Durability of Geopolymer Mortars (Fly Ash/Slag-Based) in Sulfate Environments

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Never Stand Still

SUPPHATUCH UKRITNUKUN

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  - Elemental Analysis of Test Solution
  - pH of the solution
  - Linear Expansion

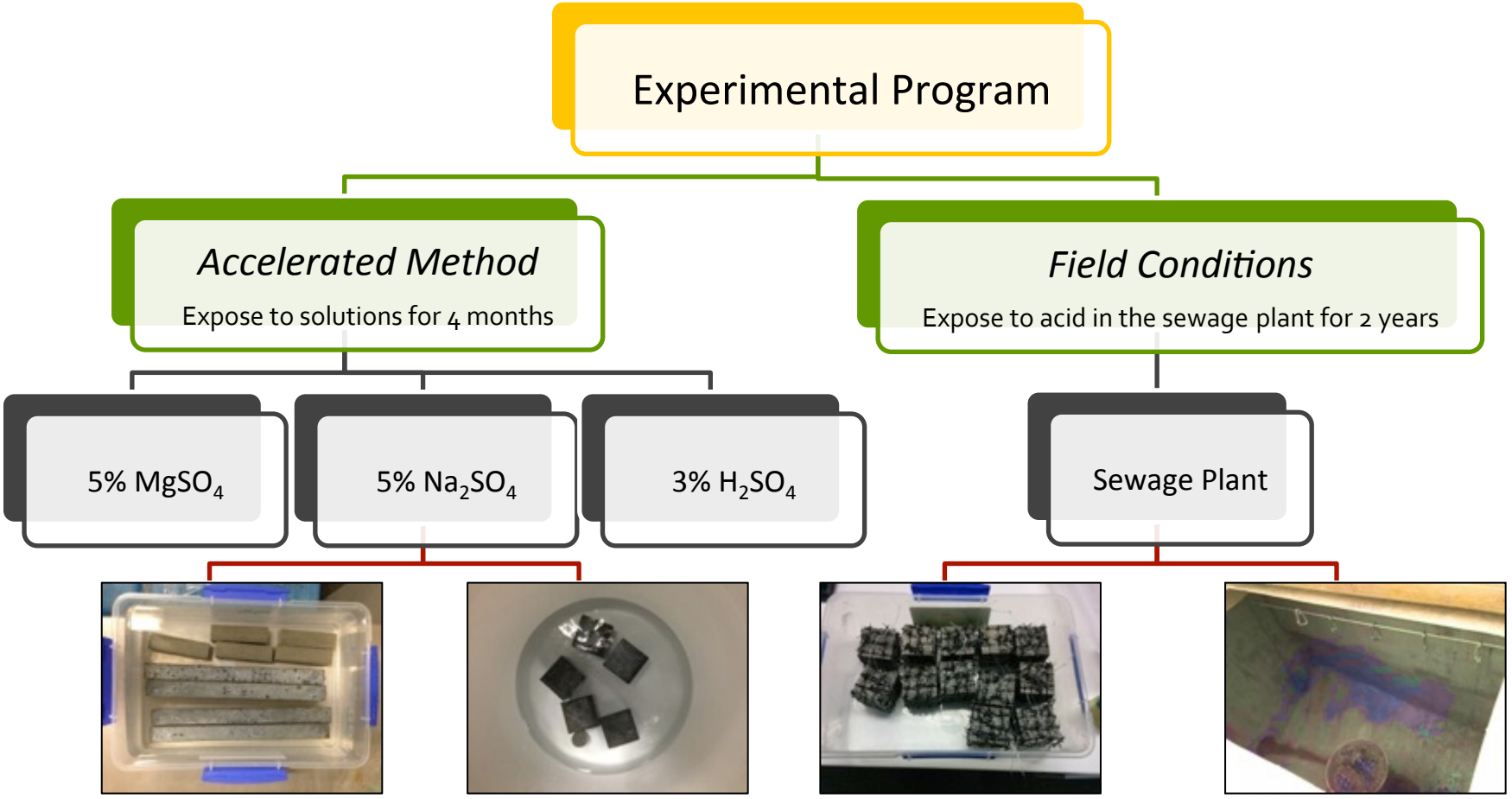
Aim of Research Work

- To investigate the deterioration mechanisms of geopolymer mortars (fly ash/slag-based) in sulfate solution ($MgSO_4$, Na_2SO_4) and acid solutions (H_2SO_4)

