## External Evaluation Panel Meeting, Jan 19, 2016

# Chemo-mechanical properties of Hardened cement pastes prepared with volcanic ash and Ordinary Portland cement.

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# Research Significance

- Volcanic ash has been considered a potential cement additive because it is similar to fly ash, a commonly used additive
- The goal of this study is to characterize cement paste made of Ordinary Portland Cement (OPC) and volcanic ash of different particle sizes and concentrations.
- Impact: This work will help engineering cements when volcanic ash is used as a partial substitute to OPC

# Background: Tests

- Particle Size Distribution using a Laser-based Particle Size Analyzer
- Compression Test (ASTM Standard)
- Mercury Intrusion Porosimetry (MIP) using alcohol suspension
- SEM/BSE Imaging at CMSE
- Future Tests
  - Nanoindentation
  - Synchrotron X-Ray Diffraction

# Specimen Preparation Chart

| | OPC (%) | IP (%) | FA (%) | Nomenclature |
|---|---|---|---|---|
| 1 | 100 | 0 | 0 | OPC |
| 2 | 50 | 50 | 0 | IP-50 |
| 3 | 60 | 40 | 0 | IP-40 |
| 4 | 70 | 30 | 0 | IP-30 |
| 5 | 80 | 20 | 0 | IP-20 |
| 6 | 90 | 10 | 0 | IP-10 |
| 7 | 50 | 0 | 50 | FA-50 |
| 8 | 60 | 0 | 40 | FA-40 |
| 9 | 70 | 0 | 30 | FA-30 |
| 10 | 80 | 0 | 20 | FA-20 |
| 11 | 90 | 0 | 10 | FA-10 |

FA = 6 μm mean particle size of VA
IP = 17 μm mean particle size of VA

- Water to Binder Ratio of 0.35
- Specimens cured for 28 days
- Specimens submerged in acetone to arrest the hydration

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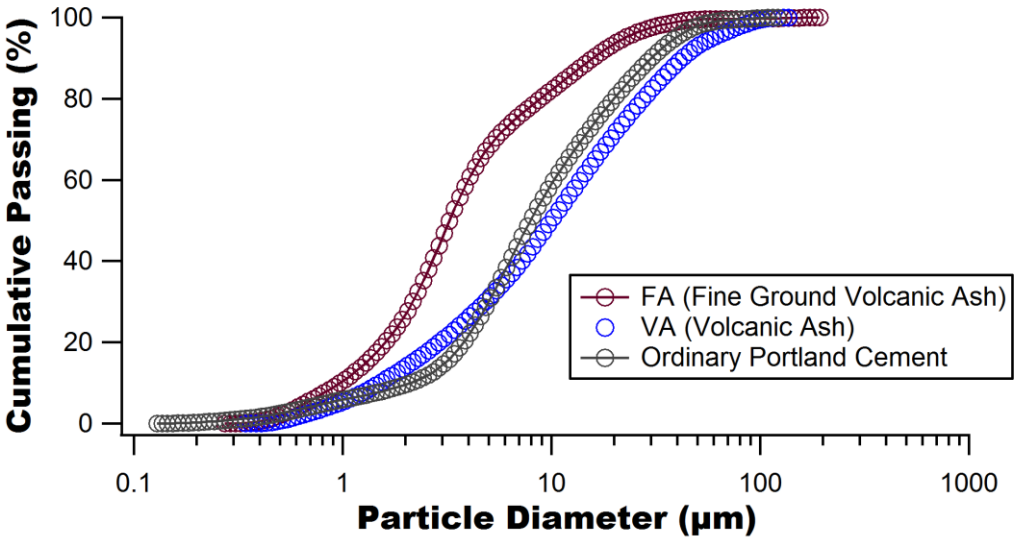
# Particle Size Distribution (Laser-based Particle Size Analyzer)




| Binder Type | Nomenclature | Mean (µm) | Median (µm) | Mode (µm) | Diameter for selected percentiles by volume | | |
|---|---|---|---|---|---|---|---|
| | | | | | D 90 (µm) | D 50 (µm) | D10 (µm) |
| Volcanic Ash | IP | 17.14 | 10.00 | 13.27 | 42.46 | 10.00 | 1.50 |
| Volcanic Ash | FA | 6.00 | 3.25 | 2.977 | 15.79 | 3.25 | 0.973 |
| Portland Cement | OPC | 12.73 | 7.94 | 6.65 | 30.10 | 7.94 | 2.12 |

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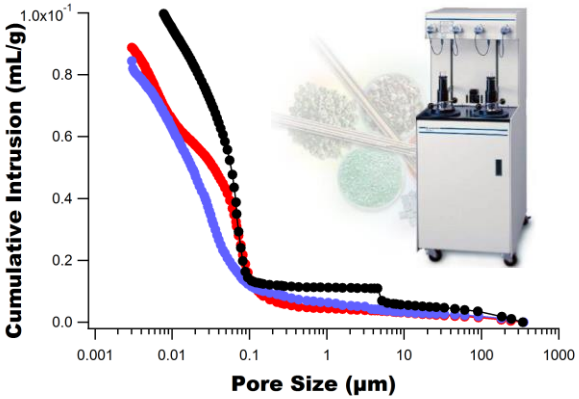
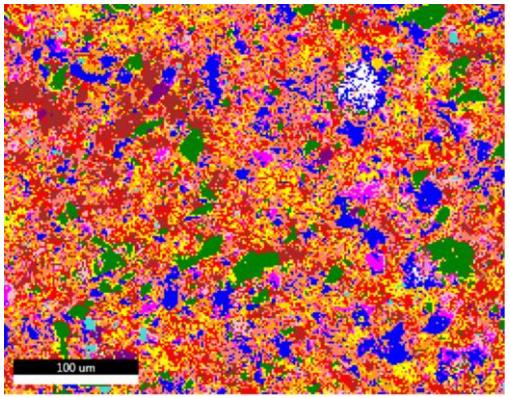
# Experimental Evaluation

