Alkali Leaching Properties of Waste Glass-Based Geopolymers

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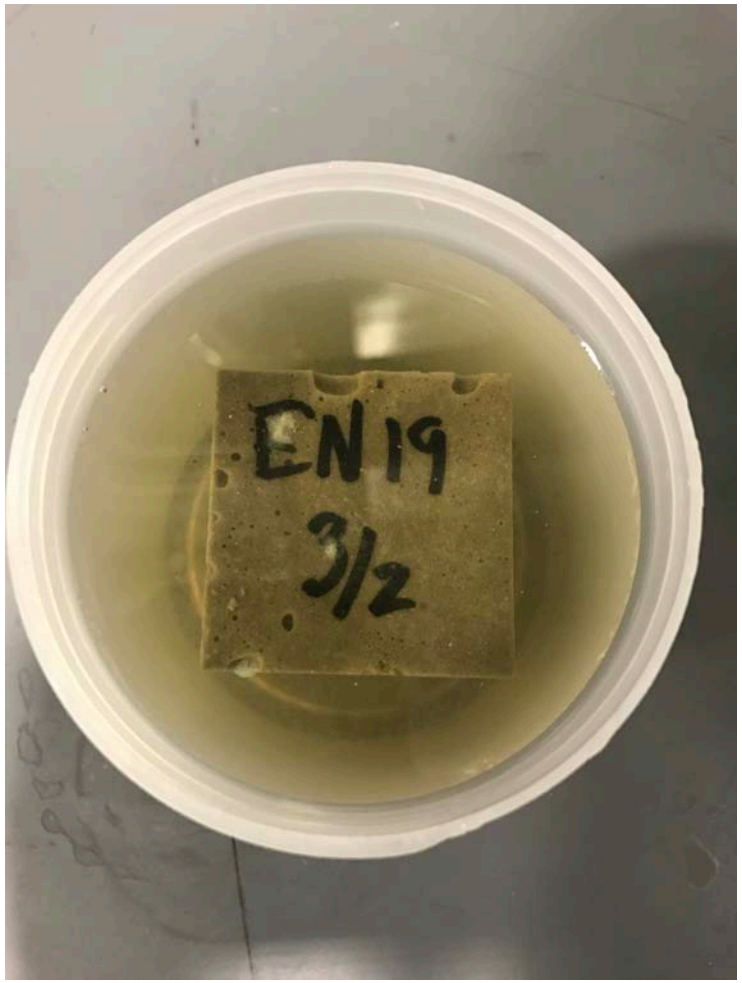
UMD University of Minnesota Duluth
Driven to Discover

Overview

- Project Motivation
- Goal and Research Questions
- Geopolymer Background
  - Glass Motivation
- Experimental Plan
  - Materials
  - Methods
- Results
- Observations and Next Steps



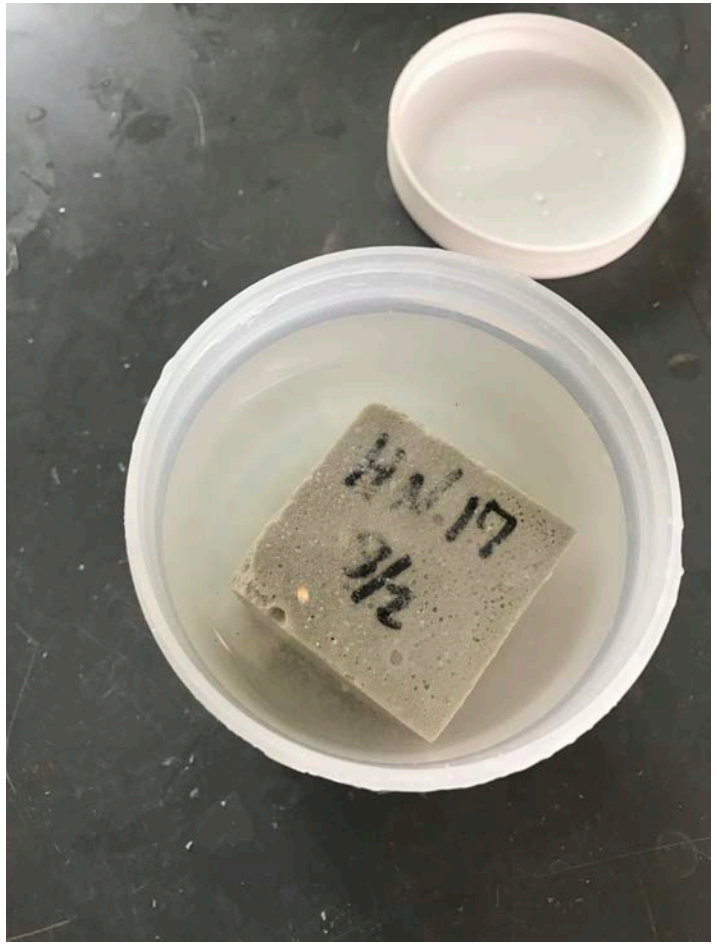
About Myself

- From Spring Valley, WI
- Bachelor's Degree from UMD in Civil Engineering
  - May 2016
- Master's Degree from UMD in Civil Engineering
  - December 2017?
- Presented research at American Concrete Institute (ACI) Convention in Detroit, MI
- Presenting at Transportation Research Board (TRB) Summer Workshop in Duluth, MN
- Will present research at ACI Convention in Anaheim, CA

[2]

Project Motivation

- Production of portland cement (PC) accounts for 6-7% of global anthropogenic carbon footprint [3]
- Looks like and performs similar to PC concrete
- Aluminosilicate source is generally an industry by-product or waste material
- Can posses unique properties
  - High heat resistance
  - Sulfate resistance
  - Chloride Resistance



Goal

Investigate the effect of chemical composition on the leaching properties of glass-based geopolymer mortars in order to improve the water stability

[1]

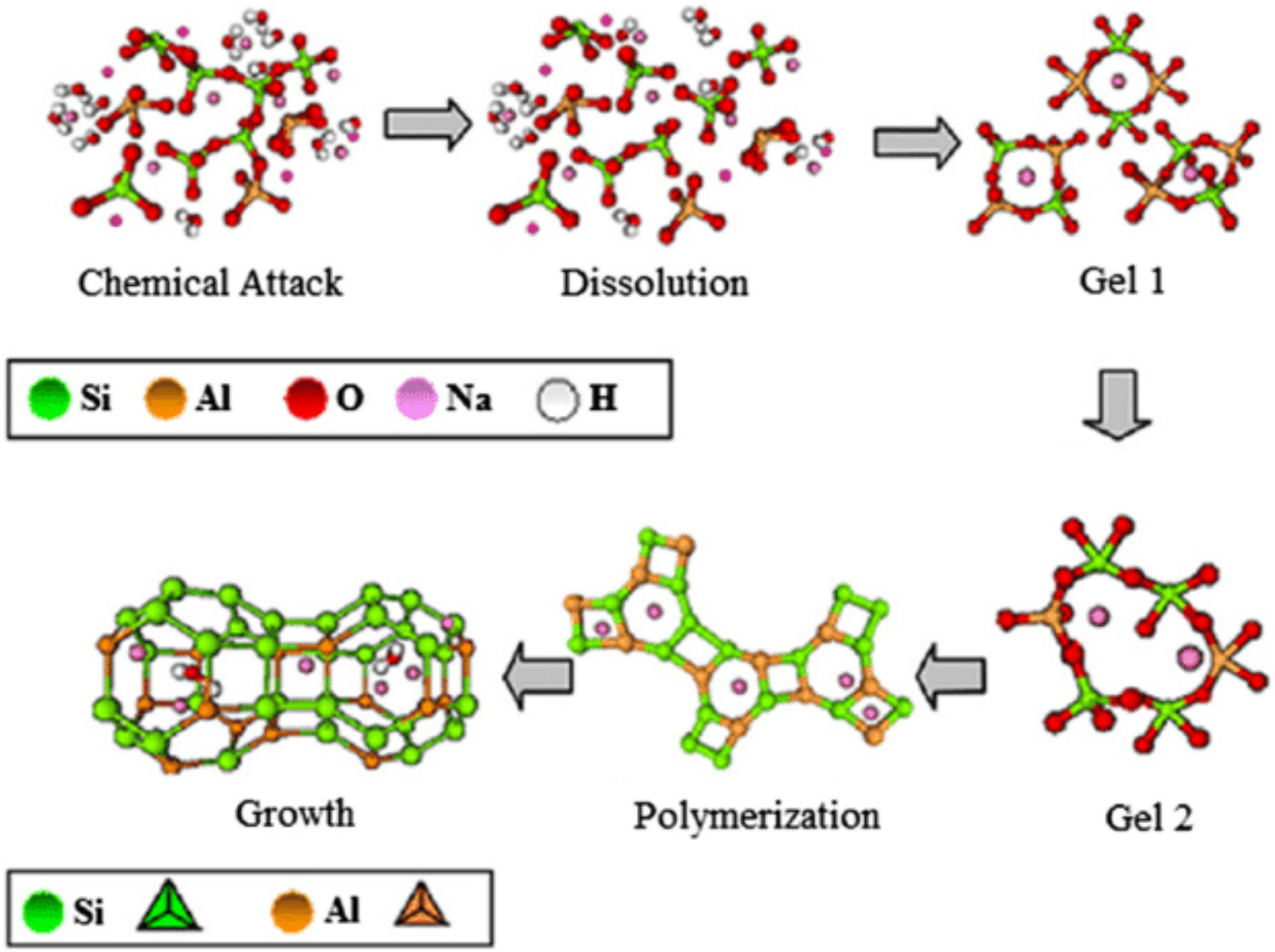
Research Questions

- What are the alkali leaching properties of glass-based geopolymers?
- What is the correlation between composition, stoichiometry, and leaching properties of glass-based geopolymers?
- Does lowering the alkali concentration or using a blend of activators improve the mechanical properties of glass-based geopolymers?
- Does the addition of an alumina source enhance the mechanical performance?

