Sludge Settling Rate Observations and Projections at the Savannah River Site – 13238

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ABSTRACT

Since 2004, sludge batches have included a high percentage of stored sludge generated from the H- modified (HM) process. The slow-settling nature of HM sludge means that the settling is often the major part of the washing tank quiescent period between required pump runs to maintain flammability control. Reasonable settling projections are needed to wash soluble salts from sludge in an efficient manner, to determine how much sludge can be washed in a batch within flammability limits, and to provide composition projections for batch qualification work done in parallel with field preparation. Challenges to providing reasonably accurate settling projections include (1) large variations in settling behavior from tank-to-tank, (2) accounting for changing initial concentrations, sludge masses, and combinations of different sludge types, (3) changing the settling behavior upon dissolving some sludge compounds, and (4) sludge preparation schedules that do not allow for much data collection for a particular sludge before washing begins. Scaling from laboratory settling tests has provided inconsistent results.

Several techniques have been employed to improve settling projections and therefore the overall batch preparation efficiency. Before any observations can be made on a particular sludge mixture, projections can only be made based on historical experience with similar sludge types. However, scaling techniques can be applied to historical settling models to account for different sludge masses, concentrations, and even combinations of types of sludge. After sludge washing/settling cycles begin, the direct measurement of the sludge height, once generally limited to a single turbidity meter measurement per settle period, is now augmented by examining the temperature profile in the settling tank, to help determine the settled sludge height over time. Recently, a settling model examined at PNNL [1,2,3] has been applied to observed thermocouple and turbidity meter readings to quickly provide settling correlations to project settled heights for other conditions. These tools improve the accuracy and adaptability of short and mid-range planning for sludge batch preparation.

INTRODUCTION

Radioactive waste at the Savannah River Site (SRS) is stored in aging underground waste storage tanks. This waste is a complex mixture of insoluble solids, referred to as sludge, and soluble salts. The sludge is currently being stabilized in the Defense Waste Processing Facility (DWPF) through a vitrification process that immobilizes the waste in a borosilicate glass matrix. Sludge feed to DWPF is prepared in batches of about 500,000 gallons with about 10 to 17 weight percent of insoluble solids. Preparation includes washing of the sludge by repeatedly adding water or dilute supernatant liquid, gravity settling the sludge solids to the bottom portion of the waste tank, and then decanting the solids-free upper layer with a telescoping steam jet. The sequence is repeated until the pre-determined target salt content that supports DWPF operation is attained. A typical sludge batch provides feed for DWPF for about two years.

Projection of settled sludge heights as a function of settling time is of particular importance for efficient and timely sludge batch preparation at SRS. Due to the hydrogen generation rates in the sludge slurry, periodic slurry pump runs are required to ensure release of accumulated hydrogen for flammability control [4]. Therefore, washwater addition, settling, and decant steps that remove soluble salts must be planned to fit within the quiescent time period between pump runs.

Washwater addition volumes and settling times must be planned to meet quiescent time limitations, make efficient use of available decant storage space, integrate with transfer route availability, and minimize batch preparation time to the extent practical. By knowing the height of the settled turbid sludge layer as a function of washwater volume added and settling time provided, one knows how much and when decant volume is available. The volume and time can be selected to best meet the Facility conditions and schedules. Furthermore, hydrogen generation rates increase with nitrate and nitrite dilution for successive washes [4], so the shrinking quiescent time and settling window must be properly anticipated to the degree that the planned sludge mass can be washed and concentrated enough to meet the DWPF feed composition target. Also, the degree of settling that can be attained impacts the final batch slurry composition, which must be projected for batch qualification studies. A number of settling models, measurement techniques, and calculation approaches have been applied to enhance settling projections and therefore provide better bases for sludge preparation plans.

DISCUSSION

Measurement of Settled Sludge Height

The detector head of the device used to measure the height of a tank's settled layer consists of a sealed assembly with a small light bulb mounted a few inches from a photoresistor. Since light measured is transmitted directly through the slurry rather than scattered from a 90 degree angle, it is not a typical turbidity meter. A cable long enough to lower into the tank is connected to a six volt battery and an ohmmeter. A weight at the detector end of the cable and a measuring tape along the cable length allow the detector to be lowered to specific depths in the tank. The detector and cable are inexpensive and disposable. As the detector is lowered through the liquid, a sudden increase on the order of hundreds of kilo-ohms indicates a minimum of about 0.05 weight percent solids [5], well below the maximum solids content allowed to be classified as "non-sludge slurry". The movable transfer jet is placed at least 24 inches above the identified turbid height to ensure that no significant amount of solids are entrained in the decant stream.

Projecting Settling Results for PUREX Sludge

The first four sludge batches prepared for feed to DWPF consisted primarily of PUREX sludge from processing to recover uranium and plutonium from irradiated targets or fuel slugs containing a core of natural or depleted uranium. E.D. Lee [6] applied a settling model provided by Eli Barnea [7] that uses data of settled height vs. time to determine settling rates for each of three solids concentration regimes. The "unhindered" or "constant rate" regime exhibits a uniform rate of settling of the liquid/sludge interface. This is followed by a "hindered" regime where the increased solids concentration increasingly impedes the downward flow of solids particles through the upward flow of interstitial liquid. Finally, a "compressive" regime occurs where very slow settling continues due to the pressure of the settled solids on lower parts of the settled layer. The model holds that plotting the logarithm of the interface settling rate vs. the dimensionless settled height, or

$$(Z_t - Z_\infty)/Z_\infty ,$$

where Z_∞ is the fully settled height, and Z_t is the height at a given time,

identifies a linear relationship for each regime. Application to Sludge Batch 1A (SB1A) settling data yielded the relationships shown in Table I [6]. Z_∞ was obtained by direct measurement after about a year of settling.