Compression Test → Pore Structure → Microstructure

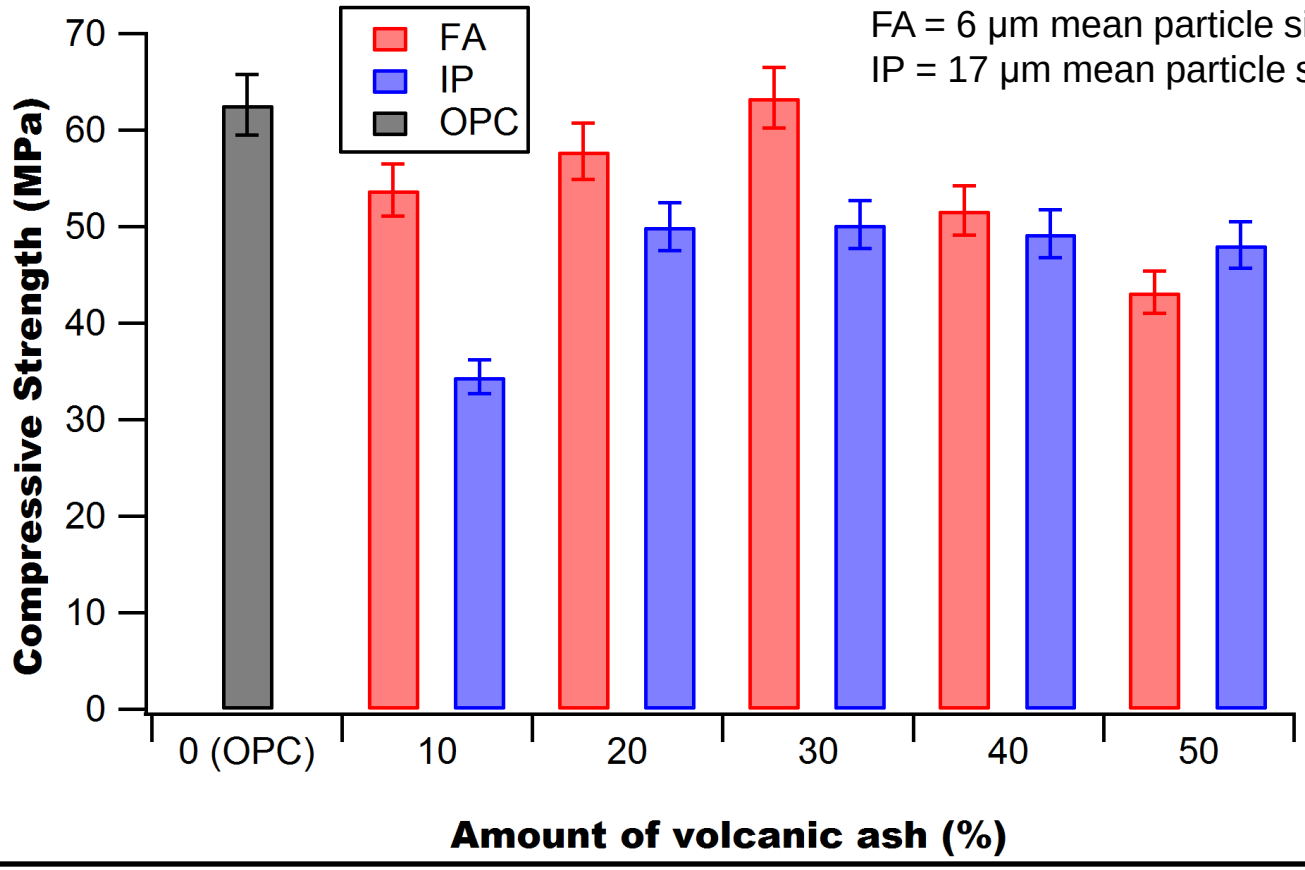



**Pore structures studies were performed using Mercury Intrusion Porosimetry**

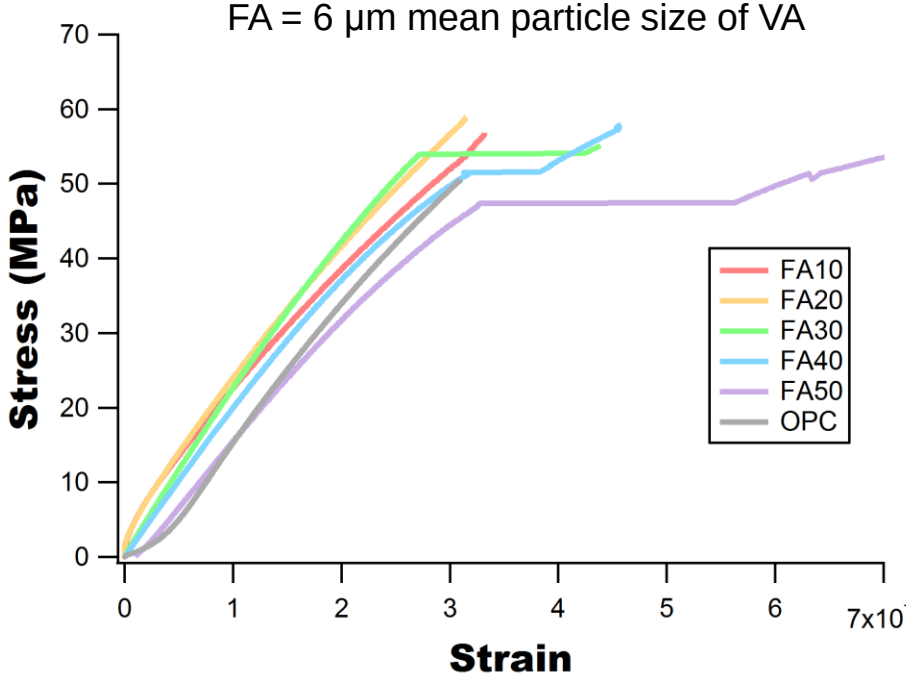
**Phase mapping performed using Back Scattered Electron Spectroscopy (BSE)**

