

Utilization of Nanotechnology for Mature Fine Tailings Treatment

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The 54th Annual Clay Mineral Society Conference

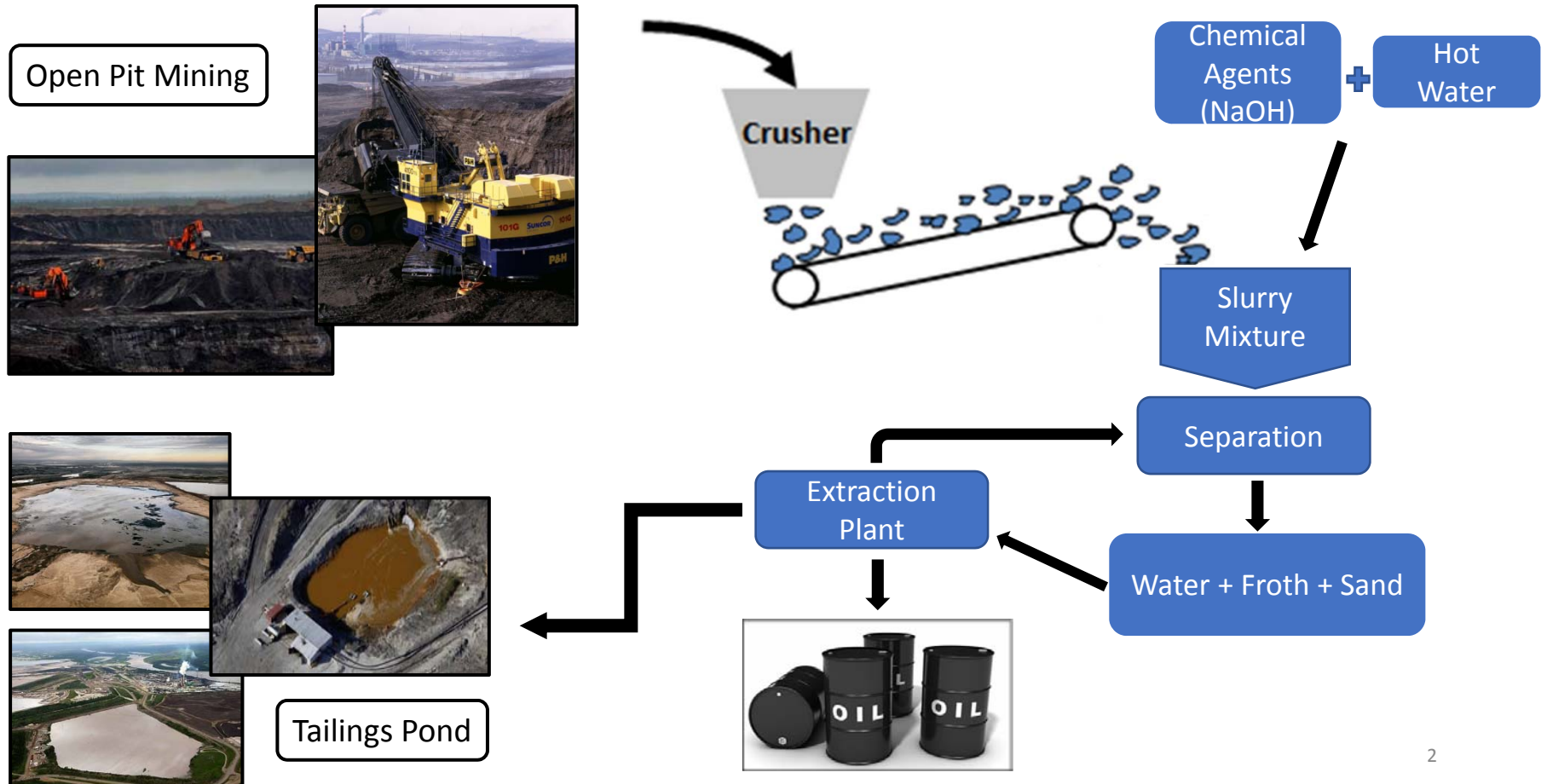
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Introduction



Mature fine Tailings

- Composition:
 - 30-50% solid content - 82 % sand ($>44\text{ }\mu\text{m}$)
 - 17 % fines ($<44\text{ }\mu\text{m}$)
 - 1-2 % bitumen
- Fines: silt ($2\text{-}44\text{ }\mu\text{m}$) and clay ($<2\text{ }\mu\text{m}$)
- Clay: 40% to 70% kaolinite, 30% to 45% illite with up to 10% mixed layer illite/smectite
- Poor settling ability after consolidation to 30 wt.% of fine tails

