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Filtration, Washing, and Caustic Leaching of Hanford Tank AZ-101 Sludge

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Abstract: Approximately 4.3 kg of slurry from Hanford Tank AZ-101 were evaluated by the pretreatment processes of cross-flow filtration, washing, caustic leaching, and rinsing. The filterability was measured with a 0.1-μm sintered metal filter using a single-element, cross-flow filtration system. During testing, the permeate flux and slurry axial velocity, pressure, and temperature were monitored every 10 min. The slurry temperature was maintained at 25°C ± 5°C, except during the elevated-temperature leaching step. The test results show that cross-flow filtration provides excellent separation of solids and liquids, as evidenced by the permeate flux and the measured activity of nearly insoluble ^{241}Am. The average permeate flux measured was higher than the Waste Treatment Plant target of 0.014 gpm/ft^2, and the average decontamination factor for ^{241}Am was 985,000 during testing.

INTRODUCTION

To convert radioactive waste stored in underground tanks into glass, the world's largest vitrification facility is being designed and constructed at the U.S. Department of Energy's (DOE) Hanford Site near Richland, WA. Flow sheets developed for the Waste Treatment Plant (WTP) include the use of washing and caustic leaching to pretreat Hanford sludge before high-level waste (HLW) vitrification. These pretreatment steps reduce the quantity of HLW generated by removing components such as aluminum, chromium, sodium, and phosphorus that are soluble in water or high-temperature caustic solutions, or both, and often limit the waste loading in the glass. So that they may be

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disposed as low-activity waste, the goal is to have the activity of the wash and leach solutions below Class C levels after cesium ion exchange. Cross-flow filtration is specified for the initial dewatering and to separate the wash and leach solutions from the solids. In cross-flow filtration, the majority of the filter cake is swept away by the fluid flowing across it. This filtration method is especially beneficial when there are very fine particles and when system simplicity is required.

The filtration, washing, and caustic leaching characteristics of sludge from Hanford Tank AZ-101 were evaluated (a) to determine if the WTP-targeted permeate flux of 0.014 gpm/ft^2 was attainable and (b) to determine the removal efficiencies of key components in the actual waste for subsequent input into the WTP flow sheet. The tests were conducted in a cells unit filter (CUF), a single-element, cross-flow filtration system used by the project to validate assumed permeate flux and removal efficiencies on actual tank waste. The chemical and radiochemical compositions of the permeate and the final leached solids were measured to determine the efficiency of the filtration, washing, and leaching processes.

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TEST APPARATUS

The CUF system is used for pretreatment and cross-flow filtration testing of highly radioactive tank waste and is used at DOE sites with significant defense wastes, most notably Hanford and the Savannah River Site. The design of tank waste simulants, used for larger-scale filtration testing, is based on characteristic data obtained in CUF testing on actual waste. The CUF is designed for hot cell operations with cross-flow filters of active lengths between 6 and 24 in. A 0.1-μm Mott sintered metal filter with a 24-in. active length and 3/8-in. bore was used in this testing. Figure 1 shows a photograph of the CUF apparatus.

To test materials in the CUF, slurry feed is introduced into the system through the slurry reservoir. An Oberdorfer progressive cavity pump (powered by an air motor) pumps the slurry from the slurry reservoir through the magnetic flow meter and the 24-in. active length filter element. The axial velocity and transmembrane pressure (TMP) are controlled by the pump speed and the throttle valve position. Permeate that passes through the filter can be sent to the back pulse chamber; can be reconstituted with the slurry in the slurry reservoir; or can be removed. The permeate flow rate is measured by means of a graduated glass-flow monitor that is fill-and-drain operated. Filter back pulsing is conducted by partially filling the back pulse chamber with permeate, pressurizing the back pulse chamber with air, and forcing the permeate in the chamber back through the filter.

