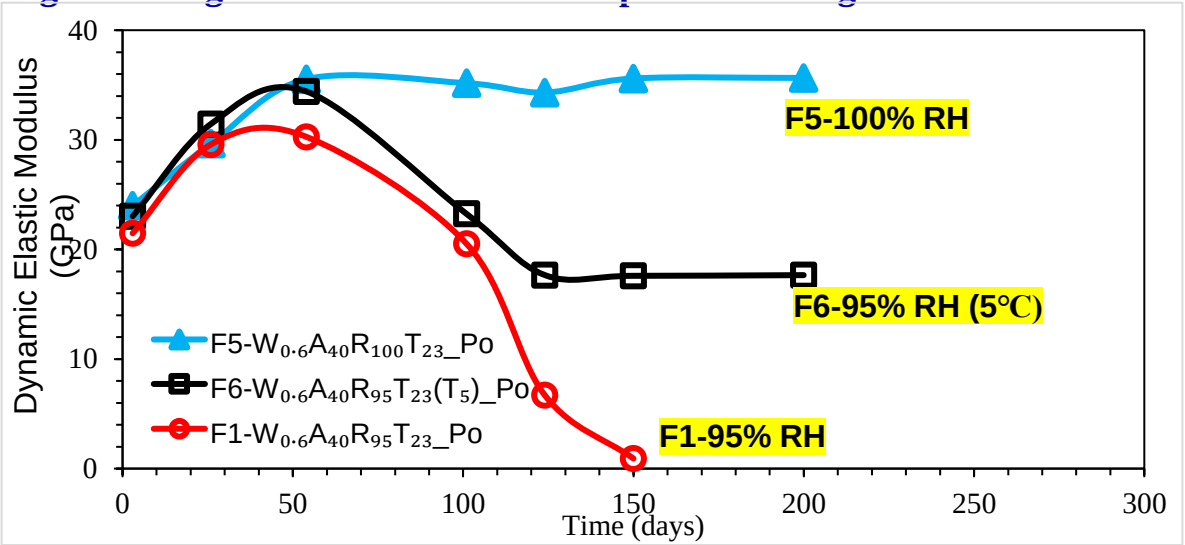
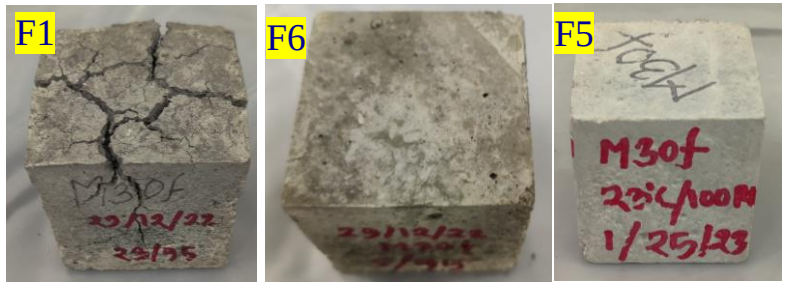


Figure 7: Degradation of Dynamic Elastic Modulus



Concrete cubes specimen at 8 months

Compressive Strength test (ASTM C 109)

Dynamic Elastic Modulus Resonance Frequency Test (ASTM C215)

Effect of concrete cross –section and air-entrainment

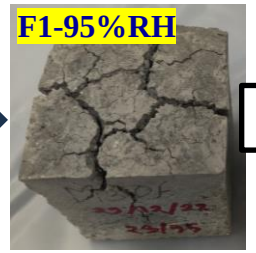
25x25 mm

Mortar bar

Start cracking before 3 months (Age=5 months)

50x50 mm

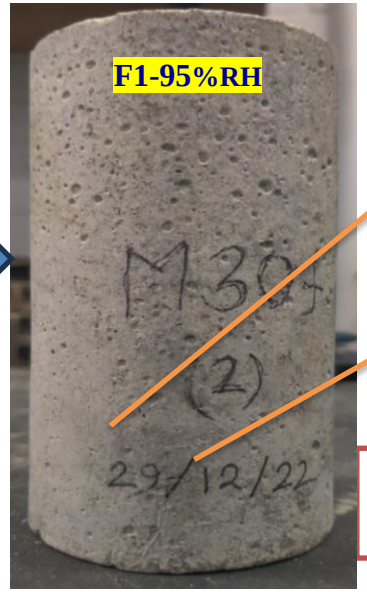
Cube

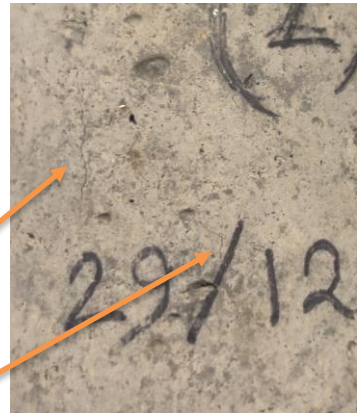


Start cracking after 3 months (Age = 8 months)

D (dia)=75 mm

Cylinder





Start Cracking at 10 months (Age = 10 months)

