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# **Insoluble Solids from Salt Dissolution: Characterization and Testing**

**S. C. Hunter**

October 2023

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## EXECUTIVE SUMMARY

Savannah River National Laboratory (SRNL) has further characterized insoluble solids that were observed in a variable depth sample from a salt dissolution campaign in Tank 9H. The insoluble solids were determined to be predominately gibbsite, a mineral form of aluminum hydroxide. From a review of salt dissolution testing and field experience, SRNL provided a realistic estimate of 8 vol% for solids of this type is formed per volume of saltcake dissolved. This estimate was doubled to 16 vol% to account for dissolution test uncertainty and differences between in-tank settling and laboratory testing. Savannah River Mission Completion (SRMC) is currently assessing the solids formed during salt dissolution as slurried sludge for hydrogen retention and release, which is driving flammability controls during salt dissolution activities. SRMC has requested SRNL perform a gas retention and release study to better understand the impact of the insoluble solids on waste tank flammability, and to provide a more accurate estimate of their ability to retain and release flammable gases.

The particle size and shape of the insoluble solids were determined by scanning electron microscopy (SEM) and laser diffraction. A representative sample of the insoluble solids was sought by synthesizing gibbsite, as well as procuring gibbsite from a manufacturer (Huber). The particle size distribution of the manufactured gibbsite more closely matched that of the Tank 9H insoluble solids. The manufactured gibbsite was used in rheology tests to determine yield stress of a gibbsite slurry and the shear strength of a settled layer of the solids. Previous testing showed that no assertion should be made about negligible gas retention in layers thinner than 18 inches. A settled layer of the gibbsite showed an increasing shear strength over time. At 23 days, the shear strength of the gibbsite was 53 Pa. There is currently no modeling of the gas retention from a settled layer of gibbsite, or any gas retention tests of a settled layer of gibbsite to date. Without these, what is reported here can only be compared to past testing at SRS and Pacific Northwest National Laboratory (PNNL) Hanford gas retention studies. Due to the shear strength of the settled layer, the insoluble solids would be expected to retain gas based on Hanford data with waste of a similar shear strength. While the amount of retained gas for a settled layer of insoluble solids is not expected to be as high as seen in Hanford simulant tests at a similar shear strength, an actual retained gas estimate would require additional testing. Without additional testing, a less conservative assessment for gas retention, such as settled sludge compared to slurried sludge, is not recommended.

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## LIST OF ABBREVIATIONS

ACSM	Analytical Characterization and Sample Management
CSTF	Concentration, Storage, and Transfer Facilities
D10	Point on the particle size distribution curve below which 10% of the particles fall
D50	Point on the particle size distribution curve below which 50% of the particles fall, also called median particle size
D90	Point on the particle size distribution curve below which 90% of the particles fall
DI	Deionized
EDX	Energy Dispersive X-ray Spectroscopy
PNNL	Pacific Northwest National Laboratory
SEM	Scanning Election Microscope
SRMC	Savannah River Mission Completion
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
SSSS	Settled Solids Shear Strength
TTR	Task Technical Request
XRD	X-ray Diffraction

## 1.0 Introduction

Savannah River National Laboratory (SRNL) received surface and variable depth samples from Savannah River Mission Completion (SRMC) during a Tank 9H saltcake dissolution campaign. The Tank 9H variable depth sample (HTF-09-21-42) pulled on May 17, 2021 had an unexpectedly high quantity of insoluble solids (10.14 wt %).<sup>1</sup> These insoluble solids were found to be mostly gibbsite, a mineral form of aluminum hydroxide. These undissolved solids from saltcake dissolution must be assessed for their ability to retain flammable gases to ensure safe operations. SRNL performed a review of historical data on insoluble solids remaining from salt dissolution campaigns at the Savannah River Site (SRS) and provided a conservative basis for quantifying the undissolved solids. It was recommended that a 16 vol% of residual insoluble solids be considered formed per volume of saltcake dissolved.<sup>2</sup>

These insoluble solids formed during salt dissolution are conservatively assessed as slurried sludge for hydrogen retention and release purposes. Slurried sludge is considered to have a maximum bubble gas volume percent of 20%. This bounding value is based off of in-situ gas measurements in actual Hanford tank waste.<sup>3</sup> It should be noted that for SRS tank waste, slurried sludge typically has between 1-6% volume retained gas.<sup>4</sup> In comparison to slurried sludge, settled sludge (based on trapped gas fraction data from Tank 8F, 13H, and 40H) and saltcake (from gas fraction analysis of salt tanks 36H and tank 41H) are considered to have 10 and 11% maximum bubble gas volume percent respectively.<sup>5,6</sup> Using these conservative assumptions in flammability analyses will drive conservative controls during salt dissolution activities, supernate transfers, evaporator operations, and other activities credited for releasing trapped gas. A more representative classification of the insoluble solids, such as settled sludge or saltcake, is sought. SRMC has requested, via a Technical Task Request (TTR),<sup>7</sup> a Gas Retention and Release study be performed by SRNL to better understand the impact of insoluble solids on waste tank flammability.<sup>8</sup>

## 2.0 Experimental Procedure

### 2.1 Tank 9H Insoluble Solids Particle Characterization

A scanning electron microscopy (SEM) sample of the Tank 9H solids was prepared in the following way. Solids from the original Tank 9H sample (HTF-09-21-42) had been collected on a 0.2  $\mu\text{m}$  cellulose filter after filtration of the sample. These solids were then placed in a vial with water (15:1 water:solids) to see if the solids would dissolve. This sample vial, labeled “HTF-09-21-42 solids 15:1 water dissolution”, was shaken to disperse insoluble solids throughout solution. A transfer pipette was dipped into the slurry to collect a small drop of sample. The pipette tip was then placed on the carbon tape atop a SEM stub to transfer the drop to the tape. The sample was allowed to dry, and solids were visually confirmed on the SEM tape before placing into green shielded bottles and transferring to SRNL Analytical Characterization and Sample Management (ACSM) personnel for SEM analysis.