EXPERIMENTAL

Tank AZ-101 contains high-level radioactive waste that was primarily generated from past plutonium production at the Hanford Site. The AZ-101

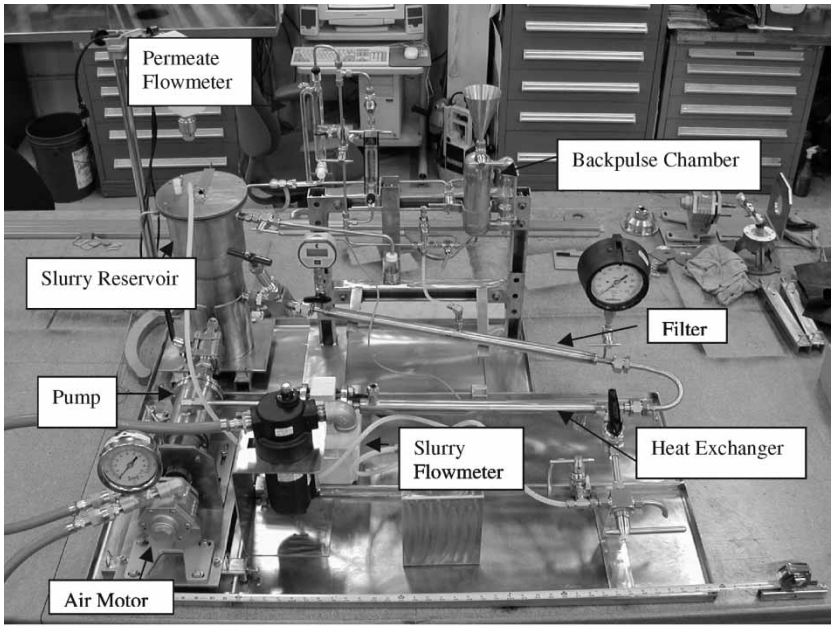

Figure 1. Cells unit filter system before installation in shielded cell.

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waste is mainly composed of metal oxides and hydroxides. The major components of the sludge (on a dry weight basis) include Na (13 wt %), Al (9.5 wt %), and Fe (5.3 wt %). Tables 1 and 2 provide component concentrations of the original slurry.

The material obtained for this testing was from a core sample retrieved from Tank AZ-101 in August 2000. The sample was homogenized into a single slurry composite and characterized chemically, radiochemically, and physically (1).

It is important for the reader to understand that, under most testing conditions, the permeate is recycled back to the slurry after the flux measurement. Reconstituting the slurry is necessary, because the small sample size would allow only very short tests if the permeate were removed. Removing the permeate from the slurry is referred to as dewatering.

To begin the testing, clean water flux was measured to benchmark filter performance. AZ-101 slurry at two different solids concentrations (one nominally 7.6 wt % undissolved solids and one dewatered to 17.9 wt%) was tested using a matrix consisting of 13 different conditions (Fig. 2) of various transmembrane pressures (TMPs) and cross-flow velocities. The first condition (center point) was held for 3 h before conditions were changed with a back pulse each hour. The center point was then repeated in the middle and at the end of testing to assess the effect of filter fouling

Table 1. Nonradioactive component concentrations in the original slurry (dry weight basis)

Analyte	Concentration, μg/g
ICP-AES results	
Ag	[457]
Al	95,000
Ba	382
Ca	[2,805]
Cd	3,785
Ce	[4,430]
Cr	1,555
Cu	[54]
Fe	52,750
K	[7,600]
La	1,560
Li	[115]
Mg	[410]
Mn	1,445
Mo	[160]
Na	130,000
Nd	1,185
Ni	2,760
P	1,715
Pb	[590]
Pd	[1,600]
Rh	[480]
Si	[4,855]
Sn	[1,600]
Sr	904
Ti	[54]
Y	[103]
Zn	[86]
Zr	14,300
IC results	
Br^-	1,083
Cl^-	<516
$C_2O_4^{-2}$	3,867
F^-	3,739
NO_2^-	112,930
NO_3^-	100,812
PO_4^{-3}	1,753
SO_4^{-2}	45,378

Notes: (1) Overall error greater than 10-times detection limit is estimated to be within $\pm 15\%$. (2) Values in brackets [ ] are within 10-times detection limit with errors likely to exceed 15%.