Geopolymers

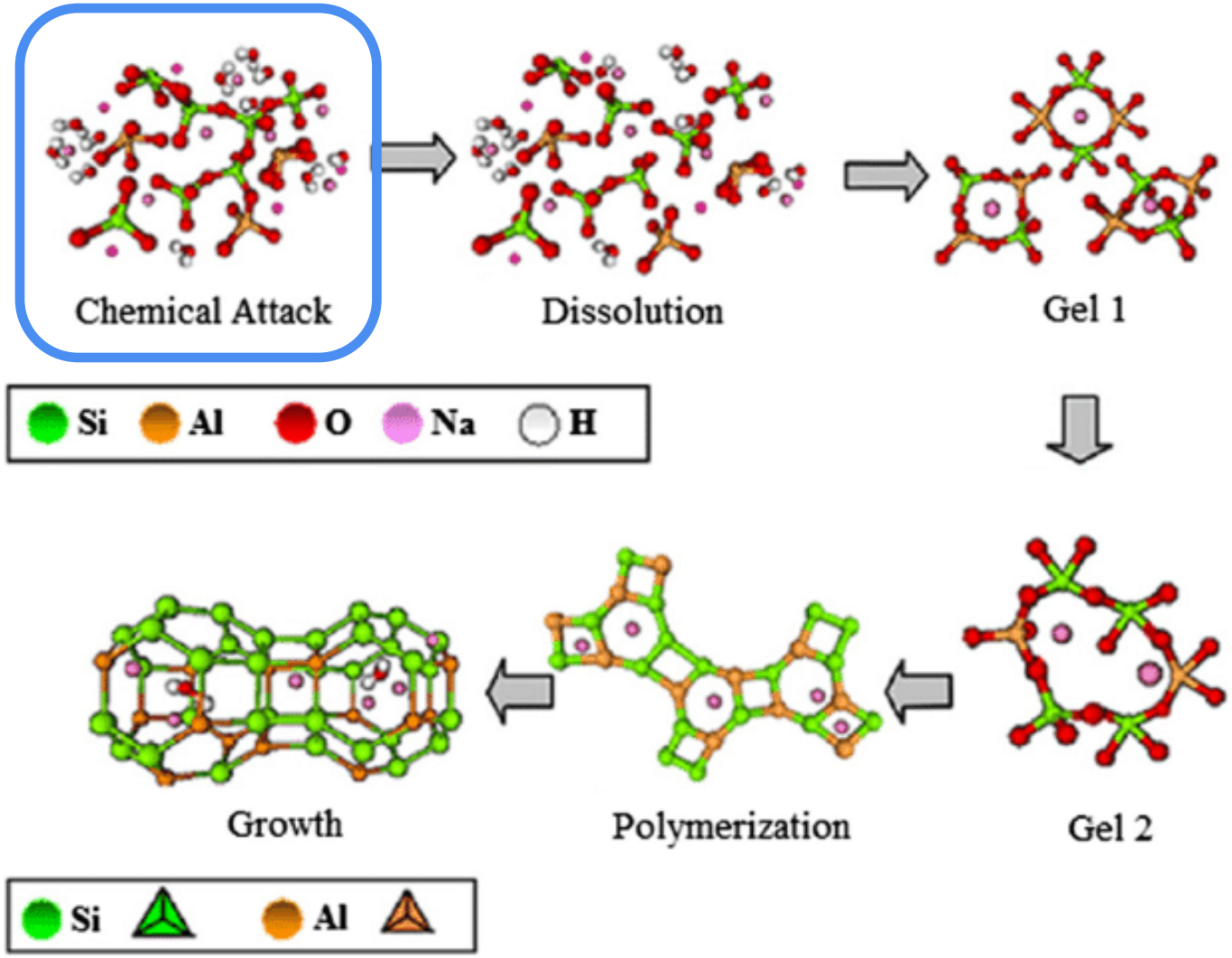
An inorganic polymer binder created from the alkali activation of a finely divided aluminosilicate source

Geopolymerization



[3]

Chemical Attack



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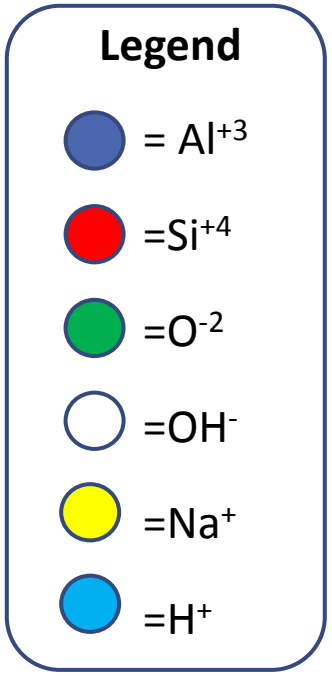
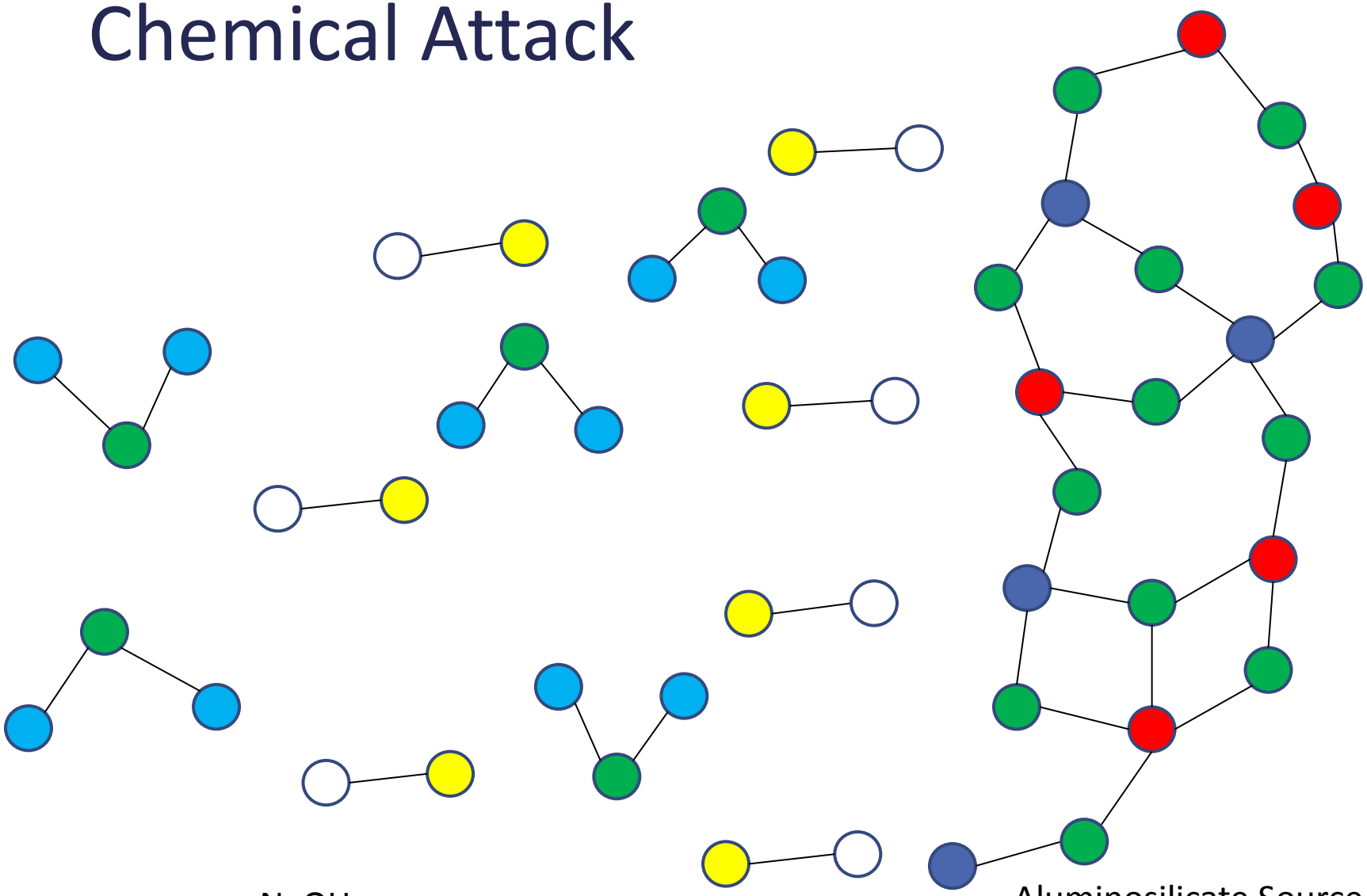
Chemical Attack