Table I: SB1A (PUREX) Sludge Layer Height vs. Settling Time

Settling Mechanism	Equation (Settling time t in days, Z in inches)	Applicable Range
Constant Rate	$t = (Z_i - Z_f)/144 \quad (Eq.1)$	$Z_t \geq 2.85\ Z_\infty$
Hindered	$t = \frac{Z_\infty}{1.827}\left[10^{(4.429-1.827\ Z_f/Z_\infty)} - 10^{(4.429-1.827\ Z_i/Z_\infty)}\right]/24\ (Eq.2)$	$Z_t \leq 2.85\ Z_\infty$, $Z_t \geq 1.78\ Z_\infty$
Compression	$t = \frac{Z_\infty}{3.029}\left[10^{(6.631-3.029\ Z_f/Z_\infty)} - 10^{(6.631-3.029\ Z_i/Z_\infty)}\right]/24\ (Eq.3)$	$Z_t \leq 1.78\ Z_\infty$

In Figure 1, the settling model output is compared to the test data from which it was generated. This model has been used to predict the settling of other PUREX sludge relatively well. For example, Table II shows settling observations from SB3. Two turbid height readings are provided for one sludge mass present in the settling tank, from the same initial slurried waste height. Two other readings are provided after additional sludge mass had been sent to the tank, settled from two different initial slurried waste heights. In both mass cases, the first turbid height reading, corresponding settle time, and initial waste height are substituted into the PUREX model of Table I to obtain a value for Z_∞. Then, that value is used in the model with the next setting time and initial slurry height to predict Z_t for the next settle. Table II shows that the predicted Z_t is close to the measured Z_t, even though it was projected from a measurement taken when settling was quite rapid and therefore potentially less accurate.

Figure 1: PUREX Settling from SB1A Measurements

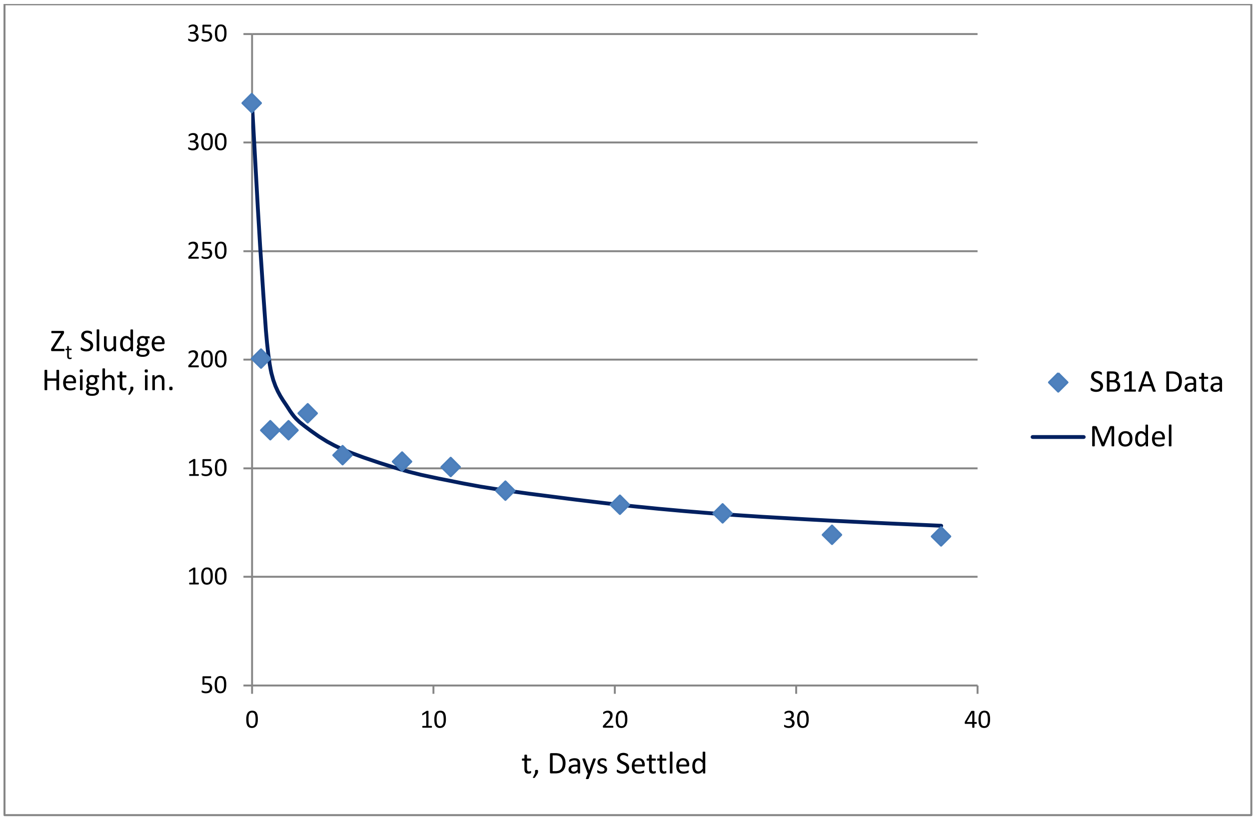


Table II: SB3 Settling Prediction using the PUREX Model

	Data			Prediction	
	Z_0	t	Z_t	Z_∞	Z_t
Mass A	273.5 273.5	4.75 16.3	109.3 **93.8**	51.4	**100.5**
Mass B	291.3 226.5	5.95 20.0	107.1 **88.1**	51.7	**90.9**

