Media Comparison of Filtration with Hanford Tank AP-107 Supernate

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ABSTRACT

The present experimental work was performed to investigate media performance in both cross-flow and dead-end filtration of 9 L of Hanford tank AP-107 supernatant. Media grades 0.1 and 5 were tested for each the cross-flow filter (CFF) and dead-end filter (DEF). The CFFs were tested in a cells unit filter (CUF) system in recycle mode; the DEFs (in-line and disc) were used to dewater the feed. Visual inspection of the feed material indicated essentially no settled insoluble solids in the feed material. The grade 5 CFF produced stable filter fluxes while the flux for the grade 0.1 CFF steadily decayed. The grade 5 BDEF (backpulsed dead-end filter) was effective at removing solids from the AP-107 feed and when backpulsed, fully restored the filter flux, indicating that the solids did not irreversibly foul the filter. The grade 0.1 DEF was used to filter the final 2 L of AP-107 feed drained from the CUF and to collect solids on the filter media for post-test characterization. Additional DEF filtering was performed on 4 L of BDEF permeate in order to assess whether small particles that cause fouling in the grade 0.1 filter passed through the grade 5 filter. There was no indication of fouling and limited solids were observed on the filter. A direct comparison of filtration rates between the 0.1 and 5 media grade filters for both the CFF and DEF showed the grade 5 filter flux was, on average, 200 times higher than the 0.1 media grade filter while still effectively filtering the solids.

Keywords: filtration; dead-end filtration; cross-flow filtration; tank waste; Hanford tank waste pretreatment

INTRODUCTION

Over 55 million gallons of Hanford tank waste is to be vitrified in a Low Activity Waste (LAW) melter for waste stabilization. To convert the radioactive waste to glass, the U.S. Department of Energy (DOE) is working to construct the world’s largest vitrification facility, the Waste Treatment and Immobilization Plant (WTP). The Low-Activity Waste Pretreatment System (LAWPS) provides for the initial production of immobilized low-activity waste by feeding Hanford tank supernate from tank farms to

WTP's LAW facility for immobilization.

Washington River Protection Solutions (WRPS) requested that Hanford tank waste collected from tank 241-AP-107 (hereafter called AP-107) be processed using conceived pre-treatment steps (suspended solids removal by filtration, and Cs removal by ion exchange) and then vitrified. WTP will use filtration to assist in waste remediation and vitrification efforts by removing the high dose contributor solids. Bench-scale filtration testing of 9.5 liters of AP-107 supernatant was conducted using two different crossflow filters (CFFs) and two different dead-end filters (DEF).. Media grades 0.1 and 5 were tested in crossflow and dead-end configurations to provide feed to the ion exchanger (IX) and vitrification and demonstrate media comparison in both the CFF and DEF configurations [1]. Previous LAWPS filtration studies have been limited to media grade 0.1 filter analysis, however, a shift in operating basis has made LAWPS facilities less restrictive on allowable particle size. Little performance data exists for media grade 5 filter and was unclear if grade 5 was a technically sound choice with respect to filter performance, both in terms of rate of filtration and with respect to solids retention. Comparing the media grades 0.1 and 5 helps define the technical sufficiency of the grade 5 filter.

EXPERIMENTAL

Test Apparatus

Cells Unit Filter (CUF)

Figure 1shows a schematic of the CUF. The CUF has flexibility for changing out the filtration media. For these tests, media grade 0.1 and 5 Mott sintered stainless steel filters each 6-in long with ½-in internal diameter, 5/8-in outside diameter were installed in parallel. These filter elements are isolated from each other and can be operated independently. The double filter assembly is shown in Figure *2*.