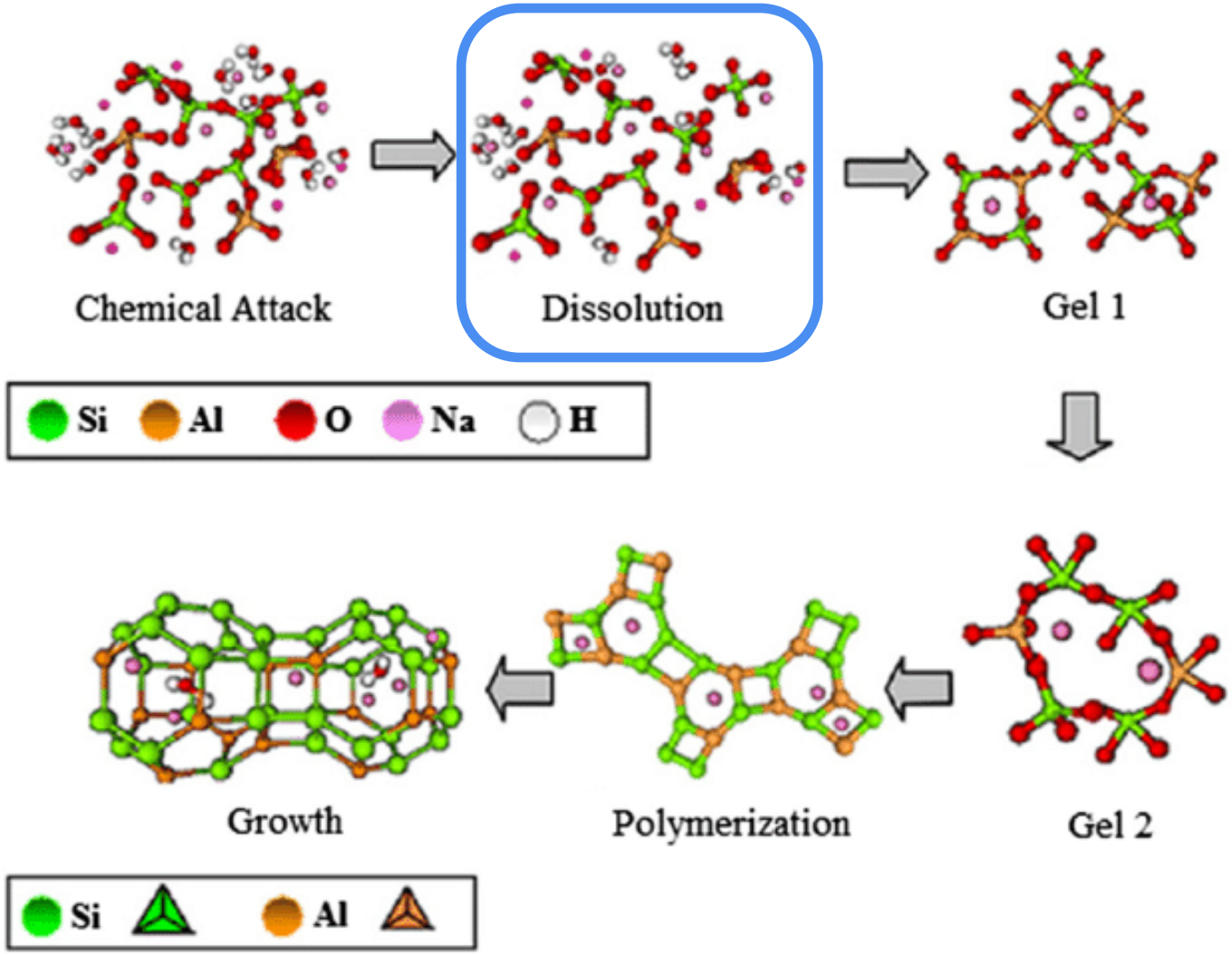




NaOH

Aluminosilicate Source

Dissolution



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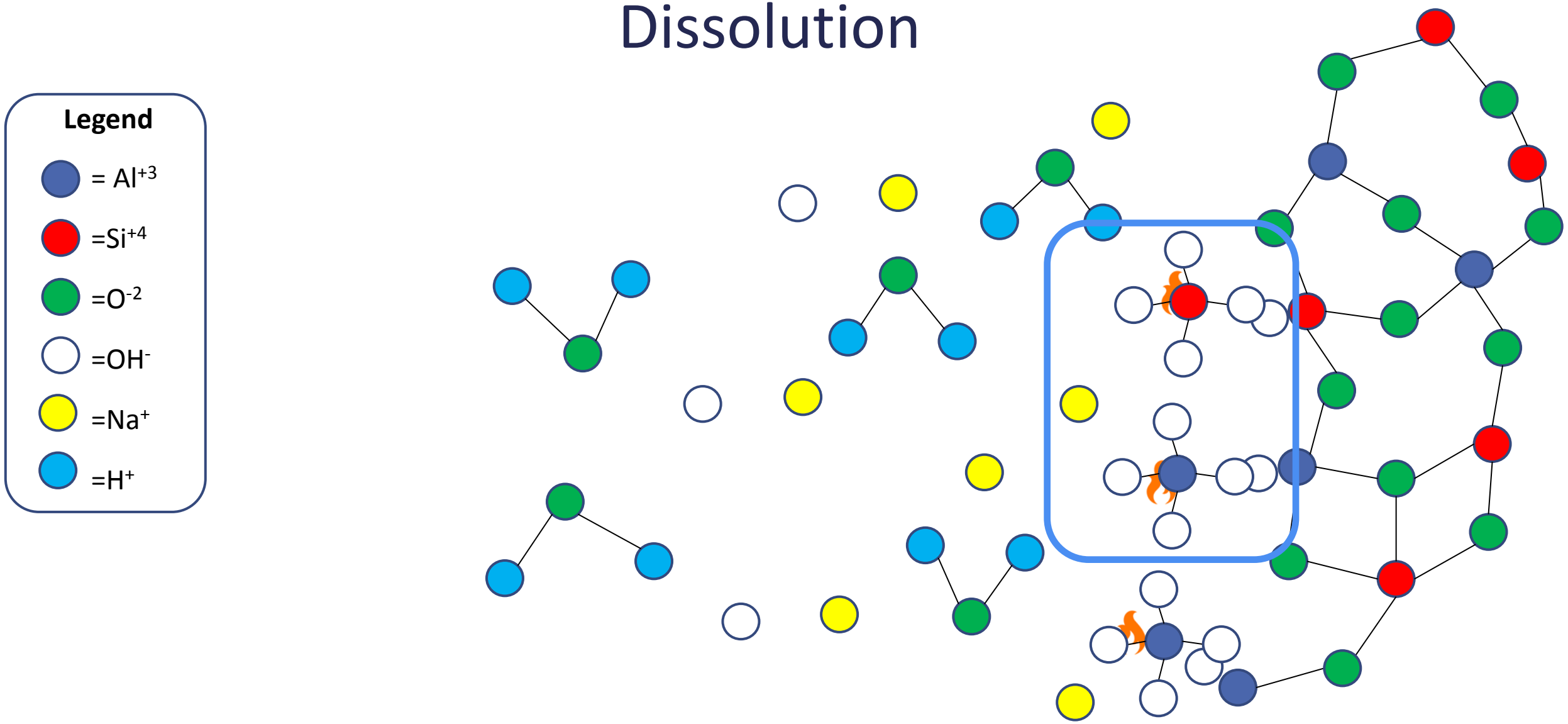
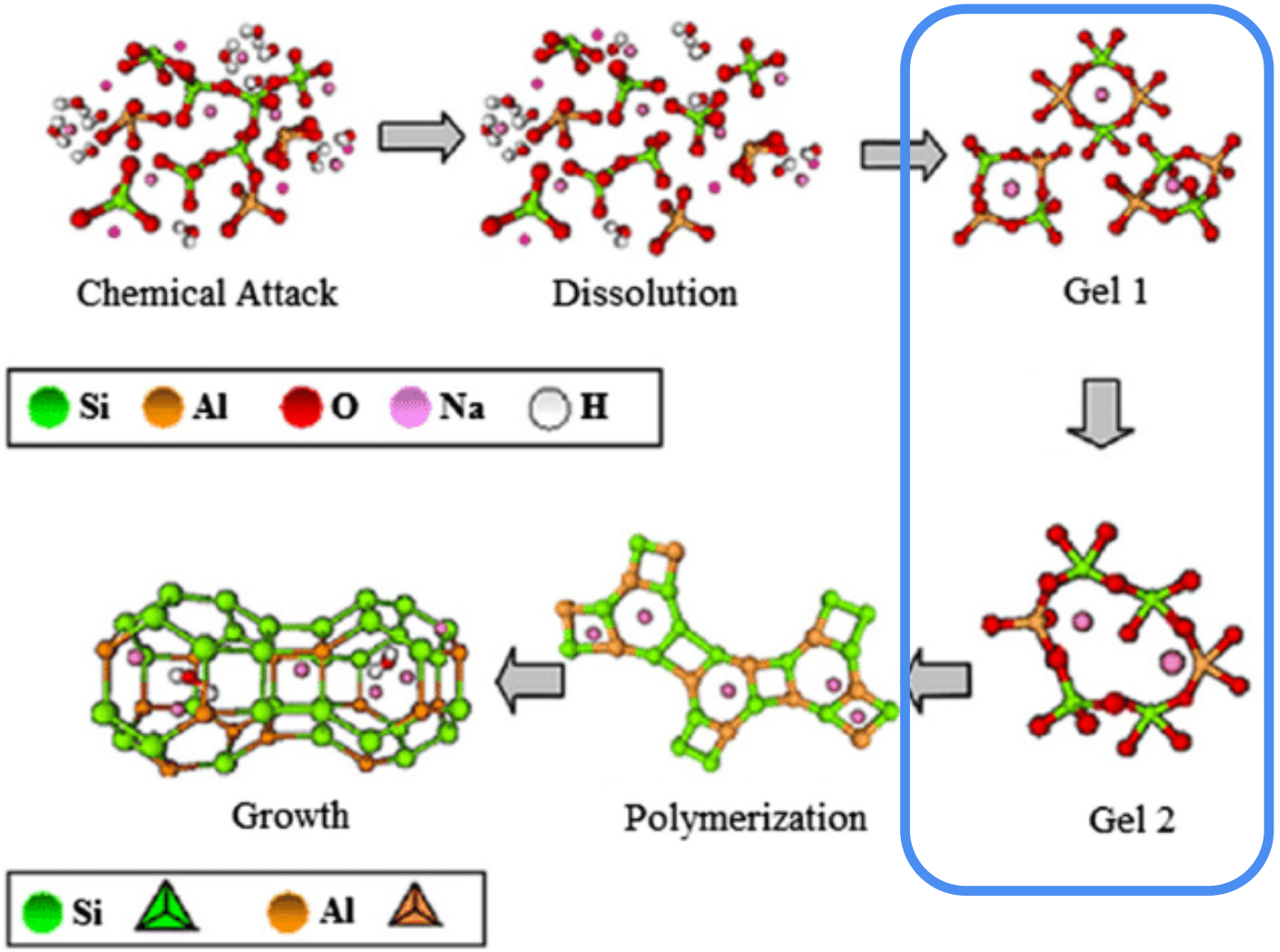
Dissolution

