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Pilot-scale cross-flow filtration test for a radioactive waste simulant with high solids loading and sodium molarity[†]

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Abstract

BNFL Inc. was contracted by the Department of Energy to design a facility to stabilize liquid radioactive wastes that are stored at the Hanford Site in the state of Washington. Because of its experience with radioactive waste stabilization the Savannah River Technology Center (SRTC) of the Westinghouse Savannah River Company worked with BNFL to help design and test certain parts of the waste treatment facility. One part is the separation of the highly radioactive solids from the liquid wastes by cross-flow filtration.

This paper discusses the results of a cross-flow filter in a pilot-scale experimental facility that was designed, built, and run by the Experimental Thermal Fluids Laboratory of SRTC. To test this filter a waste simulant was developed to prototypically represent the chemical and physical characteristics of one radioactive waste stream. The insoluble solids loading of the simulant was varied from 0.5 wt% to 8 wt%, the aqueous solution was high in sodium and free hydroxide with total solids loadings up to 36 wt%, and the particle sizes ranged from 0.5 to 5 microns in diameter. The simulant matched the waste with a pH > 14, and molarities of sodium, nitrates, and nitrites, of approximately 5.5 M, 1.4 M, and 1.2 M respectively. The cross-flow filter unit was made of seven 316L stainless steel sintered-metal tubes in parallel. Each tube was identical, with a porous length of 1.01 meter, 9.5 mm inside diameter, 12.7 mm outside diameter, and a total filter area of 0.211 m². The manufactured pore-size rating was 0.1 micron.

The flow conditions for the test varied to match the expected operation of the waste processing plant. That is, the axial slurry velocities varied from 1 m/s to 4.6 m/s and the transmembrane pressures (TMP) varied from 120 kPa to 480 kPa at a temperature of 298 K. The experimental results showed good permeability and separation. Under the optimum flow conditions of a slurry velocity of 3.8 m/s and a TMP of 300 kPa, the Filtrate Flux was above 24 cm/hour; better than the plant design need of 9 cm/hour.

Keywords: Filtration; Cross-flow; Radioactive; Hanford, Pre-treatment

[†] This work was carried out at the Savannah River Technology Center, managed by the Westinghouse Savannah River Company for the U.S. Department of Energy under contract DE-AC09-96SR18500

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1. Introduction

One important part of a plant to process radioactive slurry waste is the separation of the waste into its various constituents for more efficient handling. A first step in radioactive waste treatment is to separate most of the liquid from the solids, since it is the solids that make up the majority of the radioactive contamination. There are a large number of technologies available to choose from, but when solids need to be separated in a continuous mode there is a limited number. One way to separate is to move the liquid while retaining the solids on a permeable barrier. This method of separation is called filtration. The most widely used form of filtration is referred to as dead-end, where all the liquid of a slurry must flow through the collecting solids and then through the underlying permeable barrier. The obvious disadvantage to such a process is that the build up of collecting solids will eventually cause the flow to stop; the barrier must either be cleaned, or replaced to continue the separation process.

Another form of filtration is called cross-flow, where the turbulent flow (Fritz and Pahl, 1996) of slurry runs parallel to the permeable surface, which helps to keep the surface clean by continually shearing the solids away from the permeable barrier (Murkes and Carlsson, 1988). If the main separation objective is to concentrate a slurry by the separation of some of its supernatant, then, cross-flow filtration is an appropriate technology and is economical for certain industries (Roeleveld and Maaskant, 1999). However, cross-flow filtration is not generally the most economical means of filtration but there may be very limited options when there is no easy access to replace the filter, like when it is used in a radioactive environment. Even, when a cross-flow filtration system is designed for replacement in a radioactive environment (Gutman et al., 1989) it is always desired to minimize the exposure of personnel.