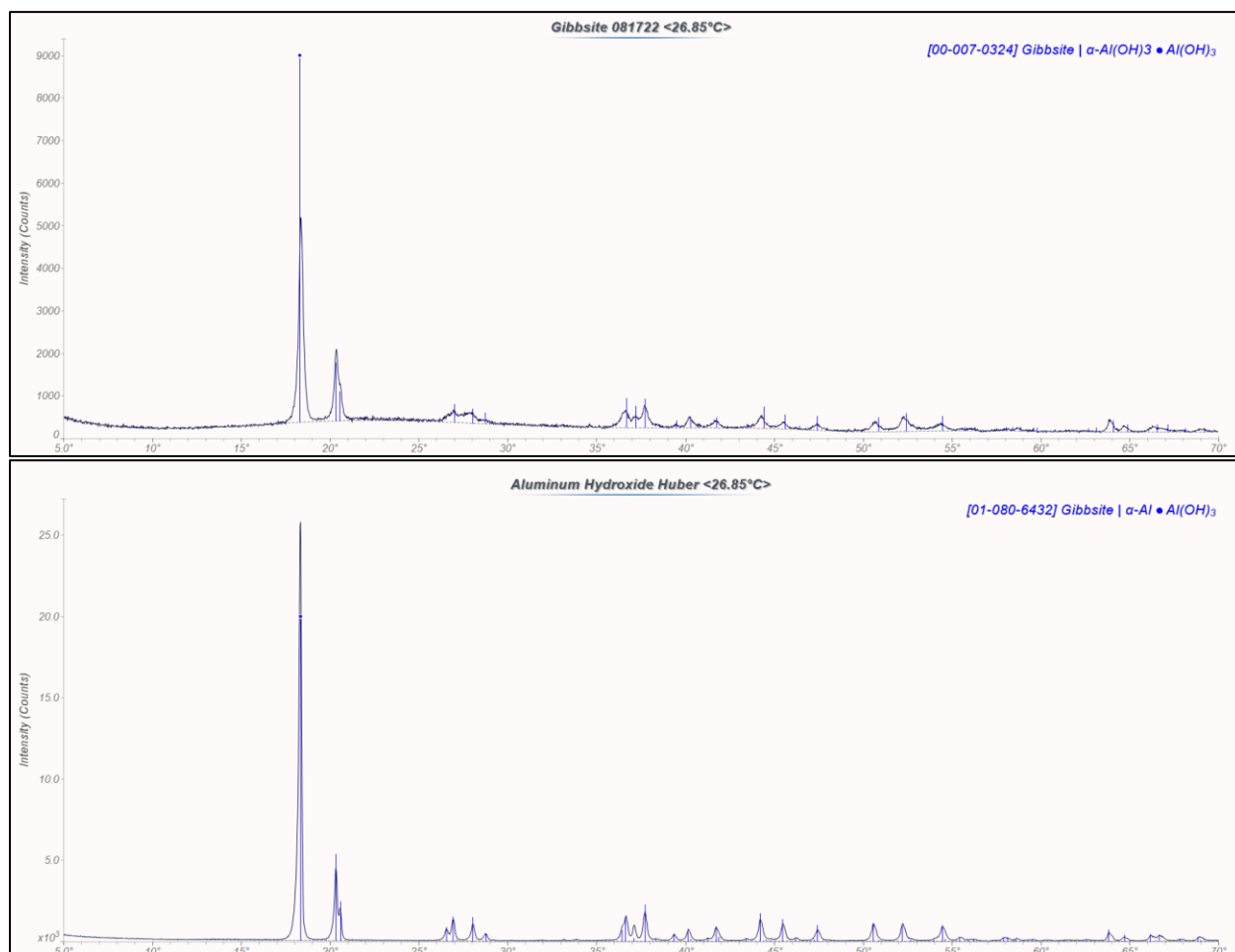
For particle size analysis, sample vials of HTF-09-21-42 (water dissolution vial mentioned above, along with a vial with the slurry labeled “HTF-09-21-42 slurry”) were left undisturbed to allow settling of solids. The supernate in each vial was then decanted without disturbing the solids. The sample vials with the solids were then sent to ACSM for analysis. A Tank 9H simulant was provided to ACSM for the particle size analysis.

### 2.2 Synthesis/Procurement and Characterization of Synthetic Gibbsite

Gibbsite was synthesized following a Hanford method.<sup>9</sup> Sodium aluminate was dissolved in ~80 °C deionized (DI) water. Sodium Nitrate and Sodium Nitrite were then added to solution at 80 °C and were allowed to dissolve before filtering the hot solution. The solution was then allowed to sit at ambient conditions for 2-3 weeks, at which point gibbsite precipitation was noted.

Manufactured gibbsite was procured from Huber under the product name Aluminum Trihydrate (Product# SB-432, Lot# FMTFR22717).

Powder X-ray Diffraction (XRD) and particle size analysis were obtained by ASCM personnel for both the manufactured and synthesized gibbsite. XRD diffractogram for the manufactured and synthesized gibbsite is shown in Figure 2-1.



**Figure 2-1. Top: XRD of the synthesized gibbsite showing a match for gibbsite mineral phase. Bottom: XRD of the manufactured gibbsite procured from Huber showing a match for the gibbsite mineral phase.**

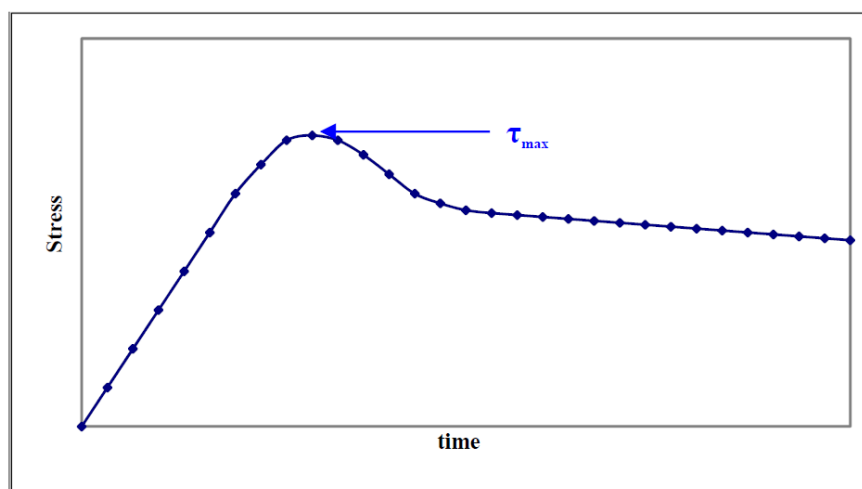
### 2.3 Rheology Measurements

Rheology testing was performed on both the manufactured and synthesized gibbsite in a Tank 9H simulant shown in Table 2-1. The Tank 9H simulant was prepared by adding 50 wt % sodium hydroxide, aluminum trinitrate nonahydrate, sodium nitrate, sodium nitrite, sodium sulfate, sodium carbonate, and sodium oxalate to DI water. The density of the simulant was 1.38 g/mL and was measured by weighing a known volume of the simulant using an M&TE autopipette and an M&TE balance. The rheology tests were performed using a Haake RheoStress 600 Rheometer. The flow curve measures the shear stress as a function of shear rate. The flow curve measurements were obtained on well-mixed 10 wt % gibbsite slurries.