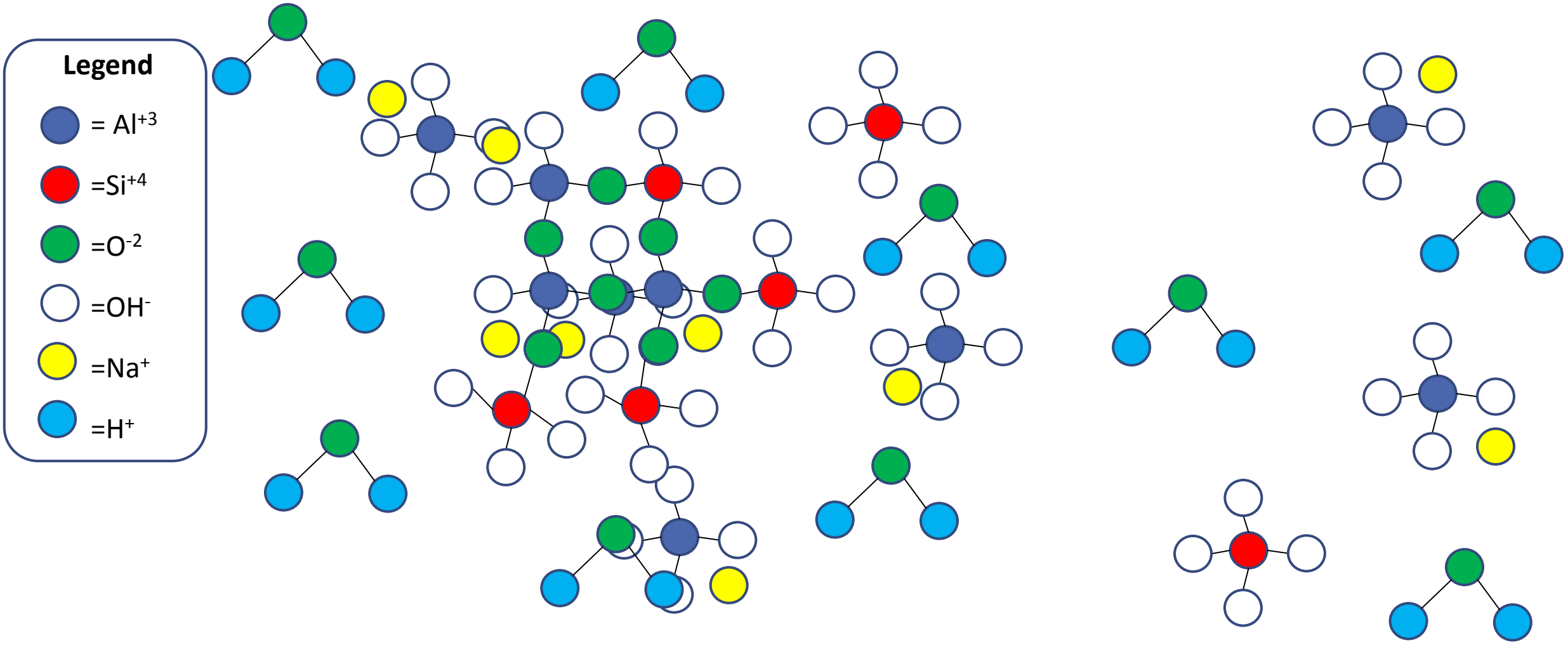


Gelation

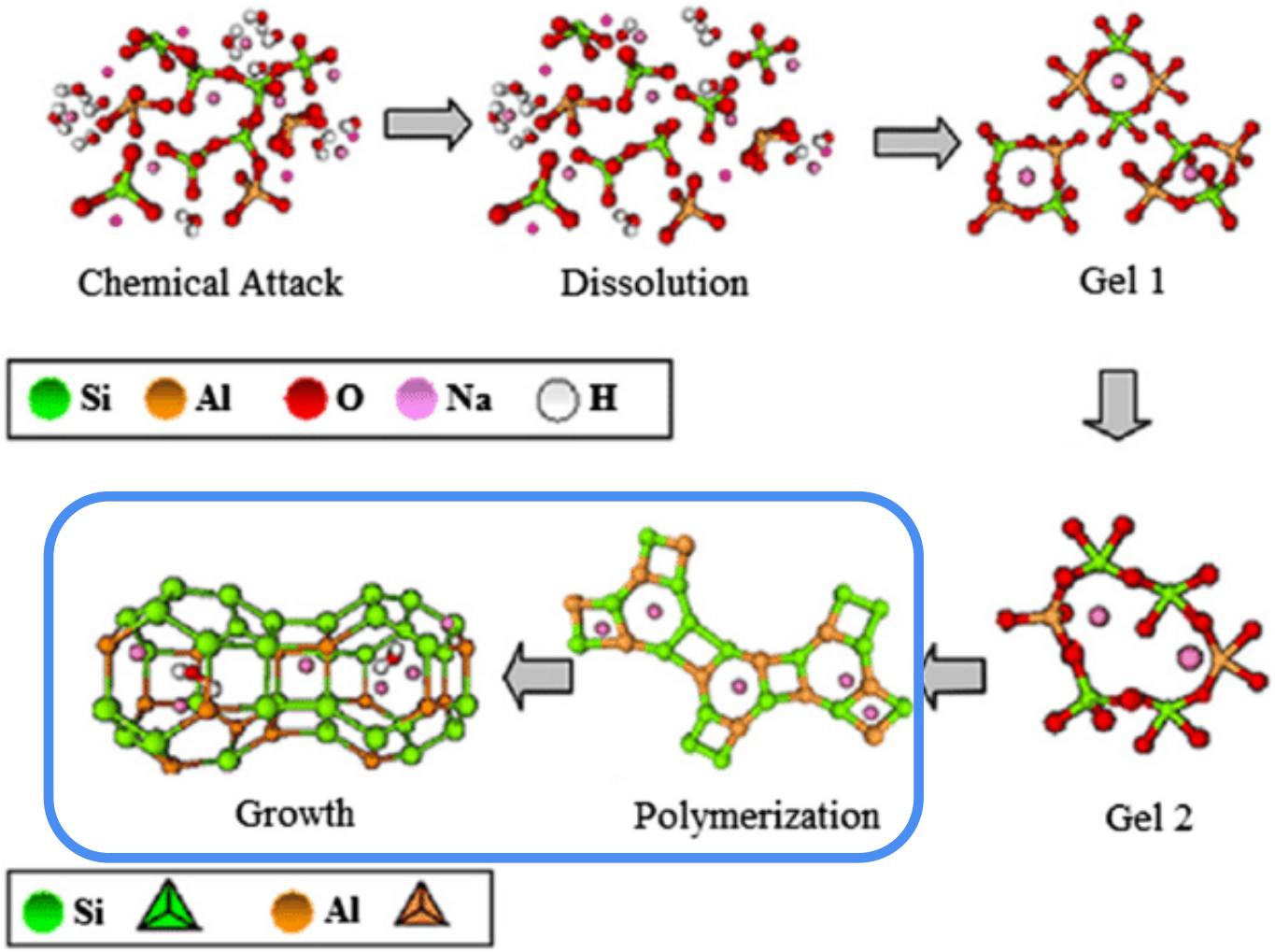

Chemical Attack
Dissolution
Gel 1
Si
Al
O
Na
H
Gel 2
Polymerization
Growth
Si
Al

[3]