Besides access, cross-flow filtration is preferable to dead-end filtration when the slurry to be filtered can easily plug, like those that contain gums (Carrère et al., 1998), colloidal solids (Sundaram and Santo, 1977), or other non-Newtonian flows (Carrère and René, 1998). Since the mechanism of cross-flow filtration tends to keep the accumulation of solids on the permeable barrier to a minimum, it will generally have less maintenance than the dead-end filter. Unfortunately, to date predicting the cross-flow filtration parameters for easily plugging slurries is still years away and must be studied through experimentation. For simple slurries (e.g., sand and water) there exist today a certain level of sophisticated analyses that can be used to predict cross-flow filtration parameters (Song, 1998; Fritz and Pahl, 1996; Lu, Hwang, and Ju, 1993; Stamatakis and Tien, 1993; Murkes and Carlsson, 1988). Such analyses that make simplifying assumptions about particle types, cake compressibility, cake uniformity, etc. do not apply to slurries that may change in character with time or flow conditions. However, with better observational techniques (Li, et al., 1998) a better understanding of these challenging slurries will emerge.

BNFL, Inc. (BNFL) was contracted by the Department of Energy (DOE) to design a facility to stabilize liquid radioactive waste that is stored at the Hanford Site. Because of its experience with radioactive waste stabilization, the Savannah River Technology Center (SRTC) of the Westinghouse Savannah River Company is working with BNFL to help

design and test certain parts of the waste treatment facility. One part of the process is the separation of radioactive solids from the liquid wastes high in sodium by cross-flow filtration. However, because of the expected plugging characteristics of the waste a cross-flow filter was tested with a simulated radioactive waste, made to prototypically represent the waste chemical and physical characteristics, while not being radioactive.

The goal of this work is to show that chosen cross-flow filter can filter better than plant need of 9 cm/hour with a radioactive waste simulant which has a sodium molarity of approximately 5.5M, a pH ~ 14, an insoluble solids concentration as high as 8 wt%, and viscosity of up to 3 cP. It is also the hope of the authors that this information will be a useful addition to the growing body of data on hard-to-filter solutions.

2. Experimental

2.1. Test rig

Figure 1 is a schematic of the entire test rig. It stood approximately 7.6 meters in height and was serviced by a two-level mezzanine. The test rig was much taller than the meter-tall filter element because it originally was used to test a taller filter. The entire rig was made of 300 series stainless steel with the majority being of 304 stainless steel.

The test rig was made up of three basic flow loops:

1. Slurry loop, which contains the filter and its housing and serves as the primary flow path for circulating slurries. This loop had an internal volume of approximately 20 liters, excluding the reservoir tank.
2. Filtrate loop, which begins at the filter housing and allows the separated filtrate liquid to flow up through the backpulse piston before returning to the top of the slurry loop to close the circuit. This loop had an internal volume of approximately 6 liters.
3. Cleaning loop, which enables cleaning of the filter in place without having to remove the slurry from the test rig by correctly orienting two 3-way valves. This loop had an internal volume of approximately 15 liters.

Two other flow circuits that are subsections of the other loops are the backpulse and the recirculations loops:

1. The backpulse loop was part of the filtrate loop and stood ready to reverse the flow of filtrate. A pulse forces filtrate (0.06 liters) back through the seven filter elements in order to knock off built-up slurry cake on the inside diameter of the porous tubes.
2. The recirculation loop was part of the slurry loop. This loop is used to: better control the slurry flow, increase mixing, and keeps the slurry well-mixed when the flow through the filter needs to be stopped.

Three 3 hp pumps were used to attain the head of 550 kPa at slurry flow of 150 lpm.

2.2. Cross-flow filter

The heart of this experiment was the cross-flow filter unit. The unit was made by the Mott Metallurgical Corporation from 316L stainless steel (sintered metal), had a pore size