Figure 8: Effect of concrete cross-section

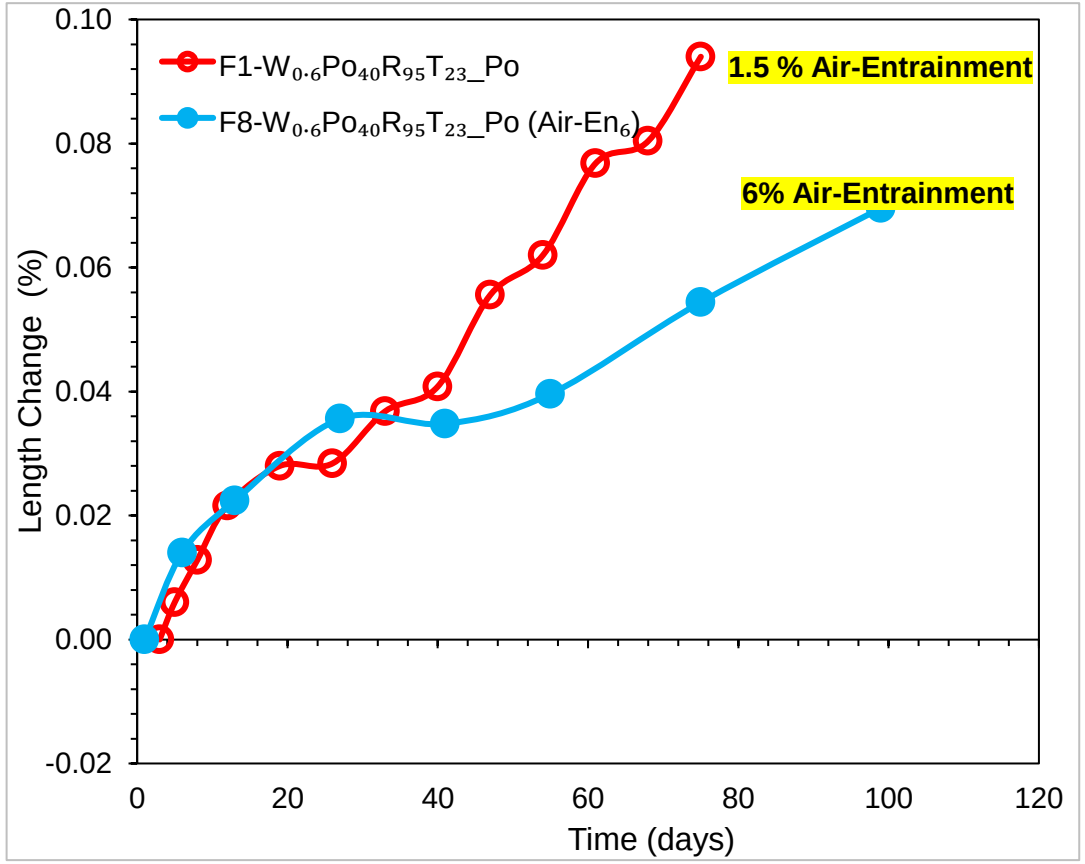


Figure 9: Effect of air-entrainment

Pyrrhotite rock (Po)

Effect of Aggregate Size and Temperature (5 to 80°C)