Another way to project settling results for an estimated PUREX sludge mass for which no previous settling measurements have been obtained is to use an historical average for the settled sludge compaction after the sludge is well beyond the rapid settling period. For example, an observed compaction of 292 grams per liter of sludge solids after 20 days of settling is a documented [8], somewhat average PUREX behavior. Applying that value to an estimated sludge mass of 250,000 kg would provide a 20-day settled height in Tank 51 (with a tank diameter corresponding to 3510 gal/in) of

$$250000 \text{ kg} / (0.292 \text{ kg/L}) / (3.785 \text{ L/gal}) / (3510 \text{ gal/in.}) = 64.4 \text{ in.}$$

For a given initial slurry height Z_0, time t of 20 days, and final height Z of 64.4 in., the equations of Table I can be used to determine Z_∞, which in turn can be used to construct a settling projection for any Z_0 and t.

Projecting Settling Results for High-Heat H-Modified Sludge

The H-modified (HM) process is similar to the PUREX process, but was tailored for recovery and separation of uranium and neptunium from burned enriched uranium fuel. HM sludge has more aluminum and less iron than PUREX sludge.

Since SB3 processing by DWPF in 2004, sludge batches prepared have consisted primarily of HM sludge. HM sludge has demonstrated much slower rates of settling than PUREX sludge. The slower settling rates means that more settling time is required. Settling time is often limited by the slurry pump run frequency required for flammability controls, and sludge batch sizes must be smaller in general to credit more tank vapor space for hydrogen accumulation in order to extend settling times. Slow-settling behavior, higher radiolytic heat content, more stringent flammability controls, and faster DWPF production rates have all made settling projections more critical.

Settling of sludge for SB4 from Tank 11 is displayed in Figure 2 alongside settling of the same 258,000 kg mass for a typical PUREX sludge. The SB4 settling curve shown was fitted from historical data by A. L. Pajunen:

$$Z_t = 50\left[1 - ln\left(0.00934 \times 1.22638 \times \frac{24t}{50} + e^{-\frac{1.22638(Z_0-50)}{50}}\right)/1.22638\right] \qquad (Eq. 4)$$

The model is of the same type applied previously to PUREX sludge. In this case, no compressive settling mode is considered, since that mechanism occurs outside of the normal range of settling times and is of little importance to sludge batch preparation. Table III demonstrates that Eq. 4 predicts actual SB4 sludge settling well.

The last data point in Table III was actually obtained during SB5 preparation, for which a large part of the washed SB4 sludge was retained for Low Temperature Aluminum Dissolution (LTAD). That settled height prediction employed a method to apply to settling results from one

