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EXPERIMENTS ON CAKE DEVELOPMENT IN CROSSFLOW FILTRATION FOR HIGH LEVEL WASTE

Mark R. Duignan and Charles A. Nash
Savannah River Nuclear Solutions
Savannah River National Laboratory
Aiken, South Carolina, USA

ABSTRACT

Crossflow filtration is a key process step in many operating and planned waste treatment facilities to separate undissolved solids from supernate slurries. This separation technology generally has the advantage of self cleaning through the action of wall shear stress, which is created by the flow of waste slurry through the filter tubes. However, the ability of filter wall self cleaning depends on the slurry being filtered. Many of the alkaline radioactive wastes are extremely challenging to filtration, e.g., those containing compounds of aluminum and iron, which have particles whose size and morphology reduces permeability.

Low filter flux can be a bottleneck in waste processing facilities such as the Salt Waste Processing Facility at the Savannah River Site and the Waste Treatment Plant at the Hanford Site. Any improvement to the filtration rate would lead directly to increased throughput of the entire process. To date, increased rates are generally realized by either increasing the crossflow filter axial flowrate, which is limited by pump capacity, or by increasing filter surface area, which is limited by space and increases the required pump load.

In the interest of accelerating waste treatment processing, DOE has funded studies to better understand filtration with the goal of improving filter fluxes in existing crossflow equipment. The Savannah River National Laboratory (SRNL) was included in those studies, with a focus on startup techniques and filter cake development. This paper discusses those filter studies.

SRNL set up both dead-end and crossflow filter tests to better understand filter performance based on filter media structure, flow conditions, and filter cleaning. Using non-radioactive simulated wastes, which were both chemically and physically similar to the actual radioactive wastes, the authors performed several tests to demonstrate increases in filter performance. With the proper use of filter flow conditions filter flow rates can be increased over rates currently realized today.

This paper describes the selection of a challenging simulated waste and crossflow filter tests to demonstrate how performance can be improved over current operation.

INTRODUCTION

Crossflow filtration is a well established technology, but the method of use and the efficiency of its separation vary widely for each different industrial application and indeed within production-end product categories. For the DOE Complex the stored radioactive wastes are being prepared for long-term storage and disposal with many technologies. Treatment of much of that waste begins with the separation of suspended solids from the liquid by filtration, including crossflow filtration. Those wastes can be very challenging to filters causing a bottleneck for an entire processing cycle. A better understanding of crossflow filtration with such wastes may help to increase filter performance and thus overall

waste treatment throughput. This study finds that filter performance can be improved with the existing hardware in current treatment plants.

The two items of focus for crossflow filtration are the filters themselves and the waste to be treated. Details of each are given later, but highlights are described here. Figure 1 is a diagram of a typical crossflow filter arrangement, which is shown in a horizontal orientation but could be vertical or at some other inclination. The arrows in the center, parallel to the walls, represent the slurry flow or the axial velocity (AV) of the slurry. The walls are the porous filter medium that separates permeate from the slurry. The arrow, perpendicular and outside the top wall represents the permeate. The motive force that drives the liquid through the filter wall is the difference in pressure from the slurry to the permeate and is referred to as the transmembrane pressure (TMP).

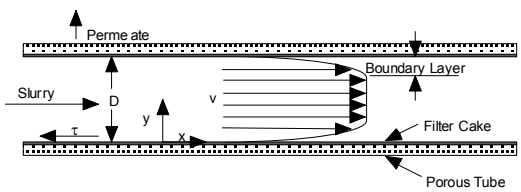

FIGURE 1. TYPICAL CROSSFLOW FILTER ARRANGEMENT

In the past many different filter media have been used at waste treatment sites. Currently, two large treatment plants are under construction – the Waste Treatment Plant at the DOE Hanford Site and the Salt Waste Processing Facility at DOE Savannah River Site. For the former the crossflow filters are 0.0127-m (1/2–inch) inside diameter stainless steel tubes and for the latter they are 0.0095-m (3/8-inch) inside diameter stainless steel tubes. This study determines differing performances between these filters. Both of these filters are made with a 0.1 micron nominal pore rating and of a symmetric sintered metal design. Because the only difference between these two filters is geometry another tube design was added for comparison. This third tube has an asymmetric design, a 0.0095-m (3/8-inch) inside diameter, and a 0.1 micron absolute pore rating. While this last tube is still made primarily of sintered stainless steel, the inner tube surface is coated with a 10-micron thick layer of zirconia. In order to elicit a side-by-side performance of all three filter tubes they were placed in parallel in a test facility such that the same test simulant would flow through each at the same time. Because the properties of wastes to be treated can change with time it is important to remove the issue of aging, which could confound results.