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Table 2. Radioactive component concentrations in the original slurry (dry weight basis)

Analyte	Concentration, μCi/g
^{3}H	8.43E − 2
^{14}C	5.72E − 3
^{90}Sr	15800
^{60}Co (GEA)	2.06
^{137}Cs (GEA)	2130
^{125}Sb (GEA)	8.89
^{154}Eu (GEA)	24.5
^{155}Eu (GEA)	28.7
^{241}Am (GEA)	47.7
^{241}Am (AEA)	41.4
$^{243/244}$Cm (AEA)	<2E-1
$^{239/240}$Pu (AEA)	2.44
^{126}Sn (ICP-MS)	0.078 μg/g
^{238}U (ICP-MS)	1.11 E-3

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over the course of testing. The system was back pulsed once between each condition.

Once the testing with the first matrix was completed, the system was run for ~10 h at 40-pounds-per-square-inch differential (psid) TMP and 11 ft/s axial velocity without back pulsing. After the extended run, representative

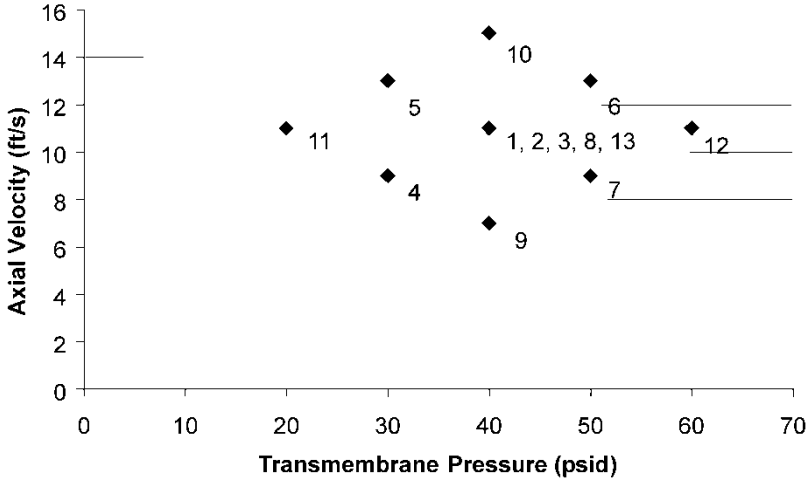

Figure 2. Experimental steps (conditions 1–13) for Hanford Tank AZ-101 cross-flow filtration test.

permeate samples were taken; the slurry was dewatered; and 2448 g of permeate were collected in five bottles labeled AZ-101. At this point, the solids concentration in the CUF was 17.9 wt% undissolved solids. The test matrix and the extended run were repeated at 17.9 wt% undissolved solids loading, and representative samples of the slurry were taken for analysis.

When the second matrix was completed, the slurry was further dewatered, and 656 g permeate were collected. At that point, the slurry was 24.7 wt % undissolved solids, which was close to the rheological limit of the filtration apparatus. Further concentration of the slurry would have resulted in plugging. The slurry was batch-washed twice with a volume of inhibited water (0.01 M NaOH) equal to the slurry volume (1 L). The purpose of the wash is to remove and displace the interstitial liquid and dissolve soluble sodium salts. The slurry was dewatered after each batch addition and a total of 1678 g of solution were removed. Permeate samples were taken during each wash and slurry samples were taken after the second wash. After the two washes, the measured undissolved solids concentration in the slurry was 19.1 wt%.

The remaining 1.4 L of slurry was caustic leached by the addition of the equivalent of 1.5 L of 5 M NaOH solution. The slurry was heated with agitation to 85°C for 8 h. The main purpose of the leach is to dissolve aluminum, chromium, and phosphorus. The calculated hydroxide concentration during the leach was 2.8 M (targeted value was 3.0 M). After leaching, 1793 g of permeate was removed by dewatering, and the remaining 1.3 L of slurry had an undissolved solids content of 8.8 wt%.

The slurry was batch-rinsed three times with 1.2 L of inhibited water (0.01 M NaOH), with the targeted volume of each rinse equal to the slurry volume. The purpose of the rinse is to remove and displace the soluble aluminum, chromium, and phosphorus dissolved during the leach. A total of 3600 g of rinse solution was added during the three rinses, and 3812 g removed, including permeate samples taken between each rinse. After dewatering following the third rinse, representative slurry samples were taken for physical, chemical, radiochemical, and rheological analysis. The final concentration of undissolved solids measured 10.9 wt%.

