

SEM-MSIA TO QUANTIFY THE COMPOSITION OF FLY ASH GLASSY PHASES AND REACTIVITY IN ALKALINE SOLUTIONS

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Background

- Fly ash

- Crystalline: 10 – 40 %

- Measure with XRD + Rietveld analysis

- Glassy: 60 – 90 %

- Difficult to characterize

- Disordered structure

- Many compositions of glass coexist in a single fly ash, even in a single particle

Background: SEM x-ray mapping (NIST)



- Field emission SEM with two energy dispersive spectroscopy (EDS) detectors
- 10 kV accelerating voltage, 60,000-80,000 counts per second, 1024 x 768 EDS maps, 256 μ s dwell time per pixel with a line average of 2
- 6 minutes to collect each map; four maps were collected and summed
- The saved data represent the actual counts at each pixel location on the specimen

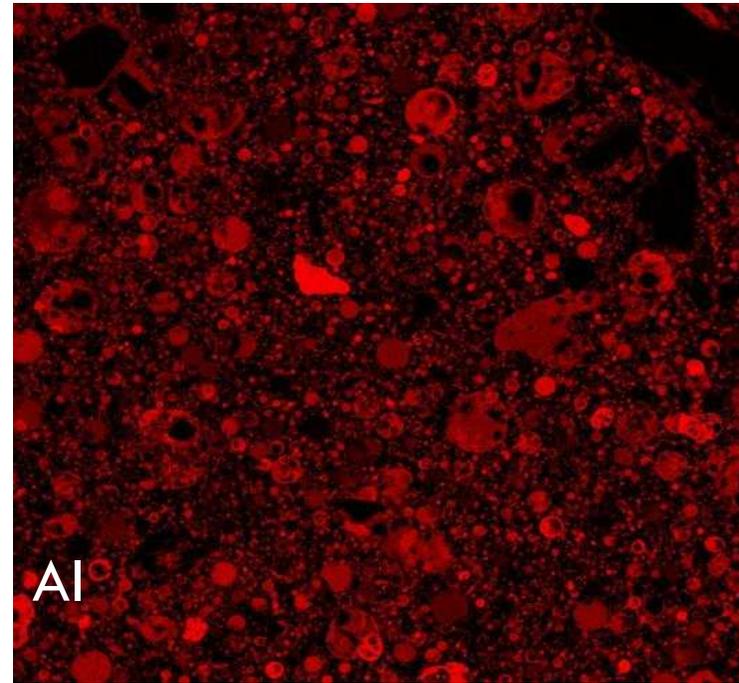
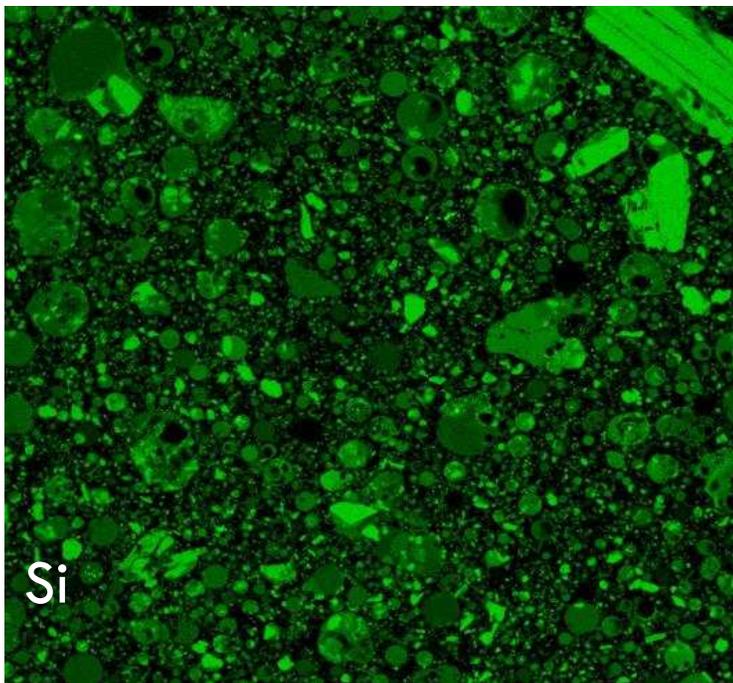
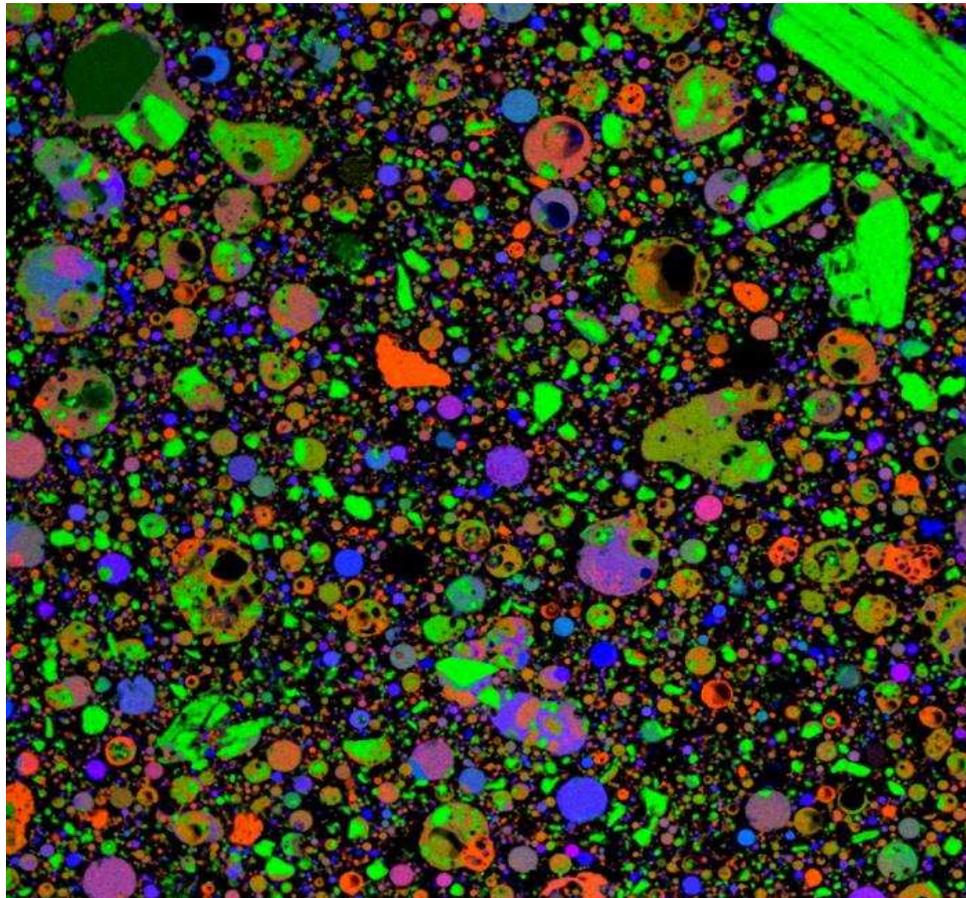
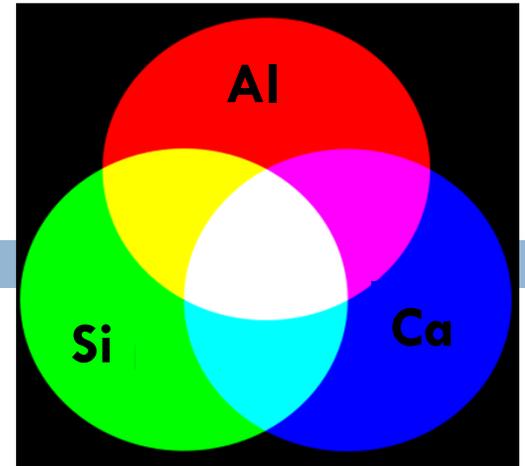