- *Issues*
  - Limited understanding of the sulfate exposure in geopolymer systems
  - Verification of reliability of ASTM C1012 (Portland cements) for use in geopolymer systems

Sulfate Solutions	• pH measurement • Elemental Analysis (ICP)
Paste / Mortar	• Microstructural Analysis (SEM) • Mineralogical Analysis (XRD) • Compressive Strength Measurements • Expansion Measurements

Experimental Program



Accelerated Method in laboratory

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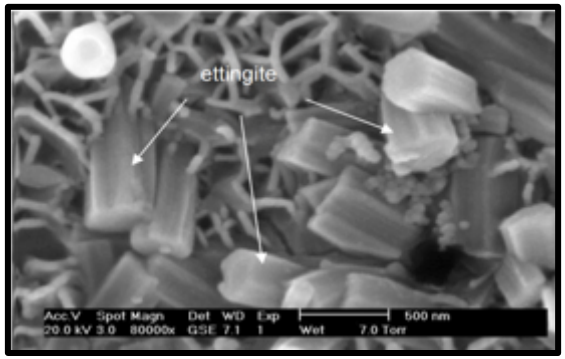
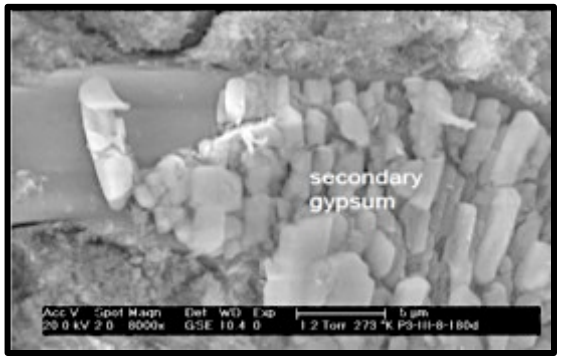
Mechanism of Sulfate Attack in OPC

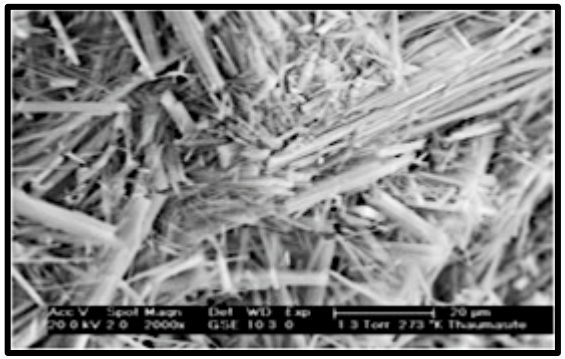
- **Sources: Aggressive soils ($MgSO_4$, Na_2SO_4), sewage plant (H_2SO_4)**
- **Most Susceptible Phases**
  - CSH, CH, and C_3A (Ca-rich phases)
  - Forms ettringite, gypsum, and/or thaumasite
  - Resulting expansion causes spalling and cracking of concrete