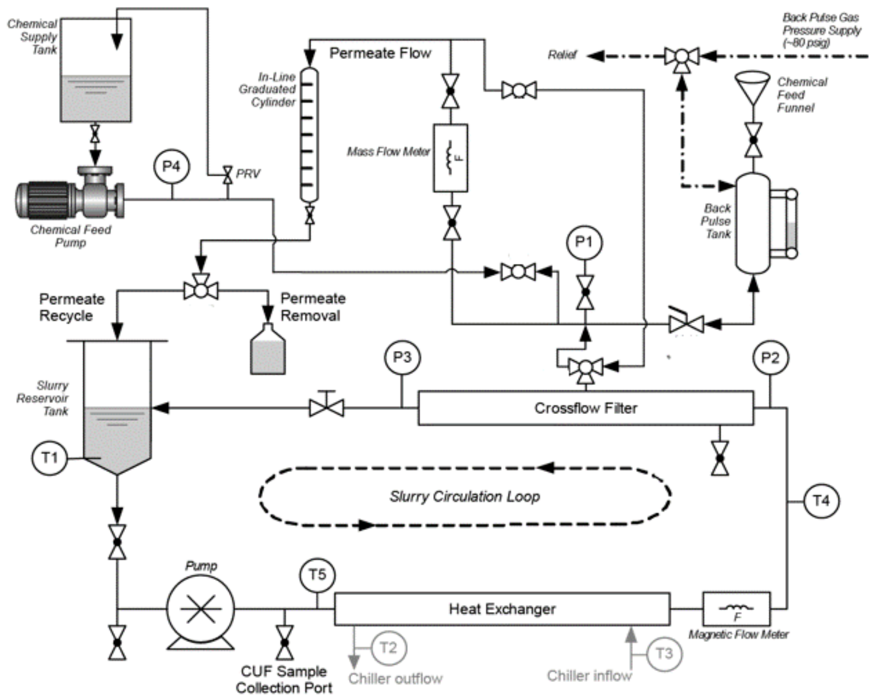

Figure 1. CUF Schematic

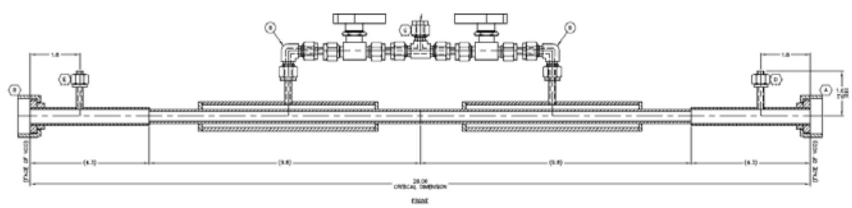

Figure 2. Double In-Line CFF Assembly, Grade 0.1 Upstream of the Grade 5 CFF

To test materials in the CUF, slurry feed is introduced to the system through the slurry reservoir. A rotary lobe pump (powered by an air motor) pumps the slurry from the slurry reservoir through a magnetic flow meter, a shell and tube heat exchanger, and the double 6-in. CFF assembly. The transmembrane pressure (TMP) and axial velocity are controlled by the air motor supply pressure. Permeate passed through the filter can be sent to the back pulse chamber, reconstituted with slurry in the slurry reservoir, or removed from the system via dewatering. The permeate flow rate is measured by means of a graduated glass-flow monitor and/or a Coriolis mass flow meter. Filter back pulsing is conducted by partially filling the back pulse chamber with permeate, pressurizing the back pulse chamber with air, and forcing permeate in the chamber back through the filter. During testing, the slurry temperature was maintained at 25 ± 5 °C by a 1000 W chiller that circulates chilled water through the heat exchanger.

Backpulse Dead-End Filter (BDEF)

The test filter for the backpulse dead-end filter (BDEF) was the Mott 6480 modified[1] to 2.75-in. filter active length [2]. The filter is cylindrical with dimensions of 3/8-in. diameter x 2.75-in. length and has a filtration area of 3.24 in^2. Figure 3 shows a sketch of the Series 6480 filter.

The BDEF test apparatus relied on the CUF to hold and agitate the feed, deliver it at the targeted pressure, and measure the BDEF permeate flux. Figure 4 shows a schematic of the BDEF assembly. The CUF provides pressurized feed to the BDEF through a connection to the CUF sample collection port. The feed is filtered as it flows through the BDEF. Filtered feed is then introduced into the CUF permeate system to measure the flowrate through the glass flow meter and/or the Coriolis flowmeter. When the CUF is operating in "BDEF mode," the CUF rotary lobe pump is operating in recirculation to provide pressurized feed to the BDEF, but the CUF CFF permeate valve is closed to prevent filtration. Thus, only filtrate from the BDEF is flowing through the CUF permeate metering and collection system. The BDEF may be backpulsed by closing the feed valve (Valve A), opening the drain valve (Valve B), and then using the CUF backpulse chamber and pressured air to backpulse the BDEF.

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[1] The filter was cut approximately in half and a new non-porous end cap was welded on. The weld was inspected and approved prior to use.

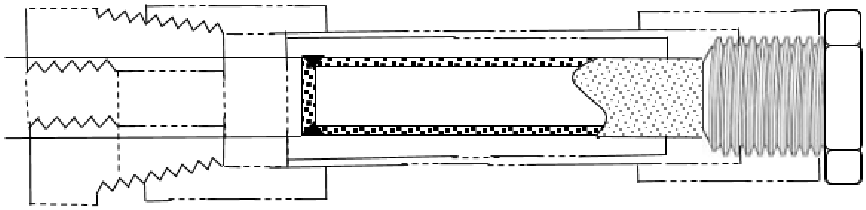

Figure 3. Mott 6480 Line Filter

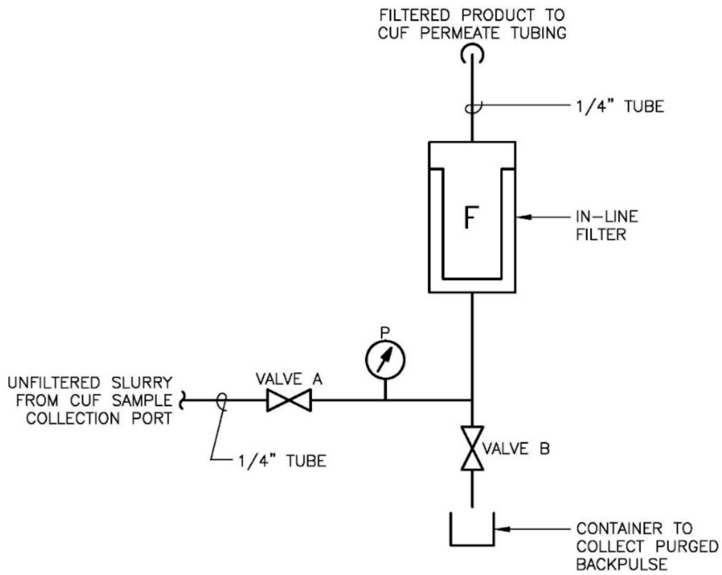

Figure 4. BDEF Schematic

Dead-End Filter (DEF)

The filter used for dead-end filtration used was a Mott 70-mm disc test filter with a filter area of approximately 4.4 in^2. The Mott filter media is stainless steel sintered metal with media grade 0.1 filter.

A schematic of the DEF test apparatus is shown in Figure 5. The dilute slurry feed is introduced to the system through the feed reservoir. Compressed air supplied to the top of the reservoir, at a controlled pressure, pushes the feed through the test filter. The TMP and filtrate mass are measured as a function of time. This filter system has the advantage of having no minimum volume necessary to operate. Therefore, the DEF was used to filter the final ~1.5 liters of AP-107 feed drained from the CUF, as the CUF requires >1.5 liters of feed to operate the pump.