Gelation



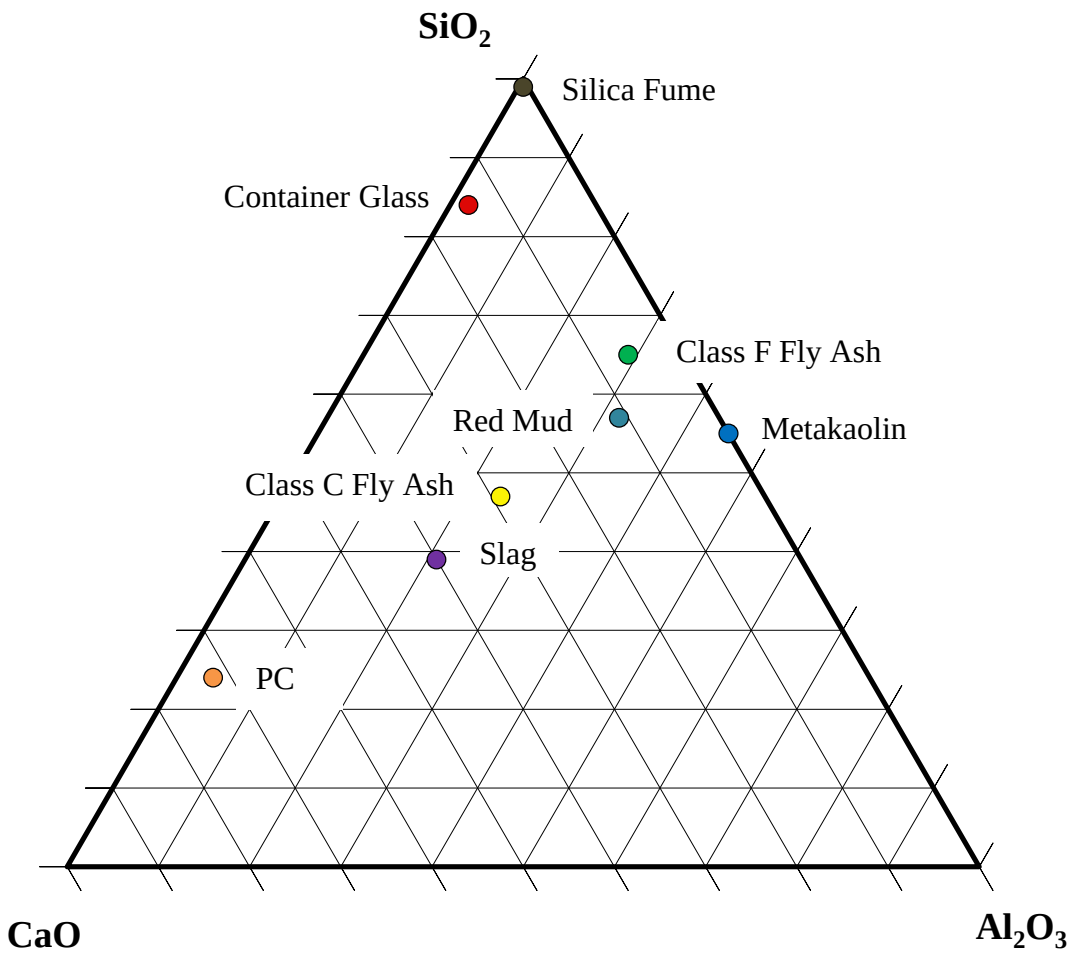
Polymerization



[3]

Source Materials

- Materials used in geopolymers
  - Fly Ash (Class F and Class C)
  - Metakaolin
  - Slag
  - Glass
- Composition is key



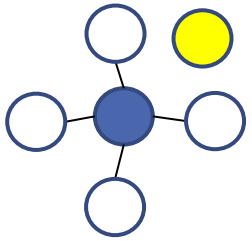
Activator

- Roles of activator:
  - Provides a medium for reactions to occur
  - High pH for rapid dissolution
  - Alkali cation balances charge of Al monomer
  - Silica in the solution can accelerate geopolymerization
- Common Activators:
  - Caustic alkalis ($NaOH$, $Ca(OH)_2$, or KOH)
  - Nonsilicate weak acid salts (Na_2CO_3)
  - Silicates (Na_2SiO_3) **[3]**

[4]

Stoichiometry

- M^+ → Either Na^+ or K^+
- Al → Al_2O_3
- Si → SiO_2
- $M^+/Al = 1$ ★
- Si/Al = 2-5



Researcher(s)	M^+/Al	Si/Al
Davidovits 1979	0.8-1.2 (~1.0)	3.5-4.5 (~4.0)
Rowles and O'Connor 2003	1.3	3.0
Duxson et al. 2005; Fernández-Jiménez et al. 2006	1.0	1.8-2.0
Duxson et al. 2007	1.0	1.0-5.0

[5-9]

Waste Glass

- Rich in reactive silica
- Amorphous structure
- Recycled waste product
  - Municipal Solid Waste (MSW)

Product Category	Generation	Recycled		Landfilled
	(Thousand Tons)	(Thousand Tons)	(% of Generation)	(Thousand Tons)
Durable Goods	2,280	Negligible	Negligible	2,050
Containers and Packaging	9,200	2,990	32.5	6,110
Total Glass	11,480	2,990	26.0	8,390

[10]

Glass Types

- Soda-lime
  - Container
  - Plate
- Borosilicate
- Glass Fibers
- Television (Tube)

	Container	Plate	Borosilicate	Glass Fibers	Television
SiO_2 (% weight)	74.0	73.0	81.0	52.0-56.0	62.0
Al_2O_3 (% weight)	1.5	0.1	2.0	12.0-16.0	2.0
B_2O_3 (% weight)	0.0	0.0	13.0	5.0-10.0	0.0
CaO (% weight)	11.0	9.0	0.0	16.0-25.0	< 2.0
PbO (% weight)	0.0	0.0	0.0	0.0	0.0-2.0
Na_2O (% weight)	13.0	14.0	4.0	0.0-2.0	7.0
K_2O (% weight)	0.3	0.0	0.0	0.0	9.0
MgO (% weight)	0.2	4.0	0.0	0.0-5.0	< 1.0
Fe_2O_3 (% weight)	0.0	0.1	0.0	0.0-0.8	0.0

[11]

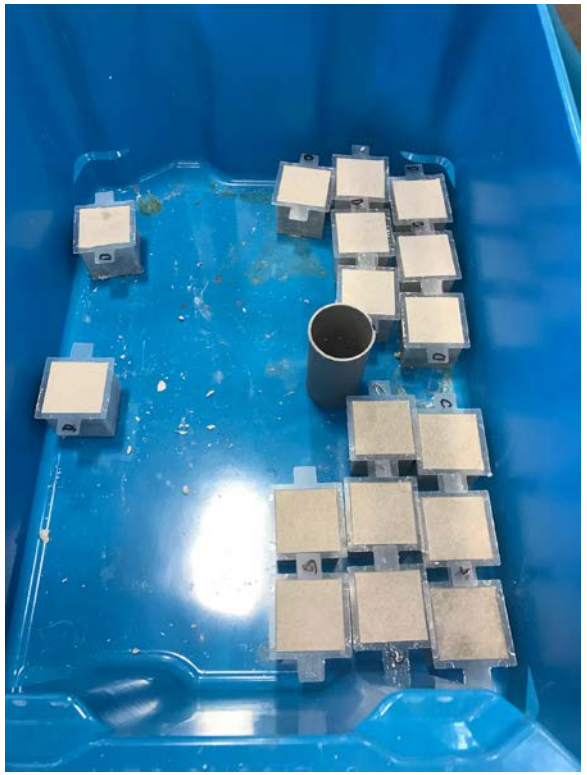
Experimental Plan

- Phase I – 16 mortar mixtures each using a different glass and activated with 10M NaOH were designed to investigate the effects of stoichiometry on the leaching and mechanical properties.

- Phase II – Stoichiometric design-based mixtures using glass in combination with other varying aluminosilicate sources to provide supplementary Al and various chemical activators were designed to apply the concepts learned in Phase I.

Mixture Parameters

- Mixture Design
  - 0.5 water/solids ratio
  - 3:1 aggregate to aluminosilicate ratio
  - 2:1 aluminosilicate to activator ratio
- Curing
  - Placed in 80°C oven for 24 hours
  - Then:
    - 23°C, 95% Relative Humidity
    - Deionized (DI) water bath