Ettringite
$3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 32H_2O$

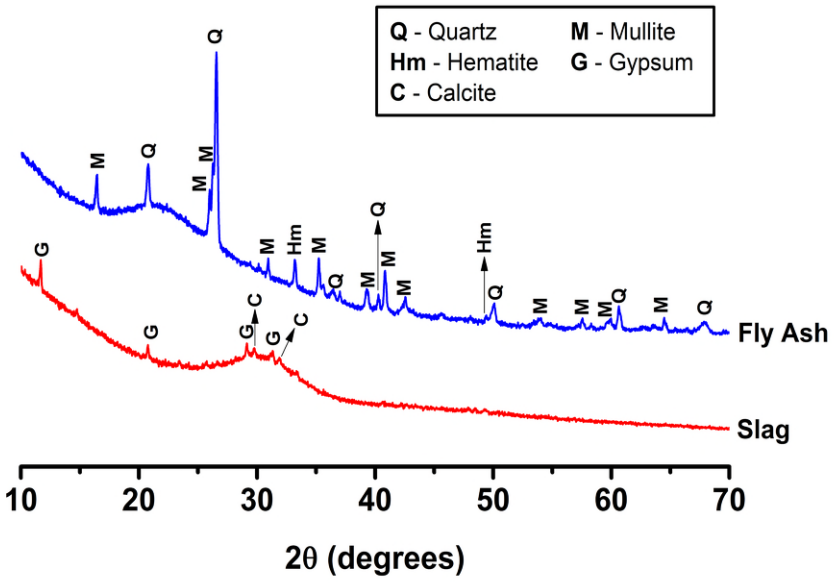


Gypsum
$CaSO_4 \cdot 2H_2O$



Thaumasite
$CaSiO_3 \cdot CaCO_3 \cdot CaSO_4 \cdot 15H_2O$

Geopolymer Fabrication Methodology
Raw Materials



Compound	Fly Ash (wt%)	Slag (wt%)
SiO_2	66.1	33.6
Al_2O_3	22.4	13.8
CaO	1.7	41.5
MgO	0.7	5.6
LOI	1.3	0.1

Compound	Grade D Sodium silicate (wt%)
SiO_2	29.4
Na_2O	14.7
H_2O	55.9

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Geopolymer Mortar Fabrication

Premix in Hobart Mixer

Fly Ash (F)

SSD Sand*

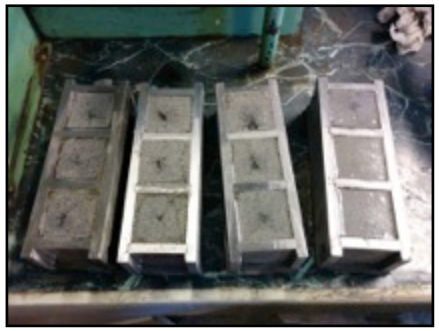
Sand/ (FA+Slag) = 2

12 M NaOH : Na_2SiO_3

+ water

Slag

Cast into moulds



50×50×50 mm^3 cubes

Compression Test

25×25×285 mm^3 bars

Expansion Test

Vibrate for 5 mins

Compressive Strength

Note:

