



# Development of an accelerated method on concrete to evaluate the deleterious oxidation potential of sulfide-bearing aggregates

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**Co-supervisor- Josée Duchesne, Ph.D.**





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Introduction

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Problem statement

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Research objectives

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Experimental program

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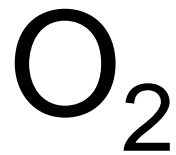
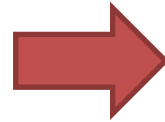
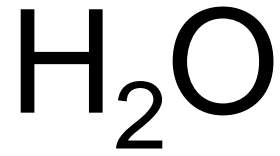
Preliminary results

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Conclusions and perspectives

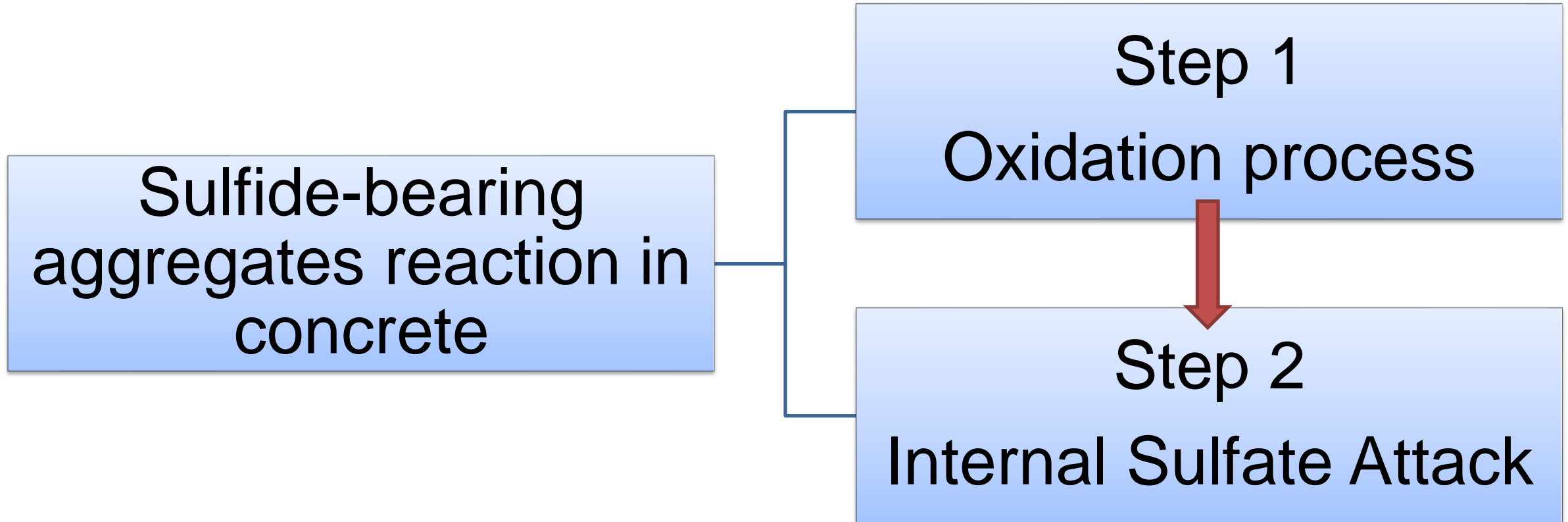


# Introduction - Iron sulfides





# Introduction – Sulfide-bearing aggregates





# Introduction

## Oxidation Process



Mikhlin et al. (2002)  
Belzile et al. (2004)



# Introduction

## Internal Sulfate Attack



**H<sub>2</sub>SO<sub>4</sub> (sulfuric acid)**

- Sulfuric acid + portlandite → **gypsum**
- C<sub>3</sub>A (cement) + gypsum “sulfates” + water → **ettringite**
- “Sulfates” + carbonates + “C-S-H” + water → **thaumasite**

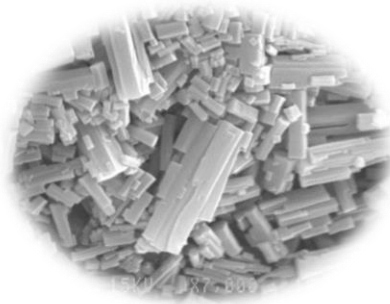
Calcium Hydroxide  
[Ca(OH)<sub>2</sub>]



**Gypsum**



**Ettringite**



**Thaumasite**



Shayan A. (1998)  
Rodrigues et al. (2015)



# Problem Statement – Trois-Rivières



Rodrigues et al. (2012)



# Problem Statement – Trois-Rivières



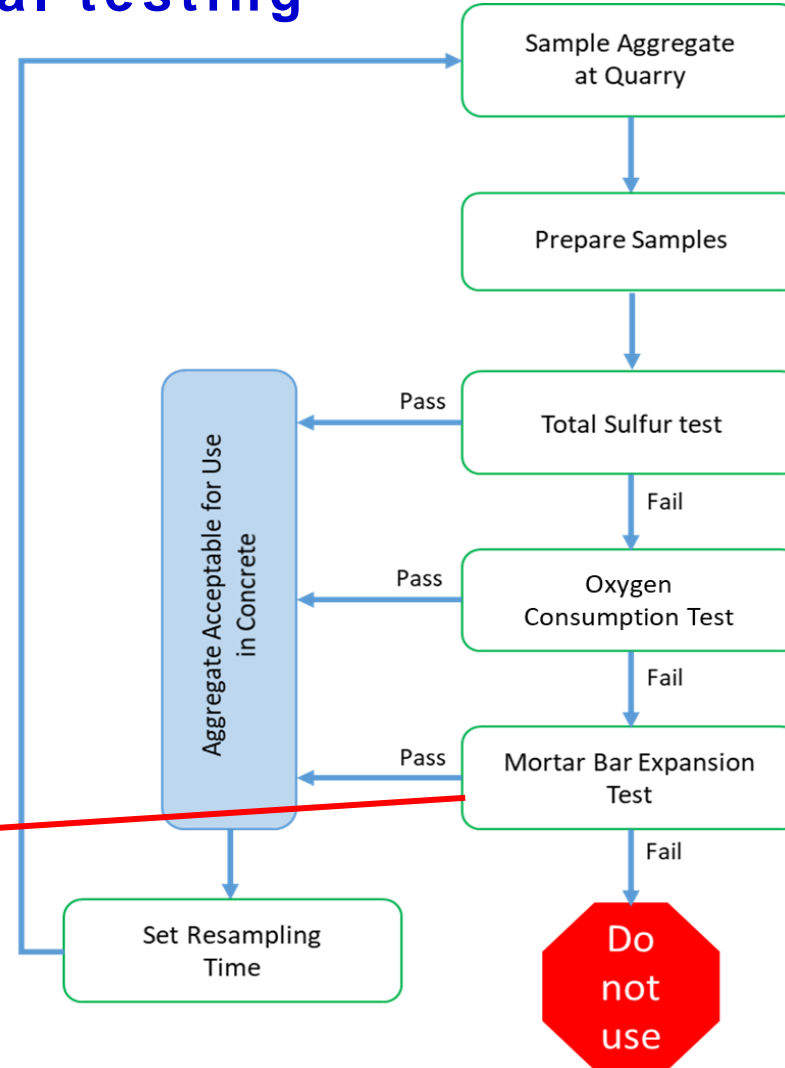
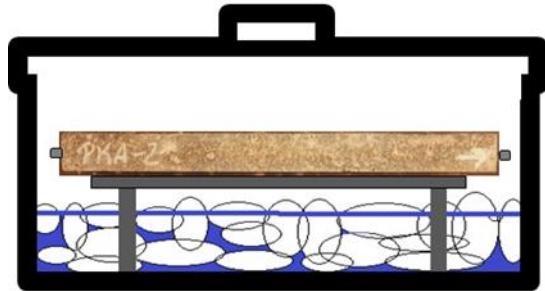
Photos taken by Rodolfo C. (2019-2020)





# Problem Statement

## Actual testing





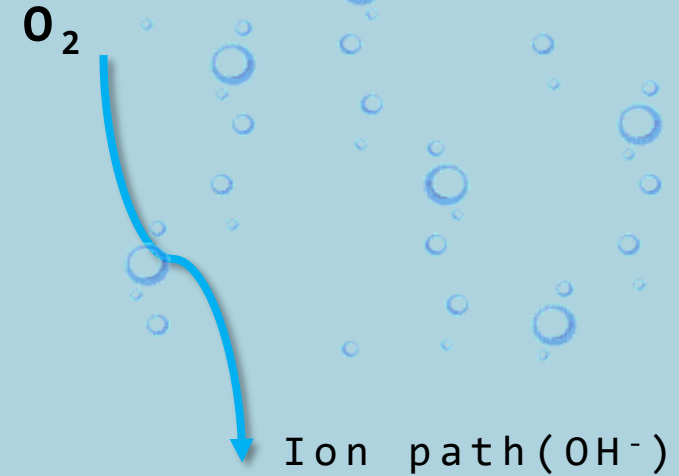
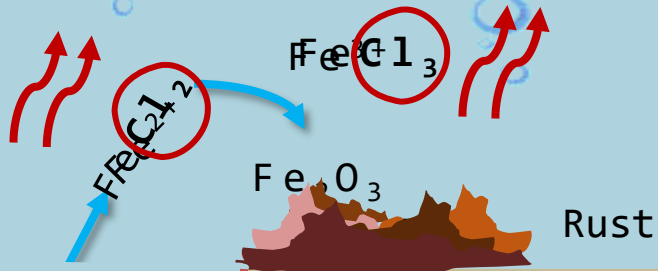
# Problem Statement