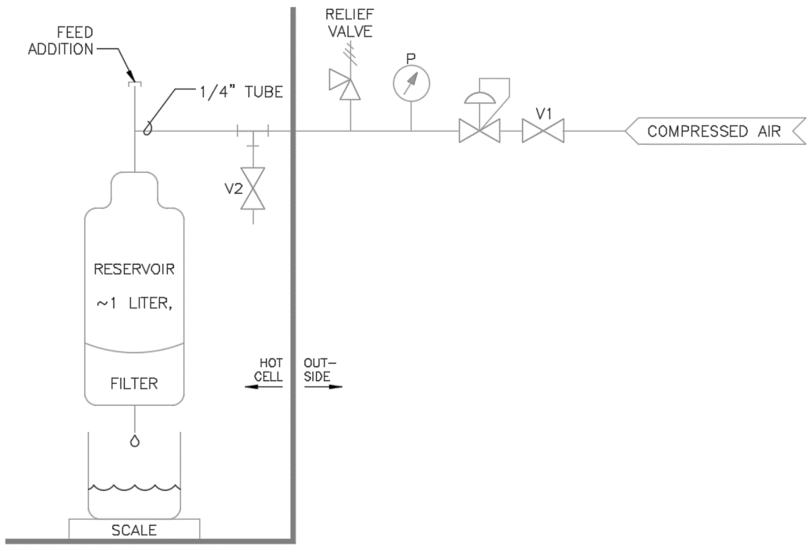

Figure 5. Schematic of Dead-End Filtration Test Setup

Test Conditions

Tank AP-107 contains low-level radioactive waste primarily generated from past plutonium production at the Hanford Site. Thirty-six samples (~250 mL each) were received from the upper supernate layer of Tank AP-107. A sample of the first and last bottles were obtained to validate the target feed composition. The major components of the waste composed of Na (91%), Al (6%) and K (1.6%). Table 1 provides component concentrations of the original AP-107 feed. Note: from looking at the samples through the hot cell window, there were no visible solids in the feed samples.

Table 1. Composition of AP-107 Feed

Analyte	Average (μg/L)	Molarity
Al	9825	0.364
K	3793	0.097
Na	129250	5.622
B	36	0.003
Ca	31	0.001
Cr	493	0.009
P	650	0.021
S	1698	0.053
Si	40	0.001

The CUF was operated in two primary modes – recycle mode where permeate was returned to the feed vessel and dewatering mode where permeate was not recycled. The media grade 0.1 and 5 CFFs were only tested in recycle mode; all dewatering of the slurry was performed with the dead end filters (media grade 5 BDEF or the media grade 0.1 DEF). A clean water flux (CWF) was measured at the start of testing to benchmark filter performance and verify the system had no leaks. AP-107 slurry was tested using a matrix consisting of various transmembrane pressures (TMPs) ranging from 1.7 psig to 20 psig at a constant nominal axial velocity (AV) of 4.48 m/s. The first matrix passed 3.2 L AP-107 through the media grade 5 CFF and held it in recycle mode for 28 hours without back pulsing.

Once the testing with the first matrix was completed, the feed was dewatered through the grade 5 BDEF and 971 g of permeate was collected. Additional AP-107 was added to the slurry reservoir; the slurry was dewatered through the BDEF; and 4608 g of permeate were collected in four bottles.

An additional 2 L of AP-107 was added to the feed vessel. The 0.1 micron filter was then tested in recycle mode at TMPs of 10, 15, 20 and 10 psig for 7 hours each. The system was backpulsed once between each condition. After completion of the matrix, the system was dewatered through the grade 5 BDEF and 2920 g of permeate was collected. Additional AP-107 was added to the slurry reservoir and dewatered through the BDEF; 1314 g of permeate was collected.

After both matrices were completed, the BDEF was backpulsed and the solids, concentrated in the back flush, were analyzed for particle size. The remaining 2373 g of AP-107 slurry in the reservoir was drained from the CUF to be filtered through the grade 0.1 DEF.

The DEF testing was performed in two steps. The first step began by obtaining a CWF using 0.1 M NaOH to provide a baseline measurement of the filter resistance at 10 psig. The DEF slurry reservoir was then loaded with 1198 g of AP-107 drained from the CUF and was passed through the media grade 0.1 DEF at 10 psig. The second matrix passed the remaining 1175 g of AP-107 as-received feed through the DEF at 20 psig. In addition to the unfiltered AP-107 slurry, six bottles of the BDEF dewatered permeate were run through the DEF in order to collect additional solids on the media grade 0.1 filter that may have passed through the grade 5 BDEF filter.

FILTRATION RESULTS

Cross Flow Filtering

A graph of the pressure normalized permeate flux for the media grade 0.1 and 5 CFF's is shown in Figure 6. The media grade 5 CFF ran for 28 hours in recycle mode The flux rate during the grade 5 testing did not decline during the duration of the test which indicated limited filter fouling occurred using this filter material. At the conclusion of CUF testing, the AP-107 slurry drained from the CUF was filtered through a dead-end filter and solids were collected. Solids collected off the dead-end filter weighed 0.5670 g. Using this mass, we can conclude the AP-107 feed had approximately 154 ppm solids (0.5670 g solids/3.68 kg AP-107 feed). Due to the relatively low solids concentration, there may have been insufficient solids in the 3.2 liters of feed to develop significant fouling of the filter. The permeability of the grade 5 filter is approximately 50 times higher than the grade 0.1 filter, so fouling may not manifest without significantly more feed.

The media grade 0.1 test matrix evaluated filter performance at four pressure set-points: 10, 15, 20, and 10 (repeat) psi. At each pressure set-point, the CUF was run in recycle mode for 7 hours. Figure 6 shows the pressure normalized permeate flux under the four conditions on a continuous time axis. Each of the data sets started with a back pulse and ran for approximately 7 hours. Back pulses were conducted at approximately 80 psi. Although immediately effective, the benefits from back pulsing in terms of a sustainable increased flux are short in duration. The media grade 0.1 CFF exhibited a flux decline consistent with previous testing and the flux after 28 hours of recycle testing was approximately 0.002 gpm ft^{-2} psi^{-1}.