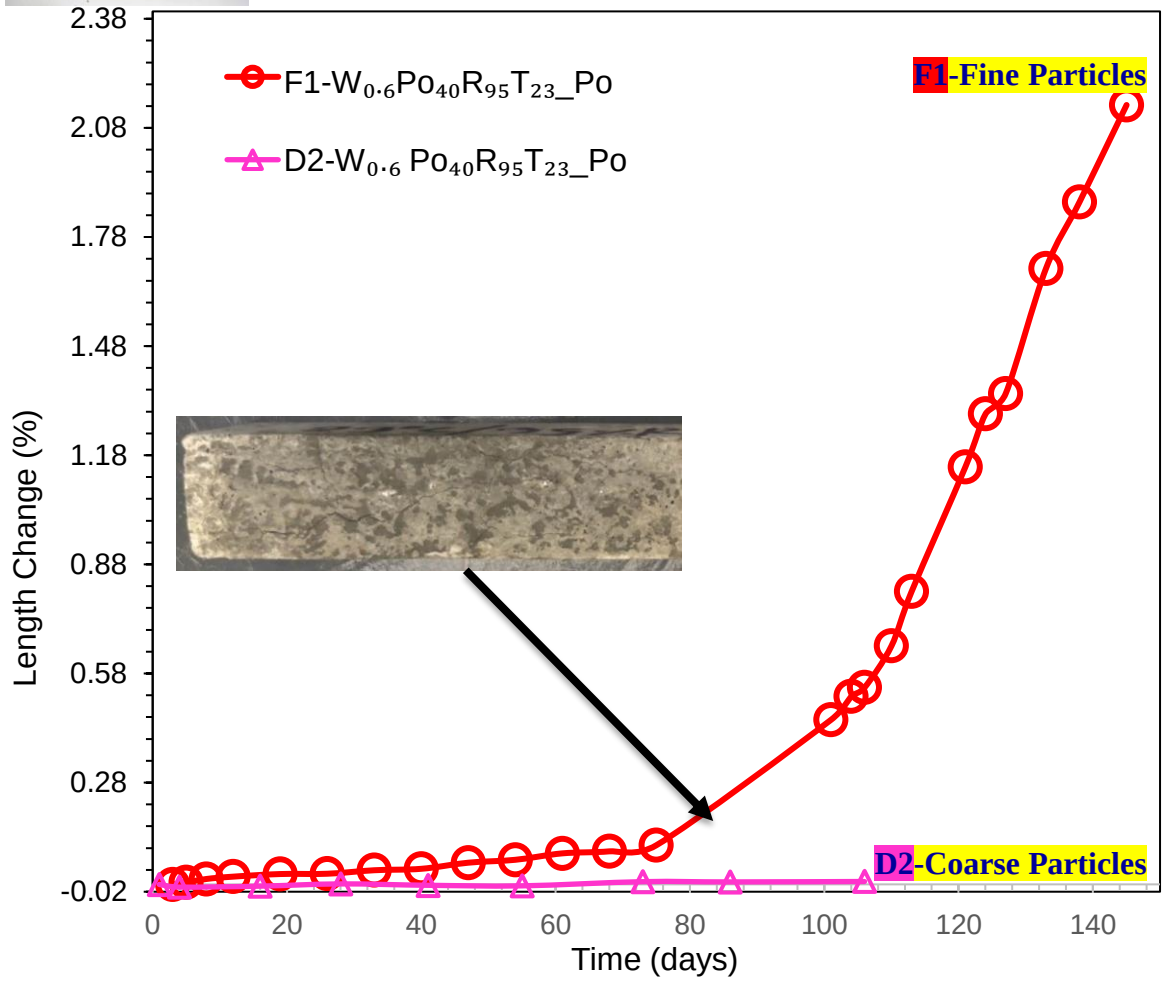


Figure 10: Sulfide bearing aggregate Size

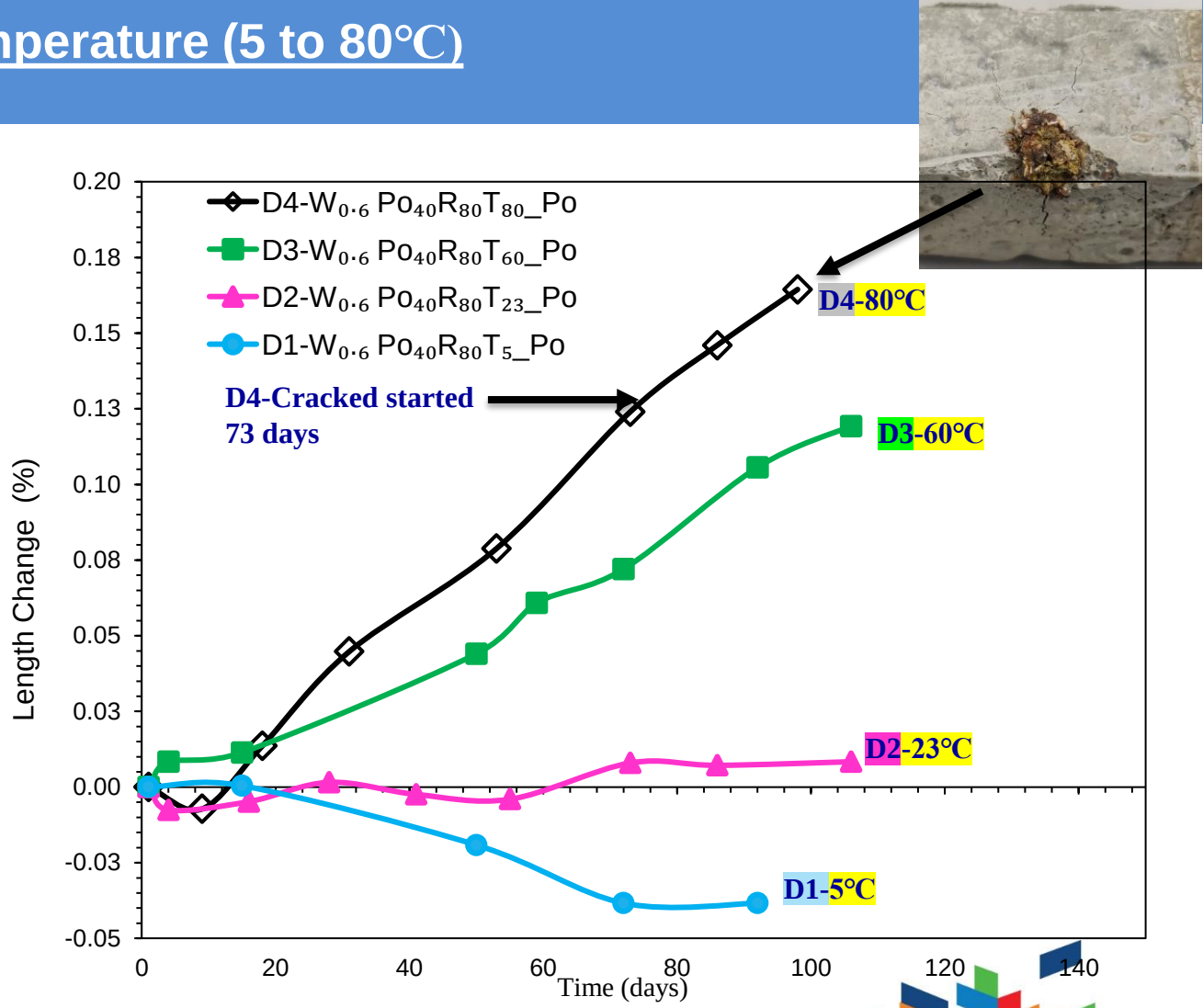


Figure 11: Temperature (5 to 80°C)

Effect of the type of Iron-sulfide and Galvanic interaction

Pyrrhotite rock (Po)

Pyrite (Py)