## Corrosion

Water (H<sub>2</sub>O)

Chlorides (Cl<sup>-</sup>)

O<sub>2</sub>

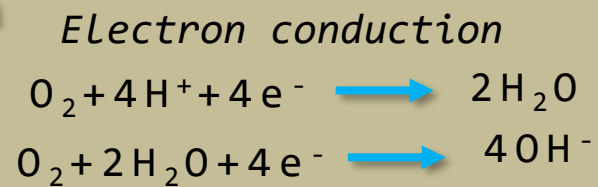


Steel

**Anodic area (+)**  
Where metal dissolves  
 $Fe \rightarrow Fe^{2+} + 2e^{-}$

Electron path (e<sup>-</sup>)

**Cathodic area (+)**



Kosmatka et al. (2002)  
Broomfield (2003)  
Bohni. (2005)



# Research Objectives

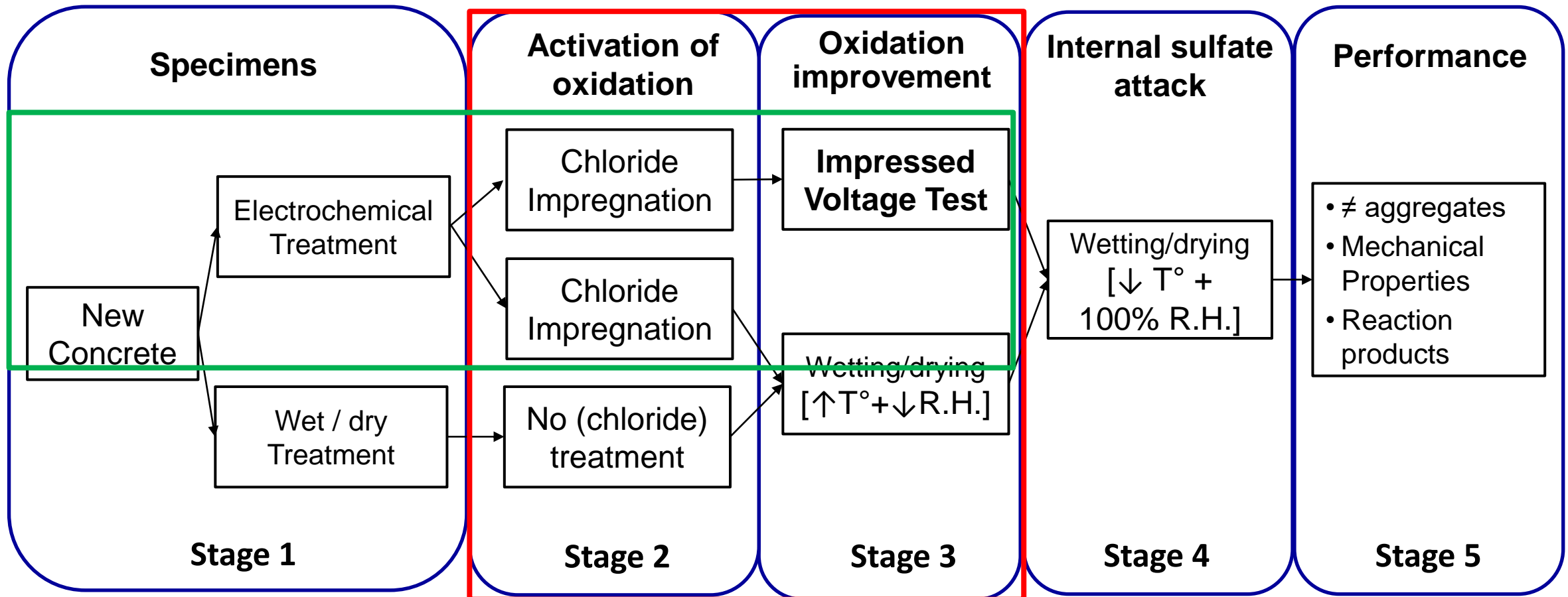
Understand the effect of moisture content and relative humidity

Develop a new accelerated methodology in concrete

Evaluate the damage due to SBA oxidation reaction



# Experimental Program - Methodology





# Experimental Program

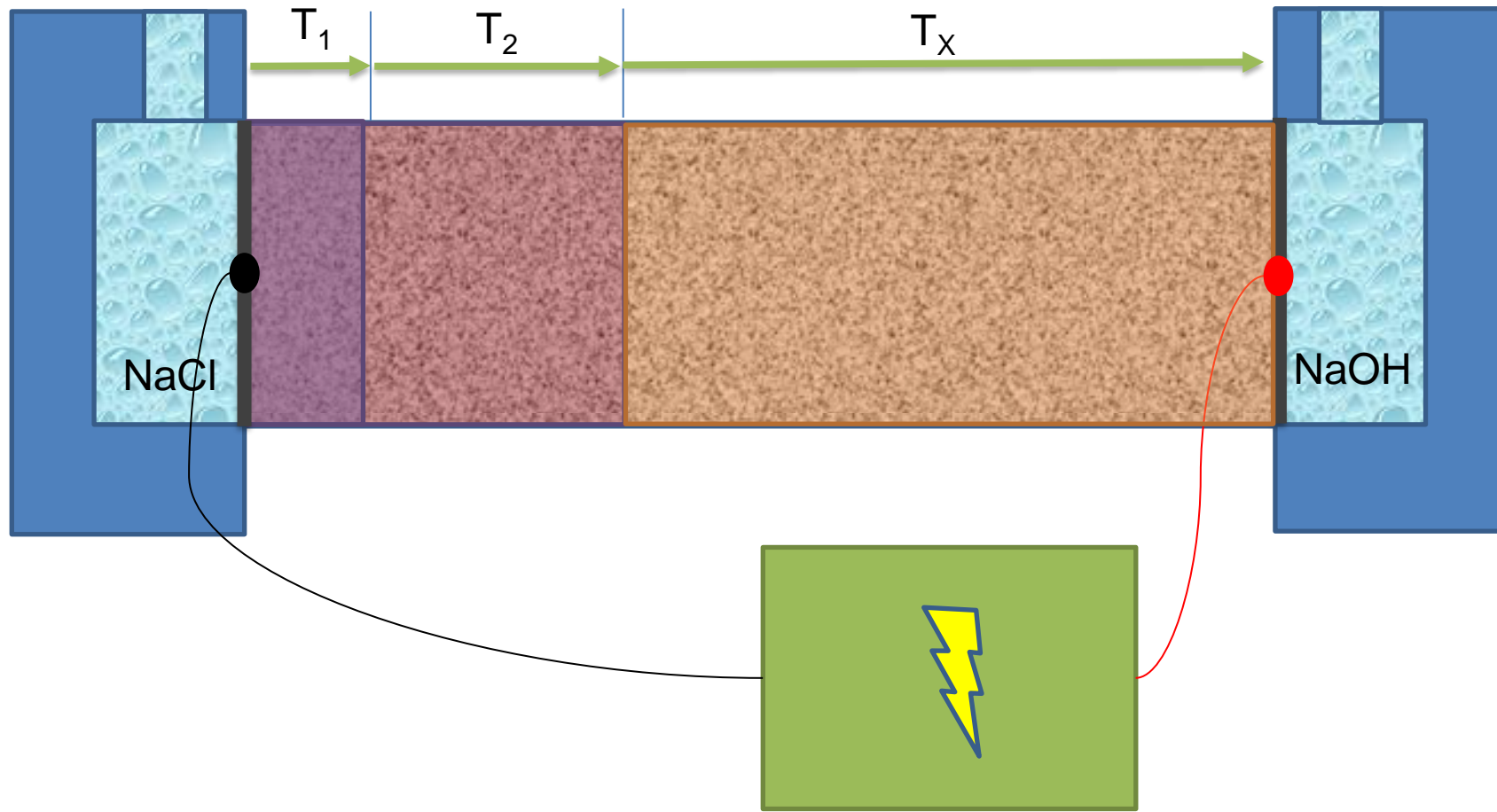
## New Concrete

- $W/C = 0.65$
- A high-alkali General use (**Type GU**) cement
- Coarse aggregate (5-20 mm)
  - High Purity Limestone (HP) / (Total Sulfur : [0.0])
  - Anorthosite (PKA) / (Total Sulfur : [0.0])
  - Crushed granitic gneiss (LT) / (Total Sulfur : [0.04])
  - Anorthosite Gabbro (MSK) / (Total Sulfur : MSK-R [1.86] & MSK [0.9])
- Non reactive granitic sand
- Based on experimental work by Durand (Hydro-Quebec)





