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Pilot-Scale Testing of a Rotary Microfilter with Irradiated Filter Disks and Simulated SRS Waste

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SUMMARY

The processing rate of high level radioactive waste treatment processes at the Savannah River Site (SRS) is limited by the flow rate of the solid-liquid separation process (crossflow filtration). If SRS could identify and develop a solid-liquid separation technology with a higher processing rate, they could increase the throughput of these processes and complete treating those waste streams in a shorter time, with a significant reduction in life-cycle cost. Savannah River Technology Center (SRTC) personnel identified the rotary microfilter as a technology that could significantly increase filter flux, with improvements of as much as 10X. SRTC is evaluating and developing the rotary microfilter for radioactive service at the SRS. One aspect of this work is evaluating the impact of radiation on the filter unit and filter disks.

The authors performed pilot-scale simulant filtration tests with irradiated filter disks. They employed three types of filter disks for the tests (0.5 µ stainless steel, 0.1 µ stainless steel, and 0.1 µ ceramic/stainless steel). They analyzed the filter's structural material, Ryton®, for hardness, and irradiated the entire disk with an estimated 2.5 - 5 year (83- 165 MRad) radiation dose. They measured the hardness of the Ryton® after the irradiation of the disk. Following irradiation, they placed the filters in the pilot-scale rotary microfilter unit and tested them with feed slurries containing 0.29 and 4.5 wt % solids.

The conclusions from this work follow.
- None of the nine disks tested experienced a catastrophic failure from radiation exposure, but some evidence of delamination existed. Separation started to occur between the epoxy and the base Ryton® material.
- The flux with the irradiated filter disks was 35 – 40% lower than the flux measured with unirradiated filter disks. A likely cause of this difference is the feed to the irradiated filter disks had a smaller particle size than the feed to the unirradiated filter disks. Another plausible cause is that personnel did not thoroughly clean the filter disks following the test with irradiated disks.
- The 0.1 micron ceramic/stainless steel filter produced the highest flux.
- The 0.1 micron stainless steel filter produced higher flux than the 0.5 micron stainless steel filter at the lower solids loading, and the same flux at the higher solids loading. Scanning electron microscope pictures show particles filling the pores of the 0.5 micron filter, and solid particles on the surface of the 0.1 micron filter.
- With the exception of one sample, all filtrate samples showed turbidity less than 5 NTU. The sample with high turbidity occurred during the test with the 0.1 micron stainless steel filter. We are uncertain of the cause.

INTRODUCTION

The processing rate of high level radioactive waste treatment processes at the Savannah River Site is limited by the flow rate of the solid-liquid separation process. The baseline filtration rate for the crossflow filter is 0.02 gpm/ft^2 under the anticipated operating conditions. If the SRS could identify and develop a solid-liquid separation technology with a higher filter processing rate, they could increase the throughput of these processes and complete treating these waste streams in a shorter time, with a significant reduction in life-cycle cost.

SRTC personnel identified the rotary microfilter as a technology that could increase filter flux, with improvements between 2 and 10X.[1,2,3] The rotary system combines centrifugation with membrane filtration. The system contains a group of flat disks with filter media on both sides. The feed slurry flows across the surface of the disks, and a pressure gradient forces the liquid through the filter. Solids are removed at the membrane surface, and the centrifugal force acts to keep the surface clean, minimizing the formation of a filter cake.

The rotary microfilter disks can be constructed with most commercially available filter media. Centrifugal filter systems are commercially available (Spintek, ASPECT USA, Pall, Canzler) and have been used in radioactive service both at Los Alamos National Laboratory (LANL)[4] (i.e., for Low-Level Waste) and in Russia (for High-Level Waste).

SRTC researchers tested the rotary microfilter as an alternative to the crossflow filters. Table 1 summarizes the results of scoping testing and Figures 1 and 2 show some of the data from the actual waste testing and pilot scale testing.[2,3] The data show significant improvement in filter flux with the rotary microfilter over the baseline crossflow filter (2.5 – 6.5 X during the scoping tests, up to 10 X in the actual waste tests, and approximately 2 X in the pilot-scale tests).

Table 1. Comparison of SpinTek Filter with Conventional Crossflow Filter from Vendor Scoping Tests

Solids (wt %)	Rotary (gpm/ft^2, measured)	Crossflow (gpm/ft^2, predicted)	Ratio
0.05	0.21	0.08	2.6
0.22	0.19	0.07	2.7
1.0	0.15	0.04	3.8
4.8	0.13	0.02	6.5

The SRTC received funding from the DOE-HQ, Office of Cleanup Technologies, via the NETL, to continue developing the rotary microfilter for SRS high level waste applications. This development includes evaluating the impact of radiation on the filter unit and the filter disks. The authors performed pilot-scale simulant filtration tests with irradiated filter disks. We conducted the tests with filter disks irradiated with an estimated 2.5 - 5 year (83- 165 MRad) radiation dose. The tests provide additional operating data, allow us to evaluate the impact of radiation on the rotary microfilter, and help us assess feasibility of using SpinTek's existing filter disk design in radioactive waste applications.

