Testing of a Rotary Microfilter for Hanford Applications - 9121

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ABSTRACT

Savannah River National Laboratory (SRNL) researchers are investigating and developing a rotary microfilter for solid-liquid separation applications with emphasis on deployment in radioactive services. The Department of Energy (DOE) Office of Waste Processing employed the SRNL team to evaluate the use of this rotary microfilter for the Hanford Supplemental Pretreatment process. The authors tested a full-scale, 25-disk filter unit containing 0.5 □ filter media using a Hanford Tank AN-105 simulant at solids loadings of 0.06, 0.29, and 1.29 wt %.

Based on recommendations from prior tests, the authors modified the filter unit by replacing the primary mechanical seal with an air seal. They also replaced the bushing with alternate materials of construction aimed at extended mean time between maintenance events.

The testing provides the following conclusions.

- The rotary filter produces a higher flux than the crossflow filter for the Hanford simulant. The gain in performance is less than previously seen for Savannah River Site simulants.
- Filtrate clarity proved excellent with turbidity of <4 NTU[*] in all samples.
- Inspection of the primary mechanical seal faces after ~ 140 hours of operation showed an expected minimal amount of initial wear, no passing of process fluid through the seal faces, and very little change in the air channeling grooves on the stationary face.
- Some polishing of surfaces occurred at the bottom of the shaft bushing. The authors recommend improving the shaft bushing by holding it in place with a locking ring and incorporating grooves to provide additional cooling.
- The authors recommend that Hanford test other pore size media to determine the optimum pore size for Hanford waste.
- During final facility operation, the filter should be rinsed with filtrate or dilute caustic and drained prior to an extended shutdown to prevent the formation of a layer of settled solids on top of the filter disks.

INTRODUCTION

SRNL researchers identified and tested the rotary microfilter as a technology to increase solid-liquid separation throughput. [1, 2, 3, 4] The testing showed significant improvement in filter flux with the rotary microfilter over the baseline crossflow filter (i.e., 2.5 – 6.5X during the scoping tests, as much as 10X in actual waste tests, and approximately 2X in pilot-scale tests).

SRNL received funding from DOE Office of Waste Processing (formerly Office of Cleanup Technologies), to develop the rotary microfilter for high level radioactive service. The work focused on

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[*] NTU = nephelometric turbidity units

evaluating alternative rotary microfilter vendors, redesigning the equipment for radioactive service, engineering studies to evaluate the risks, determining downstream impacts, assessing costs and benefits of deploying this technology, performing actual waste and pilot-scale testing of the technology, and evaluating alternative filter media. The work led to the decision to design, fabricate and perform testing on a full-scale rotary microfilter for potential SRS Tank Farm applications.

SRNL performed the additional tests to evaluate the rotary microfilter. The tests demonstrated flushing of the filter housing and effective removal of soluble and insoluble contaminants. Testing examined the rotary microfilter performance with simulated small column ion exchange feed and observed ~ 6X improvement in filter flux with the rotary microfilter over a crossflow filter with similar feed. Following that test, they conducted simulated sludge washing and found the rotary filter unit behaved as a continuous stirred tank reactor. They concentrated the feed to 20 wt % solids, and the rotary microfilter flux was ~ 6X the flux measured with a crossflow filter at similar solids loadings. [5]

Because of the success of that testing, the Hanford Site is evaluating the use of the rotary microfilter for its Supplemental Pretreatment process. [6] The authors received funding from DOE Office of Waste Processing to continue the development of the rotary microfilter and to evaluate its suitability for being the solid-liquid separation technology for Supplemental Pretreatment.

