

**CROSS-FLOW FILTRATION OF SIMULATED HIGH-LEVEL WASTE SLUDGE  
(TANK 8F)**

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## SUMMARY

The Filtration Research Engineering Demonstration (FRED) at the University of South Carolina performed engineering-scale tests to determine crossflow filter performance with a 5.6-M sodium solution containing varying concentrations of sludge and monosodium titanate (MST). The current tests investigated filter performance with slurry containing simulated Tank 8F Sludge at concentrations between 0.044 wt % and 4.80 wt %. Testing used a slurry containing 3.5 wt % Tank 8F simulated sludge and a target concentration of 0.96 wt % MST.

The tests evaluated filter performance at processing conditions approaching the limitations of the pilot plant in three phases. Phases I and II determined the operating limits of the facility. Phase III measured the filter performance at statistically chosen conditions. In addition to determining filter flux as a function of operating conditions, the study investigated hysteresis by evaluating the ability of the filter to recover to a previously obtained filter flux. If the testing could not recover previously measured filter performance when operating at the same conditions, then personnel initiated a sequence of recovery procedures that included scouring and backpulsing.

Test #1 with Tank 8F Sludge and MST stopped when the filtrate flux rates proved excessively low. Test #2 with Tank 8F Sludge and MST cleaned prior to filtering but stopped due to the appearance of particles in the filtrate. Diagnostic testing and repair of the equipment continues.

The key findings of the investigation are the following:

- Feed solutions containing only simulated sludge filtered more slowly than feed solutions containing sludge and MST. We expected this result since the MST has a larger mean particle size and a narrower particle size distribution than the simulated sludge.
- Filter flux depends strongly on transmembrane pressure. Increases in flux correlate with increases in transmembrane pressure, consistent with crossflow filtration theory.
- Filter flux decreases with increasing insoluble solids concentration, as expected and in agreement with crossflow filtration theory.

## INTRODUCTION

The Salt Disposition Systems Engineering Team selected three cesium removal technologies for further development to replace the In Tank Precipitation process: Small Tank Tetraphenylborate Precipitation, Non-elutable Ion Exchange using crystalline silicotitanate (CST) as the sorbent, and Caustic-Side Solvent Extraction.

As a pretreatment step for the CST and solvent extraction flowsheets, the operation contacts the incoming salt solution containing entrained sludge with MST to adsorb strontium and plutonium. The process then filters the resulting slurry to remove the sludge and MST. The baseline solid-liquid separation technology is crossflow microfiltration with a 0.5  $\mu$  Mott filter. Cesium removal occurs by contacting with CST in an ion exchange column or processing through a solvent extraction system.

Previously, researchers investigated cross-flow filtration of simulated waste stream for this project.<sup>1,2</sup> The current tests follow the guidance provided by Westinghouse Savannah River

Company<sup>3</sup> and in part fulfills the requirements for the current work scope matrix (Items 6.3 and 6.5)<sup>4</sup>. The test objectives involved identifying the impact of removing MST from the feed solution and investigating the impact of changes in sludge composition on filter performance. The testing occurred in the FRED facility shown in Figure 1. The FRED facility contains a filter element with seven Mott filter tubes. Each tube is made from sintered stainless steel, 0.75 inches OD, 0.625 inches ID, 10 feet long, and nominal 0.5 micron pore size.



**Figure 1. Filtration Research Engineering Demonstration**

## **EXPERIMENTAL APPROACH**

### **Test with Tank 8F Sludge Only in 5.6M Sodium Solution**

Prior to filtering, personnel cleaned the system with oxalic acid followed by sodium hydroxide. Following cleaning, researchers measured the clean water flux using deionized water to verify the cleaning effectiveness.

The simulated waste slurry contained 5.6 M sodium “average” Savannah River Site salt solution prepared according to the instructions provided by WSRC.<sup>3</sup> Table 1 shows the concentrations of the components in the solution.

**Table 1. 5.6 M Sodium Average Salt Solution**

<u>Species</u>	<u>Concentration (M)</u>
Na	5.6
K	0.015
Cs	0.00014
OH	1.91
NO <sub>3</sub>	2.14
NO <sub>2</sub>	0.52
AlO <sub>2</sub>	0.31
CO <sub>3</sub>	0.16
SO <sub>4</sub>	0.15
Cl	0.025
F	0.032
PO <sub>4</sub>	0.01
C <sub>2</sub> O <sub>4</sub>	0.004
SiO <sub>3</sub>	0.004
MoO <sub>4</sub>	0.0002

Personnel then added Tank 8F simulated sludge to reach a measured concentration of 0.044 wt %. Testing occurred with the temperature of the solution controlled to 35°C ( $\pm 3^\circ\text{C}$ ).

Phase I filtration involved filtering the solution with axial velocities that incrementally decreased from a maximum to a minimum and then incrementally increased back to the maximum while measuring TMP and filtrate flow. The maximum axial velocity (~ 26 ft/s) occurred with the Filter Feed Pump at 90% of maximum speed, and the Slurry Flow Control Valve completely open, corresponding to minimum hydraulic resistance. Personnel set the minimum axial velocity for Phase I at 18 ft/s. Personnel also measured filter performance at two points equidistant between the maximum and minimum axial velocity. We obtained axial velocities below the maximum by only reducing the Filter Feed Pump speed. On increasing axial velocity, if the filtrate flow did not return to 95% of the value previously obtained on decreasing axial velocity, personnel took remedial action. The remedial action involved scouring at increasing axial velocities until the target flux was reached. If these scouring proved insufficient, then personnel performed as many as two backpulses. If these previously defined remedial actions proved insufficient, personnel performed various combinations of additional scouring and backpulsing.

Phase II filtration involved filtering the solution with axial velocities incrementally decreased from a maximum to a minimum and then incrementally increased back to the maximum. The maximum axial velocity occurred with the Filter Feed Pump at 90% of maximum speed, and the Slurry Flow Control Valve completely open. Personnel set the minimum axial velocity for Phase II at 12 ft/s. As in Phase I, personnel also measured filter performance at two points equidistant between the maximum and minimum. We obtained axial velocities below the maximum by placing the Slurry Flow Control Valve in automatic control with a setpoint that corresponded to the desired axial velocity. The Filter Feed Pump speed remained at 90% of full speed for the duration of this Phase. On increasing axial velocity, if the filtrate flow did not return to 95% of the value obtained on decreasing axial velocity, personnel took remedial action. Remedial action included the same protocols as for Phase I.

