



# Characterization of Cement-Geomaterial Interfaces for Subsurface Applications

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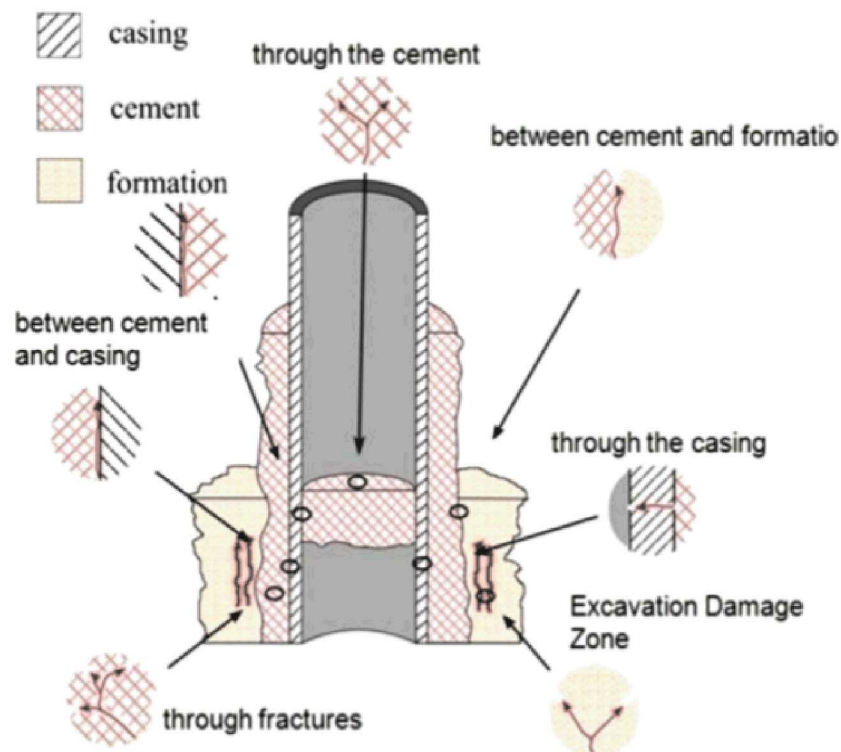
August 2018





# Introduction

- Carbon dioxide capture and storage (CCS) is a technology that captures and compresses  $\text{CO}_2$  at industrial locations, transports the supercritical fluid, and stores  $\text{CO}_2$  in subsurface geologic deposits.
- Important considerations:
  - Scale
  - Material
  - Well integrity
  - Suitable geologic structures



Bai et al., 2016



# Objective

- The objective of this study is to characterize geomaterials through pore structure analysis and more advanced electron image analysis to offer baseline data and basic insights to the chemistry that will affect wellbore stability.
- Specifically, this characterization will provide input parameters for reactive transport simulations, as well as validation of these simulations.

