Effects of fluid handling components on slurry health

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Outline

• Introduction
• Particle measurement procedures
• Effects of components on slurry particle size distribution
  – Pumps
  – Valves
  – Shipping containers
• Insights gained
Particle size measurement

• “Working” particle size distribution
  – Measured using dynamic light scattering
  – All particles in a defined volume illuminated simultaneously
  – Particles are sized by measuring their diffusion coefficient
  – Measures relative concentrations
  – Sensitive to about 1% by volume
  – Instrument used – NICOMP 380ZLS (Particle Sizing Systems)

• “Large particle tail” size distribution
  – Measured using single optical particle counters.
  – Requires dilution
    • Slurry contains about $10^{15}$ working particles/ml
    • The large particle tail contains about $10^5$ particles/ml ($\geq 0.56\mu$m)
  – Instruments used:
    • AccuSizer 780 (Particle Sizing Systems)
    • LiQuilaz-S05 (Particle Measuring Systems)
Working particle size distribution

Mean diameter = 138 nm

99th Percentile = 276 nm
On-line dilution system for large particle tail measurement
Initial size distribution of the large particle tail
Effect of pumps
Types of pumps evaluated

- Bellows with pulse dampener
- Diaphragm with pulse dampener
- Centrifugal
Schematic of system used to measure effect of pumps on slurry PSD

- Nitrogen
- humidifier
- flowmeter
- pulse dampener
- sample port
- drain
- chiller
Test conditions

- Test system volume: 12 L of slurry
- Pump air supply or speed was adjusted to achieve the following test conditions:
  - Flow rate: 30 Lpm
  - Outlet Pressure: 30 psig (gradual reduction to ambient pressure)
- Tank blanketed with humidified N₂: RH > 90%
- Slurry temperature: 20 ± 2°C
Effect of pumps on the working particle size distribution

Turnovers

Volume-Weighted Diameter (nm)

Mean: Bellows pump
Mean: Diaphragm pump
Mean: Centrifugal pump
99th Percentile: Bellows pump
99th Percentile: Diaphragm pump
99th Percentile: Centrifugal pump

As-received slurry plotted at 1.1 turnovers
Effect of bellows pumps on the large particle tail

Cumulative Concentration (#/mL)

Particle Diameter (µm)

- 0 turnovers
- 3.7
- 9.8
- 30
- 101
- 210
- 314
- 574
- 1105
Effect of pumps on particles $\geq 0.56 \mu m$
Effect of pumps on particles $\geq 10 \, \mu m$

![Graph showing the effect of different types of pumps on particle concentration.]

- **Bellows pump**
- **Diaphragm pump**
- **Centrifugal pump**

The graph illustrates the ratio of measured particle concentration to initial particle concentration over a range of turnovers. The data points show a linear relationship, indicating that Bellows pumps increase particle concentration more significantly than Diaphragm and Centrifugal pumps.
Effect of pumps on particle concentrations

<table>
<thead>
<tr>
<th>Particle Size</th>
<th>Particle Concentrations Relative to the Initial Particle Concentrations (C_T/C_I)</th>
<th>100 Turnovers</th>
<th>1,000 Turnovers</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Bellows</td>
<td>Diaphragm</td>
<td>Centrifugal</td>
</tr>
<tr>
<td>≥ 0.56 µm</td>
<td>2.9</td>
<td>2.3</td>
<td>1.0</td>
</tr>
<tr>
<td>≥ 1.0 µm</td>
<td>5.3</td>
<td>4.1</td>
<td>1.0</td>
</tr>
<tr>
<td>≥ 2.0 µm</td>
<td>5.6</td>
<td>5.7</td>
<td>1.1</td>
</tr>
<tr>
<td>≥ 5.0 µm</td>
<td>4.4</td>
<td>6.3</td>
<td>1.0</td>
</tr>
<tr>
<td>≥ 10 µm</td>
<td>4.3</td>
<td>7.7</td>
<td>0.9</td>
</tr>
</tbody>
</table>
Particle generation by pumps

Particle Diameter (µm)

Number of Particles Generated per Pump Stroke

- bellows pump
- diaphragm pump

CTA 831 1151
Effect of pump type of filter life

- **Test method**
  - Pressure drop measured across a new 10” Mykrolis Planargard™ CMP3 filter located downstream of the pump

- **Test conditions**
  - Test system volume: 28 L of slurry
  - Pump air supply or speed was adjusted to achieve the following test conditions:
    - Flow rate: 30 Lpm
    - Pump outlet pressure: 22-37 psig (depending on ΔP across filter)
  - Tank blanketed with humidified N₂: RH > 90%
  - Slurry temperature: 20 ± 2°C
Schematic of system used to measure effect of pump type on filter life
Effect of pumps on filter life