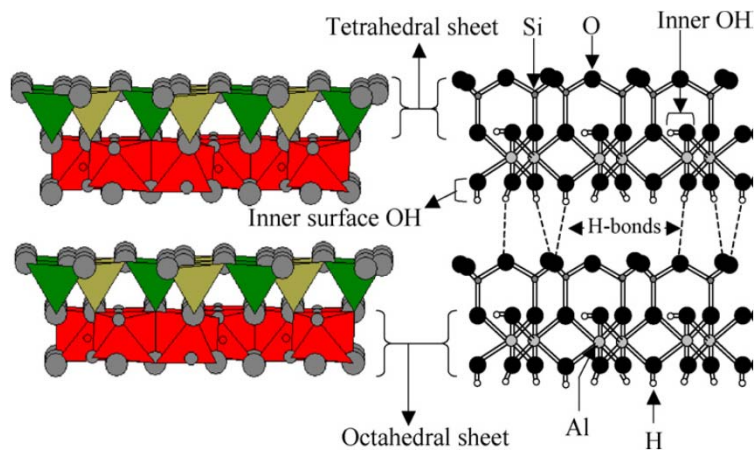


Efficient treatment  Better understanding of the treatment mechanism
and interactions between MFTs

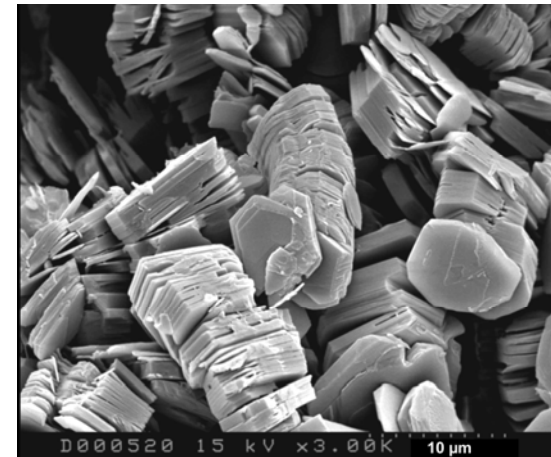
Mature fine Tailings

Main causes of MFT stability are:

- Wide particle surface charge distribution
- Wide particle size distribution
- High viscosity of the suspension
- Different types of clays in the MFT (Kaolinite being the dominant fraction)
- Residual Bitumen trapped in MFT



Structure of Kaolinite

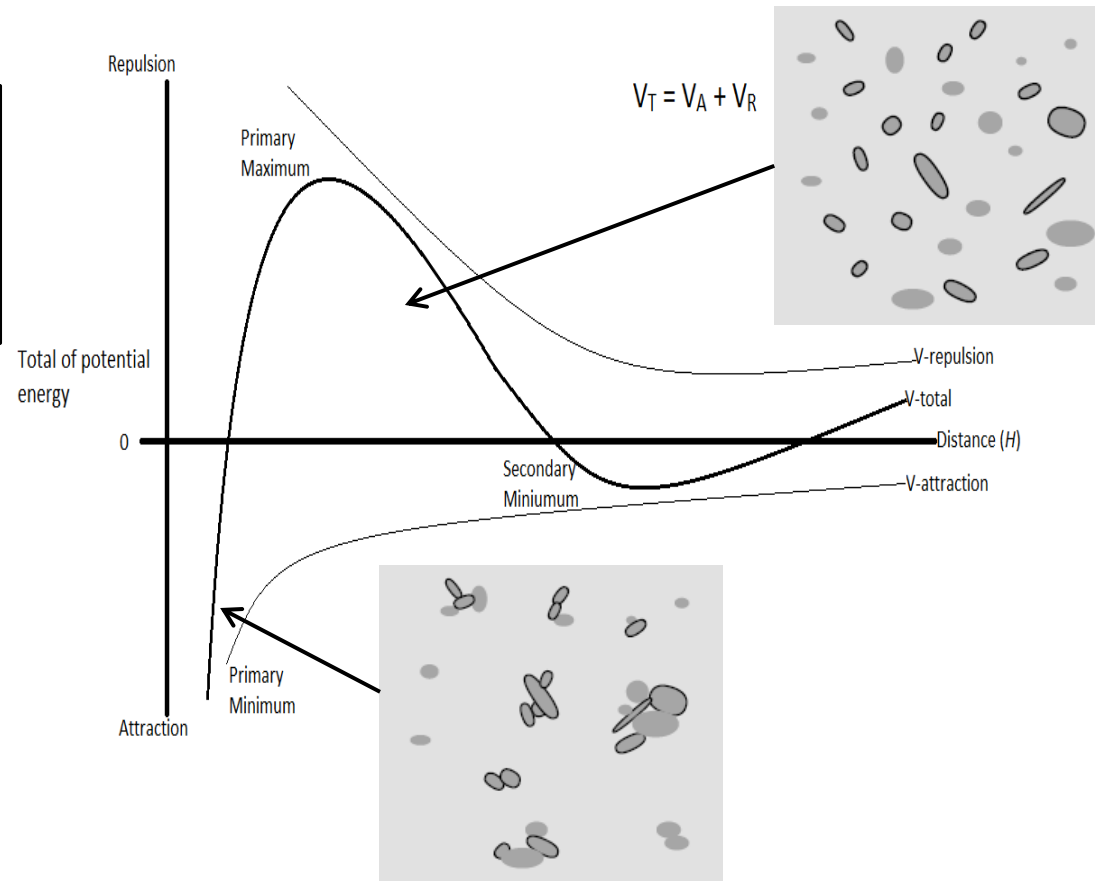


SEM image of Kaolinite

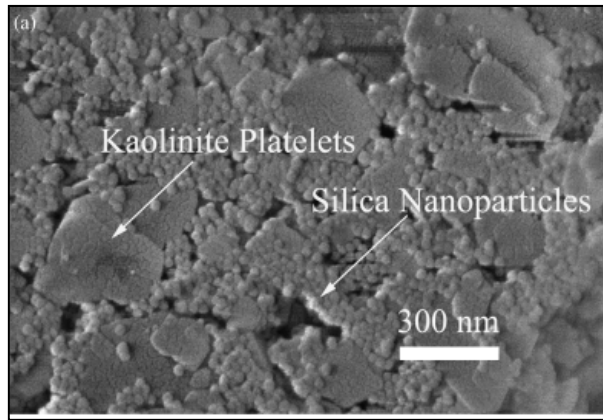
Governing Forces in Mature fine Tailings