Image Processing

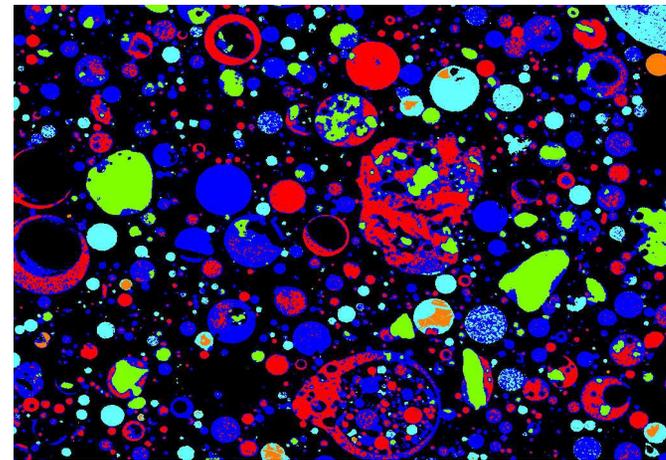
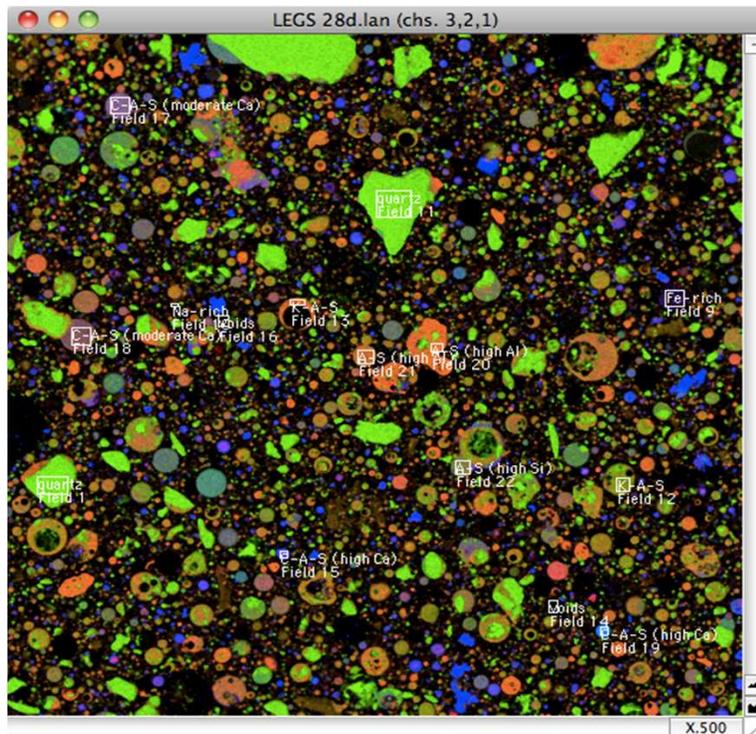
- Post-processing of x-ray maps
 - ▣ Median filter applied (radius = 1)
 - ▣ Thresholded to remove noise
- Multispectral image analysis (MSIA)
 - ▣ Virtually stacked images can be analyzed based on contributions from all images in the stack to group statistically similar pixels

MSIA- Image Stacks



MSIA- Pixel Assignment

- Select training class pixels for each phase
- Multispec assigns all pixels in the image to one of the training classes using an algorithm