The SpinTek high shear rotary filter used in this testing has 25 filter disks covered with 0.5 μ pore size (nominal) sheet membranes (0.018 cm or 0.007 inch thick) manufactured by Pall Corporation (PMM050). The filter area of each disk is 0.089 m^2 (0.96 ft^2). The disks are physically mounted on and are hydraulically connected to a common hollow rotating shaft. The entire stack of membrane disks is enclosed within a vessel. Feed is fed into the filter vessel through the inlet on the side of the vessel wall. A pressure is set in the tank by restricting the outlet flow typically using a gate valve on the concentrate piping. This applied pressure forces liquid through the filters on the filter disk. Between each disk is a set of baffles or turbulence promoters. These turbulence promoters cause strong currents and eddies at the surfaces of the membrane inhibiting the formation of a filter cake. Filtrate flows through the media and along a mesh inside the disk into the hollow shaft. The filtrate then flows through the shaft to the rotary joint which allows the spinning shaft to couple to stationary piping. The concentrated slurry exits the vessel through an outlet on the bottom. **Figure 1** illustrates the flow paths across the filter disks during filtration.

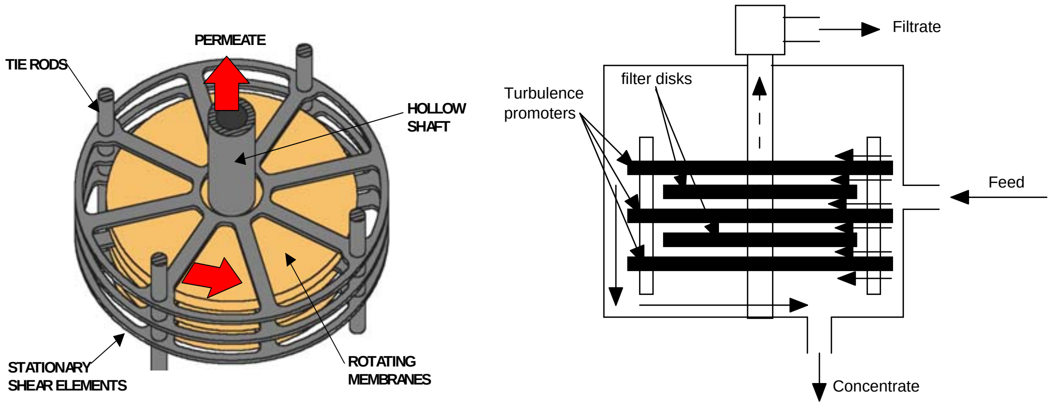

Figure 1. Diagram of Rotary Filter Principle of Operation

The advantage of the rotary microfilter compared to other membrane processes results from the high shear acting on the boundary layer next to the membrane. This shear greatly reduces fouling of the membrane surface thereby increasing fluid flow through the membrane. Pressure is decoupled from the feed flow rate, allowing more control over the driving force pressure and independent control of the shear applied to the filter cake. This feature allows the direct application of shear force with a magnitude significantly greater than that available in conventional membrane systems. The membranes rotate at a tip speed of 18.3 m/s (60 ft/s) in close proximity to the turbulence promoters. For comparison, previous cross-flow filter testing used axial velocities ranging from 0.9 to 7.6 m/s (3 to 25 ft/s). [1-4] This velocity creates high speed currents and eddies near the membrane surface. These eddies create a great deal of turbulence at the membrane surface decreasing the buildup of filter cake on the membrane. The SpinTek rotary filter unit uses 0.28-m (11-inch) diameter disks and typically operates with a rotational speed of 1170 rpm.

TESTING

The authors performed the rotary filter testing with a full-scale, 25-disk unit that had been used in previous testing to support the small column ion exchange and sludge washing applications for SRS. [5] The pump used in testing was a six stage centrifugal booster pump that had been used in the previous testing. It produced a flow rate of 68-95 L/min (18 – 25 gpm) with a feed pressure of 0.41-0.69 MPa (60 – 100 psi). Pressure was measured using manual dial pressure gages. Feed and filtrate flow were measured using Fischer Porter Magnetic flow meters. The temperature of the process fluid was measured in the feed tank with a Type K thermocouple. All data taken during testing was recorded by hand on data sheets. To minimize the amount of feed slurry needed, the concentrate and filtrate streams are recombined in the feed tank. The feed tank is mixed by recirculation of the concentrate and filtrate streams and by a 1 hp agitator.