The range of wastes to be treated is large [1], but in general they usually have high soluble ionic salt contents and are radioactive. Due to the risk/costs of radioactivity, testing was done with a non-radioactive simulant; however, actual waste testing will be necessary in the future. The selected simulant was made in a manner that the chemical and physical properties were typical of the actual waste. It was important to choose a waste that would be difficult to filter. The waste should contain components that make filtration difficult [2], e.g., iron and aluminum oxides, and small particle size, and should have some past history of filtration so a comparison could be made. The candidate selected is a salt waste and is referred to as; Sludge Batch 6 (SB6) for which the properties will be discussed later, but was consider very challenging with respect to the compounds it contains and large range of particle sizes.

When dealing with micro or ultra-filtration, one operational issue that has often been considered is how to maintain a filter surface free of cake. The rationale is that a cake-free surface will allow the solid-liquid separation to occur faster. For crossflow filtration to maintain a surface cake-free, or minimize cake buildup, the predominant method is backpulsing. In many cases backpulsing is absolutely necessary to maintain a high permeate flux, like in the water treatment industry [3]. Some backpulsing frequencies can be quite high, for instance it could be as high as 1 Hz used by some in the biochemical industry [4] where the backpulse duration is only fractions of a second. Some [5] state that "one method of reducing membrane fouling is rapid backpulsing," where backpulsing involves the reversal of the permeate flow through the filter membrane for very short periods," and that this "can provide in situ cleaning by removing some of the foulants from the membrane surface or pores." Up to a 30-fold increase with the use of backpulsing over no backpulsing has been realized.

At issue, is an ongoing need to keep the filter surface clean, i.e., free of cake. Is this the approach method for all slurries? When waste processing plants were designed to treat stored salt wastes at the Hanford and Savannah River sites backpulsing was included to help maintain

filter fluxes high. Unfortunately during the last ten years, or so, filter tests have shown that backpulsing has not been very effective [6-8]. While a lot of time and effort was invested to design robust flow-reversing systems results have not been promising.

Along with backpulsing another method to keep the filter surface clean, or to minimize cake buildup was to flow the slurry very fast past the filter surface so that the shear stress would strip the cake from the wall. However, typical axial flowrates used in operation, e.g. 3-5 m/s, may not suffice for some suspensions that are viscous or have a strong affinity for the filtration surface. If it were possible to separate the wall shear from slurry flow, a shear rate may be attainable to keep the filter wall clean.

To address this need a concept of a rotary microfilter [9] was developed that spins the filtration surface at a rate such that the filter outer surface moves at more than 18 m/s. In fact approximately 70% of the filtration surface is kept completely free of cake.

Another attempt to prevent cake to build on the filter surface is never letting the cake settle by reversing the slurry flow every couple of minutes [10] but it takes a lot of energy for large systems to reverse flow.

Indeed, having a clean filtration surface does lead to high filtration fluxes, initially, however, when the surface is clean it is always exposed to the smallest particles in a slurry. Specifically for backpulsing it has been shown that once a cake is lifted off a filter surface the smallest particles are the first to return to the surface, which accelerates depth fouling [11-12]. Because of this fact backpulsing was recommended to be kept at a minimum [6].

Cake Development

Because of poor filter performances and the ineffectiveness of backpulsing with stored salt wastes [2, 6] tests were developed to filter without backpulsing. Furthermore, filtration began by trying to establish a cake that would be more permeable and thus lead to better filter fluxes. Of course the filter membrane is itself is a filter, but by forming a filter cake on the surface a secondary filter is established [13]. When forming a cake it is always important to take into account the nature of the slurries and sludges being filtered [14]. From past work [6] it appeared that the salt wastes adhered well to the filter surface based on the loss of the backpulse effectiveness in a very short time (hours); a time short enough that filter depth fouling was probably unlikely. Unfortunately, there is no direct evidence of surface adhesion or fast depth fouling; therefore, an assumption of good adhesion led to the method of cake development used in this study. In the past, the procedure to start and maintain filtration was to fill the filtration and slurry systems, start filtration with an immediate backpulse, then periodically backpulse when the permeate flux became unacceptably low [6-8]. Depending on the waste stream the effectiveness of backpulsing drops with time. Eventually filtration has to be stopped to chemically clean the filter membrane in order to remove the depth fouling.