# Compression Test Results

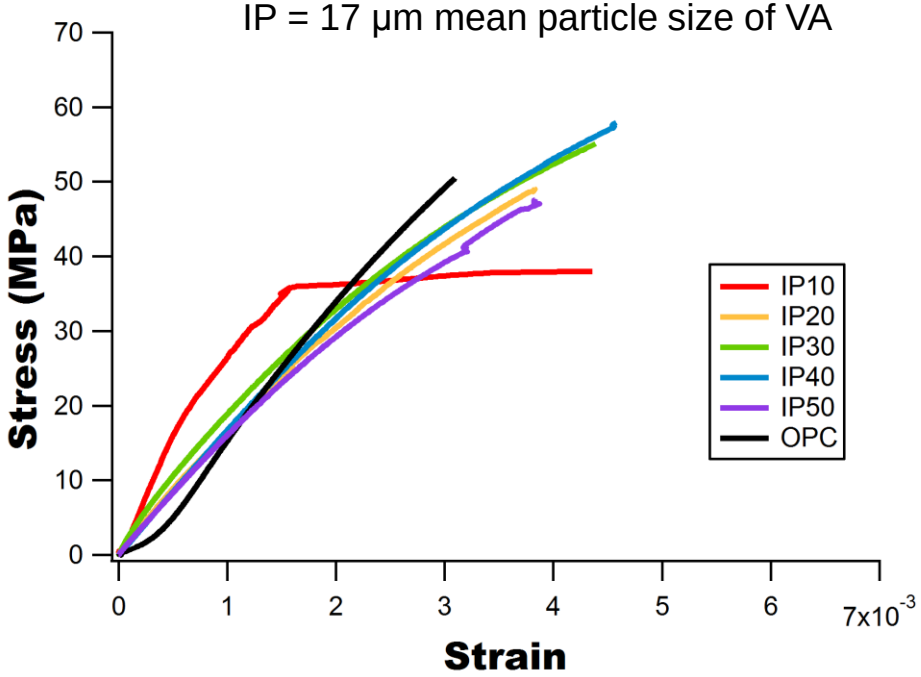


# Stress-Strain plots (Compression Testing)

FA = 6 μm mean particle size of VA

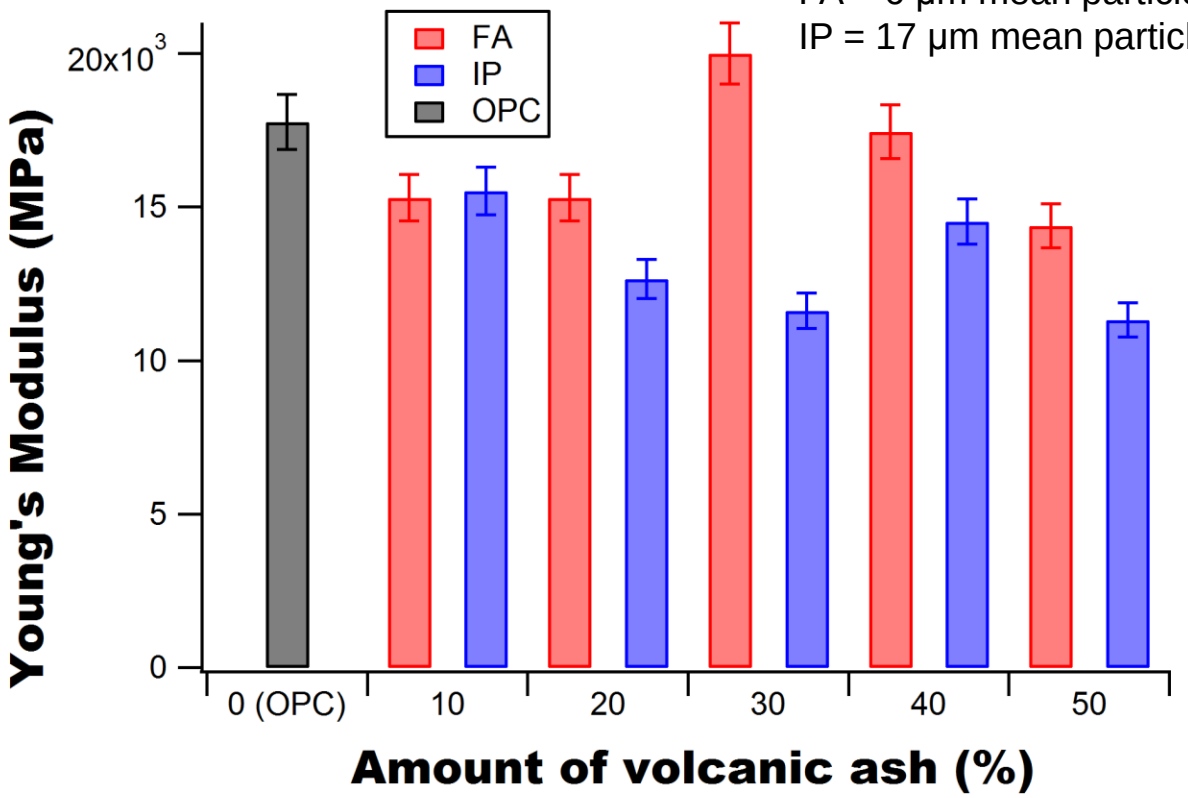



IP = 17 μm mean particle size of VA

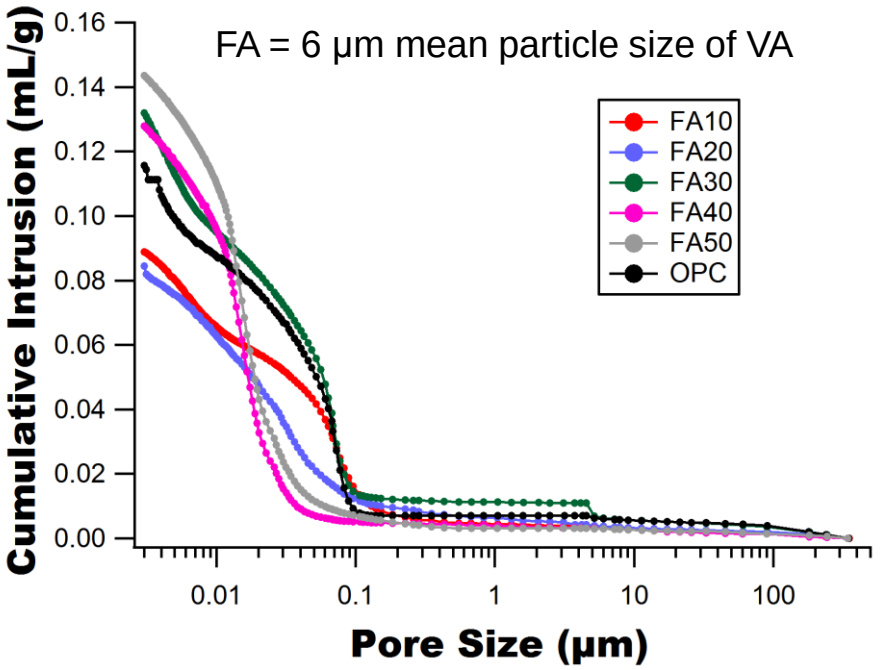


# Young's Modulus Analysis

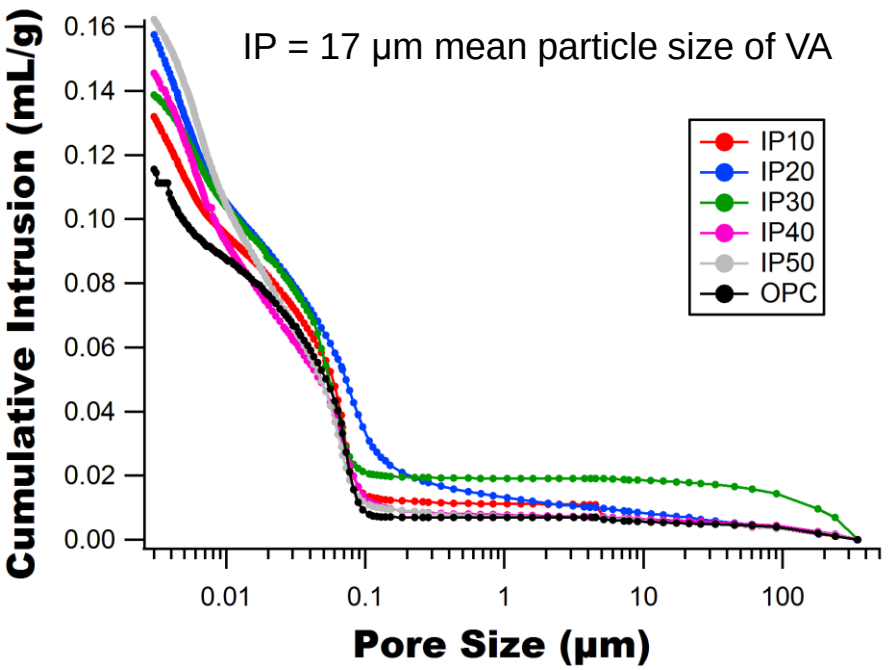
FA = 6 μm mean particle size of VA
IP = 17 μm mean particle size of VA