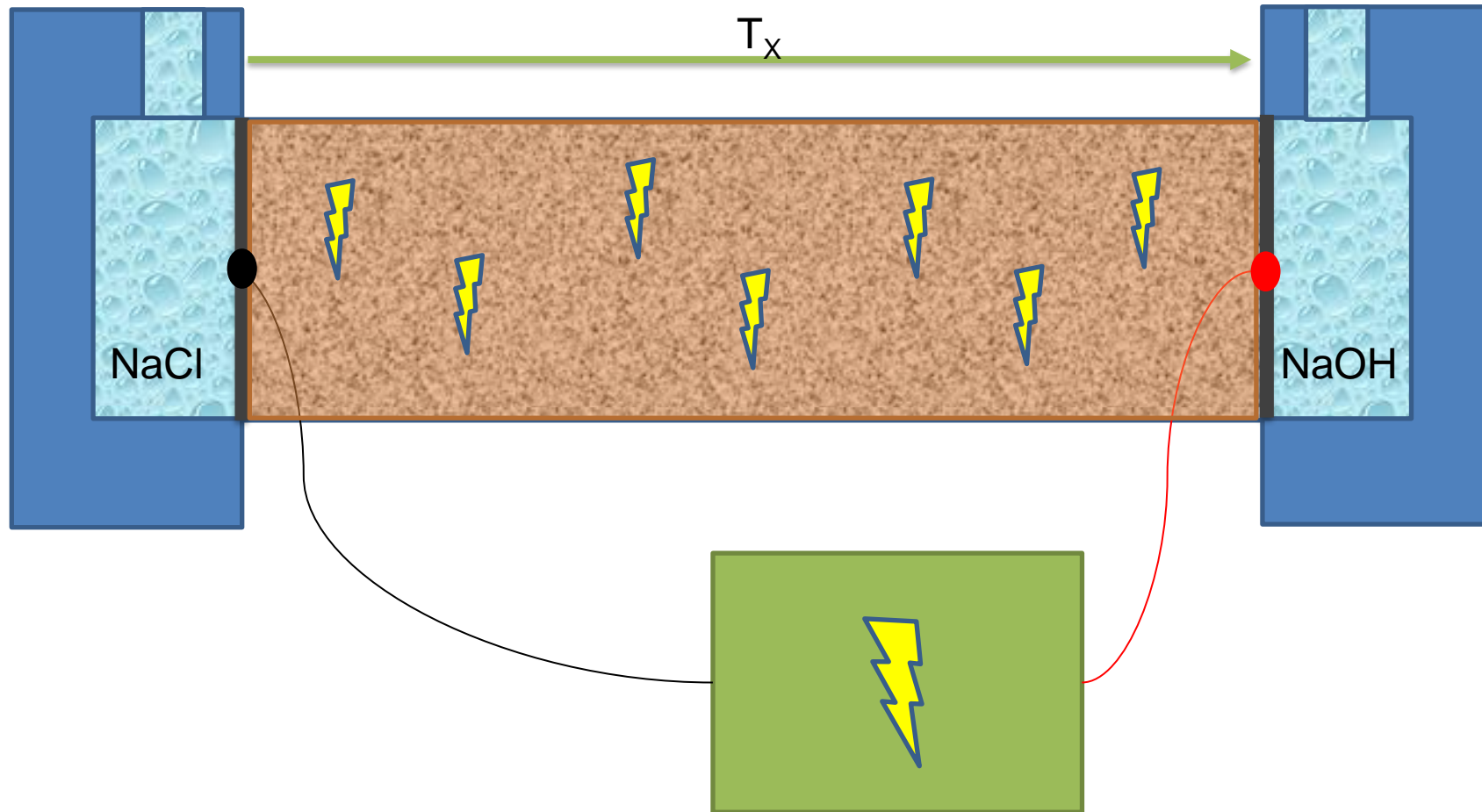
# Electrochemical Treatment: Chloride Impregnation



NT Built 492 (1999)

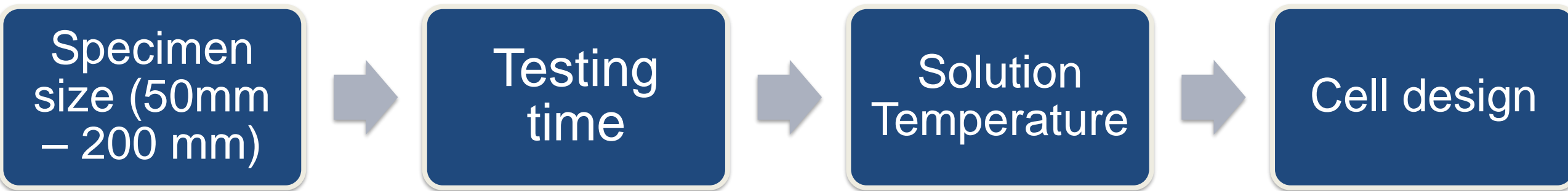


# Electrochemical Treatment: Impressed Voltage



Sivasankar (2013)