The intention of the present study was to not avoid cake buildup, but to actively establish a cake. Hopefully, the cake would be permeable and act to filter even smaller particles than what the filter membrane itself was capable of. If the filter could be started in a fashion so the smallest particles in the cake would not be near of the filter surface, Fig. 2(a), but actually closer to the top of the cake, Fig. 2(b), this may help, especially if that top layer could be periodically stripped off.

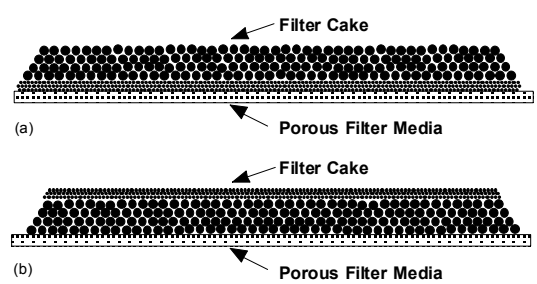

FIGURE 2. Schematic of cake on filter surface with most of the smallest particle in the cake at the: (a) bottom, (b) top

That is, to maintain the flux high a mechanism of what will be called "scouring," was tried. This is an action of stripping off some of the established cake to remove the smallest particles [15] by increasing the slurry axial velocity for a short period while no filtration is occurring.

Scouring

During the test many trials were done to see if permeate flux could be improved while filtering without the need of cleaning. A method that the authors termed scouring seemed to work the best. The scouring process begins while the

filter is in steady state, i.e., the slurry axial velocity is constant and the filter cake is well established. The permeate flux is then stopped for a short period. This has been tried by others [16]; however, an extra step was added to increase the axial velocity by 50 to 80% above the operational velocity. After being held at this higher velocity for 15 to 20 minutes the velocity is then returned to the original value while permeate flow is reestablished very slowly over a 15-minute period. The expectation was that scouring would remove the upper layer of cake that could contain small particles [15] and leave a base filter cake, free of the smallest particles. The hope was to return the filter rate to what was initially established at start up. If successful the further hope was that this process could be repeated indefinitely.

NOMENCLATURE

AV	Axial velocity of slurry being filtered
DOE	U.S. Department of Energy
NTU	Nephelometric Turbidity Unit
SB6	Sludge Batch 6, salt waste simulant
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
TMP	Transmembrane pressure, the pressure difference that drives the permeate through porous media.

EXPERIMENTAL SETUP

Crossflow Filter Equipment

Figure 3 is a schematic of the test rig, which was made up of three basic flow loops:

1. Slurry loop – contains three 0.6-m long filters and their housings, which serve as the primary flow path for circulating slurry. This "loop" was really made of three sub-loops so that the three filters could be controlled separated in order to maintain the same flow conditions in each despite their geometric differences.
2. Permeate loop – begins at the filter housing and allows the separated permeate liquid from each filter to flow to a common header that was directed back to the slurry tank.
3. Cleaning loop – allows the three filters to be cleaned without removing most of the test slurry that remained in the lower portion of the test rig during cleaning.

To circulate slurries in the test rig two 10 hp Galigher centrifugal pumps were used. The impeller and impeller housing were lined with EPDM to be compatible with both the pH > 14 slurry that was tested and the pH < 1 acid cleaning solutions. The two pumps were used in series for the slurry loop to attain a head of greater than 450 kPa at 225 lpm.