Figure 1. Rotary Microfilter Actual Waste Test Results[2]
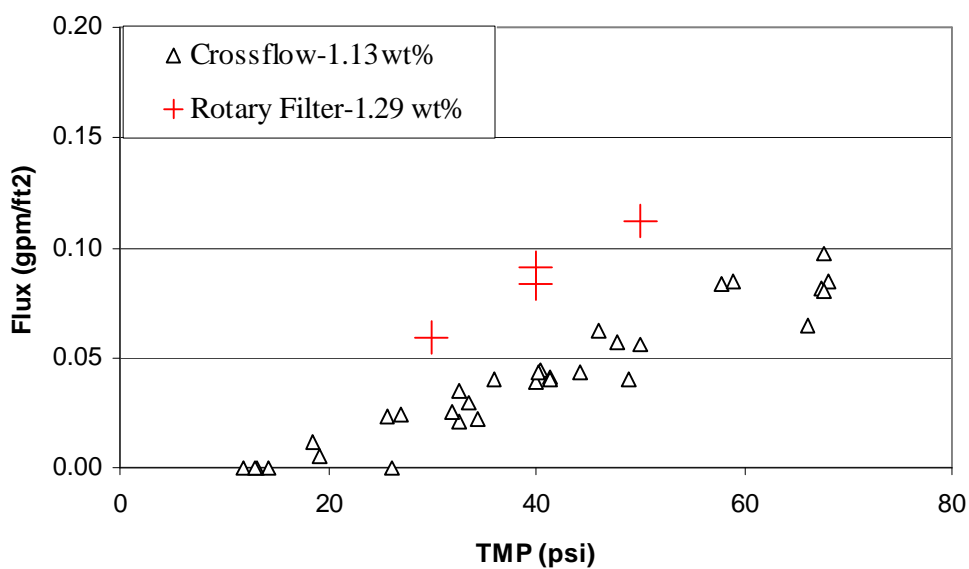

Figure 2. Rotary Microfilter Pilot-Scale Test Results[3]

TESTING

Equipment

The rotary microfilter unit currently installed at the University of South Carolina's Filtration Research Engineering Demonstration (FRED) is a SpinTek Model ST-II-3, Laboratory Test Unit with three membrane disks for a total of 3 ft^2 active membrane area (see Figure 3). The disks

spin inside a pressurized vessel with spoked turbulence promoters above and below each disk. Personnel can manually adjust the speed of the disk rotation between 500 and 1400 rpm. Increasing the rotational speed increases the shear forces at the surface of the disk. For the purpose of this test, we kept the disk rotational speed at 1170 ± 20 rpm, except where noted.

Figure 3. Pilot-Scale SpinTek Rotary Microfilter

A valve on the concentrate exit automatically controls the pressure inside the filter housing. This pressure provides the transmembrane pressure required to force filtrate through the filter membranes. For the purpose of this test, we controlled the pressure at 40 psi. Personnel added pressure sensors to the feed inlet and filtrate lines so they could collect data and calculate transmembrane pressures.

The feed slurry flows across the surface of the filter disks. A differential pressure drives the supernate through the filter membrane and into the center of the disks. The filtrate moves to the center of the disk and collects in the shaft holding the disks. The equipment provides no pressure control on the filtrate line, with only a solenoid valve to stop filtrate flow when desired. We measured filtrate flow with a magnetic flow meter.

Personnel manually controlled feed flow by adjusting the speed of the feed pump. We measured feed flow with a magnetic flow meter. For the purposes of this testing, we maintained feed flow between 3.8 and 4.2 gpm.

The feed tank has a working capacity of 115 L. The agitator in the feed tank operates at a variable speed with a single marine blade. The feed tank includes a sensor for the Lasentec® particle size analyzer.

We provided automatic temperature control for the system with a heat exchanger located on the line from the feed pump to the filter housing. Personnel supplied cooling water from a remote source and maintained the temperature with the control valve on the skid.

Materials of construction for the unit are all corrosion resistant (i.e. stainless steel, Teflon™, etc.)

The onboard Programmable Logic Controller (PLC) performs automatic control with data passed to the facility Data Control System for logging.

Test Protocol

Personnel conducted the pilot-scale simulant rotary microfilter tests as follows. We selected three types of filter disks for these tests (0.5 micron stainless steel, 0.1 micron stainless steel, and 0.1 micron ceramic/stainless steel). The ceramic/stainless steel filter contains an asymmetric ceramic membrane and a stainless steel support structure. All filters incorporated the stainless steel permeate carrier mesh. Personnel analyzed the filter structural material, Ryton[®], for hardness, and irradiated the entire disk in a Co-60 source. We also measured the hardness of the Ryton[®] after the irradiation of the disk to determine if a change in material properties occurred. The stainless steel filters received an estimated 5-year radiation dose, and the ceramic filters received a 2.5 year radiation dose. Following the irradiation, USC personnel placed the filters in the pilot-scale rotary microfilter unit and tested them with feed slurries containing 0.29 and 4.5 wt % solids. We tested each set of filter disks for at least one week. These loadings allow comparison with previous crossflow filter test data.[2,3,5]

Personnel conducted the tests with sludge plus MST slurry (sludge to MST ratio of 1.1). They prepared the feed from previously used test slurries. We selected these slurries to match the solids used in previous crossflow filter tests.[3]

We prepared the sludge plus MST slurry in the following manner. Personnel decanted the supernate (i.e., 5.6 M sodium, "average" salt solution) from drums containing sludge and MST. We analyzed the supernate for insoluble solids to determine the mass of solids needed to achieve the target concentrations. We analyzed settled solids from the drums for insoluble solids concentration and added them to the feed tank to achieve the target solids loading.

Once the feed tank contained the desired slurry (nominal 0.29 wt % insoluble solids), personnel started the rotary microfilter and circulated the feed. They set the transmembrane pressure to 40 psi. After a pre-determined operating time, personnel increased the solids loading to nominal 4.5 wt % and operated the unit with the new feed slurry.