During Phase III filtration, we investigated system performance at as many as 17 statistically selected points within the system limits developed by Phases I and II. The selected operating points complement the previous test points for the model of interest using statistical methods available in JMP® Version 3.2.2 (a commercial software product from SAS Institute, Inc. of Cary, N.C.). The model of interest is a response surface model in axial velocity and TMP, which included nonlinear effects, parameter interactions and hysteresis effects. We developed a grid of points covering the factor space for axial velocity and TMP. We used the JMP® D-Optimality algorithm to select an optimal set of test conditions (of a specified number) to augment the existing data points from the previous test phases for this response surface model.

Personnel repeated these three test phases at the following insoluble solids levels: 0.214 wt %, 0.884 wt %, and 4.8 wt %.

### **Test #1 with Tank 8F Sludge and MST**

Personnel did not clean the filter prior to the start of this test, as requested in the WSRC test plan. The slurry that remained from the end of the previous test (containing only Tank 8F sludge) served as the feedstock. We added sufficient 5.6 M sodium solution to the slurry to lower the insoluble solids loading to 3.52 wt %.

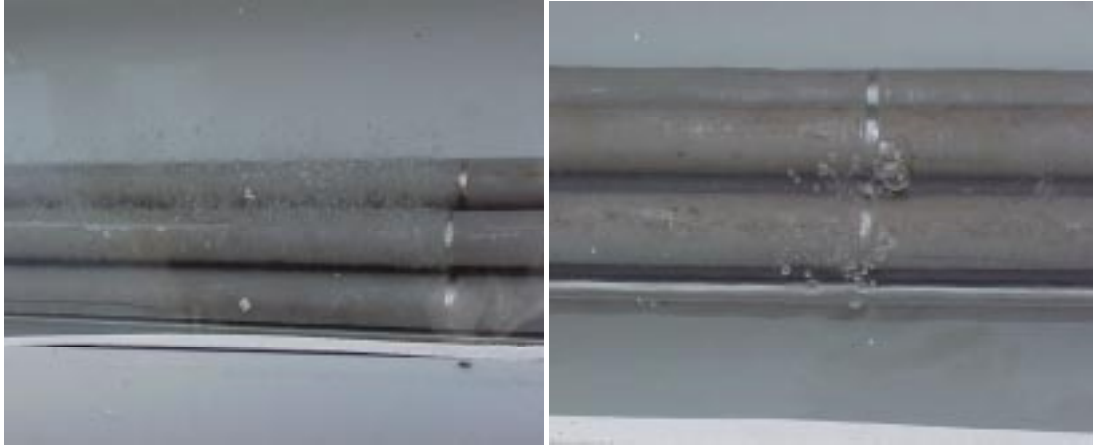
Personnel then added MST to the slurry while it circulated through the system to reach a target loading of 0.96 wt % of MST. Testing started using the Phase I filtration methodology previously described. Personnel observed no measurable filtrate flow during Phase I. Phase II testing only produced filtrate at the minimum and low axial velocities (i.e., the highest TMPs investigated). After reviewing this data, we concluded the filter needed cleaning and halted the test.

### **Test #2 with Tank 8F Sludge and MST**

We drained the slurry remaining from the previous test from the Slurry Tank and stored it in drums. The filter loop was then chemically cleaned first with oxalic acid and then with sodium hydroxide. A second water flush proved necessary before the clean water flux test gave satisfactory results. Personnel then returned the stored slurry to the Slurry Tank.

Personnel completed Phases I and II of the test using the same procedure as previous testing. The filtrate flux improved over Test #1. During Phase III, personnel observed a slight darkening of the filtrate. Analysis showed the presence of solids. Personnel suspended the test, drained the slurry to drums, and began an investigation of the filter failure.

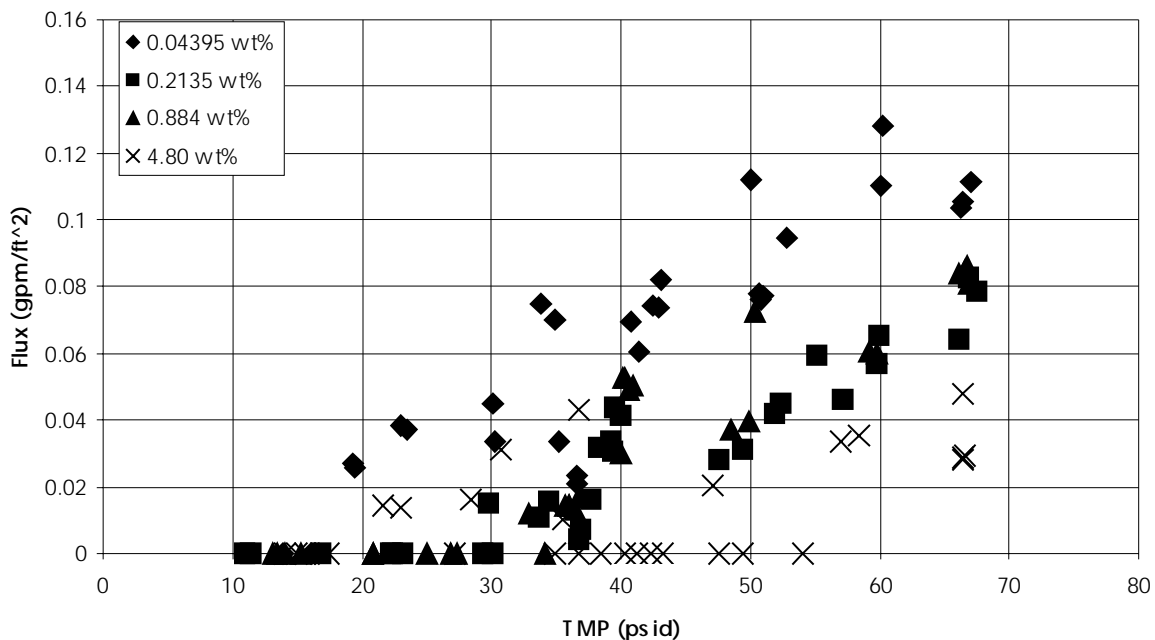
After discussions with representatives from Mott Metallurgical (the manufacturer of the filter) and WSRC personnel, FRED researchers performed a test by bubbling air through each filter tube individually with the filter submerged in water. Figure 2 shows typical photographs of the bubbles observed during testing. This test proved inconclusive, as we did not identify a location with significantly larger bubbles emerging. Bubbling was not observed along lines, indicating a possible crack, nor at a tube section weld. Rather, bubbling appeared non-uniform along any tube and at sizes consistent with theory.<sup>5</sup>