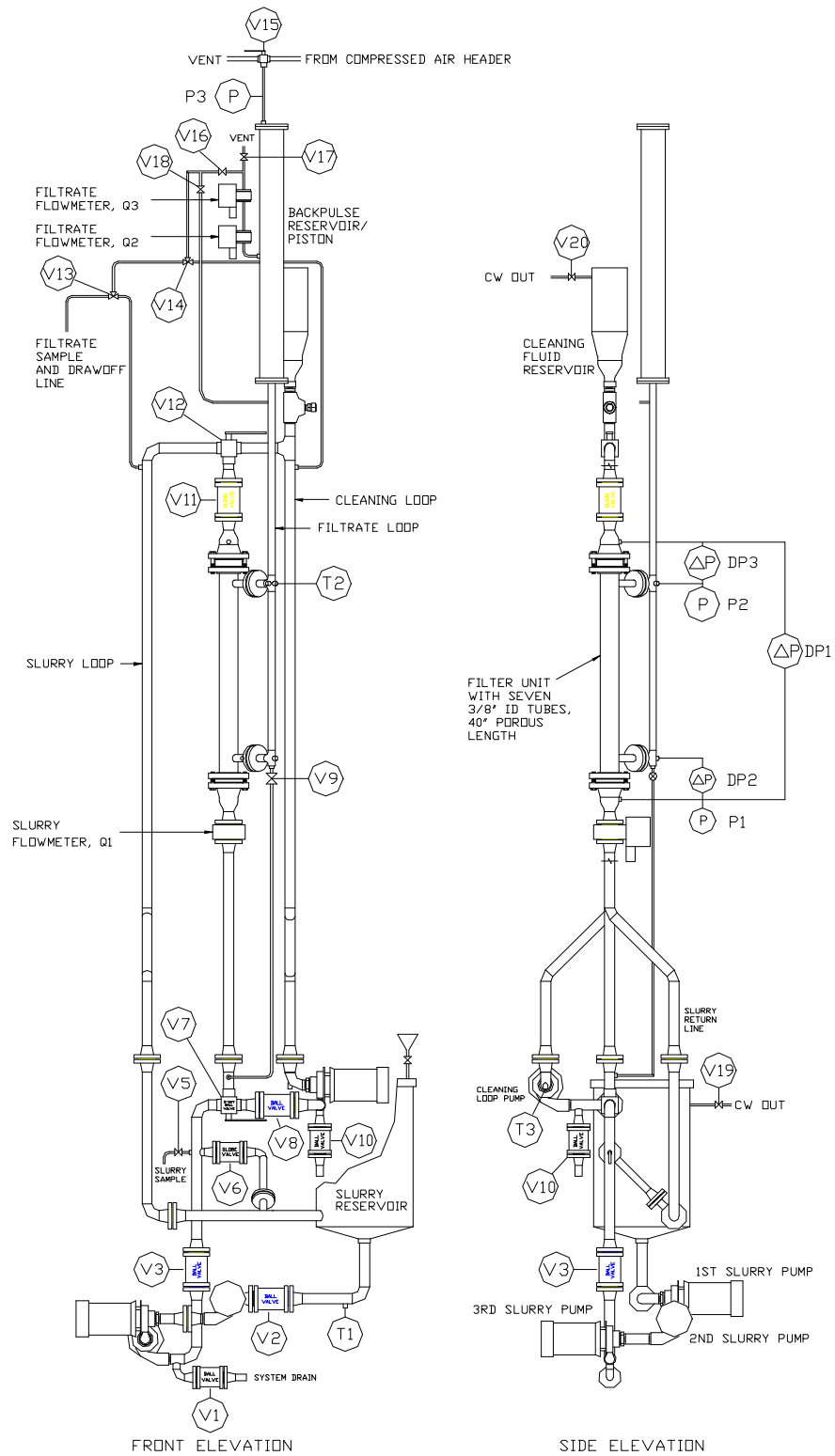


Figure 1. Schematic of the pilot-scale cross flow filtration test facility.

nominal[†] rating of 0.1 micron, had a porous length of 1.01 meter, and each of the seven porous tubes had an inside diameter of 9.5 mm and an outside diameter of 12.7 mm. The filter-tube bundle is shown in Figs. 2 and 3, and the bundle was housed inside of an 83 mm inside diameter pipe (3-inch schedule 10 pipe) as shown in Fig. 1. Besides the number of tubes, this filter unit was prototypic of the planned plant filtration unit at the time of the test.



Figure 2. 7-tube bundle and each tube had: 9.5-mm i.d., 12.7-mm o.d., 1.01-meter long.

