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Crossflow Filter Investigations to Improve SRS and Hanford Waste Treatment Plant Operations

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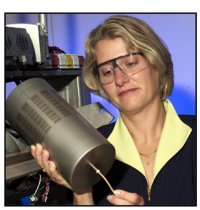
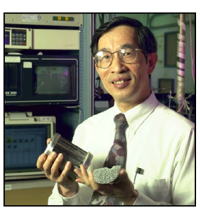
November 17, 2010

EM Waste Processing Technical Exchange 2010

EM Office of Environmental Management
safety ❖ performance ❖ cleanup ❖ closure

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EM-31 WP-2.3.6 Cross-Flow Filtration

- Cross-Flow Filtration is pinch-point for both Plants
- Improvement in flux and cleaning time sought
- Improvement will lead to increased throughput
- EM-31 funding a program to better understand installed x-flow filtration with a sludge and high-sodium supernate slurry
- To date specific studies of increasing flux or cleaning has not been directly funded. Only standard operation has been studied
- SRNL with PNNL lead are funded to study problem

EM-31 WP-2.3.6 Cross-Flow Filtration Plan

- Literature Review (Intern guided by PI) showed:
  - Cleaning with oxalic tends to be more effective and nitric acid
  - Backpulsing of thick-wall filter tubes is not effective
  - Developing a better cake may lead to better filter flux
- Dead-End Filter Test to:
  - Determine challenging slurry (SB6 or Tank 8F non-radioactive simulants are used)
  - Best filter enhancer (among 6)
- Cross-Flow Filter Test to:
  - Evaluate different filter media
  - Clean filter with just scouring and 0.5 Oxalic acid
  - Develop more permeable cake via flow conditions
  - Develop more permeable cake via filter enhancers

Experimental Matrix

Dead End Filter Testing

To Evaluate Filter Enhancers:

- Filter Aids – Filter coaters
  - SiC, TiO_2, HF, FF
    (HF, FF activated carbons)
- Body Feeds – Flocculants
  - PEO, HX200

Cross-Flow Filter Testing

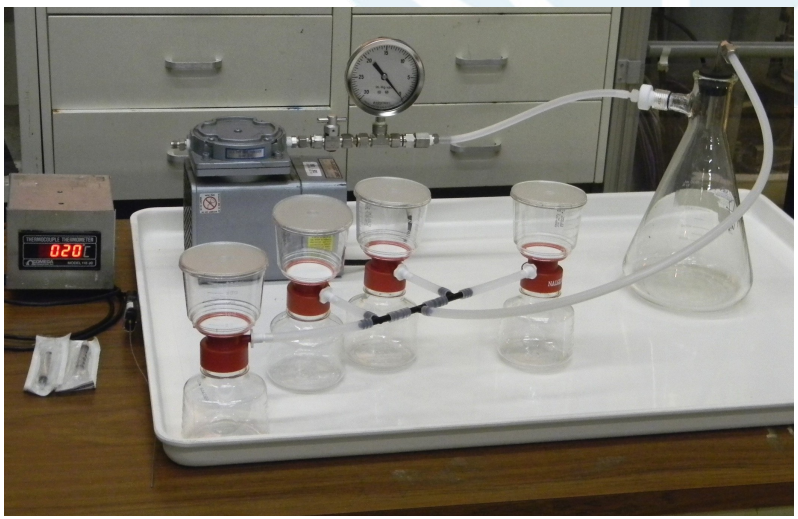
- 3 different filters
- Challenging Slurry
- Filter Pretreatment
- Cake Development
- Filter Evaluation with Filter Enhancer

Dead-End Filter Test

Evaluated:

- 2 slurries for filtration
  - Sludge Batch 6 and Tank 8F (non-radioactive simulants)
- 2 solids loading for filtration
  - 5 wt% and 0.1 wt%
- 4 filter aids
  - SiC, TiO2, and 2 activated charcoals
- 2 body feeds
  - PEO and HX200

Dead-End Test Set Up



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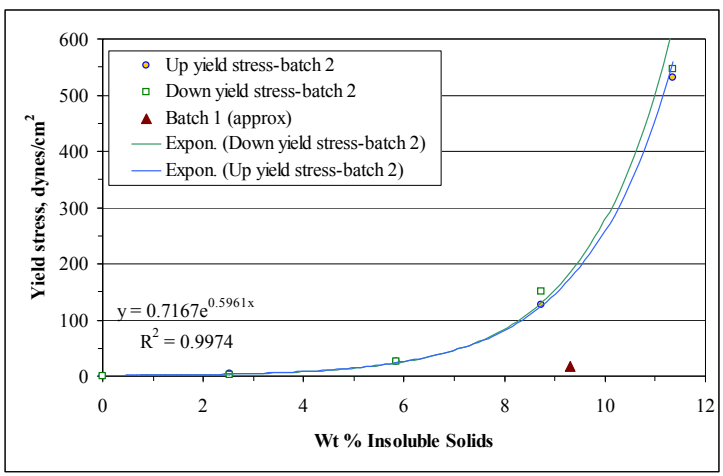
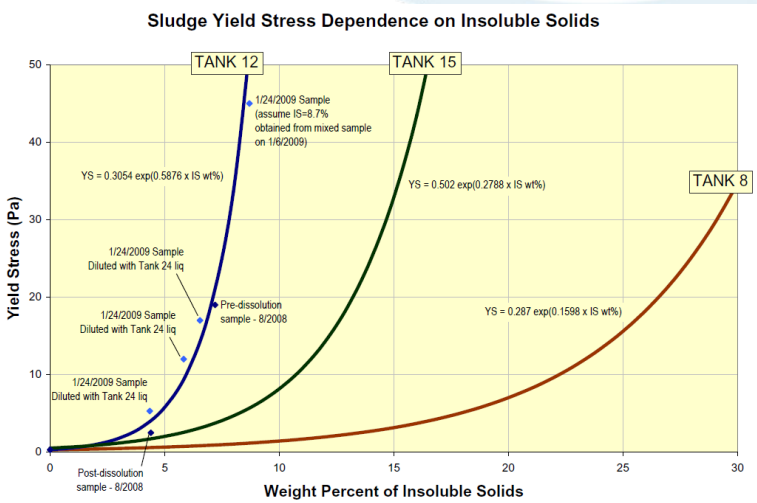
Slurries to test filter