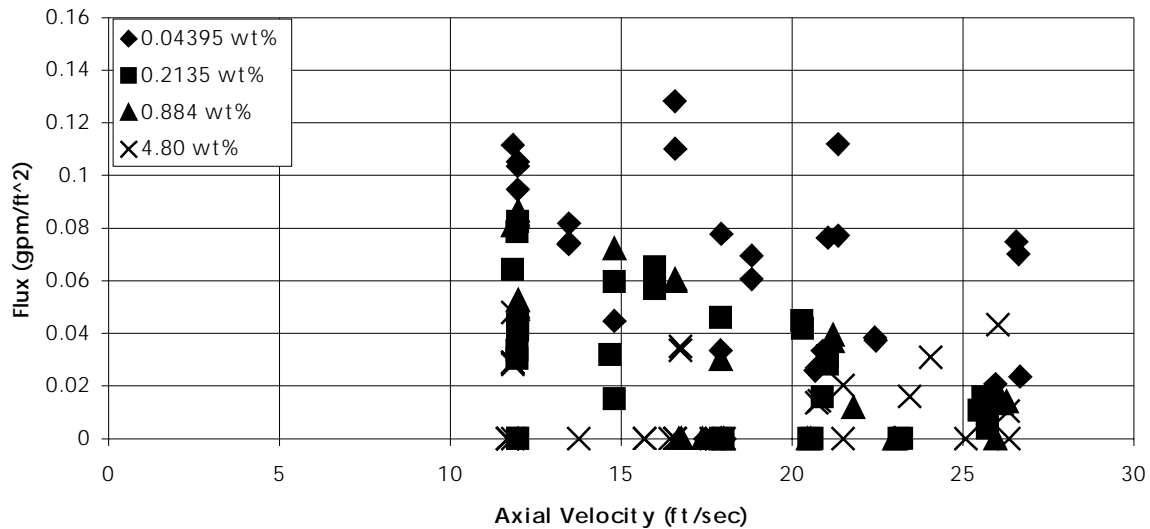
**Figure 2. Photographs of bubbles observed during testing of filter.**

## RESULTS

Figures 3 and 4 show the relationship between Filtrate Flux, TMP, and axial velocity in the Test with Tank 8F Sludge only. All insoluble solids levels are shown in Figures 3 and 4. The general trend shows that as insoluble solids concentration increases, flux decreases as expected from crossflow filtration theory.<sup>6</sup> Figure 2 shows a strong dependence of flux on TMP as expected from crossflow filtration theory.<sup>6,7</sup> No significant correlation between axial velocity and filter flux is observed in Figure 4.



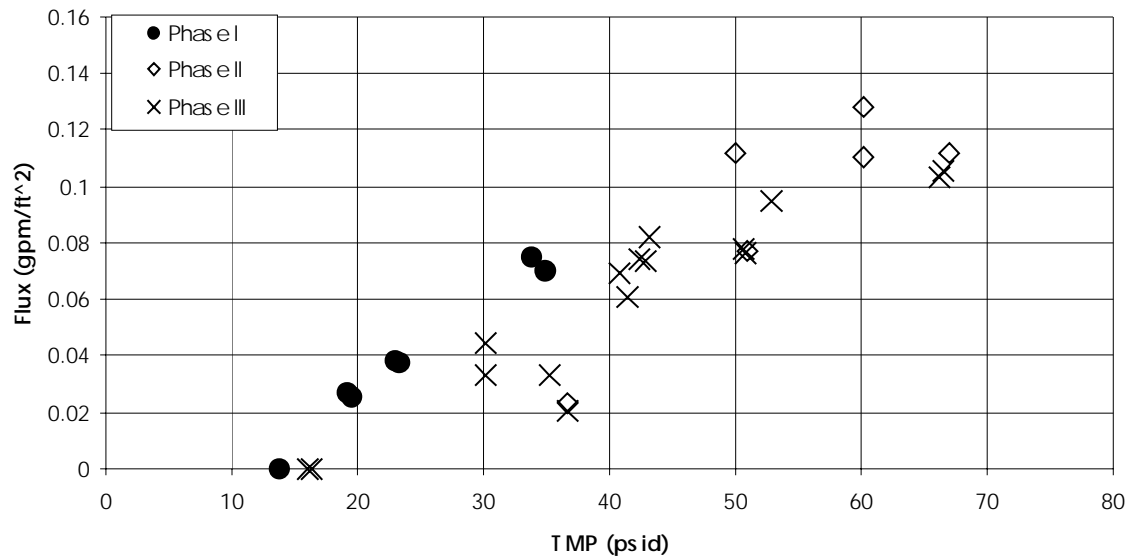
**Figure 3. Flux versus TMP during Test with Simulated Tank 8F Sludge Only**