The settled solids shear strength was measured using the vane method. An example vane measurement, which has been used at SRNL and has been described in detail previously, is shown in Figure 2-2.<sup>10</sup> To make a large enough settled layer in the Haake cup for the vane measurements, a higher wt % gibbsite loading was used. For the manufactured gibbsite the loading was approximately 38 wt % and approximately 25 wt % for the synthesized gibbsite. The gibbsite slurries were prepared using the Tank 9H simulant. Slurries were well-mixed before loading into a Haake cup and allowing the solids to settle undisturbed prior to taking measurements.

**Table 2-1. Tank 9H simulant.**

Analyte	Concentrations (M)
[OH]	0.66
[NO <sub>2</sub> ]	0.51
[NO <sub>3</sub> ]	5.36
[SO <sub>4</sub> ]	0.019
[CO <sub>3</sub> ]	0.62
[C <sub>2</sub> O <sub>4</sub> ]	0.003
[Al]	0.083
[Na]	7.89



**Figure 2-2. Example Vane Torque Versus Time/Displacement Curve (Hansen, 2012).**

## 2.4 Settling Testing

Settling tests were performed on 5, 10, and 15 wt % gibbsite slurries made with the manufactured gibbsite. The slurries were prepared using the Tank 9H simulant given in Table 2-1. The gibbsite solids settling tests were performed in 1 L graduated cylinders with approximately 900 mL of the slurries. The slurries were mixed to fully suspend the gibbsite particles. The slurry/supernatant interface was recorded at various times during settling. The settling tests were repeated to get additional interface height time points.

## 2.5 Quality Assurance

The TTR identifies the Functional Classification as Safety Class. Thus, this document was reviewed by Design Verification by Document Review. The requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2. Data are recorded in the electronic laboratory notebook: K7482-00430-26.

## 3.0 Results and Discussion

### 3.1 Tank 9H Insoluble Solids Particle Analysis

A particle size comparison of the Tank 9H solids to the manufactured and synthesized gibbsite is given in Table 3-1, and the individual results are given in Figure 3-1 and Figure 3-2. The D10, D50, and D90 given in the table correspond to the points in the particle size distribution curve below, in which a percentage of the particles fall (i.e., the D10 is 5.09  $\mu\text{m}$  for Tank 9H sample A, meaning only 10% of the particles are smaller than 5.09  $\mu\text{m}$ ). The two runs of Tank 9H solids were very similar, with a unimodal distribution. The particle size results for synthesized and manufactured gibbsite are also given in Table 3-1. The synthesized gibbsite had a bimodal distribution and did not match the Tank 9H solid particle size. The manufactured gibbsite had a unimodal distribution with similar D50 of the Tank 9H solids, while having a wider particle size distribution than the Tank 9H solids. SEM images and Energy Dispersive X-ray Spectroscopy (EDX) were additionally obtained on the Tank 9H solids. An example SEM/EDX is shown in Figure 3-3; the hexagonal plates in the SEM image, which are predominately aluminum and oxygen in composition by EDX, matches with what has previously been seen in gibbsite samples from Hanford.<sup>9</sup>

**Table 3-1. Comparison of particle size results.**

Sample	D10 ( $\mu\text{m}$ )	D50 ( $\mu\text{m}$ )	D90 ( $\mu\text{m}$ )
Tank 9H Solids Sample A	5.09	8.38	11.68
Tank 9H Solids Sample B	7.25	11.56	15.90
Manufactured Gibbsite	3.29	11.65	24.00
Synthesized Gibbsite	2.04	7.45	60.33