# Pore Structure (Mercury Intrusion Porosimetry)



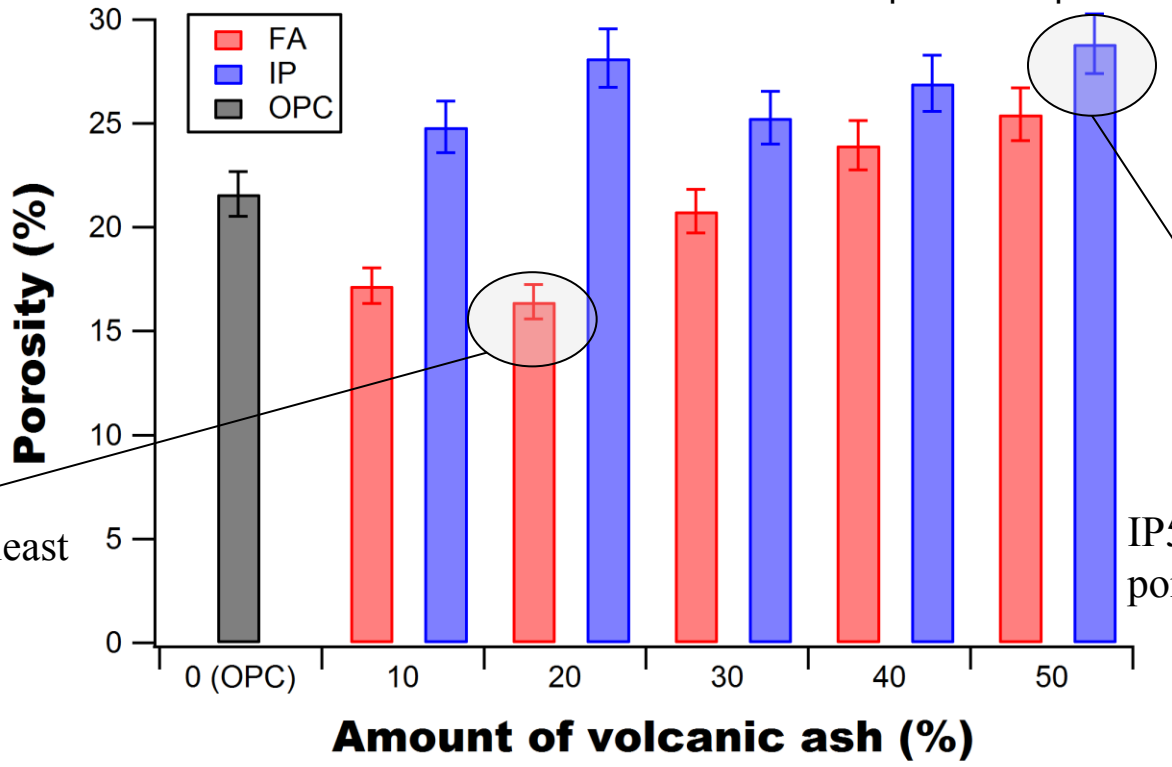

FA20 had the least intruded volume, while FA50 had the highest intrusion




OPC had the least intruded volume, while IP50 had the highest intrusion

# Porosity

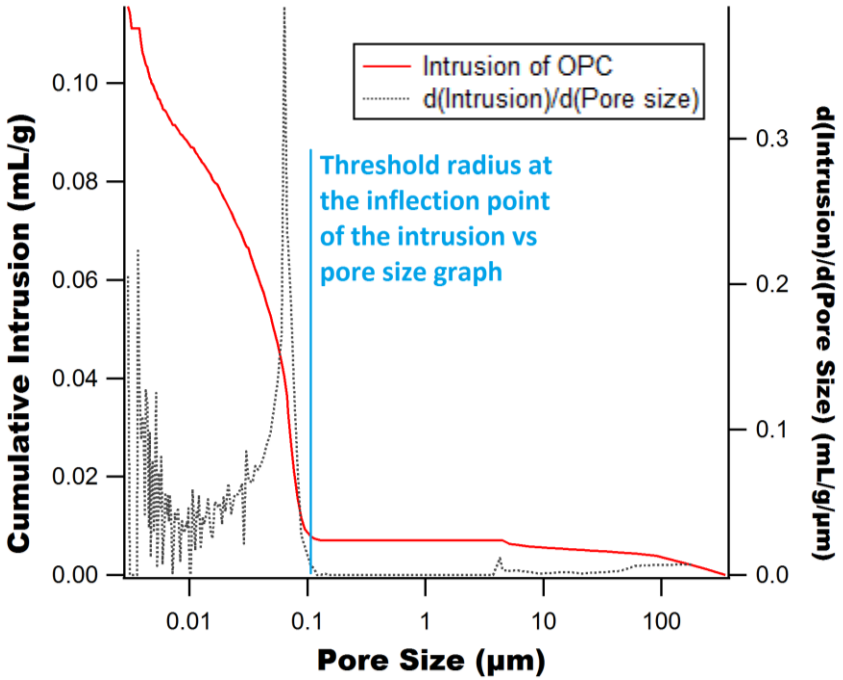
FA = 6 μm mean particle size of VA
IP = 17 μm mean particle size of VA




FA20 had the least porosity (%)

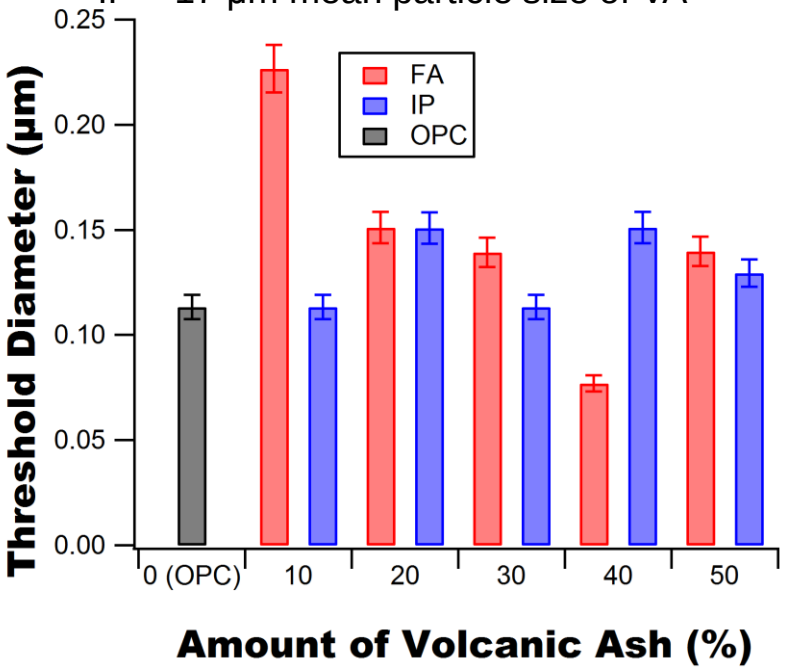
IP50 had the greatest porosity (%)