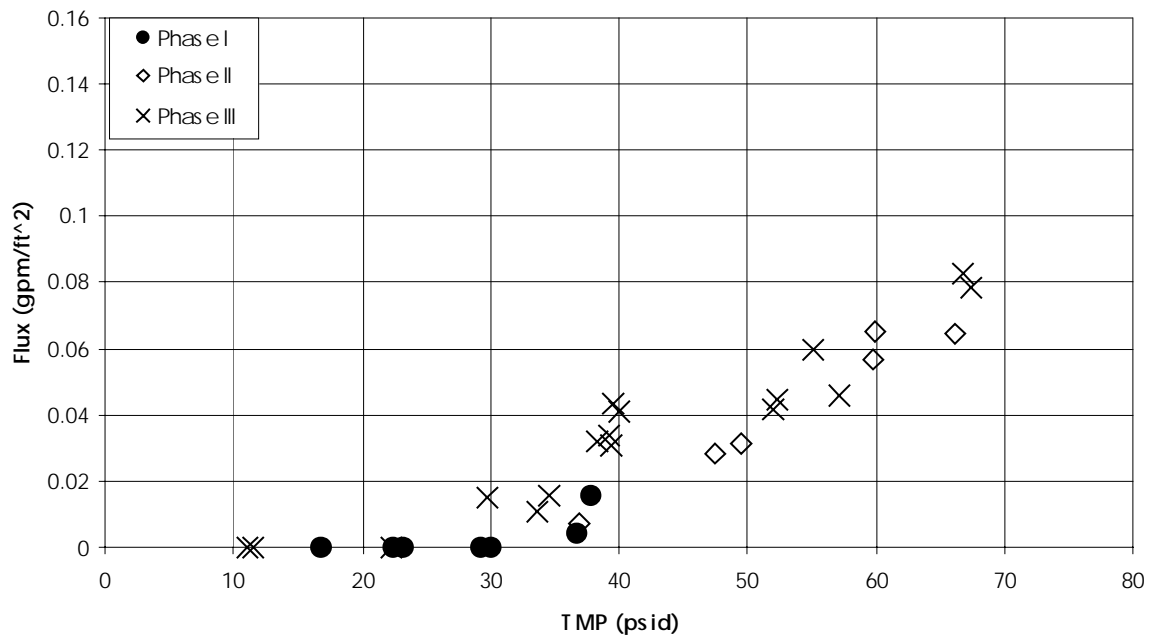
**Figure 4. Flux versus Axial Velocity during Test with Simulated Tank 8F Sludge Only**

Figures 5 - 8 show the relationship between filtrate flux and TMP for the test with Tank 8F Sludge, separated by solids loading. The results show a strong dependence on TMP as expected from crossflow filtration theory.<sup>6,7</sup> In Figure 8, a number of data points from Phase I have a higher flux than data points from other parts of the test at the same TMP. One plausible explanation is these points were the first data collected at this concentration and although the data did meet the steady state requirements defined in the test plan, it is possible that filter fouling at the high solids concentration occurs by additional mechanisms.

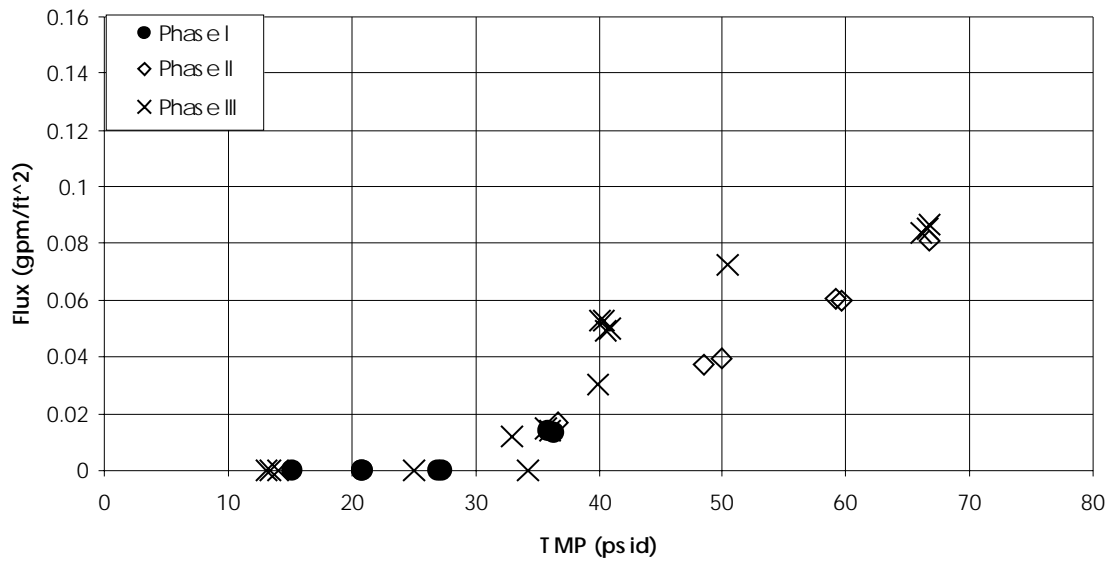




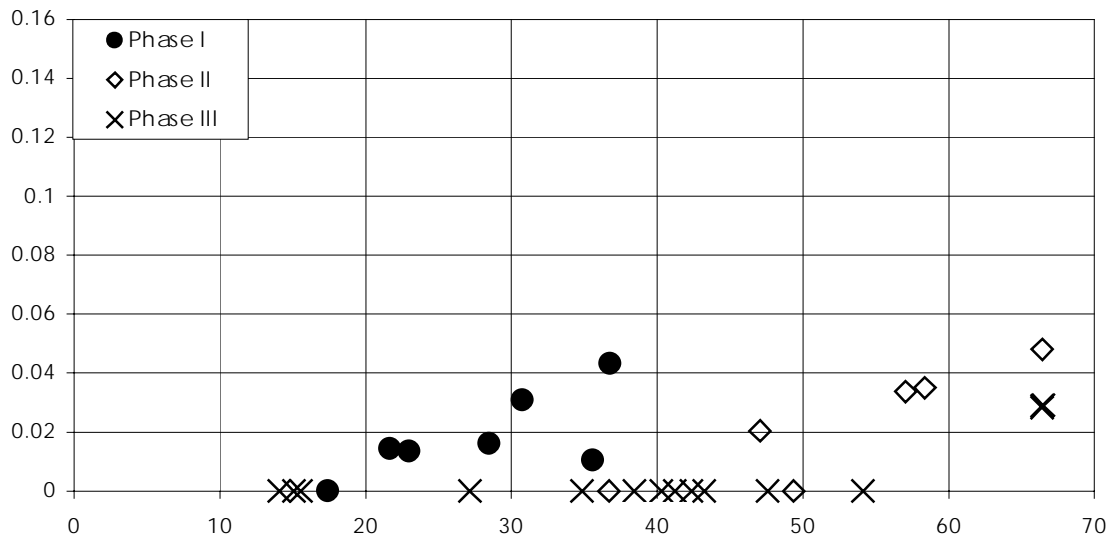
**Figure 5. Flux versus TMP with 0.044 wt % Tank 8F Sludge Only**