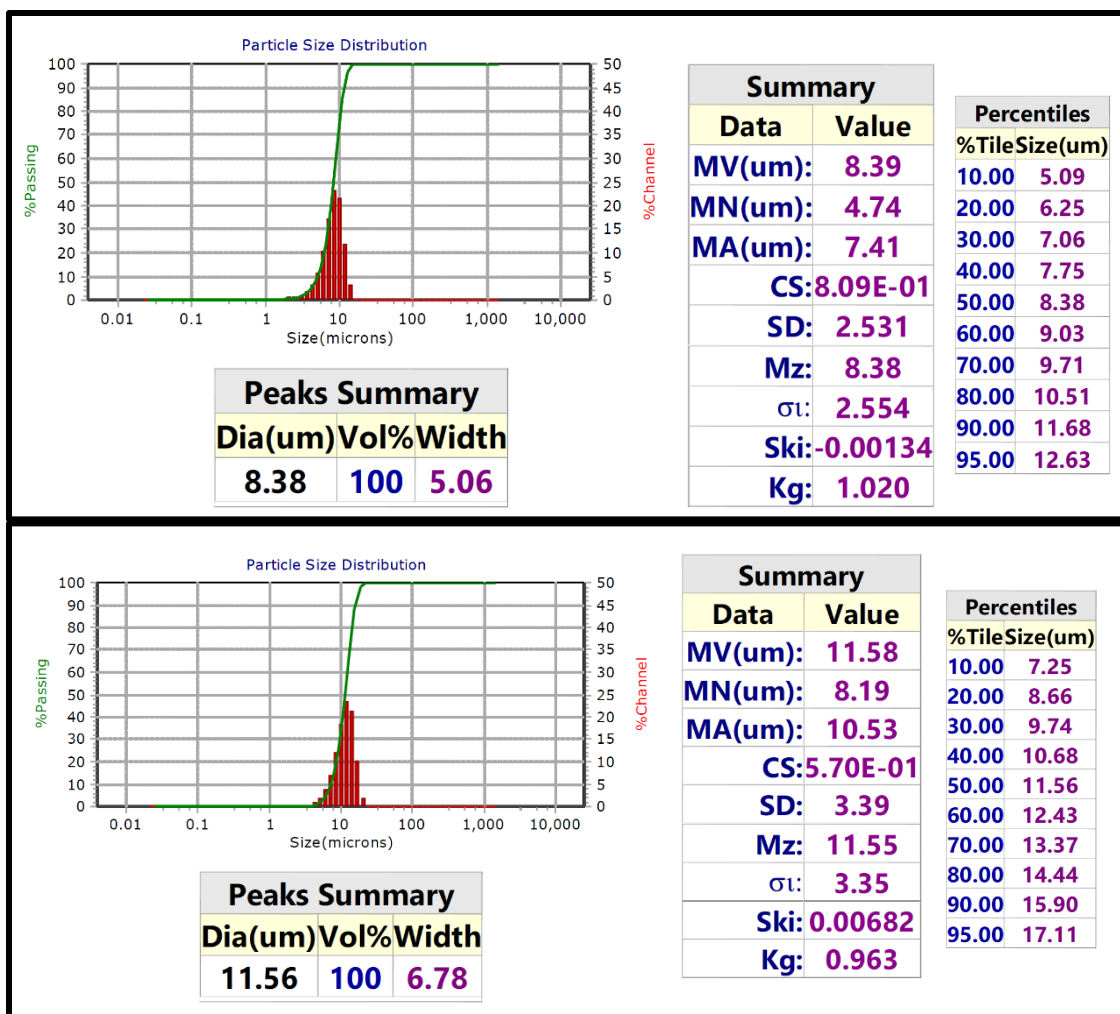


Figure 3-1. Particle size distributions for Tank 9H solids runs 1 (top) and 2 (bottom).

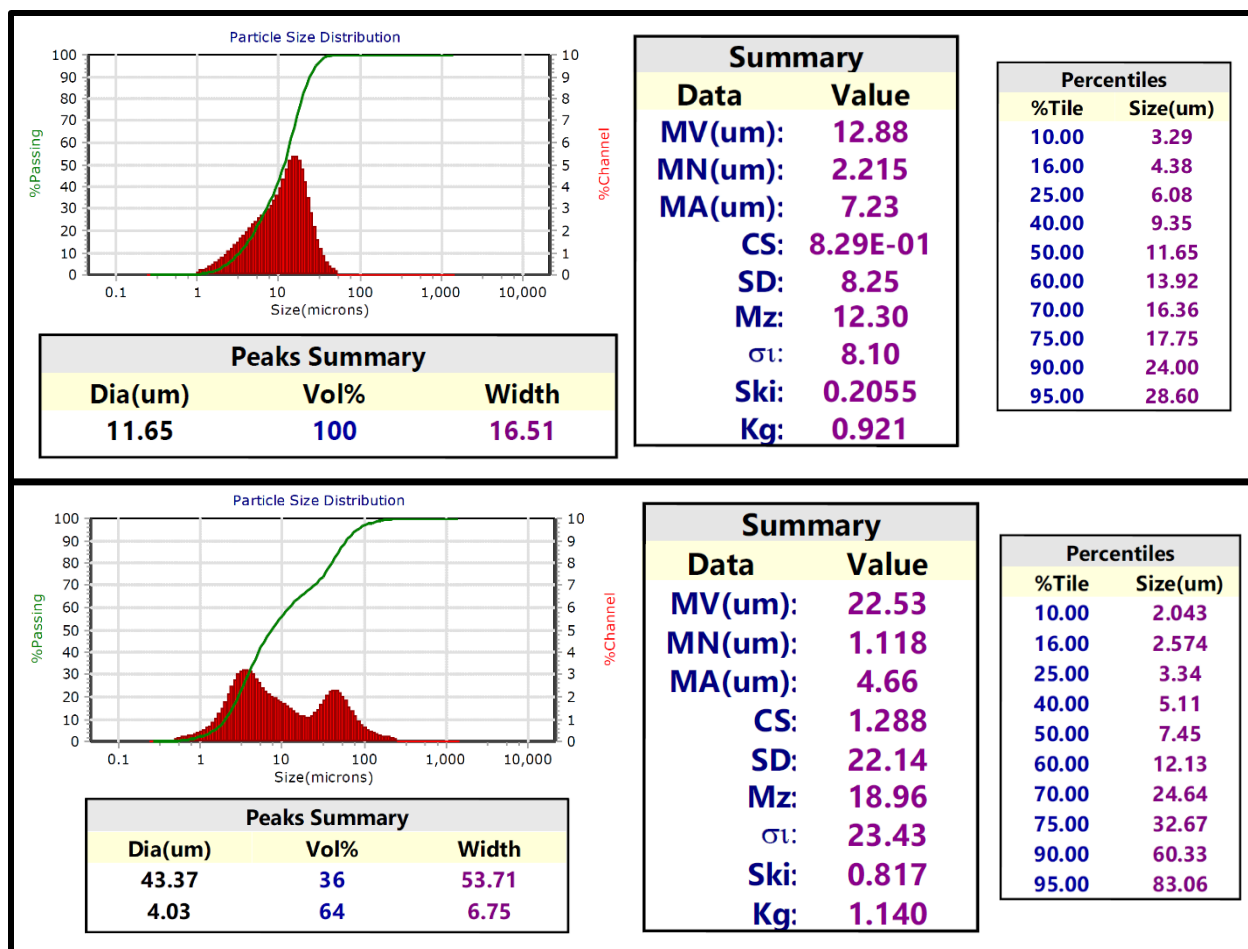
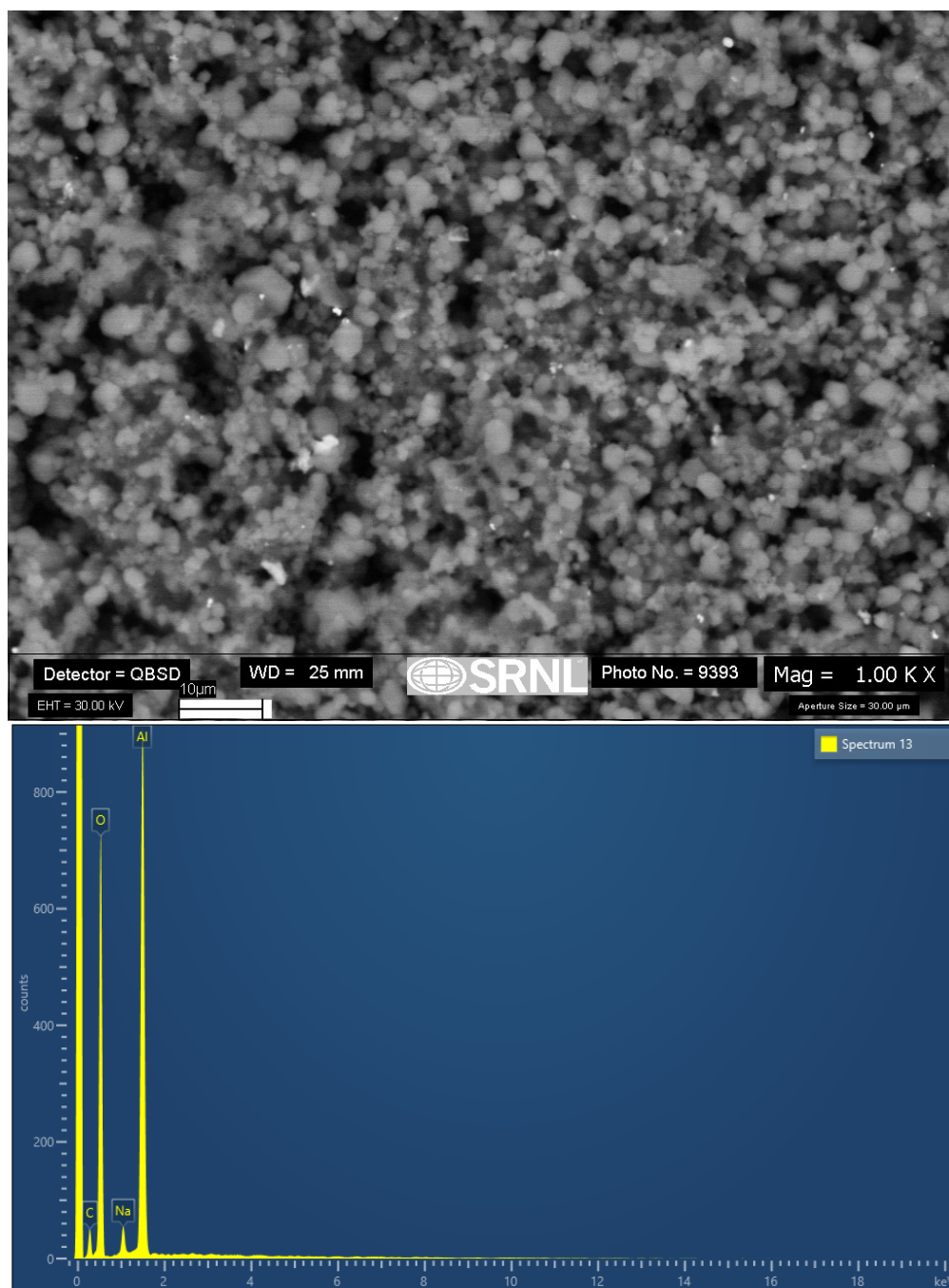