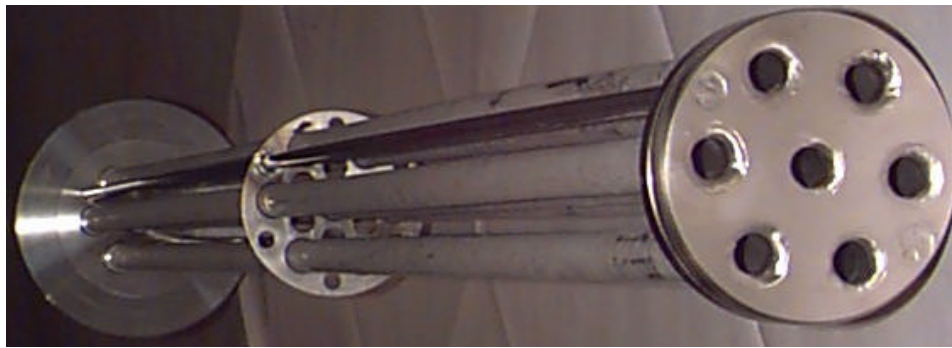


Figure 3. Upstream view of the seven-tube bundle

2.3 Instrumentation and measurement uncertainty

The measurement equipment used for this experiment was:

- Five type-E thermocouples with measurement uncertainties[‡] from 1.0 to 1.5 K
- Six variable-capacitance pressure transducers with measurement uncertainties[‡] from 0.2 to 2.6 kPa,
- Three magnetic flow meters with measurement uncertainties[‡] from 0.02 to 0.72 lpm

The measurement uncertainties (at a 95% confidence level) for the calculated quantities are:

Slurry Velocity in a Filter Tube	=	V	± 8.2 %
Transmembrane Pressure	=	TMP	± 1.1 %
Filtrate Flux	=		± 6.7 %

[†] The word “nominal” for a filter is a vague term because its meaning is manufacturer dependent. Further, a “nominal” rating does not give an exact size to a filter medium; but rather an approximation to the expected performance of a filter. In the case of Mott, a nominal rated 0.1-micron filter means that approximately 95% of particles greater than 0.1 micron will not pass the filter. However, this measurement is verified with a bubble-point test that is done to the ASTM Standard F316-80.

[‡] The measurement uncertainties are a function of the instrument and calibration. The uncertainty introduced through the use of the 16-bit data acquisition system was insignificant (<0.1% reading) and was not included in the values above. A complete analysis can be found in Duignan, 2000.

2.4. Simulated Waste Slurry

The simulant used for this task was cold (non-radioactive), but made chemically equivalent to the actual waste. The recipe for the simulated supernatant of the slurry is shown in, Fig. 4, and the simulant solids are shown in Fig. 5.

Compounds	Formula	Conc., PPM	M
Boric Acid	H3BO3	126	2.43E-03
Cesium Nitrate	CsNO3	10	6.27E-05
Potassium Nitrate	KNO3	8302	9.77E-02
Glycolic Acid	HOCH2COOH, 70 wt%	719	7.88E-03
Sodium Chloride	NaCl	6472	1.32E-01
Sodium Fluoride	NaF	181	5.14E-03
Potassium Molybdate	K2MoO4	88	4.39E-04
Ammonium Acetate	CH3COONH4	221	3.42E-03
Aluminum Trihydroxide	Al(OH)3	49574	7.56E-01
Sodium Hydroxide	NaOH	84956	2.53E+00
Sodium meta-silicate	Na2SiO3.9H2O	922	3.86E-03
Sodium Acetate	NaCH3COO.3H2O	1670	1.46E-02
Sodium Formate	HCOONa	1879	3.29E-02
Sodium Phosphate	Na3PO4.12H2O	985	3.09E-03
Sodium Carbonate	Na2CO3	9567	1.07E-01
Sodium Nitrate	NaNO3	90607	1.27E+00
Sodium Nitrite	NaNO2	71911	1.24E+00

