



UNIVERSITY OF
ILLINOIS
URBANA - CHAMPAIGN

Phase-Specific Characterization of Static and Dynamic Cementitious Systems via Raman Imaging

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University of Illinois Urbana-Champaign

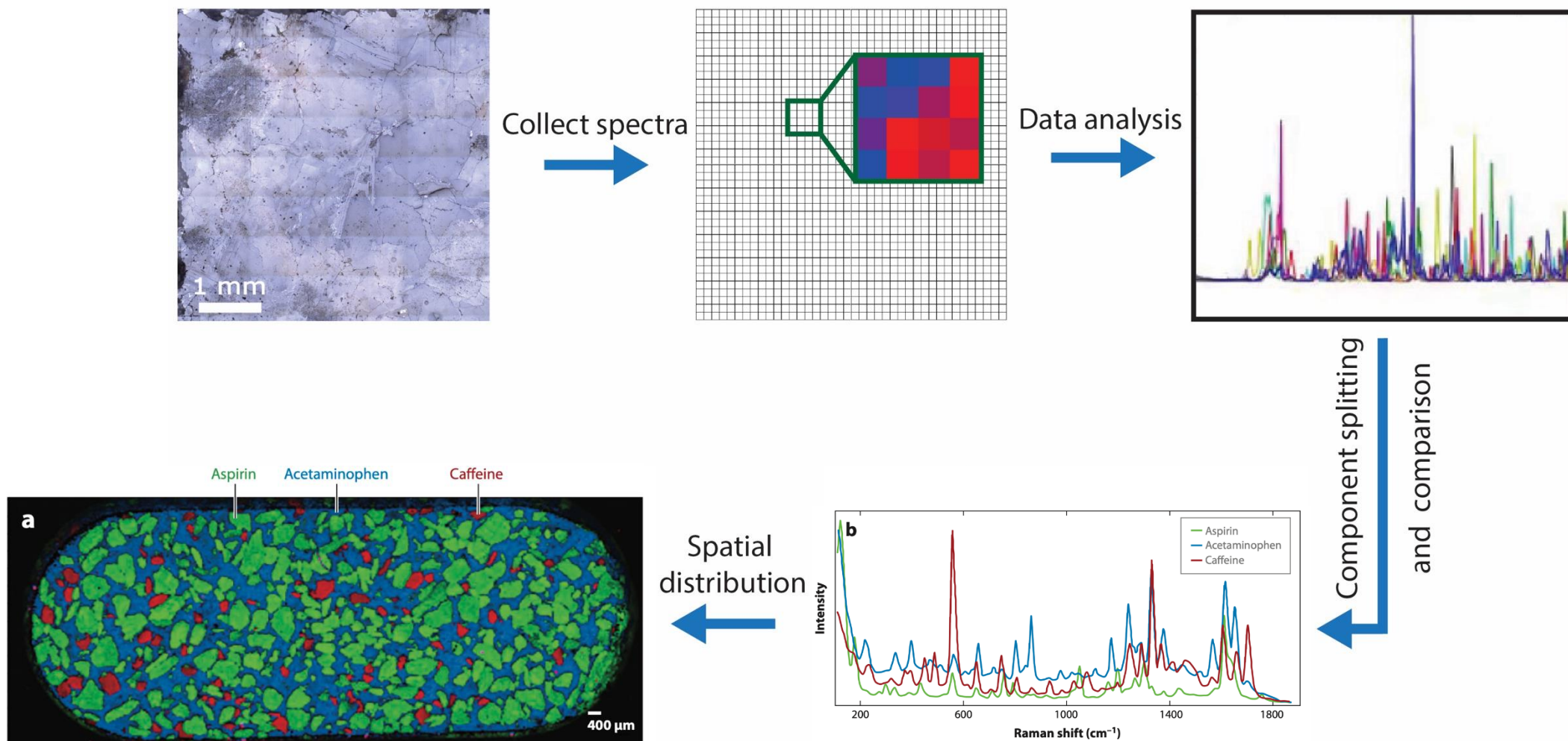
ACI Spring Convention, March 2024



Outline of this talk

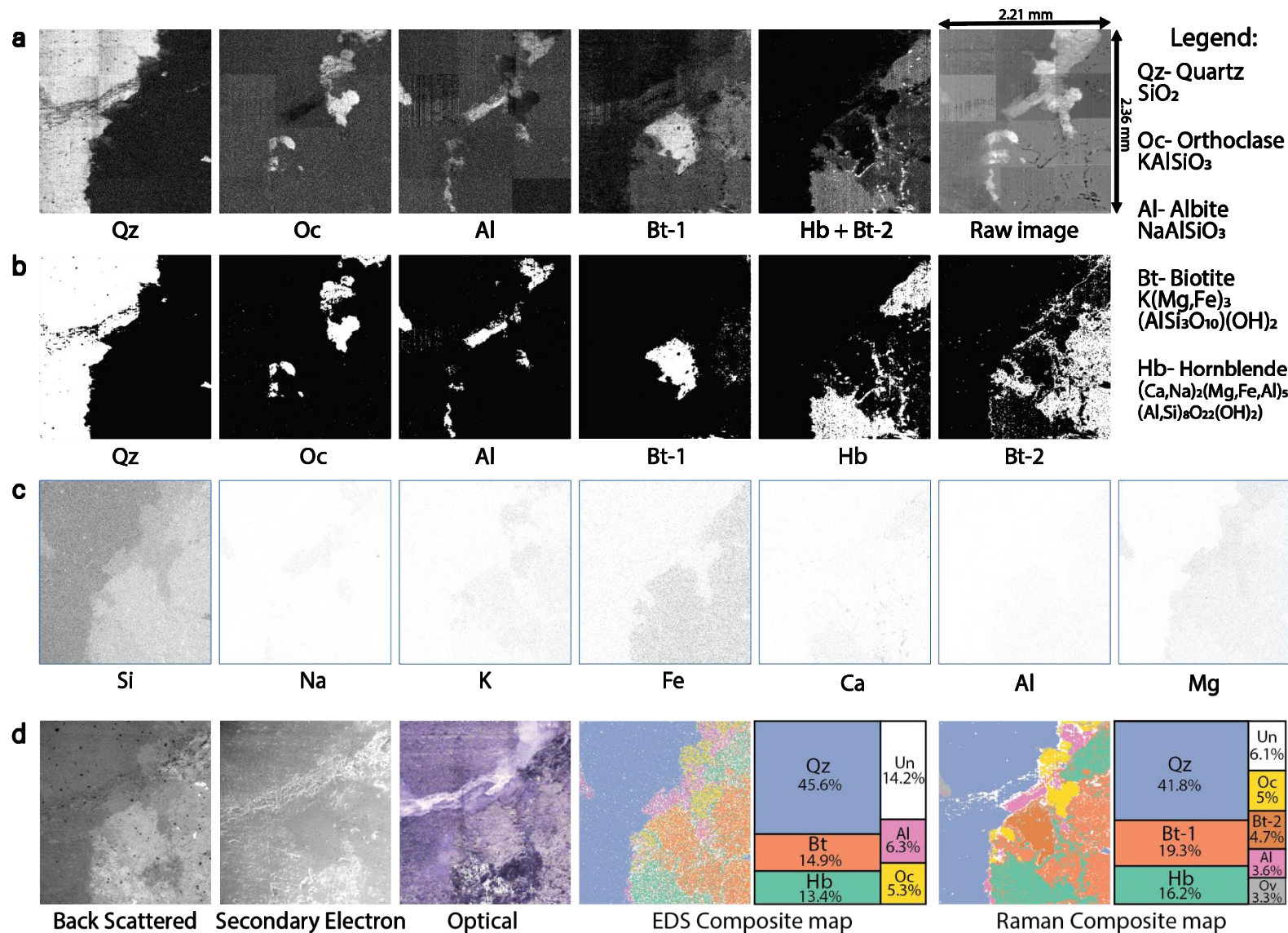
- Static Systems
 - Granite
 - Ordinary Portland Cement
 - Calcium Sulphoaluminate Cement
 - Coal-based Bottom ash
 - Waste-to-Energy Ash
- Dynamic Systems
 - Growth of alkali-silica reaction gel
 - Tracking carbonation with time