Figure 2: SB4 (HM) Settling Compared to PUREX Settling

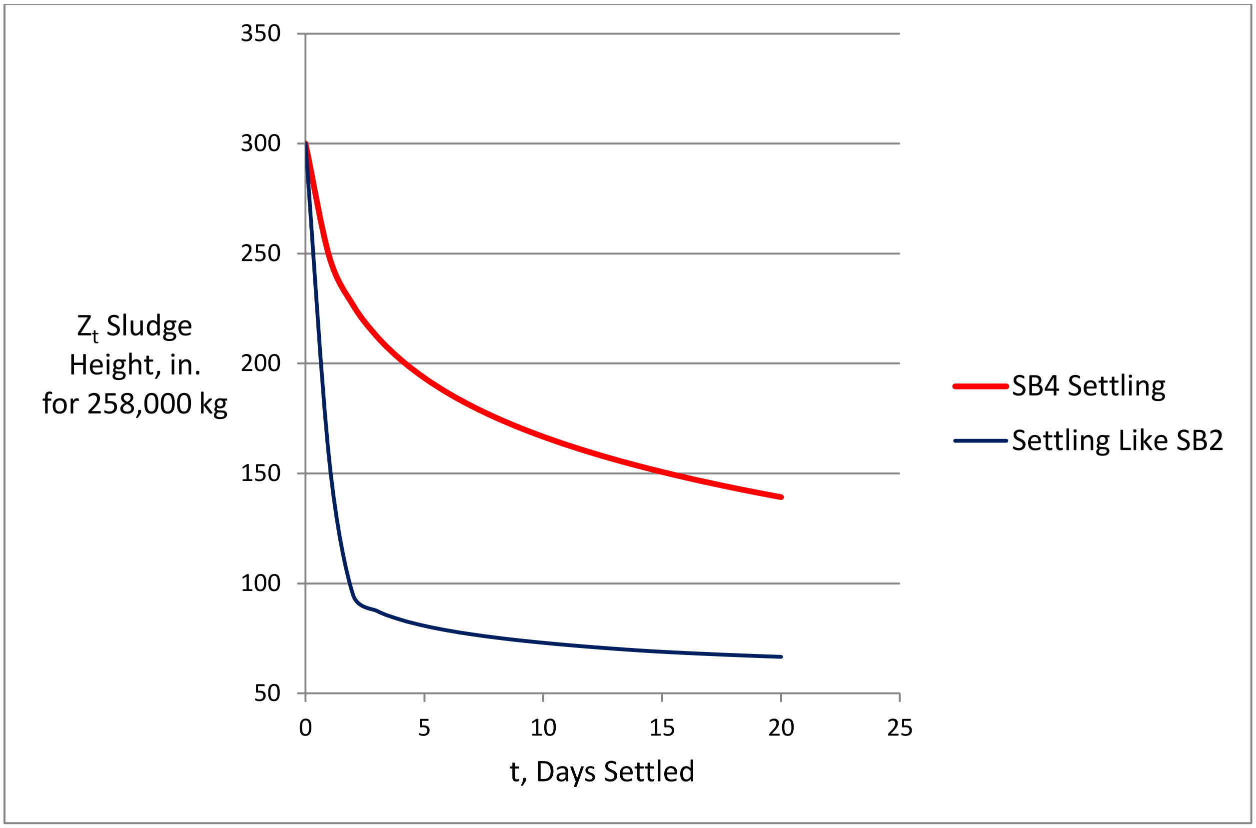


Table III: SB4 and Pre-LTAD Settling Model Predictions vs. Actual

	Date	Z_0, in.	t, days	Z_t measured, in.	Z_t predicted, in.
SB4	9/7/05	216.4	24	139.1	127.7
	1/3/06	177.6	46	102.3	99.5
	1/12/06	177.6	55	103.1	93.3
	2/27/06	217.6	22	123.1	130.9
	5/8/06	218.5	18	131.2	138.2
	6/7/06	218.5	48	101.1	101.9
	7/5/06	179.6	21	124.6	125.5
	7/19/06	179.6	35	108.6	109.2
Pre-LTAD	10/4/07	140.1	26	87.1	88.4

sludge mass to a different mass of the same sludge. Suppose that a model is used to calculate the settled height of a Mass M as a function of initial slurried waste height and settling time:

$$Z_{M,t} = f(t, Z_0) \qquad (Eq.5)$$

Consider that the plot of settled height vs. time will depend on how much sludge solids mass is present (M), and the initial sludge concentration (specified by M and Z_0). Now suppose that a new mass M' of the same sludge is obtained by addition or removal from the tank. The fully settled sludge height will be proportional to the amount of the sludge solids, so multiplying the expression for the settled sludge height by M'/M would appropriately represent the fully settled end of the new settling curve. However, the expression would not give the correct height at t = 0 or other times. For example, applying a factor of M'/M to the expression in Eq. 5 would result in an initial $Z_{M,t}$ equal to M'/MZ_0 at t = 0, when it must be Z_0 by definition. Applying a scaling factor of M/M' to the variable Z_0 in the expression keeps the impact of Z_0 in the proper proportion with M' so that the correct initial concentration is represented:

$$Z_{M',t} = f(t, Z_0 \times M/M') \times M'/M \qquad (Eq.6)$$

Before the 10/4/07 measurement of Table III was obtained, the 258,300 kg of solids analyzed in SB4 was reduced to 187,900 kg as estimated from the remaining volume after transfer of some of the fully slurried contents. Instead of Eq. 4, the expression predicting the 26-day settled height, applying Eq. 6 to Eq. 4, was:

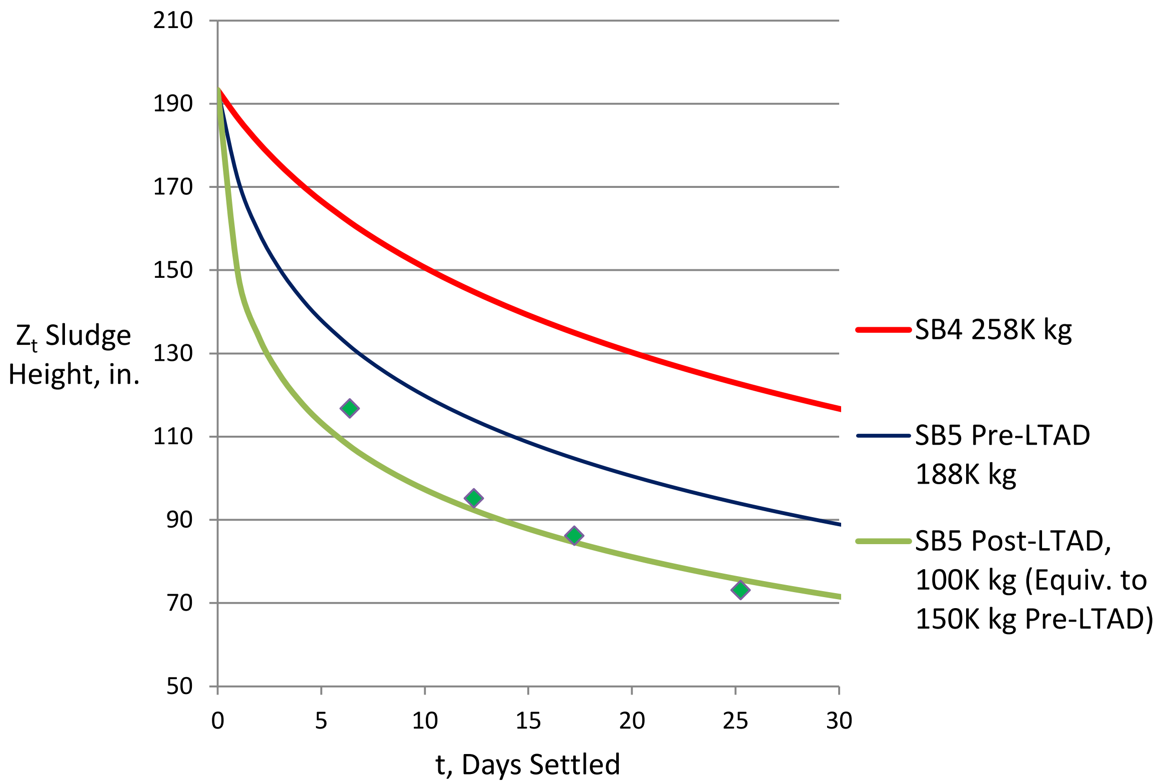
$$Z_t = 50\left[1 - ln\left(0.00934 \times 1.22638 \times \frac{24t}{50} + e^{-\frac{1.22638(Z_0 \times 258300/187900 - 50)}{50}}\right)/1.22638\right] \times 187900/258300 \qquad (Eq.7)$$