Background: Dissolution Methods

- Reactivity of glassy phases

- HF acid dissolution

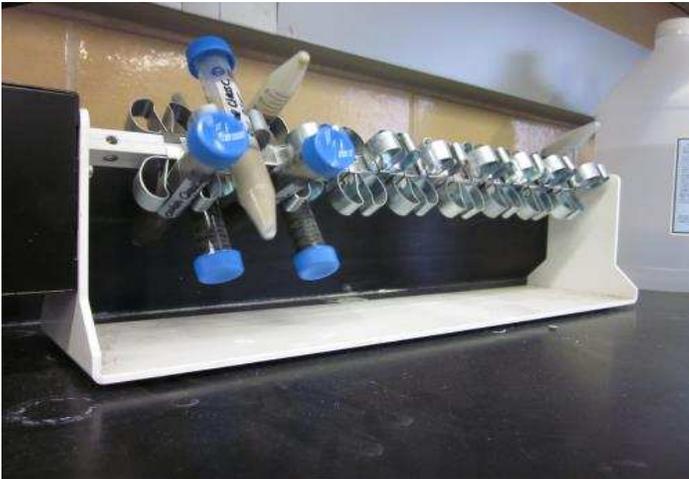


- NaOH dissolution



Dissolution Methods

- 2 g (± 0.01) fly ash + 10 mL of 8 M NaOH

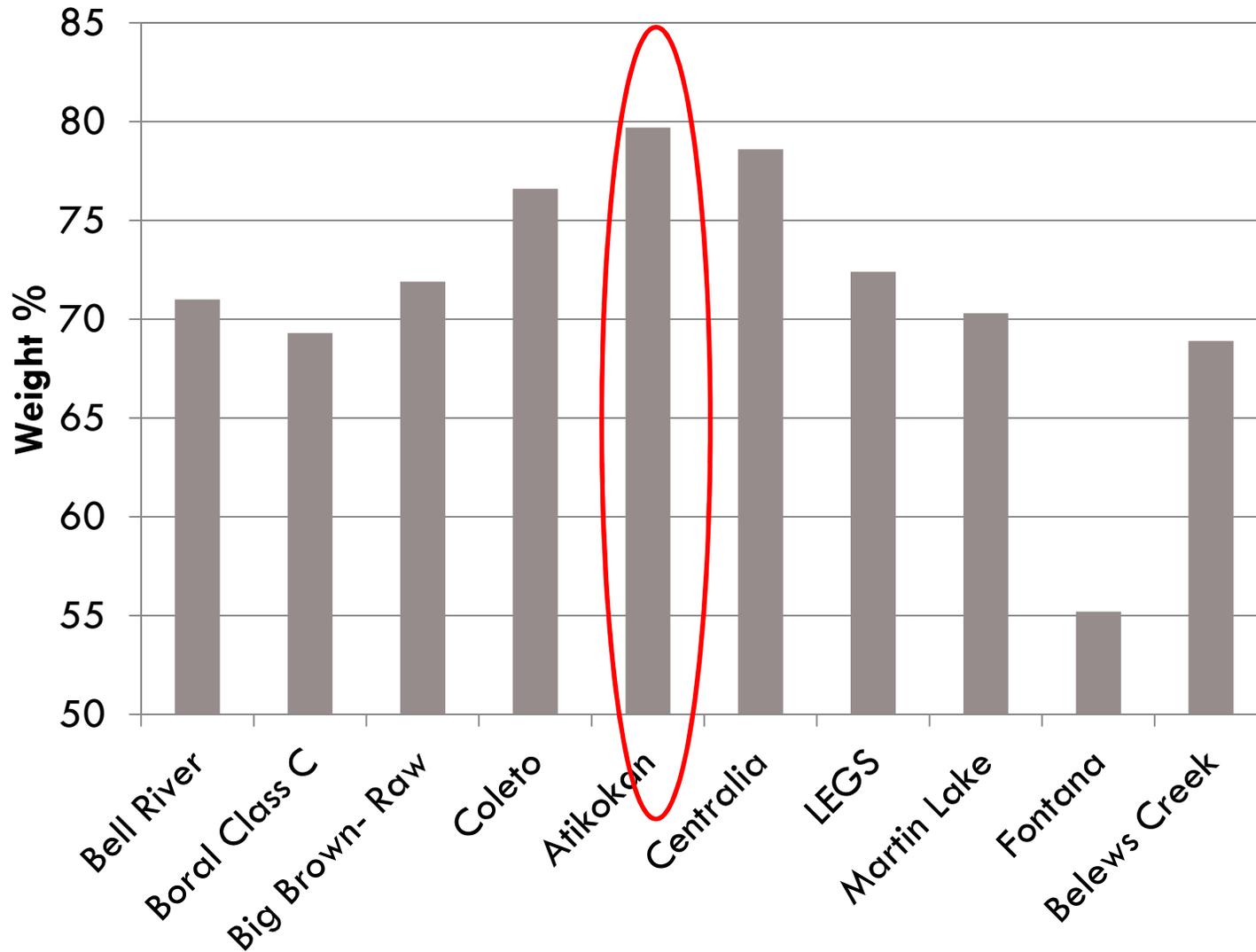


Fly ash

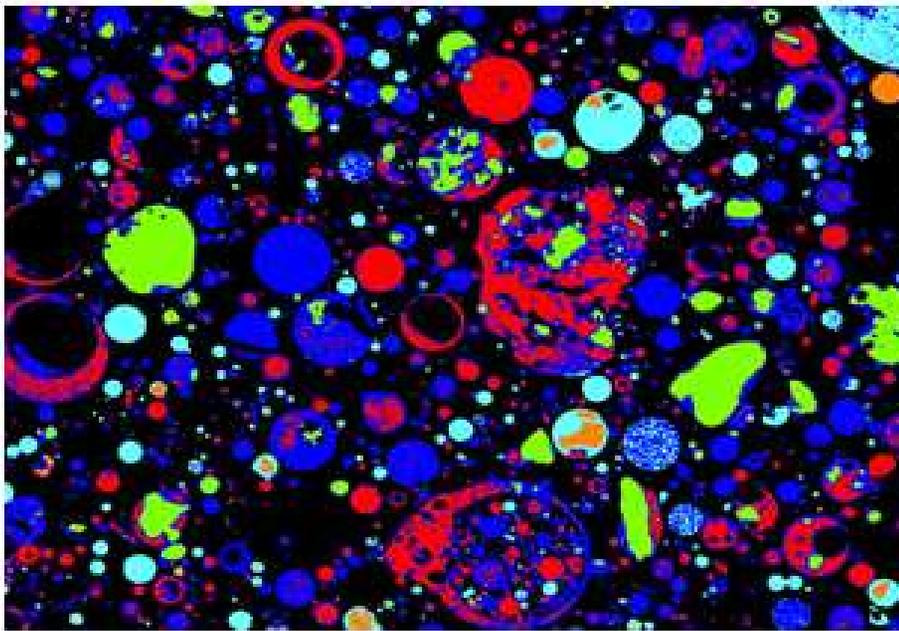
□ Atikokan Class F fly ash (Oregon, USA)