Interaction between particles:

- **Electrostatic Interaction**
- **Van der Waals Forces**
- **Excluded volume repulsion**
- **Hydrodynamic forces**

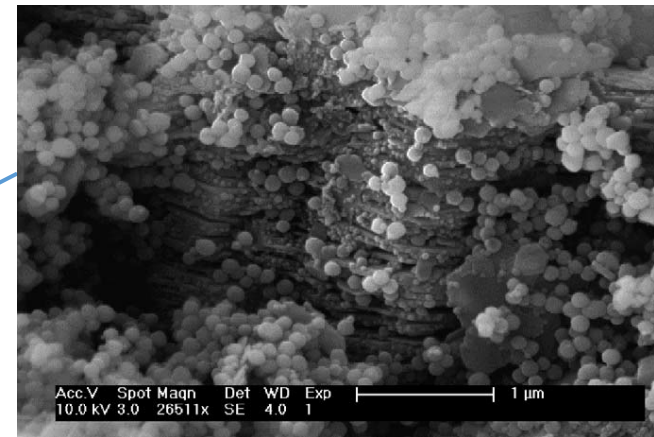


Motivation



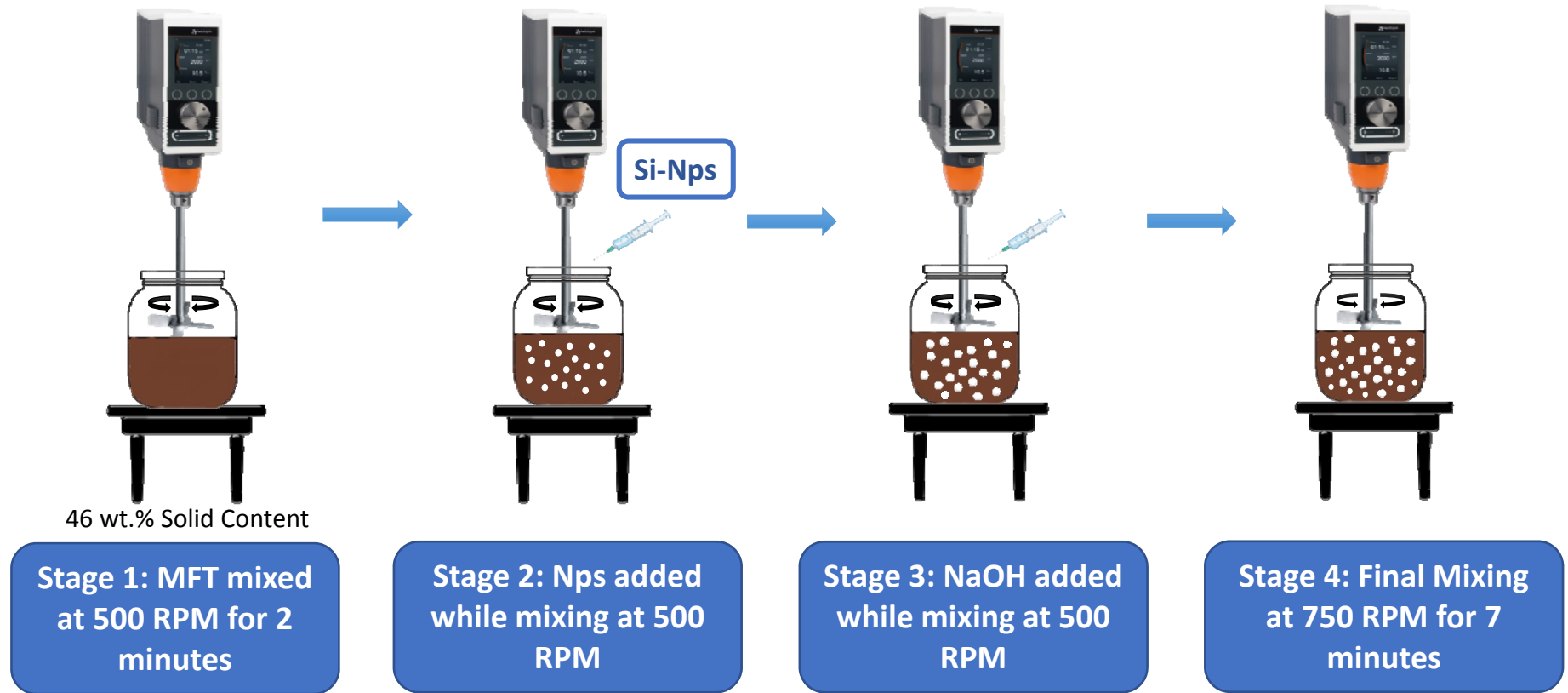
Strength of clay (kaolinite)– nanoparticle composites affected by Clay concentration, size and morphology on the microstructure

The effect on the clay stability and rheological properties due to the addition of nanoparticles to the aqueous suspension of clay and salt



Our Goal: Develop a mechanistic approach to understand the destabilization of MFTs using nanoparticles

Materials and Method



Results and Discussion

After addition of nanoparticles, layers of varying solid content appear in MFT along with a mat of bitumen.