Slurries SRS Tank 8F and Sludge Batch 6 (SB6) were evaluated to determine the more difficult to filter. SB6 with the smallest particles and complex chemistry was more difficult. Some properties* of SB6 sludge are:





Note: 10 dyne/cm^2 = 1 pascal

*Information from D. T. Herman, SRNL

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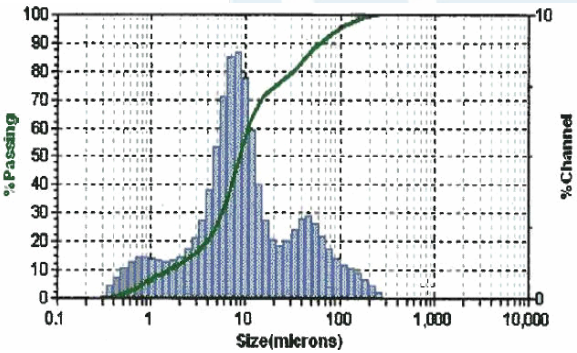
SB6 Sludge Simulant Properties*

Component	Calcined Solids Wt %	Calcined Solids Wt %
	Target	*Actual*
Al1	16.181	15.8
Ca	1.147	1.08
Ce	0.085	0.08
Cu	0.085	0.1
Fe	17.743	18.02
K	0.021	0.24
La	0.074	0.08
Mg	0.552	0.55
Mn	5.982	6.31
Na	19.305	17.77
Ni	2.231	2.3
S	0.712	0.28
Si	1.232	1.52
Zn	0.053	0.06
Zr	0.234	0.22
Sum	66.03	64.4



	Target	*Actual*
Slurry density g/mL	1.12 ±0.05	1.12
Total Solids, wt %	18.17 ± 2%	16.7
Insoluble Solids, wt %	14 ± 1%	10.4
Anions		
Nitrite, NO_2^-	8807 ±10%	11100
Nitrate, NO_3^-	6096 ±10%	6470
Phosphate, PO_4^{3-}	27 ±25%	<100
Sulfate, SO_4^{2-}	904 ±25%	1060

Simulant properties as-received
Yield Stress 54.56 Pa, Consistency 17.79 cP
Note:It was subsequently mixed with
5.6 M supernate to obtain desired solids loading.

*Information from D. T. Herman, SRNL

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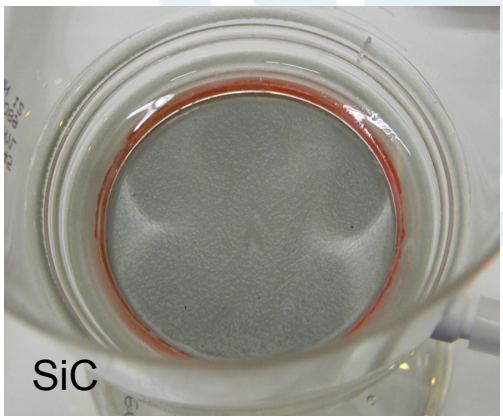
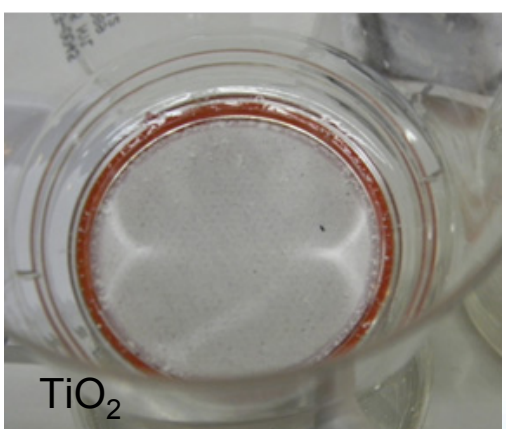
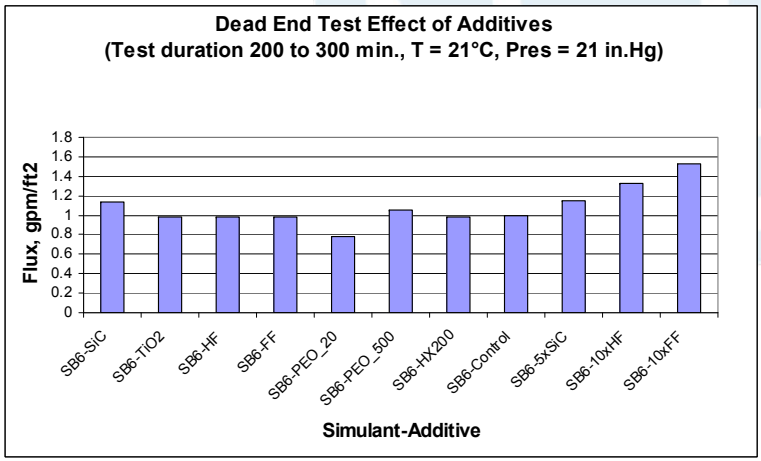
Filter Aid Application