# Electrochemical Treatment







# Electrochemical Treatment: Sample size and time of testing

Starting with 100 mm long specimen  
Same voltage proposed by NT built 492



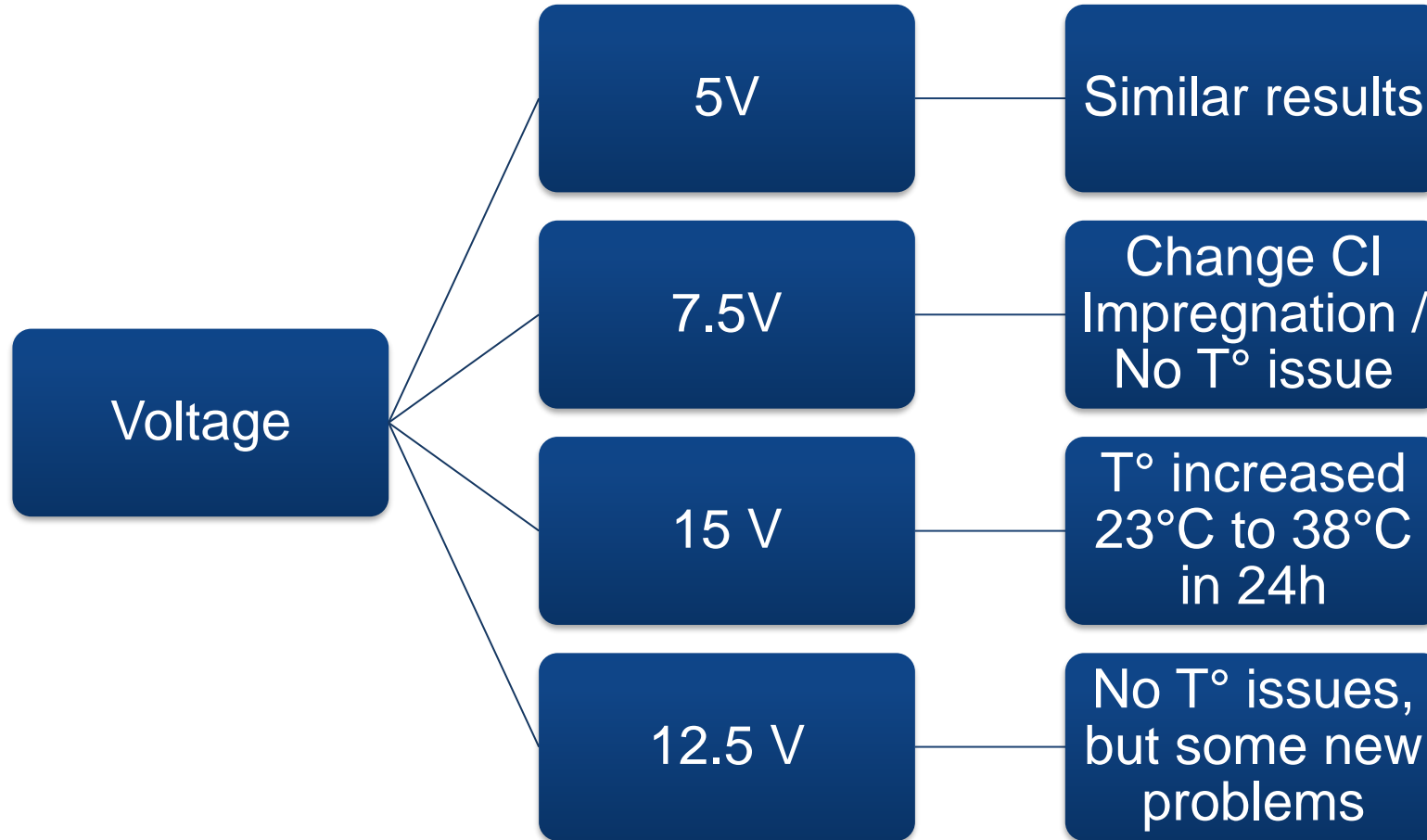
After 1 day of testing



After 5 day of testing

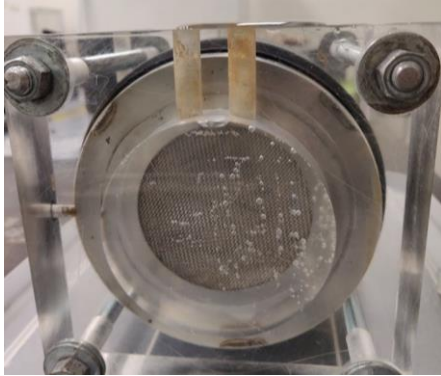


# Electrochemical Treatment: Sample size and time of testing

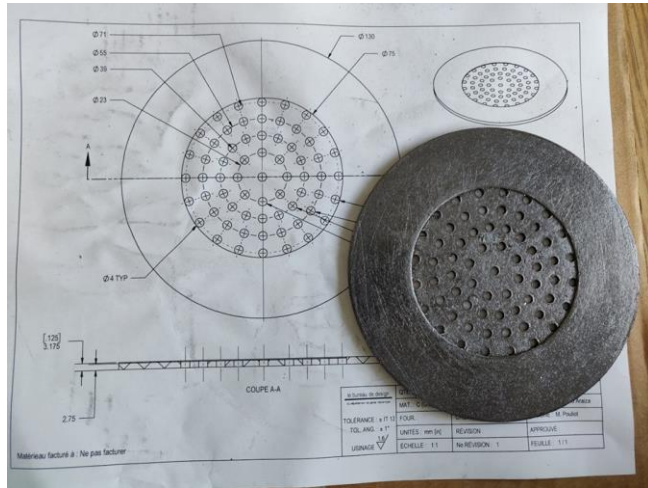




Stainless Steel



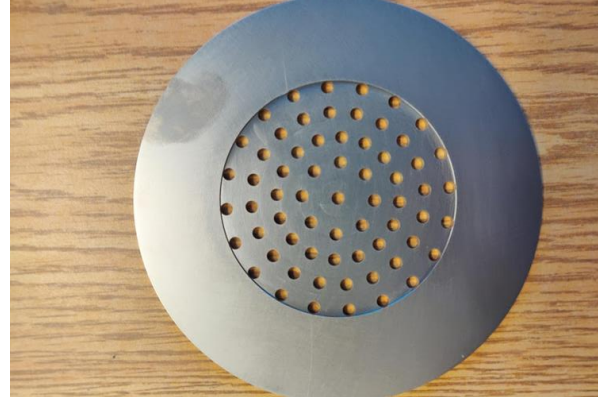
After 10 days of testing



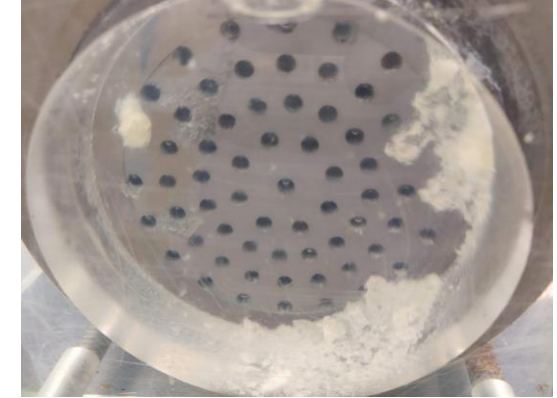
# Electrochemical Treatment:

## Electrochemical cell configuration

Titanium



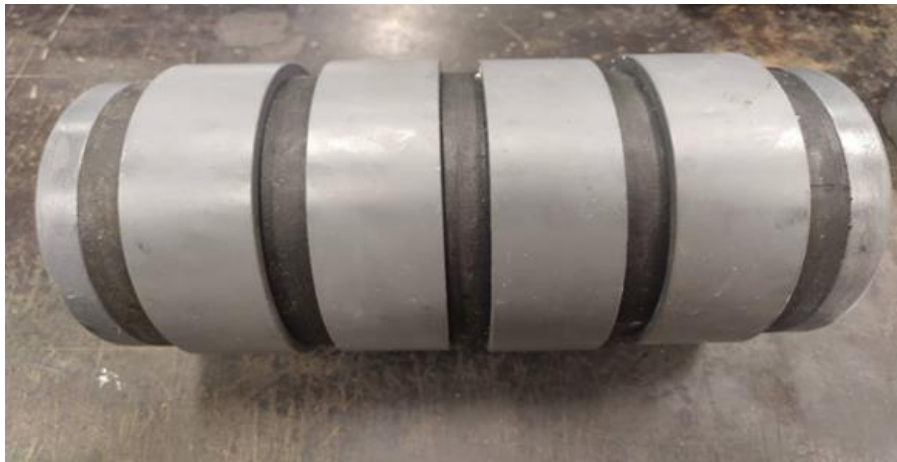
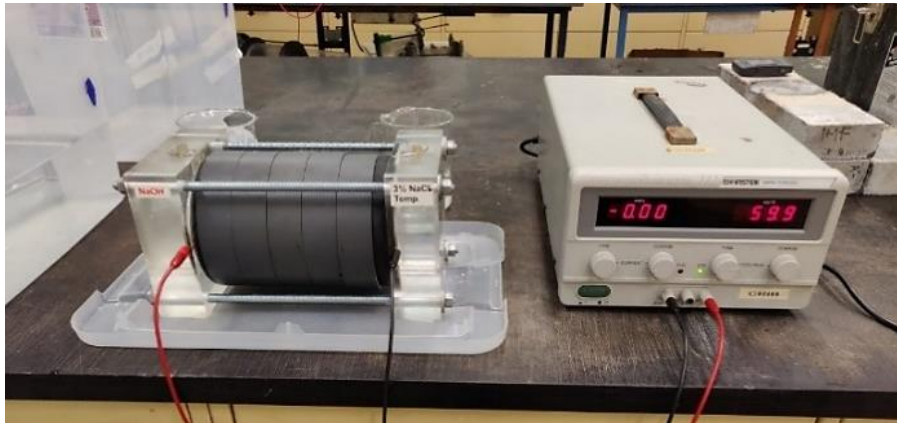
After 7 days of testing



After 14 days of testing



# Electrochemical Treatment: Electrochemical cell configuration





# Electrochemical Treatment: Chloride impregnation results



**3 days**  
6 cm



**7 days**  
13 cm



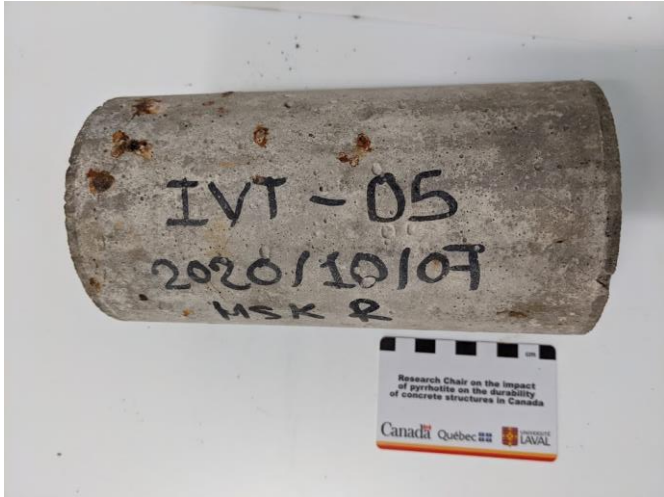
**10 days**  
17 cm



**14days**  
20 cm



# Electrochemical Treatment: Impressed Voltage



Chloride Impregnation (14d)



Chloride Impregnation (14d) + Impressed Voltage (7d)

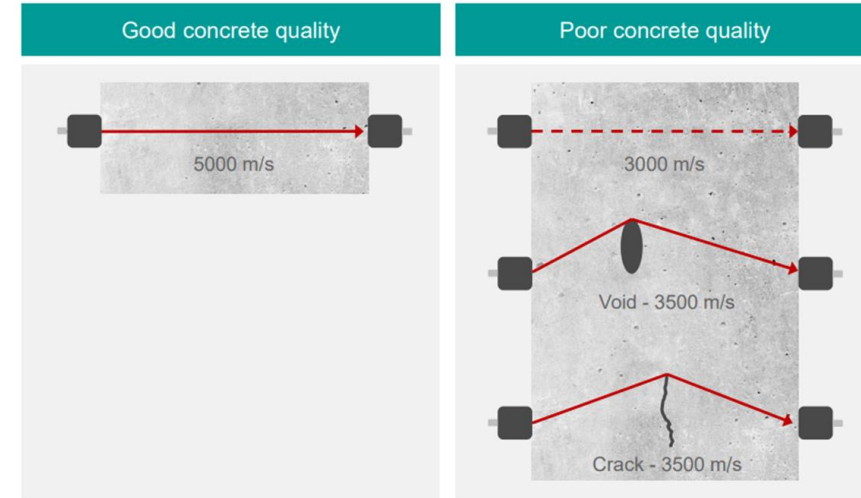
MSK-R [1.86]