**Immediately
After Flocculation**



**1 Day After
Flocculation**



Bitumen Layer

Top Layer

Middle Layer

Bottom Layer

**Solid and Bitumen
Content Analysis**

**Drying
Test**

**Toluene Insoluble
Fraction Test
(ASTM- D5546)**

Solid and Bitumen Content Analysis



- Solid and Bitumen content calculated from drying and toluene insoluble fraction test
- Zeta Potential and size distribution of solid particles in the flocculated MFTs.

Sample	Solid Content (wt.%)	Bitumen Content (wt.%)	Zeta Potential (mV)	Particle Size (nm)
Bulk MFT	46	1.5-2	-24.7	680
Top Layer	10-15	1-1.5	-25.3	349
Middle Layer	52-55	0.7-0.9	-22.2	767
Bottom Layer	70-80	0.15-0.20	-37.9	862

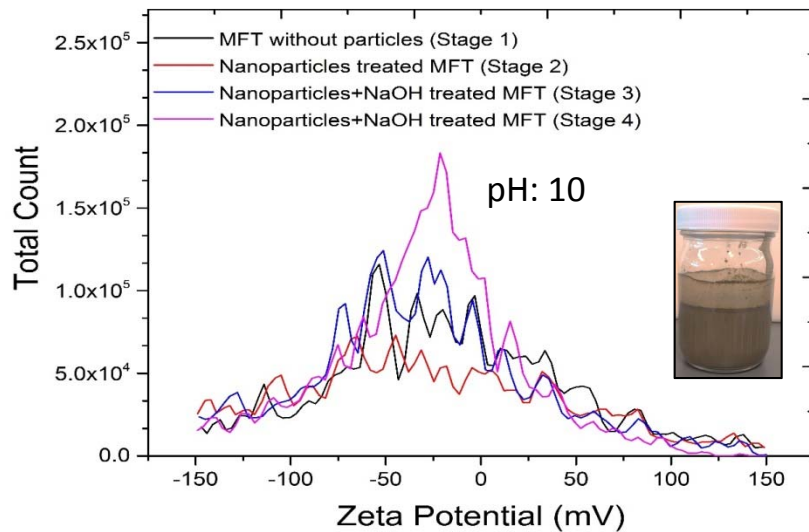
Solid and Bitumen Content Analysis



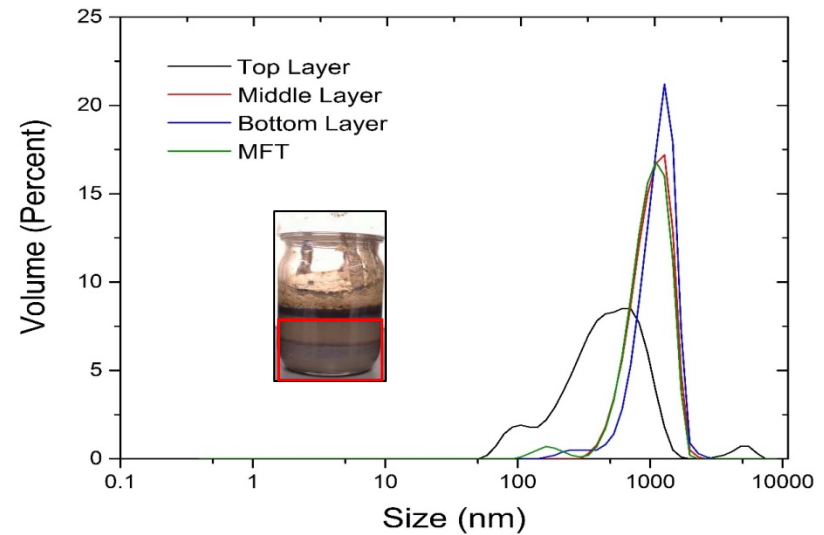
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Zeta Potential and Size Analysis

