

Testing of the Second Generation SpinTek Rotary Filter -11357

David Herman*, Michael Poirier*, Mark Fowley*, Mark Keefer**, Thomas Huff**,
William Greene***, Jason Gilmour***

*Savannah River National Laboratory, Aiken, South Carolina 29808

**Savannah River Remediation LLC, Aiken, South Carolina 29808

***SpinTek Filtration, Los Alamitos, California 90720

Abstract

The SpinTek rotary microfilter has been developed under the Department of Energy (DOE) Office of Environmental Management (EM) for the purpose of deployment in radioactive service in the DOE complex. The unit that was fabricated and tested is the second generation of the filter that incorporates recommended improvements from previous testing. The completion of this test satisfied a key milestone for the EM technology development program and technology readiness for deployment by Savannah River Remediation in the Small Column Ion Exchange and Sludge Washing processes at the Savannah River Site (SRS).

The Savannah River National Laboratory (SRNL) contracted SpinTek Filtration to fabricate a full scale 25 disk rotary filter and perform a 1000 hour endurance test with a simulated SRS sludge. Over 1500 hours of operation have been completed with the filter.

SpinTek Filtration fabricated a prototypic 25 disk rotary filter including updates to manufacturing tolerances, an updated design to the rotary joint, improved cooling to the bottom journal, decreases in disk and filter shaft hydraulic resistances. The filter disks were fabricated with 0.5 μ pore size, sintered-metal filter media manufactured by Pall Corporation (M050). After fabrication was complete, the filter passed acceptance tests demonstrating rejection of solids and clean water flux with a 50 % improvement over the previous filters. Once the acceptance test was complete, a 1000 hour endurance test was initiated simulating a sludge washing process. The test used a simulated SRS Sludge Batch 6 recipe. The insoluble solids started at 5 wt % and were raised to 10 and 15 wt % insoluble solids to simulate the concentration of a large volume tank.

The filter system was automated and set up for 24 hour unattended operation. To facilitate this, process control logic was written to operate the filter. During the development it was demonstrated that the method of starting and stopping the filter can affect the build up of filter cake on the disks and therefore the performance of the filter.

The filter performed well with the simulant. Very little drop in production was noticed between the 5 and 10 wt % insoluble solids feed. Increasing to 15 wt % had a more pronounced impact due to the rheology of the feed.

Acid cleaning was used to clean the filter disks in-situ and restore filtration rate to almost 90% of the initial clean disk rate. Eighty liters of 0.2 M nitric acid in conjunction with water rinses were used to clean the filter in less than 2 hours.

Filter testing was completed after 1000 hours of operation were performed on the final filter assembly configuration. The total run time for the testing was over 1500 hours. At the end of

the test, the sludge washing was performed successfully from approximately 5.6 M to less than 1 M sodium.

Introduction

The SpinTek rotary microfilter has been developed under the Department of Energy (DOE) Office of Environmental Management (EM) for the purpose of deployment in radioactive service in the DOE complex. Radioactive waste treatment processes rely upon solid-liquid separation to segregate and efficiently treat the high level and low level waste components. The advantage of the rotary filter over other filtration methods is its compact design, allowing the filter to be deployed in existing facilities (i.e., waste tanks) thereby reducing the overall costs of waste disposition.

The rotary filter is planned for deployment in risers of the Savannah River Site (SRS) waste tanks for both sludge washing and Small Column Ion Exchange program. In-tank deployment of a filter and ion exchange columns is a “transformational” initiative supported by Savannah River Remediation, DOE-EM, and the Savannah River National Laboratory (SRNL) to reduce the current lifecycle schedule at SRS by six years and \$3.6 billion. This process will utilize monosodium titanate (MST) for Actinides/Strontium removal and crystalline silicotitanate (CST) for cesium removal. The rotary filter will be used as a prefilter for the sorbent columns. Sludge washing will also use an in-tank deployment of the filter. Mechanical filtration would reduce processing durations and dilution water required.

Testing has been completed on single disk lab scale[1], three disk pilot scale[2] and first generation full scale twenty five disk[3,4] filter units. The technology has been primarily tested for potential deployment as a prefilter for ion exchange columns and solvent extraction, with limited testing for sludge washing applications. All testing to date has been relatively short term. Total operational time on a single 25-disk filter unit is a total of approximately 500 hours. The longest continuous run time was approximately 12 hours.