Oxides	Limestone (LEGS) wt. %
Al_2O_3	21.58
SiO_2	47.66
CaO	12.30
Fe_2O_3	4.21
K_2O	0.89
MgO	2.70
Na_2O	1.93
SO_3	1.20
TiO_2	0.97

Bulk Amorphous Content by Rietveld Analysis



MSIA pixel assignment map



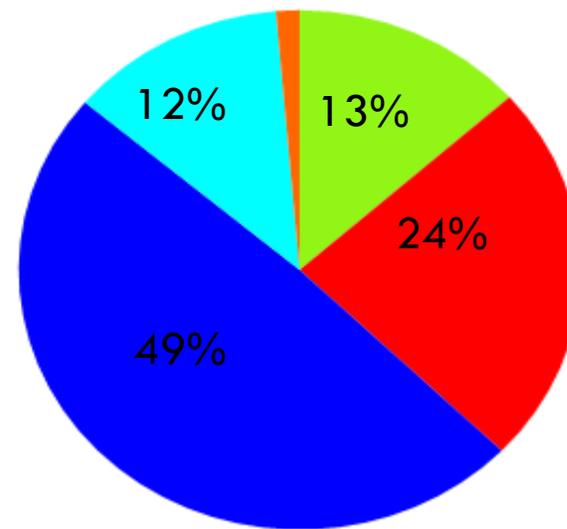
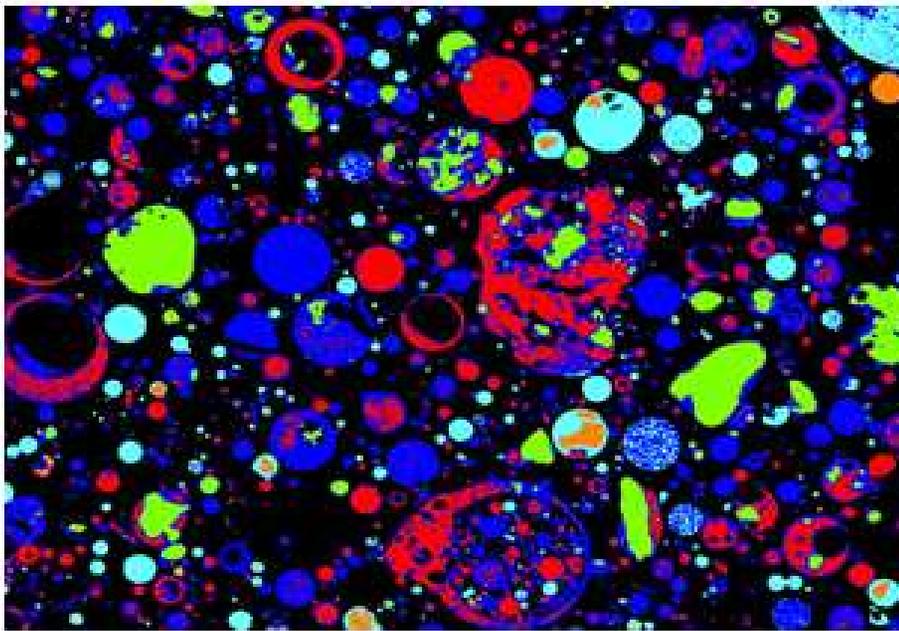
Phase designation	S / A	C / S
Aluminosilicate	0.95	-
C-A-S	0.56	0.2
C-A-S 1 (low Al)	1.28	1.0

 Aluminosilicate
 C-A-S
 C-A-S (low Al)