Prior to the tests conducted here, the filter unit was modified by replacing the silicon carbide/silicon carbide faced John Crane Type 1 mechanical seal with a John Crane Type 28LD air cooled seal. The material of the bottom shaft bushing was changed from graphite to silicon-carbide. To prevent excessive wear on the shaft, an additional silicon carbide sleeve was added so that the contact wear surfaces at the bottom of the shaft are both silicon carbide.

The filter disks used in testing were a set of 25 un-used disks.

Technicians prepared a simulated Hanford AN-105 feed slurry containing 5 M sodium. The recipe is based on a prior test simulant, but it eliminates selected heavy metals. [7]

Table 1 shows the composition of the supernate and Table 2 shows the solids fractions of the slurry. Technicians prepared 380 L (100 gallons) of supernate as follows. They added 75.6 kg of de-ionized water to a tank. Next, they added sodium aluminate, sodium hydroxide (50 wt % solution), boric acid, calcium nitrate, cesium nitrate, magnesium nitrate, potassium nitrate, zinc nitrate, sodium chloride, sodium fluoride, sodium sulfate, and potassium molybdate. They mixed the solution until all of the compounds dissolved. Next, they added sodium silicate, sodium acetate, sodium formate, sodium glycolate, sodium oxalate, and sodium phosphate, mixing the solution after the addition of each compound. They added an additional 113.4 kg of de-ionized water, and mixed the solution thoroughly. They added the sodium carbonate, and mixed thoroughly. They added the sodium nitrate and sodium nitrite, and mixed the solution thoroughly. They added an additional 146.7 kg of de-ionized water, and mixed the solution overnight.

Technicians prepared the solids fraction of the slurry as follows. They procured all of the compounds, except for sodium oxalate, with particle size less than 10 μ. The sodium oxalate was not available as less than 10 μ, so SRNL personnel ground the sodium oxalate particles using a Union Process SG-1 Attritor Mill and measured the particle size of the product with a scanning electron microscope. The analysis showed the particles to be less than 10 μ. They mixed the compounds together in the ratios shown in Table **2**.

Table 1. Hanford AN-105 Supernate

Compound	Target Concentration (g/L)
$NaAlO_2$	56.661
NaOH	64.461
H_3BO_3	0.137
$Ca(NO3)_2.4H_2O$	0.111
$CsNO_3$	0.114
$Mg(NO_3)_2.6H_2O$	0.027
KNO_3	9.030
$Zn(NO_3)_2.6H_2O$	0.022
NaCl	7.039
NaF	0.197
Na_2SO_4	0.536
K_2MoO_4	0.096
$Na_2SiO_3.9H_2O$	1.003
$NaCH_3COO.3H_2O$	2.241
HCOONa	2.044
$HOCH_2COONa$	0.706
Na_2C_2O4	0.436
$Na_3PO_4.12H_2O$	1.072
Na_2CO_3	10.405
$NaNO_3$	98.500
$NaNO_2$	78.211

Table 2. Hanford AN-105 Solids

Compound	Solids Fraction (%)
Al_2O_3	9.2
CaOxalate	5.0
Cr_2O_3	26.0
Fe_2O_3	1.1
MnO_2	0.3
NaOxalate	52.5
NiO	0.5
SiO_2	5.4

Technicians prepared the slurry as follows. They added 300 L (80 gallons) of supernate and 226.04 g of solids to the filter feed tank to produce a 0.06 wt % solids slurry. They fed the slurry to the filter at a feed flow rate of ~95 L/min (~25 gpm), a feed pressure of ~0.48 MPa (~70 psi), and a feed temperature of ~35 °C. The filtrate pressure was ~ 0.21 MPa (~30 psi), producing a transmembrane pressure of ~0.28 MPa (~40 psi). They set the rotor speed to 1170 rpm. The filter operated for ~40 hours on day shift (i.e., ~ 8 hours per day, 5 times per week), and personnel recorded the operating parameters and filtrate flow rate during the test. The operating parameters recorded were feed flow rate, filtrate flow rate, feed pressure, concentrate pressure, filtrate pressure, temperature, and rotor speed. Motor current and output power, along with the surface temperatures of the rotary joint and mechanical seal housing were measured at random intervals. They collected filtrate samples twice each day of operation to measure turbidity.