The SpinTek filter used in this testing has 0.5 μ pore size, sintered-metal membrane filter disks. Each disk contains approximately 1 square foot of filter media. During the last test campaign for the Generation-1 25 disk filter, several improvements were identified and were incorporated into the construction of this filter. These improvements included improving manufacturing tolerances, improvements to the rotary joint design, improved cooling to the bottom journal, decreases in disk and filter shaft hydraulic resistances.

Since the previous testing had also been more focused on the ion exchange prefilter application, the feed to the filter has primarily been salt solution simulants with a low concentration of large diameter sludge particulates. For the sludge washing application, the filter would be exposed to more viscous slurries and higher concentrations of varying particle sizes. This test utilized a slurry simulant more indicative of real sludge waste [5]. The simulated sludge slurry developed for this test was based on recent experience with processing of sludge from Tank 12 at SRS.

SRNL contracted SpinTek Filtration to perform a 1000 hour endurance test with a SpinTek 25-disk Rotary Microfilter as part of the EM-31 program for alternate solid-liquid separation

technologies. These filtration tests were intended to evaluate the performance of the rotary microfilter for separating sludge solids from simulated SRS supernate.

The filtration testing occurred at the SpinTek facility located in Los Alamitos California. The test facility includes the necessary process and data acquisition equipment, including a second generation full scale 25 disk Rotary Microfilter to conduct a 1000 hour endurance test. A contract was established for the fabrication of what is being called the Generation-2 rotary filter and for a 1000 hour operational test. A photo of the filter and test stand is shown as Figure 1.



Figure 1. Rotary Filter Test Stand at SpinTek Facility

Testing Characteristics

Sludge Simulant

The slurry simulant used simulates the SRS Sludge Batch 6 Tank 12 sludge. Sludge washing relies upon gravity settling of the slurry for solid-liquid separation. The Tank 12 sludge exhibited very poor particle settling rates than previous sludge batches, very high fluid viscosity and a very high yield stress. These material attributes served as the basis for the simulant.

The slurry simulant does not include Resource Conservation and Recovery Act (RCRA) metals or halides. The RCRA metals (barium, chromium, and lead) were removed from the recipe. The weight percent for all three totaled 0.37% and is considered masked by the elimination of uranium which represented over 4% of the original recipe target and is normalized out of the recipe. The halide salts (sodium chloride, and sodium fluoride) were replaced by sodium nitrate on a molar basis. The target and measured anion and cation compositions of the feed simulant is summarized in Table 1.

Table 1. Concentration of Anion/Cation Content of Sludge Batch 6 Feed Simulant

Component	Calcined Solids Wt %	
	<i>Target</i>	<i>Actual</i>
Al*	16.181	15.8
Ca	1.147	1.08
Ce	0.085	0.08
Cu	0.085	0.10
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	2.30
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
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
<u>Anions</u>	mg/kg	mg/kg
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

*The Al wt% has not been adjusted to account for replacement of Ba, Cr, and Pb. The weight percent for all three totaled 0.37% and is considered masked by the elimination of U which represented over 4% of the original recipe target. Uranium was normalized out of this recipe.

Rheology was measured by SRNL on an as-received sample of the simulated sludge. The yield stress was measured as a function of weight percent solids is shown in Figure 2. Measurements were done with in a 3 M salt solution. The rheology of the simulant was compared with that of samples of actual sludge. Curves of yield stress as a function of weight percent solids for several actual waste tank samples are also shown in Figure 2.