# Threshold Pore Diameter




Threshold diameter can be determined by finding the inflection point of the intrusion vs size graph.

FA = 6 µm mean particle size of VA
IP = 17 µm mean particle size of VA




The pore diameter depends on factors such as tortuosity and effective porosity.

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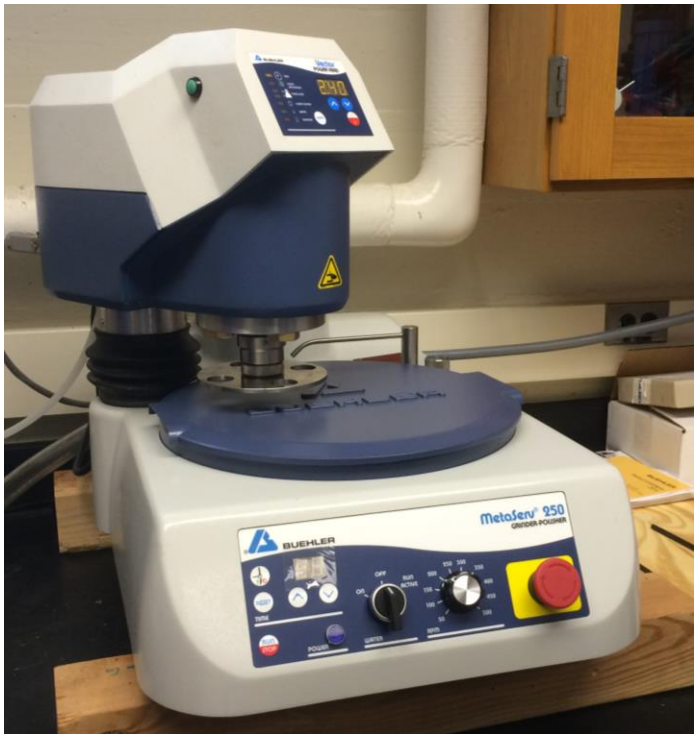
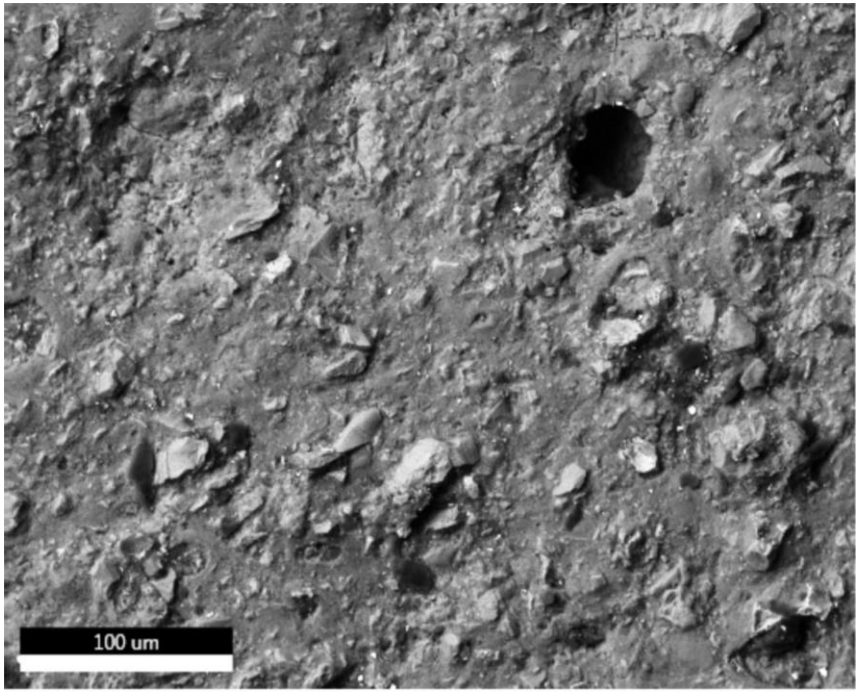
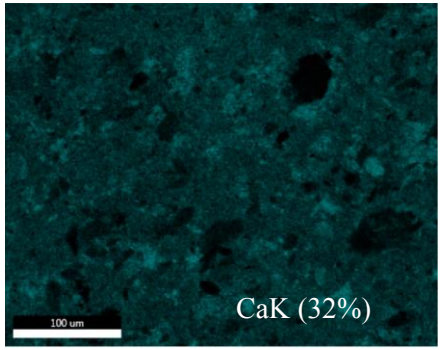
# Polishing Procedure