RESULTS

Filter Flux

Figure 4 shows the filter flux plotted as a function of time during the test. Personnel performed the first test with 0.1 micron stainless steel filter disks irradiated with 165 MRad (estimated 5 year dose). The initial feed slurry contained 0.29 wt % insoluble solids. They operated the

rotary microfilter for 60 hours. The flux after 60 hours equaled 0.060 gpm/ft^2. Personnel then increased the solids loading to 4.5 wt % and operated the filter for an additional 88 hours. After 88 hours, the filter flux equaled 0.026 gpm/ft^2.
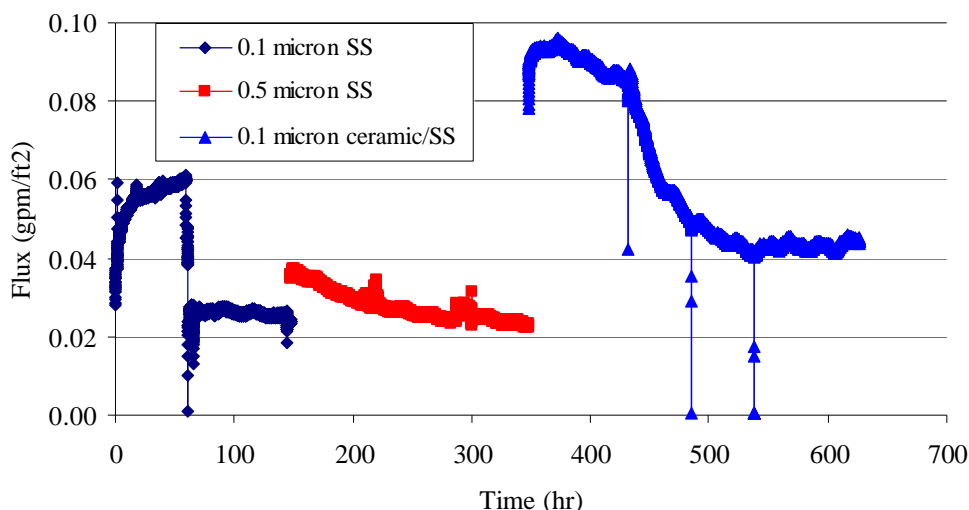

Figure 4. Rotary Microfilter Performance with Irradiated Filter Disks

Personnel flushed the filter unit with 0.01 M NaOH, removed the 0.1 micron filter disks, and installed 0.5 micron stainless steel filter disks irradiated with 165 MRad (estimated 5 year dose). The initial feed slurry contained 0.29 wt % insoluble solids. They operated the rotary microfilter for 152 hours. The flux after 152 hours measured 0.027 gpm/ft^2. Personnel then increased the solids loading to 4.5 wt % and operated the filter for an additional 48 hours. After 48 hours, the filter flux reached 0.023 gpm/ft^2.

Personnel flushed the filter unit with 0.01 M NaOH, removed the 0.5 micron filter disks, and installed 0.1 micron ceramic/stainless steel filter disks that had been irradiated with 83 MRad (estimated 2.5 year dose). The initial feed slurry contained 0.29 wt % insoluble solids. They operated the rotary microfilter for 84 hours. The flux after 84 hours equaled 0.085 gpm/ft^2. Personnel then increased the solids loading to 4.5 wt % and operated the filter for an additional 194 hours. After 126 hours, the filter flux measured 0.042 gpm/ft^2.

Figure 5 compares filter flux of the irradiated 0.1 micron stainless steel filter with the flux of an unirradiated filter from a previous test.[3] With 0.1 micron stainless steel irradiated filter disks, filter flux measured 0.06 gpm/ft^2 at 0.29 wt % solids compared to 0.10 gpm/ft^2 with unirradiated filter disks. At 4.5 wt % solids, the filter flux measured 0.026 gpm/ft^2 using irradiated filter disks compared with 0.04 gpm/ft^2 using unirradiated filter disks. The flux with the irradiated filter disks proved 35 – 40% lower than the flux measured with unirradiated filter disks.
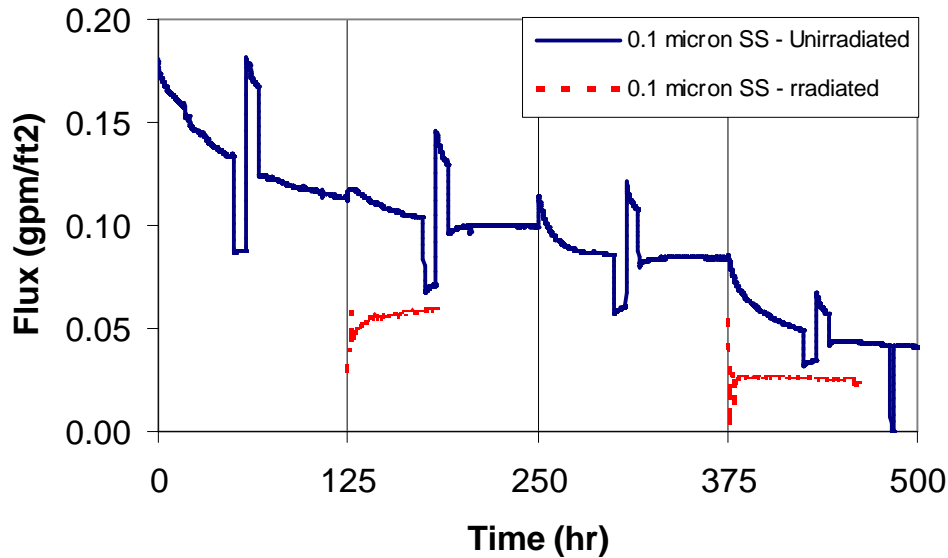
Figure 5. Comparison of Filter Flux between Irradiated and Unirradiated Filter Disks

This result is surprising, because radiation should not affect the stainless steel filter media. A likely cause of the difference is that personnel did not thoroughly clean the filter disks following the 2002 rotary microfilter test.[3] The sludge and manganese dioxide solids that adhered to the filter in that test likely dried and set in the filter. Presence of these solids may have lowered the flux in the more recent tests.