Figure 3-2. Particle size distributions for manufactured gibbsite (Huber, top) and from synthesized gibbsite (bottom).



**Figure 3-3. Top: SEM image of Tank 9H insoluble solids showing hexagonal shaped gibbsite particles. Bottom: Typical SEM/EDX showing predominately Al and O elemental composition for the hexagonal particles.**

### 3.2 Gibbsite Rheology Data and Comparison to PNNL Hanford Studies

Rheology testing was not performed on the actual Tank 9 solids due to an insufficient amount of sample for the testing. Flow curve measurements were performed to determine the viscosity and yield stress of approximately 10 wt % slurries of both the synthesized and manufactured gibbsite in a Tank 9H simulant. Both slurries were found to be Newtonian and their viscosities are given in Table 3-2. The table gives the



viscosities of the Up and Down curves, as well as the viscosity from the Hold point, where the shear rate was held at 600 1/s.

**Table 3-2. Viscosity of 10 wt % slurries and Tank 9H simulant.**

Sample	Viscosity (cP)								
	Up			Down			Hold		
	1	2	avg	1	2	avg	1	2	avg
10% Synthesized Gibbsite	5.30	-	<b>5.30</b>	5.29	<b>5.45</b>	<b>5.37</b>	5.23	<b>5.43</b>	<b>5.34</b>
10% Manufactured Gibbsite	4.94	4.93	<b>4.94</b>	4.77	4.78	<b>4.77</b>	4.88	4.89	<b>4.89</b>
Tank 9H Simulant	4.21	4.21	<b>4.21</b>	4.11	4.21	<b>4.16</b>			

Vane measurements were performed to determine the shear strength of a settled layer of the manufactured gibbsite. The results of these measurements are given in Table 3-3. Two Haake cups with a settled layer of the manufactured gibbsite were prepared. The 120 hours measurement was repeated and labeled SSSS-3 and 4 in the Table 3-3. All measurements were performed after allowing the solids to settle for a set amount of time. The settled solid shear strength (SSSS) for the manufactured gibbsite was found to increase over time. After 24 hours, the settled layer of manufactured gibbsite had an average SSSS of 38.5 Pa. After 120 hours the SSSS was 49.4 Pa, a 28% increase in the shear strength compared to the 24-hour measurement. After 553 hours the SSSS was 53.4 Pa, a further 8.21% increase in the shear strength compared to the 120-hour measurement. For the synthesized gibbsite, Table 3-4, the SSSS also increased over time; however, the shear strength of the settled layer was about four times smaller than that of the manufactured gibbsite. This may be indicative of the importance of particle size on the shear strength of the settled layer. Shear strength has been shown to increase with an increase in particles <5  $\mu\text{m}$  in size.<sup>11,12</sup>

**Table 3-3. Manufactured gibbsite settled solids vane measurement results.**

Material	Manufactured Gibbsite					
Time	SSSS-1 (Pa)	SSSS-2 (Pa)	SSSS-3 (Pa)	SSSS-4 (Pa)	Average (Pa)	% change
24 hours	37.3	39.8	-	-	<b>38.5</b>	-
120 hours	48.6	55.2	47.7	45.9	<b>49.4</b>	28.2%
23 days (~553 hours)	55.3	51.5	-	-	<b>53.4</b>	38.7%

**Table 3-4. Synthesized gibbsite settled solids vane measurement results.**

Material	Synthesized Gibbsite			
Time	SSSS-1 (Pa)	SSSS-2 (Pa)	Average (Pa)	% change
24 hours	9.2	8.7	8.9	-
72 hours	12.6	13.2	12.9	44.7%