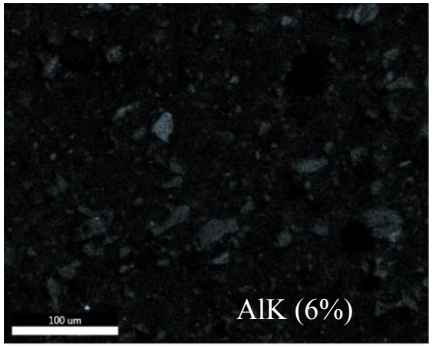


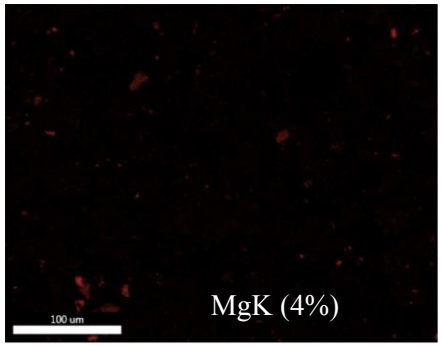

| GRIT | TIME (min) | LOAD (lb) |
|---|---|---|
| 120 | 4 | 5 |
| 240 | 8 | 6 |
| 320 | 8 | 6 |
| 400 | 8 | 6 |
| 600 | 8 | 6 |
| 800 | 8 | 6 |
| 1200 | 16 | 8 |
| 9 µm | 60 | 8 |
| 3 µm | 60 | 8 |
| 1 µm | 60 | 8 |

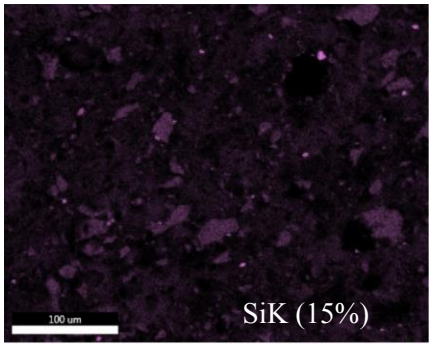
# Back Scattered Electron (BSE) Imaging with Elemental Mapping