 C-A-S (high Al)
 Fe-rich
 Quartz

 Voids

MSIA area percentages



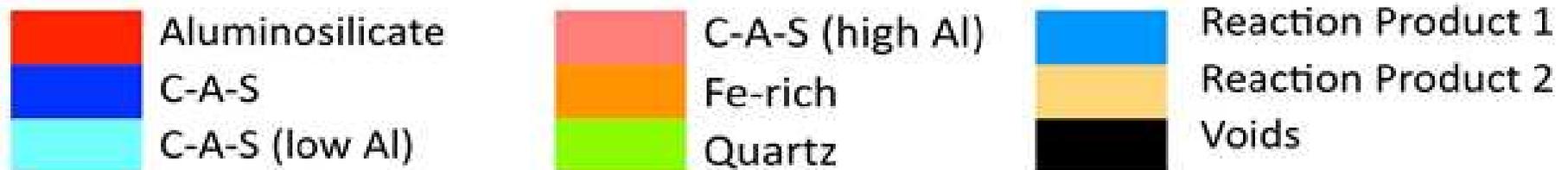
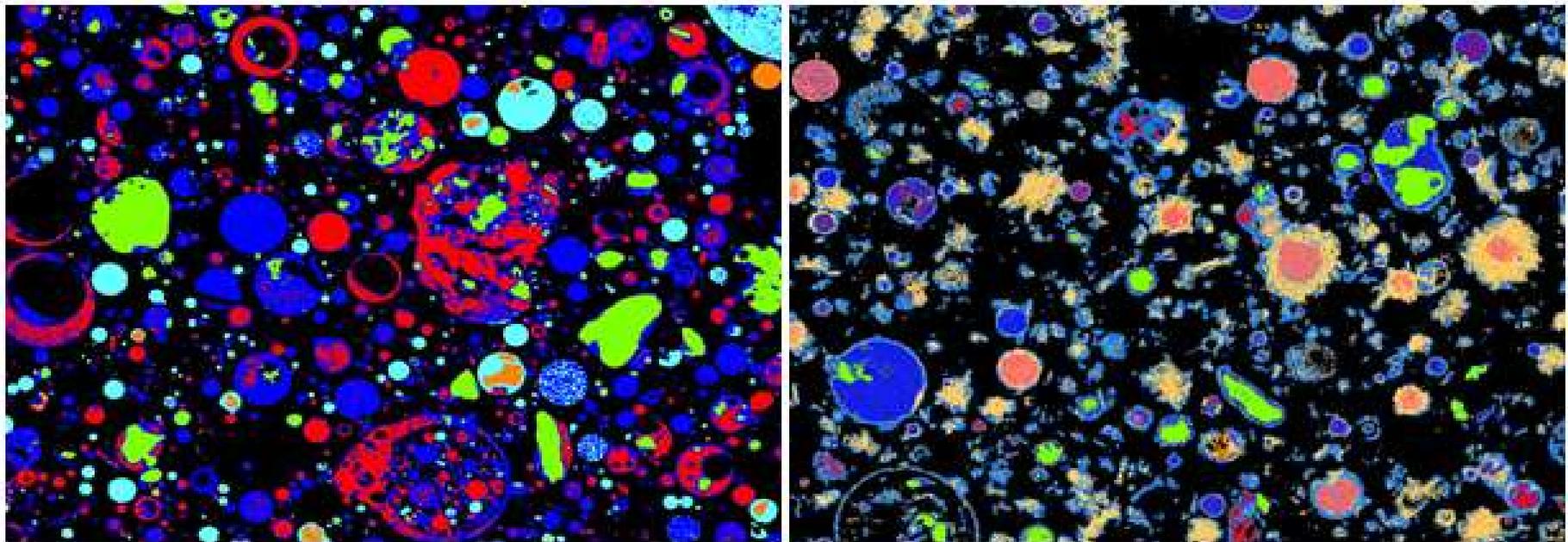
- quartz
- Aluminosilicate
- C-A-S
- C-A-S (low Al)
- Fe-rich

Aluminosilicate
C-A-S
C-A-S (low Al)