*SSD = Saturated surface dry

All the ratios are mass ratio

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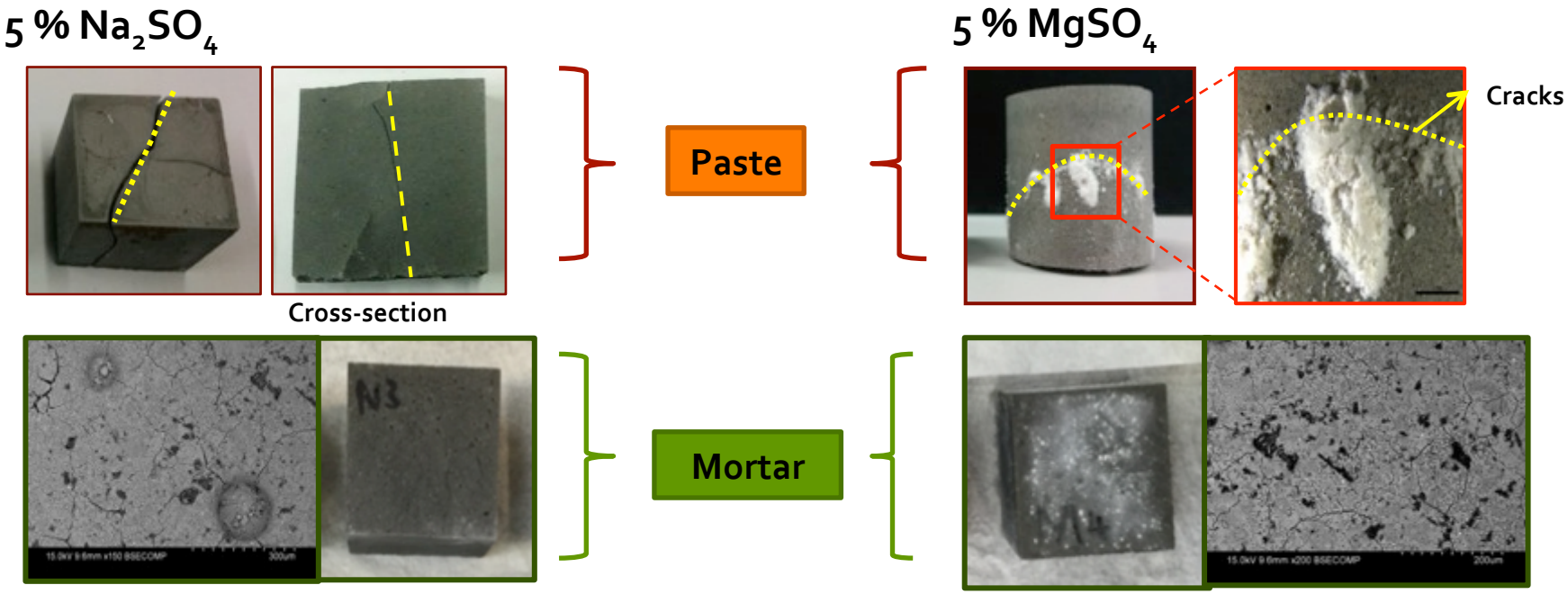
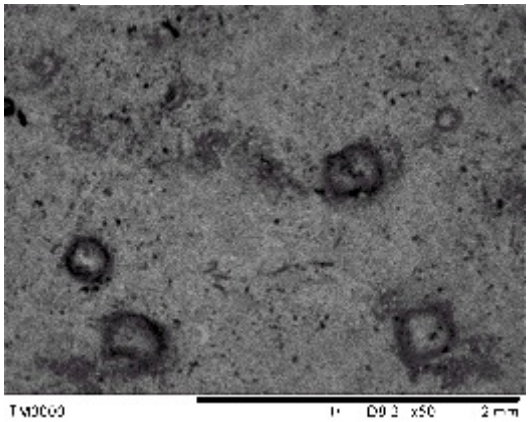
Physical Appearance - Geopolymers

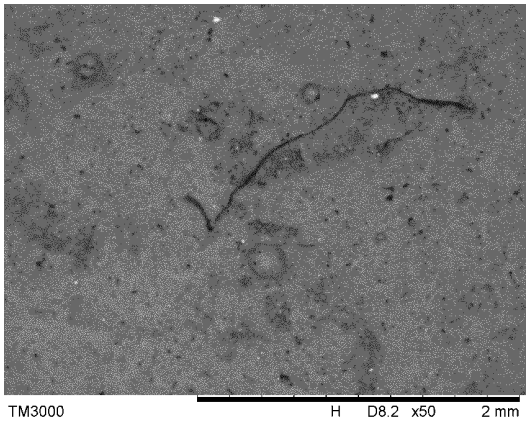


- Pastes cracked in halves within 1 week of exposure in both Na_2SO_4 and $MgSO_4$
- Mortar remain intact even though micro-cracks were present
- White crystalline precipitate (gypsum) was found in the samples exposed in $MgSO_4$