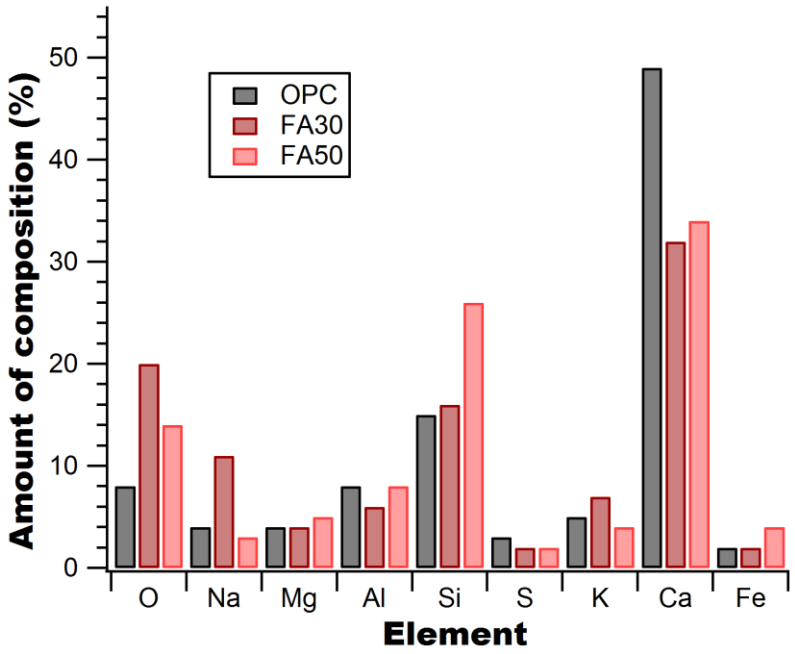



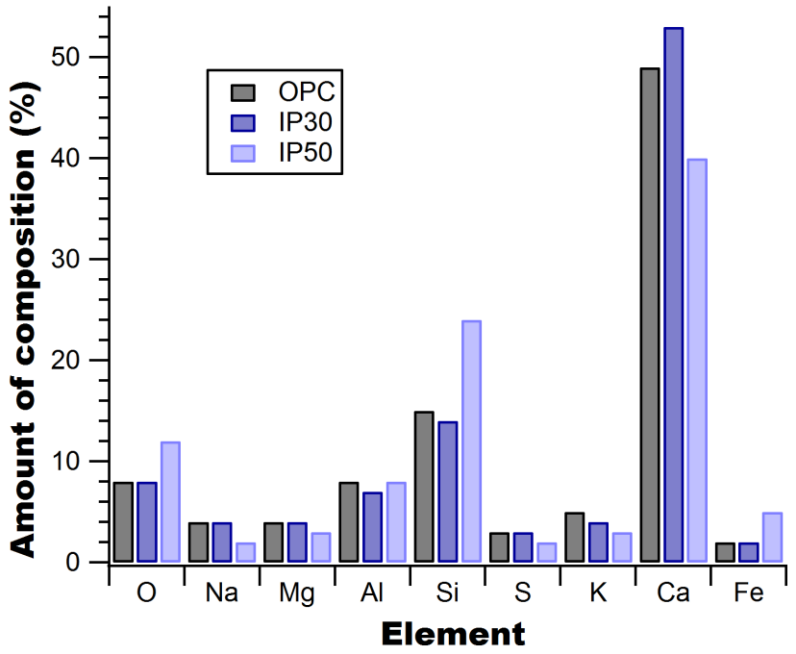



# Elemental composition (SEM/BSE)

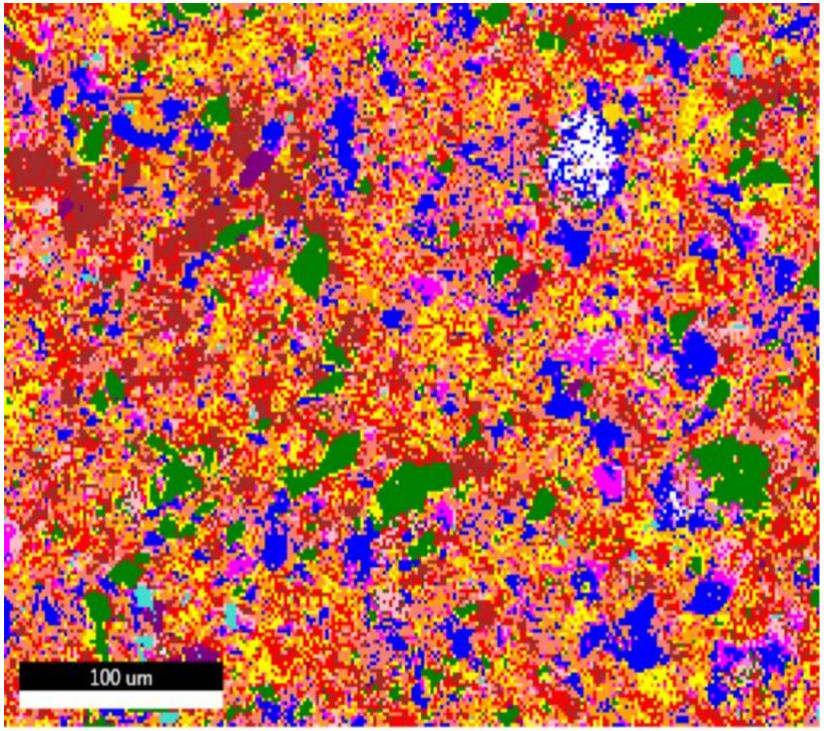
FA = 6 μm mean particle size of VA



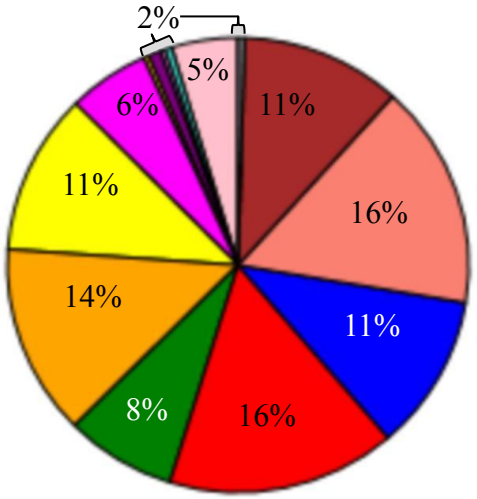

IP = 17 μm mean particle size of VA



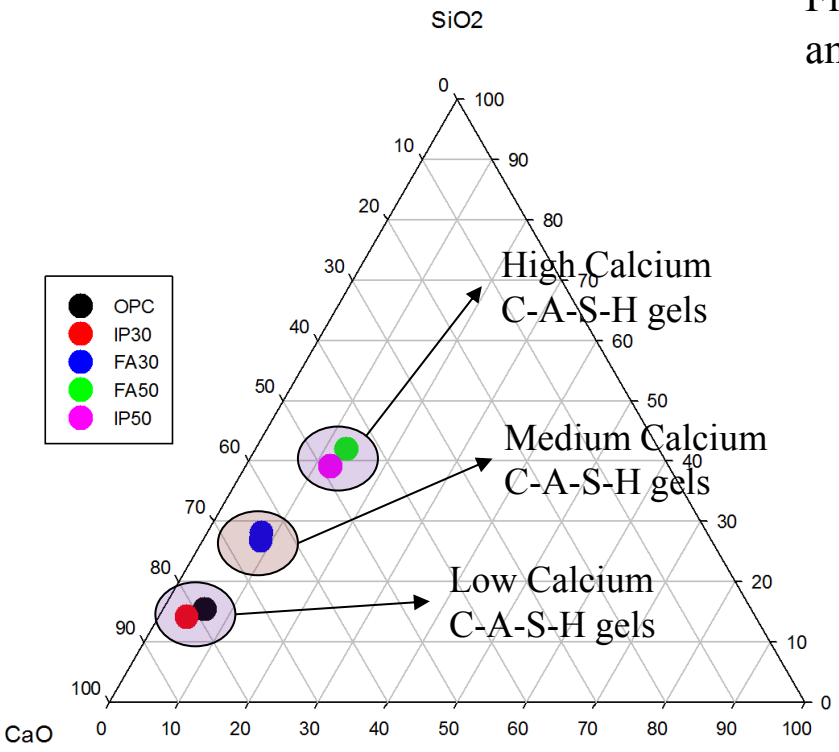
# Phase Mapping (SEM/BSE)