Another plausible explanation for the difference in flux between the irradiated and unirradiated filters is differences in particle size distribution for the two feeds. We will discuss this hypothesis later.

Figure 6 compares the flux measured with the different filter media. The 0.1 micron ceramic/stainless steel filter produced the highest flux at both solids loadings. The 0.1 micron stainless steel filter produced higher flux than the 0.5 micron stainless steel filter at the lower solids loading, and the same flux at the higher solids loading. The result is consistent with previous crossflow filter test results with this feed that showed a 0.1 micron stainless steel filter produces higher flux that a 0.5 micron stainless steel filter.
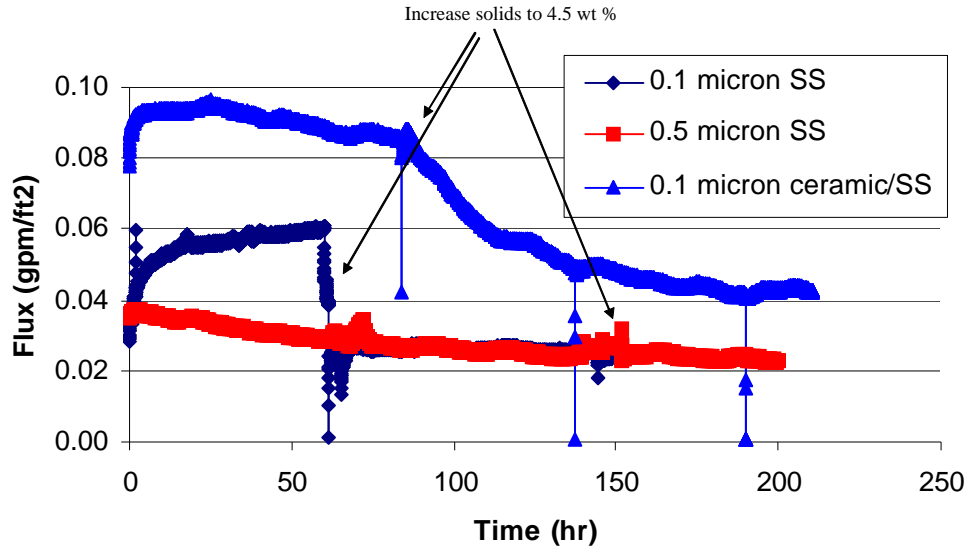
Figure 6. Comparison of Filter Media

Figure 7 shows Scanning Electron Microscope (SEM) pictures of the "new" 0.5 micron stainless steel (SS) filter, the "fouled" 0.5 micron SS filter, and the "fouled" 0.1 micron SS filter. The "new" 0.5 micron SS filter shows a very open pore structure. After testing, the 0.5 micron SS filter SEM picture shows a less open structure with many particles filling the pores. The SEM picture of the 0.1 micron SS filter after testing shows solid particles located on the filter surface. If the particles form a filter cake rather than accumulating in the filter pores, the resistance will be lower and filter flux higher.
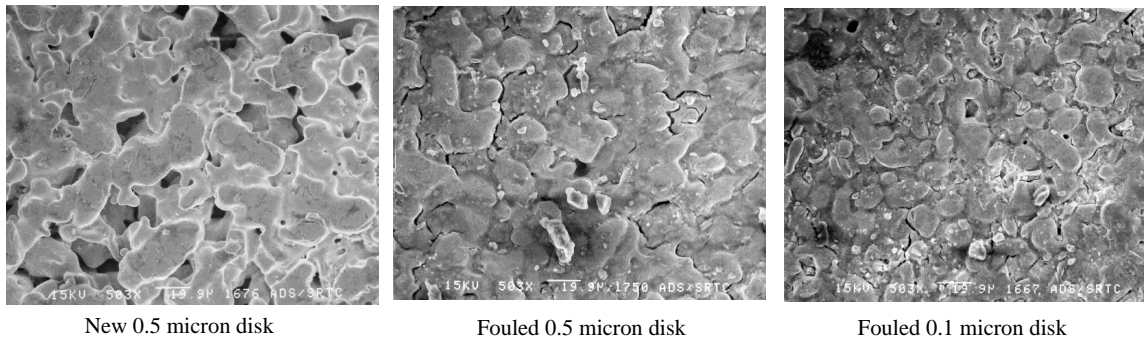

New 0.5 micron disk   Fouled 0.5 micron disk   Fouled 0.1 micron disk

Figure 7. SEM Photos of Stainless Steel Filter Media

Particle Size Data

Personnel collected particle measurements with a Focused Beam Reflectance Measurement (FBRM) probe (Lasentec®). The probe works in the following manner. Personnel installed the probe in the feed tank. The laser beam projects through the window of the FBRM probe and focuses just outside the window surface. This focused beam follows a path around the circumference of the probe window. As particles pass by the window surface, the focused beam will intersect the edge of a particle. The particle will backscatter laser light. The particle will

continue to backscatter the light until the focused beam reaches the opposite edge of the particle. The instrument collects the backscattered light and converts it into an electronic signal.

The FBRM isolates the time of backscatter from one edge of an individual particle to its opposite edge. The software records the product of the time multiplied by the scan speed as a chord length. A chord length is a straight line between any two points on the edge of a particle or particle structure (agglomerate). FBRM typically measures tens of thousands of chords per second, resulting in a robust number-by-chord-length distribution.

The chord-length distribution provides a means of tracking changes in both particle dimension and particle population. The calculations do not assume a particle shape. The chord-length distribution is essentially unique for any given particle size and shape distribution. Assuming the average particle shape remains constant over millions of particles, changes to the chord-length distribution reflect solely a function of the change in particle dimension and particle number.