**Figure 6. Flux versus TMP with 0.21 wt % Tank 8F Sludge Only**



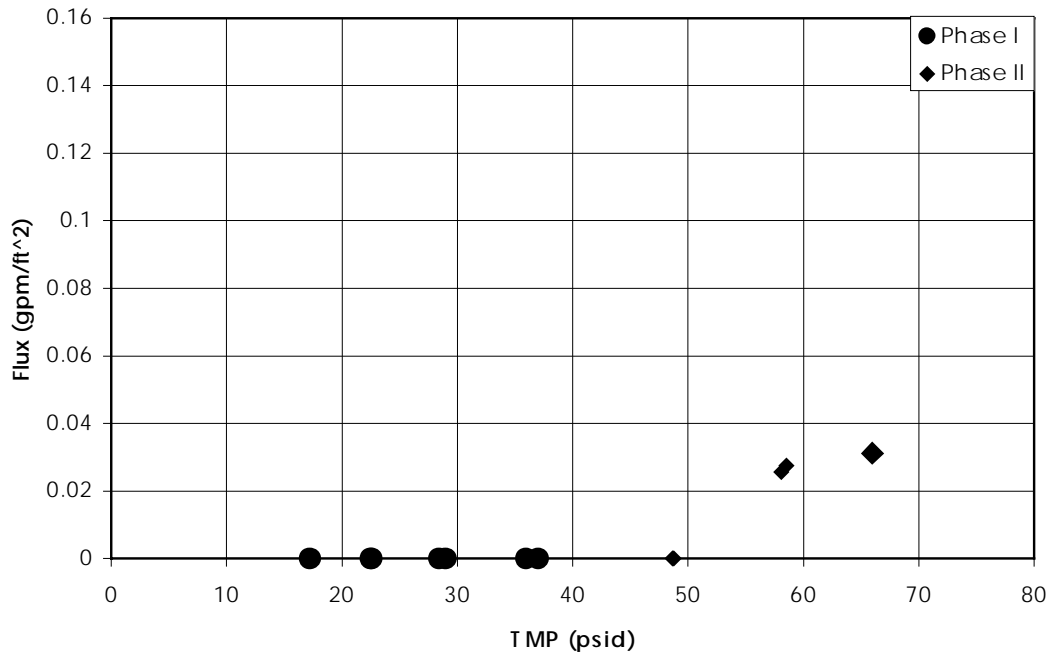
**Figure 7. Flux versus TMP with 0.88 wt % Tank 8F Sludge Only**



**Figure 8. Flux versus TMP with 4.8 wt % Tank 8F Sludge Only**

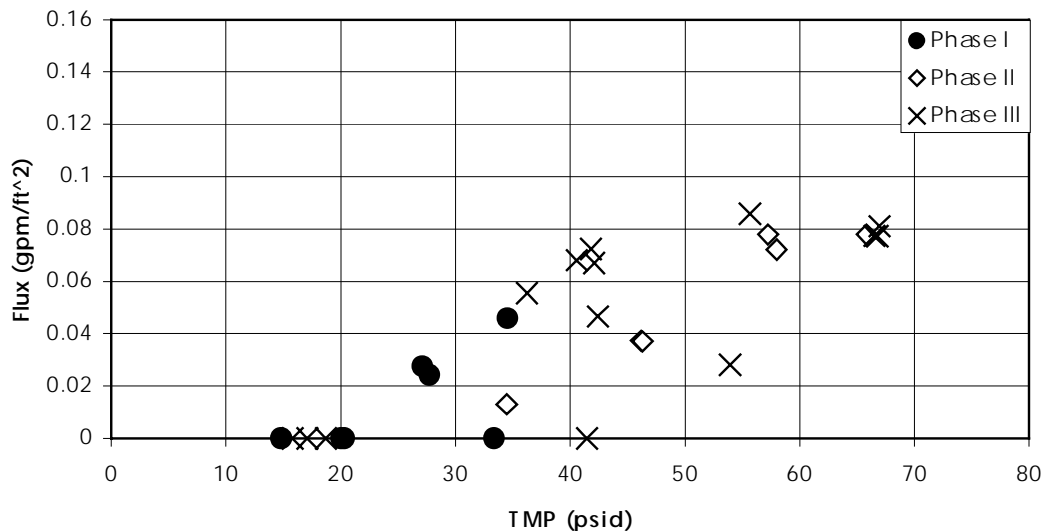
Figure 9 shows the relationship between filtrate flux, TMP, and axial velocity for Test #1 with (3.52 wt %) Tank 8F Sludge and (0.96 wt %) MST. The filter flux remained very low, and below the detection limit in many cases. This data suggests a severely fouled filter. Since we

did not clean the filter prior to this test, the filter fouling from the previous test affected the filter flux in this test as expected.



**Figure 9. Flux versus TMP during Test #1 with 3.52 wt % Simulated Tank 8F Sludge and 0.96 wt % MST**

Figure 10 shows the relationship between filtrate flux, TMP, and axial velocity for Test #2 with Tank 8F Sludge and MST. The feed for this test was the same as Test #1 with Tank 8F Sludge and MST. The filter flux is much higher than the filter flux during Test #1. The higher flux likely resulted due to the cleaning, as expected.