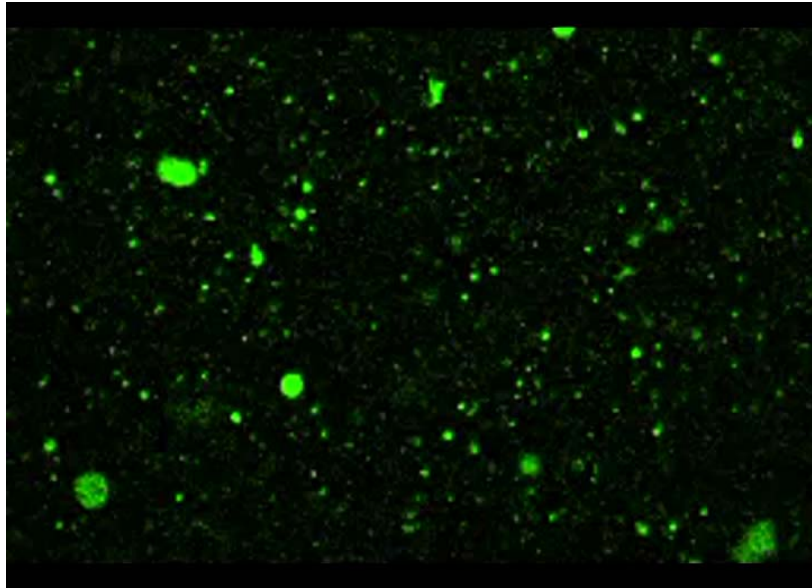


pH dependent destabilization for efficient treatment of tailings via synergy of nanoparticles and NaOH

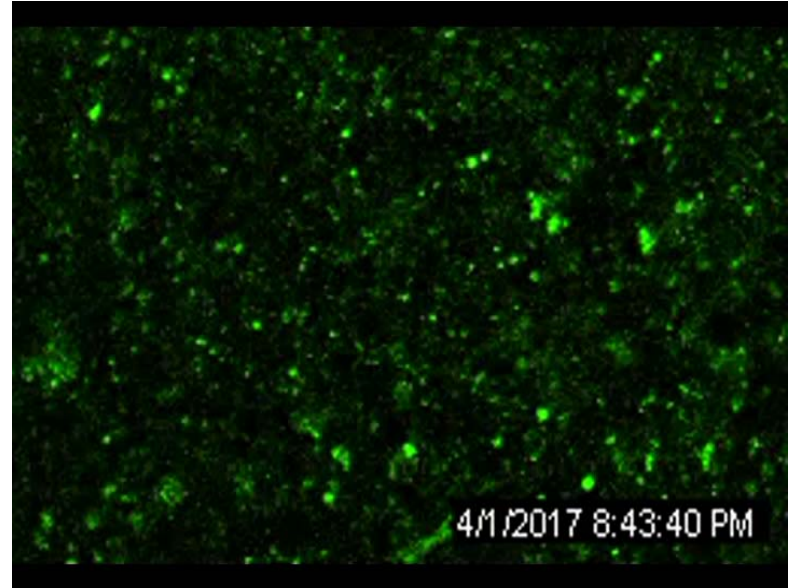


Particle size increase from the top to the bottom layer

Bitumen Mobility in Different Layers

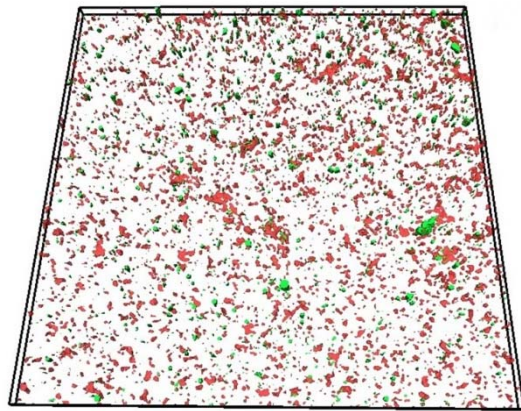


Bitumen in top layer

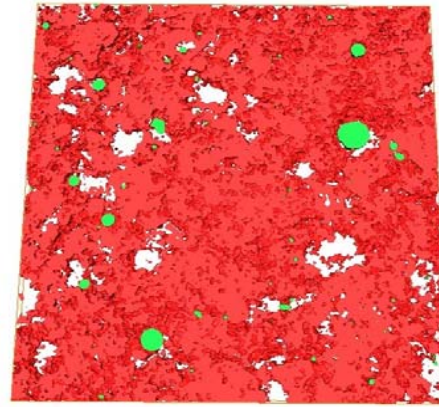


Bitumen in bottom layer

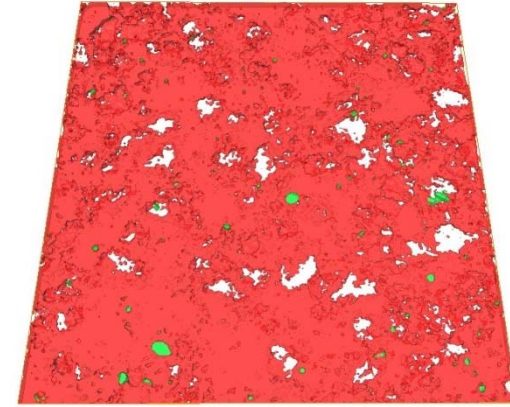
Fluorescence/Reflectance Microscopy



Top Layer



Middle Layer



Bottom Layer

5 μm
145 μm 145 μm

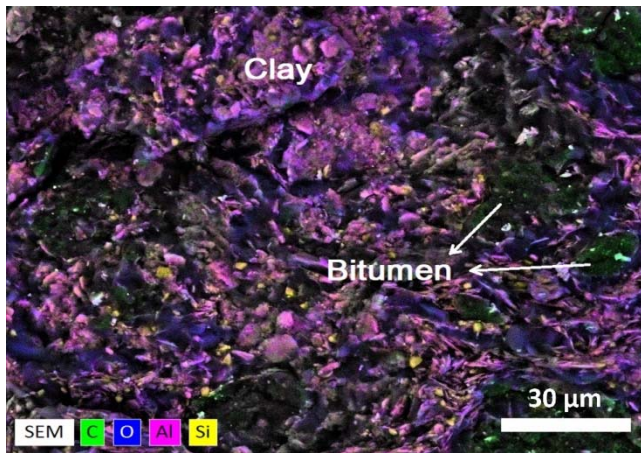
■ Bitumen ■ Clay + Nanoparticles □ Water (transparent)