Figure 8 shows the particle size distribution during the test with the 0.1 micron stainless steel filter. The initial 0.29 wt % feed slurry had a median particle size of 13 micron. Following 60 hours of filtration, the median particle size decreased to 12 micron. The figure shows a reduction in the number of large particles, which is likely due to the shearing caused by the filter feed pump. Reducing the rotor speed from 1170 rpm to 600 rpm did not impact the particle size. The curves for those data sets overlap. The median particle size of the initial 4.5 wt % slurry equals 9 micron, which is less than the median particle size of the 0.29 wt % slurry. After 88 hours of filtration, the median particle size increased to 10 micron.
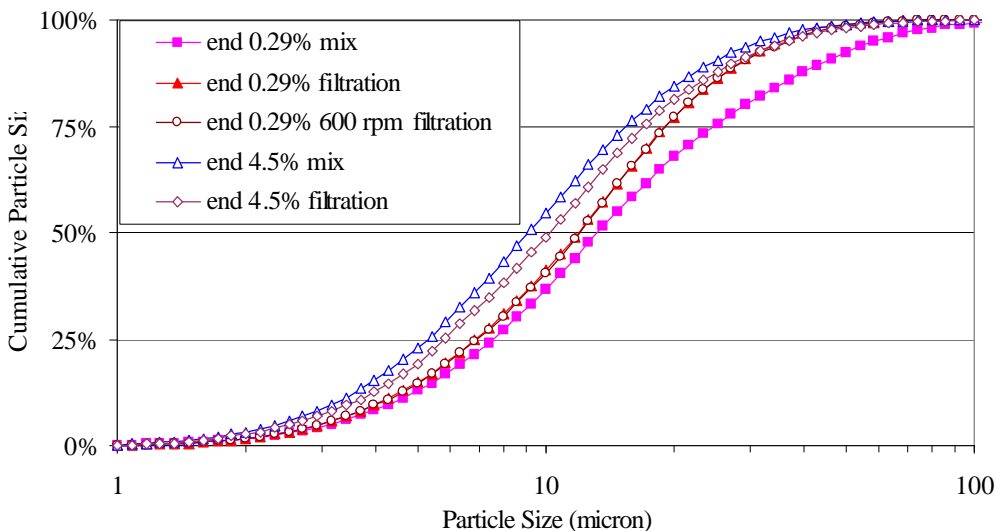

Figure 8. Particle size data from the SpinTek Test with 0.1 Micron SS Filter

Figure 9 shows the particle size distribution during the test with the 0.5 micron stainless steel filter. The initial 0.29 wt % feed slurry had a median particle size of 10 micron. Following 151 hours of filtration, the median particle size increased to 13 micron. The figure shows no significant change in the fraction of large particles. The median particle size of the initial

4.5 wt % slurry equals 9 micron, which is less than the median particle size of the 0.29 wt % slurry. After 49 hours of filtration, the median particle size increased to 10 micron.

Figure 10 shows the particle size distribution during the test with the 0.1 micron ceramic/ stainless steel filter. The initial 0.29 wt % feed slurry had a median particle size of 15 micron. Following 84 hours of filtration, the median particle size decreased to 14 micron. The median particle size of the 4.5 wt % slurry equals 10 micron, which is less than the median particle size of the 0.29 wt % slurry. After 194 hours of filtration, the median particle size remained 10 micron.
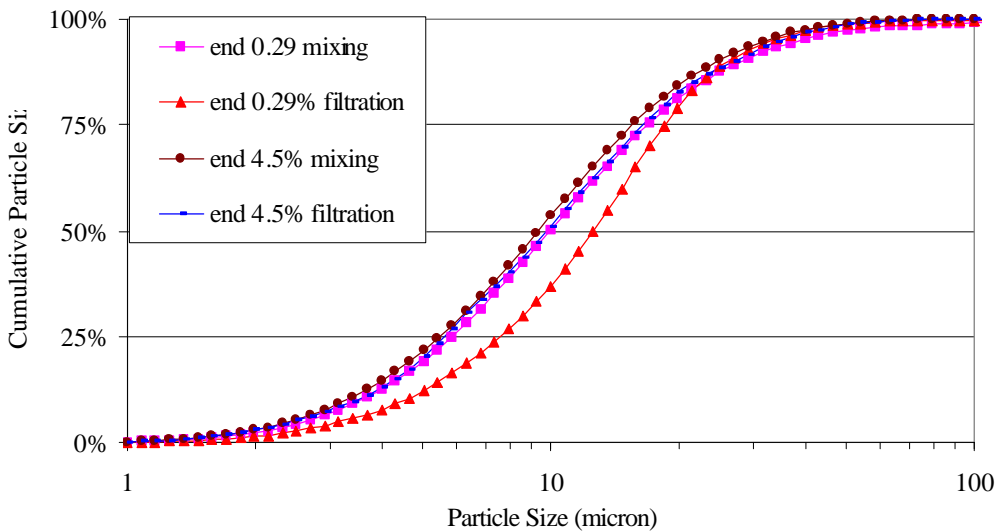