Hanford has typically related shear strength of a layer to its ability to retain and release hydrogen, as shown in Figure 3-4.<sup>13</sup> The black dots in the figure indicate the measurements from tests with Bentonite Clay. The blue images shown at the bottom of the figure represent how the gas is released at different shear strengths. Additionally, PNNL Hanford gas retention and release studies have shown that when gas bubbles are larger than the waste particles, they will displace the waste and be retained by the strength of the waste. An example of this type of bubble retention is shown at the top of Figure 3-5, which are images of bubbles in bentonite clay simulant at different shear strengths. The bottom of Figure 3-5 shows an example of the bubble displacing mechanism and a second potential mechanism for gas retention in which the gas bubbles

are held within pores by capillary forces. For Hanford sludges, the gas retention peaks at about 40% by volume with a shear strength of 50 Pa. However, this data should not be used as an estimate of the gas retained in a settled layer of gibbsite, this data was obtained in simulant tests that may not be indicative of real waste tank conditions. Additionally, these simulant tests used different gas generation methods (ex. hydrogen peroxide and iron particles) that may produce bubbles at different rates and sizes than how gas is generated in tank waste. It was suggested in testing at PNNL by R.C. Daniel et al. that the bubble generation method impacts gas retention and release behavior.<sup>14</sup> Real waste tank gas retention measurements at SRS have been much lower, 1-6 vol%, than the Hanford simulant data. The data does show that the shear strength of a material is an important parameter when looking at its ability to retain gas. Our rheology data shows that a settled layer of gibbsite is strong enough to retain gas.

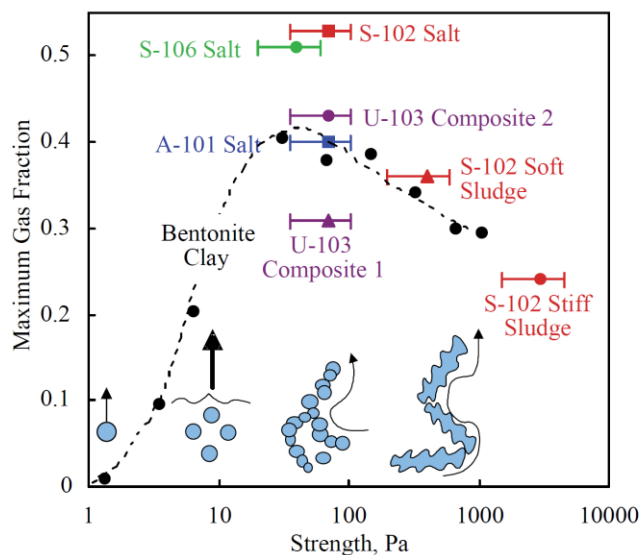


Figure 3-4. Maximum gas fraction vs. shear strength in simulant and actual Hanford waste.<sup>13</sup>

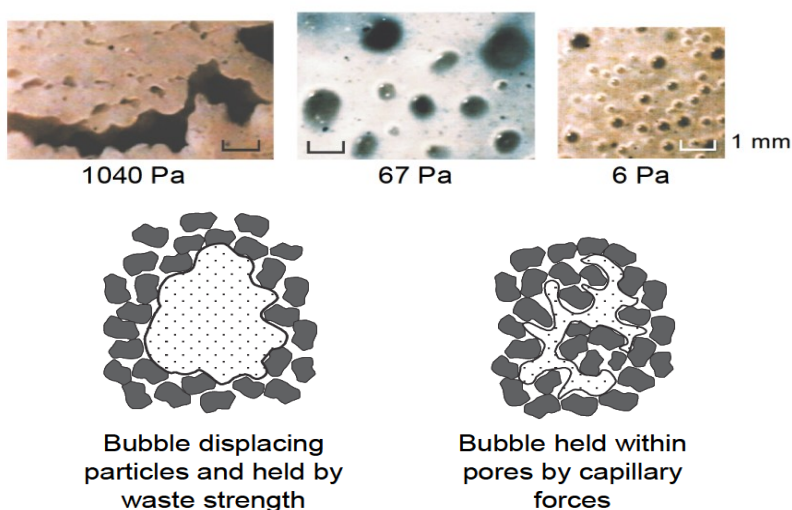


Figure 3-5. Top: Retained bubbles at a range of shear strengths. Bottom: Two possible bubble retention mechanisms in waste.<sup>13</sup>

It has been previously reported that 18 inches of sludge are required before an appreciable amount of gas can be trapped.<sup>15</sup> This was based on a 1998 correspondence with Hanford in which it was stated that the yield stress is essentially zero at the top and increases linearly with depth of the sludge.<sup>4</sup> However, it was mentioned in the same correspondence that if the settled sludge has a significant strength (> 50 Pa), then this would not be the case. Testing by PNNL in 2014 disproved a similar theory that gas retention increases significantly at deeper depths.<sup>16</sup> While it would seem reasonable that at some point, a very thin settled layer would no longer be able to retain the gas bubbles, no data was found in the literature at this time. As such, no assertion should be made about negligible gas retention in layers thinner than 18 inches. Additionally, the impact of a higher gas-retaining settled layer forming on top of a lower gas-retaining settled layer could not be found in literature.

### 3.3 Modeling

The M-Star software has been benchmarked and used to determine retained hydrogen release from a Bingham plastic fluid in an impeller mixed tank. SRNL attempted to simulate hydrogen release from a Bingham plastic fluid in an unagitated vessel. These efforts proved unsuccessful.

The software does not impose a true yield stress on the fluid, rather, it calculates an apparent viscosity as a function of the rate of strain throughout the vessel. It sets a maximum viscosity which is much larger than the apparent viscosity in the regions of interest in the vessel. Having a finite yield stress would only be important in a very small fraction of the vessel, near the boundaries.

With an unagitated vessel, this feature of the software creates difficulties. The fluid in the simulation will not have a yield stress, but rather a very large viscosity. This very large viscosity allows the retained gas bubbles to rise very slowly. The maximum holdup volume in the slurry becomes a function of the maximum viscosity set by the software user. The maximum holdup should be a function of slurry yield stress rather than a user input to the software.