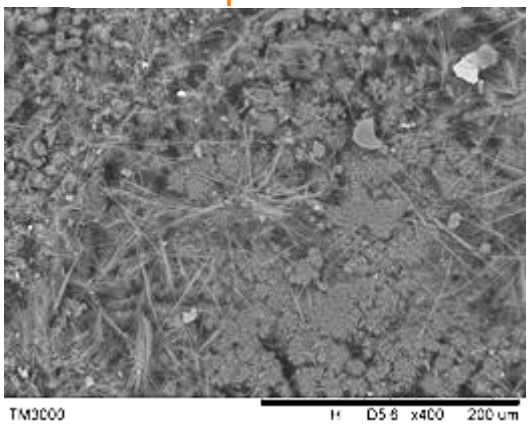
Microstructure

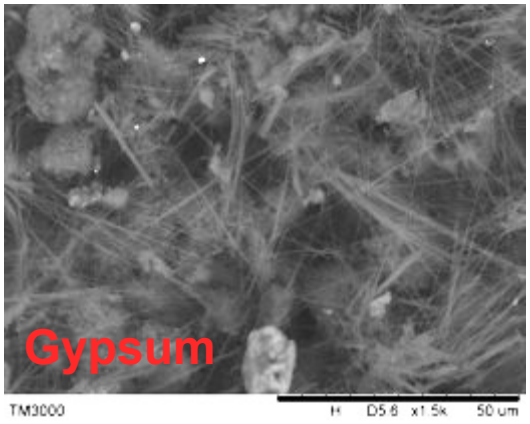
Before Exposure



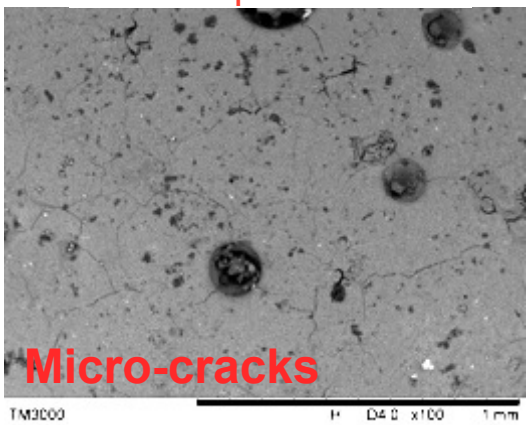


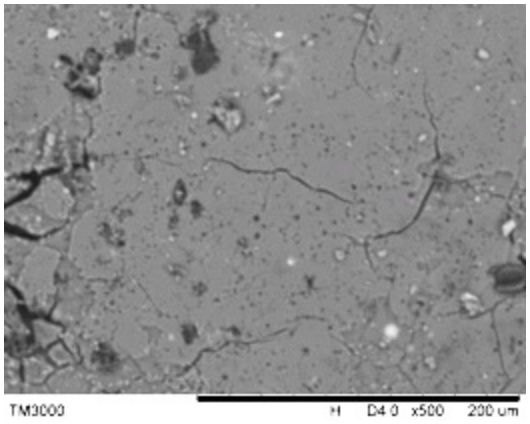
$MgSO_4$ (4 weeks)