During testing, the permeate flux and pressure, slurry axial velocity and pressure, and slurry temperature were monitored every 10 min. The slurry temperature was maintained at 25°C ± 5°C, except during the elevated-temperature leaching step. Flux data were corrected to 25°C, using the following formula (based upon the Andrade correlation (2) with constants provided by the client) to correct for viscosity and surface tension changes:

$$Flux_{25C} = Flux_T e^{2500(1/273+T-1/298)} \quad (1)$$

where $Flux_{25C}$ is the corrected permeate flux, and T is the temperature (in °C) at the flux measurement ($Flux_T$).

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FILTRATION RESULTS

The average permeate flux from the 13 test conditions (excluding the first 10 min of operation) is provided in Table 3. A graph of the permeate flux for the 7.6 and 17.9 wt% solids slurries as a function of time for conditions 1, 2, 3, 8, and 13 (the center points of the matrix) is shown in Fig. 3. The benefits from back pulsing in terms of increased flux are minor and short in duration. The flux immediately after back pulsing decreases with run order, but the flux beyond ~30 min after back pulsing shows little dependency with run order. This lack of dependency with run order is in contrast with previous cross-flow filtration studies on Hanford tank wastes (3–6), in which permeate flux degraded with time.

The permeate flux may be limited either by the viscous resistance of the fluid passing through the porous media or by the capability of the fluid to transport solids away from the filter membrane. Back transport of solids away from the membrane and into the bulk stream is required to prevent cake thickness from continually increasing. If mass transport of solids away from the filter membrane does not limit flow, then the permeate flux at steady state should vary proportionately with pressure in accordance with Darcy's Law for pressure filtration. If mass transport is causing the limiting resistance, then increased axial velocity will serve to increase the permeate flux as it aids in sweeping the solids away from the membrane surface.

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Table 3. Average permeate flux for low solids matrix

Condition #	Targeted axial velocity (ft/s)	Targeted transmembrane pressure (psid)	7.6 wt% Slurry average permeate flux (gpm/ft^2)	17.9 wt% Slurry average permeate flux (gpm/ft^2)
1	11	40	0.033	0.022
2	11	40	0.030	0.022
3	11	40	0.030	0.022
4	9	30	0.028	0.016
5	13	30	0.023	0.023
6	13	50	0.031	0.023
7	9	50	0.029	0.014
8	11	40	0.031	0.020
9	7	40	0.024	0.011
10	15	40	0.026	0.025
11	11	20	0.018	0.019
12	11	60	0.036	0.018
13	11	40	0.031	0.019

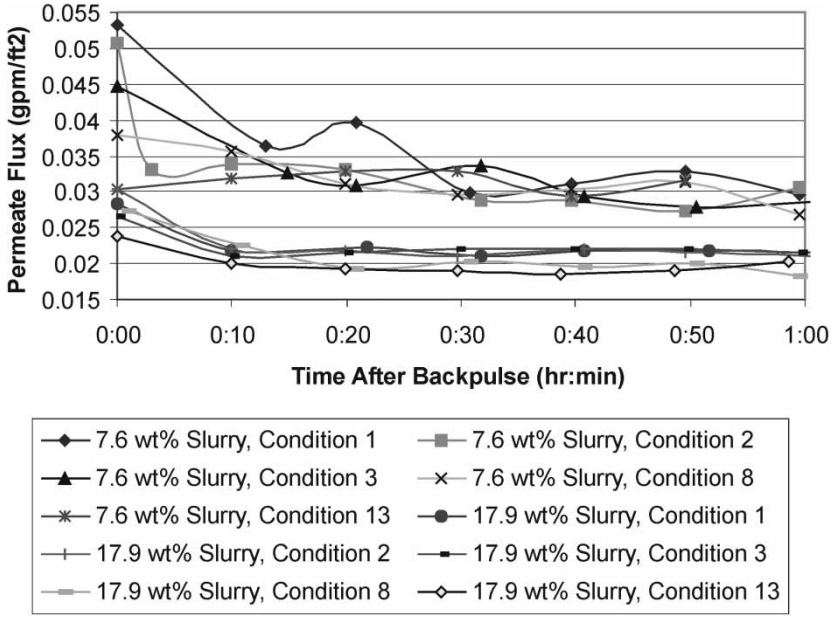

Figure 3. Permeate flux as a function of time.

