Evolution of the Microstructure and Rheological Properties during Tailings Consolidation Process

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IOSCT Conference, Lake Louise, Alberta
December 5, 2016
Motivation

Effect of type of flocculant and processing conditions on the microstructure and rheology of clay dispersions

Effect of inorganic nanoparticle additives

McFarlane, J. Addai-Mensah and K. Bremmell, Rheology of flocculated kaolinite dispersions (2005), Korea-Australia Rheology Journal, 17(4), 181-190


Goal: Establishing a connection between evolving microstructure and bulk rheology during consolidation process
Preparation of Model Tailings

Northern Alberta Bitumen (mixed with 2/3 of kaolinite at 150 rpm)

Heated to 90°C

Kaolinite

Nile blue

Untagged 0.05% NaHCO₃ solution in DI

Overhead mixing at 650 rpm

Model MFT (30% clay)

Dilution

Model MFT (10% clay, 3% bitumen)
Flocculation Procedure

- Overhead stirrer
- Model MFT (10% clay)
- A310 impeller

100 g/t of clay solids
0.1 wt% Flopam A3338 solution
in 0.05 wt % NaHCO₃ DI solution

Sample placed in the aging funnel
Approach – Confocal Rheology

Rheometer with 25 mm plate measuring system

Confocal Microscope

Linking rheology and 3D structure via:
- Flocc network characterization
- Pore volume quantification
- Bitumen/Clay connectivity
Confocal Microscopy

3D imaging for evaluation of:
Size, shape and distribution of flocs
Pore volume distribution
Confocal Microscopy

3D imaging for evaluation of:
- Size, shape and distribution of flocs
- Pore volume distribution
Microstructure of Flocculated Tailings

Water and Polymer are not tagged

Bitumen - Autofluorescence
Clay – Tagged with Nile Blue
Monitoring of Flocculation Process

Floccs form instantaneously and break under continued mixing.
Smaller particles not able to join bigger floccs.
Flocs Microstructure During Consolidation Process

Day 1

Day 22

Day 5

Day 30
Effect of Aging on Microstructure

<table>
<thead>
<tr>
<th>3D Microstructure</th>
<th>Pore Volume Fraction (%)</th>
<th>Surface Coverage of Bitumen by Clay</th>
<th>Floc Characteristic Length</th>
</tr>
</thead>
<tbody>
<tr>
<td>Day 1</td>
<td>65.2, 68.2, 73.3</td>
<td>94.4, 98.3, 91.0</td>
<td>( \lambda = \frac{1}{Q} = \frac{V}{\sum_{i=1}^{N} A_i} )</td>
</tr>
<tr>
<td>Day 5</td>
<td></td>
<td></td>
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<tr>
<td>Day 22</td>
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<tr>
<td>Day 30</td>
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</tr>
</tbody>
</table>

- Clay
- Bitumen

- 10.2 µm
- 160 µm
- 160 µm

\( \lambda \) = \frac{1}{Q} = \frac{V}{\sum_{i=1}^{N} A_i}
Effect of Aging on Rheology of Flocculated Sample

Increasing trend in Storage and Loss Modulus but decreasing in crossover frequency
Effect of Aging on Rheology of Flocculated Sample

Yield Stress increases with aging
Summary and Future Work

Microstructural Evolution during Consolidation Process

3D Image Quantification

Rheology

- Rheology of real MFT
- Influence of pre-shear time on the rheology
- Thixotropic study of the microstructure
- Floc Fractal dimension evaluation from 3D Images

Confocal Rheology

Rheometer

LSCM
Acknowledgment
Kaolinite tagged with Nile Blue

Zeta potential (average of 3 runs):

-41 mV ± 13.3 mV

Adsorption of the fluorescent dye does not change the surface properties of kaolinite
Rheology comparison between different samples

10% model MFT sample prepared by heating method and removing the emulsified bitumen shows the same rheology as the real MFT sample diluted to 10% clay.
Laser Scanning Confocal Microscopy

• 3D imaging for evaluation of:
  • Size, shape and distribution of flocs
  • Pore volume size distribution

\[
\lambda = \frac{1}{Q} = \frac{V}{\sum_{i=1}^{N} A_i}
\]
Floccs Characteristic Size
Imaging

Bitumen – Autofluorescence
Clay – Nile blue tagged
Water - untagged

Emission spectra of bitumen and labeled clay

405 nm laser excitation
638 nm laser excitation

nm
Future focus

4D study of flocculation dynamics
Hydrodynamic effects on floc structure and subsequent dewatering
Time depended rheology and its relationship to the microstructure
Flocs aging – preliminary results
Flocs aging – preliminary results – Oct 19

LVE of OCT 19 floccs over time

Storage Modulus, Pa
Loss Modulus, Pa

Shear Strain, %

Day 1

Day 5
Flocs aging – preliminary results – Oct 19

LVE of OCT 19 floccs over time

Storage Modulus, Pa
Loss Modulus, Pa

Shear strain, %
Flocs aging – preliminary results – Oct 19

LVE Oct 19 aging

Storage Modulus, Pa
Loss Modulus, Pa

10^5

G'

10^4

G''

Day 5

Day 22

Day 10

Day 1

Shear Strain, %

10^{-4}

10^{-3}

10^{-2}

10^{-1}

10^0

10^1

10^2
Flocs aging – preliminary results – Oct 19

Day 1
Floccs aging – preliminary results – Oct 19

Day 1 63x
Flocs aging – preliminary results – Oct 19

Day 5 10x
Flocs aging – preliminary results – Oct 19

Day 22 63 x
Flocs aging – preliminary results

Shear strain vs. shear stress - effect of aging

Day 1

Day 2

Day 3

Shear stress, Pa

10^{-2} 10^{-1} 10^{0} 10^{1} 10^{2}

10^{-4} 10^{-3} 10^{-2} 10^{-1} 10^{0} 10^{1} 10^{2}

Shear strain, %

294.1 Pa, 3.9%

463.4 Pa, 10%

31.5 Pa, 2.3%
Flocs aging – pore size distribution and connectivity analysis