Besides Tank 11 sludge that was used to prepare SB4 and SB5, another HM sludge that has been washed is that from Tank 12, most of which went into SB6. It was evident that Tank 12 sludge was much slower-settling than even Tank 11, although only a bit of settling data was collected before the sludge was altered by the aluminum dissolution process. Both sludges had high boehmite (AlOOH) content, but Tank 12 had much more than Tank 11 [9, 10]. Apparently, settling behavior of different HM sludges can vary widely, so a single expression like Eq. 4 for HM sludge is inadequate of estimating all HM sludge settling behavior.

Observations of the Impact of Aluminum Dissolution on Settling

LTAD was performed on the 187,900 kg remaining in Tank 51 after SB4 preparation. Dissolution in caustic at around 60 ^oC for 46 days dissolved about 88,000 kg of boehmite [9]. Post-dissolution settling results, all from near the same initial height, are shown in Figure 3. For comparison the settling curves from the same height for SB4 (using Eq. 4) and SB5 pre-dissolution sludge (using Eq. 7) are shown as well.

Figure 3: SB5 Post-LTAD Settling Compared to Pre-LTAD Masses



After SB5 LTAD was completed, settling results indicated a settling curve similar in character to that in Eq. 4 and Eq. 7 for the SB4 solids from which the remaining solids were derived. Figure 3 shows that the settling curve resembles what one would expect from a lower mass of the SB4 solids, despite the fact that little more than just the boehmite component was dissolved. Also, the 100,000 kg of solids remaining after dissolution settled as one would roughly expect for about 150,000 kg of SB4 solids. While the settling was of course faster for the much lower mass of remaining sludge, the post-LTAD sludge settles much slower than SB4 if compared at the same solids concentration.

Another observation can be made on the settling of sludges having undergone aluminum dissolution. Settling rates generally improved with successive washes over time. Table IV shows the settling results of SB5 and SB6 during the washing period when sludge was not transferred into or out of the washing tank. In the case of both sludge batches, the sludge remaining after aluminum dissolution was mixed with a smaller proportion of PUREX sludge before washing.

Table IV: Settling Results during Washing of SB5 and SB6

SB5	Date	3/31/08	4/3/08	4/10/08	4/30/08	5/6/08	5/29/08	7/14/08
	Z_0, in.	165.3	165.8	165.3	158.7	158.7	160.2	159.5
	t, days	10.0	13.2	20.0	11.5	17.4	15.4	14.1
	Z_t, in.	105.1	99.4	86.6	97.1	87.1	82.1	85.1
	Date	8/4/08	8/25/08	8/27/08	9/8/08	10/1/08	10/4/08	10/20/08
	Z_0, in.	152.7	166.0	166.0	146.3	135.2	135.2	117.1
	t, days	14.3	11.3	13.1	8.1	9.1	11.9	11.2
	Z_t, in.	88.6	77.1	72.1	71.4	95.1	93.1	85.1
SB6	Date	11/3/09	11/30/09	12/8/09	1/4/10	1/27/10	2/22/10	3/17/10
	Z_0, in.	145.2	184.6	184.6	183.7	186.0	186.1	186.7
	t, days	26	11	19	16.5	14.8	13.9	11.5
	Z_t, in.	119.1	143.1	131.1	127.1	127.1	119.1	119.1
	Date	5/5/10	6/3/10					
	Z_0, in.	185.9	184.7					
	t, days	18	16					
	Z_t, in.	107.1	95.1					

Table IV shows settles of both sludge batches in chronological order. When multiple turbidity readings are taken during the same settle, those table entries are grouped by shading. Fortunately, many settles were begun from near the same initial slurried waste height Z_0. Therefore it is possible to compare settled heights Z_t when the settling times are about the same, or to compare settling times t when settled heights are about the same.

For SB5, it appears that settling trend was fairly consistently faster/deeper (with the possible exception of the 8/4/08 reading), until the last three settles. Laboratory washing of the batch qualification sample demonstrated the same improvement followed by a rise in the settled height near the end of washing. One explanation proposed to explain the higher sludge height at the end of SB5 washing is dilution of the batch to the point that aluminum in solution could have re-precipitated to a degree. SB6 shows consistent settling improvement over its washing period.

The reason for the general increase in settling during the washing of SB5 and SB6 is not known. Perhaps it is related to the aluminum dissolution operation to which both batches were subjected. It has been described how the sludge remaining after aluminum dissolution appears to settle slower (for a given mass) than pre-dissolution sludge. During LTAD, slurry pumps are operated fairly continuously for many weeks in order to raise the waste temperature. The reduced settling rate of the remaining sludge may be a result of the high amount of shear imposed on the sludge solids. It could also be that those solids slowly re-consolidate over time or with repeated settling cycles.

It may be noted that SB7A, which included a significant fraction of Tank 12 sludge but also various sources of PUREX sludge, also exhibited a trend toward faster settling over time. SB7A included a sizable amount of sodium oxalate solids from tank chemical cleaning operations. Sodium oxalate is sparingly soluble and will dissolve during the latter sludge washes. This could largely explain the improving settling trend for that sludge batch.