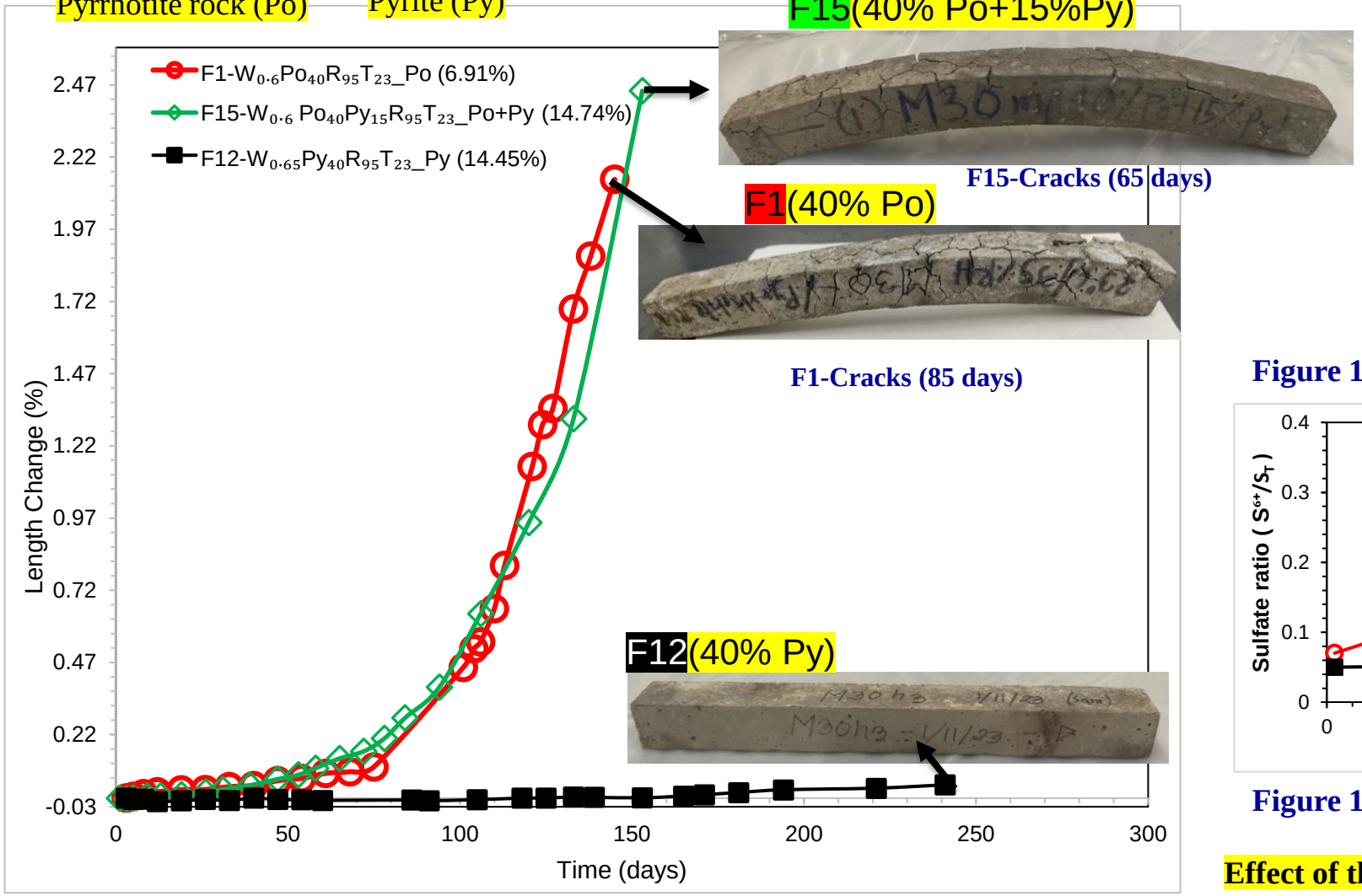


Figure 12: Iron-sulfide type (Pyrrhotite and Pyrite) and galvanic interaction

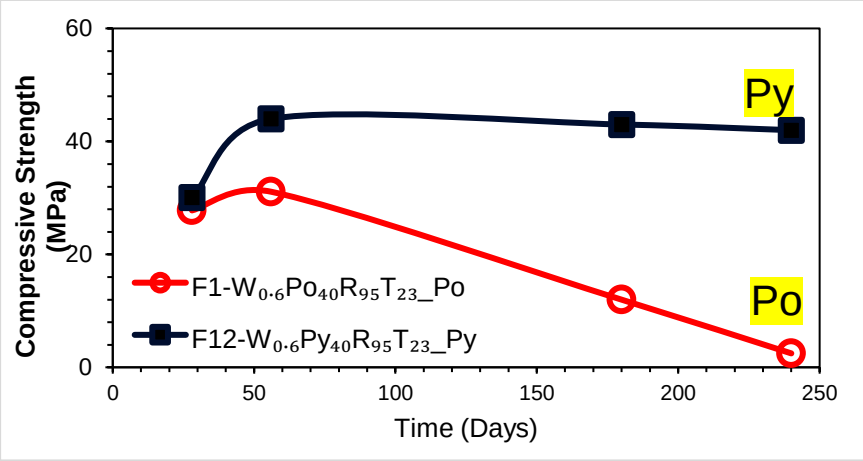


Figure 13: Compressive strength

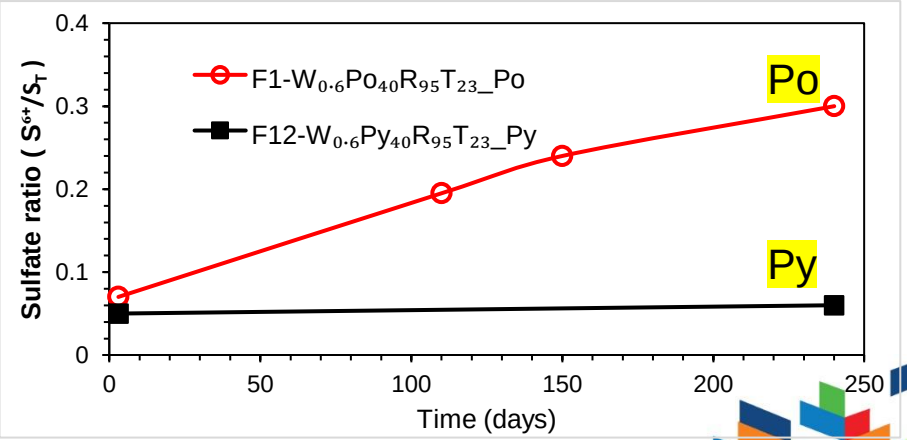


Figure 14: Sulfate generated over time

Effect of the type of iron-sulfide (Pyrrhotite and pyrite)

Conclusion (Part 1)

- Expansion, concrete compressive strength, dynamic elastic modulus, and sulfate generated over time can be measured to quantify concrete damage caused by iron sulfide oxidation.
- An excellent correlation exists between expansion, concrete compressive strength, and dynamic elastic modulus including sulfate generation in concrete.
- Concrete specimens with smaller particle sizes, less air entrainment, and smaller cross-sections exhibited the fastest expansion and cracking due to iron-sulfide oxidation
- Oxidation rates increase with increasing pyrrhotite content, galvanic interaction, and a temperature of 80°C.
- A 95% rh showed the greatest expansion in contrast to a 100% rh.
- Pyrrhotite exhibited almost 100 percent faster oxidation rate than pyrite