Volume fraction (%)	Top Layer	Middle Layer	Bottom Layer
Clay+ Nanoparticles	12.71	49.38	73.13
Bitumen	1.4	0.83	0.26

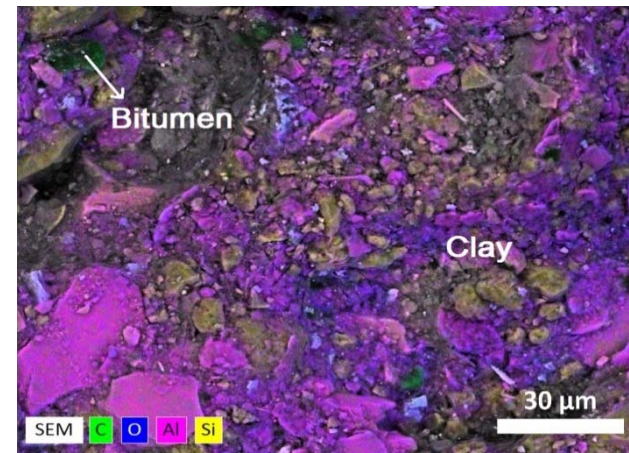
The volume fraction of clay-nanoparticles flocs and bitumen obtained from 3D LCSM images strongly correlate with the drying and toluene test results.



Cryo-SEM of Flocculated Layers



Middle Layer

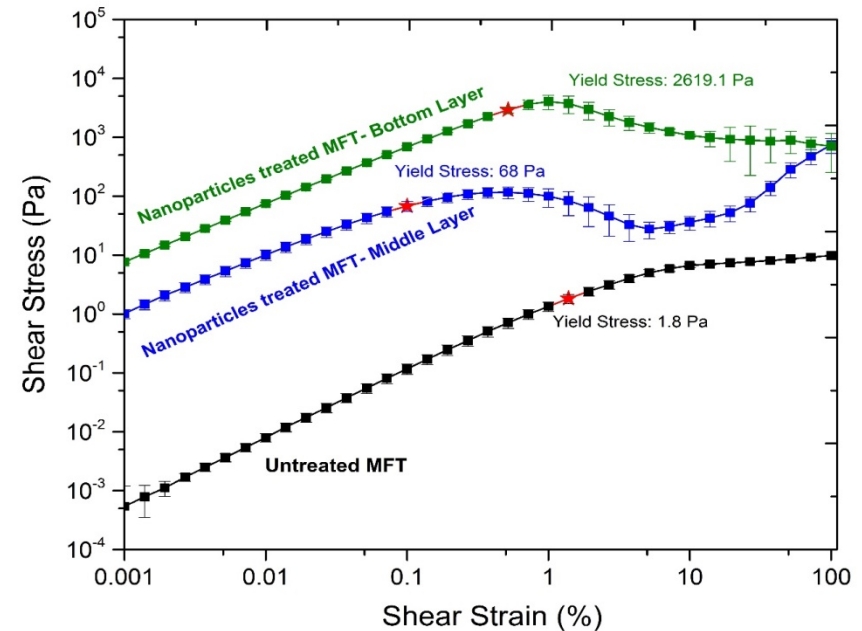
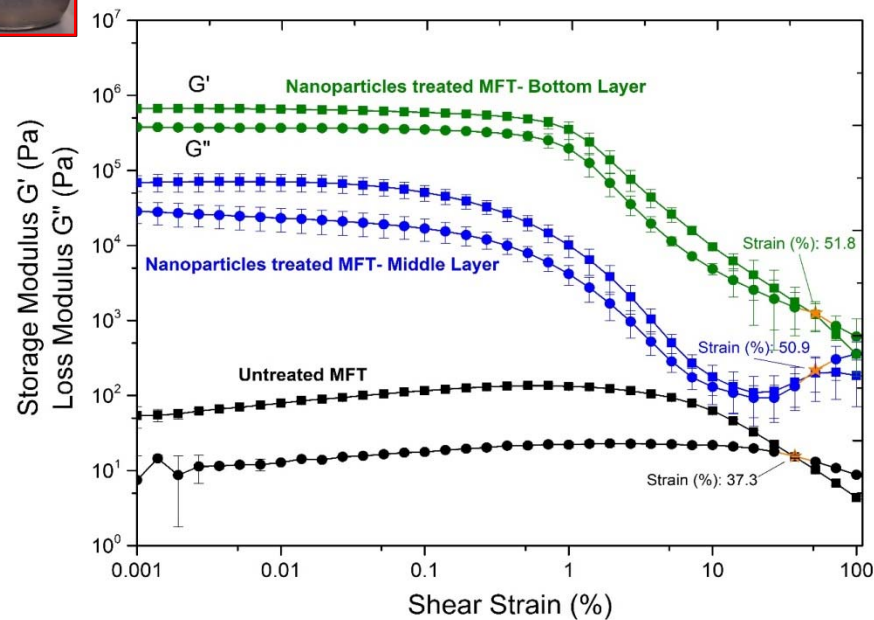


Bottom Layer

Cryo-SEM with EDX images of these different layers further confirmed the formation of densely packed structures with minimal amount of bitumen in the bottom layer, while more bitumen is present in the middle layer.