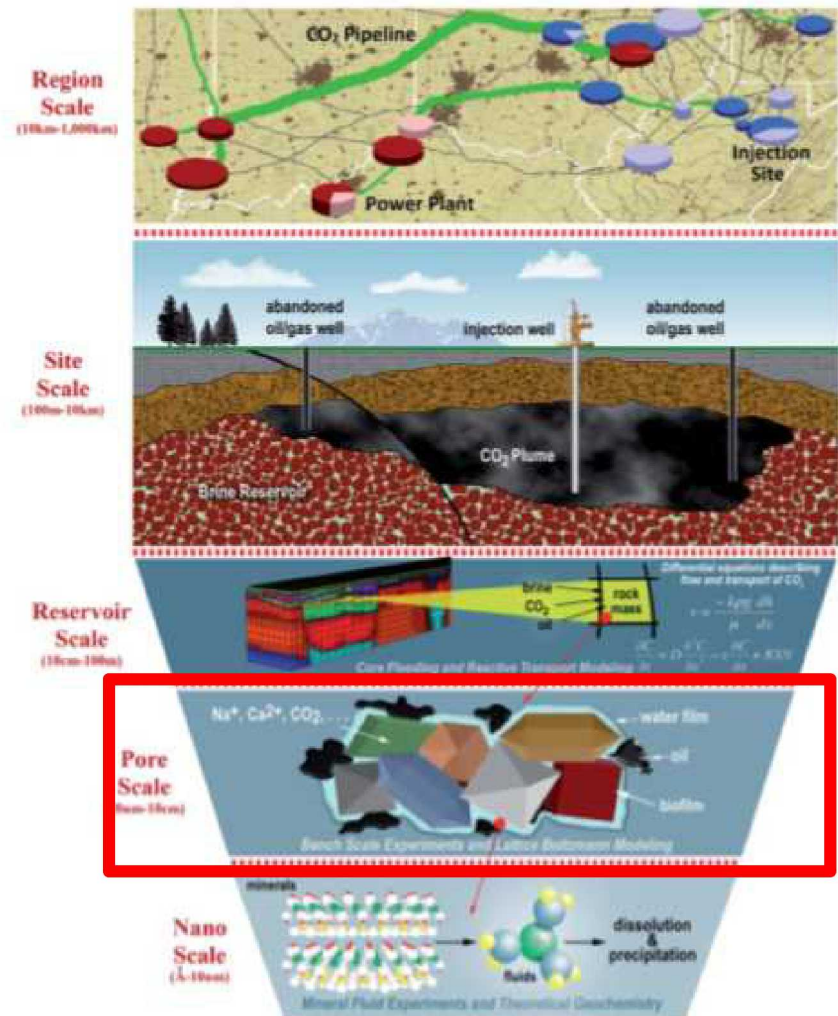


Fig. 1 Illustration of the multiscale aspects of CO<sub>2</sub> storage science.

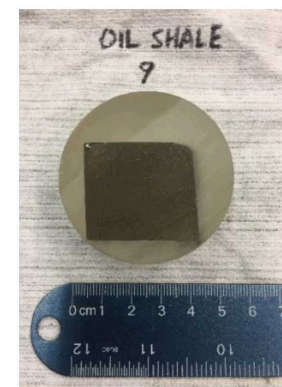
Middleton et al., 2012





# Materials

- Five different geologic samples were used in this study.
  - Samples consisted of sedimentary and carbonate rocks, from the Cretaceous age, in the Mount Scopus formation in the Negev Desert of Israel.
  - Sandstone (SST), chalk (C), marl (M), phosphorite (P), and oil shale (OS).
  - Contain large amounts of organic material.
- Two types of rock samples will be referenced: baseline material and samples that have undergone EPA method 1315 leaching test.





# Methods

- Nitrogen Gas Adsorption Experiment
  - Utilizing BET theory, gives the measurement of specific surface area of materials through gas ( $N_2$ ) adsorption.
- MIP
  - A useful method to characterize porous material
  - Samples are evacuated to remove air, then sample cell is filled with mercury as the pressure in the system slowly increases, intruding mercury into smaller and smaller pore spaces
- EPA Method 1315
  - A mass transfer rate, tank leaching test.
- Backscatter SEM
  - A TESCAN VEGA3 SEM and EDS software EDAX TEAM was used in low vacuum for imaging and elemental analysis.
- Petrographic Microscope
  - Leitz Orthoplan microscope with a Leica DFC425 camera and Leica Application Suite software





# Results and Discussion

- BET and MIP Data

Rock	BET Surface Area (m <sup>2</sup> /g)	Porosity (%)	Average Pore diameter (um)	Total Pore Area (m <sup>2</sup> /g)	Total Intrusion Volume (mL/g)	Bulk density (g/mL)	Tortuosity (Calc)	Cumulative Pore Volume (mL/g)
Sandstone	0.4926	8.2597	0.3953	0.345	0.0341	2.4201	26.9447	0.0314
Marl	5.5523	31.6708	0.13764	4.983	0.1715	1.8471	87.5473	0.1318
Chalk	0.6428	6.8169	0.10467	1.056	0.0276	2.4672	67.6575	0.253
Phosphorite	15.0786	34.8568	0.03646	26.415	0.2408	1.4476	4.3768	0.121
Oil Shale	15.9254	32.8374	0.02751	25.318	0.1741	1.8858	64.966	0.1018