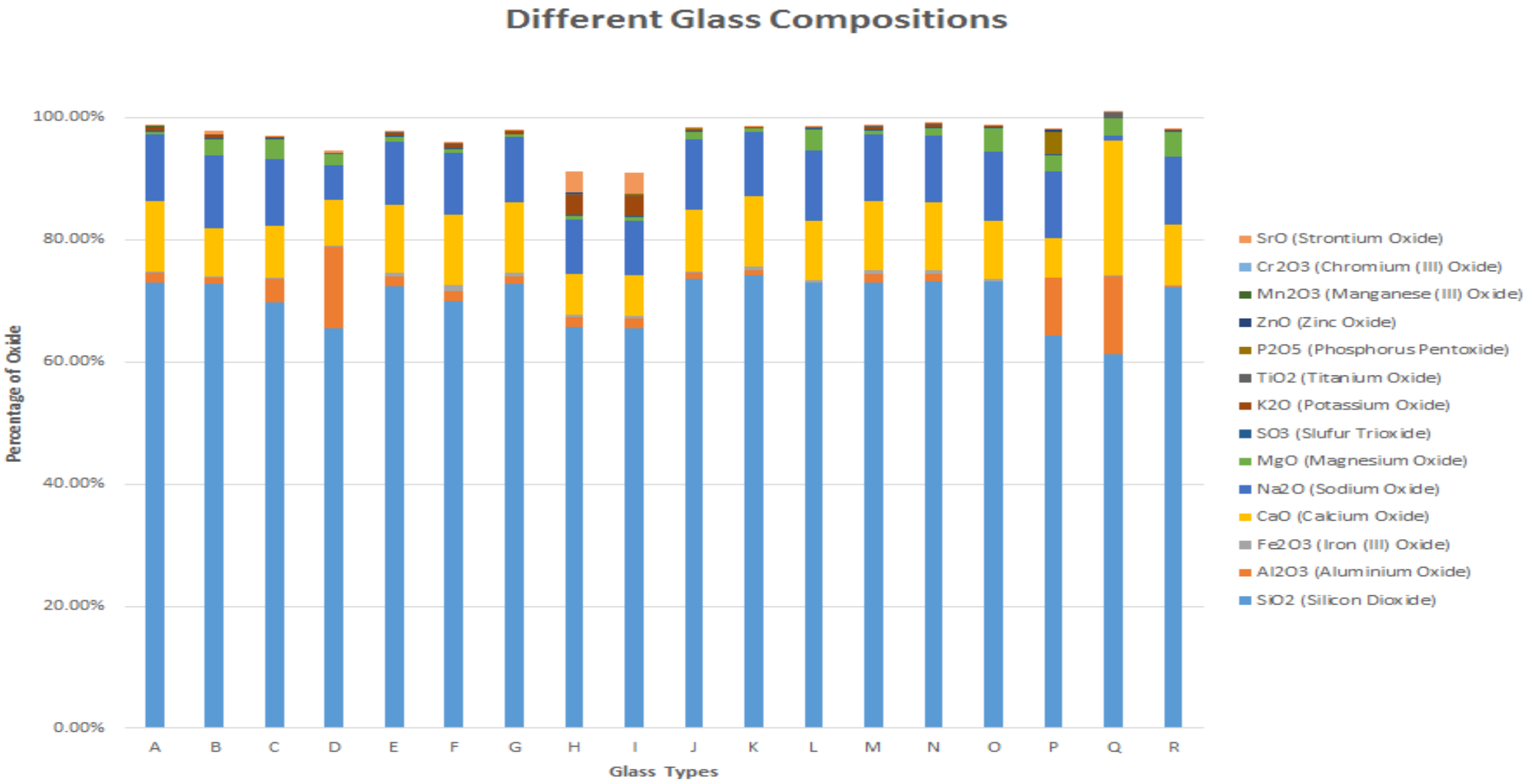
Materials

- Aluminosilicate
  - Glass
- Activator Solution
  - 10M NaOH
- Silica sand
- Water

Glass Samples

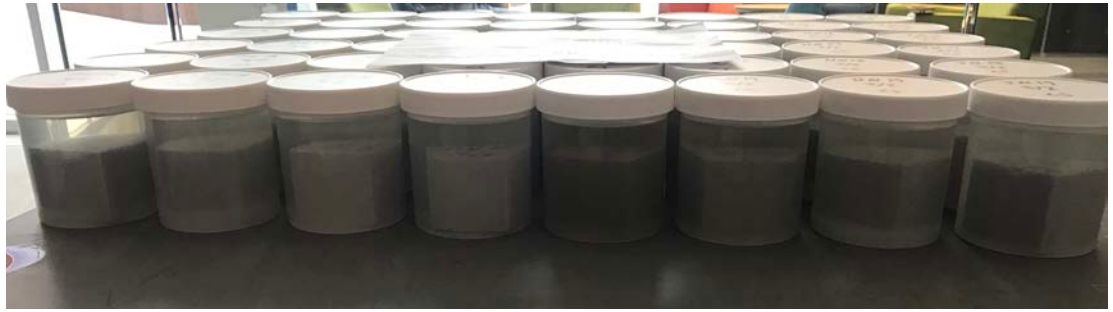
- 16 glasses were collected and analyzed by x-ray fluorescence (XRF)
  - Eight different locations
  - Multiple waste streams
- Glasses were ground to desired particle size
  - d90 < 45 µm
  - d50 = 8-14 µm
  - d10 = 1.5-2.5 µm

Glass ID	Origin Location	Waste Stream
A	Utah	Consumer
B	New York	Consumer
C	Tennessee	Consumer
D	Tennessee	Industrial
E	Ohio	Consumer
G	Minnesota	Consumer
H	Indiana	Consumer
J	Missouri	Consumer
K	Missouri	Consumer
L	Missouri	Consumer
M	Minnesota	Consumer
N	Minnesota	Consumer
O	Minnesota	Consumer
P	Tennessee	Consumer
Q	Tennessee	Manufactured
R	Tennessee	Industrial



Scope

- 100% waste glass geopolymer mortars
- Leaching Properties
- Compressive Strength
- Solution Compositional Analysis
- Microstructural Characterization

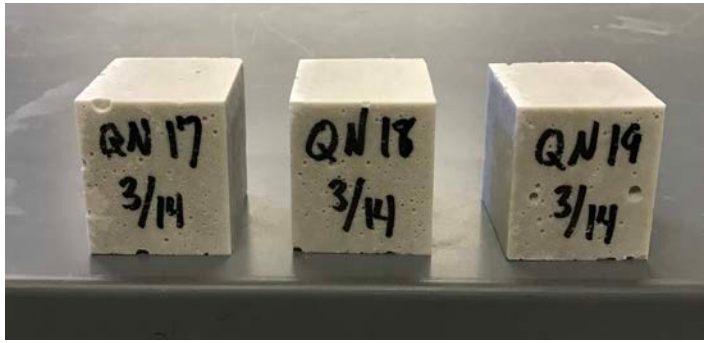


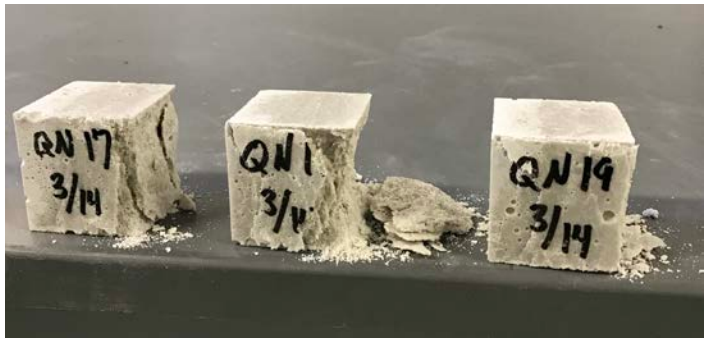
Leaching Properties

- Containers filled with 250 mL DI water
- pH
- Conductivity
- Early data was collected (1, 3, and 7 days) and then every week until 56 days
- Solution samples collected (7, 28, and 56 days)

Compressive Strength

- ASTM C109
- Cubes stored in DI water (wet) and cubes cured ambiently (dry)
- 7, 28, and 56 days