Entrained Solids

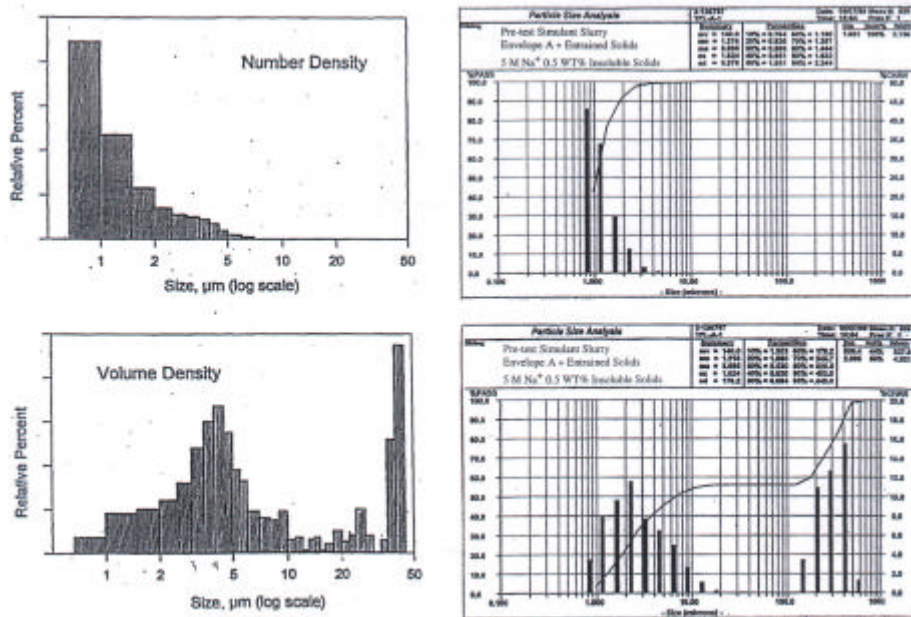
Compound* Name	Compound Formula	Concentration g/100g solids
Alumina	Al2O3	9.2%
Calcium Oxalate	CaC2O4	5.0%
Chromium Oxide	Cr2O3	26.0%
Ferric Oxide	Fe2O3	1.1%
Manganese Oxide	MnO2	0.3%
Sodium Oxalate	Na2C2O4	52.4%
Nickel Oxide	NiO	0.5%
Silicon Oxide	SiO2	5.4%
Total		100.0%

* All particles sized between 0.5 and 5 microns

Figure 4. Simulant: supernate only[†].

Figure 5. Simulant: Solids Only[†].

Besides matching the chemistry of the actual waste for filtration, it was important to closely match the insoluble solids in size and quantity. Obtaining a perfect match was impossible but the simulant had a similar solids distribution as shown in Fig. 6.



(a) Solids Distribution in Actual Waste Sample

(b) Solids Distribution in Waste Simulant

Figure 6. Insoluble solids distribution.

[†] This simulant represents the waste that is being stored in the Department of Energy Hanford Site tank number 241-AN-105 (Herting, 1997; Herting, 1999; Eibling and Nash, 2000).

In general, the simulant properties at 298 K were:

Slurry Density: ~1.23 g/mL at 0.5 wt% insoluble solids and 30 wt% total solids
 Slurry Density: ~1.28 g/mL at 8 wt% insoluble solids and 36 wt% total solids
 Slurry Viscosity: ~3 cP at 0.5 wt% insoluble solids and 30 wt% total solids
 Slurry Rheology ~Newtonian (at high insoluble solids loadings Bingham plastic characteristics are present, i.e., yield stress)
 Supernate Density: ~1.21 g/mL at 36 wt% soluble solids
 Supernate Viscosity: ~1.8 cP at 36 wt% soluble solids
 Sodium [Na⁺] Conc: ~5.5 M

2.5 Test procedure

The following is a summary of the test procedure. It assumes the slurry was ready for use and in the test rig. The sequence of test runs was to do a series of test runs at a low (~ 0.5 wt%) insoluble solids concentration (runs 1-13) to determine the optimum operating conditions, i.e., the slurry velocity and TMP which gave the highest filtrate flux, then one test run at a high (~ 8 wt%) insoluble solids concentration (run 14) at those optimum conditions.

For the majority of the test runs the test procedure was:

1. Circulate the slurry in the recirculation loop until the temperature reached 298 K.
2. Allow the slurry to flow through the cross-flow filter.
3. When the slurry and filtrate loops establish steady flows adjust the backpulse piston pressure to at least 200 kPa over the slurry pressure in the filter.
4. Backpulse the filter, hold for 10 seconds, then allow the filtrate flow to return.
5. Allow the test rig to run for at least 2 hours.
6. Backpulse the filter once again.
7. End the test run.