Raman Imaging for phase mapping of composite materials

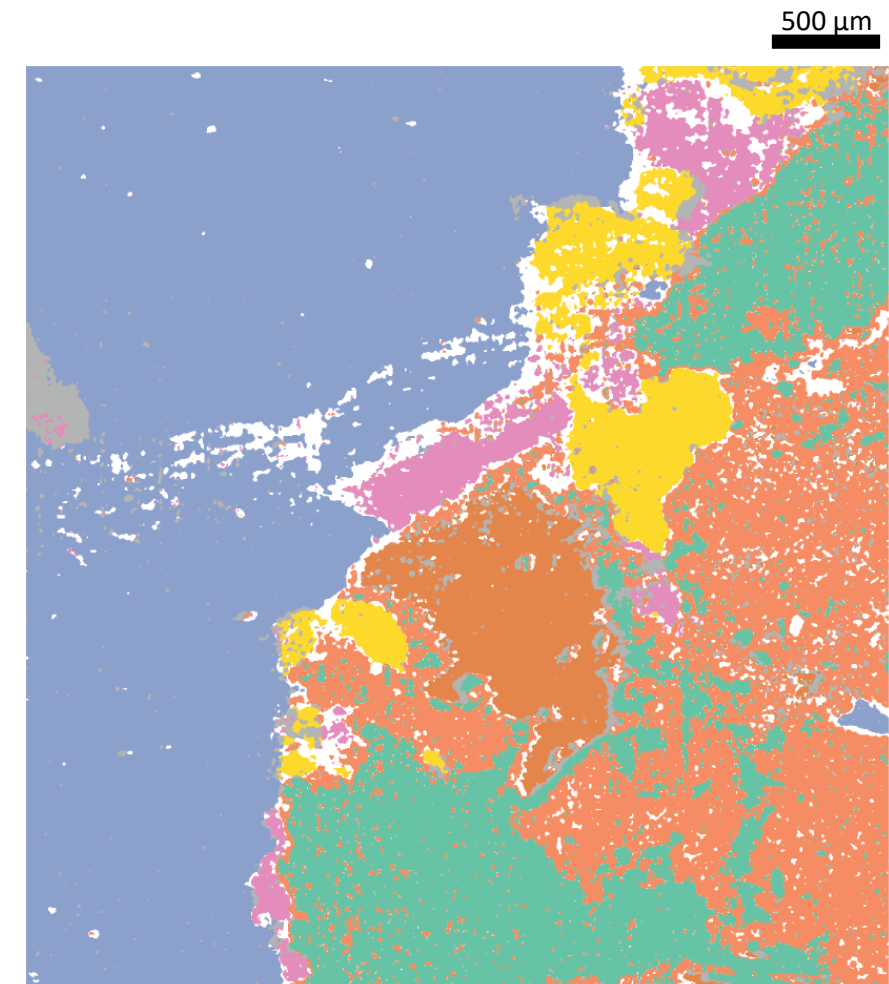


Stewart et al., *Ann. Rev. Anal. Chem.* **2012**

Granite



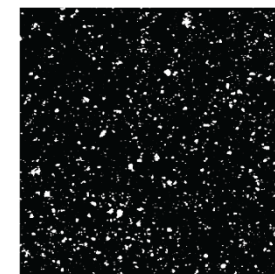
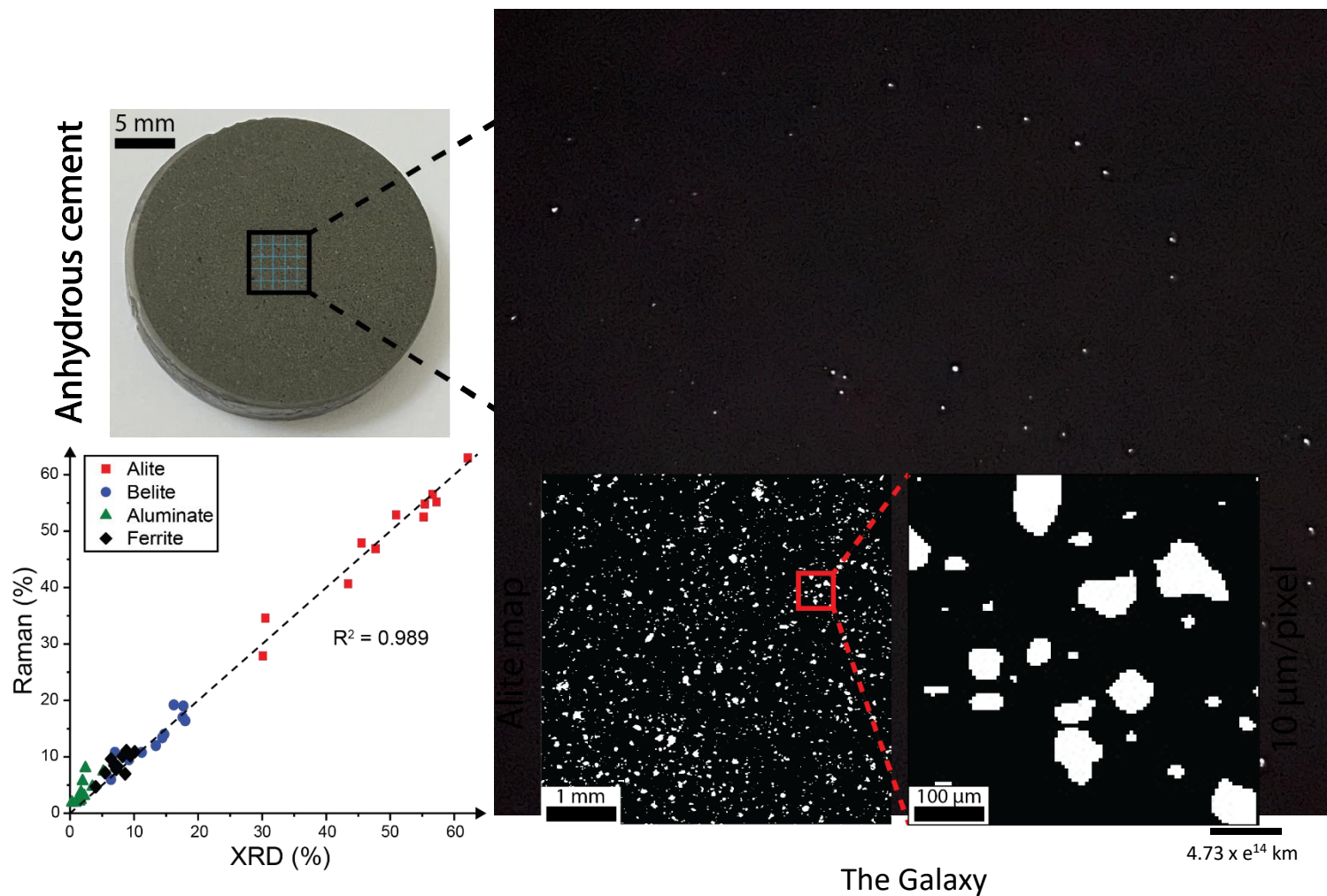
Comparing EDS and Raman Imaging