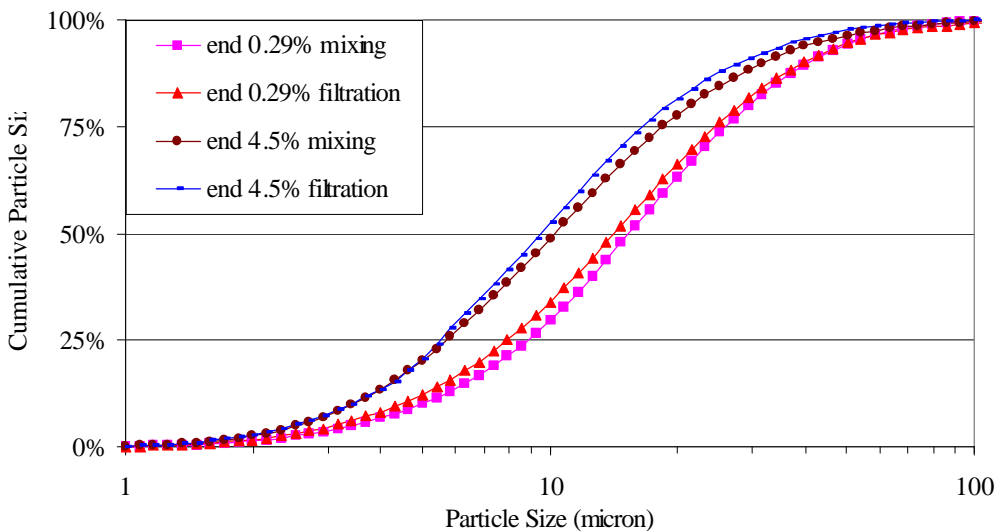**Figure 9. Particle size data from the SpinTek Test with 0.5 Micron SS Filter**

Figure 10. Particle size data from the SpinTek Test with 0.1 Micron Ceramic/SS Filter

Figure 11 shows the particles size distribution of the feed slurry following the tests with 0.1 micron stainless steel filter disks. One test used disks irradiated with 165 MRad, and the other

test used unirradiated disks.[3] The median particle size in the test with the irradiated filter disks
equals 10 micron compared with 17 micron in the test with unirradiated filter disks. This
difference in particle size could explain the difference in flux observed in the two tests with the
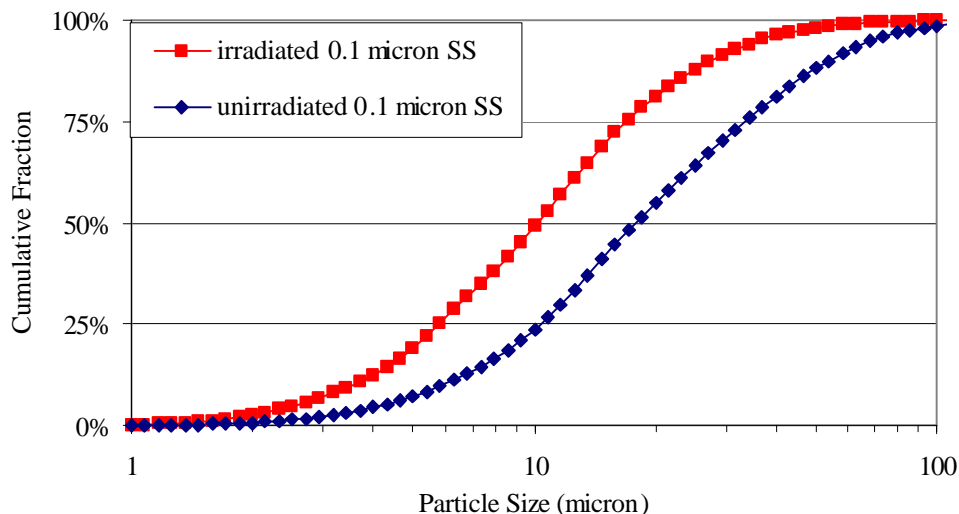same filter media.

Figure 11. Particle Size Following Tests with 0.1 Micron Filter Disks

Table 2 and Figure 12 compare the particle size distributions of the initial feed slurries
(0.29 wt % solids) used in each of the filter tests. The feed slurry in the 0.5 micron stainless steel
filter test had the smallest median particle size, and the feed slurry in the 0.1 micron
ceramic/stainless steel filter test had the largest median particle size. Coincidentally, the 0.1
micron ceramic/stainless steel filter achieved the highest flux, and the 0.5 micron stainless steel
filter produced the lowest flux.

Table 2. Median Particle Size

Filter	Insoluble Solids (wt %)	Time (start/finish)	Median Particle Size (micron)
0.1 SS	0.29	Start	13
0.1 SS	0.29	Finish	12
0.1 SS	4.5	Start	9
0.1 SS	4.5	Finish	10
0.5 SS	0.29	Start	10
0.5 SS	0.29	Finish	13
0.5 SS	4.5	Start	9
0.5 SS	4.5	Finish	10
0.1 ceramic	0.29	Start	15
0.1 ceramic	0.29	Finish	14
0.1 ceramic	4.5	Start	10
0.1 ceramic	4.5	Finish	10
0.1 SS-unirrad.	0.06	Start	17
0.1 SS-unirrad.	4.5	Finish	12

Figure 13 compares the particle size distributions of the 0.29 wt % feed slurries following
filtration. The feed slurries in the 0.5 micron and 0.1 micron stainless steel filter tests had
approximately the same median particle distribution. The feed slurry in the 0.1 micron

ceramic/stainless steel test had a slightly larger median particle size and a larger fraction of large
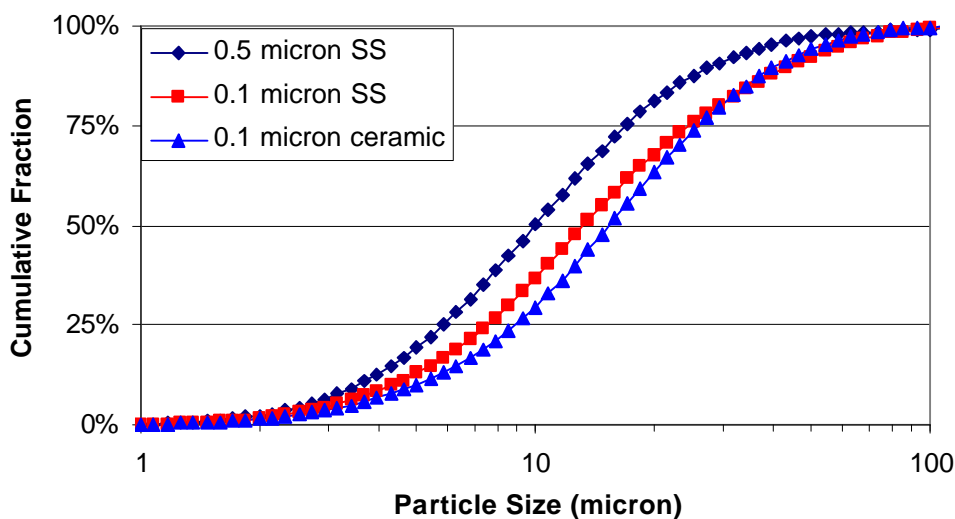particles.