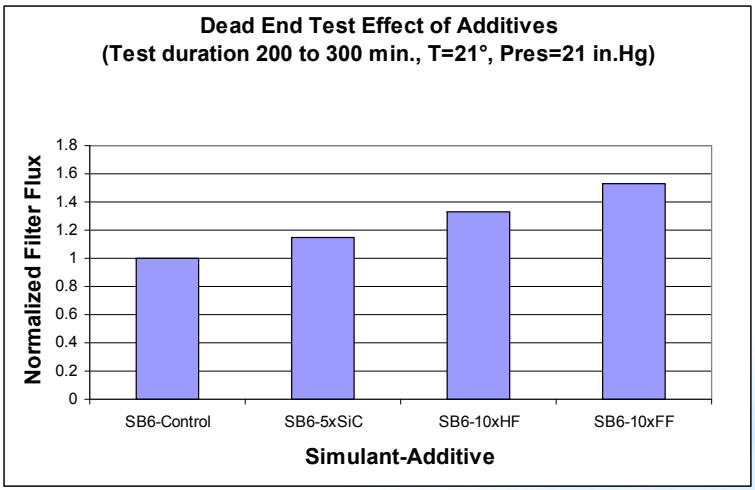




Dead-End Test Results

- Top figure compares all the filter enhancer against the Control
- Control = filtering without any enhancer
- Bottom figure shows the most promising candidates





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Dead-End Test Conclusions

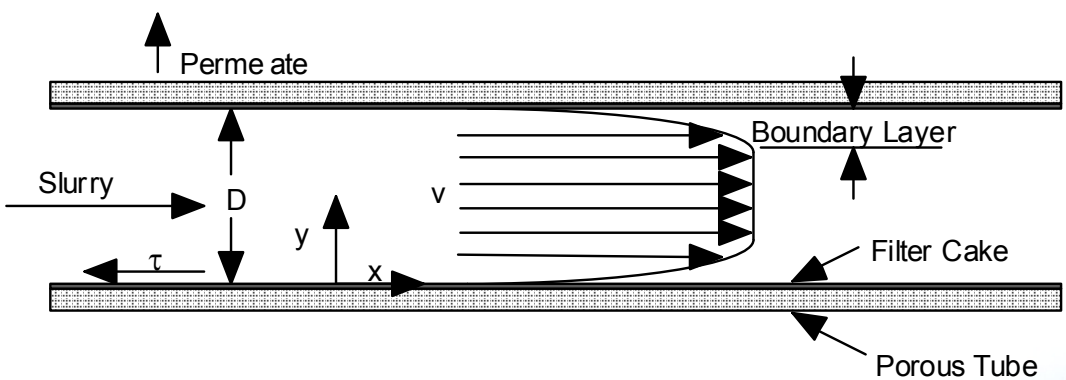
- The nature of the substance to be filtered has a significant effect on filter performance
- Dead-end filter element can be thoroughly coated with filter aids
- The structure of filter cake significantly affect filter performance as evidence with filter aids
- SB6 is more difficult to filter thank Tank 8F
- The higher solids loading is more difficult to filter
- Activated Charcoal resulted in the best filter rate
- Body feeds had no effect

Cross-Flow Filtration

- Results from the dead-end filter test are used in the cross-flow test
- Most challenging slurry: SB6 (of two evaluated)
- Most challenging solids loading: 5 wt% (of two evaluated)
- Most effective filter enhancer: (Norbit) DARCO S-51FF Activated Carbon (of 6 tested.)
- Test with different 0.1 micron media
  - Symmetric and Asymmetric designs
  - Different inside diameters
- Use standard flow conditions:
  - Axial Velocity = 12 ft/s, Transmembrane Pres. = 40 psid
  - Axial Velocity = 15 ft/s, Transmembrane Pres. = 50 psid

Clean by Scouring

- Scouring is running the filter at a high axial velocity and very little transmembrane pressure to use wall shear to clean surface fouling
  - Slurry
  - Water Only
- Velocities to try are 18 ft/s and 22 ft/s
- Standard WTP and SWPF velocities are 12 to 15 ft/s



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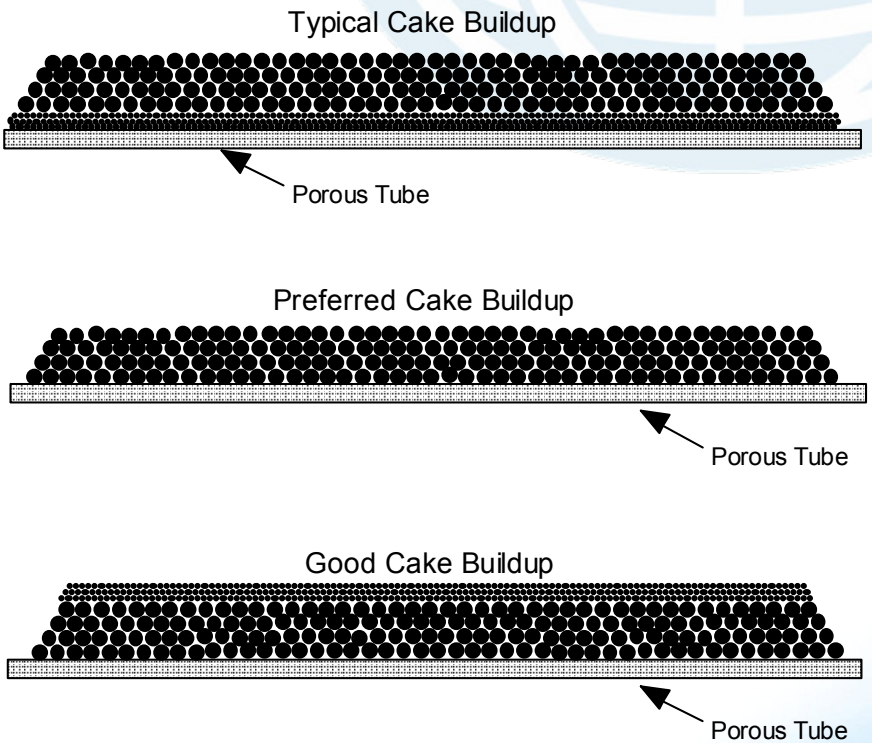
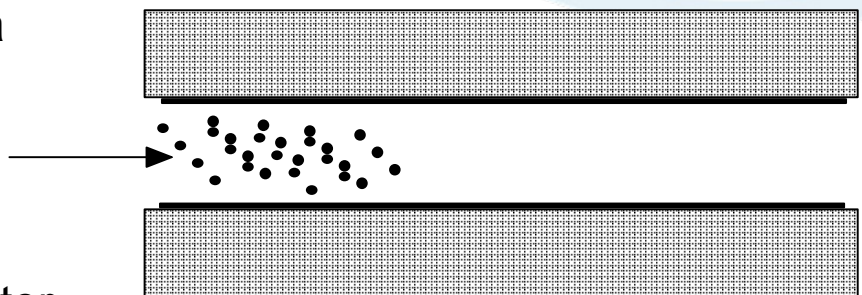
Develop Permeable Cake with Flow Conditions