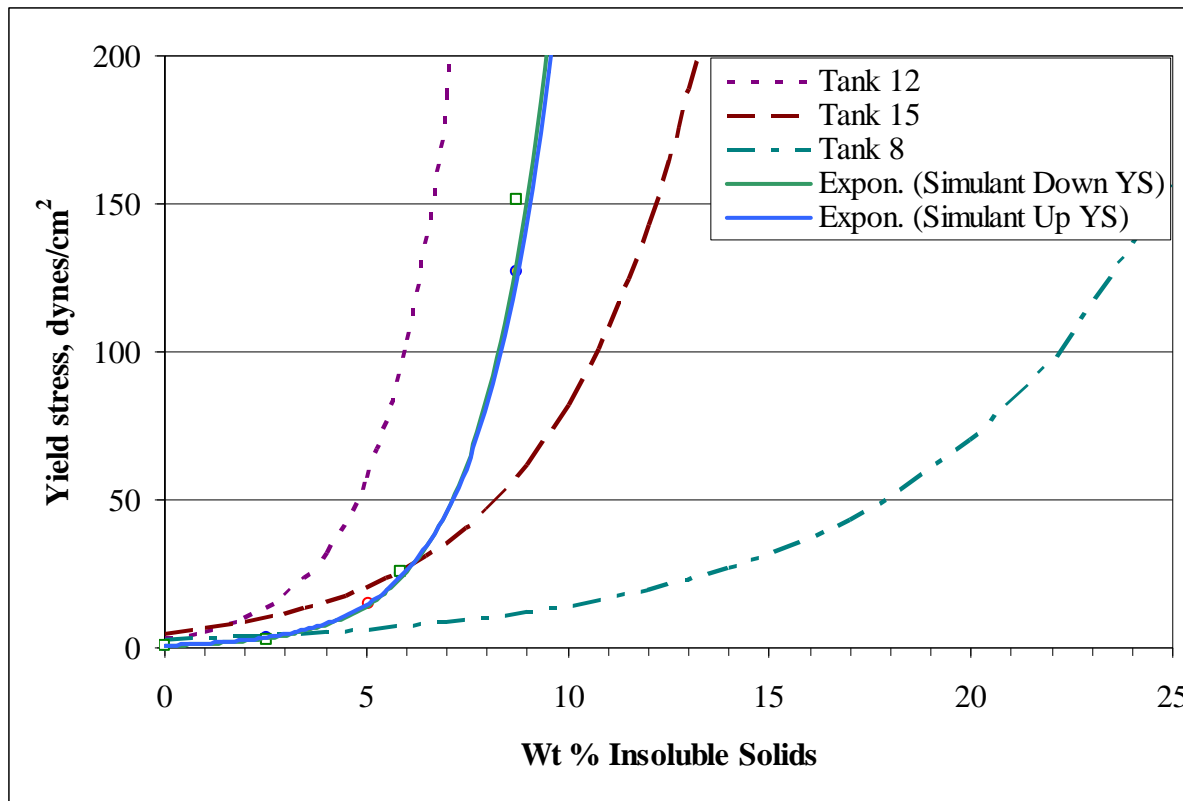


Figure 2. Rheology of the SB6 Simulant Compared to Rheology of Waste Tank Samples

Particle size distribution was also measured for the simulated sludge and found to be a tri-modal distribution with peaks at 0.8, 9 and 50 microns with a mean particle size of 8.7 microns.

Supernate Simulant

Soluble salts were added to the simulated sludge to produce a simulated supernate of 5.6 M sodium. The target composition of the supernate is shown in Table 2. Bulk Supernate Composition

Table 2. Bulk Supernate Composition

Component	Concentration (M)
Free OH ⁻	1.33E+00
NaNO ₃	2.60E+00
NaAl(OH) ₄	4.29E-01
NaNO ₂	1.34E-01
Na ₂ SO ₄	5.21E-01
Na ₂ CO ₃	2.60E-02
Total Na	5.6

Experimental Procedure

The task was to conduct an extended duration (over 1000 hours) test with the filter unit to simulate sludge concentration and washing. The test covered the range of (nominally) 5 to 15 wt % insoluble solids using simulated SRS sludge. To utilize a more manageable volume of liquid, the filtrate was recycled into the feed tank. Instead of a gradual concentration of insoluble solids in the feed, the insoluble solids were increased in steps.

During the test, feed samples were analyzed for particle size, rheology, and composition. Filtrate samples were collected and turbidity measurements were made to assure filtrate quality. Filter flux was measured as a gauge of filter performance. The nominal operating conditions were 95 – 190 liters per minute (lpm) feed flow rate, 200 – 415 kPa net pressure difference across the filter, 35 - 40 °C temperature, and 1170 rpm rotor speed.