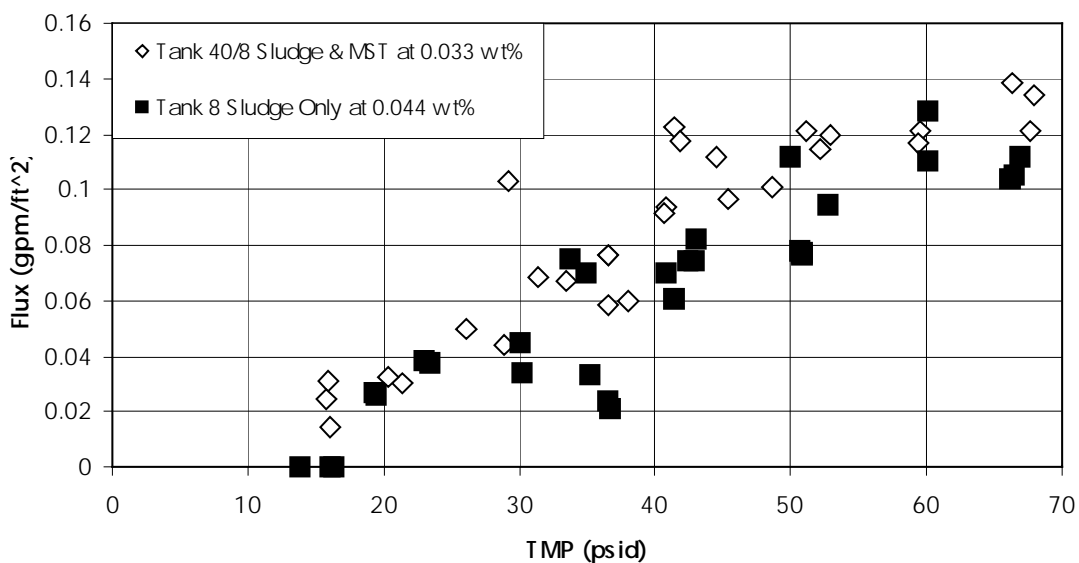


**Figure 10. Flux versus TMP during Test #2 with 3.52 wt % Simulated Tank 8F Sludge and 0.96 wt % MST**

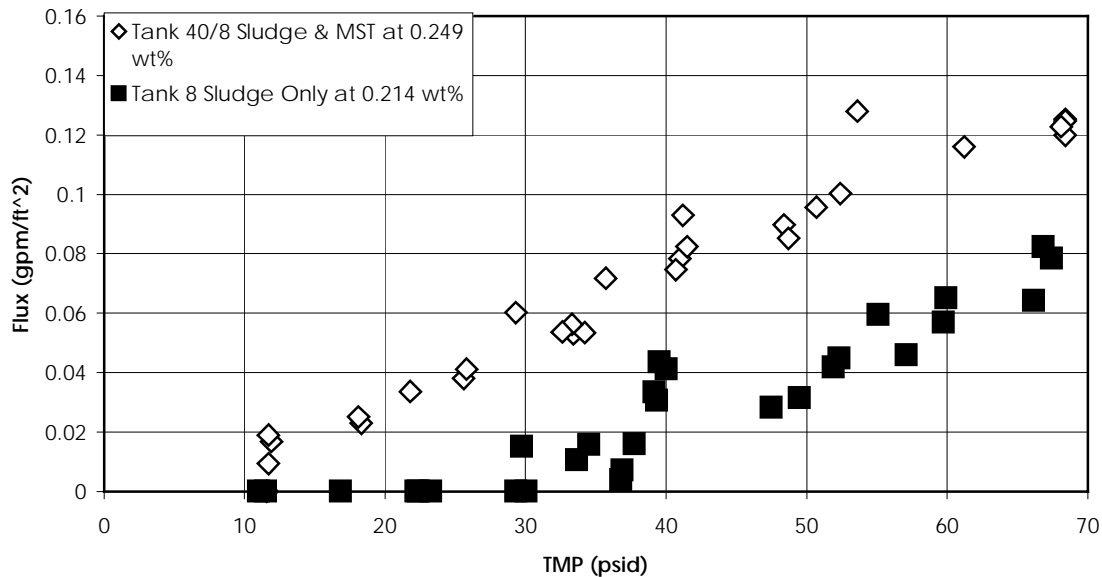
## COMPARISON BETWEEN TESTS

Figures 11 - 14 compare performance between the Tank 8F sludge test and the previous Tank 40H/8F sludge with MST test.<sup>2</sup> The figures show a lower filter flux with sludge only than observed with blend sludge and MST. This behavior occurs at each insoluble solids concentration and agrees with expectations. The sludge has a smaller mean particle size than MST (2.5  $\mu$  vs 10  $\mu$ ). In addition, the MST has a narrower particle size distribution. Crossflow filtration theory predicts larger particles yield higher flux.<sup>7</sup> Since the MST has a narrower particle size distribution, it will pack less efficiently, which will create a more open filter cake with a higher porosity. The higher porosity leads to higher filter flux.<sup>7</sup>

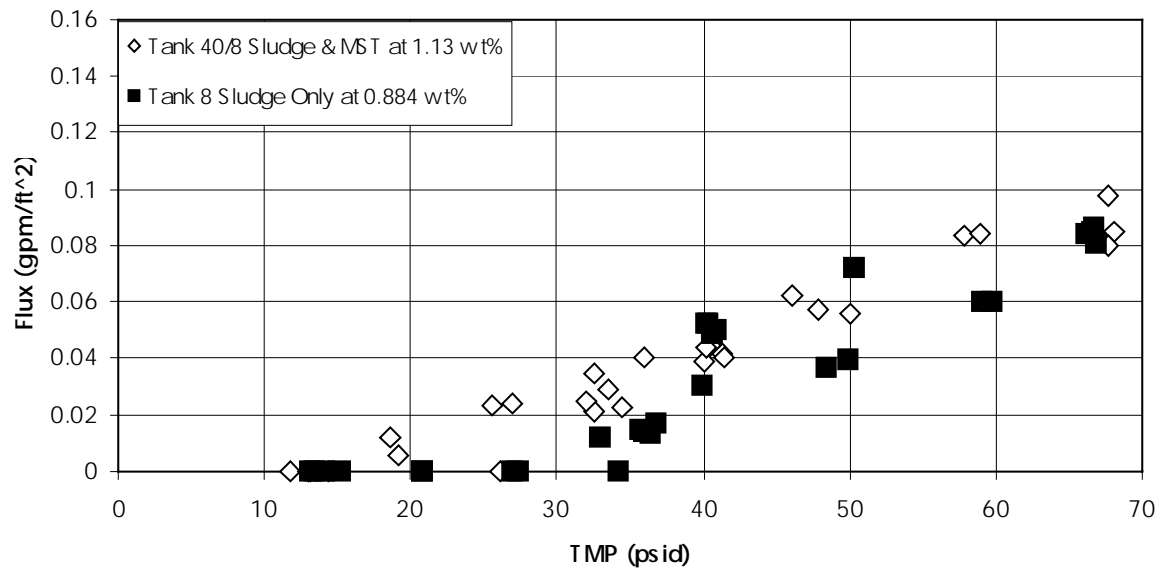
The comparison between Test #1 with Tank 8F sludge and MST and Test #2 with Tank 8F sludge and MST shown in Figure 15 supports the statement that a clean filter will have higher filtrate flux than a dirty filter.