- Develop more permeable cake via flow conditions by:
  - Start with no filtrate flow
  - Low TMP
  - Then increase standard flow conditions
  - Very slowly establish filtrate flow



Improve Permeability with Filter Aides

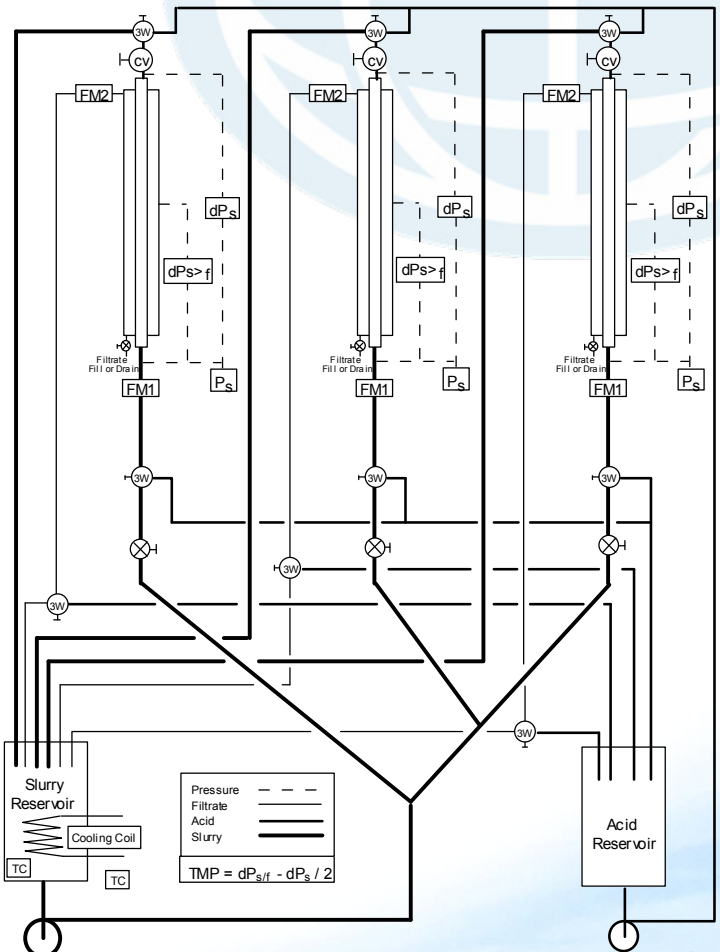
- Some filter enhancer, like filter aids, are made to coat the filter surface to build a more permeable cake



- The black coating on the filter in the figure at right indicates the precoat of filter aid waiting to develop the filter cake

Cross-Flow Filter Test Equipment

Three different filters fed from the same source at the same time for all three filters to experience the sample slurry and flow conditions to make a side be side comparison.



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Why Three Tubes?

Two study the performance of existing equipment:

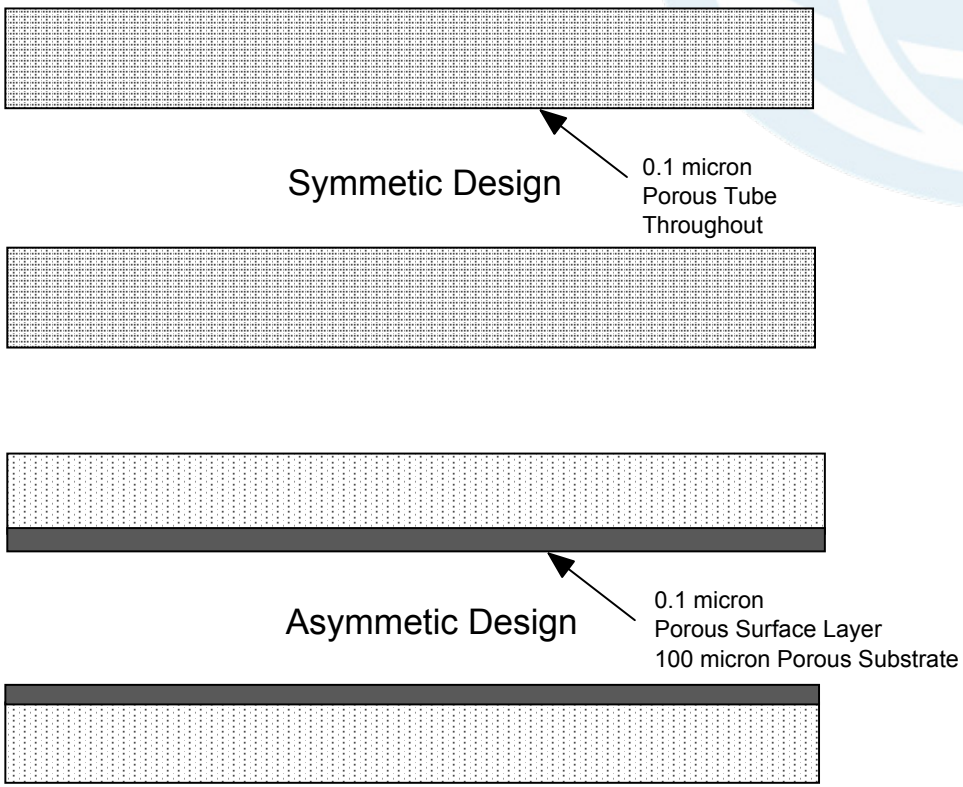
- Mott 3/8-inch ID Symmetric (SWPF)
- Mott 1/2-inch ID Symmetric (WTP)

and the performance of a different design:

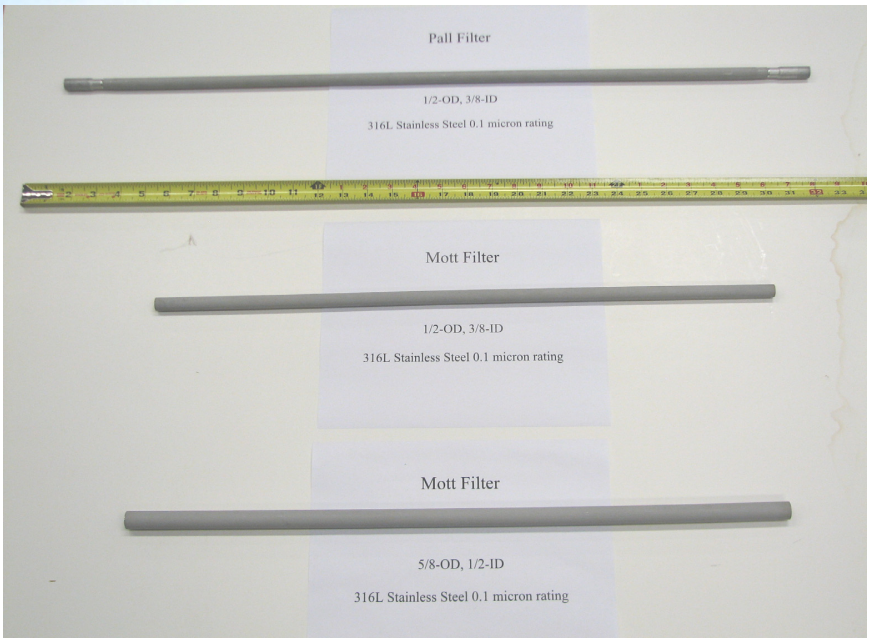
- Metal Substrate w/ceramic (ZrO_2) inner coating
  - Pall (Asymmetric)

Notes: The metal of all three tubes is 316L Stainless Steel, the Mott tubes are a nominal 0.1 micron rated pore size, and the Pall tube is an absolute 0.1 micron rated pore size

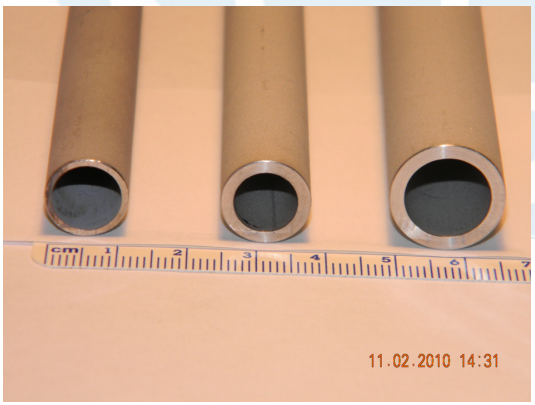
Why Three Tubes?



Actual Filter Tubes Tested



All tubes were cut to the same length and the active filter length was 22.5 inches



- Above figure shows tube at:
  - Left: 3/8” I.D. Pall, asym
  - Middle: 3/8” I.D. Mott, sym
  - Right: 1/2” I.D. Mott, sym

I.D.=Inside Diameter

Asym = asymmetric design

Sym = symmetric design

Cross-Flow Filter Test Equipment

Photographs show the three installed filter tubes and the to the left show a close up of 3/8-inch Mott tube in its filter housing.

Cross-Flow Filter Test Equipment

Photograph shows computer and the two 10 hp slurry pumps used to operate the cross-flow filter test.

Photograph shows reservoir charged with SB6 slurry at a solid loading of 5 wt% and the filtrate of the combined three filters returning

X-Flow Preliminary Results (Test still in Progress)

- Slow start up with clean filters to properly coat surface:
  - Filtrate Flow is closed
  - Slowly fill filter tubes and allow the slurry flow system to reach steady state at approximately 1 ft/s axial velocity and at the minimum slurry pressure
  - Very slowly open filtrate valves to allow filtrate system to fill. (Noted was the fact the system was partially full already as liquid pushed air out of the filtrate system and into the slurry and very slow is 10 to 15 minutes from no to full filtrate flow.)
  - Once the filtrate system is full, shut the filtrate valves.
  - Bring the system to the standard flow conditions, e.g., AV = 12 ft/s, TMP = 40 psid; AV = 15ft/s, TMP = 50psid
  - Very slowly open the filtrate valves to allow filtrate flow to begin.

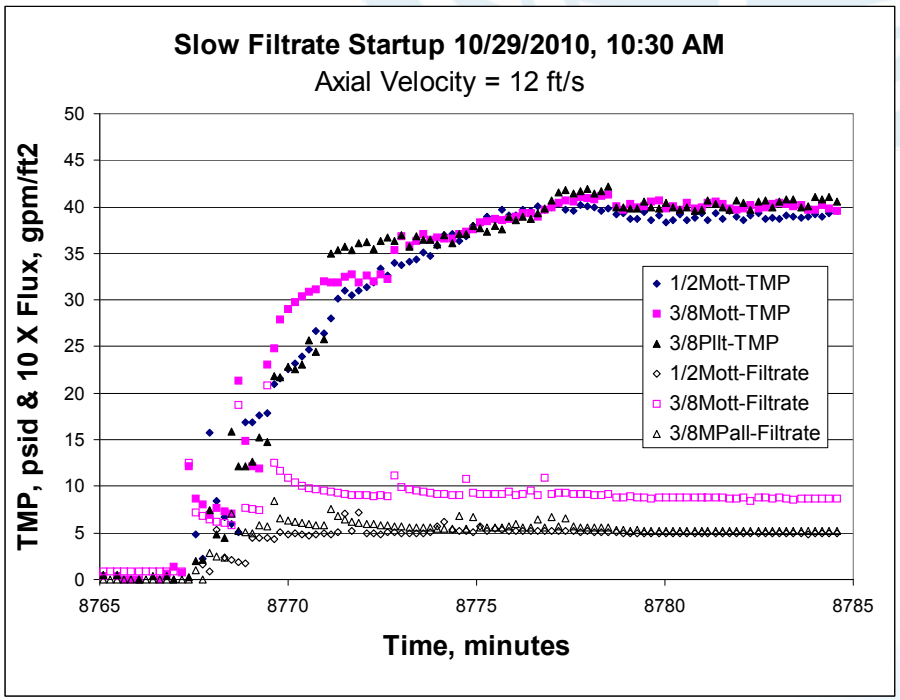
AV = Slurry Axial Velocity in filter tube