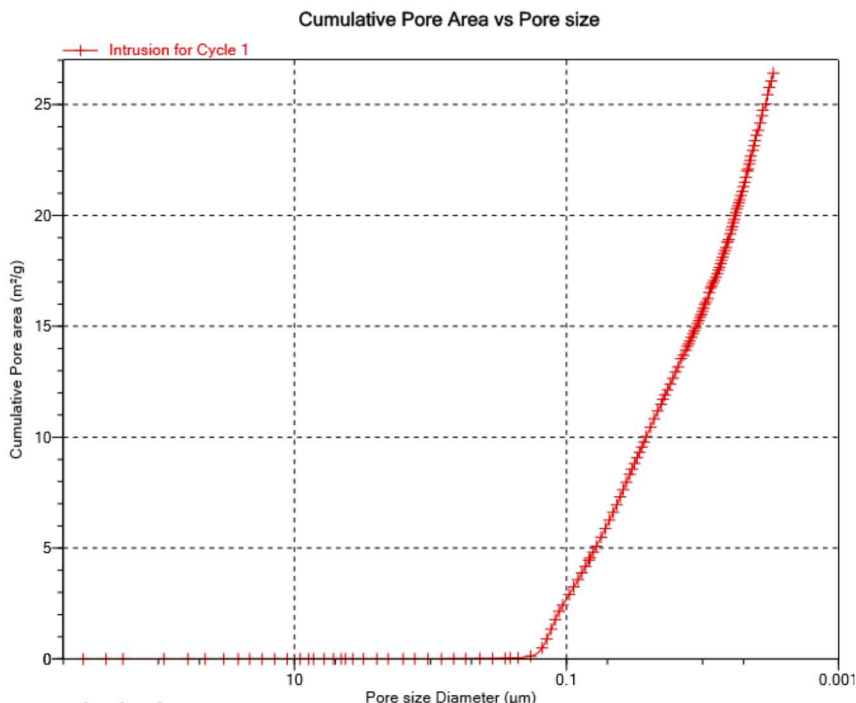
- Marl, Phosphorite, and Oil Shale = highest porosity % and BET surface areas
- Chalk and Sandstone = lowest porosity % and BET surface areas