Figures 4 and 5 show the average flux plotted as a function of TMP and axial velocity, respectively. For the 7.6 wt% slurry, the flux is principally dependent on the TMP, which is typical of low solids slurries and indicates that back transport of solids is not limiting. Indeed, the axial velocity shows almost no influence on the flux. In contrast with the low solids slurry, the

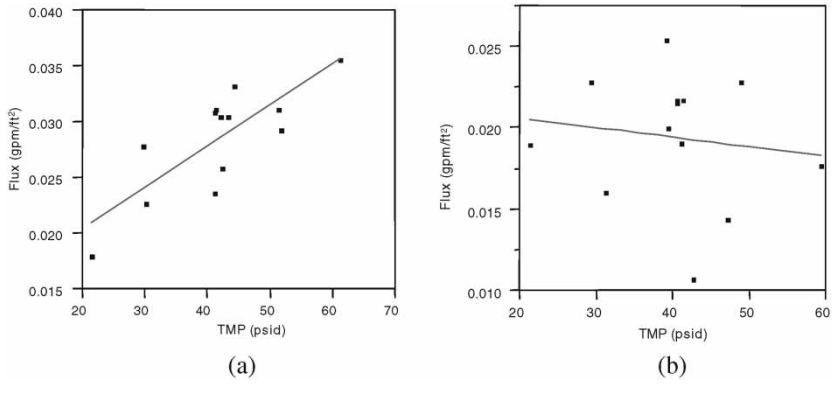

Figure 4. Effect of transmembrane pressure on permeate flux: (a) 7.6-wt% slurry and (b) 17.9-wt% slurry.

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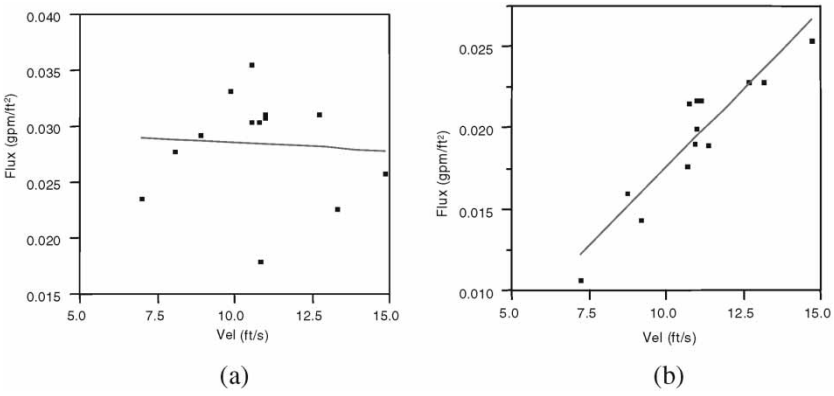

Figure 5. Effect of axial velocity on permeate flux: (a) 7.6-wt% slurry and (b) 17.9-wt% slurry.

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17.9 wt % slurry flux is principally dependent on the axial velocity, with almost no dependency on the TMP, indicating that back transport of solids is the limiting resistence.

The lines in Figs. 4 and 5 represent a linear regression through the data. In Figs. 4b and 5a, the linear regression is meant to highlight the lack of trend, which is so pronounced that the experimental design (test matrix, shown in Fig. 1) can be seen. In Fig. 4b, this lack of trend occurs because the TMP has so little impact on the 17.9 wt % slurry flux and velocity is so highly correlated. Likewise, in Fig. 5a, the lack of trend results from the low impact of the axial velocity on the 7.6 wt % slurry permeate flux and the highly correlated TMP. It is somewhat surprising that a slurry of 7.6 wt % solids behaved in a manner typical of much lower solids slurries.

Extended Runs and Dewatering of Untreated AZ-101

After the tests 1–13 with the low- and high-solids slurries, the feed was tested for ~10 h at 11 ft/s axial velocity and 40 psid TMP, without back pulsing. The results are shown in Fig. 6. After the first 100 to 200 min, the slurry flux generally stopped decreasing and held within a range. The range for the high-solids slurry showed less variability. The average permeate flux (excluding the first 200 min) was 0.026 and 0.016 gpm/ft^2 for the low- and high-solids slurry, respectively.