After operating for 40 hours, they added an additional 866.5 g of solids to the feed tank to produce 0.29 wt % solids slurry. They fed the slurry to the filter at a feed flow rate of ~95 L/min (~25 gpm), a feed pressure of ~0.48 MPa (~70 psi), and a feed temperature of ~35 °C. The filtrate pressure was ~ 0.21 MPa (~30 psi), producing a transmembrane pressure of ~0.28 MPa (~40 psi). They set the rotor speed to 1170 rpm. The filter operated for ~40 hours on day shift, and personnel recorded the operating parameters and filtrate flow rate during the test. They collected filtrate samples daily to measure turbidity.

After operating for 40 hours, they added an additional 3767.38 g of solids to the feed tank to produce 1.29 wt % solids slurry. They fed the slurry to the filter at a feed flow rate of ~95 L/min (~25 gpm), a feed pressure of ~0.48 MPa (~70 psi)i, and a feed temperature of ~35 °C. The filtrate pressure was ~ 0.21 MPa (~30 psi), producing a transmembrane pressure of ~0.28 MPa (~40 psi). They set the rotor speed to 1170 rpm. The filter operated for ~40 hours on day shift, and personnel recorded the operating parameters and filtrate flow rate during the test. They collected filtrate samples daily to measure turbidity.

RESULTS

Mechanical Performance, Flux and Filtrate Clarity

Operation of the filter proceeded with no mechanical upsets or interruptions. **Figure 2** shows the flux data for the various slurries. For the 0.06 wt % slurry, the filter flux approached a steady value in approximately 10 hours. The flux averaged 10.6 L m^{-2} min^{-1} or 23.7 L/m total (0.26 gpm/ft^2 or 6.25 gpm total). Additional solids were added to the feed to raise the insoluble solids concentration to 0.29 wt %. After reaching a near constant value, the filter flux averaged 6.9 L m^{-2} min^{-1} or 15 L/m total (0.17 gpm/ft^2 or 4 gpm total). The filter reached a near constant value after approximately 15 hours. Personnel added solids to reach 1.29 wt % slurry. After reaching a near constant value, the filter flux averaged approximately 4.1 L m^{-2} min^{-1} or 9.1 L/m total (0.10 gpm/ft^2 or 2.4 gpm total). The filter flux reached a near constant value after approximately 25 hours. All filtrate samples had turbidity less than 4 NTU.