TMP = Transmembrane Pressure

X-Flow Preliminary Results (Test still in Progress)

- Very Slow Filtrate Flow Engagement Defined:
  - First the flow standard slurry flow conditions are set. In the case at the right that was AV = 12 ft/s and TMP = 40 psid.
  - Then over approximately 15 minutes the filtrate flow is established

Note: The filtrate fluxes are multiplied by 10 to be located along side the TMP data. Also the minutes being in the 6th day of this test.



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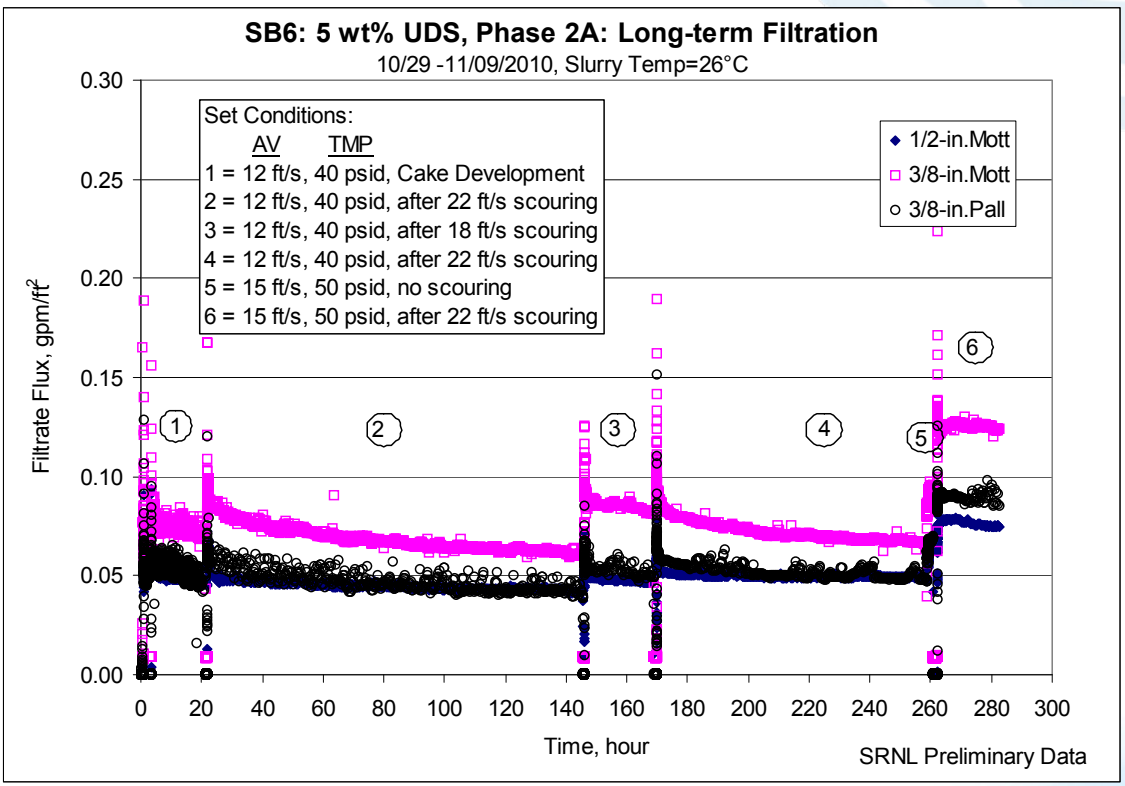
X-Flow Preliminary Results (Test still in Progress)

- This long term test was done at a constant solids loading
- The individual zone are highlight in the following slides