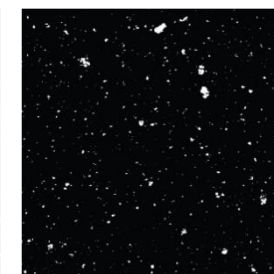
Raman imaging can result in high resolution maps with low scan times for large area scans

Polavaram and Garg, *Sci. Rep.* 2021

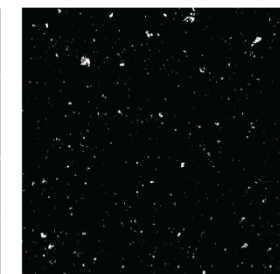
Ordinary Portland Cement



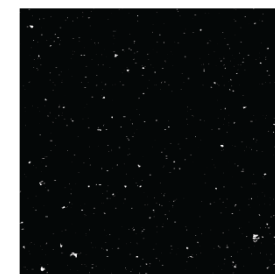
Alite (55.5 %)



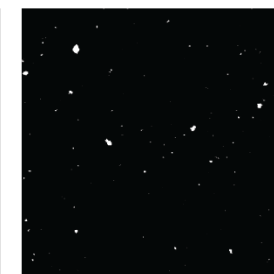
Belite (17.2%)



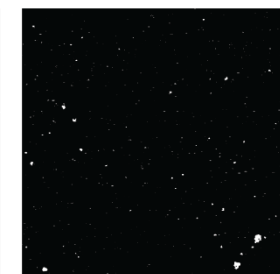
Ferrite (10.0 %)



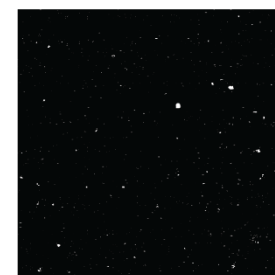
Aluminate (3.3 %)



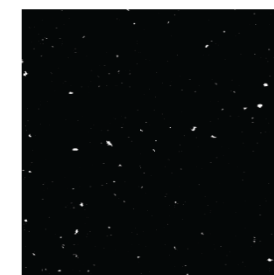
Anhydrite (3.0 %)



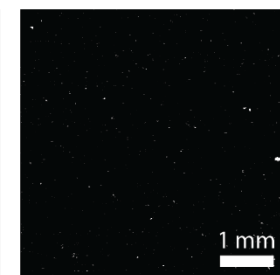
Calcite (2.8 %)



Gypsum (2.4 %)



Dolomite (1.1 %)

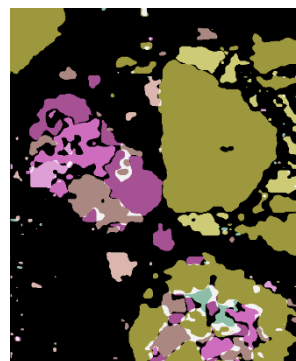
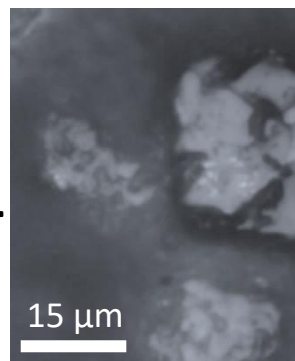


Quartz (1.0 %)

Polavaram and Garg, *Cem. Concr. Res.* **2021**

Calcium Sulphoaluminate Cement

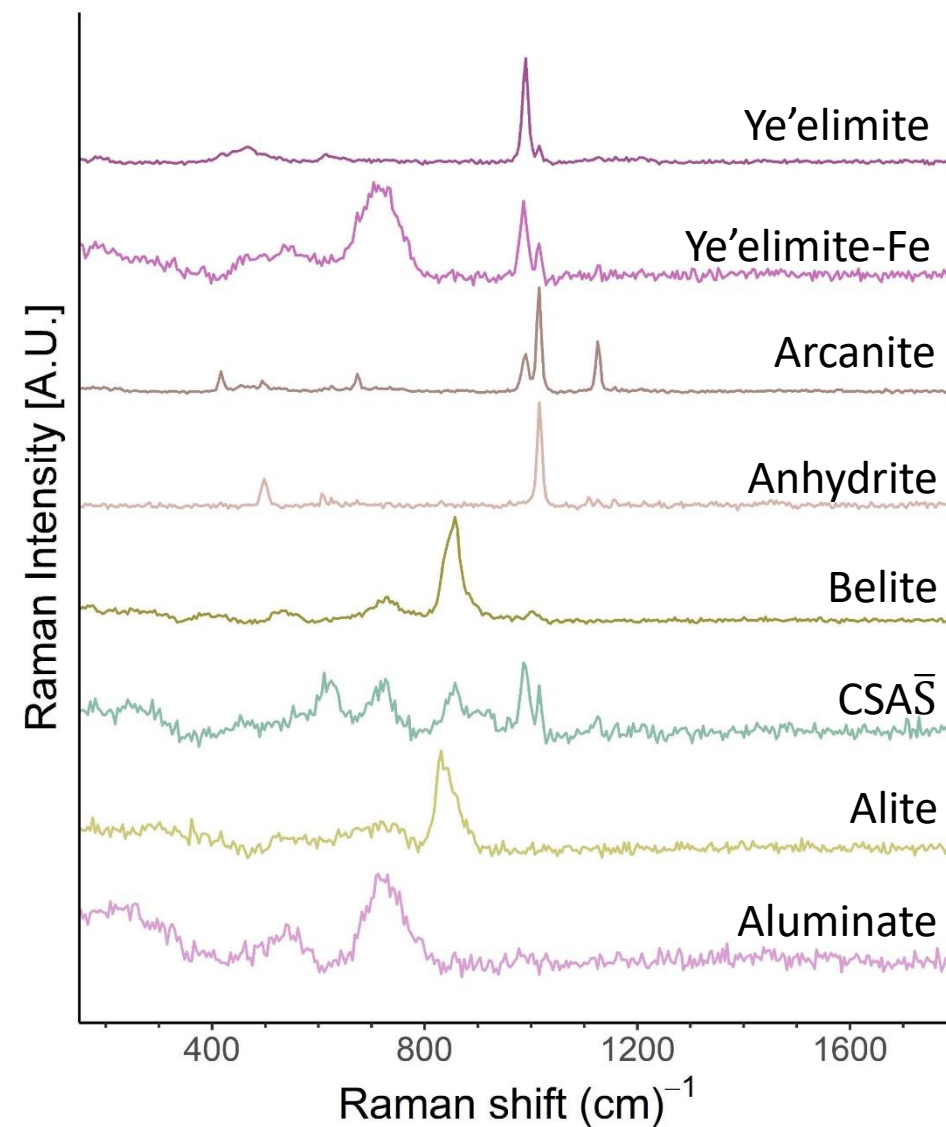
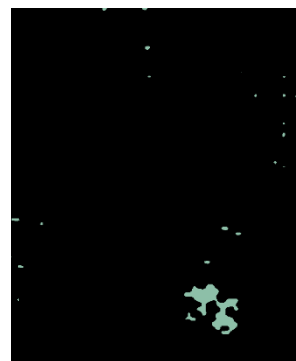
Optical