Note: MIP data obtained from Micromeritics Instrument Corporation

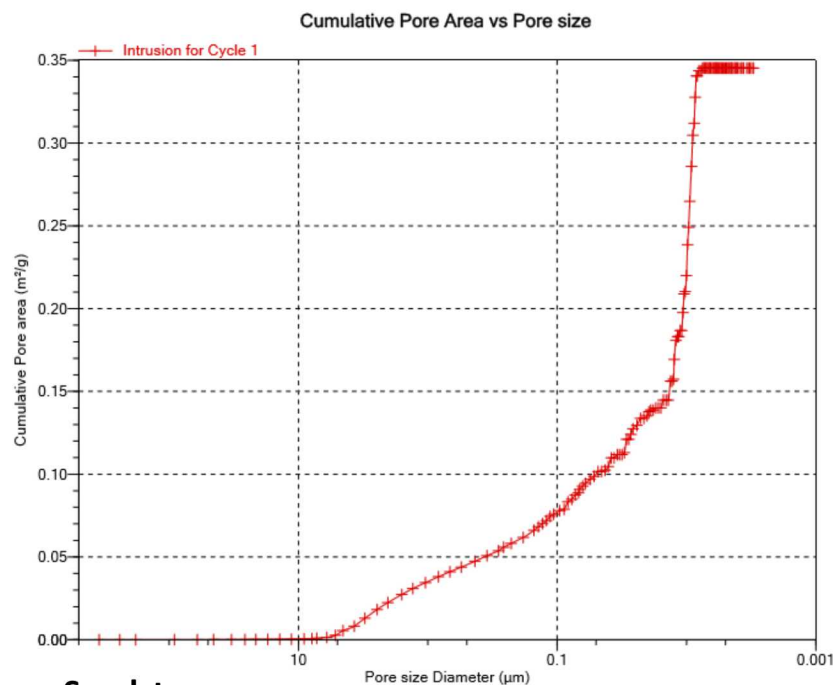




# Results and Discussion



Oil Shale



Sandstone

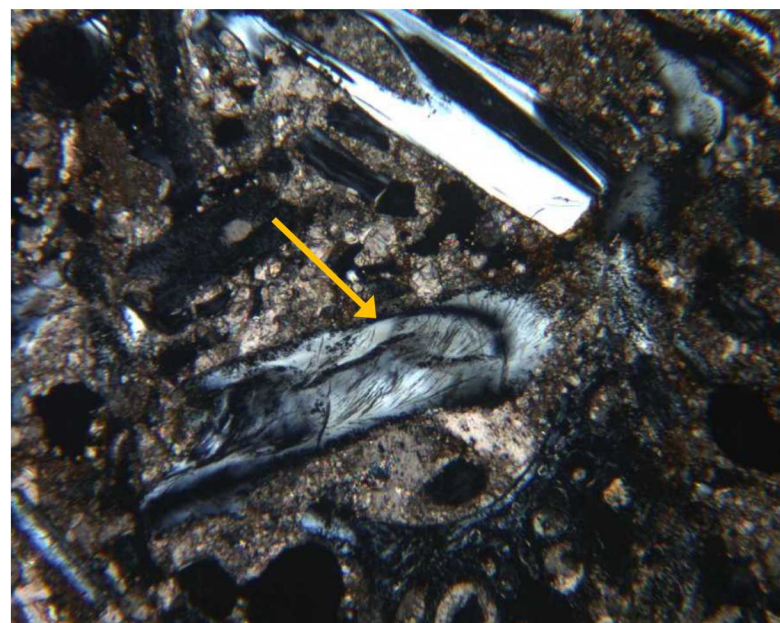
## Summary:

- OS, P, C, M – all comprised of mostly micropores (pore size radius between 0.1 – 0.001 microns)
- SST – some transitional pores, fewer micropores

Note: MIP data obtained from Micromeritics Instrument Corporation



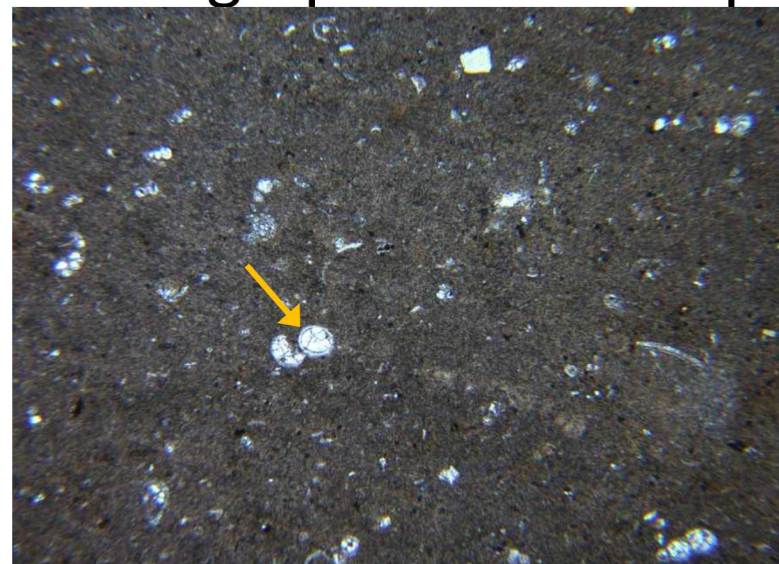
## Results and Discussion: Petrographic Microscope