# What to measure?

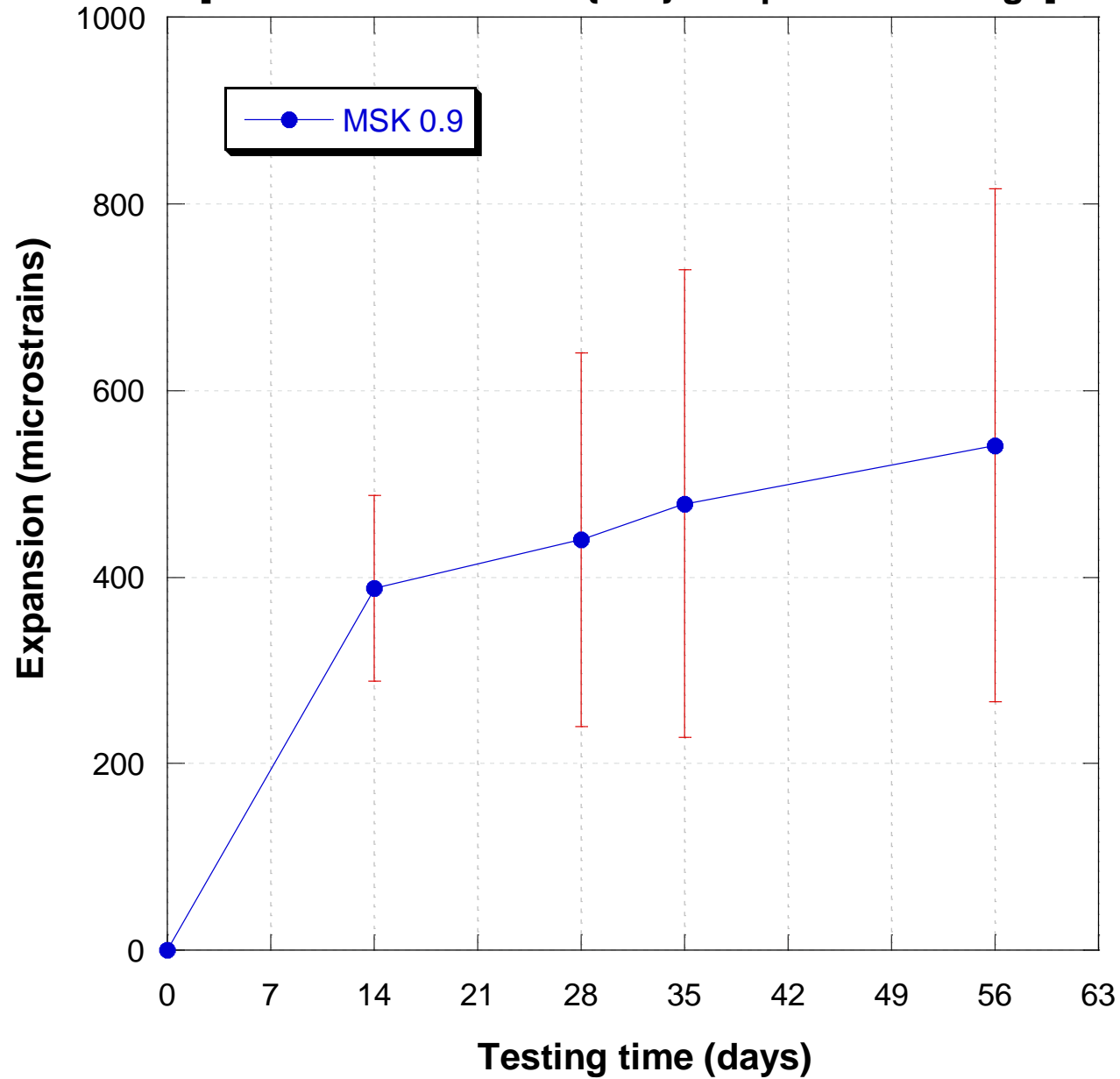
- Compressive Strength
- Stiffness Damage Test (SDT)
  - Modulus of Elasticity
  - Stiffness Damage Index (SDI)
- Damage Rating Index (DRI)
- Ultrasonic Pulse Velocity
- Electrical Resistivity
- Expansion