Composite Image

Arcanite: K_2SO_4

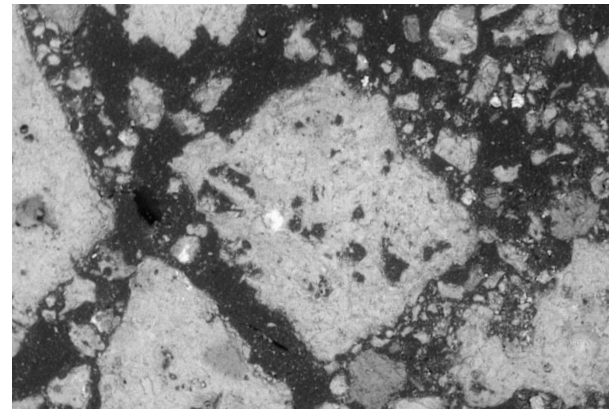
CSĀ: Calcium Silicon
Aluminium Sulfur Oxide

Kothari and Garg, *Under Review* 2024

Coal Based Bottom Ash

(10 μm resolution per pixel)

SEM-BSE

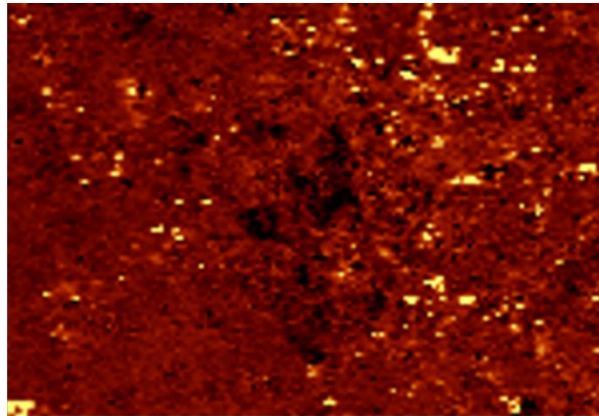


200 μm

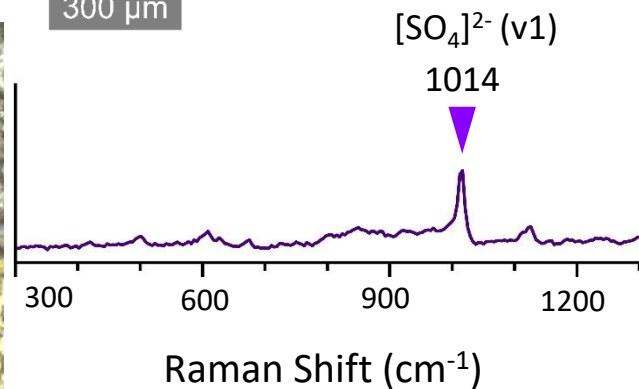
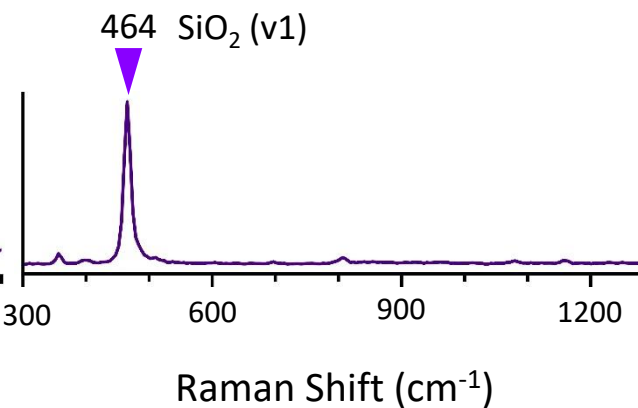
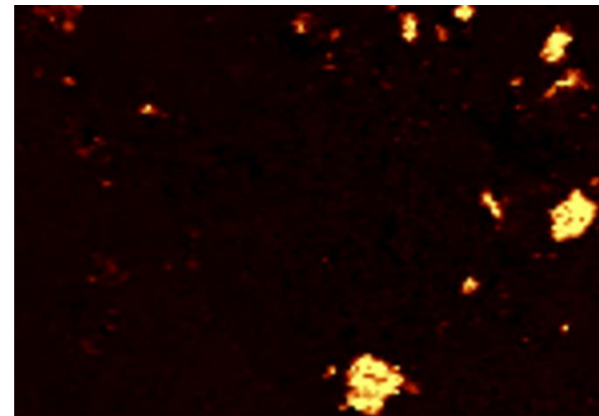
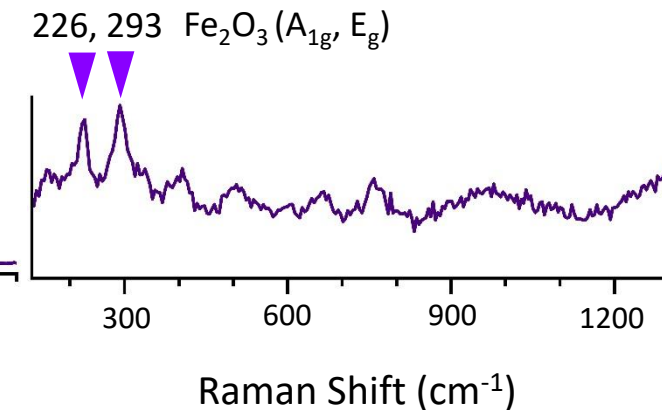
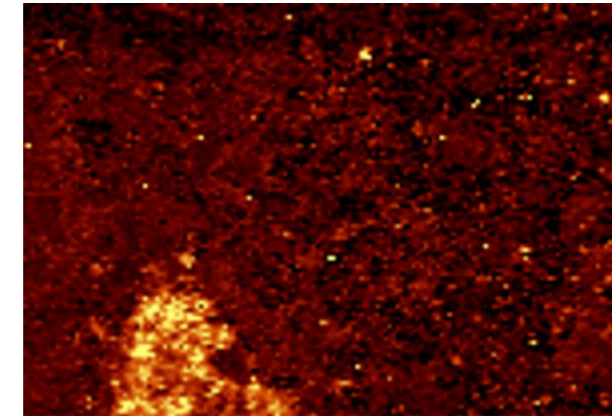
Optical