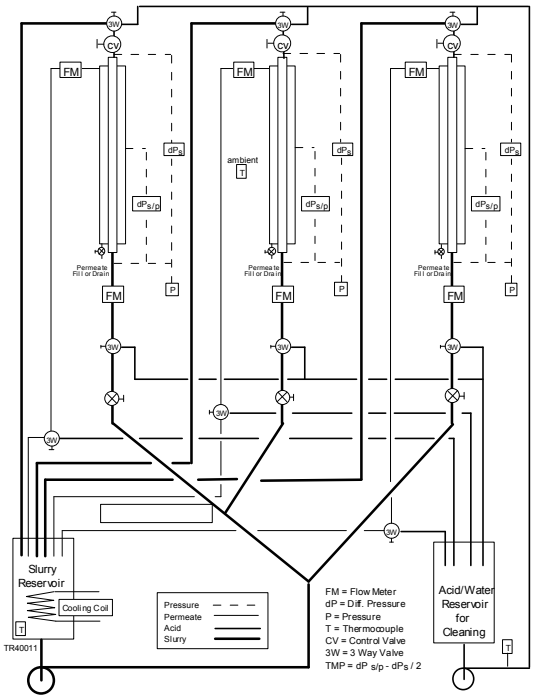

FIGURE 3. SCHEMATIC OF THE CROSSFLOW ULTRAFILTRATION TEST FACILITY

TABLE 1. FILTER TUBES SPECIFICS

Filter[a]	Actual Inside Diameter (m)	1 x Standard Deviation (m)	Actual Outside Diameter (m)	Medium Design[b]	Primary Material	Active Length (m)	Filter Surface Pore Rating[d]
Mott	0.01237	6.62E-05	0.01658	Symmetric	316L Stainless	0.572	0.1 micron nominal
Mott	0.00923	6.91E-05	0.01301	Symmetric	316L Stainless	0.572	0.1 micron nominal
Pall	0.00994	8.32E-05	0.01218	Asymmetric[c]	316L Stainless	0.572	0.1 micron absolute

a. Mott refers to the Mott Corporation and Pall refers to the Pall Corporation.
b. Symmetric = filter has same material and pore rating throughout, Asymmetric = filter has two or more materials and pore ratings.
c. Pall filter consists of a 10-micron thick inner surface made of zirconia and a stainless steel substrate that has a much larger pore rating.
d. The word "nominal" for a filter rating 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 approximately 95% of particles greater than 0.1 micron will not pass the filter. For the 0.1 micron absolute rate 100% of particles greater than 0.1 micron will not pass the filter. A rough approximating between the two ratings is a 0.1 micron nominal has been equated to 0.7 micron nominal rating [17].

Crossflow Filters

Details for the three crossflow filters tested are found in Tab. 1. Figure 4 shows one end view of the three filter tubes after they were machined to fit in the filter housing. Note the filter wall of the Mott tubes, which is of a symmetric design, are thicker than the Pall filter that had the asymmetric design with an inner coating of zirconia.

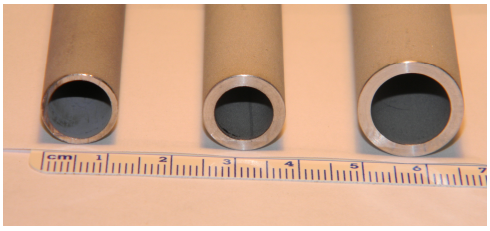

FIGURE 4. THREE FILTER TUBES STUDIED, LEFT TO RIGHT: 0.0095-m PALL, 0.0095-m MOTT, 0.0127-m MOTT

Once installed in the test facility all three tubes had an active filter length of 0.57 m and can be seen in Fig 5. A close up of one of the tubes is shown in Fig. 6.

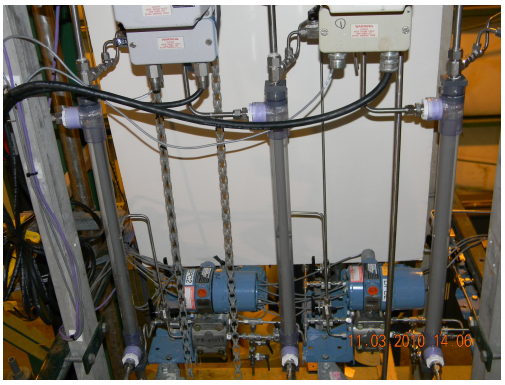

FIGURE 5. THREE FILTER TUBES IN FILTER HOUSINGS

FIGURE 6. CLOSE UP OF ONE OF THE THREE TUBES

Instrumentation

The measurement equipment used for this experiment was:

5 Type E thermocouples
accuracies*: 0.6 to 1.1°C,
6 Differential pressure transducers
accuracies*: 0.14 to 0.83 kPa
3 Gauge pressure transducers:
accuracies*: 0.28 to 0.41 kPa
3 Magnetic flow meters (permeate):
accuracies*: 1.89 to 6.06 E-6 m^3
3 Magnetic flow meters for slurry
accuracies*: 1.14 to 2.27 E-4 m^3
1 Turbidity meter:
accuracy: ±2% Reading or 0.01 NTU, whichever is greater

*accuracies 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.