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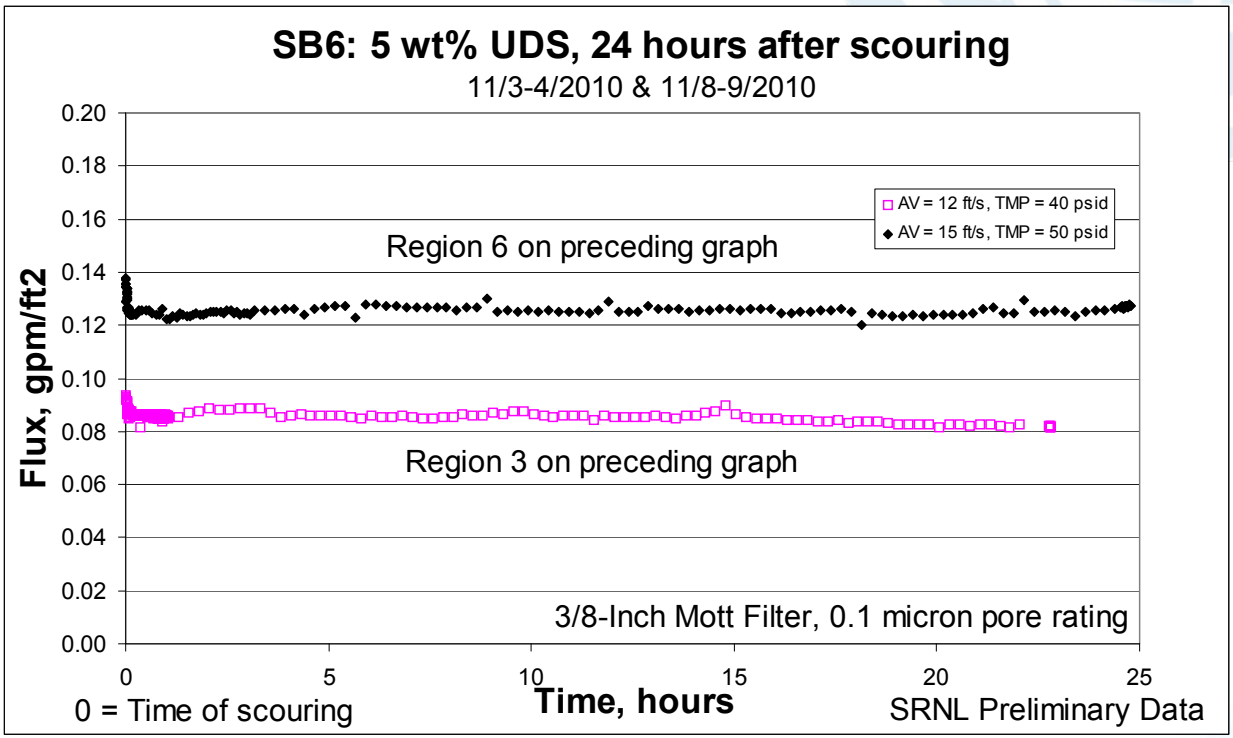
X-Flow Preliminary Results (Test still in Progress)

- After cake is developed filtrate flux is high, long-lasting, and steady after 15 minutes of scouring



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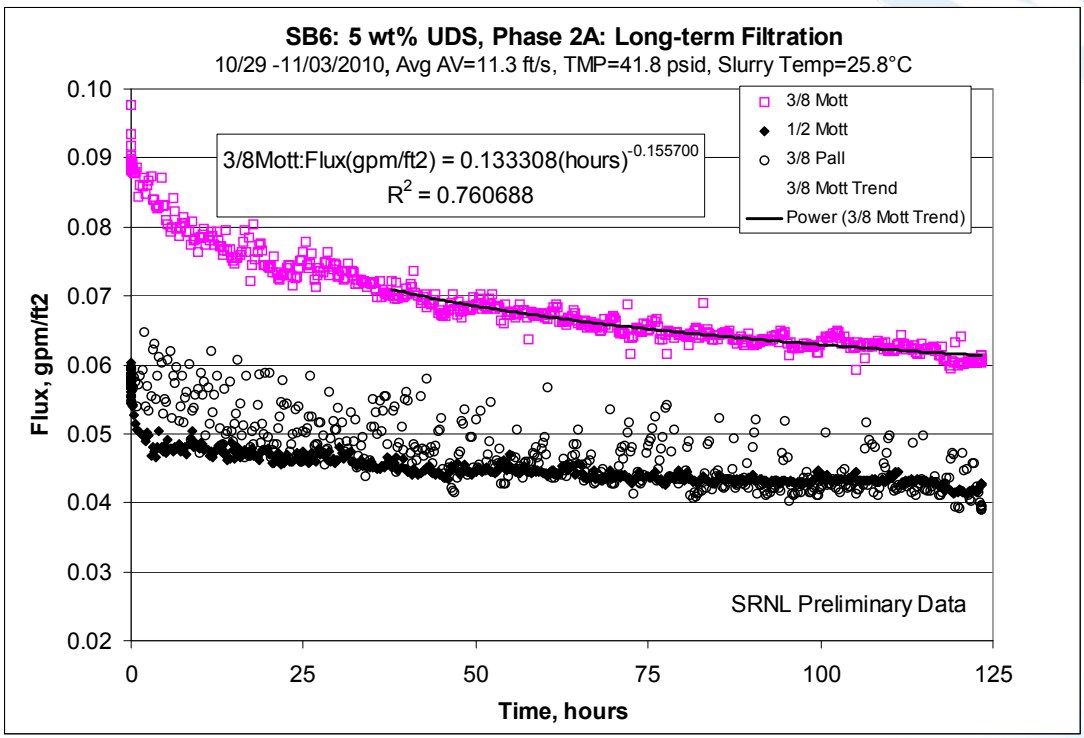
X-Flow Preliminary Results (Test still in Progress)

- Using the 125 hours of zone 2, shown on the preceding slide, a long-term trend can be estimated, shown below
- The power-law fit was done over the one-hundred-hour period where the decay was uniform.



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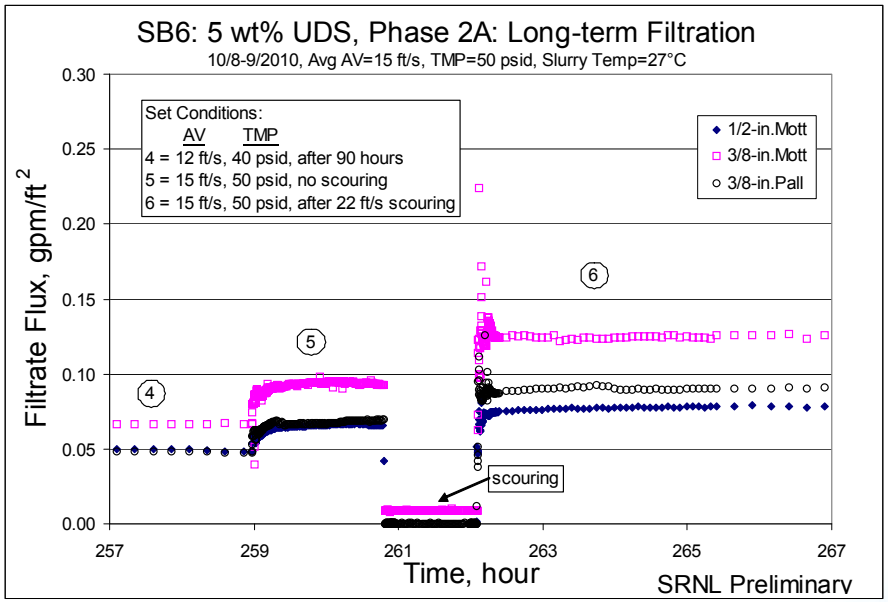
X-Flow Preliminary Results (Test still in Progress)

- Increasing flow conditions from AV = 12 ft/s and TMP = 40 psid to 15 ft/s and 50 psid the following was observed:
  - Increasing flow conditions the filtrate flux increases 40% over the previous conditions, however the increase was only 10% from when the previous conditions stated.
  - By scouring leads to a more significant recovery of filtrate flux than simply increasing the slurry flow conditions.