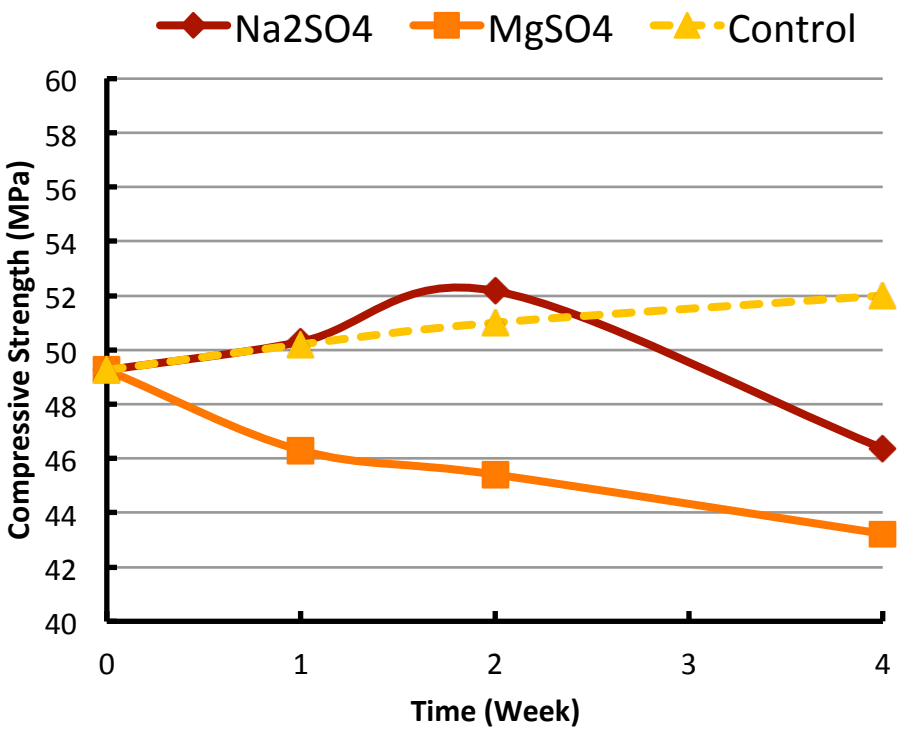


Na_2SO_4 (4 weeks)





Compressive Strengths



- **Control:** Gradual increase in strength from 49 to 52 MPa over four weeks
- **Na_2SO_4:** In first two weeks, strength increases to ~ 52 MPa;
  - Na might be reacting to enhance geopolymerisation which compensates for leaching losses;
- After four weeks, strength falls to 46 MPa:
  - Geopolymerisation ceases; leaching continues causing cracking
- **$MgSO_4$:** Compressive strength declines from first week of exposure to 44 MPa
  - Decalcification from the matrix

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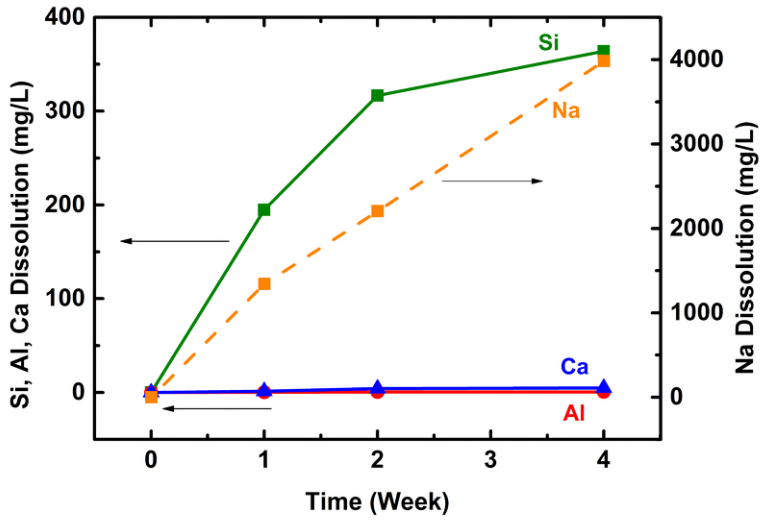
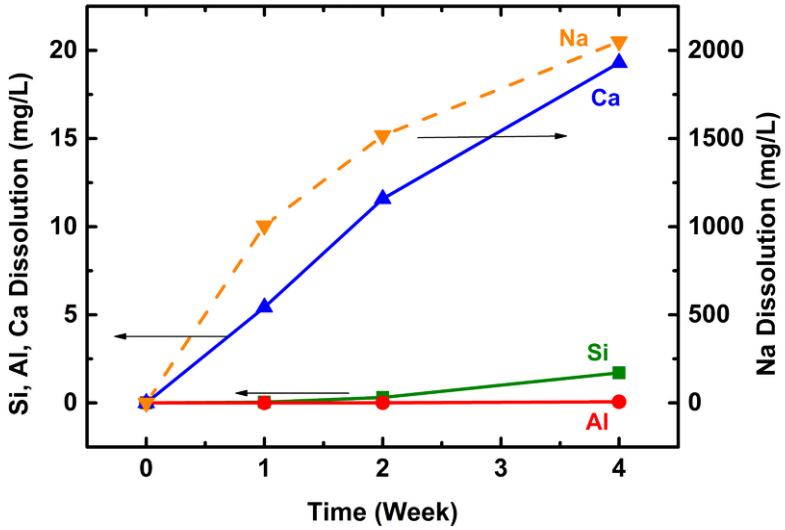
Elemental Analysis - ICP