Part II: Electrochemical Acceleration Method

- **Overview of Acceleration Method**
- **Accelerated Test Setup Description**
- **Sample Preparation**
- **Results**
- **Conclusion**

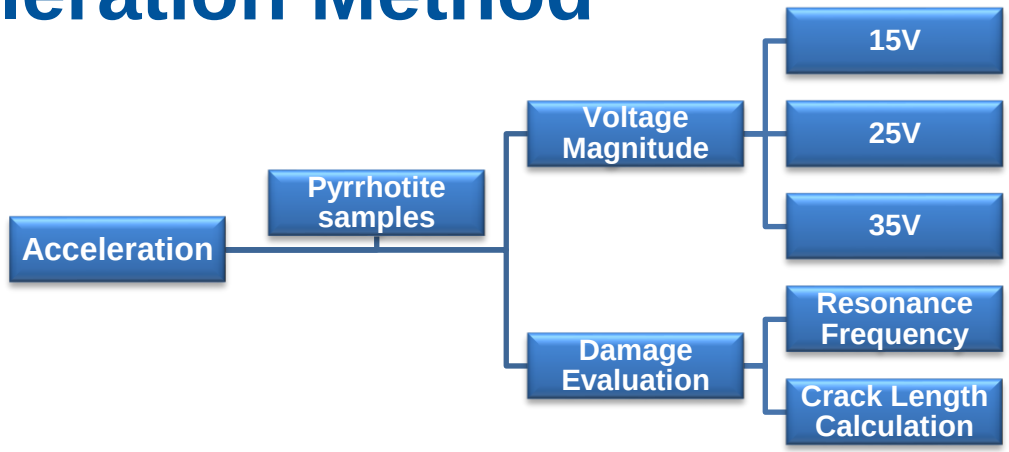
Overview of Acceleration Method

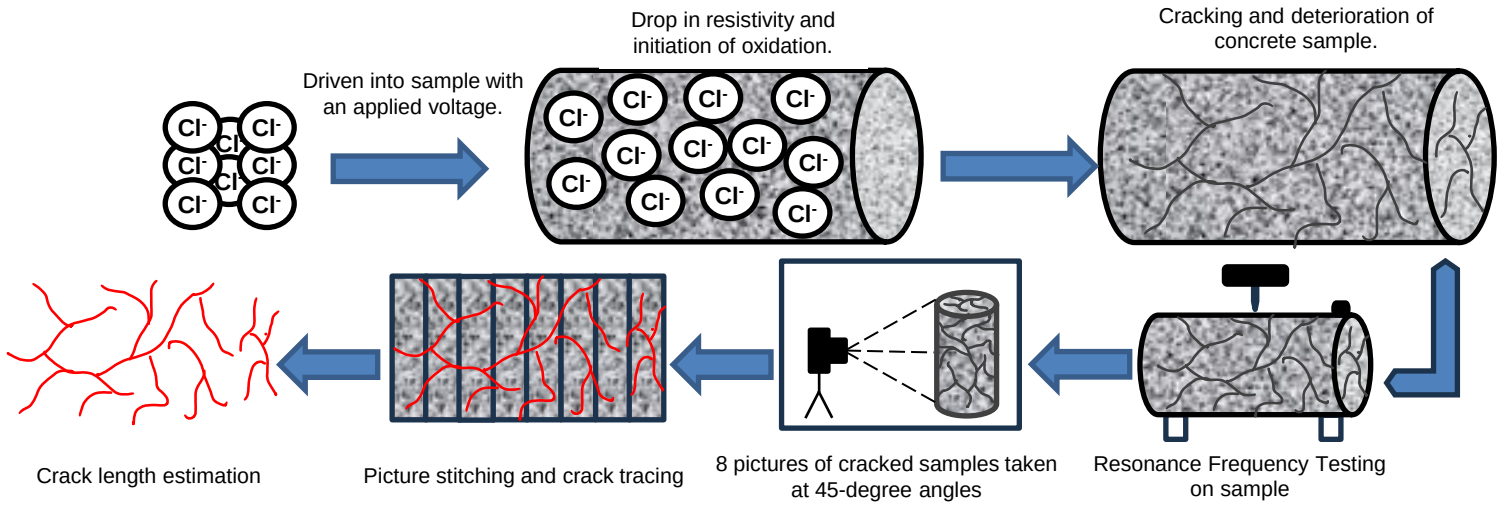
Problems

- Interconnected and complicated damage mechanisms.
- Unfeasible timeframe for replicating damage process in a laboratory environment.

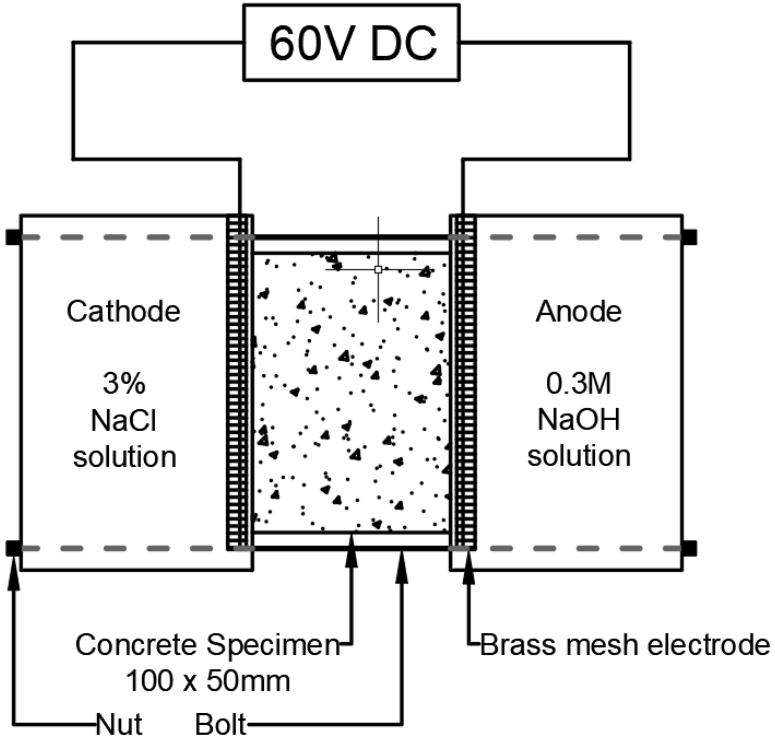
Goals

- Identify and define damage parameters.
- Investigate and verify damage parameters of samples in comparison to field deterioration.