25.4 microns

**Phosphorite**

**Marl**



25.4 microns

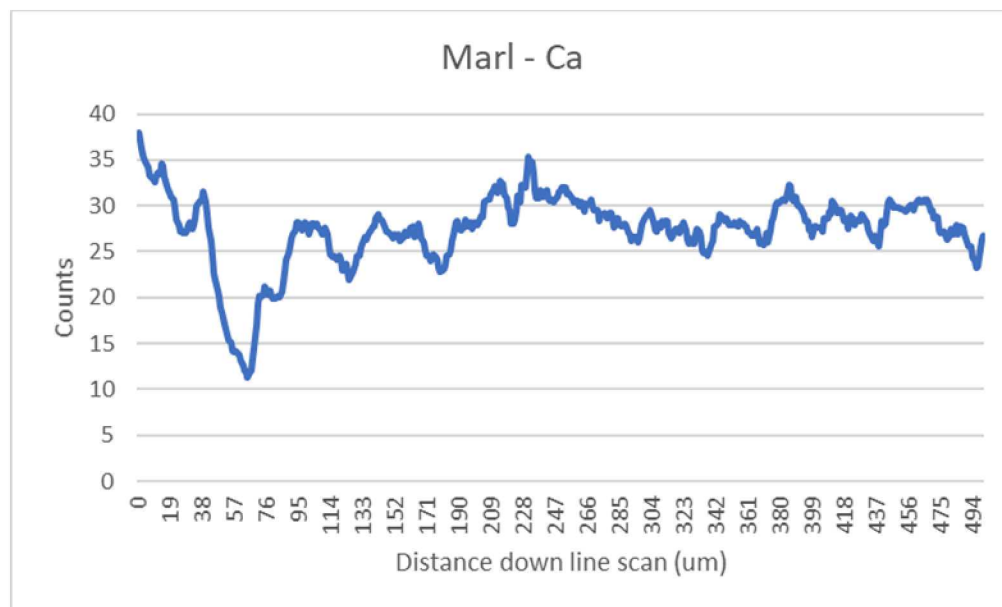
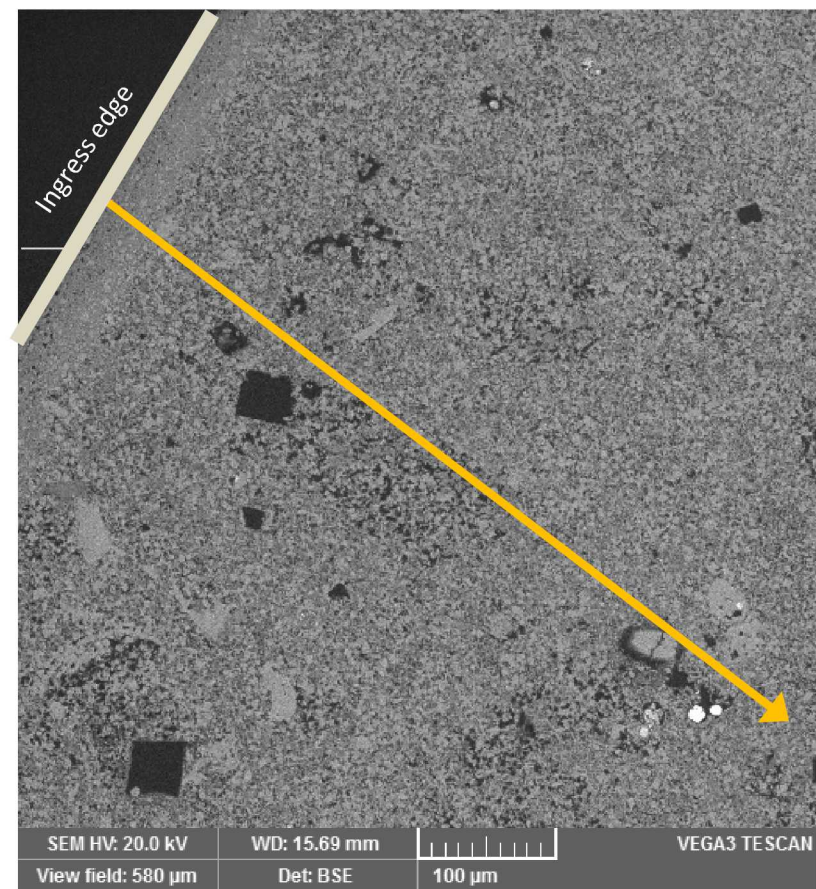
**Oil Shale**