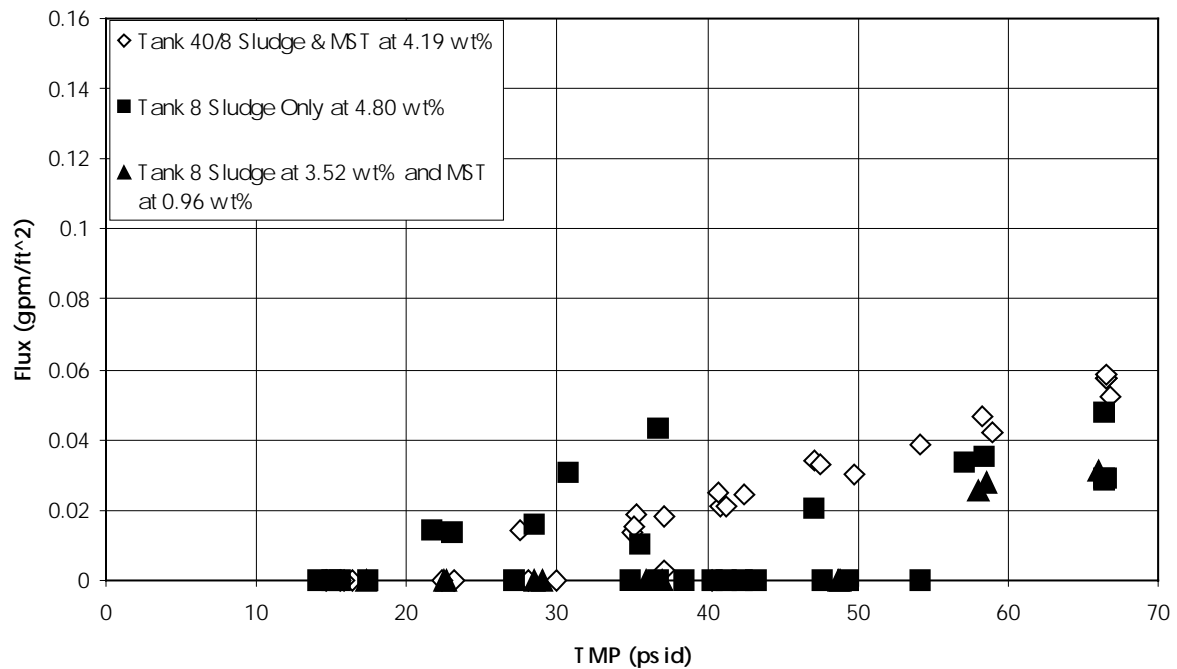
**Figure 11. Comparison of Filter Performance between Feed Containing Tank 40H/8F Sludge and MST and Feed Containing Tank 8F Sludge Only**



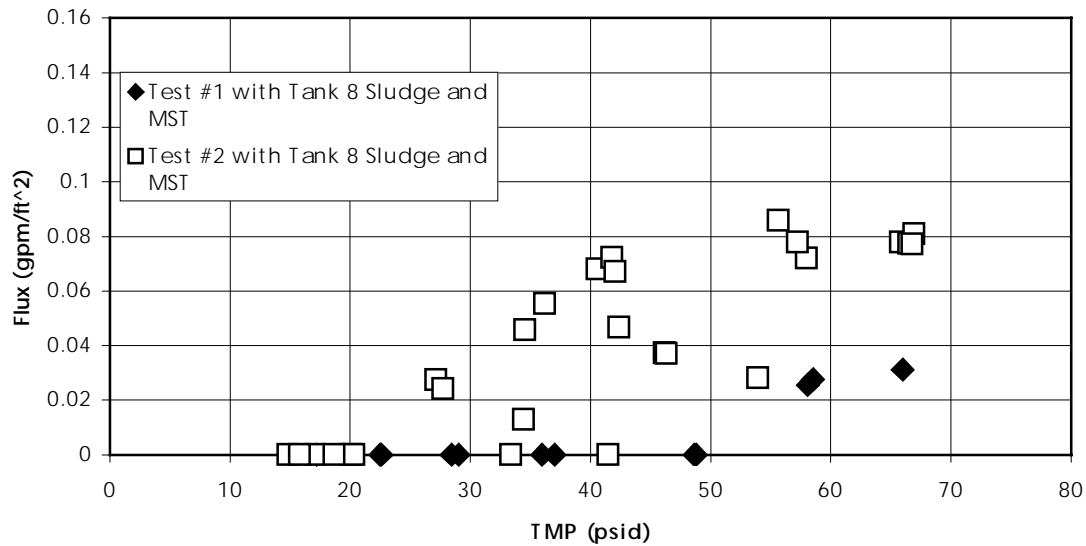
**Figure 12. Comparison of Filter Performance between Feed Containing Tank 40H/8F Sludge and MST and Feed Containing Tank 8F Sludge Only**



**Figure 13. Comparison of Filter Performance between Feed Containing Tank 40H/8F Sludge and MST and Feed Containing Tank 8F Sludge Only**



**Figure 14. Comparison of Filter Performance between Feed Containing Tank 40H/8F Sludge and MST and Feed Containing Tank 8F Sludge Only**



**Figure 15. Comparison of Filter Performance between Test #1 with Tank 8F Sludge and MST and Test #2 with Tank 8F Sludge and MST**

### Recovery of Filter Flux

Operation required remedial steps (scouring and backpulsing) at several points in the tests. The following tables summarize the remedial actions taken with those that exceed normal actions *italicized*. In many instances the target flux could not be reached. This result contrasts with the previous testing with blended Tank 40H/8F Sludge and MST, where flux was not recovered in only one case. This data support the hypothesis that sludge only feeds filter prove more difficult to filter than sludge and MST feeds.