After each of the extended runs, the slurry was dewatered at 11 ft/s axial velocity and 40 psid TMP. The first dewatering brought the slurry from 7.6 to 17.9 wt % undissolved solids. The second dewatering, just prior to the first sludge washing, brought the slurry from 17.9 to 24.7 wt % undissolved solids. The system was not back pulsed during either dewatering.

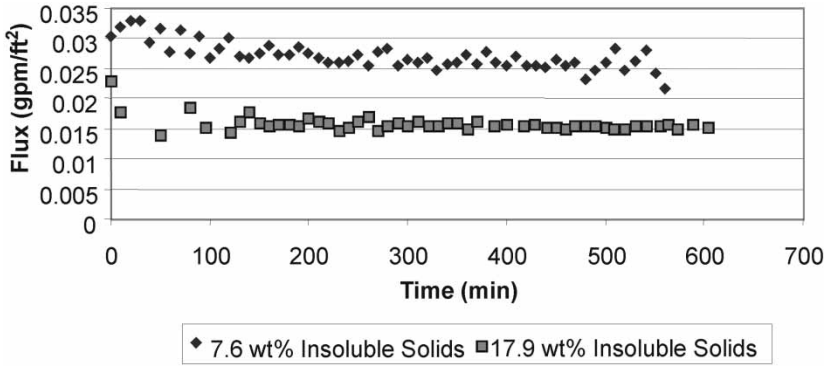

Figure 6. Extended run without back pulsing.

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Dewatering of Wash 1 and Wash 2

After the dewatering, the AZ-101 slurry (undissolved solids content = 24.7 wt %) was batch-washed twice with a volume of inhibited water (0.01 M NaOH) equal to the slurry volume (1 L). Because all dewaterings were conducted at 11 ft/s axial velocity and 40 TMP, the results are directly comparable. Figure 7 shows the permeate flux measured after wash 1 and wash 2 as a function of the log of the solids concentration, C_s. Also shown is the average flux measured from the untreated slurry during the 3 h extended runs. The flux during the dewatering from wash 1 and wash 2 was significantly higher than the dewatering of the original supernatant, resulting in a

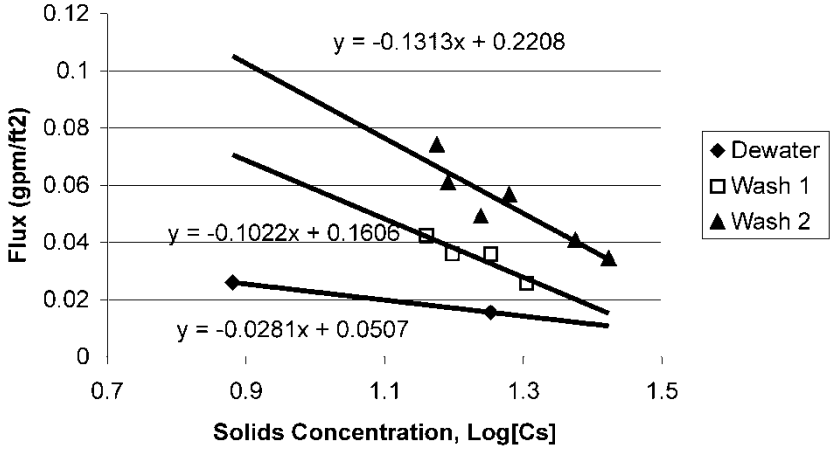

Figure 7. AZ-101 flux at various solids loadings.

steeper line. The increase in flux is attributed to the decreased viscosity of the fluid.

Figure 8 displays the permeate flux measured from the original slurry and during wash 1 and wash 2 as a function of the permeate viscosity. Not unexpectedly, the data indicate that the permeate flux is proportional to $(\text{viscosity})^{-1}$. The linear fits shown have a forced zero intercept.

Dewatering of the Leached Slurry and Subsequent Rinses

After the 8 h leach, the slurry was batch-rinsed three times, 1.2 L inhibited water (0.01 M $NaOH_{aq}$)/batch, and dewatered. All of the dewaterings were at 11 ft/s axial velocity and 40 psid TMP. The flux during each of these dewaterings is shown in Fig. 9. It is interesting that after the leach, the flux displayed little or no decrease with increasing solids concentration. The cause for this is unknown. The flux of each rinse was higher than the previous rinse, which again is most likely due to decreasing viscosity of the permeate. In all cases, the permeate flow rate was high and did not require any back pulsing.