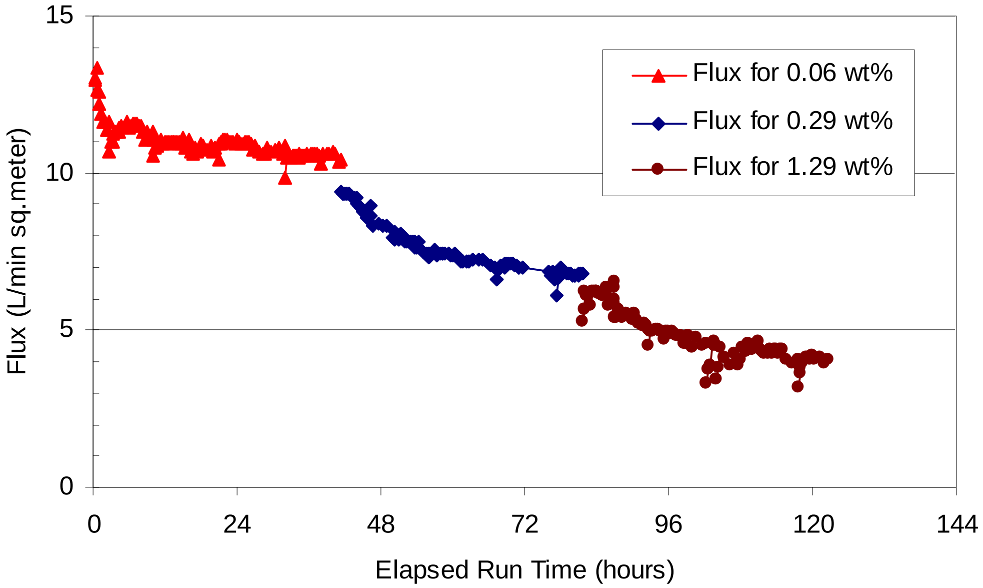

Figure 2 Flux for 0.06 wt %, 0.29 wt % and 1.29 wt % Insoluble Solids at TMP of 0.28 MPa (40 psi)

One can compare the flux of the AN-105 simulant to the flux measured during a prior test with simulated SRS sludge (see **Figure 3**). [5] The comparison is appropriate since both sludges contain similar compounds (e.g., metal oxides) and have relatively similar particle size (mean 1 – 5 μ). At the start of the testing with 0.06 wt % solids, the AN-105 simulant had a higher flux than the SRS sludge simulant. When we increased the solids loading to 0.29 wt %, the flux with the AN-105 simulant was initially higher. By the end of that test, the flux was approximately the same for both feed slurries: 6.9 L m^{-2} min^{-1} for a total filtration rate of ~15 L/min at this solids loading. When we increased the solids loading to 1.29 wt %, the flux with SRS simulant remained approximately the same, while the flux with AN-105 decreased. Both simulants had approximately the same starting flux of 4.1 L m^{-2} min^{-1}. The flux with the AN-105 simulant continued to decay until reaching approximately 9.1 L/min of filtrate for the entire unit.