Measurement Uncertainty

The measurement uncertainties (95% confidence level), for the important calculated quantities are:

Slurry Velocity in a Filter Tube:	± 9 %
Transmembrane Pressure:	± 1 %
Permeate Flux:	± 12 %

Simulated Waste Slurry

Two waste simulants were obtained for this test: a HM Waste Simulant – Sludge Batch 6 (SB6) and a Purex Waste Simulant – SRS Tank 8F[†].

In a separate dead-end filter test the SB6 was found to filter significantly slower than the Tank 8F waste. Therefore, SB6 was chosen for the crossflow filter test and the properties of the SB6 sludge properties are shown below in Figs. 7-9 and Tabs. 2-3. The yield stress of 54.6 Pa for SB6 simulant is equivalent to waste tanks with the highest yield stresses and therefore should be conservative in this aspect.

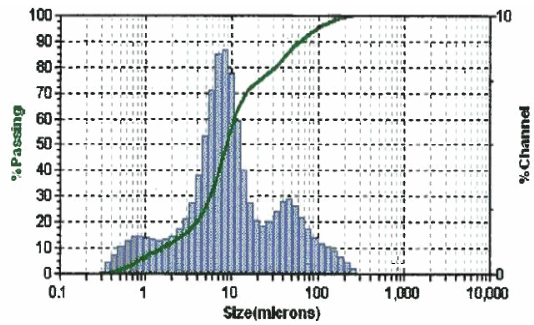

FIGURE 7. PARTICLE SIZE DISTRIBUTION OF SLUDGE SIMULANT

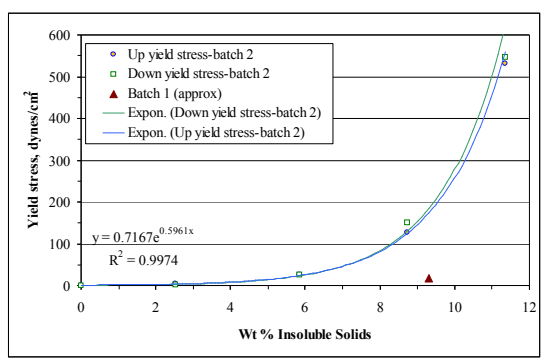

FIGURE 8. SLUDGE BATCH 6 WAS USED FOR THE TEST. NOTE: 10 dyne/cm^2 = 1 Pa

[†] Nuclear Waste Definitions: A HM Waste refers to a liquid waste that came from the H-Canyon (at SRS) Modified Purex process and a Purex Waste resulted from the **P**lutonium **UR**anium **EX**traction process.

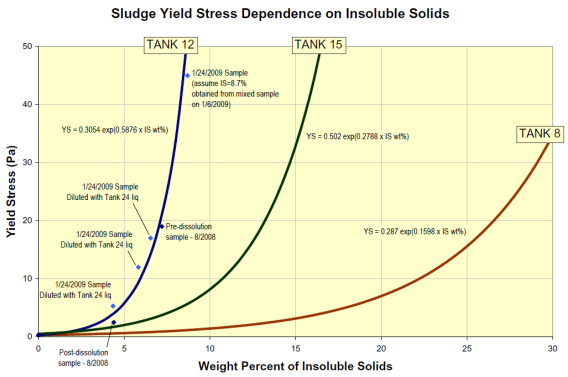

FIGURE 9. RANGE OF YIELD STRESS OF WASTES STORED IN SRS TANKS

TABLE 2. SB6 SLUDGE CONSTITUENTS

Component	Calcined Solids, wt%	
	Target	*Actual*
Al	16.181	15.80
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	**66.0**	**64.4**

TABLE 3. PROPERTIES OF SB6

	Target	*Actual*
Slurry density, g/mL	1.12 ± 0.05	1.12
Total solids, wt%	18.2 ± 2%	16.7
Insoluble solids, wt%	14.0 ± 1%	10.4
Anions, mg/Kg		
Nitrite	8807 ± 10%	1110
Nitrate	6096 ± 10%	6470
Phospate	27 ± 25%	<100
Sulfate	904 ± 25%	1060

Notes for Tab. 3:
Simulant properties "as-received"were:
Bingham Yield Stress 54.6 Pa
Bingham Consistency = 17.8 cP
The SB6 was mixed with a 5.6 M supernatant to obtain a 5 wt% solids loading before testing.