Anhydrite



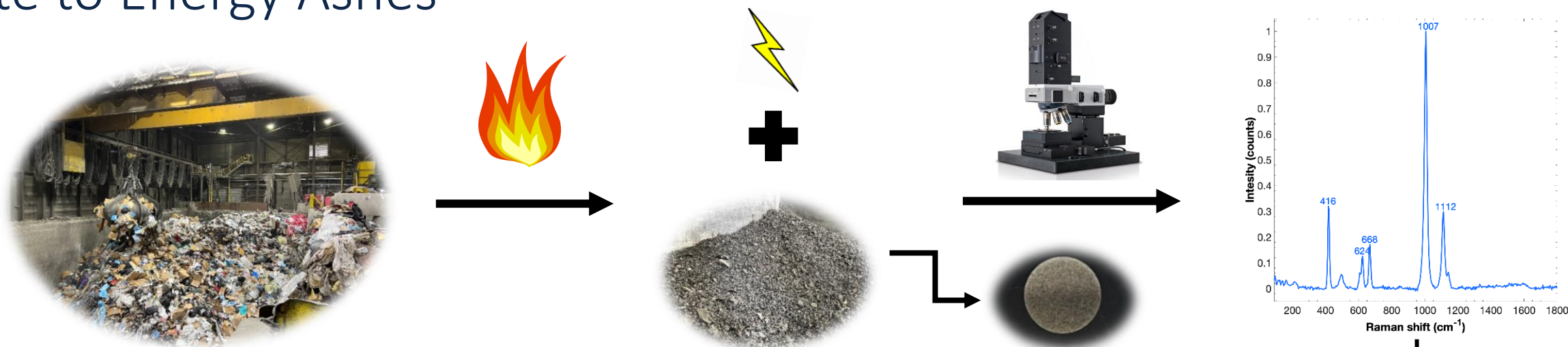
300 μm

Quartz SiO_2 Hematite Fe_2O_3 

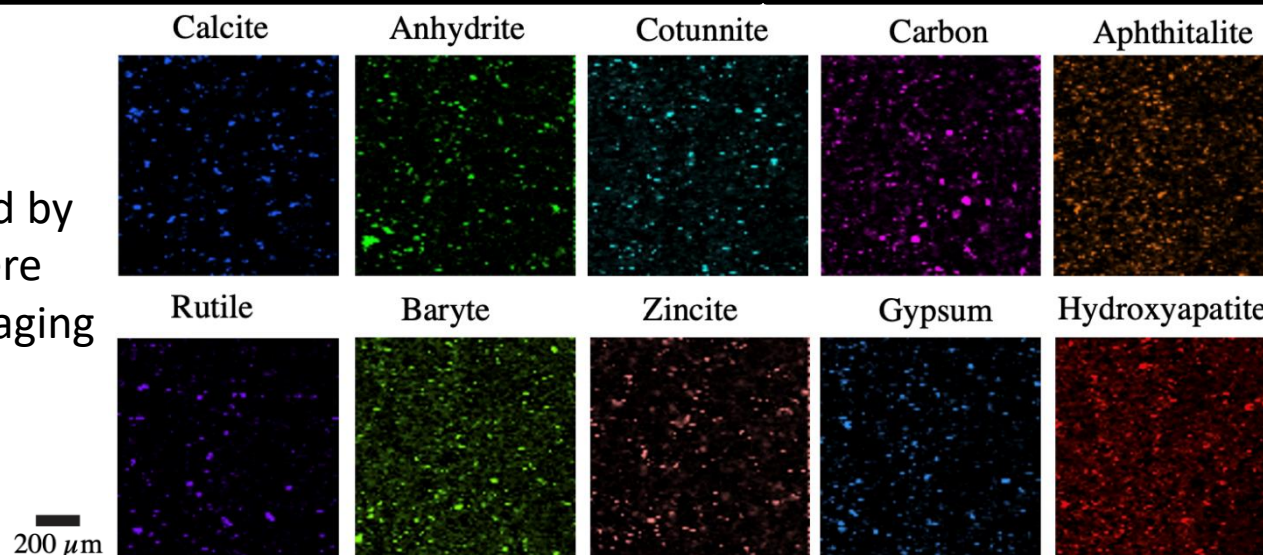
Phases are consistent with spectra from RRUFF database

Witte and Garg, *Under Review* 2024

Waste to Energy Ashes



6 phases were detected by XRD, and 10 phases were detected by Raman imaging



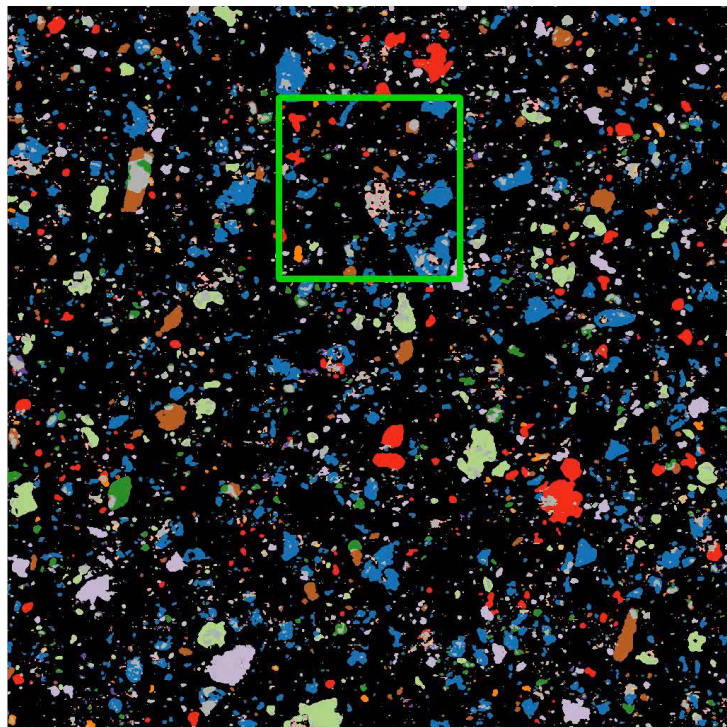
Cotunnite (PbCl_2) and baryte ($\text{Ba}(\text{SO}_4)$) are only detected by Raman imaging and contain toxic elements (lead and barium)