**Table 2 Summary of Remediation Steps for Test with Tank 8F Sludge Only**

Nominal Loading (wt%)	Phase	Low Axial Velocity	High Axial Velocity	Maximum Axial Velocity
0.06	I	None Required  <b>Target Reached</b>	None Required  <b>Target Reached</b>	None Required  <b>Target Reached</b>
	II	Scour @ Low Velocity Scour @ High Velocity Scour @ Max. Velocity  <b>Target Reached</b>	Scour @ High Velocity Scour @ Max. Velocity 1 sec. Backpulse 3 sec. Backpulse <i>3 sec. Backpulse</i> <i>3 sec. Backpulse</i> <i>Scour @ Max Velocity</i> <i>Scour @ 95% Pump Speed</i>  <b>Target NOT Reached</b>	Scour @ Max. Velocity Scour @ 95% Pump Speed 1 sec. Backpulse 3 sec. Backpulse  <b>Target NOT Reached</b>
0.28	I	None Required  <b>Target Reached</b>	None Required  <b>Target Reached</b>	None Required  <b>Target Reached</b>
	II	Scour @ Low Velocity Scour @ High Velocity Scour @ Max. Velocity 1 sec. Backpulse  <b>Target Reached</b>	None Required  <b>Target Reached</b>	Scour @ Max. Velocity Scour @ 95% Pump Speed 1 sec. Backpulse 3 sec. Backpulse  <b>Target NOT Reached</b>
1.29	I	None Required  <b>Target Reached</b>	None Required  <b>Target Reached</b>	Scour @ Max. Velocity  <b>Target Reached</b>
	II	Scour @ Low Velocity  <b>Target Reached</b>	None Required  <b>Target Reached</b>	None Required  <b>Target Reached</b>
6.00	I	Scour @ Low Velocity Scour @ High Velocity Scour @ Max. Velocity 1 sec. Backpulse 3 sec. Backpulse  <b>Target Reached</b>	Scour @ High Velocity Scour @ Max. Velocity 1 sec. Backpulse 3 sec. Backpulse  <b>Target NOT Reached</b>	Scour @ Max. Velocity Scour @ 95% Pump Speed 1 sec. Backpulse 3 sec. Backpulse  <b>Target NOT Reached</b>
	II	Scour @ Low Velocity  <b>Target Reached</b>	Scour @ High Velocity Scour @ Max. Velocity 1 sec. Backpulse 3 sec. Backpulse <i>3 sec. Backpulse (no filtrate flow after backpulse)</i> <i>Scour @ 95% Pump speed</i> <i>Scour @ 95% Pump speed</i> <i>3 sec. Backpulse (no filtrate flow after backpulse)</i> <i>Scour @ 95% Pump speed</i> <i>Scour @ 95% Pump speed</i> <i>3 sec. Backpulse (no filtrate flow after backpulse)</i> <b>Target NOT Reached</b>	Scour @ Max. Velocity Scour @ 95% Pump speed 1 sec. Backpulse 3 sec. Backpulse <i>3 sec. Backpulse (no filtrate flow after backpulse)</i> <i>Scour @ 95% Pump speed</i> <i>Scour @ 95% Pump speed</i> <i>3 sec. Backpulse (no filtrate flow after backpulse)</i> <i>Scour @ 95% Pump speed</i> <i>Scour @ 95% Pump speed</i> <i>3 sec. Backpulse</i> <b>Target NOT Reached</b>



**Table 3 Summary of Remediation Steps for Test #1 with Tank 8 Sludge and MST**

Nominal Loading (wt%)	Phase	Low Axial Velocity	High Axial Velocity	Maximum Axial Velocity
3.52 wt% Sludge and 0.96 wt% MST	I	None Required  <b>Target Reached</b>	None Required  <b>Target Reached</b>	None Required  <b>Target Reached</b>
	II	Scour @ Low Velocity Scour @ High Velocity Scour @ Max. Velocity  <b>Target Reached</b>	<u><b>Test Halted</b></u>	<u><b>Test Halted</b></u>

**Table 4 Summary of Remediation Steps for Test #2 with Tank 8 Sludge and MST**

Nominal Loading (wt%)	Phase	Low Axial Velocity	High Axial Velocity	Maximum Axial Velocity
3.52 wt% Sludge and 0.96 wt% MST	I	None Required  <b>Target Reached</b>	None Required  <b>Target Reached</b>	Scour @ Max. Velocity Scour @ 95% Pump speed 1 sec. Backpulse 3 sec. Backpulse <i>Scour @ 95% pump Speed (2 h)</i>  <u><b>Target NOT Reached</b></u>
	II	Scour @ Low Velocity Scour @ High Velocity Scour @ Max. Velocity 1 sec. Backpulse  <b>Target Reached</b>	None Required  <b>Target Reached</b>	None Required  <b>Target Reached</b>

## CONCLUSIONS

The conclusions from these tests follow.

- Feed solutions containing only simulated sludge filtered more slowly than feed solutions containing sludge and MST. We expected this result since the MST has a larger mean particle size and a narrower particle size distribution than the simulated sludge.
- Filter flux depends strongly on transmembrane pressure. Increases in flux correlate with increases in transmembrane pressure, consistent with crossflow filtration theory.
- Filter flux decreases with increasing insoluble solids concentration, as expected and in agreement with crossflow filtration theory.
- Prior testing without intervening filter cleaning can affect test results.

**APPROVALS:****Authors:**

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S. D. Fink, TFA System Lead  
WPT L4 Manager & Co-author

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W. L. Tamosaitis, WPT Manager

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Date**REFERENCES**

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<sup>1</sup> Ralph Haggard et al., "Final Report on the Crossflow Filter Testing for the Salt Disposition Alternative", USC-FRED-PSP-RPT-09-0-010, Rev. 0, December 4, 1998.

<sup>2</sup> V. Van Brunt, Carol Stork, and Travis Deal, "Final Report on the Crossflow Filter Optimization with 5.6 M Sodium Salt Solution," USC-FRED-PSP-RPT-09-0-015, December 20, 2000.

<sup>3</sup> M. R. Poirier, Inter-Office Memorandum, SRT-WHM-2000-010, November 9, 2000.

<sup>4</sup> Westinghouse Savannah River Company, "Science & Applied Technology Integration Work Scope of Work Matrix for Alpha Removal (Demonstration Phase)," HLW-SDT-2000-00047, November 9, 2000.

<sup>5</sup> Clift, R., J. R. Grace, and M. E. Weber, Bubbles, Drops and Particles, Academic Press, NY (1978)

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<sup>7</sup> M. C. Porter, Handbook of Industrial Membrane Technology, Park Ridge: Noyes, 1990.