SLUDGE WASHING AND CAUSTIC LEACHING RESULTS

The removal efficiencies for both the initial sludge washing and combined washing and caustic leaching of the nonradioactive components are shown in Table 4. The results in columns 2 and 3 are based on the measured amount removed in the permeate compared with the amount in the original slurry. The results for column 4 are based on the measured amount remaining in the slurry after treatment compared with the amount in the

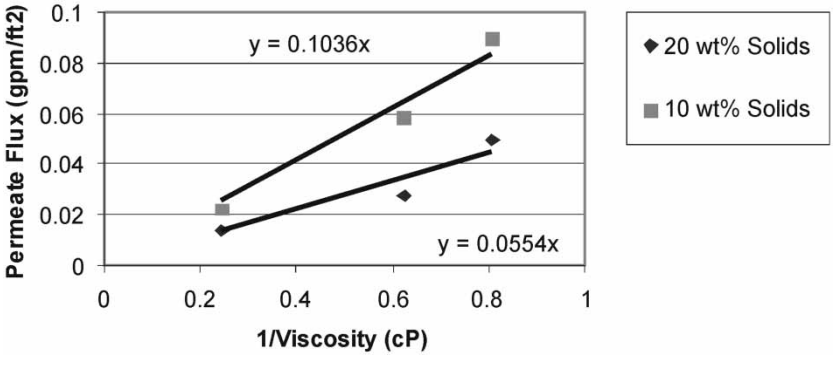

Figure 8. Effect of viscosity on the permeate flux.

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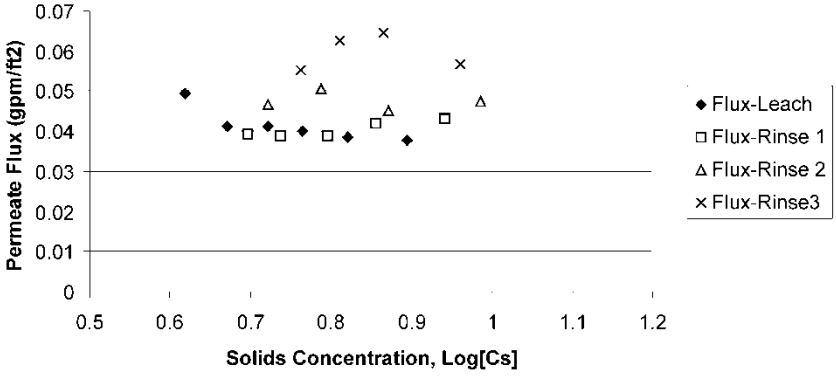

Figure 9. Dewatering of leached and rinsed AZ-101 slurry.

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original slurry. Comparing columns 3 and 4 provides a range of the amount removed based on what was measured as removed (column 3) and what was measured as remaining (column 4). The percent recovery, which is simply a mass balance for each analyte, is provided in column 5.

For the two washing steps, 1 L of inhibited water (0.01 M NaOH) solution was added to the slurry, and then an approximately equivalent amount of permeate was removed through the filter. Results indicate that 85% of the sodium was removed from the slurry during the water washing steps. The majority of the soluble anions (fluoride, nitrite, nitrate, sulfate, and oxalate) were removed during the first two water washes. Other components with significant removal efficiencies during the water wash were chromium with 56% removal and phosphorus with 42% removal.

The equivalent of 1.5 L of 5 M NaOH was added for the caustic leach, resulting in a calculated 2.8 M NaOH concentration. The three subsequent rinses were each performed with 1.2 L of 0.01 M NaOH, resulting in a calculated 1.45, 0.65, and 0.26 M NaOH solution, respectively. The overall amount removed in the washes, leach, and rinses is also shown in Table 4. While only 9% of the aluminum was removed during the washing, a total of 70% was removed by washing and caustic leaching. Caustic leaching also significantly improved the amount of chromium and phosphorus removed.