### 3.4 Settling Testing with Procured Gibbsite

Settling tests were performed in 1 L graduated cylinders using either 5, 10, and 15 wt % gibbsite in a Tank 9H simulant. The height of the interface between the supernatant and slurry was visually measured during settling. Settling typically occurs in three stages: free settling, in which particles are not hindered by other particles as they settle; hindered settling, in which the interaction between particles hinders the rate of settling; and compression settling, in which the weight of overlaying solids drive out the interstitial fluid between the solids. At 5 wt %, the gibbsite appeared to be free settling for the first five hours, forming a settled layer. Particles will settle at different rates based on their size, and this was more clearly evident in the 5 wt % slurries, where bands appeared to be forming during settling tests. This settled layer further settled over the next 5 hours. The 10 and 15 wt % gibbsite loading slurries appeared to quickly go into a hindered settling regime, with a clearly visible interface between the cloudy gibbsite slurry and the clear supernatant, taking up to two days to settle. Table 3-5 gives the overall results of the tests. The third column in the table gives the final measured height of the slurry (or settled layer). The fourth column is the vol % of the settled layer (final height/initial height). The last column gives the wt % gibbsite in the settled layer which is calculated using the following equation:

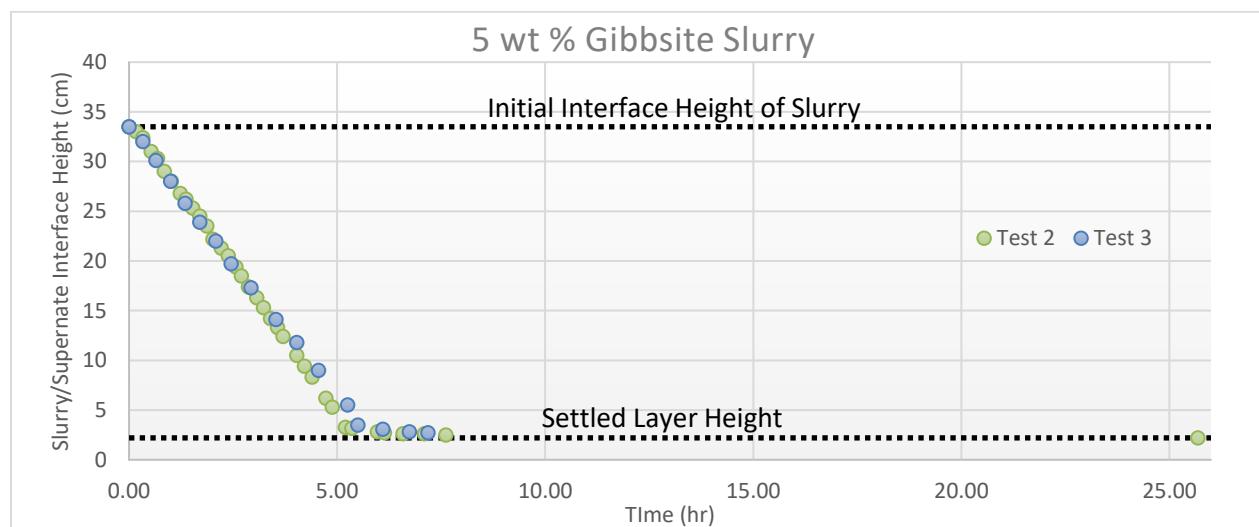
$$\text{wt \% Gibbsite of settled layer} = \frac{\text{wt \% Gibbsite of slurry}}{(\text{Settled Layer Height} / \text{Initial Slurry Height})}$$

Figure 3-6 gives the slurry/supernatant interface height as a function of time for the 5 wt % tests, Figure 3-7 for the 10 wt % tests, and Figure 3-8 for the 15 wt % tests. Multiple tests were run for each gibbsite loading to get additional time points and are included in the respective figures. Test 1 of the 5 wt % test is not included, as due to the multiple bands of solids settling in all of the 5 wt % cases, it was difficult

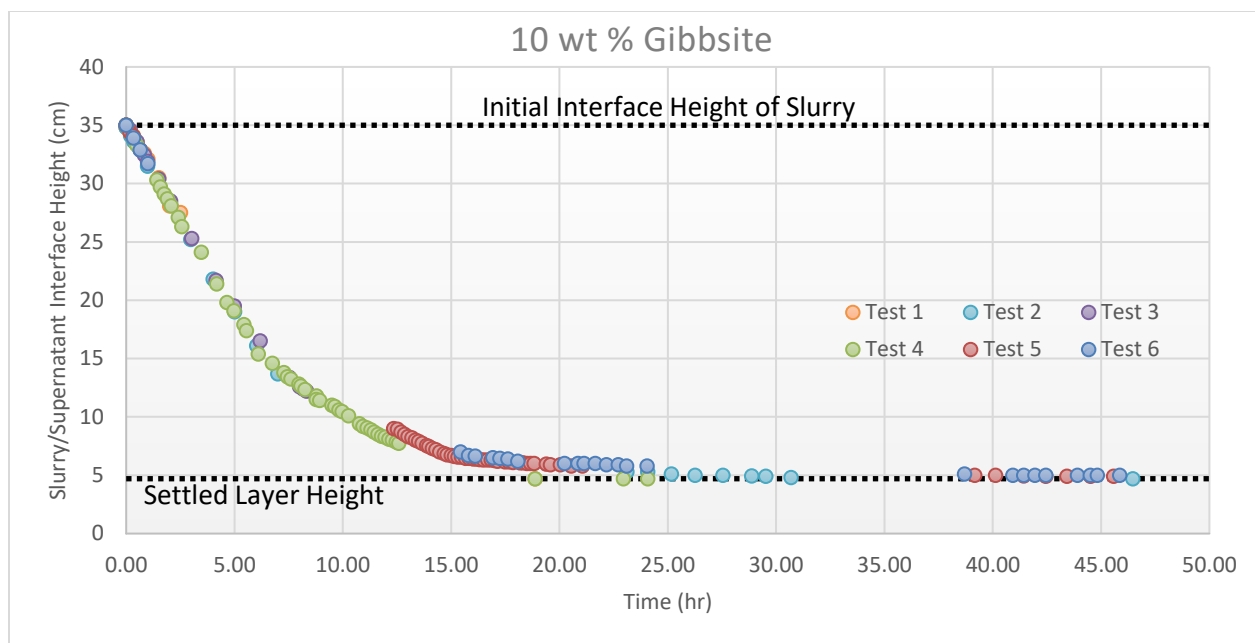
following the interface height. All three appeared to have settled within two days; however, further compression settling is probable over a much longer time frame (weeks to months). Further compressibility may be hinted at by the rheology results given above; the results showed an increasing SSSS over a longer time frame than the settling tests reported here. This increase in SSSS may signal further compressibility of the solids layer; however, Hanford work has also shown that a slurry's shear strength may increase over time through an aging mechanism that is independent to changes in the insoluble solids concentration due to settling or compaction.<sup>17</sup> It should be noted that the settling times reported here are indicative of the testing vessel; as the distance the particles must travel in a vessel increases, so does the time to fully reach sediment state. Additionally, as the density of the solution increases, the settling velocity of the particles would decrease. The density of Tank 9 simulant used in these tests was 1.38 g/mL.