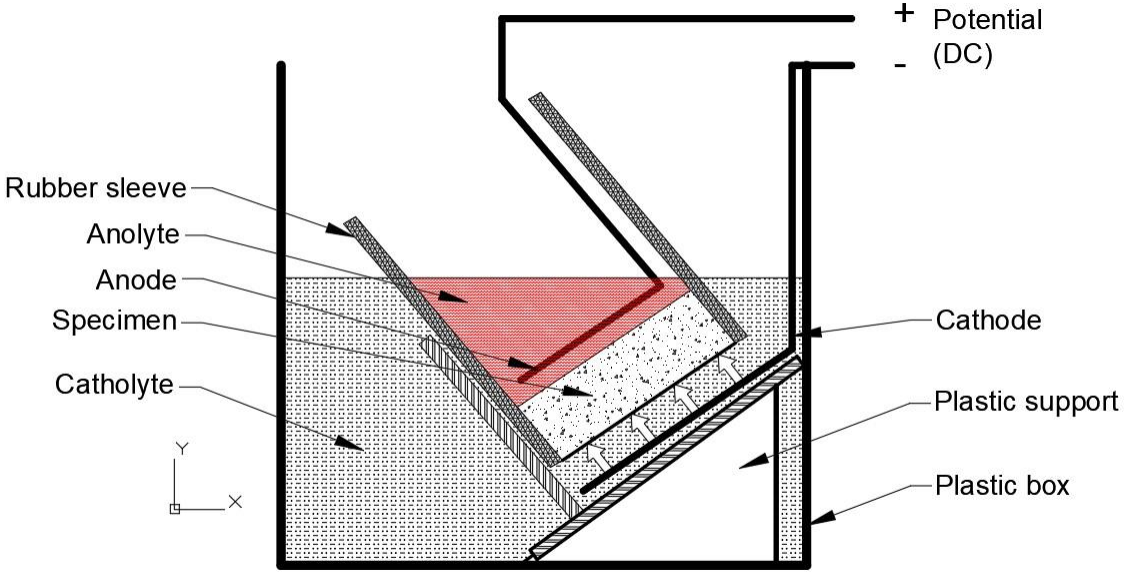




Accelerated Test Setup

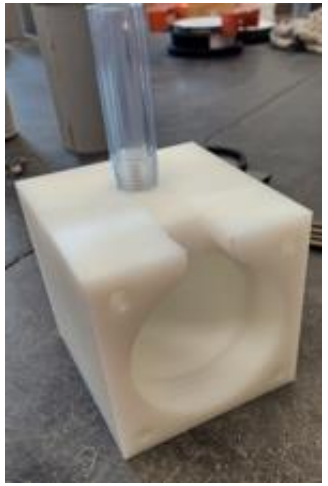


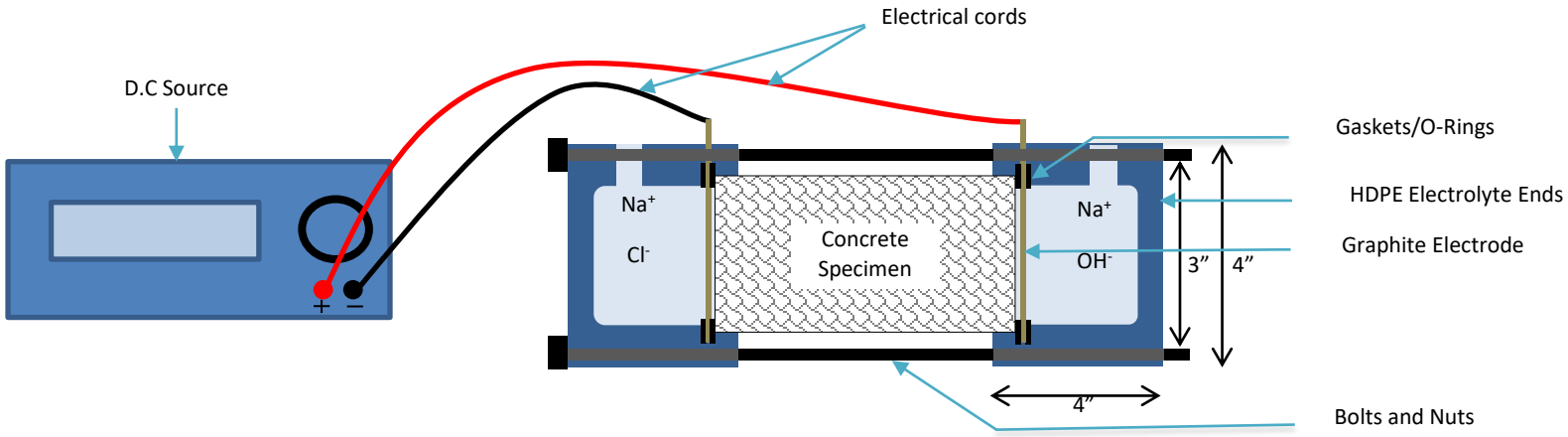
ASTM C1202 – 22 Setup

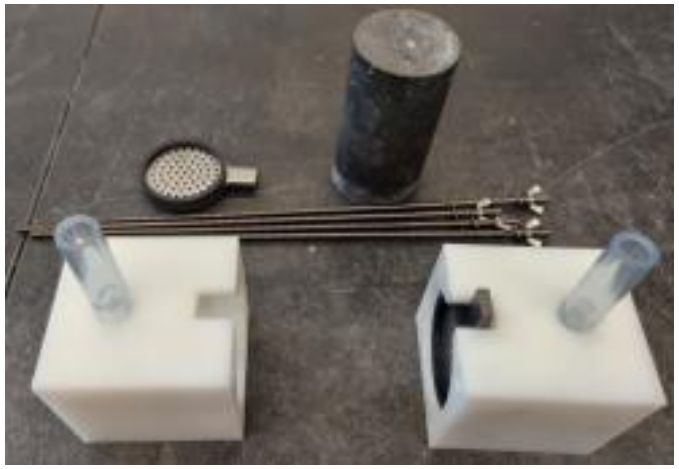


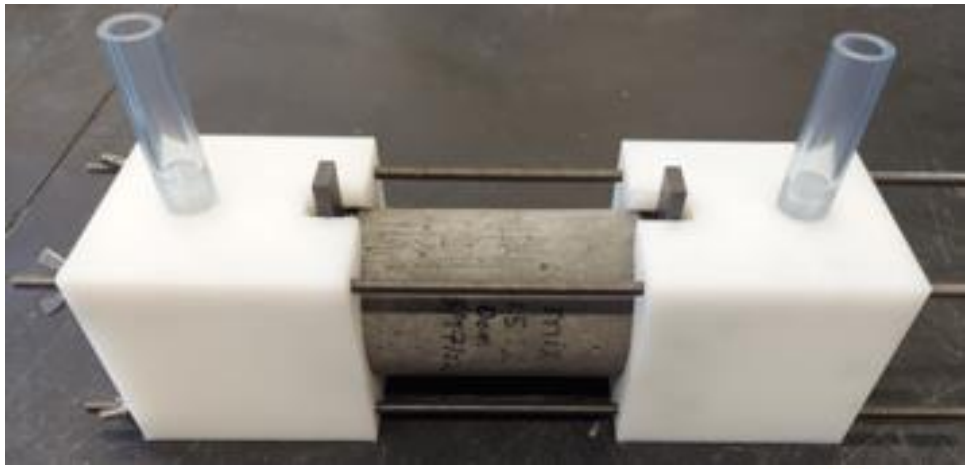
NT BUILD 492 – 99 Setup

Accelerated Test Setup









Sample Preparation

Pyrrhotite Aggregates (**1/2 – 5/8”**)
Total Sulfur: **1.7%**