Projecting Settling Results for Sludge Blends

A method has been devised to estimate the settling rates of a blend of two different sludges for which setting models already exist. The method assumes that the two sludge types still behave independently upon combination. While that may be an oversimplification, it at least provides a starting basis for anticipating combined settling behavior, when no previous settling measurements are available.

The methodology can be understood by envisioning the sludge receipt tank divided vertically into two parts, each part containing a particular mass of different sludge. Suppose that each sludge has a model that describes its settled height as a function of intial slurried waste height and settling time:

$$Z_1 = f_1(t, Z_0) \qquad (Eq. 8)$$

$$Z_2 = f_2(t, Z_0) \qquad (Eq. 9)$$

Consider that the settled height of each sludge will be greater if confined to only part of the area of the tank. Also consider that in order to keep the variable Z_0 in proportion to represent the correct initial sludge concentration, it must be scaled by the same area ratio as the overall settling function. If A is the fractional area apportioned to a particular sludge,

$$Z_1 = f_1(t, Z_0 A_1)/A_1 \qquad (Eq. 10)$$

$$Z_2 = f_2(t, Z_0 A_2)/A_2 \qquad (Eq. 11)$$

This scaling is analogous to that used for Eq. 6. In both cases, the adjustment is accounting for a different solids mass per unit area. Now, since the two fractional areas sum to 1, and the two "areas" of the tank must settle to the same height in the same time,

$$f_1(t, Z_0 A_1)/A_1 = f_2\left(t, Z_0\,(1 - A_1)\right)/(1 - A_1) \qquad (Eq. 12)$$

The projected combined settling height corresponds to the value of A_1 that satisfies Eq. 12, which can be substituted into Eq. 10 or Eq. 11 to obtain the height.

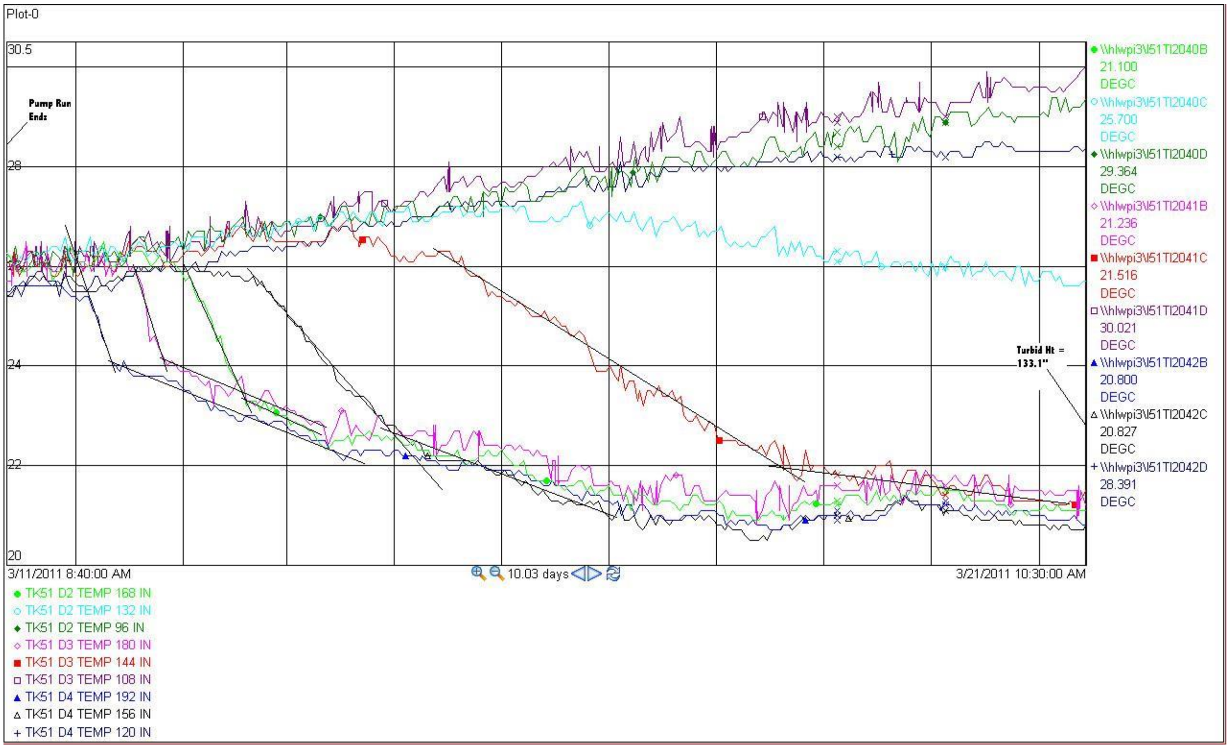
One application of this method has been to initially project results of combining PUREX and high-heat HM sludge [11]. It has also been used to model the effect of dissolving sodium oxalate solids on settling and washing of sludge. Settling results for the neutralization tank that collected spent oxalic acid from chemical cleaning of Tanks 5 and 6 and had a high percentage of sodium oxalate solids seemed to match the PUREX settling model well. Assuming that sodium oxalate solids settle like PUREX sludge, the composite settling model could be adjusted for the expected dissolution of sodium oxalate as washing proceeded.