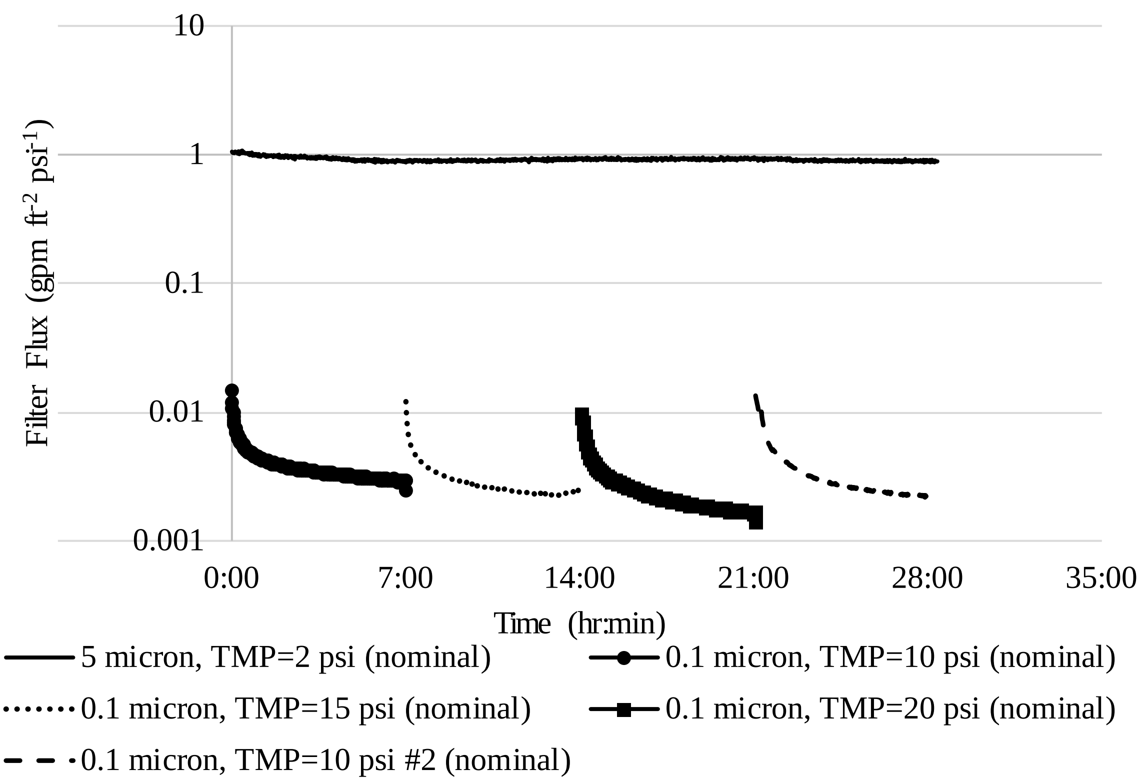

Figure 6. Pressure Normalized Permeate Flux for an AP-107 Supernatant Filtered Through a Mott Grade 0.1 and 5 CFFs

The media grade 5 filter flux was, on average, 200 times higher than the media grade 0.1 filter while still effectively filtering the solids. The high flux rates and lack of fouling with the 5 media grade filter caused speculation that small particles that fouled the 0.1 media grade filter were passing through the 5 media grade filter. In order to assess this theory, four effluent bottles that were cross flow filtered with the 5 media grade filter and dewatered through the 5 media grade BDEF were additionally passed through the 0.1 media grade DEF. No recoverable solids were found on the filter. This indicates that solids may be getting trapped in the 5 media grade CFF, but not enough to noticeably impact the flux as it does with the 0.1 media grade CFF.

Backpulse Dead End Filtration

The grade 5 BDEF was only run in dewatering mode. Note that BDEF dewatering events did not occur continuously in the operations described above. The disparate BDEF dewatering events were knitted together on a continuous time axis and shown on Figure 7.The TMP floated during testing due to throttling the permeate flow to prevent the Coriolis flowmeter from being over-ranged past 30 lph. The discontinuities in the data on Figure 7 are when filtration was stopped to add more AP-107 feed to the CUF and changing permeate containers (the permeate was collected in 1.5 L bottles that had to be changed when full).

The following dewatering events shown in Figure 7 are outlined below:

(1) <u>Dewatering Event 1:</u> Composited AP-107 feed in CUF and ran CUF in recycle (TMP: 1.7 psig; AV: 14.7 ft/s) for 28 hours with no backpulsing. Aligned BDEF (Filter 3) in dewater mode and dewatered AP-107 to produce ion exchange (IX) feed.

(2) <u>Dewatering Event 2:</u> Composited AP-107 feed in CUF run in dewater mode with BDEF to produce IX feed. Composited additional AP-107 feed and continued to dewater.

(3) Dewatering Event 3: Composited AP-107 feed and performed with 0.1 media grade CUF. Ran CUF in recycle (TMP: 10 psig; AV: 14.7 ft/s) for 7 hours. Backpulsed, then continued running in recycle (TMP: 15 psig; AV: 14.7 ft/s) for 7 hours. Backpulsed, then continued running in recycle (TMP: 20 psig; AV: 14.7 ft/s) for 7 hours. Backpulsed, then continued running in recycle (TMP: 10 psig; AV: 14.7 ft/s) for 7 hours. Aligned BDEF in dewater mode and dewatered AP-107 to produce IX feed.

(4) Dewatering Event 4: Composited AP-107 feed in CUF. Backpulsed BDEF (saved sample for particle size distribution analysis) and then ran in dewater mode with BDEF to produce IX feed.

The flux remained constant during the first dewatering event occurring immediately after the media grade 5 CFF, indicating no solids built up on the filter. This supports the idea that the 28-hour recycle test with the media grade 5 filter did indeed remove solids from the system. When fresh feed is added to the CUF/BDEF system and dewatered without CFF recycle (dewatering event 2), the BDEF filtration flux declines with time.

An anomaly occurs during dewatering events 2 and 3 after the 28-hour recycle test with the media grade 0.1 filter: the flux starts off higher than expected. It is suspected that during the testing of the CFF, the BDEF cake may have settled off the surface of the filter on termination of BDEF flow, resulting in a higher than expected normalized flux relative to filtration data from dewatering event 3. The decrease in filter flux with time could be attributed to the pre-existing filter cake from the previous dewatering events.

Additional fresh AP-107 feed was added to the CUF/BDEF system and the BDEF was backpulsed after the 3rd dewatering event (time 0:23 on Figure 7). The flux was fully restored indicating that the backflush was effective at removing the filter cake. The backpulse was conducted at 30 psi and 140 mL of flush fluid containing the concentrated solids from the backflush were collected and removed from the CUF/BDEF system. The final dewatering event was performed on the fresh feed and, consistent with results from event 2, showed a flux decline with time as solids built up a filter cake onto the surface of the filter.