Control Aggregates (**1/2 – 5/8”**)
Total Sulfur: **0.04%**

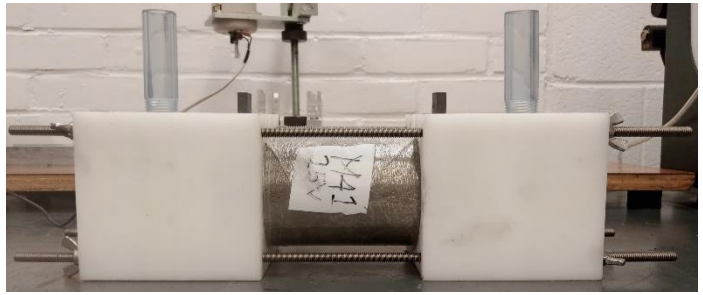
Casting molds for
(3 x 6)” specimens

Saw-cutting specimen to 4.5”

specimen grinding
for even surface

Ground specimens

Specimens wrapped in plastic
sheets



Specimen in accelerated testing
setup

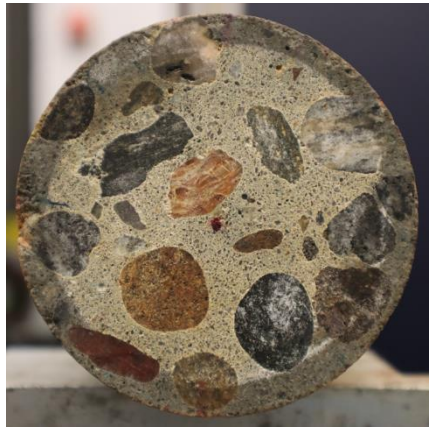
Accelerated Testing Results (Day 35 at 35V)