Tools to Improve Short-Term Settling Projections

As discussed earlier, accurate sludge settling predictions help to define an efficient washing scheme and to project final batch composition. Even when applying the historical data and reasonable calculation techniques, there are significant uncertainties and occasional surprises. One limitation is that for practical reasons (time, cost, and personnel exposure) the number of turbid height measurements is restricted to the minimum necessary to process the batch. Receipt of a “new” sludge, aluminum dissolution operations, and sparingly soluble solids from chemical cleaning operations all introduce more uncertainty.

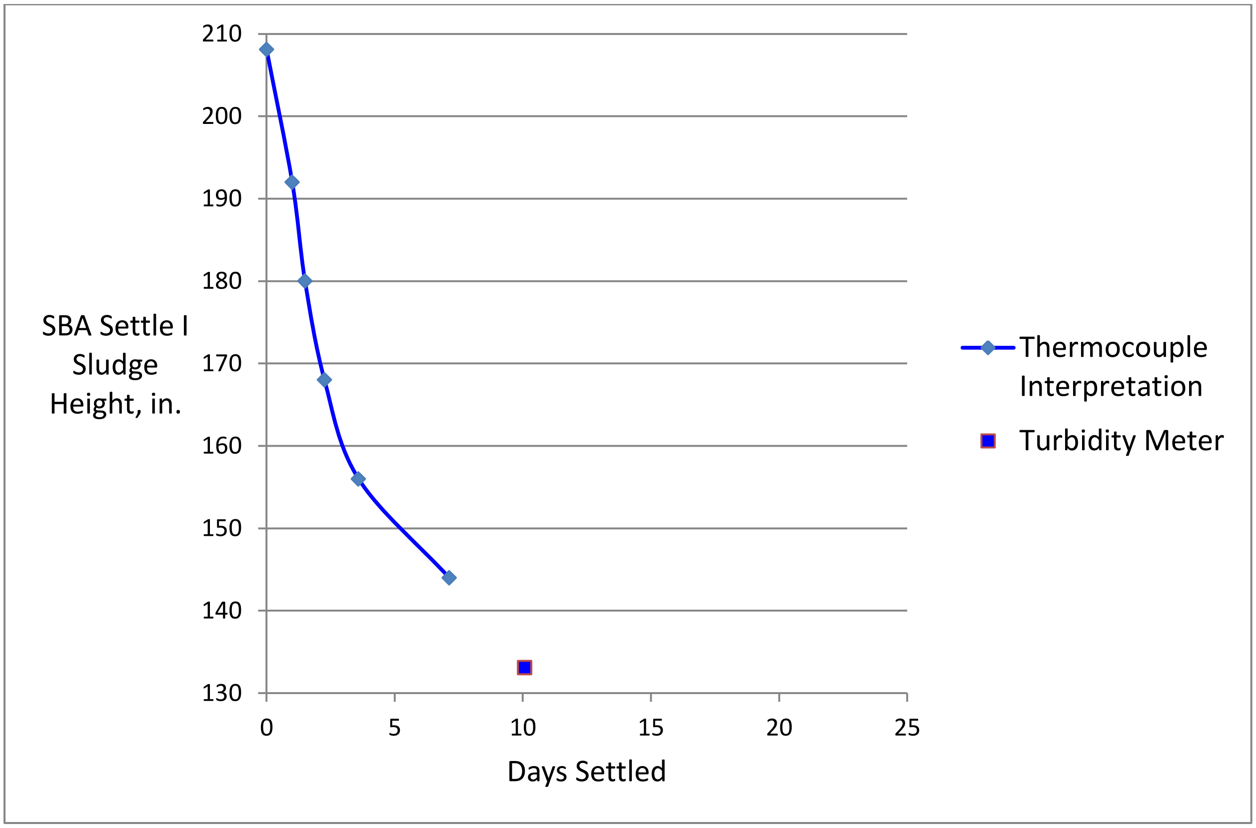
One method has recently been applied to determine the settling behavior of sludge as it settles instead of collecting disparate pieces of turbid height data over an extended time. Tank 51 thermocouples, suspended at various elevations, seem to indicate which elevations are in the hotter settled sludge layer, and which are in the cooler liquid layer. Figure 4 shows an example of the thermocouple readings over time as sludge settles. When the sludge is fully slurried (left axis), the thermocouple outputs are clustered together, as expected. As settling proceeds, the higher thermocouples, one by one, diverge from the hotter lower ones that are near the heat-generating sludge layer. Finally, there are two temperature clusters, one seemingly representing the sludge layer, and one representing the liquid layer. A thermocouple or two may output an intermediate temperature, indicating a transition region.

Figure 4: Typical Tank 51 Thermocouple Response While Settling



Interpretation of the thermocouple output is somewhat subjective, but if plotting the thermocouple height against the time difference between the end of the pump run and the first temperature deflection after divergence from the high-temperature cluster, one gets a plot as in Figure 5. The plot resembles a credible settling curve. When attempting to use a plot like Fig. 5 to predict a turbidity height measurement, a 5 to 10 inch discrepancy is not unusual. In this case, the turbidity measurement of 133 inches occurred when the 132-inch thermocouple output had diverged from the bulk sludge temperature was not yet near the liquid-phase thermocouples. Nevertheless, the thermocouples provide a useful indication of the settling progress. A 5 to 10 inch discrepancy is not excessive for a sludge of unknown settling behavior. Also, the relatively rapid initial settling rate can be estimated without taking multiple turbidity readings.