5 % Na_2SO_4



- Significant leaching of Na and Si from alkaline activator
- Aluminosilicate raw materials remain undisturbed (based on FTIR analysis)
- Leaching of Ca and Al is not noticeable

5 % $MgSO_4$



- Mg decalcifies the Ca-rich phase i.e. generate cracks and leads to the formation of gypsum around the cracks
- Mg replaces Ca-rich phase
- Leaching of Si and Al is not significant

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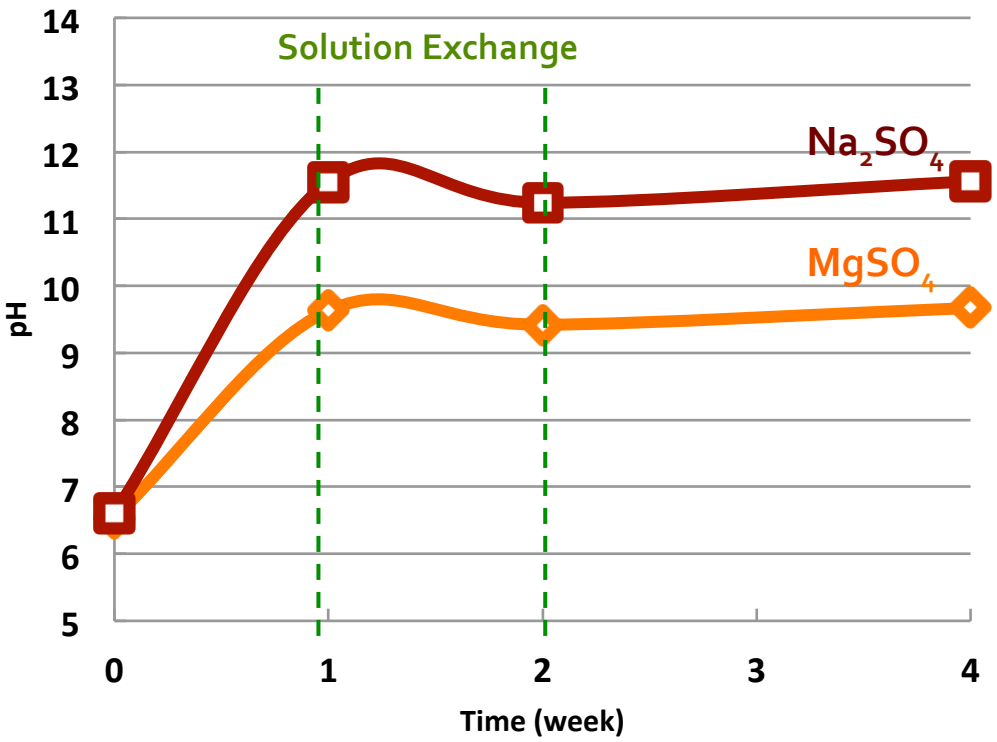
XRF Analysis of the mortar