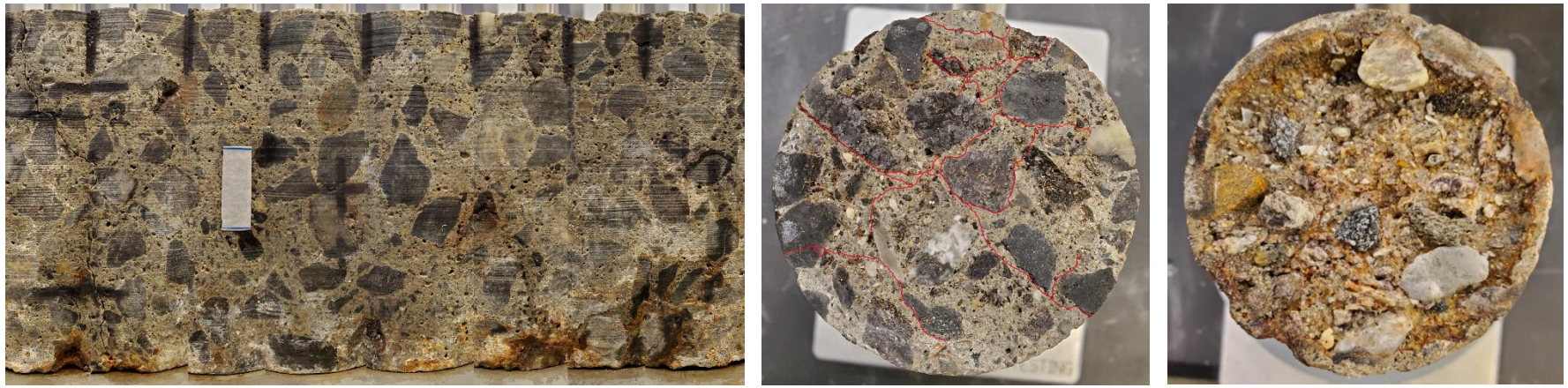
Pyrrhotite-bearing Sample

Day 84

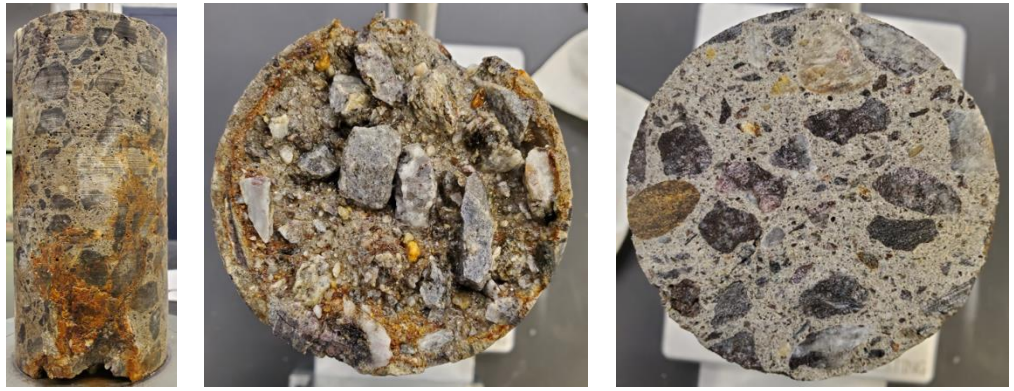



Control Sample

Accelerated Testing Results – Field Sample



Field Sample – Day 112

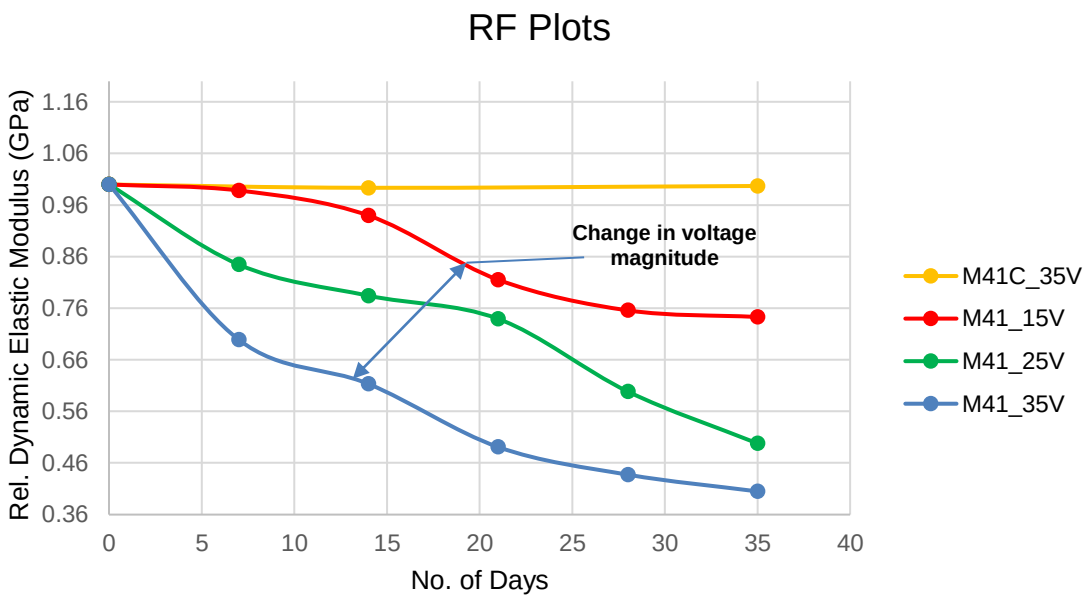


Field Sample – Day 56

Dynamic Elastic Modulus Results from Resonance Frequency

Resonance Frequency Testing

Day	M41_35V			M41_25V			M41_15V			M41C_35V		
	RF (Hz)	E (Gpa)	Rel. E (Gpa)	RF (Hz)	E (Gpa)	Rel. E (Gpa)	RF (Hz)	E (Gpa)	Rel. E (Gpa)	RF (Hz)	E (Gpa)	Rel. E (Gpa)
0	12361	40.21	1.00	12452	40.48	1.00	12347	38.53	1.00	8025	32.24	1.00
14	9681	24.66	0.61	11029	31.76	0.78	11970	36.21	0.94	8026	32.03	0.99
35	7814	16.29	0.41	8721	20.16	0.50	10644	28.63	0.74	8023	32.15	1.00



$$E = CMn^2$$

M = Mass of specimen (kg)

n = Fundamental transverse frequency (Hz)

C = correction factor constant

E = Dynamic elastic modulus

CONCLUSIONS

Employing the adopted electrochemical method effectively accelerated the oxidation and deterioration of laboratory-cast and field concrete samples.

As exposure time to accelerated oxidation increased, RFs and Es in the pyrrhotite-bearing samples and field samples declined, while the control sample remained stable, showing no measurable sign of deterioration.

Higher applied voltages were directly associated with increased oxidation, deterioration rates, and greater reduction in RFs and Es in pyrrhotite-bearing samples.

These findings will aid in understanding the damage evolution, will allow for evaluating the potential risk of concrete failure and will be critical for the study of potential mitigation strategies towards the stability and longevity of residential structures.

Acknowledgments

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