Figure 5: Tank 51 Settling from Figure 4 Thermocouple Interpretation



Another tool recently used to facilitate near-term settling projections is a simplified settling model that requires much less data than is needed to construct and fit settling models of the type represented by Eq. 1-3 and Eq. 4. The model was presented by Renko [2,3] and described in a PNNL data summary [1]. It does not model a constant-rate or compressive settling modes, which are less likely to be of value for washing of slow-settling sludges.

$$\frac{Z_t}{Z_0} = \frac{C\Phi_0}{\alpha} + \left(1 - \frac{C\Phi_0}{\alpha}\right) e^{-\frac{\alpha}{\Phi_0 Z_0}t} \qquad (Eq.13)$$

where: C and α are constants for a particular sludge found by fitting the model, and Φ_0 is the initial solid-phase volume fraction in the mixed suspension

Eq. 13 can be re-arranged as

$$Z = \frac{Z_0\Phi_0 C}{\alpha} + \left(Z_0 - \frac{Z_0\Phi_0 C}{\alpha}\right) e^{-\frac{\alpha}{\Phi_0 Z_0}t} \qquad (Eq.14)$$

In Eq. 14, it can be seen that as t approaches ∞, Z approaches $Z_0\Phi_0 C / \alpha$, so

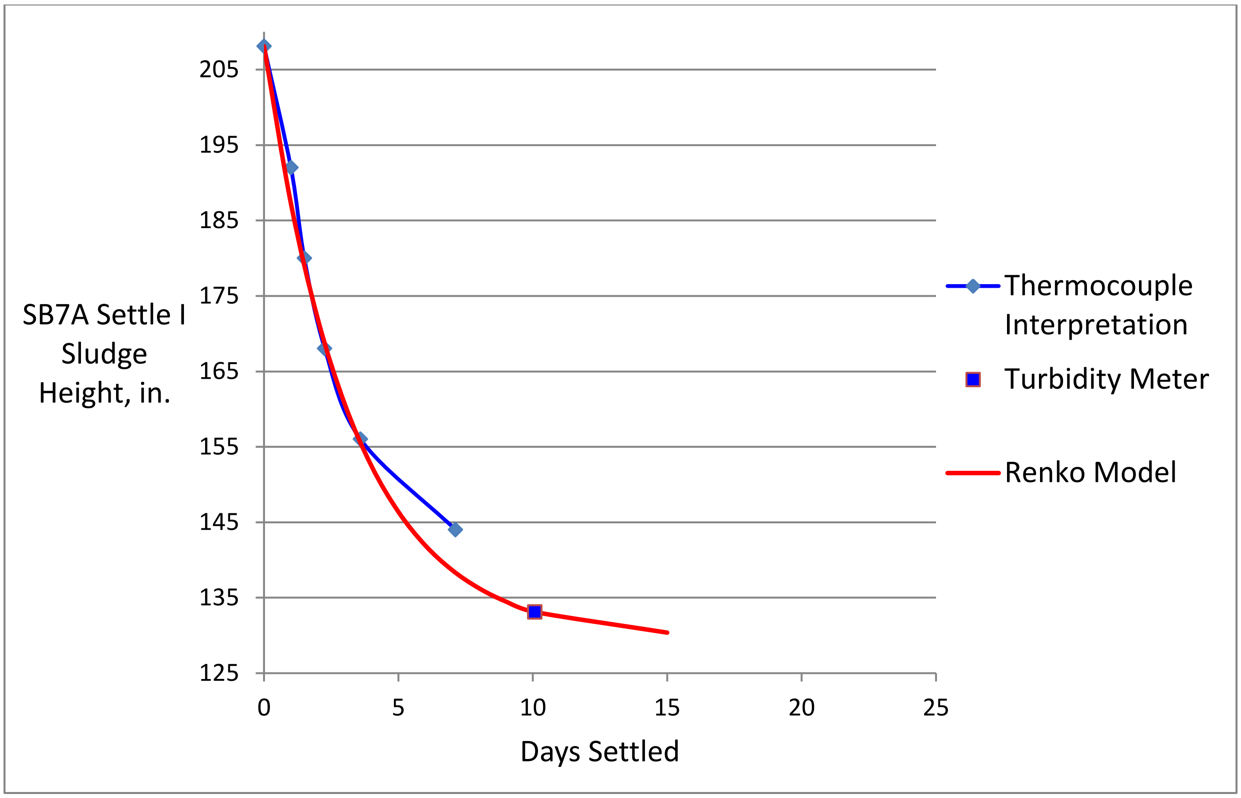
$$Z_\infty = Z_0 \Phi_0 C/\alpha \qquad \text{(Eq. 15)}$$

Substituting Eq. 15 into Eq. 14,

$$Z_t = Z_\infty + (Z_0 - Z_\infty)e^{-\frac{C}{Z_\infty}t} \qquad \text{(Eq. 16)}$$

In Figure 6, the data from Figure 5 is fitted to Eq. 16. This demonstrates that with the thermocouple data, a single turbidity meter reading (preferable taken after the settling rate has begun to decay), and Eq. 16, even limited settling experience can be used to provide a more informed projection for a future wash than a single turbidity meter reading alone.

Figure 6: Example Application of Renko Model to Preliminary Settling Data



CONCLUSIONS

Historical experience with settling of PUREX and HM sludge has been documented, but those models alone do not provide a basis for timely and accurate predictions given the wide range of settling behavior experienced with HM sludge, short settling windows due to flammability controls, and more demanding sludge preparation schedules. Additional techniques of scaling results to changes in the sludge mass, estimating the expected settling behavior of combinations of sludge types, using the tank temperature profile to read settling progress, and using a simplified settling model have improved the accuracy and flexibility of short- and mid-range process planning for sludge preparation.

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