3. Results and discussion

3.1. Analytical Test Results

3.1.1. Filter Effectiveness

A test objective was to determine if the nominal rated 0.1-micron Mott filter could remove all the insoluble solids from the simulant. As already mentioned in the preceding experimental section, the insoluble solids were made of 7 compounds: 6 metals (Al, Ca, Cr, Fe, Mn, and Ni) with Chromium being the largest quantity by mass, and Sodium Oxalate, which was the largest single quantity by mass (Fig. 5). Based on a real-sample analysis (Fig. 6a), most of the particle sizes ranged from 0.5 to 5 microns. Fig. 6b confirms that the sizes of solids used in the simulant were close to the target values. From Tbl. 1 it is apparent that the filter removed all the insoluble solids.

Run No.	Solids in Slurry, wt%	Solids in Filtrate, wt%
1	0.45	<0.003
13	0.29	<0.003
14	8.2	<0.01

Table 1. Insoluble solids in the slurry and the filtrate throughout the test.

The filter was very effective in removing all the insoluble solids. Furthermore, the basic sizes of the solid particles did not change during the course of the experiment. Particles smaller than 0.1 micron could have compromised the filter. Figure 7 shows that the particle diameter (by a volume distribution analysis) started at approximately 2.5 microns and remained that size. The standard deviations were on the same order of magnitude as the particle diameters. This was expected since the purchased solids were requested to be between 0.5 and 5 microns. In general, the particle sizes did conform to the required sizes and the particle sizes remained basically intact throughout the experiment.

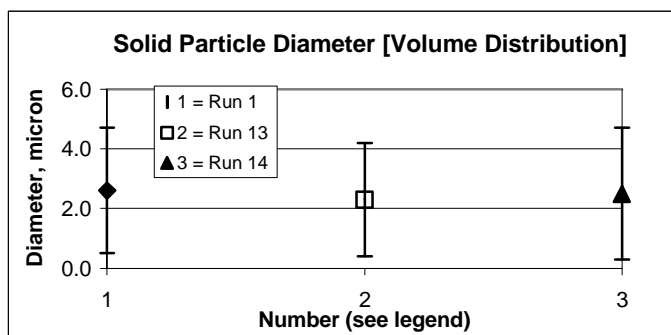


Figure 7. Particle size based on a volume distribution.

3.2. Hydraulic Test Results

3.2.1 Low Solids Concentration

From Fig. 8, at a slurry velocity of 3.7 m/s, there was an approximate 15% increase in filtrate flux when the TMP was increased from 162 kPa to 483 kPa. Since the measurement uncertainty of the filtrate flux was $\pm 7\%$ this increase was just barely significant and most of the increase was realized by the time the TMP reached approximately 290 kPa.

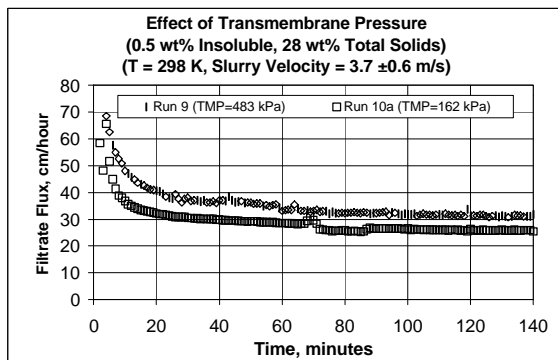


Figure 8. Effect of TMP at a slurry velocity.

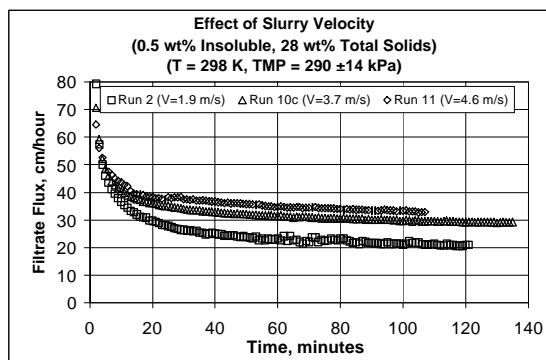


Figure 9. Effect of axial slurry velocity.