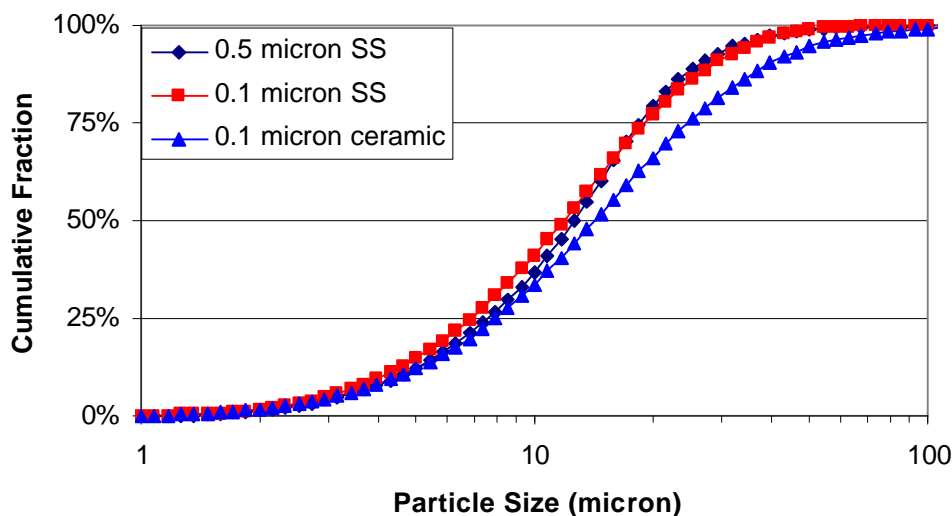**Figure 12. Comparison of Initial Feed Slurries for Filter Tests**

Figure 13. Comparison of 0.29 wt % Feed Slurries Following Filtration

Figure 14 compares the particle size distributions of the feed slurries (4.5 wt % solids) at the
conclusion of each of the filter tests. The distributions are essentially the same. Coincidentally,
at the conclusion of the tests, the 0.1 micron and 0.5 micron stainless steel filters produced
approximately the same flux (0.026 gpm/ft^2 versus 0.023 gpm/ft^2). The ceramic/stainless steel
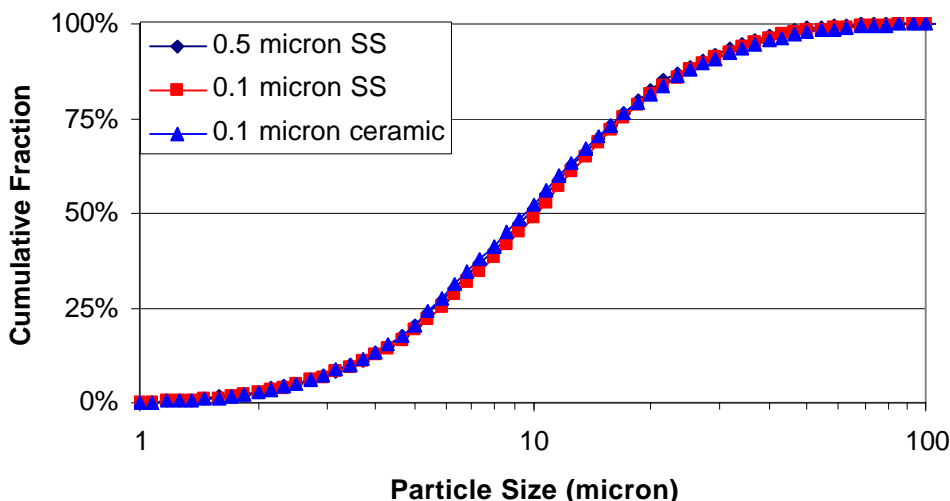filter produced a higher flux (0.042 gpm/ft^2).

Figure 14. Comparison of Feed Slurries at Conclusion of Filter Tests

Figures 12 – 14 show that the initial feed in the tests with the 0.5 micron stainless steel filter had a smaller median particle size than the initial feed in the tests with the 0.1 micron stainless steel filter. A smaller median particle size produces a filter cake with higher resistance, which reduces filter flux. However, by the end of the tests, the particle size distributions appeared the same for the three tests. After reviewing this data, as well as the scanning electron microscope data, we believe the reason for the lower flux with the 0.5 micron stainless steel filter is its larger pores allowed fine particles to enter the pores where they became trapped.

Reliability

During the testing, personnel collected filtrate samples periodically and analyzed them for turbidity (target < 5 NTU). During testing with the 0.1 micron SS disk, one sample showed high turbidity (> 200 NTU). Subsequent samples, collected every six hours, showed turbidity less than 5 NTU. We are uncertain of the cause of the high turbidity, but it did not persist through the test. All filtrate samples from tests with the 0.5 micron SS filters and the 0.1 micron ceramic filters showed turbidity less than 5 NTU.