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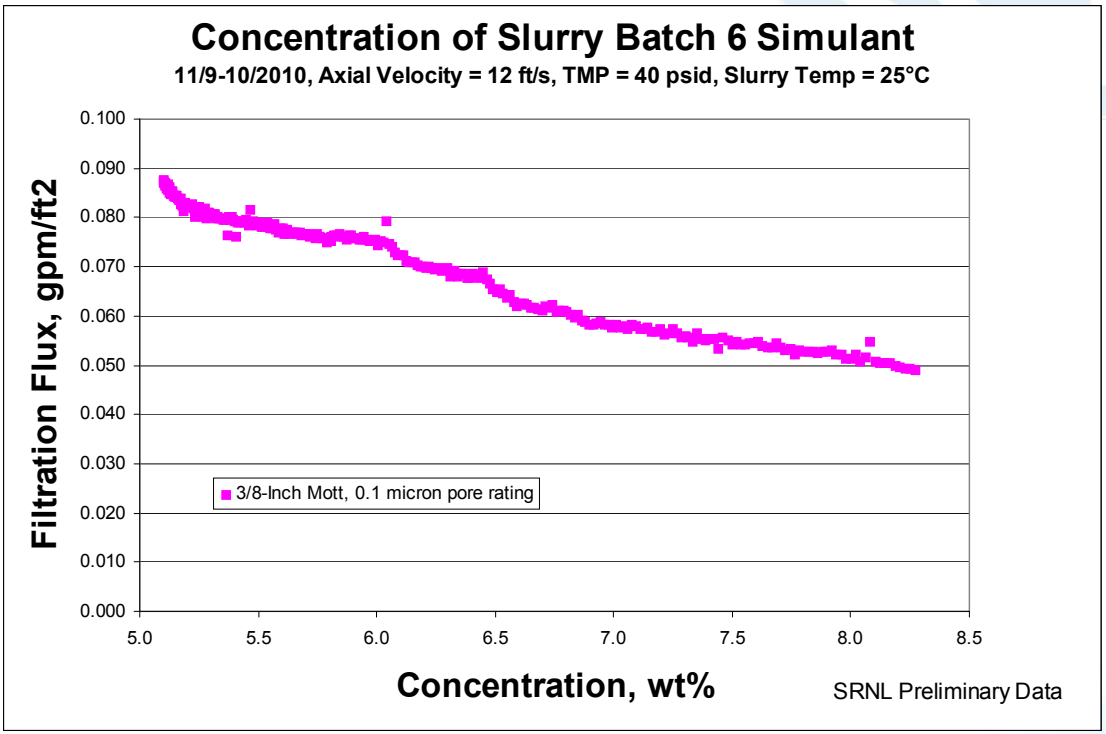
X-Flow Preliminary Results (Test still in Progress)

Initial results indicate that even when a waste is concentrated the filtrate flux remains strong



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X-Flow Preliminary Conclusions (Test still in Progress)

- To obtain a high, long lasting, and relatively stable filtrate flux it is fundamental to start the filter properly in order to develop a more permeable cake.
- Once the cake is established it is the basis for all filtrate fluxes
  - Shutting and opening filtrate flow creates not loss in flux, e.g.g open and closing a valve, pump temporary failure, etc
  - Scouring after any length of period, e.g., 1, 10, 100 hours, returns the flux to what it was at last scouring when using the same axial velocity and transmembrane pressure
  - Changing the slurry flow conditions without scouring at time x increases or decreases the filtrate flux to what it would have been if started at the new flow conditions

Preliminary Conclusions (Test is still in Progress)

- Of the three filters tested side-by-side the 3/8-inch inside diameter Mott filter performed the best
- Increasing flow conditions (from AV = 12 ft/s and TMP = 40 psid to 15 ft/s and 50 psid) the filtrate flux increase 40% over the previous conditions, however the increase was only 10% from when the previous conditions stated.
- By scouring before setting the new conditions the increase was more than an 80% over the previous conditions of 12 ft/s and 40 psid and better than 40% from when that previous conditions stated. Furthermore, scouring allowed the new conditions to produce a filtrate flux of 30% better than the none scoured filtrate flux. This is comparable to the drop in filtrate flux drop that occurred over 90 hours at 12 ft/s and 40 psid. This implies that cake on the filter is well established and once established it is only the slurry flow conditions that control the filtrate flux.
- Flux at high solids loading is ongoing
- Flux with the use of filter enhancer to begin 11/22/2010

Preliminary Conclusions (Test is still in Progress)

- Cleaning with just water improved flux but did not return filter flux to clean filter.
- Cleaning with 0.5 M Oxalic acid it is important to give time to soak:
  - After 1 hour of cleaning improvement was minimal
  - After 24 hours of soaking improvement was significant but still much lower that expected.
  - After 36 hours of soaking improvement was very significant. The filter flux increase 3 fold.

Still to be completed

- Flux at high solids loading is ongoing
- Flux with the use of filter enhancer to begin 11/22/2010

Future Work

- Multitube Operation
- Pilot Scale (length & Tube Diameter)
- Perfect Cake Development Technique
- Perfect Scouring Technique
- Use a larger range of solids loading (1 to 15 wt%)
- Perfect need for cleaning