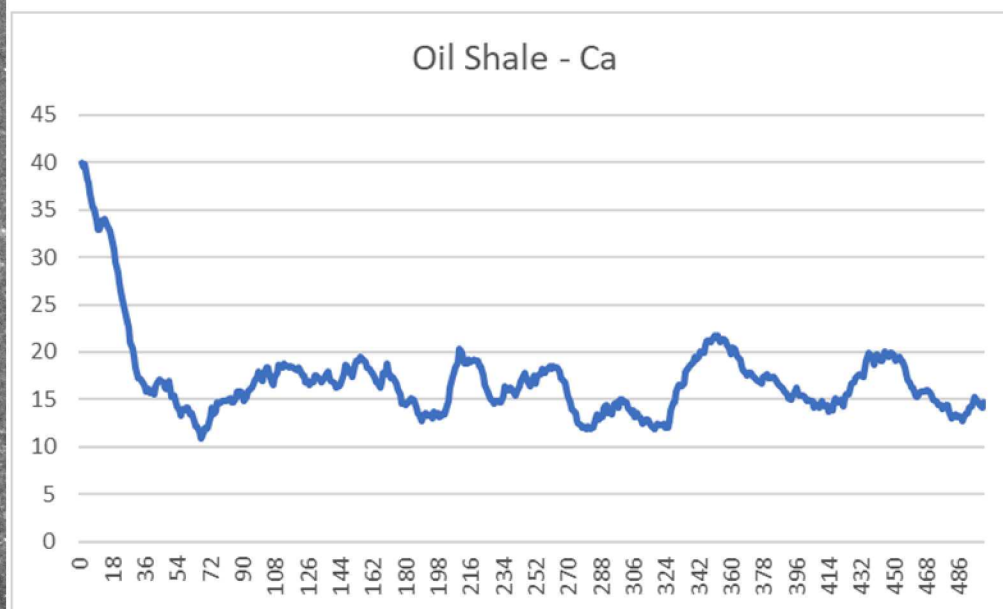
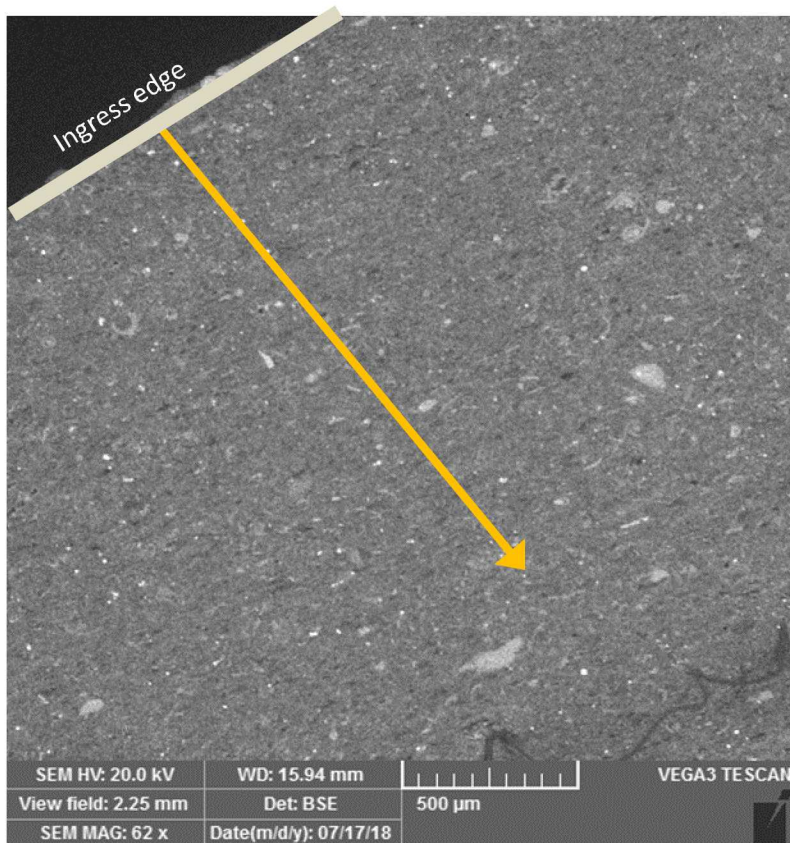


# Results and Discussion: SEM, EDS Line Scan





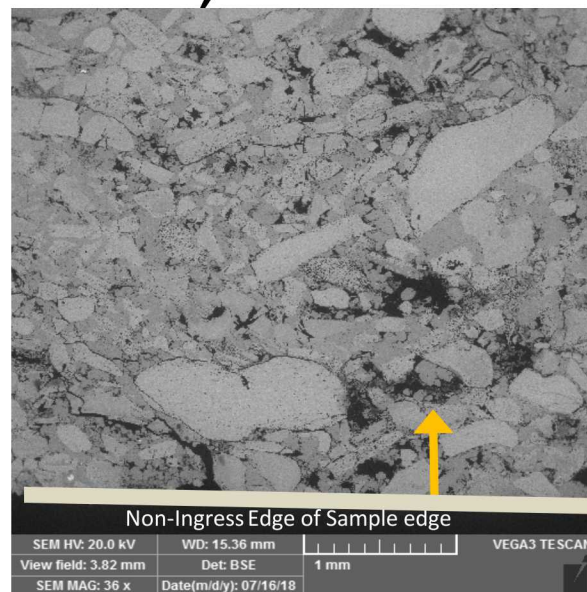
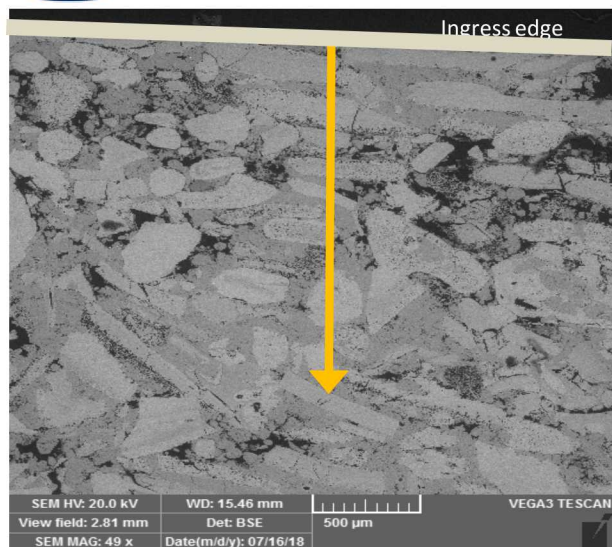
# Results and Discussion: SEM, EDS Line Scan