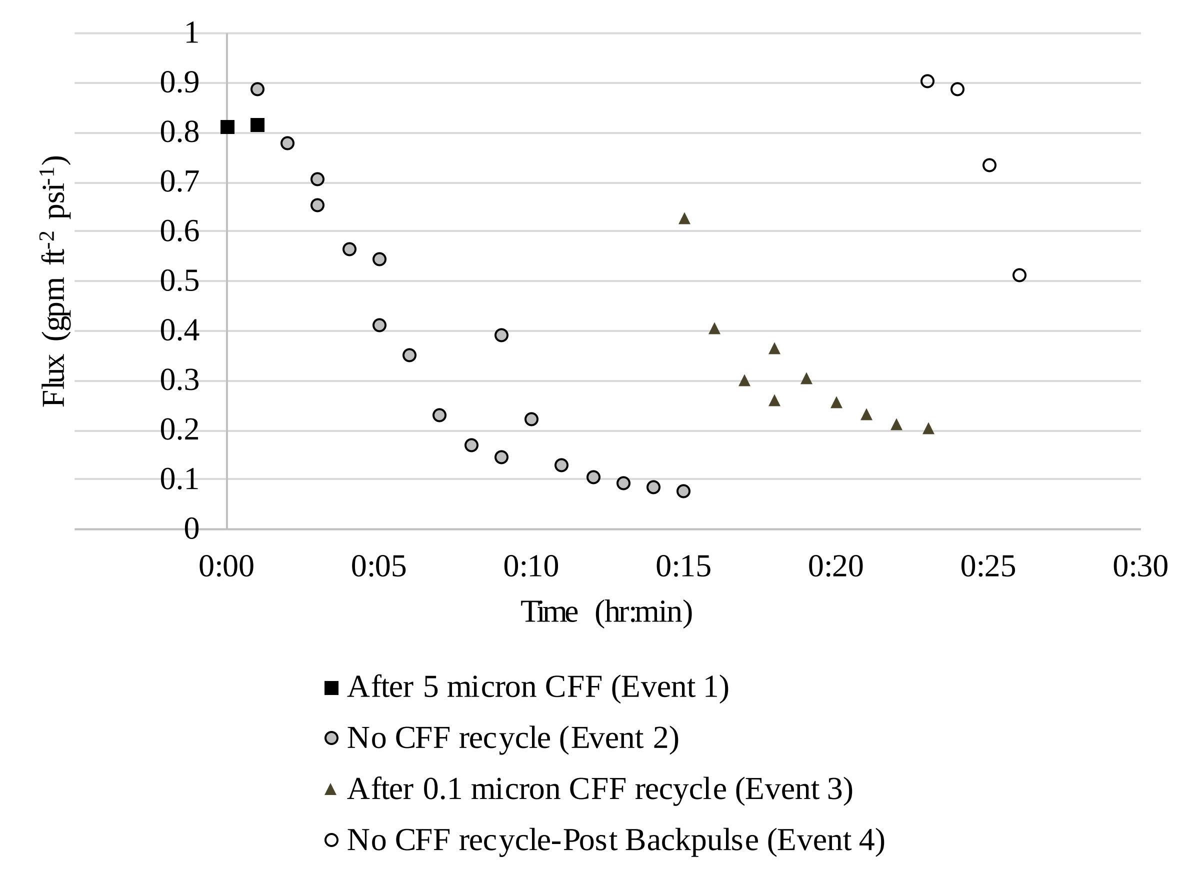

Figure 7. Disparate BDEF Dewatering Event Flux for AP-107 Supernatant Filtered Through a Mott Grade 5 BDEF

Dead End Filtration

AP-107 drained from the CUF was filtered through the DEF and solids were collected. DEF testing filtered two ~1L batches at two different transmembrane pressures, the first at 10 psi and the second at 20 psi. Flux data for this testing are displayed in Figure 8. It is readily apparent that the filter performance declines as additional material is filtered. This is likely due to the buildup of material on the surface of the filter. At the conclusion of the first batch (DEF Dewater 1), it is assumed the exterior of the filter was fully coated with particles on the surface. The second batch (DEF Dewater 2) added additional material to the filter cake forming on the surface of the dead end filter. The DEF was disassembled and the solids were scraped off the filter for further analysis.