Samouh et al, *J. Hazard. Mater.* 2023

Size and Shape Distribution of Individual Clinker Phases

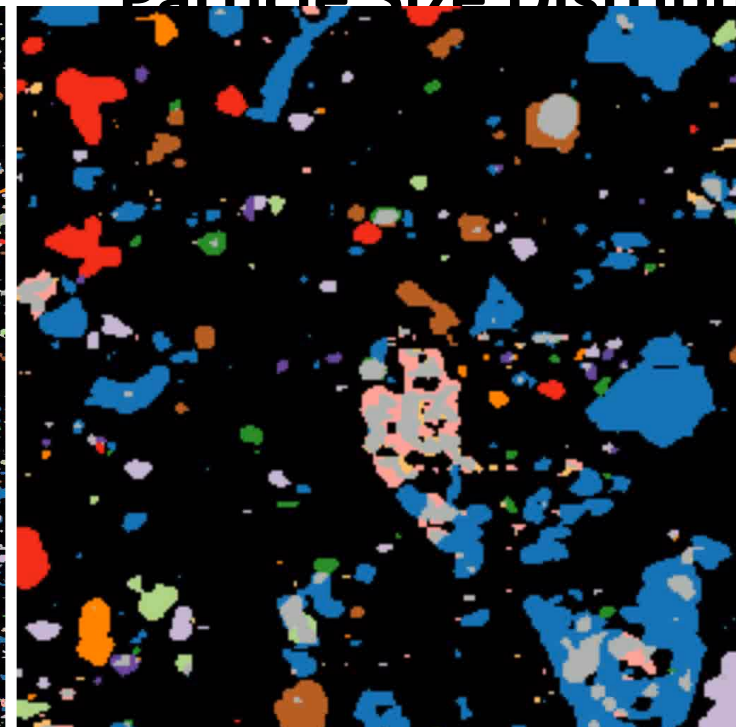
Anhydrous Cement

Raman Image

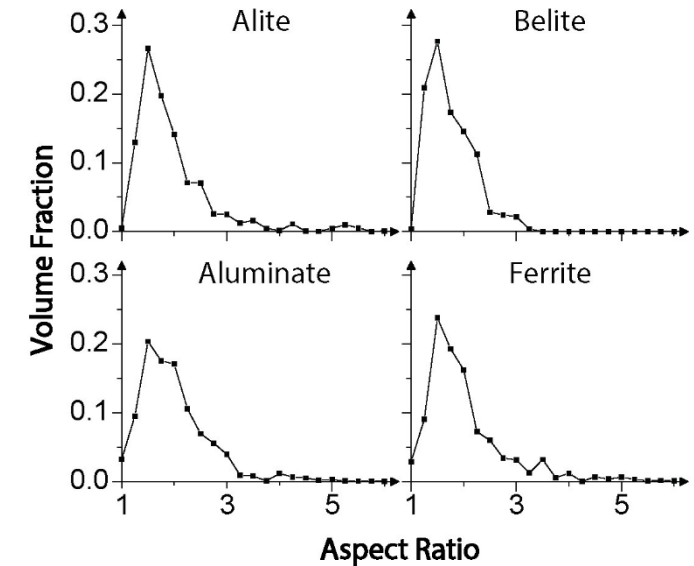
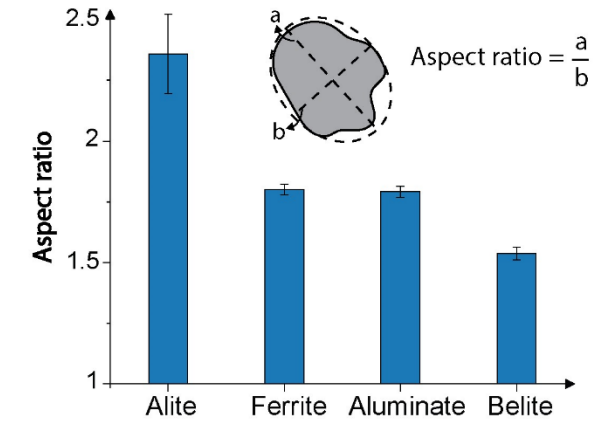
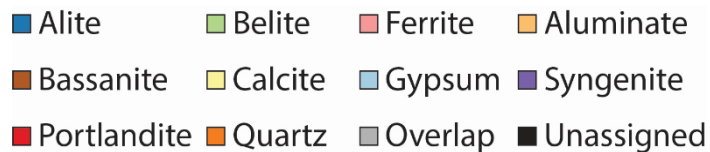


100 μm

Raman Image - Inset
Phase-Specific
Particle Size Distributions



10 μm



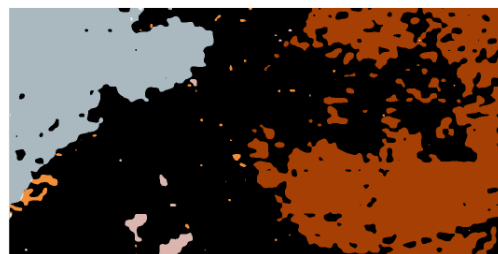
Polavaram and Garg, *J. Phys. Chem. C*. 2023

Imaging the Growth of Alkali-Silica Reaction Gel – Raman vs. EDS

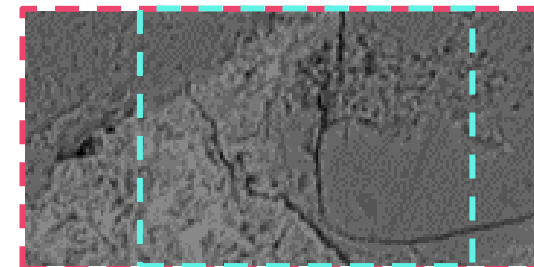
Optical



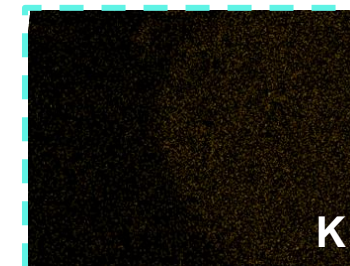
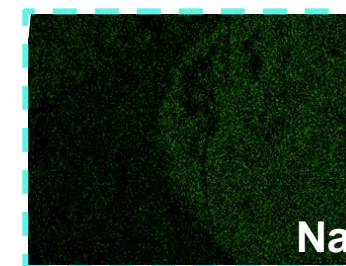
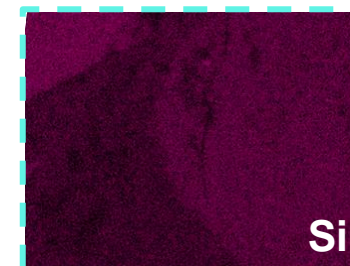
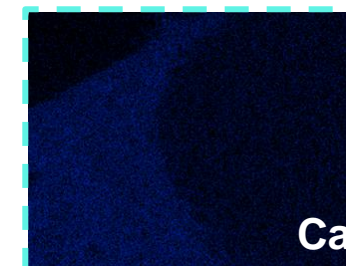
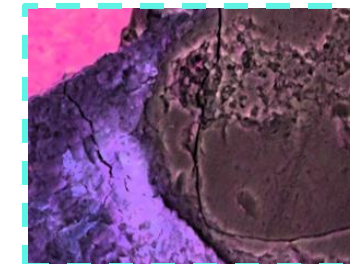
Combined Raman