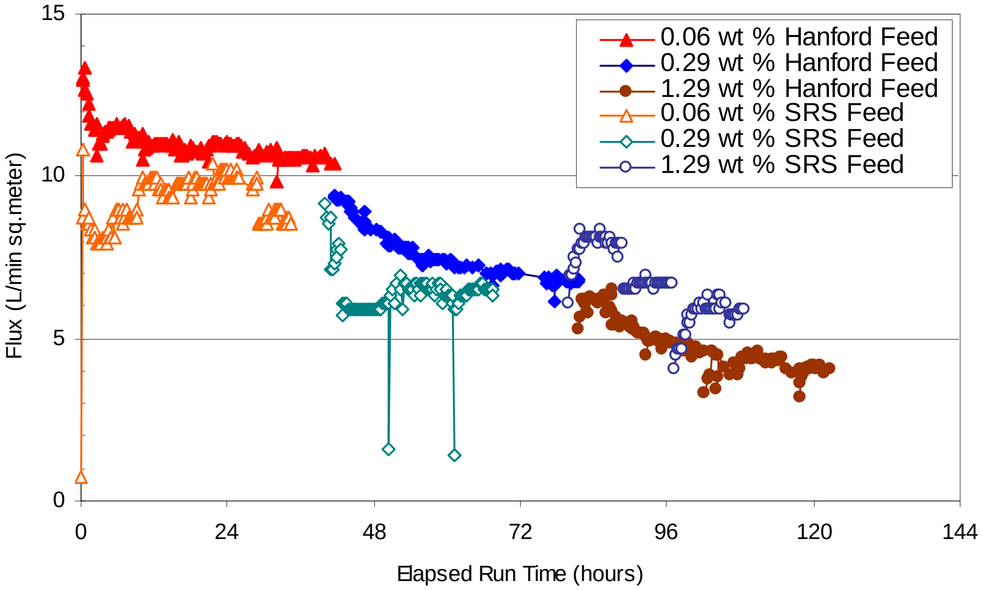

Figure 3 Comparison of SRS and Hanford Simulant Flux Rates

One may also compare the flux in this test with the flux measured during a crossflow filter test with an AN-105 simulant. [8] Because the tests used different solids loadings, different filter pore size, and differences in simulant recipe, a direct comparison is not available. Comparing the rotary filter flux at 0.06 wt % solids with the crossflow filter flux at 0.5 wt % solids shows the rotary filter flux is 1.8 – 3.0 X higher. Comparing the rotary filter flux at 0.29 wt % solids with the crossflow filter flux at 0.5 wt % solids shows the rotary filter flux is 1.15 – 2.0 X higher. Comparing the rotary filter flux at 1.29 wt % solids with the crossflow filter flux at 0.5 wt % solids shows the rotary filter flux is 0.7 – 1.25 X of the flux with a crossflow filter.

Table 3 shows the particle size distribution of the solids in the current test and in the rotary filter test using SRS sludge as measured with a Microtrac SRA-150. The table shows the volume fraction of particles in each size range and the cumulative volume fraction of particles less than each size. The carrier fluid for the measurement was simulated salt solution (i.e., SRS salt solution for SRS sludge and AN-105 salt solution for AN-105 solids). The median particle size of the AN-105 solids was 1.49 µ. The median particle size of the SRS solids was 3.32 µ. In addition, the AN-105 solids had a larger fraction of particles less than 1 µ. According to various filtration theories, filter flux increases with increasing particle size. The relationship is described by equation [1]

$$J = K\, d_p^{\,n} \qquad [1]$$

where J is filter flux, K is a constant, d_p is particle size, and n is an exponent. Various filtration models have n equal to 4/3, 2, and 3. [9] In addition, the increase in fine particles (<1 µ) would provide more particles that could penetrate the filter membrane to foul the filter pores.

A similar comparison of particle sizes is available (and reported elsewhere [10]) for the AN-105 solids from the rotary filter test and the crossflow filter test. The median particle size of the AN-105 solids in the rotary filter test was 1.49 µ. The median particle size of the solids during the crossflow filter tests was 2.32 and 2.59 µ. As described above, this larger particle size would produce higher filter flux, and may explain why the rotary filter did not show as big of an improvement in filter flux as has been observed in other rotary filter versus crossflow filter tests. In addition, the feed for the rotary filter test had a larger fraction of particles less than 1 μ than the feed for the crossflow filter tests.

Table 3. Particle Size of SRS Solids and Hanford AN-105 Solids

	SRS Solids		Hanford AN-105 Solids	
Size (□)	Fraction	Cum Fraction	Fraction	Cum Fraction
0.204	0.24	0.24	0	0
0.243	0.3	0.54	0	0
0.289	0.42	0.96	0	0
0.344	0.68	1.64	0.53	0.53
0.409	1.18	2.82	1.52	2.05
0.486	2	4.82	3.29	5.34
0.578	3.04	7.86	5.04	10.38
0.688	4.05	11.91	5.92	16.3
0.818	4.77	16.68	6.2	22.5
0.972	5.12	21.8	6.6	29.1
1.156	5.11	26.91	7.49	36.59
1.375	4.83	31.74	8.8	45.39
1.635	4.39	36.13	9.94	55.33
1.945	3.91	40.04	9.96	65.29
2.312	3.49	43.53	8.89	74.18
2.75	3.19	46.72	7	81.18
3.27	3.04	49.76	5.27	86.45
3.889	3.11	52.87	4.05	90.5
4.625	3.46	56.33	3.29	93.79
5.5	4.17	60.5	2.7	96.49
6.541	5.43	65.93	2.01	98.5
7.778	7.29	73.22	1.12	99.62
9.25	9.11	82.33	0.38	100
11	8.97	91.3	0	100
13.08	5.82	97.12	0	100
15.56	2.24	99.36	0	100
18.5	0.54	99.9	0	100
22	0.1	100	0	100
26.16	0	100	0	100
31.11	0	100	0	100