Rheology of Flocculated MFT



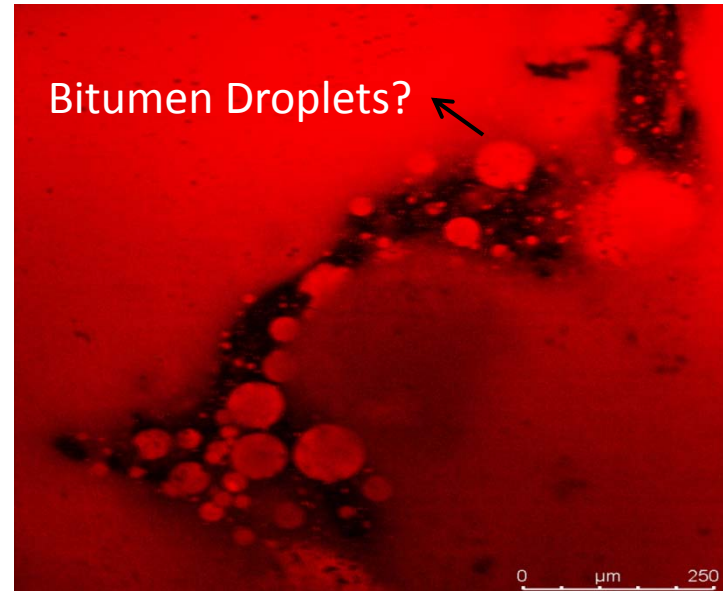
Rheological studies showed that middle layer exhibited a transition to a gel like, shear thickening structure, while high solid content and clay packing in the bottom layer resulted in remarkably high yield strength.



Bitumen Extraction from MFT?



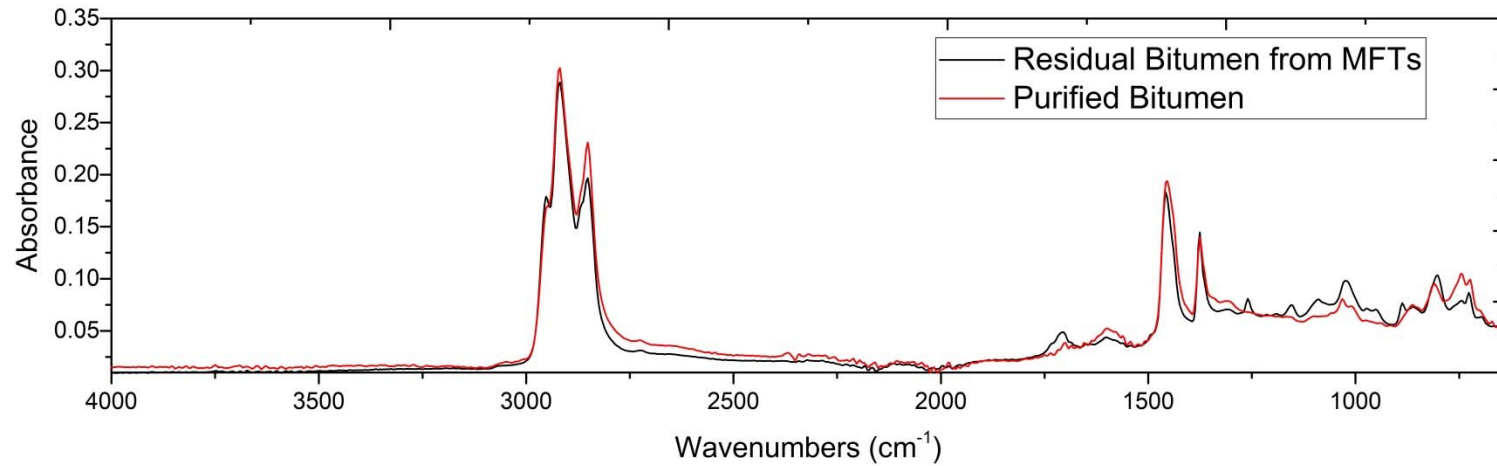
Bitumen (1-2 wt.%)
Particles: Surface modified SiNP



Bitumen (1-2 wt.%)
Particles: Si based NPs (surface modified)