SEM-BSE



SEM-EDS

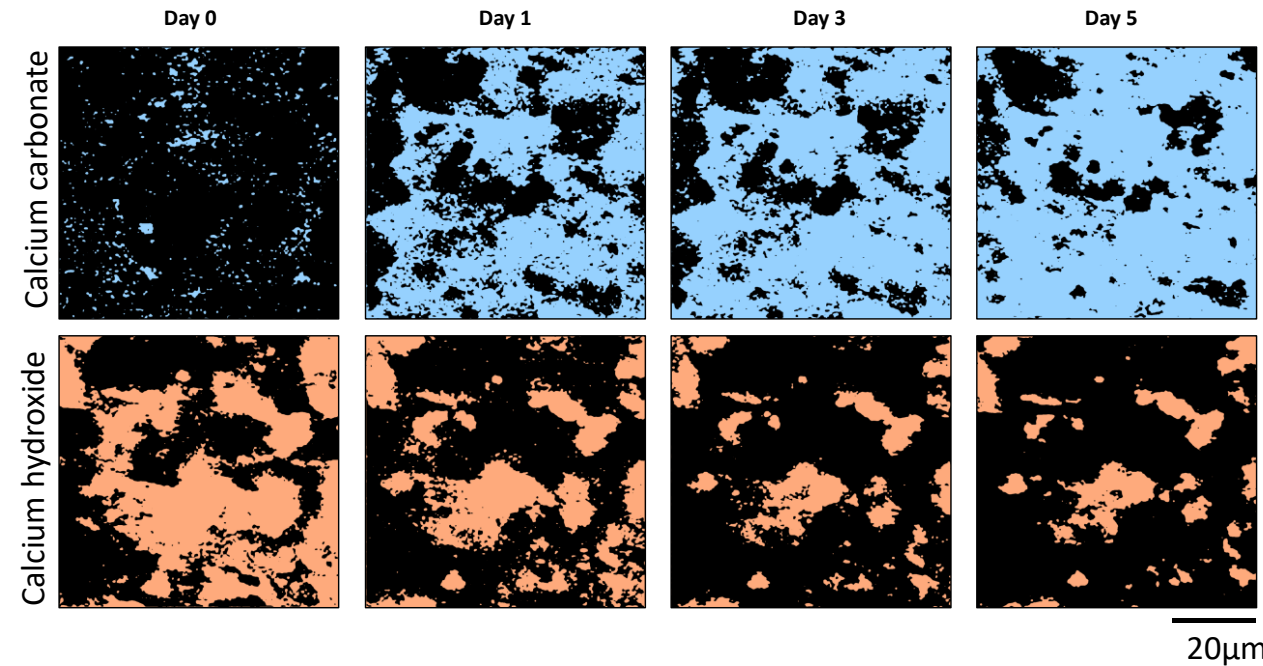


50 μm

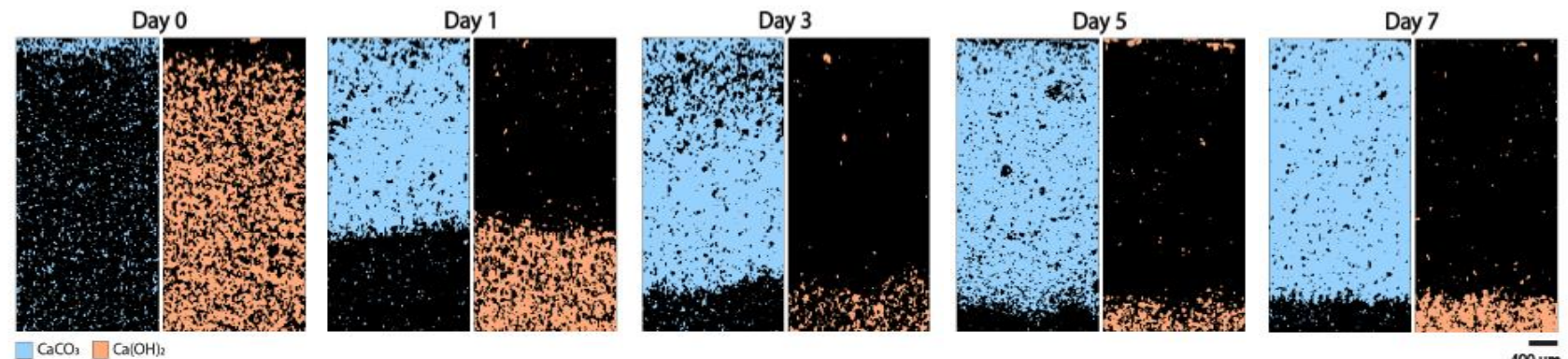
Kothari and Garg, *In Preparation* 2024

Tracking Carbonation with Time in 2D: Surface and Cross-Section

Surface study

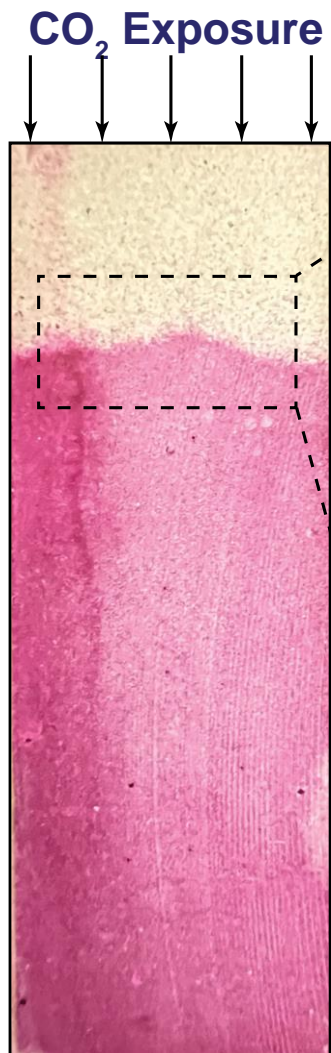


Cross-sectional study

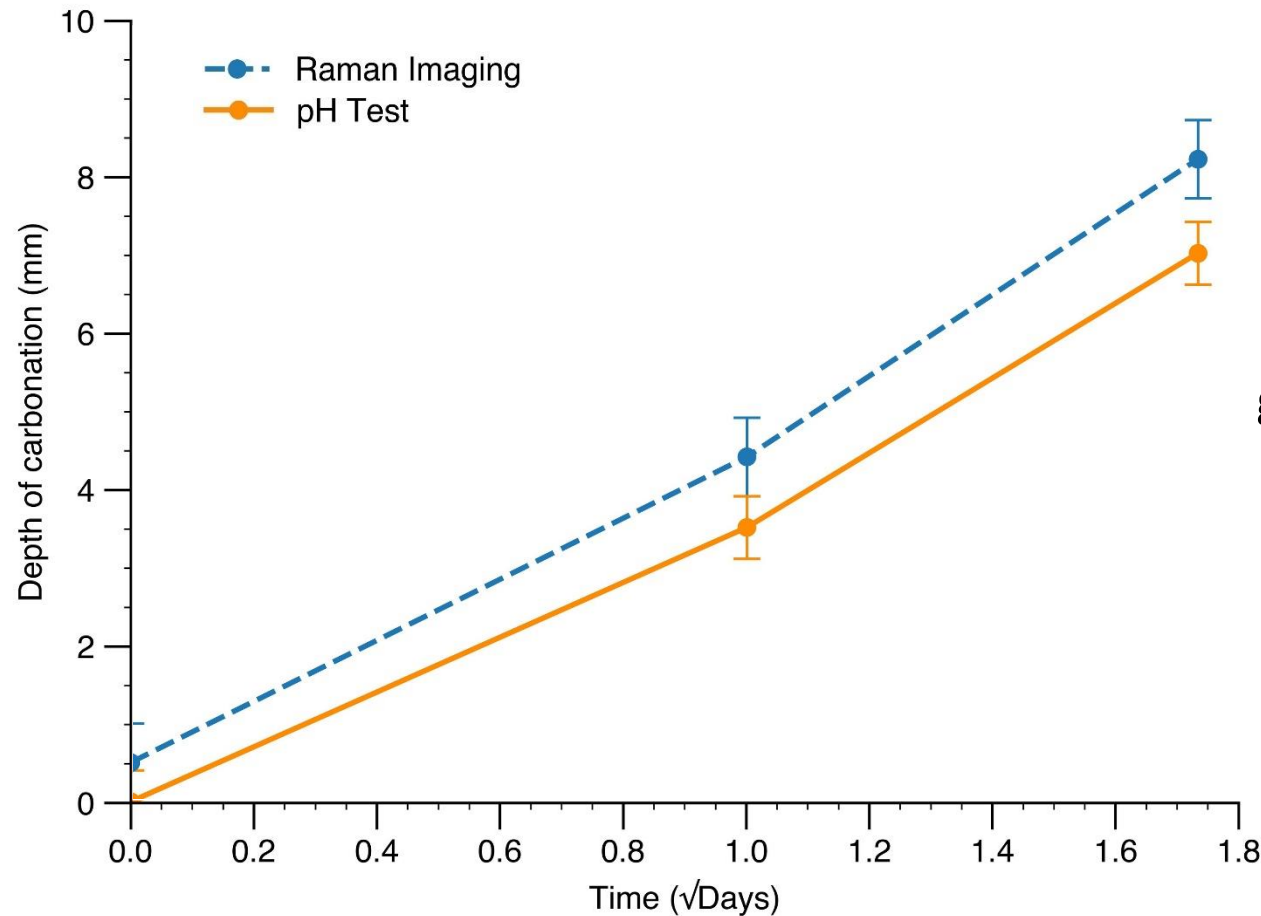


Srivastava and Garg, *J. Raman Spectroscopy* 2023

Tracking Carbonation with Time in 2D: Mapping Calcite and Portlandite



Imaging



Carbonation depth **~1.5 mm** greater than the depth obtained via phenolphthalein test