Disassembly and Inspection

After completion of the operational testing, personnel disassembled the filter disk stack. The filter vessel and disk stack was not flushed prior to disassembly. A significant difference in the filter cake between the top side of the disks and the bottom side was observed. **Figure 4** shows the top and bottom of a representative filter disk (i.e., third from the bottom in the stack). The filter-cake accumulation on the top side of the disk is due to the settling of the feed material when operation is complete. During testing, the filter was simply shut down at the end of the day without any draining or flushing. Additionally, no attempt was made to clean the disk in-situ such as achieved by dropping the TMP while maintaining the rotor speed. (In previous testing, this approach was shown to improve filter flux by a small amount.) The condition of the filter disks is consistent with previous observations with the top side of the disks showing a thicker cake of solids. This leads to the conclusion that the filter is better at preventing the accumulation of filter cake than dispersing a filter cake that has already formed. To prevent the buildup of similar filter-cake in deployment, it is recommended that the filter be drained and flushed with filtrate or dilute caustic after shutdown.

Figure 4 Top (left) and Bottom (right) Sides of Filter Disk

Seal Wear Inspection

After disassembly was completed, the shaft seal was removed and inspected. There was no indication that any of the process fluid passed the seal. Photographs of the seal surfaces – and other surfaces discussed in this section – are available in a more detailed report. [10]

The carbon face of the rotor did show polishing, or general erosion, in the area indicative of initial wear. This polishing is due to contact of the seal faces, primarily at startup and shutdown, when there is not enough velocity to cause liftoff for the faces. No evidence of the passing of process fluid was observed. Very little change to the air channeling grooves on the stationary was observed, though no depth measurements were obtained since these measurements would have required the removal of the seal stationary.

Some polishing, or general erosion, was observed on the bottom of the shaft bushing as well as the receiver bushing. The shaft bushing is not supported and is held in place by a sealant. This sealant was compromised by the process fluid allowing the shaft bushing to contact the bottom of the receiver bushing. It is recommended that the shaft bushing be updated to allow it to be held in place by a retaining ring as well as incorporated grooves to allow for additional cooling flow.

CONCLUSIONS

The following conclusions result from this testing.

- The filter flux at 0.06 wt % solids reached a near constant value at an average of 10.6 L m^{-2} min^{-1} or 23.7 L/m total (0.26 gpm/ft^2 or 6.25 gpm total).
- The filter flux at 0.29 wt % solids reached a near constant value at an average of 6.9 L m^{-2} min^{-1} or 15 L/m total (0.17 gpm/ft^2 or 4 gpm total).
- The filter flux at 1.29 wt % solids reached a near constant value at an average of 4.1 L m^{-2} min^{-1} or 9.1 L/m total (0.10 gpm/ft^2 or 2.4 gpm total).
- The rotary filter produces a higher flux than the crossflow filter for the Hanford simulant. The gain in performance is less than previously seen for Savannah River Site simulants.
- Filtrate clarity proved excellent with turbidity of <4 NTU in all samples.

- Inspection of the primary mechanical seal faces after ~ 140 hours of operation showed an expected minimal amount of initial wear, no passing of process fluid through the seal faces, and very little change in the air channeling grooves on the stationary face.
- Some polishing of surfaces occurred at the bottom of the shaft bushing. The authors recommend improving the shaft bushing by holding it in place with a locking ring and incorporating grooves to provide additional cooling.
- The authors recommend that Hanford test other pore size media to determine the optimum pore size for Hanford waste.
- During final facility operation, the filter should be rinsed with filtrate or dilute caustic and drained prior to an extended shutdown to prevent the formation of a layer of settled solids on top of the filter disks.

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