Overall, the recoveries were very good. The recovery can be represented as:

$$\text{Recovery} = \frac{\begin{array}{r}(\text{Analyte}_{\text{wash}} + \text{Analyte}_{\text{leach}} + \text{Analyte}_{\text{rinse}} \\ +\text{Analyte}_{\text{sampling}} + \text{Analyte}_{\text{residue}})\end{array}}{\text{Analyte}_{\text{initial·sludge}}} \qquad (2)$$

The recovery deviates from 100% because of the variability in the analysis, which is estimated to be $\pm$ 15%. Replicate samples were not

Table 4. Selected component removal efficiencies

Analyte	Removed in wash (%)	Total removed in wash, leach, and rinse (%)	Total removed in wash, leach, and rinse (%) (Based on slurry residue)	Recovery (%)
Al	9	70	75	99
Ba	0	0	6	107
Cd	0	0	8	104
Cr	56	93	64	132
Fe	0	0	8	104
La	0	0	11	101
Mn	0	0	11	101
Na	85[a]	91[a]	91	110
Nd	0	0	13	99
Ni	0	0	15	97
P	42	60	40	128
Sr	0	0	9	103
Zr	0	0	<0	121
^{137}Cs	>100	>100	93	137
^{154}Eu	<0.22	<0.60	2	110
^{155}Eu	<4.6	<8.3	0	120
^{241}Am (AEA)	0	0	5	107
^{90}Sr	0	0	8	104
C_2O_4	>100	>100	97	183
F	>100	>100	98	159
NO_2	>100	>100	99	140
NO_3	>100	>100	100	134
SO_4	66	93	99	94

[a]Because of the significant sodium added during the leaching, Na numbers are calculated based on what was remaining in the sludge rather than what was removed.

within the scope of this study; consequently, the specific samples contributing to the error were not identified.

The sludge washing and leaching results were thermodynamically modeled using the Environmental Simulation Program (ESP), version 6.6 (7). The model was run using the Felmy database (8), which is based upon Pitzer Equations (9,10), and the nuclear database developed at Hanford, which is based on the Bromley Zematis approach. The results of the model were in good agreement with the experimental data for the principal components in the slurry. The interested reader is referred to reference (8) for results.

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The insoluble radioactive component concentrations provide a means of measuring the capability of the filter to separate the undissolved solids from the liquids. The isotope ^{241}Am is basically insoluble in caustic solutions, and its concentration was measured for all permeates and slurries; consequently, it was used to measure filter removal efficiency in terms of a decontamination factor (DF) (DF = concentration in the slurry/concentration in permeate) for each step of the process. The ^{241}Am DFs were approximately 803,000 for the original slurry; the water wash DF was 2,160,000; the caustic leach DF was 935,000; and the final rinse DF was 40,300, for an average of 985,000. These DFs indicate good solid/liquid separations using the Mott 0.1-μm sintered metal filter; and in all cases, the resulting immobilized low-activity waste would not breach the Class C limit of 100 nCi/g for TRU (11).

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CONCLUSIONS

The results of the tests performed on sludge from Hanford Tank AZ-101 indicate that cross-flow filtration provides excellent separation of solids and liquids. The permeate flux was higher than the targeted value of 0.014 gpm/ft^2, except for a few of the conditions tested with the 17.9 wt % solids slurry. It was demonstrated that the permeate flux of the as-received and washed slurries declined linearly with the log of the undissolved solids concentration; and that as the undissolved solids concentration of the slurry increases, the targeted permeate flux can be achieved simply by increasing the axial velocity. Pretreatment of the sludge by washing removed 85% of the sodium, 56% of the chromium, 42% of the phosphorus, the majority of soluble anions (fluoride, nitrite, nitrate, sulfate, and oxalate), and ^{137}Cs. While only 9% of the aluminum was removed during washing, a total of 70% was removed by washing and caustic leaching. Caustic leaching also significantly improved the amount of chromium and phosphorus removed. Overall, the pretreatment reduced the mass of sludge by 56%. In addition, the results verify the segregation of TRU from the liquids by means of filtration. No significant problems were encountered during the testing, suggesting that the pretreatment and filtration of AZ-101 waste will not pose any unanticipated challenges to Hanford's Waste Treatment Plant.

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