Rathnakumar and Garg, *In Preparation* 2024

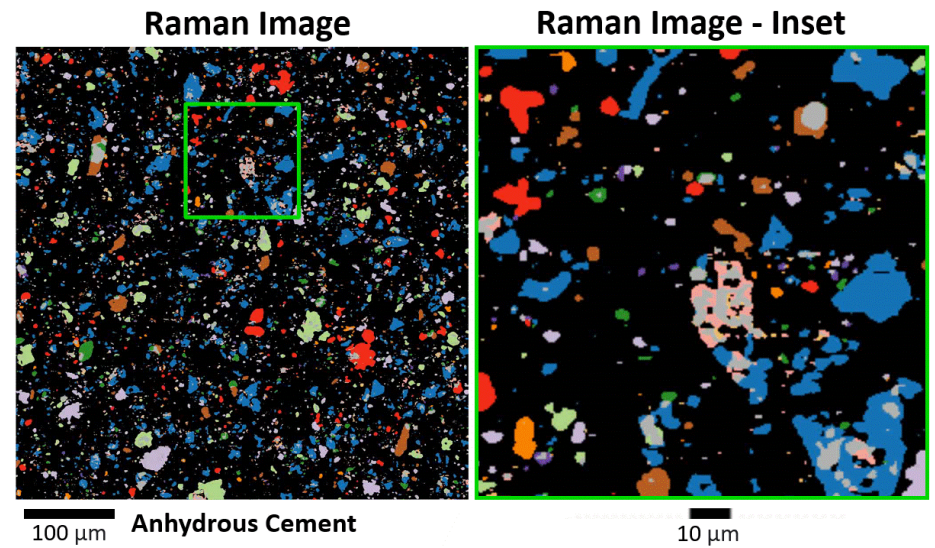
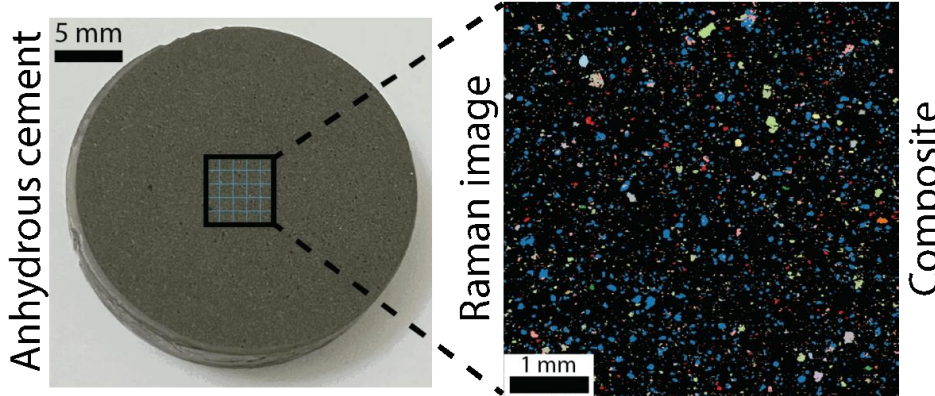
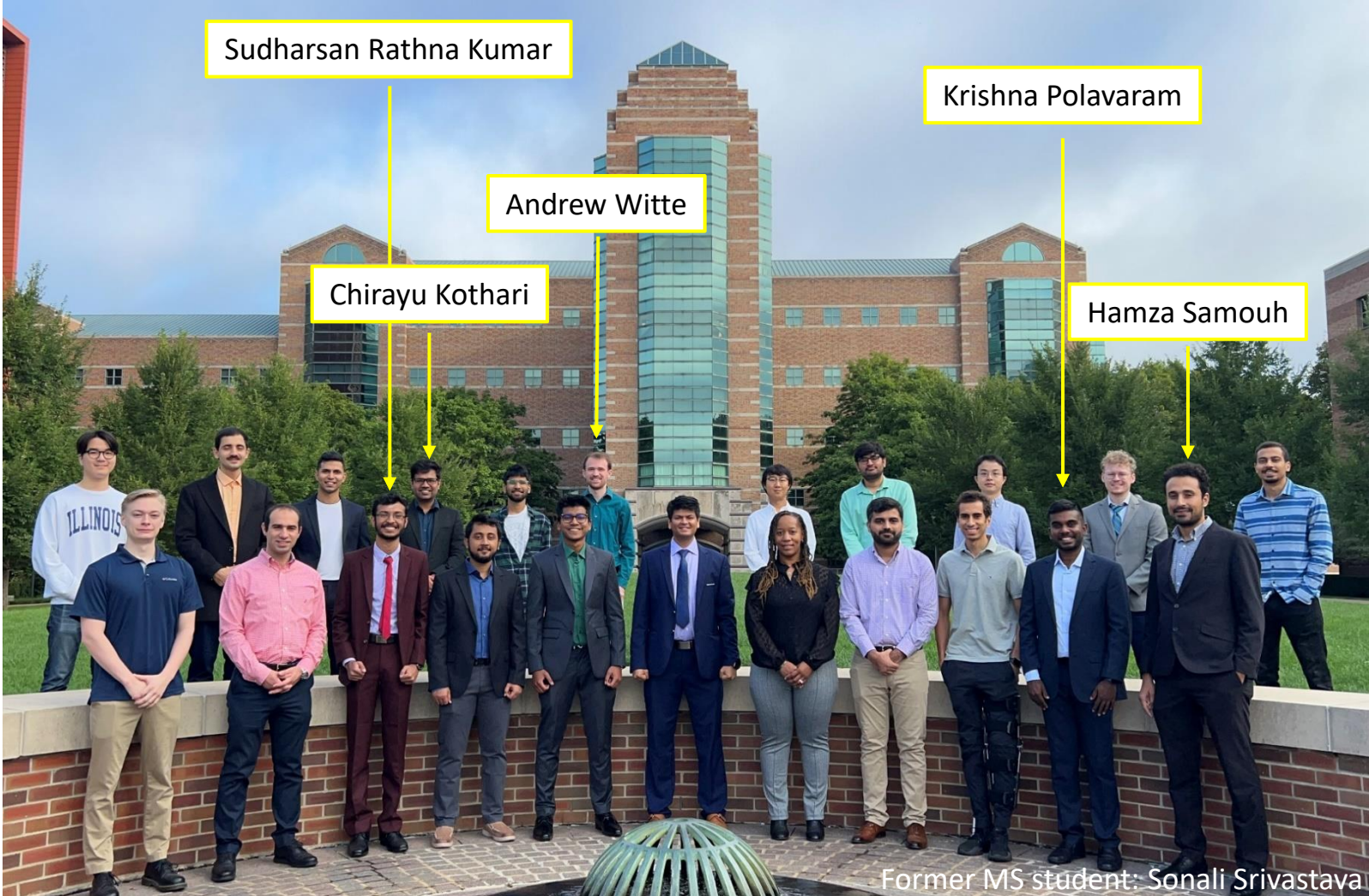
Contributions

- This imaging approach can be used to obtain high-fidelity and high-resolution (micron scale) phase images of samples over large areas (mm scale).
- Raman imaging can be used to quantify and characterize various static, multi-phase heterogenous systems such as cements, aggregates, and SCMs.
- In addition, dynamic phenomena such as ASR and carbonation can be spatiotemporally tracked using Raman imaging.
- Finally, in the near future, combined SEM-Raman imaging approach on these systems will open up new pathways towards advanced characterization of complex systems.

Polavaram and Garg, *Sci. Rep.* **2021**
Polavaram and Garg, *Cem. Concr. Res.* **2021**
Srivastava and Garg, *J. Raman Spectrosc.* **2023**
Polavaram and Garg, *J. Phys. Chem. C.* **2023**
Samouh et al, *J. Hazard. Mater.* **2023**

Kothari and Garg, *Under Review* **2024**
Witte and Garg, *Under Review* **2024**
Rathnakumar and Garg, *In Preparation* **2024**
[Polavaram et al, 77th RILEM Annual Week 2023
Raman Imaging of Cementitious Systems](#)

Acknowledgements



Garg Group
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