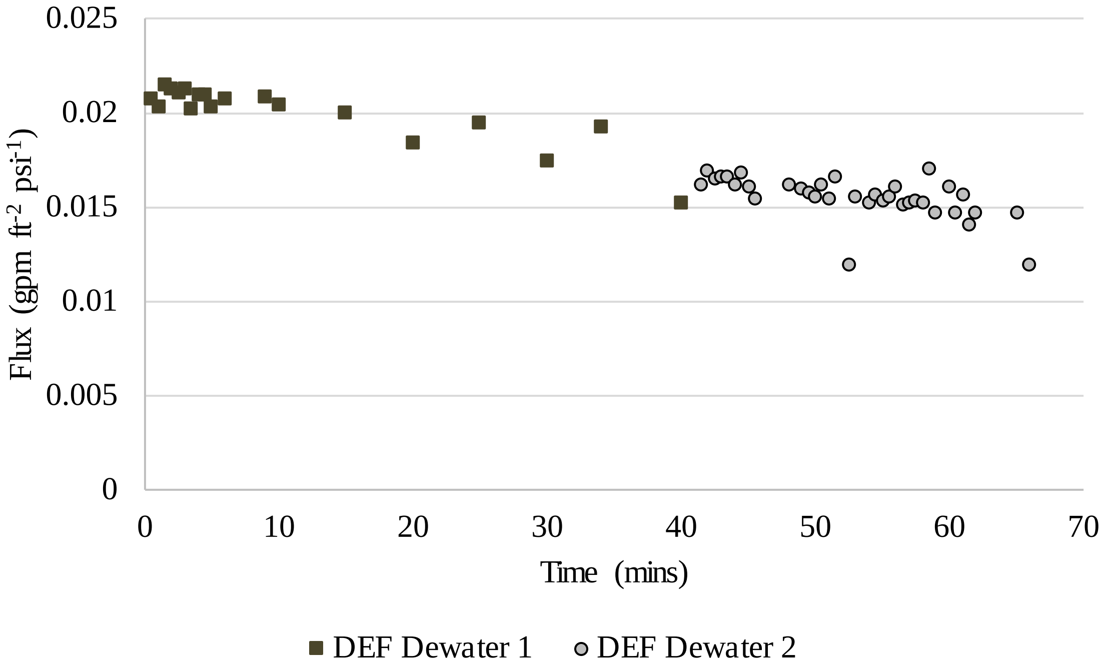

Figure 8. Flux for an AP-107 Supernatant Filtered Through a Mott Grade 0.1 DEF

In addition to the AP-107 slurry, four bottles of CUF dewatered permeate were run through the DEF in order to collect any additional solids that may have passed through the 5 media grade BDEF. Very minimal solids were found on the filter, indicating the media grade 5 filter was effective at removing solids from the permeate. A graph of the permeate flux for each bottle is shown on a continuous time axis on Figure 9. The original DEF filtration data is plotted as a flux comparison to this additional dewater data. Perplexingly, the flux shows a similar decline as to what was found with the unfiltered AP-107, however, comparatively no solids were found on the filter after the additional bottles were dewatered. One explanation is that it could be an effect of colloidal suspensions leading to filter fouling without leaving a visible filter cake behind.

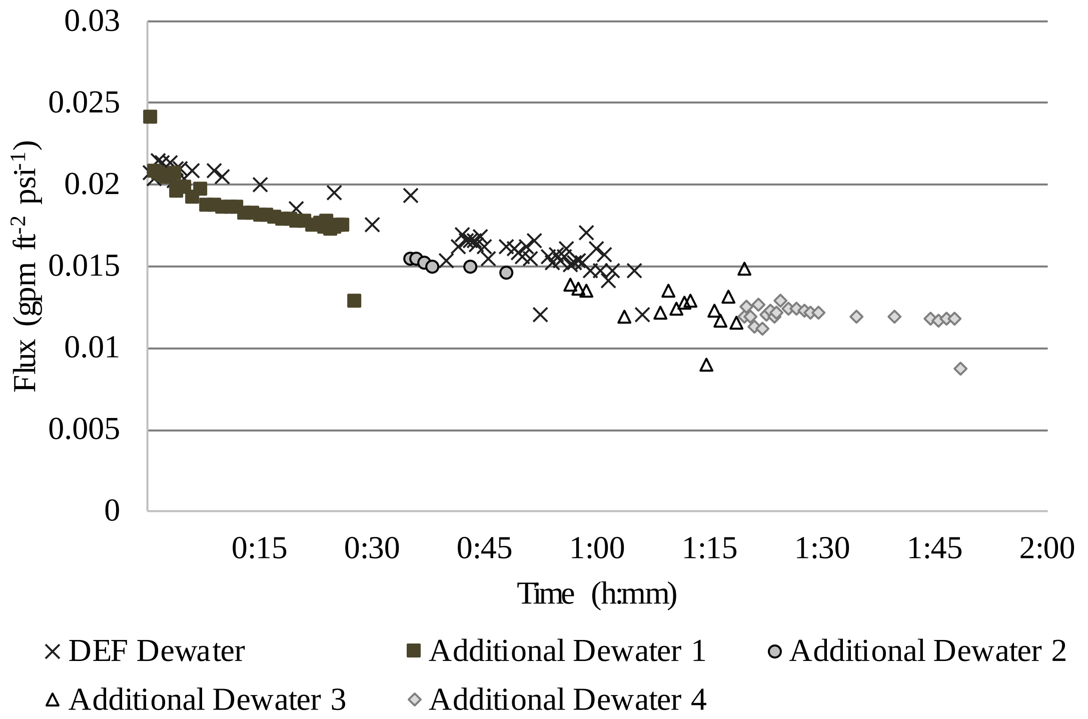

Figure 9. Permeate Flux for DEF Dewatering

Particle size distribution (PSD) was conducted on the solids backpulsed from the media grade 5 BDEF. Three sub-aliquots were analyzed for particle size distribution from the parent AP-107 slurry sample provided for analysis (i.e., AP-107 feed with backpulsed filter solids). Each sub-aliquot was measured under four conditions: 1) Pre-sonication, 2) 50% Ultrasonic power, 3) 100 % Ultrasonic power, and 4) Post-sonication. The results of the three sub-aliquots were then averaged to obtain a composite PSD for each condition evaluated. The composite PSDs of the collected AP-107 filter solids are shown in Figure 10 with select percentiles given in Table 2. As is often the case upon sonication, weak to moderate agglomerates are broken, leading to an increase in the volume contribution of smaller particles. It should be noted that at 100 % ultrasonic power, a population of 300-700 μm "particles" appear. This population disappears once sonication is stopped, i.e., not observed in the post-sonication measurement.

This population could either be weak agglomerates or an artefact of sonication. Filtration of the material appears to make agglomerates that did not exist prior to filtration.