# Expansion

## Electrochemical Treatment [Chloride Penetration {14d} + Impressed Voltage]

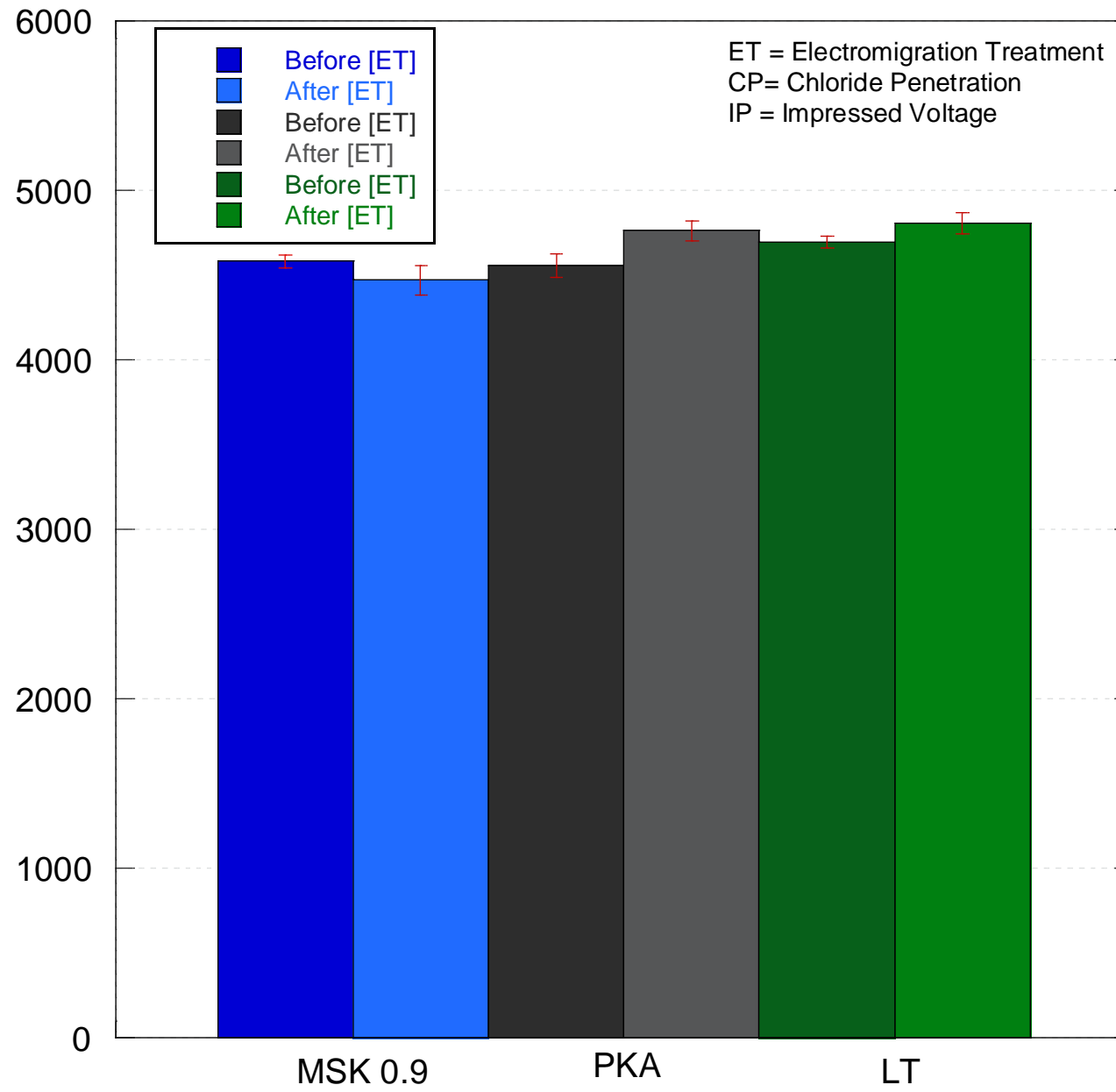




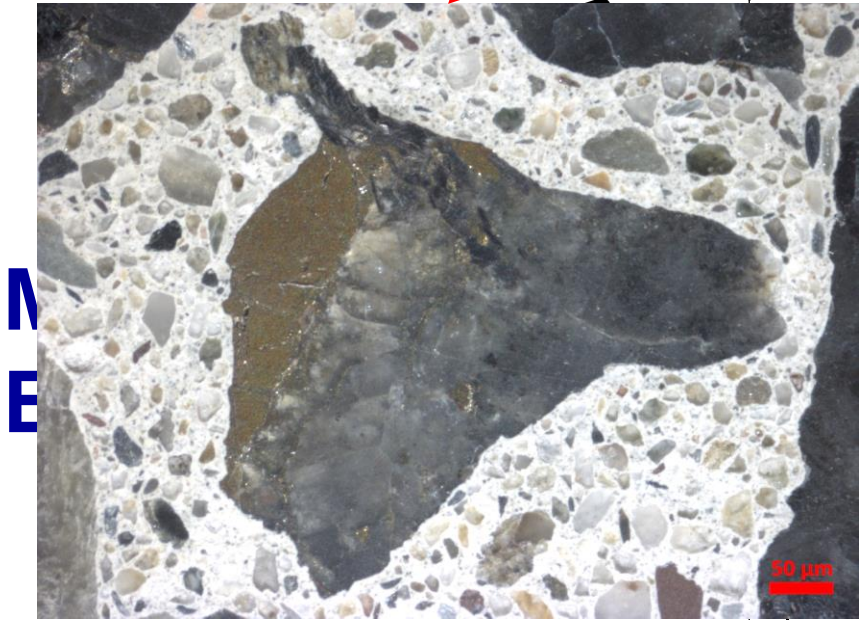


# Ultrasonic Pulse Velocity

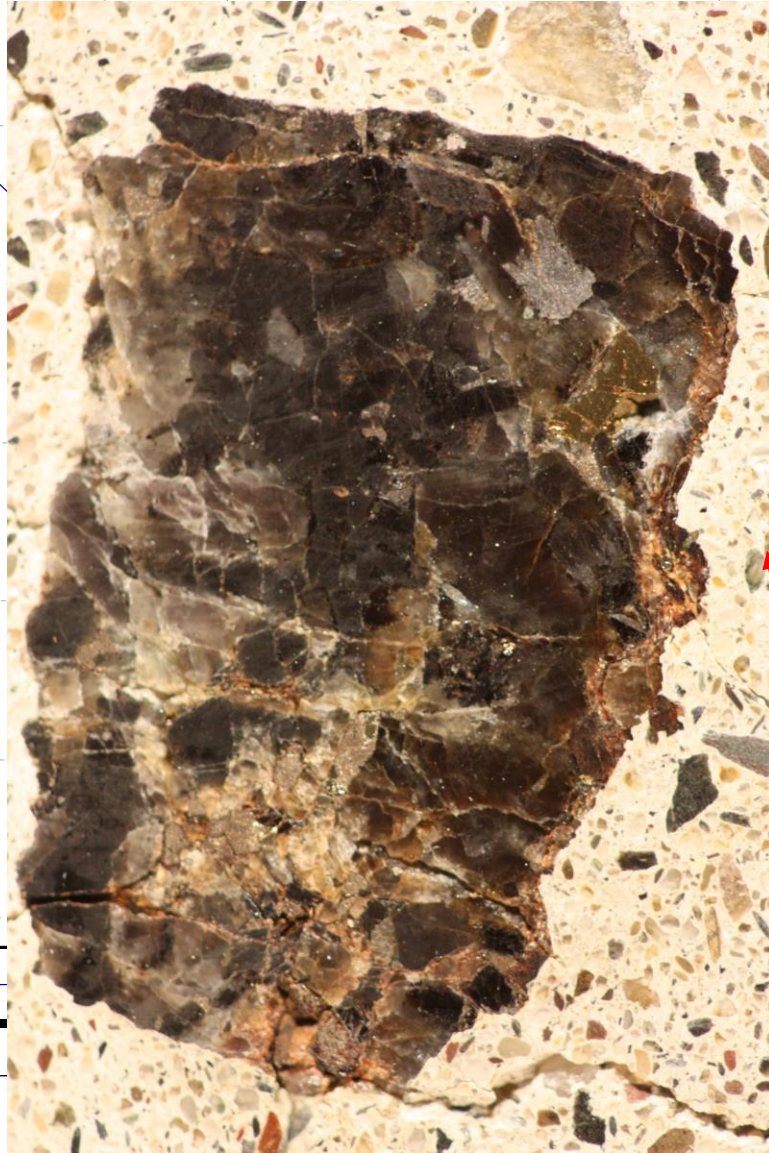
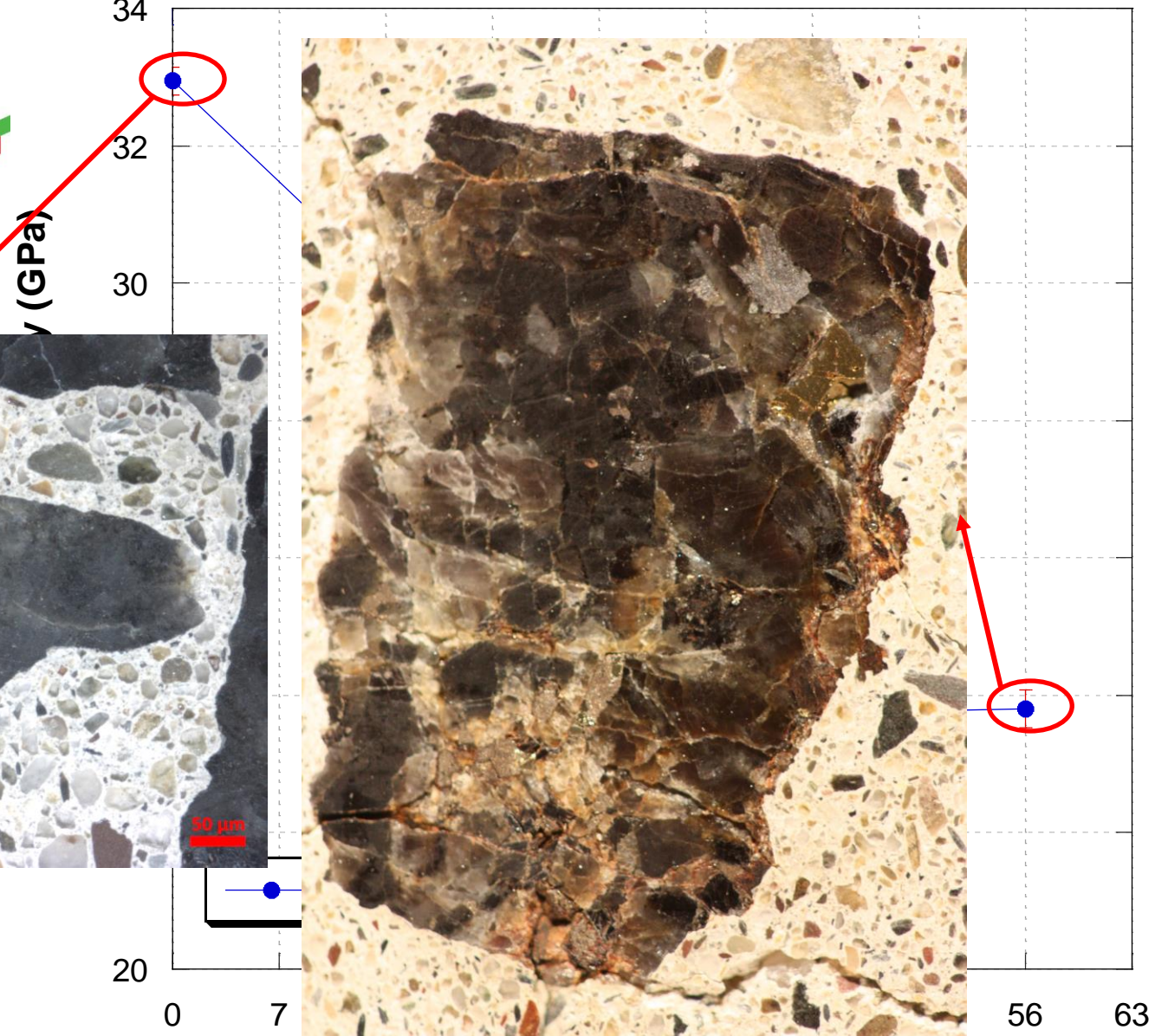
Ultrasonic Pulse Velocity (m/s)



Testing time = 35 days [14 CP + 21 IV]



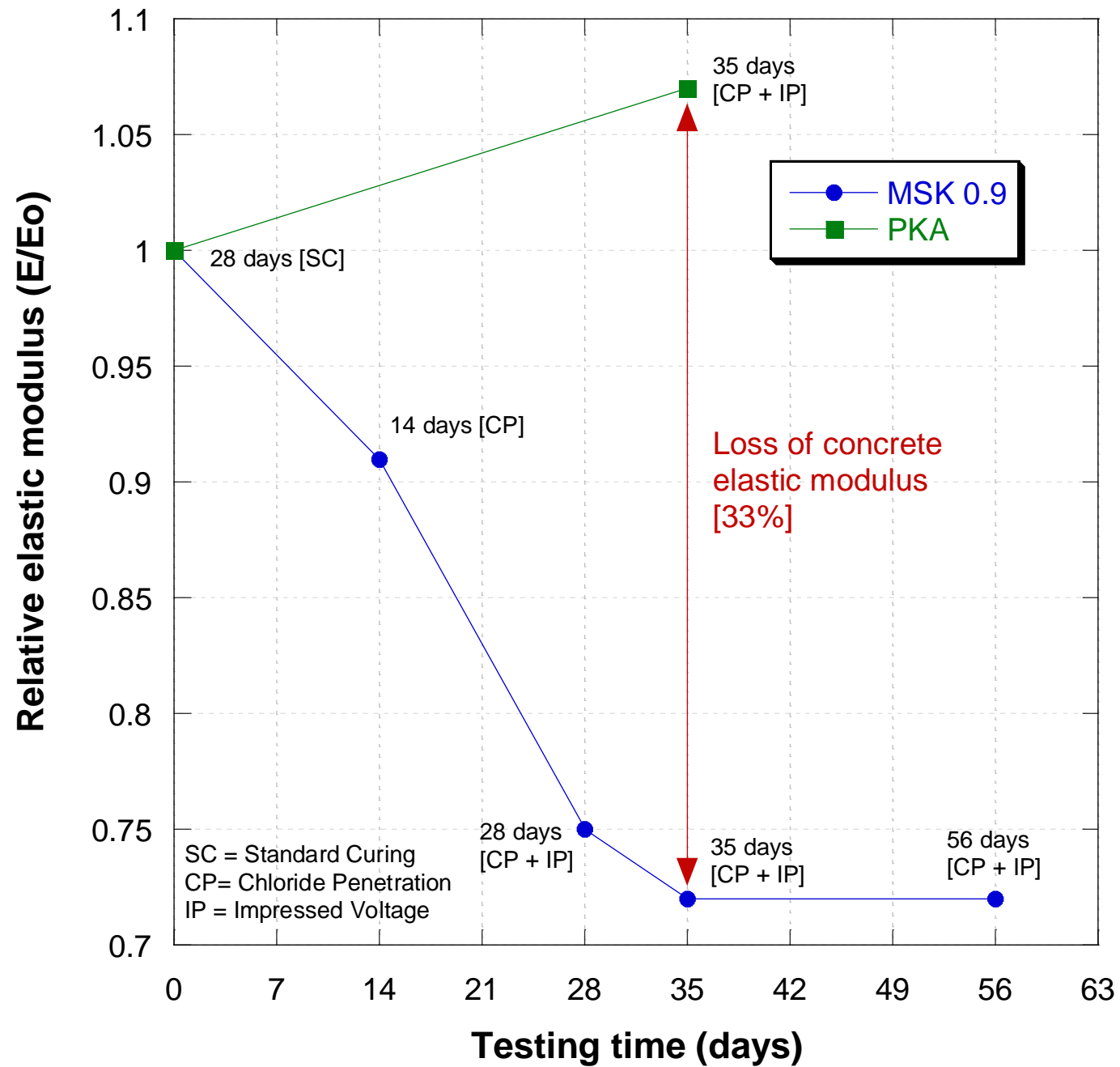
M  
E



Testing Time (days)  
[Chloride impregnation (14d) + Impressed Voltage]