At all TMPs tested the filtrate flux was found to increase linearly with increasing slurry velocity. At an average TMP of 290 kPa, Fig. 9 shows a significant improvement of filtrate flux ($\sim 30\%$) when the slurry velocity was increased from 1.9 m/s to 4.6 m/s.

Finally, backpulsing clearly improved throughput of filtrate but that improvement must be balanced against the downtime caused by the increased frequency of cleaning. That is, excessive backpulsing can be detrimental to long-term performance, depending on the type of filter used. For the filter under study, the manufacturer recommends that the backpulse frequency be no greater than once an hour; the longer the time between backpulses the better. Even though the filter elements used had a thick wall (1.6 mm as opposed to a thin membrane filter), it is made to function as a surface, and not a depth, filter. The solids that are removed from the slurry are to remain on the surface of the filter and not lodged within the filter wall. As the filter cake builds, it itself becomes a secondary filter (Murkes and Carlsson, 1988) and according to Fischer and Raasch, 1986 and Lu and Ju, 1989, it is the smallest particles in the slurry that form the cake. When a backpulse occurs, most, or all of the filter cake is knocked off the fixed filter surface, which allows a higher filtrate flux, but also exposes the filter substrate to smaller solid particles. Over time these smaller particles will cause depth fouling and a reduced performance that cannot be improved through backpulsing. Fortunately, the long-term performance, without backpulsing still gave an adequate filtrate flux. Figure 10 shows the results of a separate test done over more than seven hours between backpulses. At the end of the test, the filtrate flux was still above 25 cm/hour[†] and by the asymptotic behavior, the flux appeared to remain fairly constant after 7 hours. Possibly, the filtrate flux could have been maintained for many more hours without a backpulse.

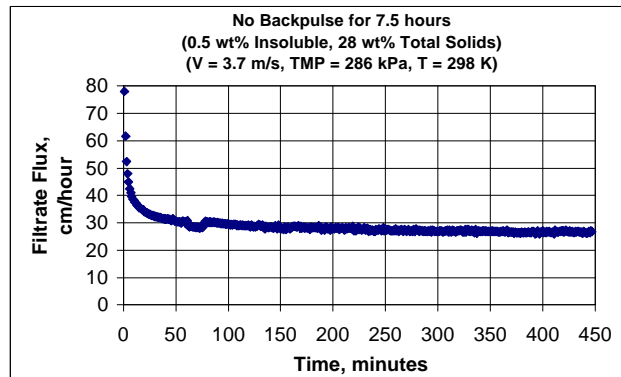


Figure 10. Long term filter operation between backpulses

Even though there are reasons to minimize backpulsing, another separate test was done to quantify how increasing the backpulse frequency increases the filtrate flux. Figure 11 shows the result of backpulsing were significant, implying that most of the filter fouling was on the surface. Increasing the backpulse frequency by a factor of approximately 90, the filtrate flux increased, but only two fold. Many studies (More et al., 2000; Gan, 1999; Levesley and Hoare, 1999; Ramirez and Davis, 1998) clearly show the advantage of increasing the backpulse frequency, even to frequencies of 10 to 20 Hz. However, filters that work well at those frequencies are very thin membranes where depth fouling is generally not a factor, i.e., a particle that manages to enter a filter pore just goes right

[†] Volumetric flow rate per filter surface area (cm³/hour/cm²) was reduced to cm/hour for convenience.

through because of the thin membrane. For a robust sintered-metal filter, depth fouling cannot be ruled out.

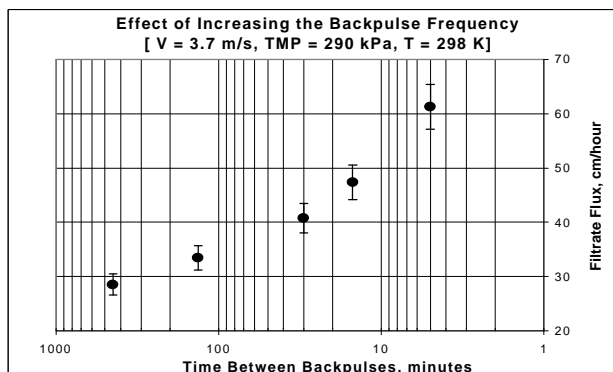


Figure 11. Filter operation with an increasing backpulse frequency

3.2.2 High Solids Concentration Test Run 14

Based on the low-solids concentration runs, the best combination of axial slurry velocity and TMP was chosen to be 3.7 m/s and 290 kPa, respectively. Figure 12 shows the results of the high concentration with a sodium level of approximately 5.5 M and an insoluble solids loading of 8 wt%. The filtrate flux over the entire run stayed at or above 24 cm/hour and in fact was very similar to the low-concentration runs.