During testing, vibration of the filter unit was measured continuously by four IFM electronic VSA001 sensors located by the main shaft bearing and the rotary joint. Maximum readings from these sensors were recorded by the PLC. Individual snap shots of the vibration section for these four sensors were also obtained. The measurements will enable the researchers to establish a baseline on the unit's vibration, and monitor changes in the vibration signature of the filter due to increase solids loading.

Results and Discussion

Endurance Testing Results

A graph of the filtration rate throughout the testing is shown in Figure 3. After the transition from 5 to 10 wt % insoluble solids the filter tripped off due to a feed tank level alarm. No indications of any problems with the filter were evident. However, when the filter stack was inspected during the outage, a chip was discovered in the journal bearing. The original journal bearing material, silicon carbide on silicon carbide, was replaced with Stellite on Nitronic 60. The test was restarted with the 10 wt % insoluble solids but the test clock was reset to zero.

After approximately 300 hours into the restart, the filter was again shut down by the controller logic due to the loss of a support system air compressor. At that point the filter was cleaned in-situ, which will be described later.

After approximately 660 hours, the insoluble solids were raised to 15 wt %. During the 15 wt % testing, three separate trips occurred due to either problems with the chiller used to cool the feed slurry or planned and unplanned power outages.

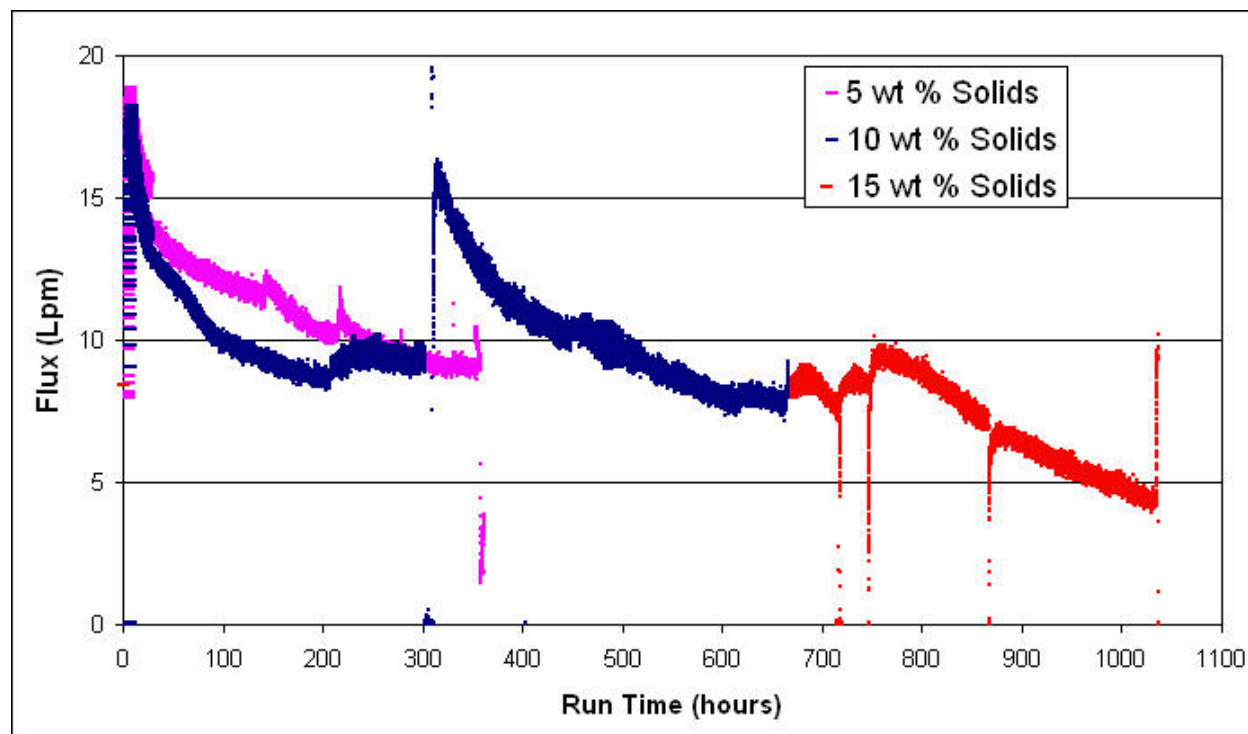


Figure 3. Permeate Flow for 1000 Hours of Operation

At the end of the testing, the feed slurry was washed by adding inhibited water, 0.01M NaOH and 0.011 M NaNO₃, at the same rate the filtrate was removed. The purpose of this was to wash the soluble salts from the feed slurry.