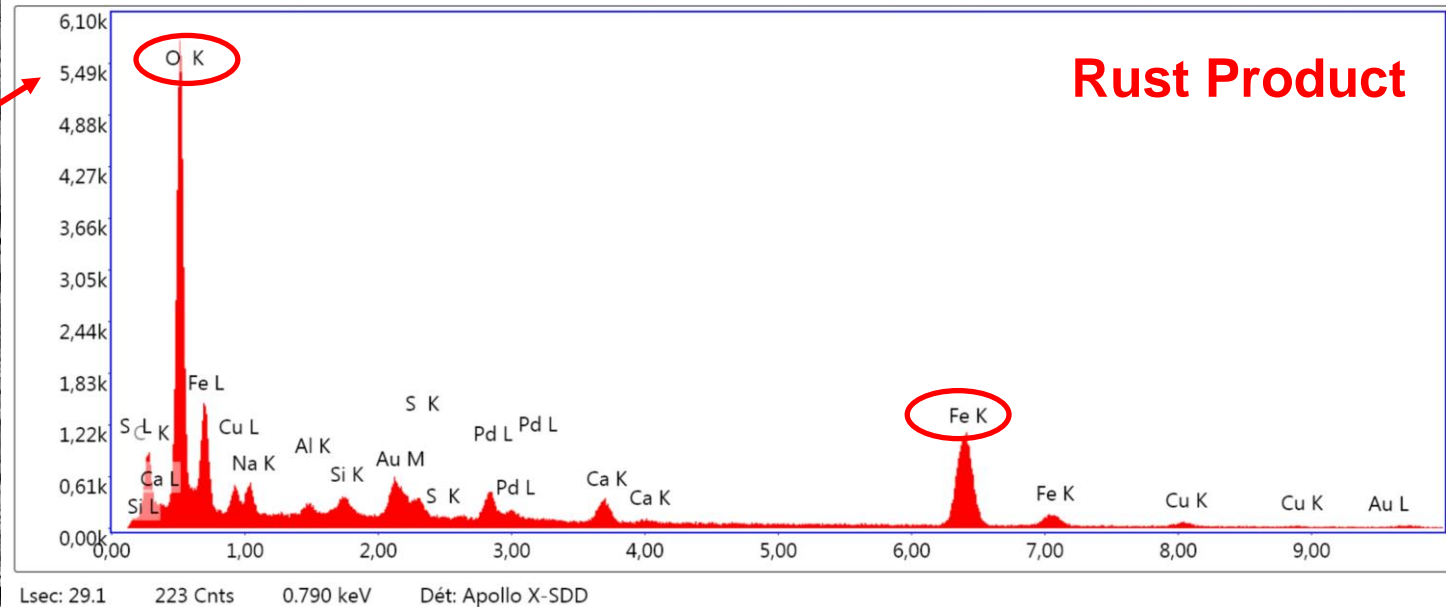
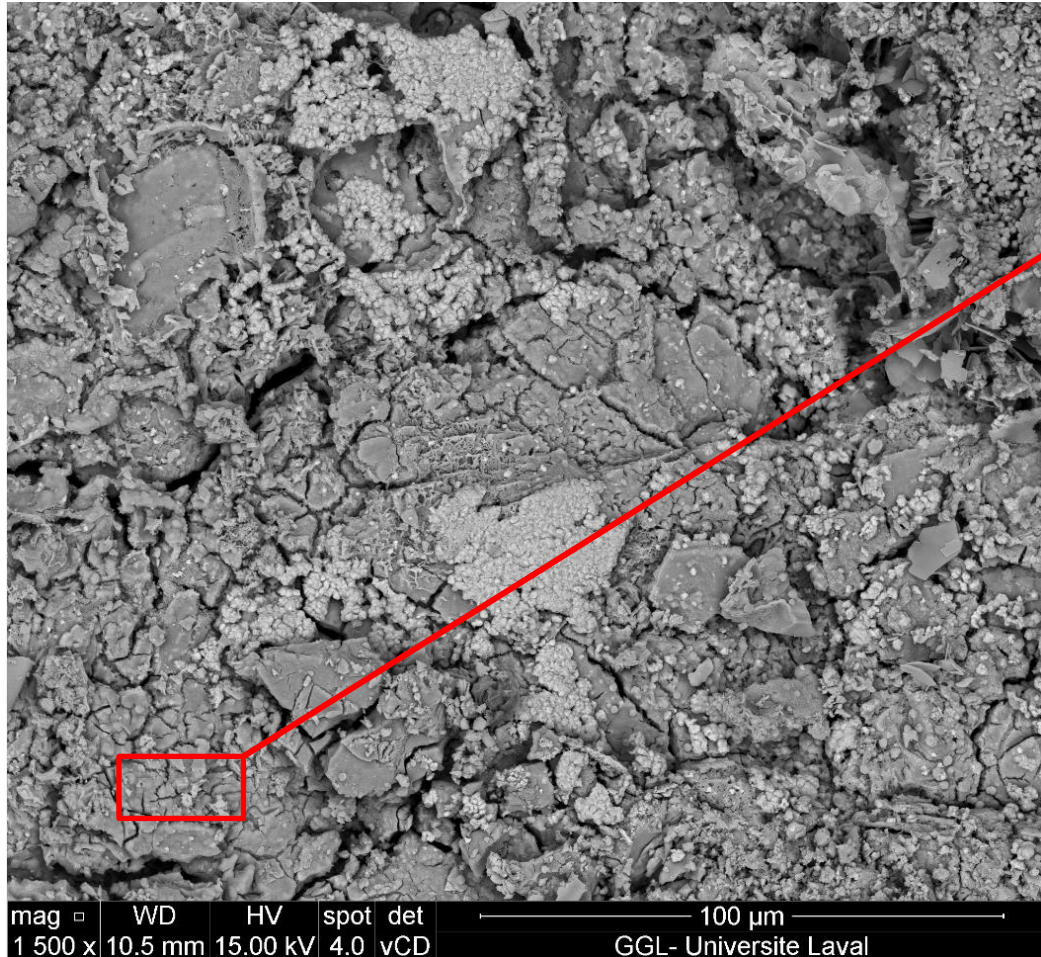


# Modulus of Elasticity





# Scanning Electron Microscopy / EDS

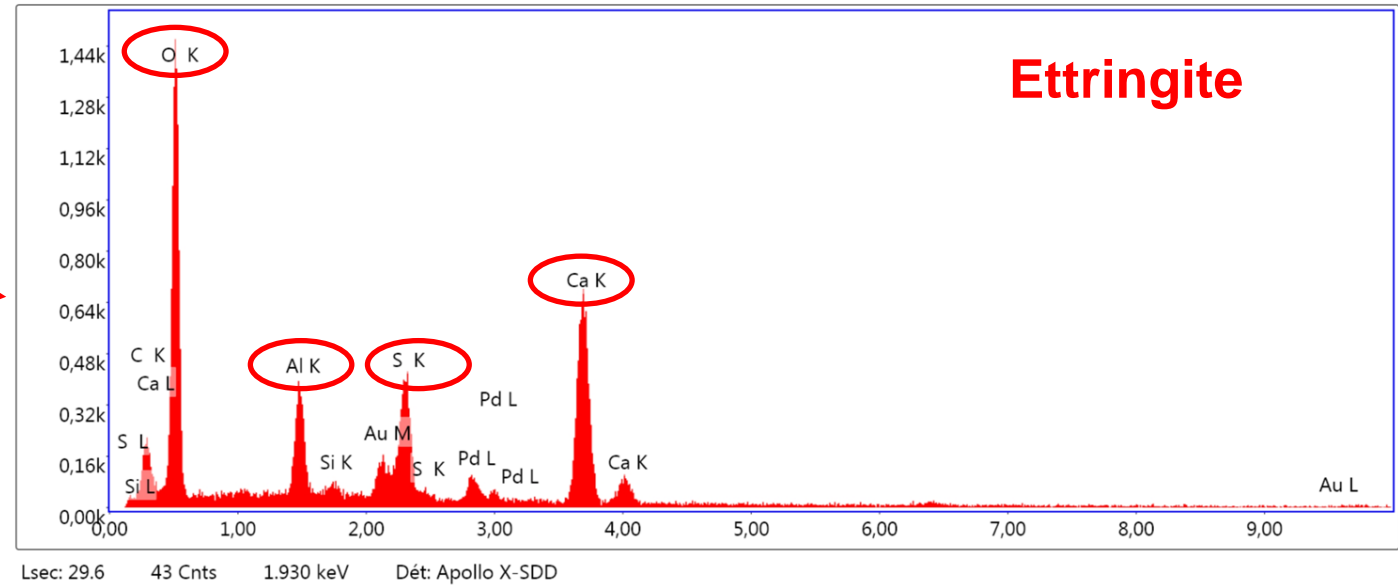
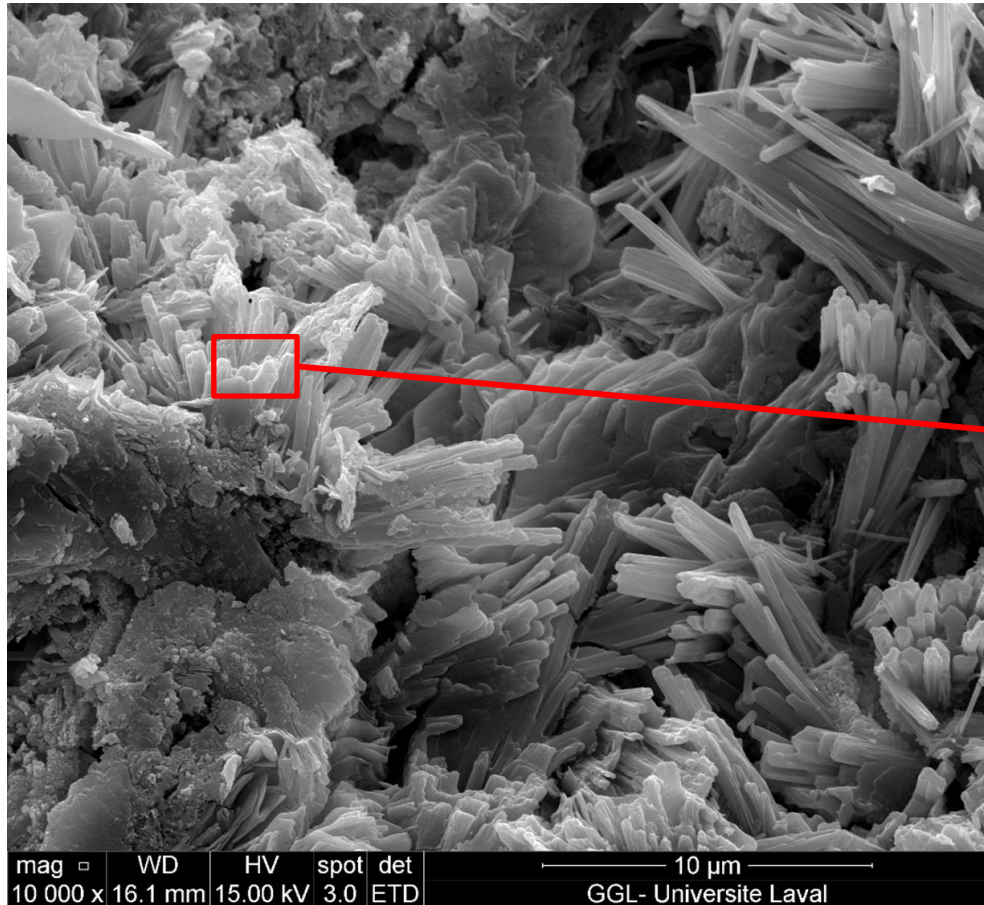