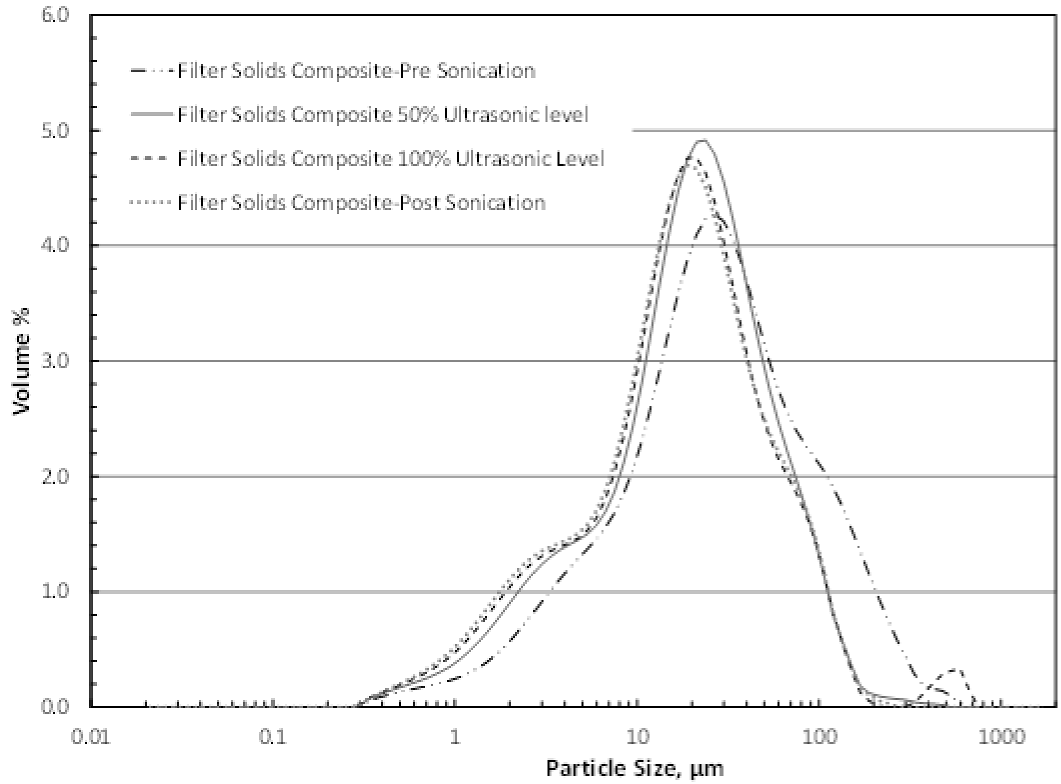

Figure 10. Composite PSDs of AP-107 collected filter solids measured in cesium-exchanged AP-107 supernatant.

Table 2. Selected Particle Size Percentiles for AP-107 Filter Solids

Percentile	d(0.05)	d(0.10)	d(0.25)	d(0.50)	d(0.75)	d(0.90)	d(0.95)
Filter Solids Composite-Pre-Sonication	2.50	4.44	12.0	26.4	57.6	122	175
Filter Solids Composite-50% Ultrasoinc Level	1.81	3.14	9.21	20.0	37.4	66.6	90.0
Filter Solids Composite-100% Ultrasoinc Level	1.59	2.73	8.25	18.3	35.3	68.9	96.2
Filter Solids Composite-Post-Sonication	1.51	2.57	7.76	17.5	34.1	65.0	88.0

Although a small percentage of the solid population, if the smaller solids (<0.1µm) are forming a gel within the permeate, this would decrease filter flux through the DEF even after passing through the BDEF. A gel layer composed of colloidal particles (300 to 400 nm) that have passed through the grade 5 filter may not be visible to the eye but could depth foul the 0.1 grade filter. This would be consistent with the flux decline observed during 0.1 grade DEF filtration (see Figure 9) described previously and attributed to colloidal fines.

CUF/BDEF 5 Media Grade Filtration

The 5 media grade filter showed a relatively consistent initial permeate flux ranging from 0.8-0.9 gpm ft^{-2} psi^{-1} for the BDEF to 1.05 gpm ft^{-2} psi^{-1} for the CFF. The first 30 minutes of the CFF recycle with the 5 media grade filter is plotted against the BDEF dewatering events in Figure 11. The CFF maintains a nominally constant normalized permeate flux over the duration of the test. Feed previously passed through the 5 media grade CFF showed no flux decline when filtered through the BDEF. However, as fresh unfiltered feed is introduced to the system, the BDEF begins to foul where the CFF did not. It is important to note that although the flux is normalized for pressure, the cross-flow testing was conducted at a third of the TMP conducted during this segment of the dead end filtration. As mentioned earlier, the flux decline in the BDEF data after the 0.1 media recycle could be attributed to the pre-existing filter cake from the previous dewater. The post backpulse fresh feed dewater flux declines at a rate much faster than the previous fresh feed flux decline.

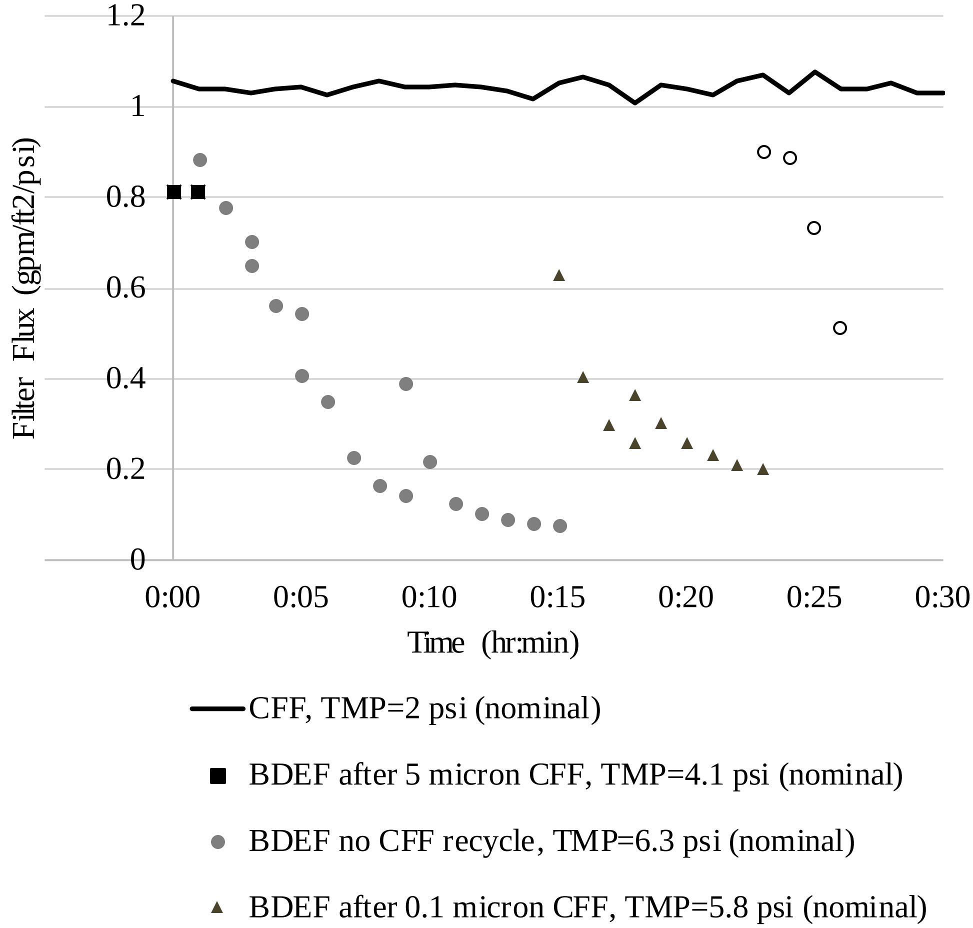

Figure 11. Media Grade 5 CFF Filtration and 5 Media Grade BDEF Data with Pressure Normalized Filter Flux