C-A-S (high Al)
Fe-rich
Quartz

Voids

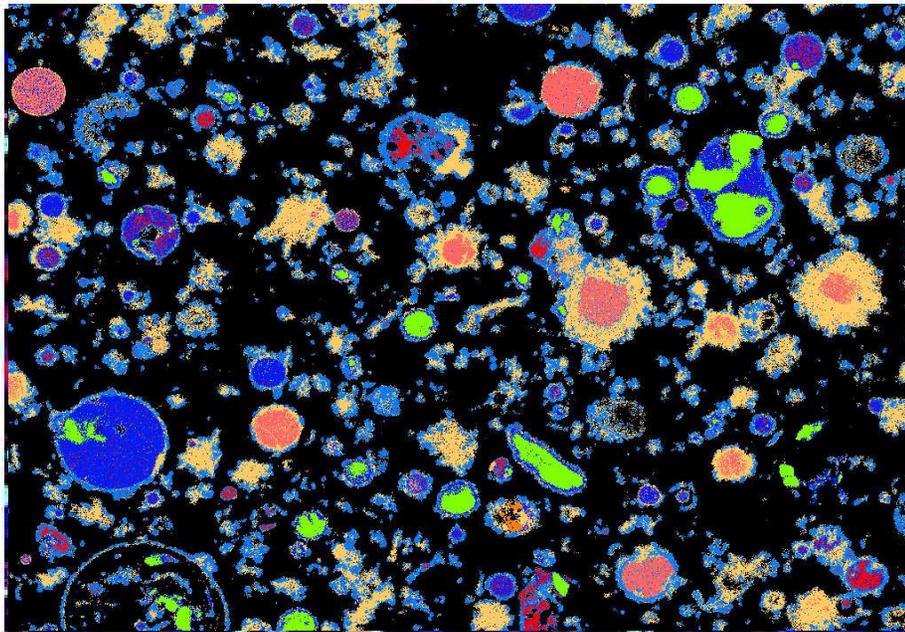
Reactivity analysis



Fly Ash

After 28 days in 8N NaOH solution

MSIA reactivity map



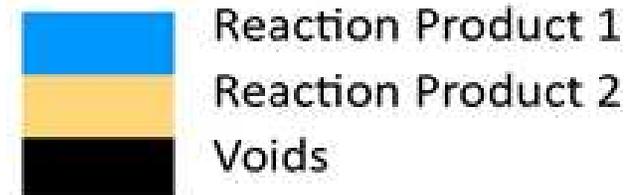
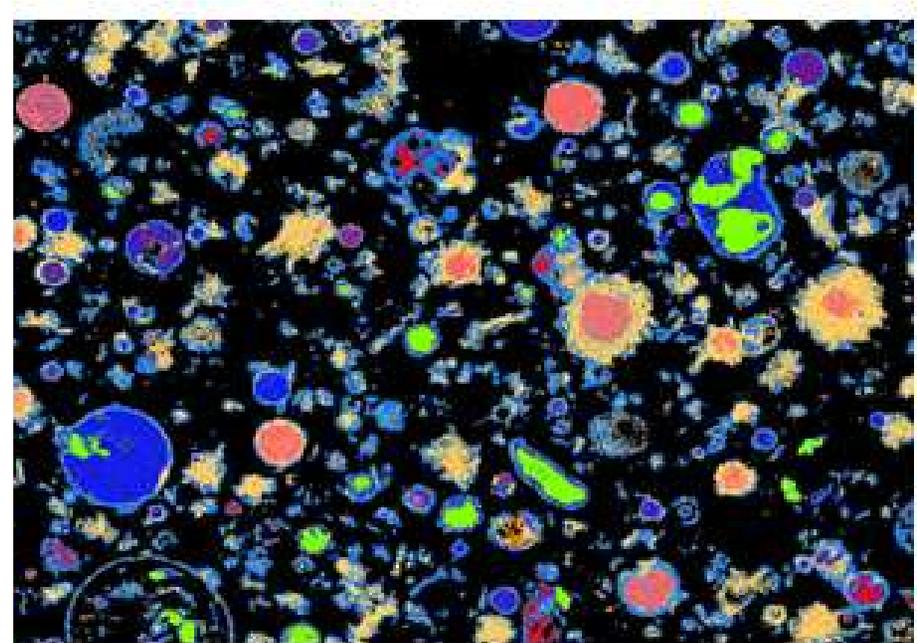
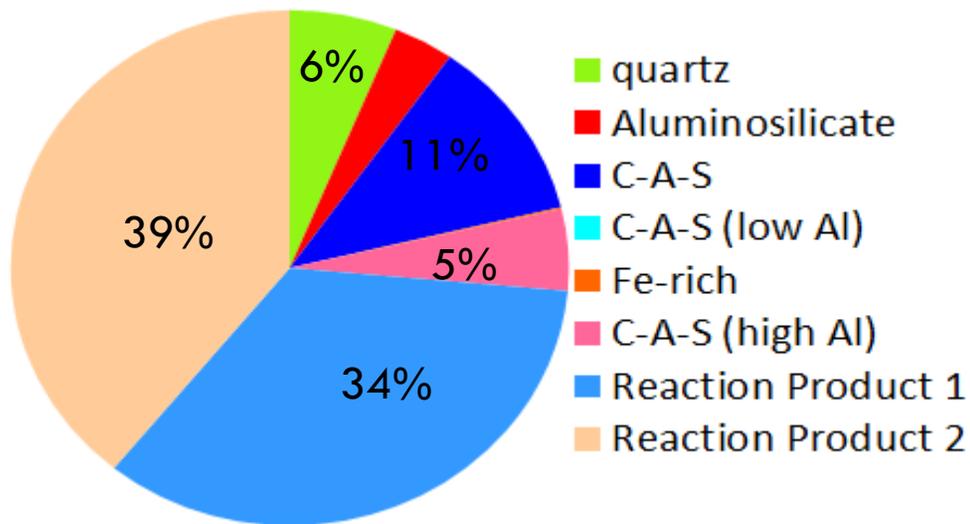
Phase designation	S / A	C / S
Aluminosilicate	0.95	-
C-A-S	0.56	0.2
C-A-S 1 (low Al)	1.28	1.0
C-A-S 2 (high Al)	0.88	0.8
Reaction Product 1	0.65	0.2
Reaction Product 2	1.56	0.9