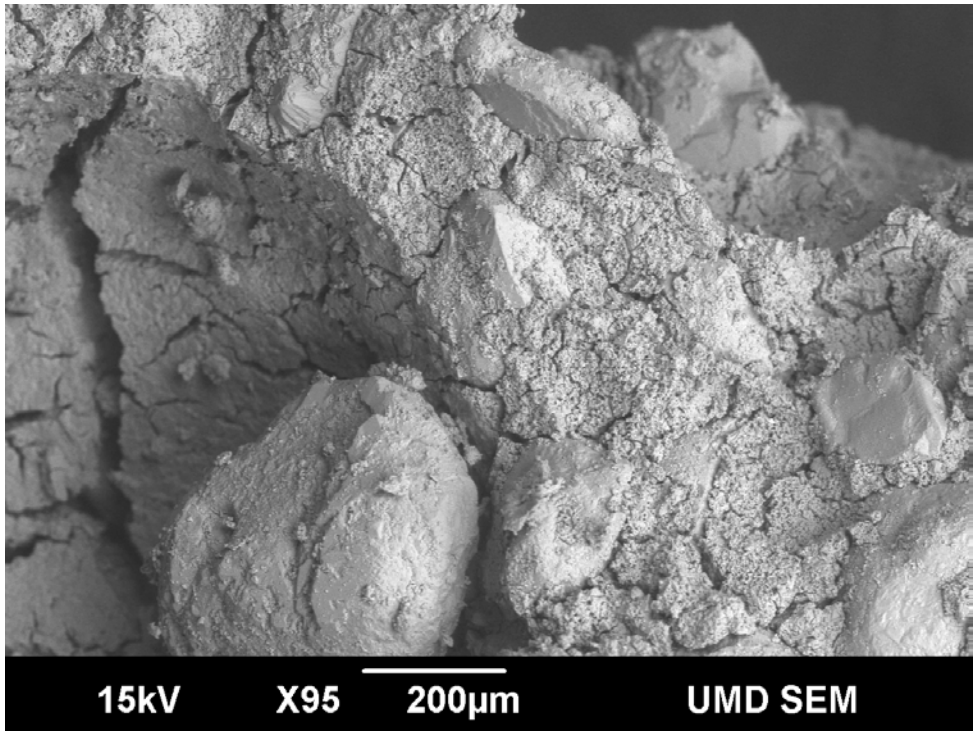


Solution Compositional Analysis

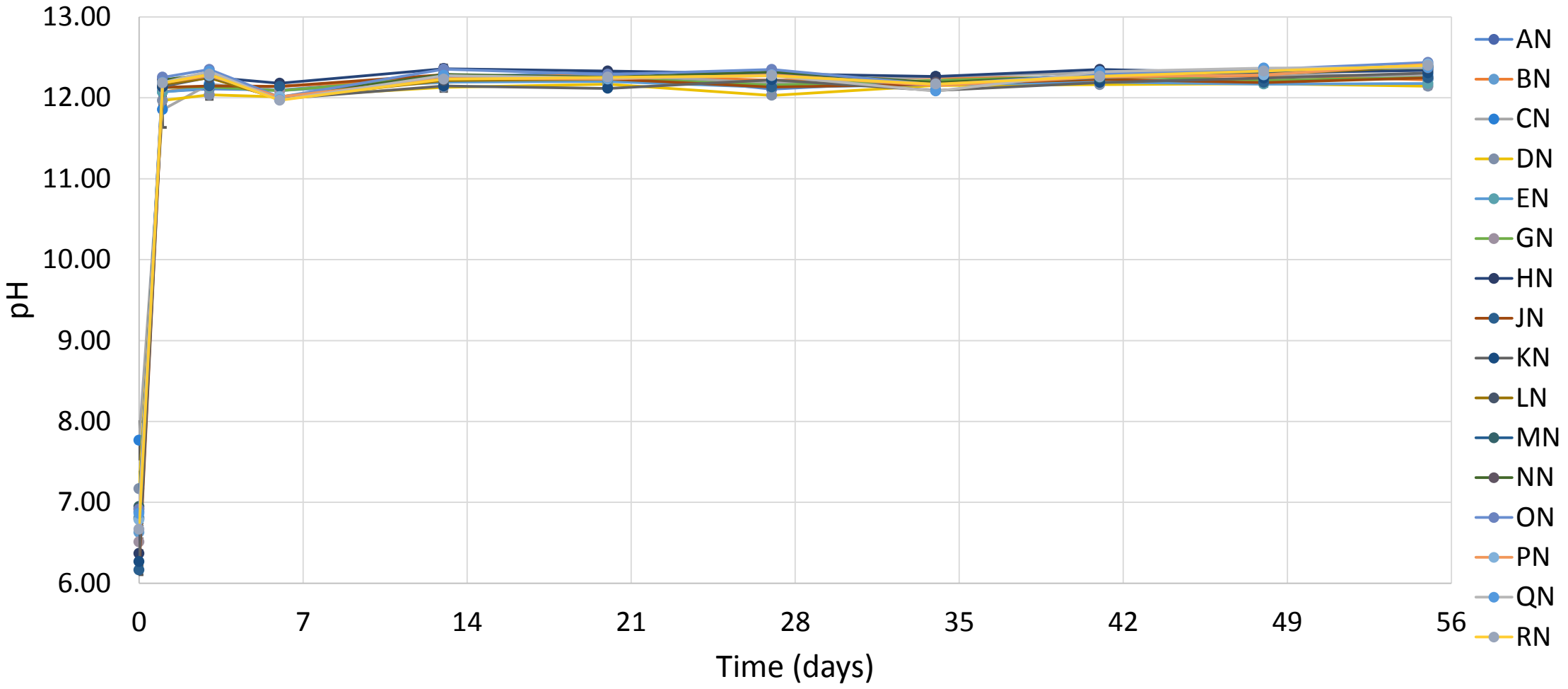
- 0.5 mL sample collected
- Selective Ion Electrode
  - Na
- Spectrophotometry
  - Si

Microstructural Characterization

- Scanning electron microscope (SEM)
  - Secondary electron imaging (SEI)
  - Energy dispersive X-ray spectroscopy (EDS)



Phase I pH


13.00
12.00
11.00
10.00
9.00
8.00
7.00
6.00
pH
0
7
14
21
28
35
42
49
56
Time (days)
AN
BN
CN
DN
EN
GN
HN
JN
KN
LN
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ON
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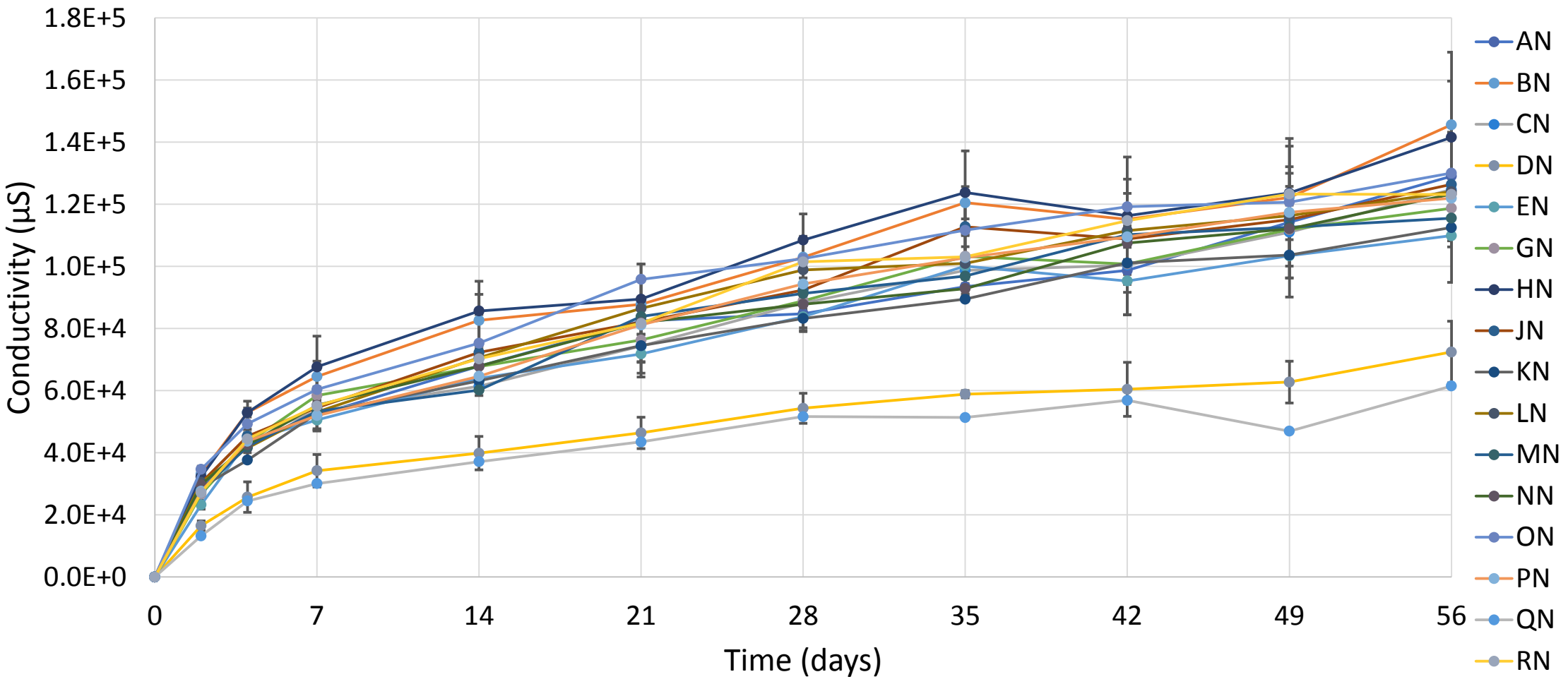
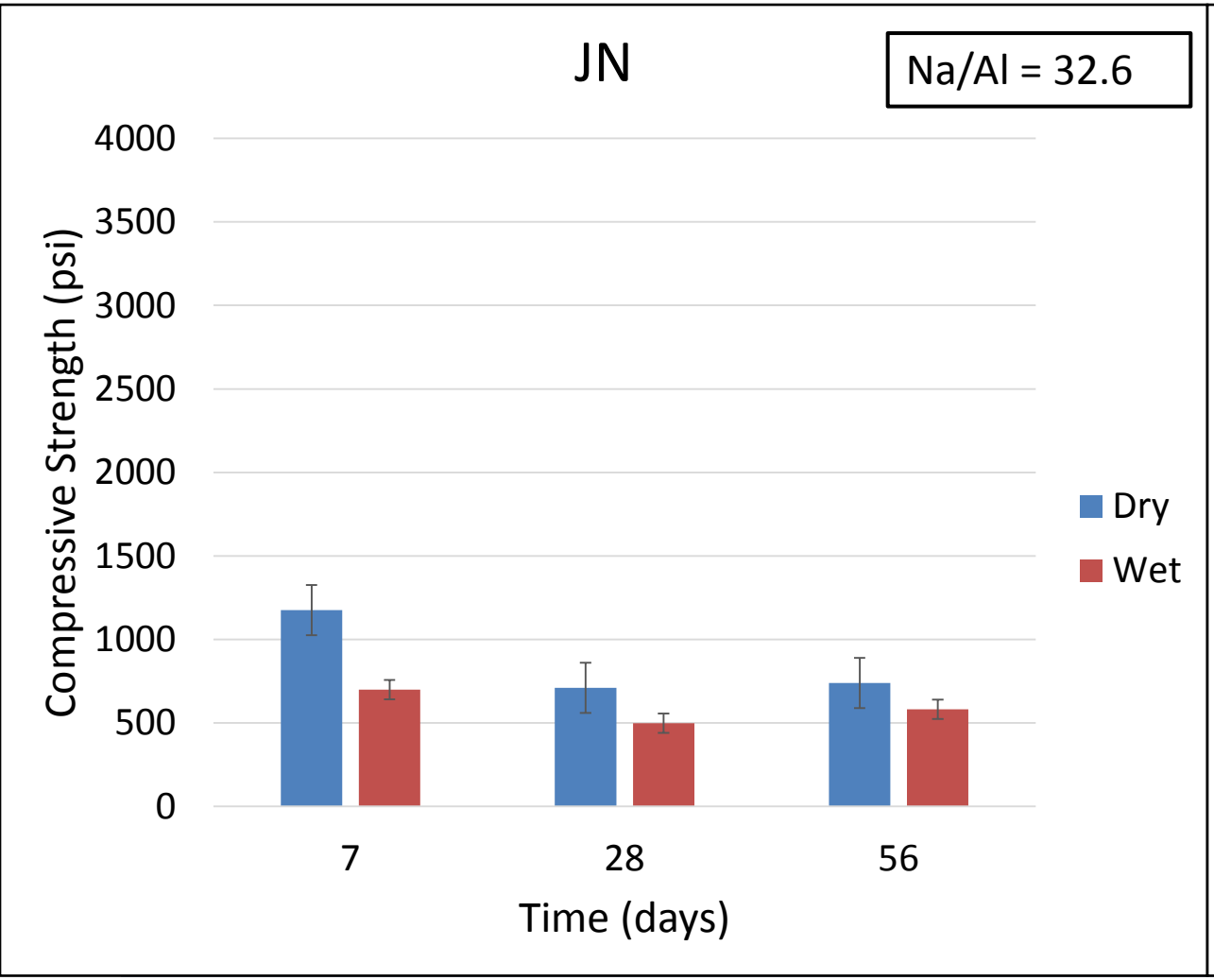
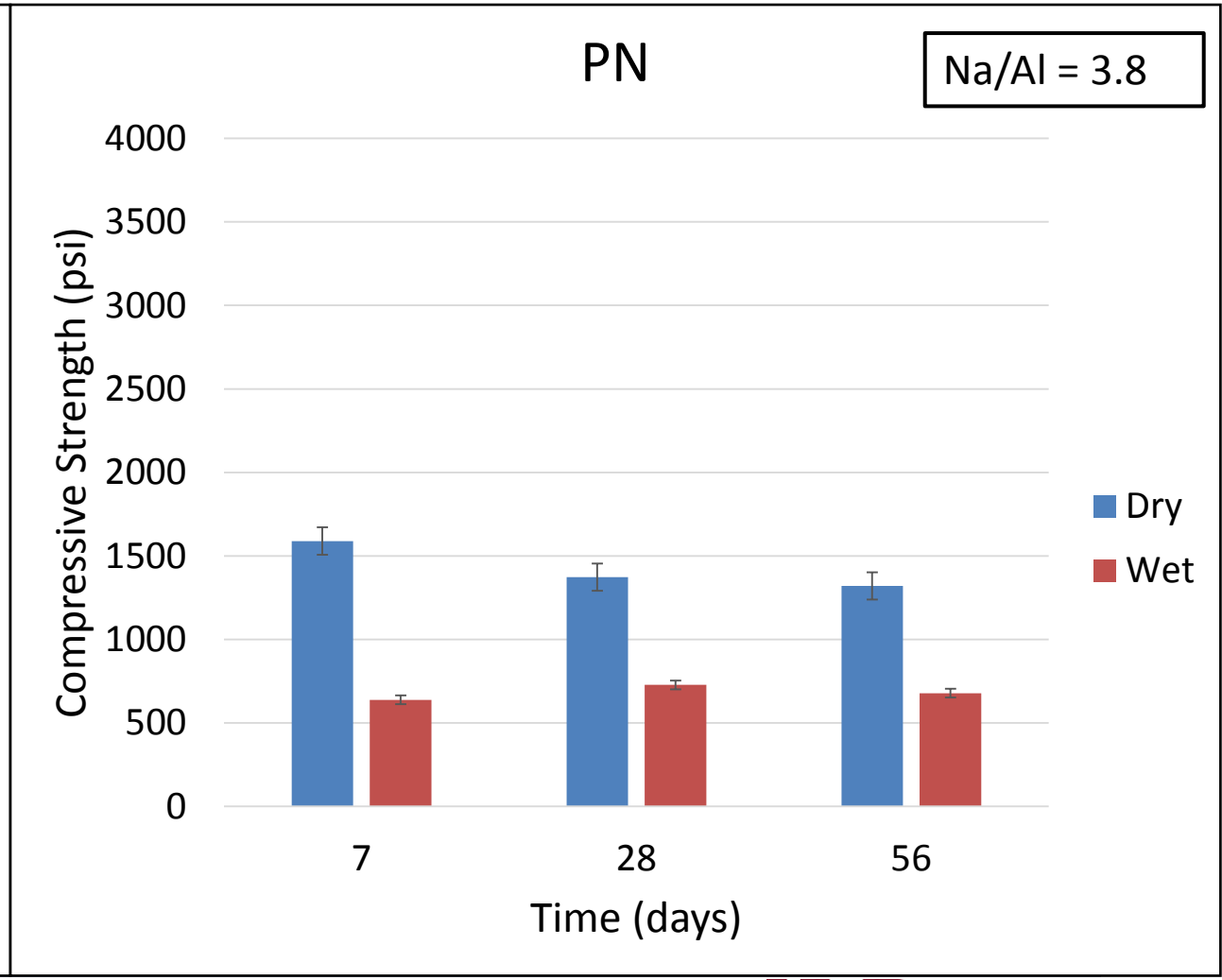
QN
RN