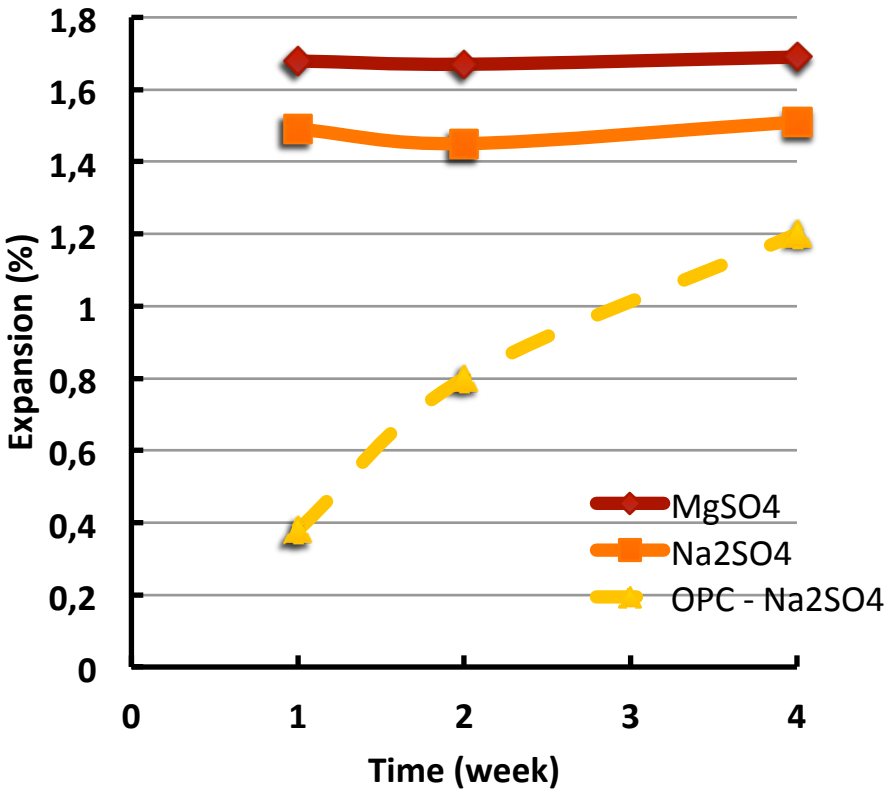
	Control	5% $MgSO_4$	5% Na_2SO_4
SiO_2	73.5	75.6	74.1
MgO	0.5	1.0	0.5
Na_2O	3.1	2.3	2.7
CaO	5.0	5.0	5.1

pH of the solutions



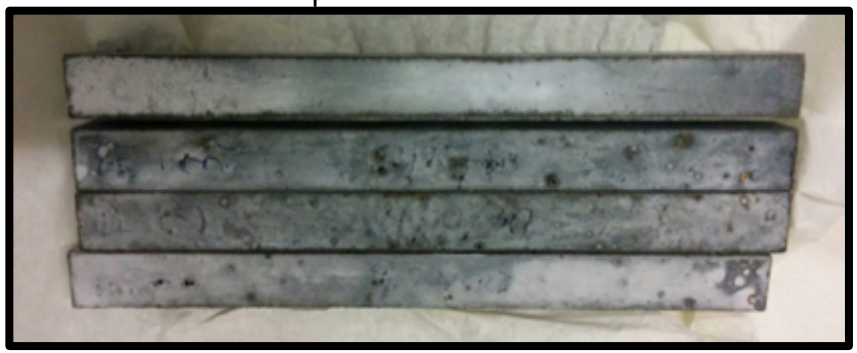
- Increased leaching of Si and Na in Na_2SO_4 results in rapid increase in pH
- Significant increase in pH in the first week in both test media
- After the solution exchange was done at the end of Week 1, there was a slight drop in pH
- After Week 4, the solution appears to have reached equilibrium and no further increase in pH was observed

Expansion Measurement



5 wt% Na_2SO_4 (4 weeks)

5 wt% $MgSO_4$ (4 weeks)



Conclusion

Fabrication Method

- High temperature curing: leads to high porosity and voids mortar due to evaporation of unbounded water
- Correct mixing step need to be implemented in order to successfully fabricate dense geopolymer mortar

Sulfate studies

- Presence of Mg decalcify the Ca-rich gel leads to the precipitation of gypsum
- $MgSO_4$ is more severe than Na_2SO_4 : reduction of compressive strength
- No visible cracks but the presence of micro cracks leads to the degradation of strength