- Centrifugal pump
- Bellows pump
- Diaphragm pump
Effect of valves
Schematic of system used to measure the effect of valves on slurry PSD

- Slurry tank
- Circulation pump
- Programmable Logic Controller
- CDA
- Humidified Nitrogen
- Test valves
- Bypass loop
Test conditions

- 8 valves tested simultaneously
- Valves tested under steady flow and cycling conditions
- Valve cycle rate: 12 cycles/min
- Mean velocity through each valve: 0.5-1.0 ft/s
- Slurry system turnover rate: 0.75 turnovers/min
- Test system volume: 25 L
- Valve outlet pressure: 22 psig (gradual reduction to ambient pressure)
Effect of valve cycling on slurry PSD

Cumulative Particle Concentration ≥ 0.6 µm (#/ml)
(measured by PMS LiQuilaz-S05)

- Spool test 1
- Spool test 2
- Spool test 3
- Valve test 1
- Valve test 2
- Valve test 3

Cycling initiated

Turnovers

 Cumulative Particle Concentration ≥ 0.6 µm (#/ml)

0 1000 2000 3000 4000 5000

0 10000 20000 30000 40000 50000 60000

Spool test 1
Spool test 2
Spool test 3
Valve test 1
Valve test 2
Valve test 3

Cycling initiated
Effect of valve cycling on slurry PSD

Cumulative Concentration (#/ml)

Particle Diameter (µm)

1,250 cycles
3,750 cycles
12,250 cycles
37,875 cycles
126,000 cycles
168,500 cycles
262,250 cycles
Particle generation by pumps and valves

Particle Diameter (µm)

Number of Particles Generated per Pump Stroke (pumps)
or per Valve Cycle (valve)

- bellows pump
- diaphragm pump
- valve
Effect of valves and pumps at POU application

<table>
<thead>
<tr>
<th>Size</th>
<th>Initial Concentration</th>
<th>Bellows pump</th>
<th>Valve</th>
</tr>
</thead>
<tbody>
<tr>
<td>um</td>
<td>#/ml</td>
<td>Concentration</td>
<td>Increase</td>
</tr>
<tr>
<td>0.7</td>
<td>158056</td>
<td>163918</td>
<td>3.7</td>
</tr>
<tr>
<td>1.0</td>
<td>32110</td>
<td>34167</td>
<td>6.4</td>
</tr>
<tr>
<td>2.0</td>
<td>6972</td>
<td>7339</td>
<td>5.3</td>
</tr>
<tr>
<td>5.0</td>
<td>2303</td>
<td>2360</td>
<td>2.5</td>
</tr>
<tr>
<td>10.0</td>
<td>704</td>
<td>711</td>
<td>0.9</td>
</tr>
</tbody>
</table>

Assume: 5 strokes (or cycles) per L
# Effect of pump in a slurry delivery system

| Size (um) | Initial Concentration (#/ml) | 1 Pass Through Component | | 100 Passes Through Component |
|----------|------------------------------|--------------------------|--------------------------|
|          |                              | Bellows pump              | Valve                    | Bellows pump              |
|          |                              | Concentration (#/ml)      | Concentration (#/ml)     | Concentration (#/ml)      |
|          |                              | Increase (%)              | Increase (%)             | Increase (%)              |
| 0.7      | 158056                       | 163918                   | 3.7                      | 158121                    | 0.04 |
| 1.0      | 32110                        | 34167                    | 6.4                      | 32139                     | 0.09 |
| 2.0      | 6972                         | 7339                     | 5.3                      | 6981                      | 0.13 |
| 5.0      | 2303                         | 2360                     | 2.5                      | 2306                      | 0.12 |
| 10.0     | 704                          | 711                      | 0.9                      | 704                       | 0.06 |
Effect of shipping containers
Effect of shipping containers on slurry PSD

• Proprietary slurry
• Measured PSD of slurry before and after simulated shipping
• Compared containers with and without headspace
Effect of shipping containers on slurry PSD

Particle Diameter (µm)

Cumulative Concentration (#/mL)

- Slurry filtrate
- Containers after shipping with headspace (Qty=9)
- Containers after shipping without headspace (Qty=8)
Summary

• Pumps can significantly increase large particle concentrations in sensitive slurries.
• Pump type is important; large particle concentrations showed little change with the centrifugal pump; large changes with bellows and diaphragm pumps.
• Filter clogging rates were consistent with changes in large particle concentrations measured in the absence of filters.
• Valves appear to cause much less agglomeration than pumps.
• Particle agglomeration is a function of how the slurry is packaged. If the headspace in a container is reduced, agglomeration is reduced.
• Causes of agglomeration:
  – Shear?
  – Cavitation?
  – Other?