The size of particles for the SB6 simulant had a large variation from 0.3 to 300 microns, which captured well the ranges expected in the actual wastes [18]. In fact particle size distribution was tri-modal, Fig. 7, with peaks at approximately 0.8, 8, and 50 microns. It was assumed this range would be very challenging to the filters.

DISCUSSION

Cake Development, Long-term Slurry Flux at 5 wt% of Undissolved Solids, and Scouring

The overall test results are shown in Fig. 10. The three filters were pre-conditioned by previously subjecting them to the test slurry, followed by a pre-acid water rinsing, an acid cleaning, and a post-acid water rinsing. This was done until the water fluxes returned to those obtained before filtering with slurry. The preconditioning was to try to put the filters in a 'used' condition to avoid the anomaly of new filter performance.

Region 1: Cake Development and Scouring

Here the filter system was very slowly filled with the test slurry while the permeate system was shut so the filters would not become challenged prematurely. However, the permeate system did slowly fill with permeate as liquid separated from the slurry into the filter housing. This was possible because the air in the permeate housing was drawn into the slurry through the filter, which then percolated through the slurry loop until it was released to the atmosphere from the slurry reservoir. Once the slurry was circulating at a very slow rate, i.e., the axial filter velocity was less than 0.5 m/s, and air stop leaving the system, then the permeate system was allowed to flow. The permeate flow was established slowly, over a 15-minute period. Once both the permeate and slurry loops were filled, the permeate flow was once again stopped. Now the flow conditions to be used for filtration were established, i.e., axial velocity (AV) = 3.66 m/s and a transmembrane pressure (TMP) = 276 kPa. (These condition were used because they had been selected from previous work [6] as the best for filtration.) Once established and stable, then the permeate flow was very slowly (15 minutes) engaged. With the permeate flow established the system was allowed to run about

2 hours to allow the filter cake to develop, as noticed by a slight drop in filter rate. After that period the filters received the initial 'scouring' for 15 to 20 minutes.

To reiterate, scouring is the process of 1. shutting the permeate flow valve, 2. increasing the slurry axial velocity to 50% to 80% above the original set velocity, 3. allowing the high velocity slurry to flow for approximately 20 minutes, then 4. returning the velocity back to the original setting, and finally 5. reestablishing permeate flow over a 15-minute period. This scouring, which was done after two hours, is hard to see in Fig. 10 but it is exactly what was done between Region 1 and Region 2. In fact, it is what was done between Regions 2 and 3, 3 and 4, 5 and 6, noted by the jump in permeate flux. That is, at no time were the filters cleaned or backpulsed, but only scoured. It can be seen that after each scouring the filtration flux return to approximately the same value, implying that no significant depth fouling had occurred. In fact, over the entire 290 hours (12 days) of continuous filtration the filters were never backpulsed or cleaned and after each scouring the filter flux always returned to its initial value at time zero.