Performance of the filter was considered to be good. Flux started to decay as filter cake would gradually build up. A true steady state was never reached though the rate of drop in flux decreased. The increase from 5 and 10 wt % insoluble solids had very similar performance. Increasing from 10 to 15 wt % insoluble solids had a much greater impact on the performance of the filter. This parallels a significant increase in yield stress between 5, 10 and 15 wt % insoluble solids as shown in Figure 2. As expected, filtration rate increased as the soluble salts were removed. Flux more than doubled from the end of the 1000 hours to the end of the sludge washing.

Feed Tank Temperature

The temperature of the feed tank was to be maintained at 35 centigrade \pm 5 centigrade except at initial start up and subsequent restarts. However, due to atmospheric conditions, the chiller used to cool the feed was unable to provide adequate cooling. Supplemental once-through cooling was used to maintain temperature. The target feed temperature was adjusted by 3 degrees to minimize the amount of supplemental cooling required. Feed temperature was increased to 38 degrees Celsius starting at approximately 575 hours into the test.

Feed Flow

Feed flow ranged between of 121 and 167 liters per minute throughout the test. Primary control of the system is based on pressure. As the system automation adjusted to maintain pressure, feed flow varied per the pump curve.

Feed and Filtrate Pressure

The pressure drop across the filter membranes was held at approximately 276 kPa throughout the test. Feed pressure was typically in the range of 448 kPa with the filtrate line at approximately 172 kPa. A back pressure of approximately 172 kPa was applied to the filtrate line by partially closing the valve in the filtrate line. The backpressure is applied since the seal manufacturer for the double mechanical seal in the rotary joint recommends at least 100 kPa across the seal faces.

Vibration

A vibration signature was measured with the filter operated with water prior to operation with simulants to establish a baseline vibration signature. Vibration was measured throughout testing with the system PLC recording the maximum vibration from four sensors. The sensors were located in the X, Y and Z plane for the main shaft bearing and the X-plane for the rotary joint. Screen shots of the vibration spectrum were obtained throughout testing.

The highest vibration was generally found in the rotary joint. Initially, the highest velocity was measured be approximately 8 mm/s. Very little change was observed transitioning from water to sludge and then with increasing sludge levels. It was determined during testing that the filter mounting frame had a large influence on the vibration magnitude. Several hundred hours into the test, two additional braces were added to the frame. The consequence was the reduction of the vibration maximum velocity to approximately 3.5 mm/sec at the rotary joint and 1.77 mm/sec at the main bearing.

Filtrate Clarity

Filtrate samples were obtained at a minimum of one per day. Turbidity was measured in Nephelometric Turbidity Units (NTU). Maximum turbidity was measured to be less than 2.5 NTU with the majority of samples less than 1 NTU. Figure 4 shows the results of the turbidity measurements.