mag 1 500 x    WD 10.5 mm    HV 15.00 kV    spot 4.0    det vCD

100 µm  
GGL- Université Laval



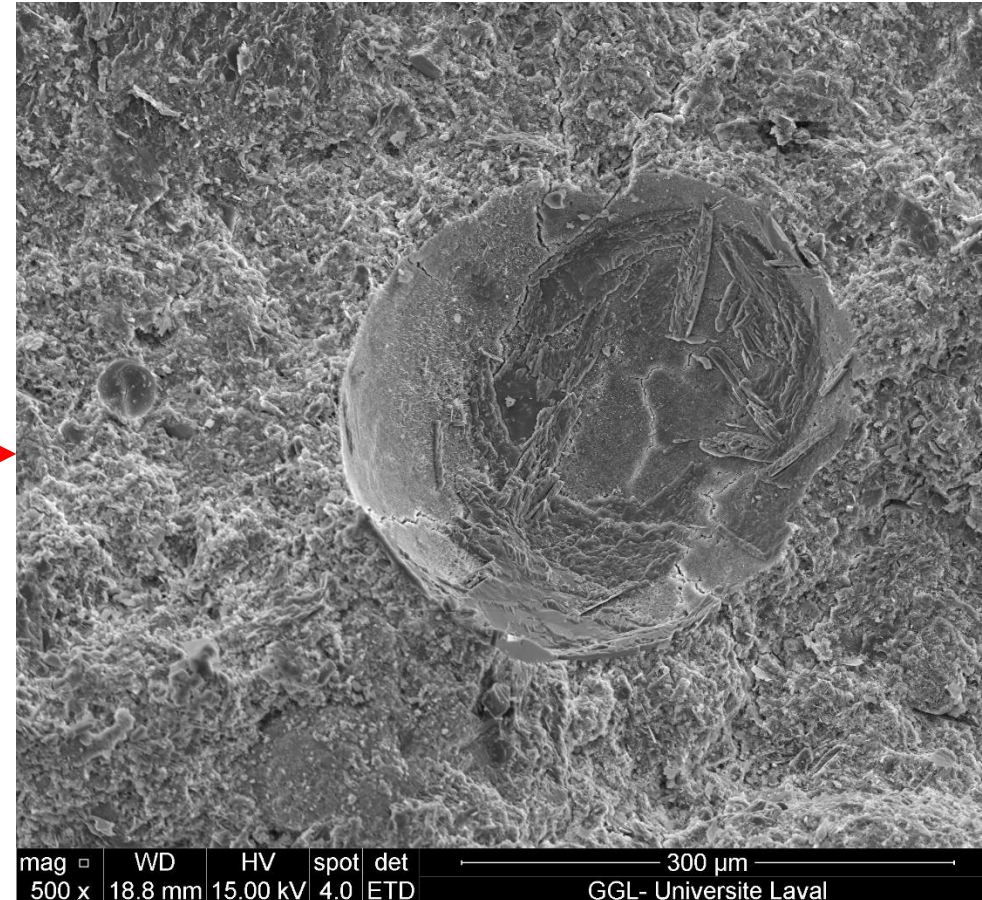
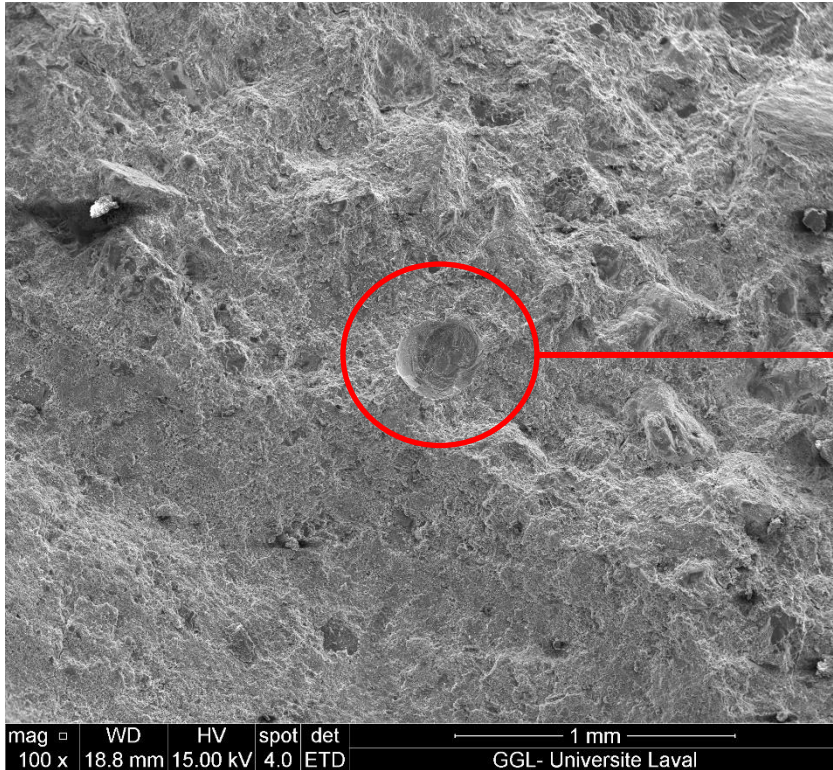
# Scanning Electron Microscopy / EDS





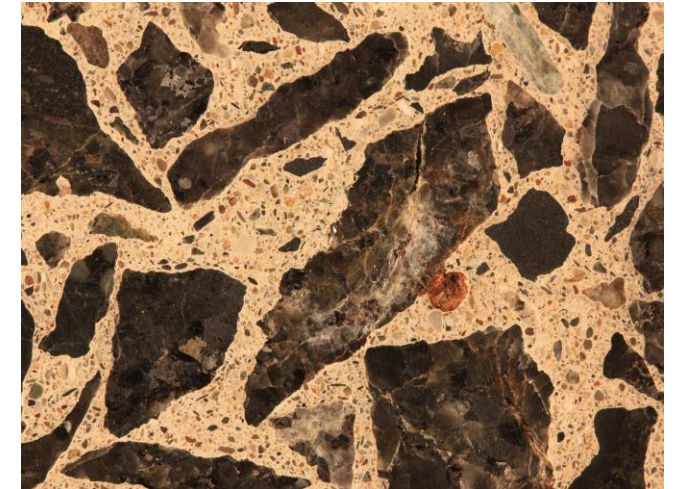
# Scanning Electron Microscopy

PKA [0.0]



# Conclusions and Perspectives

- Electrochemical treatment induces the iron sulfides oxidation process
- Chloride ions catalyze the oxidation reaction of pyrrhotite
- Secondary reaction products (ettringite and rust) are confirmed
- Modulus of Elasticity is the most promising evaluation parameter
- Testing additional aggregates (different sulfur contents)
- Develop full quality-control testing protocol



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**Contact: pierre-luc.fecteau@ggl.ulaval.ca**



# Thank you for your attention

## Acknowledgements to our team!





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