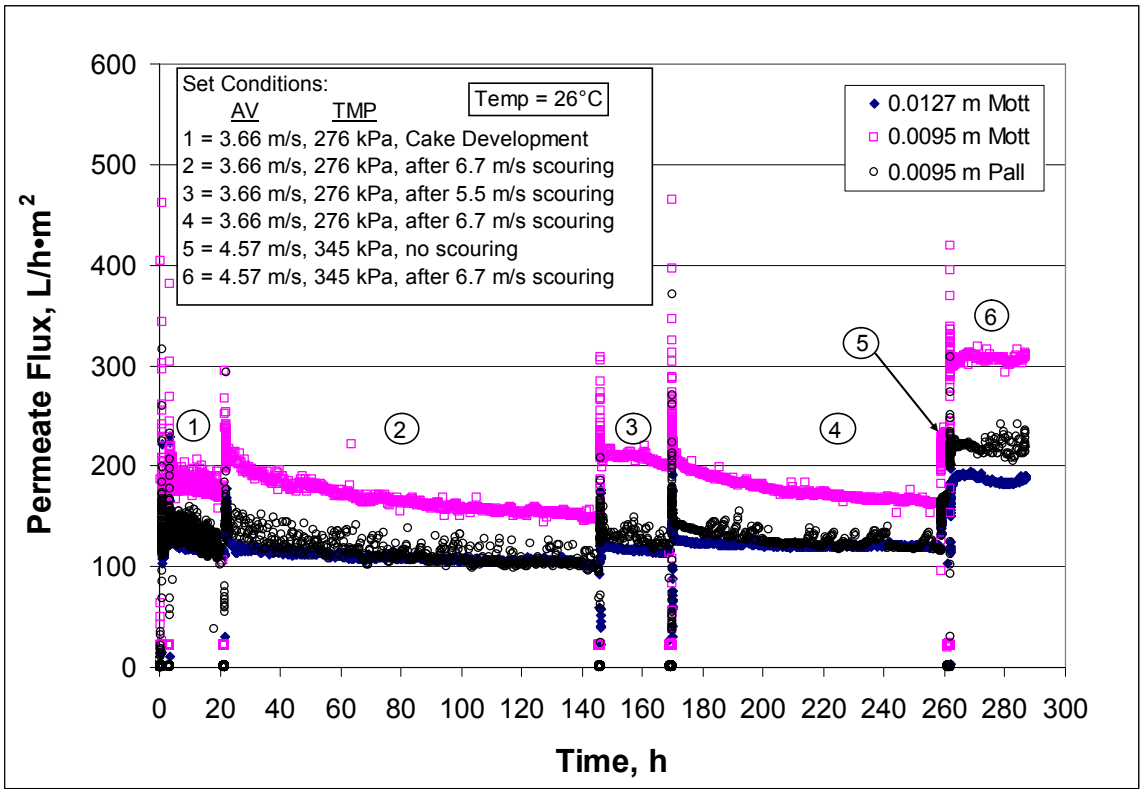

FIGURE 10. LONG-TERM 12-DAY FILTRATION TEST DONE AT THE CONDITIONS LISTED IN THE LEGEND AND AT 25°C ±2°C

Region 2: Long-term Filtration

Figure 11 is an excerpt of Region 2 from Figure 10. It depicts the better performance of the 0.0095-m Mott filter over the 0.0127-m Mott and the 0.0095-m Pall

An interesting feature is the higher flux of the smaller diameter filter tube. The 30 to 40% higher flux of the small Mott tube over the larger Mott tube was not a surprise as this has been studied previously [19] but it was never observed with the same slurry at the same time; this evidence was reassuring. The higher flux is directly related to the high wall shear for the smaller of the two tubes with the same porosity. The other interesting aspect seen in Fig 11 is the very slow rate of decline in the filter flux. The drop in flux is approximately 30% over 5 days,

which is a significant improvement to the 80% drops experienced from past works [6, 8].

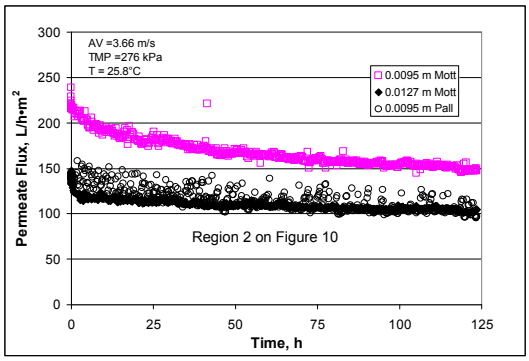

FIGURE 11. FILTER PERFORMANCE AT A SINGLE SET OF FLOW CONDITIONS AT 5 WT% SOLIDS LOADING

Finally, the large difference in flux between the two tubes with the same insider diameter of 0.0095 m must be related to the different pore structure. The Mott filter is listed as a 0.1 micron nominal pore and the Pall is a 0.1 micron absolute pore. As mentioned earlier, the 0.1-m Mott has been estimated [20] to be an approximately 0.7 micron absolute pore rating; therefore, the Pall had a much tighter pore structure. The smaller pores do a much better job to separate the smallest solid particles, but also results in a lower flux because of a much higher base membrane flow resistance. The question then is: is the much tighter pore needed for these type wastes? One way to determine this would be to measure the turbidity of the permeate. Unfortunately, the turbidity of the permeate of each filter could not be measured because all three streams were joined in a common header, as the permeate was returned to the slurry reservoir. However, the turbidity of the joined stream was measured and that would tell at least the separation efficiency for the tube with the largest pore openings:

Turbidity (±0.01 NTU)
Deionized water only: 0.26 NTU
From filters using only water: 0.25 NTU
From filter using 5 wt% SB6: 0.03 NTU

These data imply that not only does the filter cake act as a secondary filter but it prevents even the smallest particles from passing through the filter. This means that the more open pore structure is more efficient. Of course, the pore size cannot be allowed to become too big because eventually depth fouling would confound operation.

Region 3, 5, and 6: Higher Flow Conditions

Figure 12 is an excerpt of Regions 3 and 6 from Figure 10 of only the 0.0095-m Mott data. Because of the success of a much higher, and longer sustained, filter flow rate than expected, it was of interest to see if higher flow conditions would result an even higher permeate flux. After several scourings and a return to the same axial velocity and TMP, those values were increased.

At the end of a successful long term run, shown as Region 4 in Fig. 10, the flow conditions were increased to an axial velocity of 4.57 m/s and a TMP of 345 kPa, without scouring. Indeed the filter fluxes increased by about 50%, but this is only about 15% of the starting flux of Region 4. However, after a scouring was done and then a return to the conditions of 4.57 m/s and 345 kPa, the increase was 100% or about 43% above the starting point of Region 4.

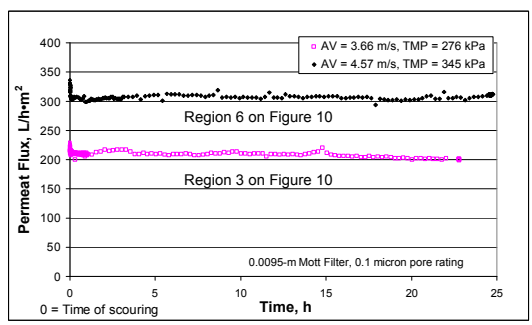

FIGURE 12. FILTER PERFORMANCE AT A SINGLE SET OF FLOW CONDITIONS AT 5 WT% SOLIDS LOADING

This 43% can be seen better in Fig. 12 which illustrates the 20+ hours of operation after a scouring that started Regions 3 and 6 with the 0.0095-m Mott filter. The permeate flux at an AV of 4.57 m/s and a TMP of 345 kPa was surprising high, better than 300 L/h•m^2 and remained high for a full 24 hours. With continual scouring this flux may be maintained for a very long time.

Finally, data were not obtained using the same filters and slurry simulant while operating in the traditional method. The traditional method entails: 1. a fast start up of slurry and permeate flow with the full TMP, 2. an initial single backpulse to clean the filter surface, followed by

3. periodic backpulses, as the permeate flux drops to a predetermined value. These data were not obtained during this test but there is a large database [21-22] with similar waste streams and filters that demonstrate past and current operation. Those data roughly show that with the traditional method the permeate flux begins at approximately 200 L/h•m^2, but within 12 to 36 hours the flux drops to below 50 L/h•m^2, which requires filtration to stop so the filters can be cleaned with acid. The scouring method described in this paper seems to result in much better filter performance.

CONCLUSIONS

Experiments that use non-radioactive simulants for actual waste always carry the inherent risk of not eliciting prototypic results; however, they will assist in focusing the scope needed to minimize radioactive testing and thus maximize safety. To that end this investigation has determined:

- Filter cake is something that should be properly developed in initial filter operation.
- Backpulsing is not necessary to maintain a good filter flux with salt wastes and metal oxide/hydroxide sludges.
- Scouring a filter without cleaning will lead to improved filter performance.
- The presence of a filter cake can improve the solids separation by an order of magnitude as determined by turbidity.
- A well developed cake with periodic scouring may allow a good filter flux to be maintained for long periods of time.
- Permeate flux decline is reversible when the concentration of the undissolved solids drops and the filter is scoured.

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