CUF/BDEF 0.1 Media Grade Filtration

The first hour of the 0.1 media grade CFF filtration data was plotted against the DEF data and shown in Figure 12. Nominal flux values for both DEF evolutions appear higher than the CFF initial flux values by nominally 30%. Although this trend is in contradiction of the results for the 5 media grade filters, the 0.1 micron filter DEF could be using a cake filtration mechanism. The larger particles form a cake layer on the filter medium and after the cake is formed, the cake becomes the primary filtration medium. The finer particles can be trapped between the interstitial space of the larger particles and thus trapping more of the small particles earlier before they pore foul the filter [3].

The flux values during the 10 psi TMP are consistently 20-30% higher than the 20 psi TMP condition for both the DEF and CFF data. These results indicate that the tendency for the membrane to foul is increased at a higher pressure and are consistent with behaviors observed in previous testing of PEP simulant filtration [4] and LAWPS simulant filtration [5] The DEF flux declined 26% over the duration of the test for both TMP conditions where the CFF declined nominally double that during the first 25 minutes of testing.

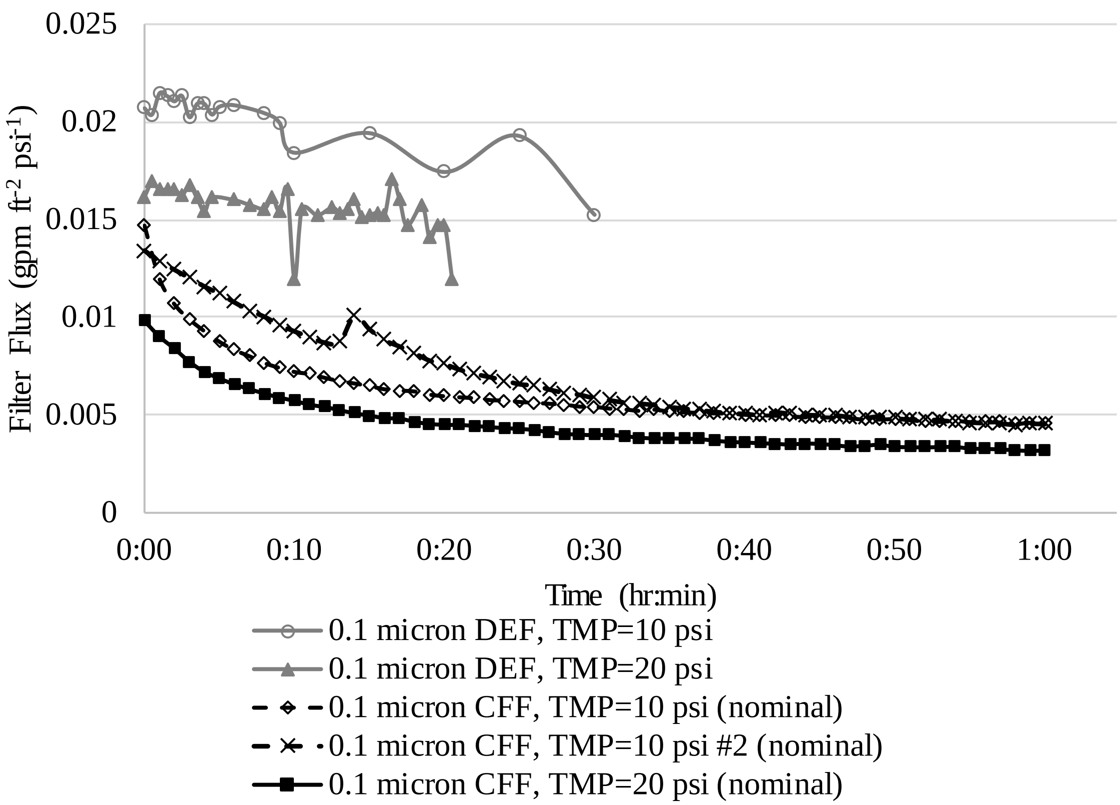

Figure 12 Media Grade 0.1 CFF Filtration and 0.1 Media Grade DEF Data with Pressure Normalized Filter Flux

CONCLUSIONS

The results of the tests performed on supernate from Hanford Tank AP-107 indicate that the media grade 5 filtration appeared effective at removing solids from the AP-107 feed. However, as part of the pretreatment steps for vitrification of the waste, cesium is removed via ion exchange and long term effects to cesium ion exchange were not examined. Although solids removal appeared effective, it is still possible that solids are getting through that may cause issues for ion exchange based upon the flux decline observed during DEF filtration from the BDEF filtered permeate. When comparing both the 5 media grade and 0.1 media grade filters in CFF, the 5 media grade filter flux was, on average, 200 times higher than the 0.1 media grade filter while still effectively filtering the solids.

Comparing the 5 media grade performance in both the CFF and BDEF showed a relatively consistent initial permeate flux ranging from 0.8-0.9 gpm ft^{-2} psi^{-1} for the BDEF to 1.05 gpm ft^{-2} psi^{-1} for the CFF. Feed previously passed through the 5 media grade CFF showed no flux decline when filtered through the BDEF. However, as fresh unfiltered feed is introduced to the system, the BDEF begins to foul where the CFF did not. CFF testing has identified a critical pressure at which fouling begins as determined by the filtration number [6]. With a large dependency on applied pressure, it is possible the low TMP tested for the media 5 CFFs could have attributed to the lack of fouling on the filter.

When relating the 0.1 media grade performance in CFF and DEF, nominal flux values for both DEF evolutions appeared higher than the CFF initial flux values by nominally 30%. The flux values during the 10 psi TMP are consistently 20-30% higher than the 20 psi TMP condition for both the DEF and CFF data and is consistent with that observed in previous testing. These results indicate that the tendency for the membrane to foul is increased at higher pressure.

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