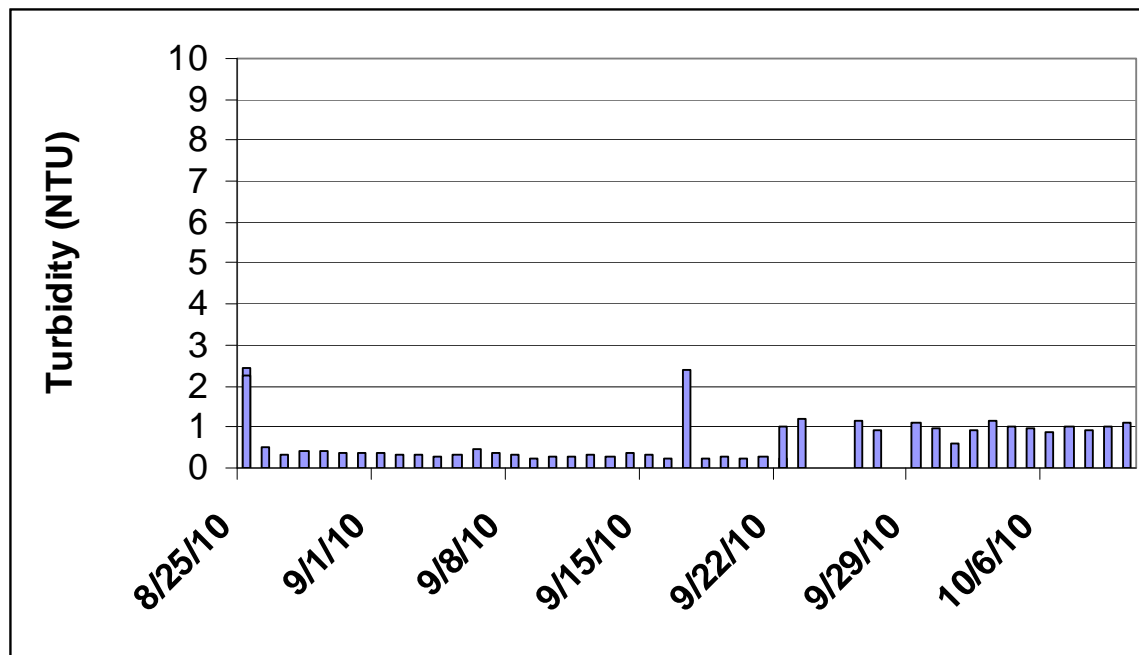


Figure 4. Turbidity Measurements

Operational Issues and Improvements

Journal Bearing

The filter was in operation for approximately 156 hours when the support system air compressor failed. This caused the rotor to trip off. The PLC logic at the time failed to trip the feed pump and allow the filtrate line to remain open. This resulted in a dead end filtration of the filter disks for 2 to 3 hours. Upon restart, flux was very poor and improved to approximately 5.7 lpm. SpinTek decided to stop the filter and inspect the filter stack. Upon stack removal, it was discovered that the bottom journal bearing had chipped.

The journal was replaced and the test clock was reset to zero. Initial insoluble solids loading was approximately 5 wt % in a 5.6 M sodium salt solution. Approximately 362 hours into restart of filter operation as the solids loading was being increased, one of the tank alarms was accidentally tripped and the filter was shut down. An inspection revealed a chip in the journal bearing similar to the first chip. As with the previous incident, the chip was retained in place. No indication of this occurrence was conclusively found in any of the running data.

The cause of the chips were determined to be a misalignment of the bottom shaft nut to the filter shaft and stress points that were designed into the journal bearing.

This journal was designed with a key drive. The keyway was determined to be a point of stress concentration. This key way has been removed from the design. The journal material was also replaced. The original material was silicon carbide on silicon carbide. This was the material configuration run successfully in previous testing [4]. The silicon carbide was replaced with

Stellite™ for the rotor portion of the journal and Nitronic 60 for the stationary. These materials were recommended based on discussions with the SRNL subject matter experts from the Materials Technology Group, Advanced Characterization Group and exhibited performance in the SRS Tank Farm in a deployed pump design. The test clock was again reset to zero since a new part was introduced.

Acid Cleaning

Approximately 663 hours into the test, the air compressor from the support system feeding the control valves in the test system failed. Note that the air compressor is a function of the test setup and not prototypic of a deployed filter. The loss of air pressure resulted in a shutdown of the filter rotor. The system responded to the PLC logic and shut the system off without incident. The filter was drained of the feed sludge. SpinTek personnel then flushed the system with de-ionized (DI) water using the supplementary pump. This water rinse was followed by an acid rinse with 80 L of 0.2 M nitric acid. The acid was flushed through the system and allowed to soak for 10 minutes. The acid was circulated a second time and allowed to soak for an additional 10 minutes. The acid was then drained and the filter was rinsed with DI water. The water was drained and the filter was restarted once the air compressor repairs were completed. The filter was down for approximately 7 hours.

Upon restart, the filter flux returned to over 15 lpm. Thus the acid flush restored flux to almost 90 % of the original clean disk flux.