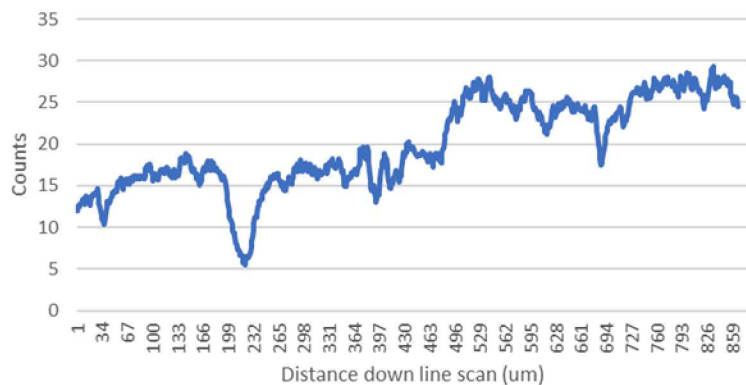




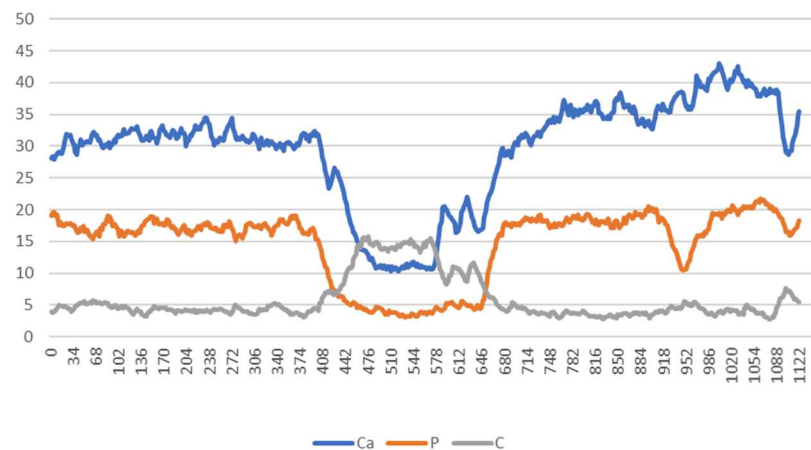
# Results and Discussion: SEM, EDS Line Scan



Phosphorite - Ca



Phosphorite - Ca, P, C







# Conclusions and Lessons Learned

- Imaging of the samples.
  - Backscatter SEM and petrographic microscope
    - Offers insight to the structure and composition of the rocks
  - BET and MIP
    - Pore size distribution and surface area of materials.
- Characterization of materials yields no surprises, and gives valuable information for modeling parameters.
- Backscatter SEM & EDS
  - Some challenges determining Br front.
    - But some insight gained by looking at other element composition.
  - Confirmation of characterization work.
- Future work
  - Finish characterization work
  - Image analysis of SEM images for porosity changes



# Acknowledgements

It is important to first thank my mentors at SNL, Ed Matteo and Carlos Jòve-Colòn, without whom I would have been totally lost. Their time and expertise were greatly appreciated as I maneuvered my way through the project and life at Sandia. I would also like to thank the numerous others who helped me with my work this summer— Melissa Mills, Jessica Kruichak, and Carlos Lopez just to name a few.



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Questions?