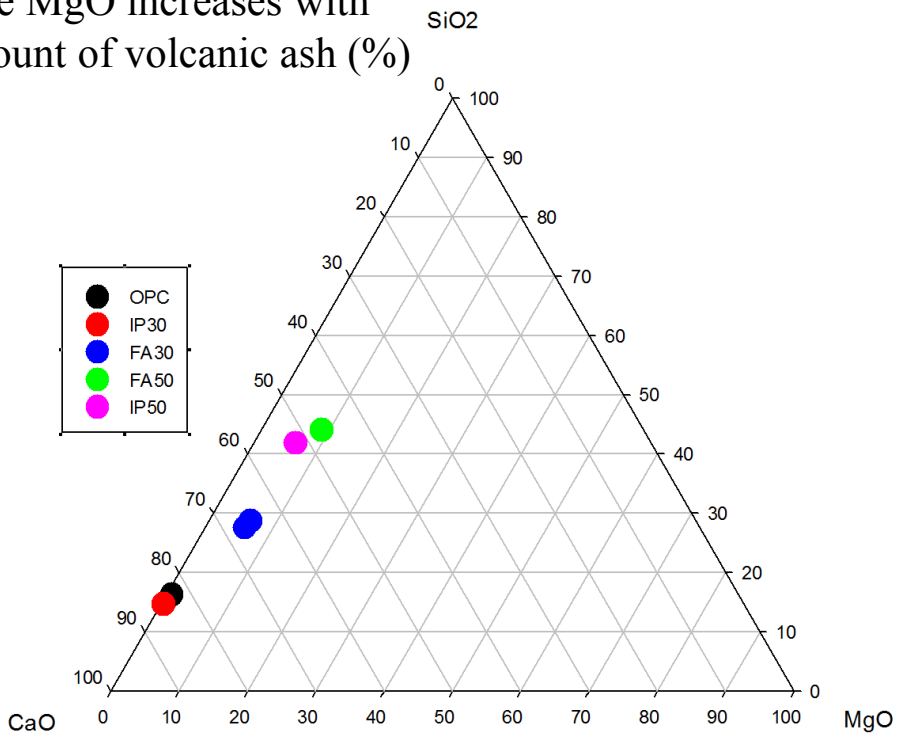

0% Unallocated (229 Pixels)
11% O K/NaK/CaK/SiK/K K (5755 Pixels)
16% CaK/O K/NaK/SiK/K K (8173 Pixels)
11% CaK/O K/K K/SiK (5670 Pixels)
16% O K/CaK/SiK/NaK/K K/AlK (8220 Pixels)
8% SiK/O K/AlK/CaK/NaK (3976 Pixels)
14% CaK/SiK/O K/NaK/K K (6946 Pixels)
11% CaK/SiK/O K/NaK/K K/AlK (5871 Pixels)
6% CaK/O K/SiK/AlK/K K/NaK (2921 Pixels)
0% SiK/CaK/O K (179 Pixels)
1% MgK/O K/SiK/CaK (451 Pixels)
0% FeK/CaK/K K/O K/SiK (168 Pixels)
1% MgK/O K/CaK/NaK/SiK (257 Pixels)
5% O K/CaK/SiK/AlK/NaK/MgK/K K (2384 Pixels)



# Ternary Phase Diagrams



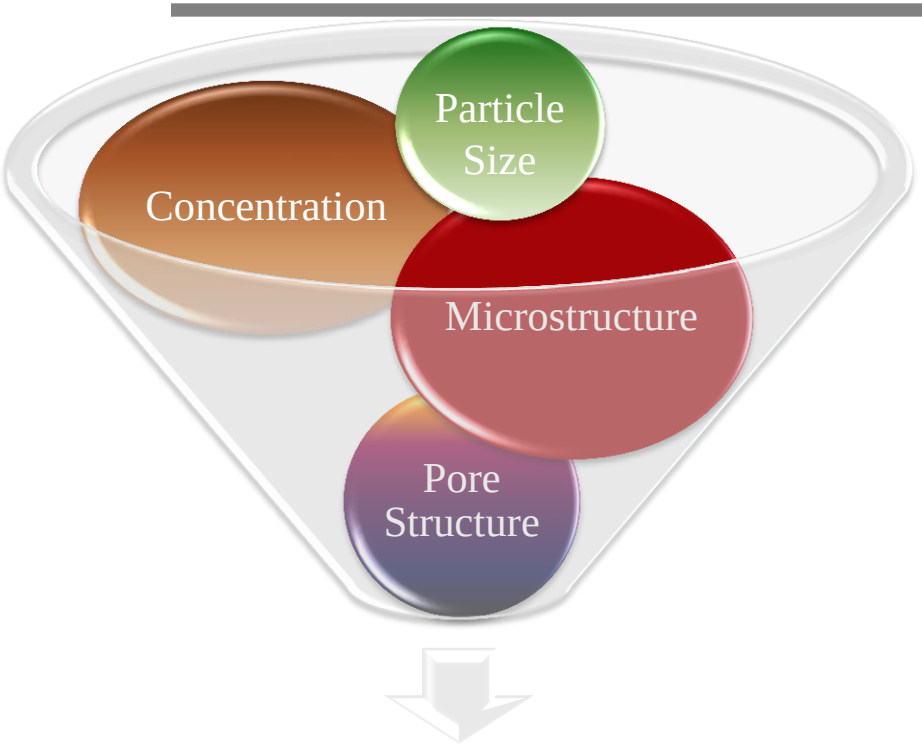

Free MgO increases with amount of volcanic ash (%)



# Ongoing work to complete this task

- Nanoindentation Polishing Procedure is being developed, and trials are being carried out.
- Since we are dealing with a complex system, it is planned to use Synchrotron XRD for the phase analysis. If the proposal is approved, we plan to perform it at Argonne National lab

# Conclusions




- The particle size and concentration of volcanic ash additive affects the microstructure and pore structure, so it can be can be chosen to optimize certain cement paste properties
- In particular, we found certain mixes that had comparable strength (FA30), phases (IP30), and porosities as our C60-OPC sample