Startup Logic

The manner of filter startup was shown to have an influence on filter performance. During the initial run with the sludge simulant, the pump was allowed to run for approximately 2 hours with the filtrate valve open and the rotor off. The PLC logic would not allow the rotor to start due to pressure swings in the instrumentation from the purging of air in the filter. Therefore the air had to be purged from the filter prior to the start of the rotor. The result was the filter being dead-ended for these two hours. This resulted in a build up of solids on the membranes of the disks. When the rotor was finally started, initial flux was low (less than 4 lpm) and increased as the operating rotor turbulence started to reduce the solids build up. It should be noted that the filter flux that was achieved with the filter starting presumably packed with solids. The operation of the rotor improved the filtration rate presumably by breaking up the filter cake.

Later in testing, the PLC logic was changed to allow a faster start to the rotor and the air was purged from the filter housing. This was accomplished by extending the alarm duration so that very short pressure surges were ignored by the alarm logic. In addition, the time lag between achieving minimum pressure and starting the rotor was significantly reduced. This combination resulted in a much higher initial flux.

During an early part of testing, the pressure drop across the system was increased to determine the effect on filter performance. The pressure drop across the membranes was increased by approximately 25 % from 275 to 345 kPa and flux responded by increasing approximately 33 %. This illustrates that the pressure drop across the disks may still be optimized to obtain better filter performance. This result is consistent with previous work. [3,4]

Conclusions

The SpinTek rotary microfilter successfully completed over 1000 hours of operation on several insoluble solids loadings of an SRS Sludge Batch 6 simulant mimicking the concentration of a large volume of feed and washing the soluble constituents.

The filter performed well with the simulant. Very little drop in production was noticed between the 5 and 10 wt % insoluble solids feed. Increasing to 15 wt % had a more pronounced impact due to the rheology of the feed.

Typical sludge washing is performed in a repetitive batch mode by performing large bulk water additions, relatively long gravity settling periods, and decantation of the diluted stream. The rotary filters would allow for near continuous washing, providing greater dilution efficiently and reducing the dilution water needs by up to 40%. The conceptual filter arrangement for sludge washing is two filters operating in parallel. A combined continual filtration rate of 4 to 5 gpm would eliminate gravity settling periods and reduce the overall duration required to complete sludge washing for a typical sludge batch by several months. Higher filtration rates will result in greater schedule improvements.

The filter system was automated and run for continuous unattended operation. It was demonstrated that the control logic performed well during several unexpected occurrences. During the development of the process logic control system, it was demonstrated that minimizing dead ending of the filter disks during startup and shutdown of the filter can improve the build up of filter cake on the disks and therefore the performance of the filter.

Acid cleaning was used to clean the filter disks in-situ and restore filtration rate to almost 90% of the initial clean disk rate. Eighty liters of 0.2 M nitric acid in conjunction with water rinses were used to clean the filter in less than 2 hours.

With the completion of the testing, the rotary filter successfully demonstrated filtration of a challenging SRS simulant up to 15 wt % insoluble solids in a 5.6 M salt simulant, sludge washing, in-situ acid cleaning with dilute acid, and an extended run time. As a result, the filter has completed an additional stage of readiness for deployment.

References

1. D. T. Herman, M. R. Poirier, and S. D. Fink, "Testing of the SpinTek Rotary Microfilter Using Actual Waste", WSRC-TR-2003-00030, Rev. 1, December 2003.
2. M. R. Poirier, D. T. Herman, S. D. Fink, R. Haggard, T. Deal, C. Stork, and V. Van Brunt, "Pilot-Scale Testing of a SpinTek Rotary Microfilter with SRS Simulated High Level Waste", WSRC-TR-2003-00071, February 3, 2003.
3. D. T. Herman, M. R. Poirier, and S. D. Fink, "Testing and Evaluation of the Modified Design of the 25-Disk Rotary Microfilter", WSRC-STI-2006-00073, rev. 0, August 2006.

4. D.T. Herman, D.B. Stefanko, M.R. Poirier, S.D. Fink, “Testing of a Full-Scale Rotary Microfilter for the Enhanced Process for Radionuclides Removal”, SRNL-STI-2009-00183, January, 2009.
5. H.H. Elder, “Study of Potential Application of the SpinTek Rotary Microfilter to Sludge Washing”, CBU-PIT-2005-00019, June 13, 2005.