Phase I Conductivity

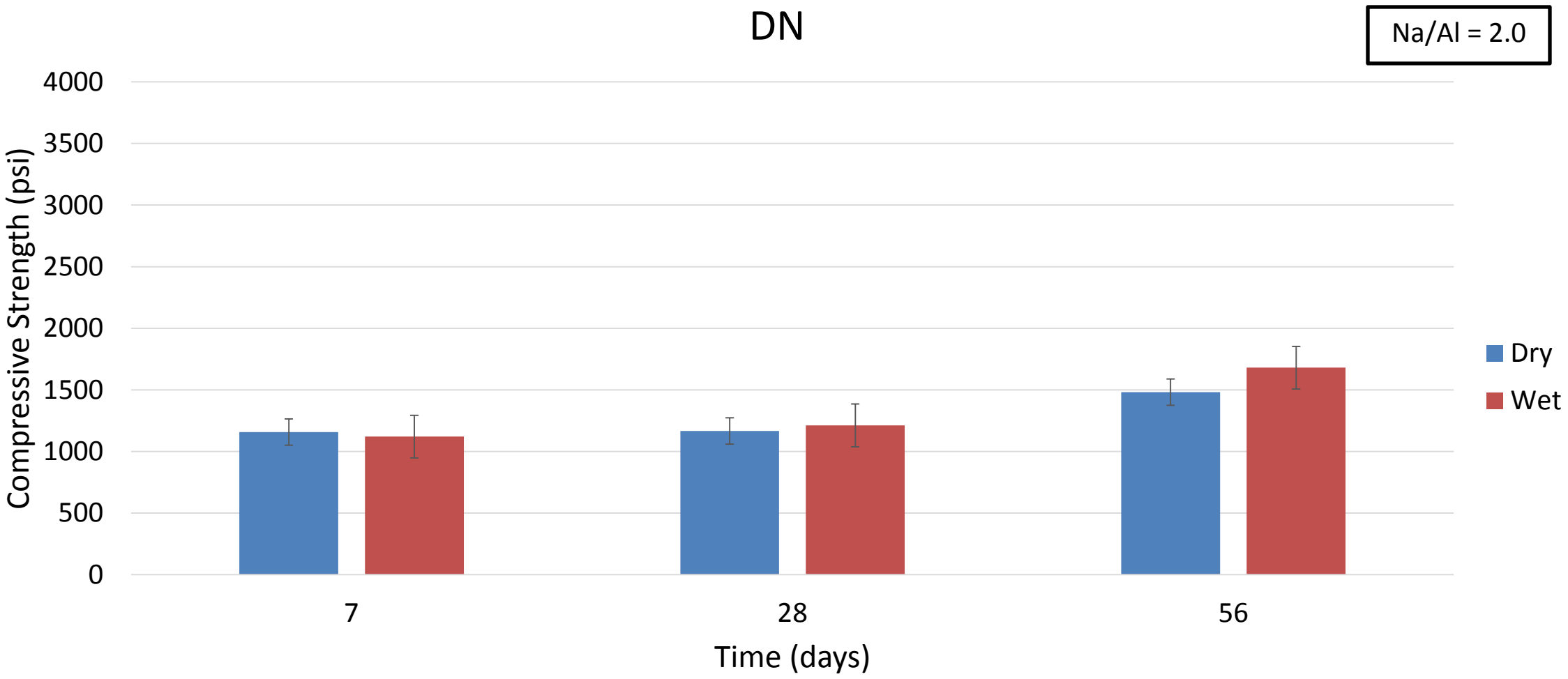


Phase I Compressive Strength





Phase I Compressive Strength



Observations

- Glasses with a higher Na/Al ratio have shown more white precipitate on wet cured samples
- More liquid precipitate in dry cured samples
- All of the mortar mixtures have shown a large air void structure

[1]

[1]

Observations

- 100% glass mixtures should be amended to account for the following:
  - Low compressive strength
  - Strength loss
  - Visible leaching of alkalis

Next Steps - Phase II

- New glass received from verified materials recycling facility (MRF)
- Explore other less caustic activators to increase compressive strength and decrease free sodium content
  - $Ca(OH)_2$
  - Na_2SiO_3
  - Na_2CO_3

[1]

Next Steps - Phase II

- Mix in additional alumina or calcium sources to obtain a Na/Al ~ 1-2
  - Metakaolin
  - Class F fly ash

Acknowledgments

- University of Minnesota Duluth
- National Resource Research Institute - Coleraine, MN
- American Engineering Testing – St. Paul, MN
- UMD Concrete Research Group
- Dr. Chanlan Chun
- Dr. Andrea Schokker
- Dr. Mary Christiansen

AMERICAN ENGINEERING TESTING, INC. [12]

[13]

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Questions?