Aluminosilicate
 C-A-S
 C-A-S (low Al)

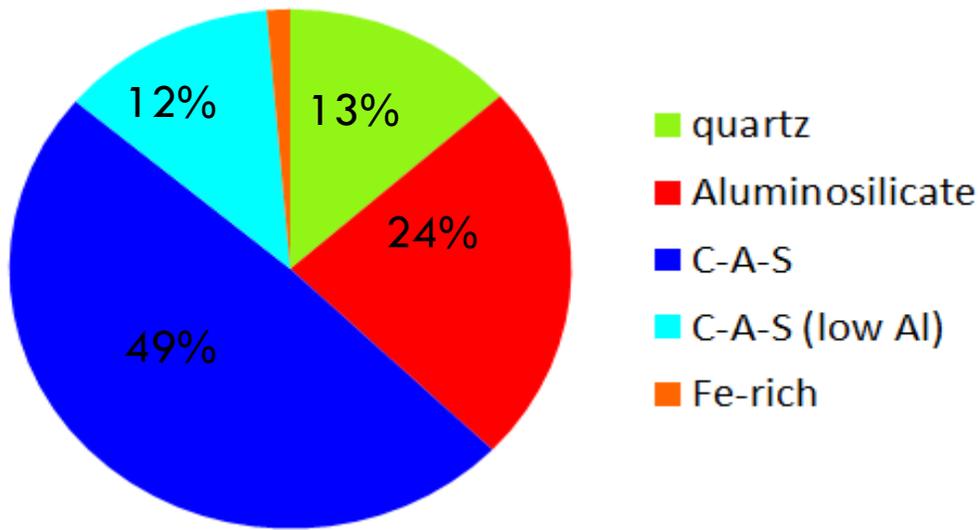
C-A-S (high Al)
 Fe-rich
 Quartz

Reaction Product 1
 Reaction Product 2
 Voids

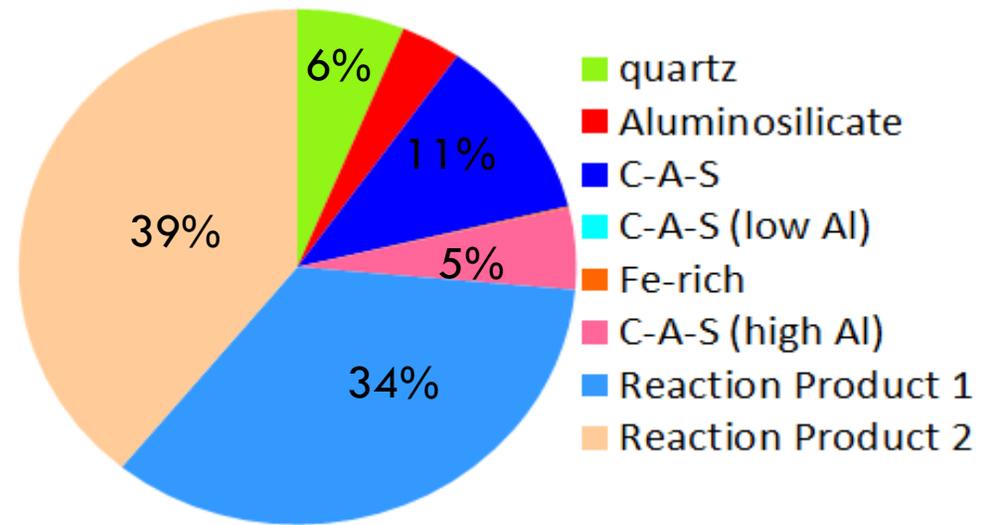
After 8N NaOH exposure



MSIA pixel assignment maps



Fly Ash



After 28 days in 8N NaOH solution

Summary

- The fly ash consisted of an aluminosilicate phase, two C-A-S phases, an iron-rich phase, mullite, and quartz.
- All of the glassy phases appeared reactive in this fly ash.
- Two types of reaction product were observed, with varied S/A ratios and C/S ratios. The high Ca reaction product (Reaction Product 2) appeared to have formed from C-A-S particles by leaching silicon and calcium.

Observations & Future Work



- SEM-MSIA allows qualitative and quantitative (area percentage) analysis of glassy phases in fly ash
- Tracking phases over time after exposure to caustic solutions enables assessment of the relative reactivity of glassy phases
- We are analyzing reactivity for 10 fly ashes

Thank you to NSF CMMI-0926627 for financial support of this work, to Aasiyah Baig for her work on the research, and to NIST for use of their scanning electron microscope.