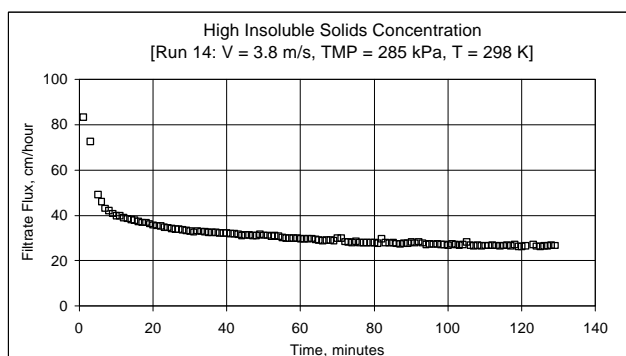


Figure 12. Run 14: 8 wt% insoluble solids; 36 wt% total solids.

3.3. Comparisons to other experiments

Surprisingly, the filtrate flux obtained for both the low and high concentration of solids of this complex slurry was relatively high (≥ 24 cm/hour). This flux is only slightly lower than that obtained by one cross-flow filtration investigation by Murkes and Carlsson, 1988; (p. 41), where a filtrate flux of approximately 40 cm/hour was measured after 2 hours of filtering for a much simpler slurry of distilled water and clay (kaolin). [The experimental conditions were: a nominal filter pore rating of 0.2 μm , TMP~200 kPa, $V \sim 2.5$ m/s, 0.9 wt% insoluble solids, particle sizes $< 2 \mu\text{m}$, a slurry viscosity of 1 cP, and a temperature of 297 K.] Conversely, a filtrate flux of 24 cm/hour was significantly higher (by more than an order of magnitude) than that of a shear-thinning slurry of guar gum

which was investigated by Carrère et al, 1998. [Their filtrate flux was approximately 1 cm/hour for the experimental conditions of: a nominal filter pore rating of 0.14 μm , TMP~200 kPa, $V\sim 3$ m/s, 0.4 w% concentration of guar gum, particle size of 0.24 μm , a slurry apparent viscosity of ~ 400 cP at 1 s^{-1} and 62 at 10^3 s^{-1} , and a temperature of 293 K.].

4.0. Conclusions

The following conclusions are for the nominal rated 0.1 micron Mott cross-flow filter which was used under the conditions stated herein at a slurry temperature of 298 K.

1. There was no measurable quantity of insoluble solids in the filtrate under any circumstance from the filtered slurry that had a solids loading from 0.5 wt% to greater than 8 wt% and for particle sizes measured as small as 0.76 micron.
2. Higher slurry velocities and higher transmembrane pressures led to higher filtrate fluxes. However, the increase in filtrate flux was strongly affected by the slurry velocity, but only weakly by TMP.
3. For a low concentration of insoluble solids (0.5 wt%), the best overall filtrate flux was 50 cm/hour at a slurry velocity of 3.7 m/s and a TMP of 290 kPa.
4. An average filtrate flux of 25 cm/hour can be maintained for at least 7 hours without backpulsing. It could be increased to 40cm/hour with a backpulse interval of 30 minutes.
5. For a high concentration of insoluble solids (8 wt%) the filtrate flux remained above 24 cm/hour for at least 2 hours after a backpulse.
6. Surface fouling of the filter was the dominant mechanism of fouling since backpulsing returned the initial filter flux but overtime depth fouling cannot ignored.

Acknowledgments

The author would like to thank all that were involved in this task including members of the Savannah River Technology Center of the Westinghouse Savannah River Company and BNFL, Inc. The work was funded by the U.S. Department of Energy under Contract DE-AC09-96SR18500.

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