Results indicate the filter continued to perform satisfactorily, though evidence of degradation is observed on some of the disks after a 5-year equivalent radiation dose. Therefore, the 2-year lifetime assumed in the risk assessment is conservative. However, the irradiation occurred in air and does not include the effect of irradiation in a caustic environment.

None of the nine disks tested experienced a catastrophic failure, but some evidence of delamination exists. Separation started to occur between the epoxy and the base Ryton® material. Personnel sectioned the disks and observed that the joint remained sealed. Figure 15 illustrates the damage to the disk.

We tested the hardness of the Ryton® support plate as received and after exposure in the cobalt source. Hardness measurements used the Rockwell B scale with a 100 kg load and 1/16" indenter. A representative test block occurred after each set of tests and all tested within range.

Figure 15. Delamination of Filter Disk

Table 3 shows the measurements and average hardness of the exposed Ryton®. The results show no pattern to the measurements. This result indicates that the damage to the Ryton® is on the surface of the Ryton® plate whereas the hardness measurement is a bulk property.

Table 3. Hardness Measurements of Ryton® Using Rockwell B Scale

Disk	As Received	8.25E8 Rad Equivalent Dose	1.65E8 Rad Equivalent Dose
0.1 SS-1	8.7	9.2	7.8
0.1 SS-2	9.7	8.8	8.3
0.1 SS-3	9.0	6.3	6.2
Average	9.1 ± 0.5	8.1 ± 1.6	7.4 ± 1.1
CER-1	4.7	8.7	NA
CER-2	6.8	10.2	NA
CER-3	7.7	7.7	NA
Average	6.4 ± 1.5	8.9 ± 1.3	NA

An FTIR analysis of the Ryton® (polyphenylene sulfide) exposed to the five year equivalent dose showed the Ryton® degrading, weakening the bond between the Ryton® and the epoxy. Comparisons of the disk material before and after irradiation from the FTIR analysis are shown as Figure 16. The unirradiated sample shows carbon-hydrogen stretches and phenyl groups,

which are indicative of polyphenylene sulfide. Following irradiation, the sample shows an O-H stretch, an asymmetric O=S-OH stretch, and a symmetric O=S-OH stretch. These peaks indicate that the phenyl ring groups are destroyed (note the dissapearance of the phenyl ring bands) while new sulfonic acid groups (oxidation and hydration of the sulfur atoms) are created on the surface.
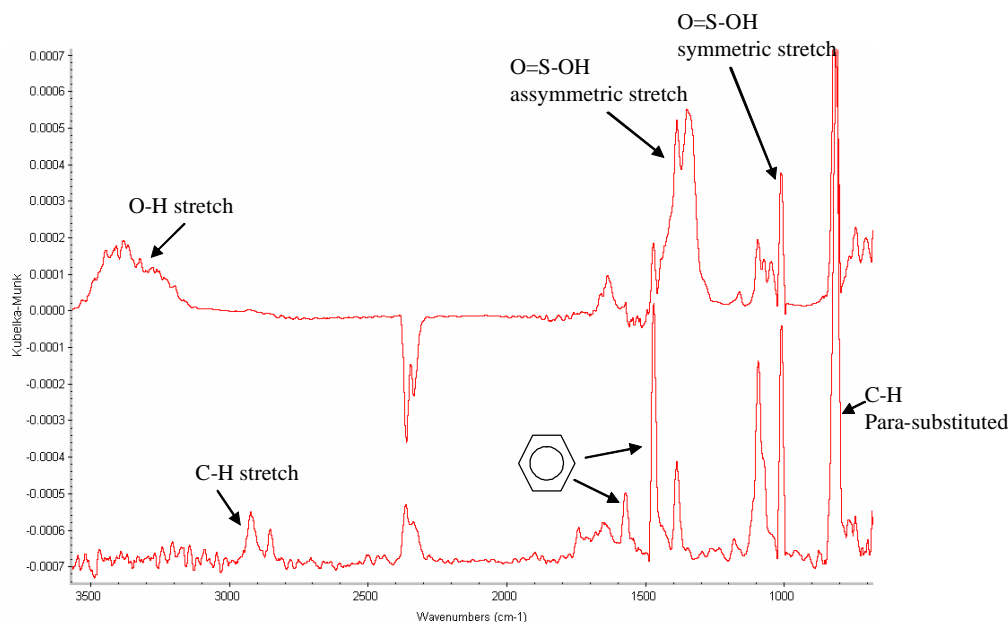

Figure 16. FTIR Analysis of Ryton

It should be again noted that irradiation of the disks occurred in air and not caustic. The caustic could potentially accelerate the degradation of the disks due to radiation damage. A five year life is anticipated for the off the shelf vendor filter disks, and the two year life assumed in the risk assessment and cost/benefit analysis is probably conservative.

CONCLUSIONS

The conclusions from this work follow.
- None of the nine disks tested experienced a catastrophic failure from radiation exposure, but some evidence of delamination existed. Separation started to occur between the epoxy and the base Ryton® material.
- The flux with the irradiated filter disks was 35 – 40% lower than the flux measured with unirradiated filter disks. A likely cause of this difference is the feed to the irradiated filter disks had a smaller particle size than the feed to the unirradiated filter disks. Another plausible cause is that personnel did not thoroughy clean the filter disks following the 2002 test.
- The 0.1 micron ceramic/stainless steel filter produced the highest flux.
- The 0.1 micron stainless steel filter produced higher flux than the 0.5 micron stainless steel filter at the lower solids loading, and the same flux at the higher solids loading. Scanning electron microscope pictures show particles filling the pores of the 0.5 micron filter, and solid particles on the surface of the 0.1 micron filter.

- With the exception of one sample, all filtrate samples showed turbidity less than 5 NTU. The sample with high turbidity occurred during the test with the 0.1 micron stainless steel filter. We are uncertain of the cause.

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