FTIR Results of Bitumen Layer



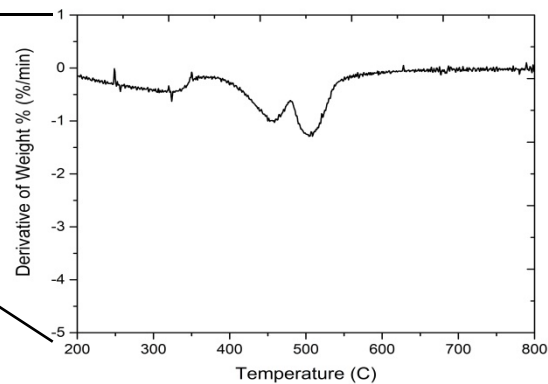
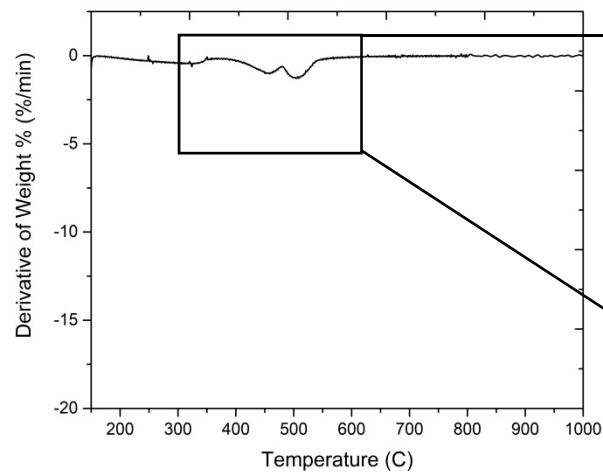
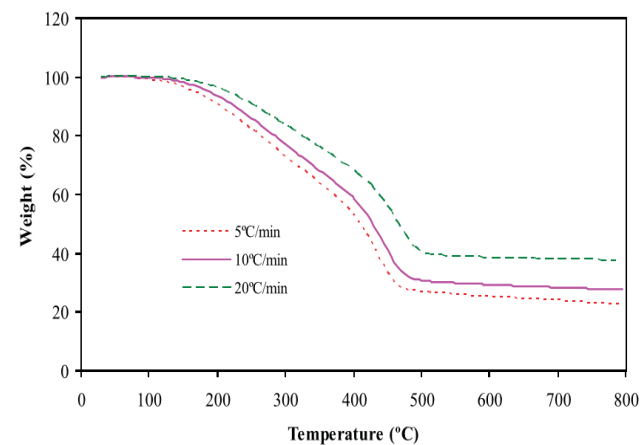
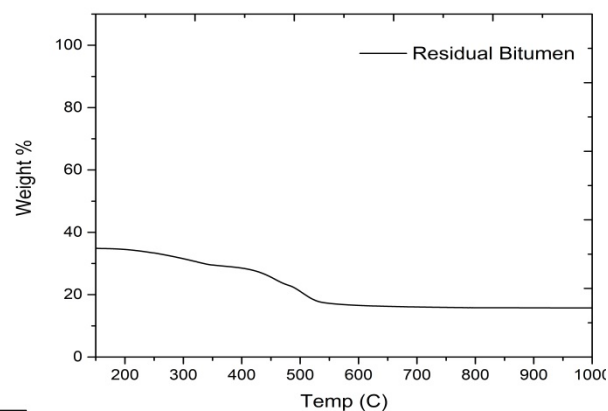
Comparative analysis of the FTIR spectra of commercial bitumen and residual bitumen extracted from tailings proves that nanoparticles addition in MFTs is releasing trapped bitumen.



- ❖ Sample initial weight: 15.56 mg
- ❖ Experiment was conducted under Air
- ❖ Heating Rate: 10 C min⁻¹

TGA Results

TGA curve of Extracted Bitumen



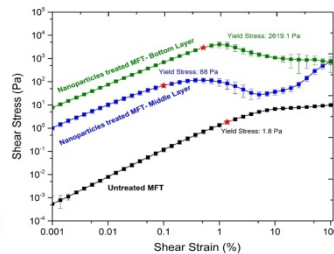
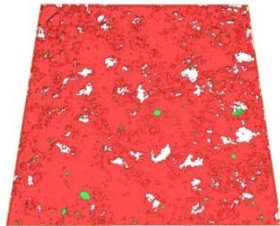
DTG curve of Extracted Bitumen

Murugan, P., Mani, T., Mahinpey, N. and Dong, M. (2012), Pyrolysis kinetics of Athabasca bitumen 18 using a TGA under the influence of reservoir sand. Can. J. Chem. Eng., 90: 315–319.

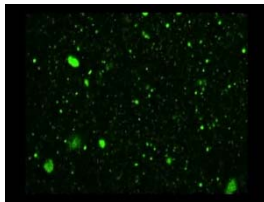
Conclusion



The synergistic effect of nanoparticles and NaOH addition with mixing dynamics indicates pH dependent destabilization and formation of layers



Interconnected 3D clay-particles structure and high clay to void ratio. Middle layer showed shear thickening effect due to the structural rearrangement



Addition of nanoparticles in MFTs resulted in liberation of trapped bitumen.

Future Work

- Optimizing the mixing regime and nanoparticles concentration
- Tagging silica nanoparticles with the fluorophore to visualize the nanoparticles distribution in the flocculated layer, study the microstructure evolution and probe the flocculation mechanism
- Extract maximum amount of bitumen from the MFTs

Acknowledgement