**Table 3-5. Settling results.**

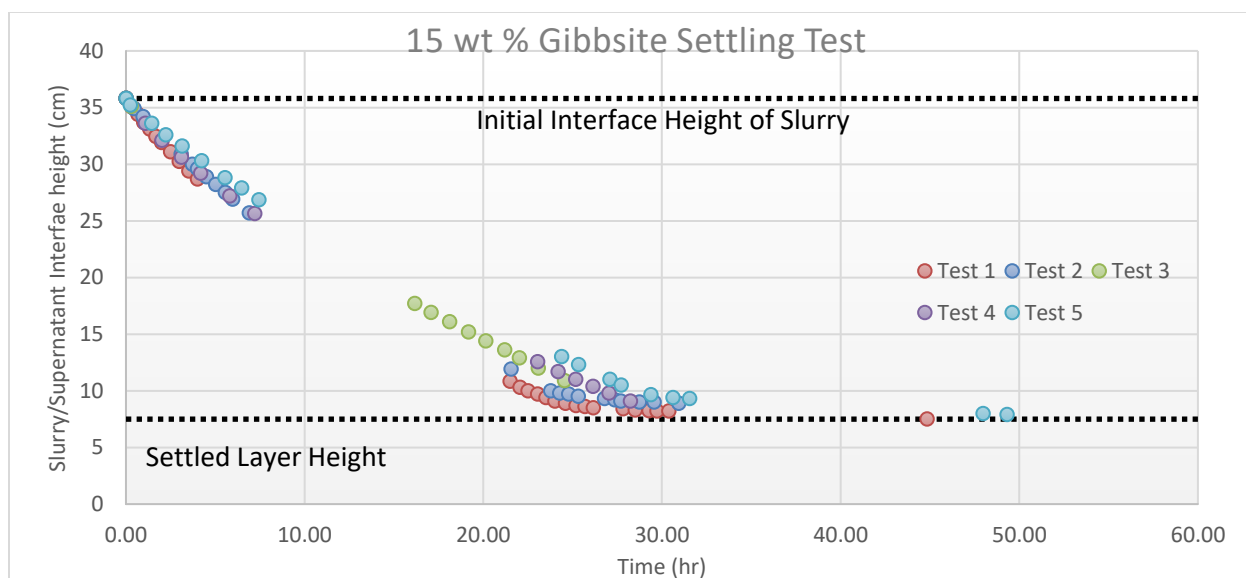
Wt % Gibbsite of Slurry	Initial Slurry Height (cm)	Final Slurry Height (cm)	Time to Fully Settle (hours)	Final Vol % of Settled Layer	Wt % Gibbsite of Settled Layer
5	33.5	2.2	~8-10	6.6	76
10	35	4.7	~18-48	13	74
15	35.8	7.3	>44	20	74



**Figure 3-6. Settling data for a 5 wt % gibbsite slurry in a Tank 9H simulant.**



**Figure 3-7. Settling data for a 10 wt % gibbsite slurry in a Tank 9H simulant.**



**Figure 3-8. Settling data for a 15 wt % gibbsite slurry in a Tank 9H simulant.**

## 4.0 Conclusions

SRNL has further characterized solids that were observed in a variable depth sample from a salt dissolution campaign in Tank 9H. SRMC is currently having to assess these insoluble solids formed during salt dissolution as slurried sludge for hydrogen retention release, which is driving flammability controls during salt dissolution activities. SRMC has requested SRNL to perform a gas retention and release study to better understand the impact of the insoluble solids on waste tank flammability.

The Tank 9H solids were further characterized using SEM and laser diffraction to determine the particle size and shape of the solids. Hexagonal plate particles in the SEM images were found to be predominately aluminum and oxygen from EDX. This data, along with the previously reported XRD data, confirms that the Tank 9H solids were predominately gibbsite. Rheology and settling tests were performed with manufactured gibbsite obtained from Huber that had a similar particle size to the Tank 9H solids. A settled layer of the gibbsite showed an increasing shear strength over time. At 23 days, the shear strength of the gibbsite was 53 Pa. Additionally, when looking at previous studies, no assertion should be made about negligible gas retention in layers thinner than 18 inches. The data reported herein was to be used in conjunction with modeling data to provide an estimate of gas retention from a settled layer of the insoluble solids. The modeling effort proved unsuccessful due to difficulties when trying to model an unagitated vessel with the M-star software, maximum gas holdup was a function of viscosity (user input) when it should be a function of the slurry yield stress. There is currently no modeling of the gas retention from a settled layer of gibbsite, or any gas retention tests of a settled layer of gibbsite to date. Without these, what is reported here can only be compared to past testing at SRS and studies performed at Hanford. Due to the shear strength of the settled layer, the insoluble solids would be expected to retain gas based on Hanford data with waste of a similar shear strength. While the amount of retained gas for a settled layer of insoluble solids is not expected to be as high as seen in Hanford simulant tests at a similar shear strength, an actual retained gas estimate would require additional testing. Without additional testing, a less conservative assessment for gas retention, such as settled sludge compared to slurried sludge, is not recommended.

## 5.0 Recommendations

The following recommendations are made as a result of this testing:

- Additional data during salt dissolution campaigns should be collected to determine a more appropriate estimate of the maximum amount of insoluble solids that can be formed during salt dissolution. Tank chemistry including saltcake composition should also be taken into account to assess whether the conditions present would produce a significant amount of insoluble solids during salt dissolution.
- A gas retention study with a settled layer of gibbsite is recommended to obtain a more representative value for how much hydrogen gas can be retained. Specifically, it is recommended to perform this testing with a simulant containing a settled layer of gibbsite in an irradiator. By using an irradiator, hydrogen gas would be produced by the radiolysis of the water in the simulant, similar to how most hydrogen gas would be produced in waste tanks. The gas retention of the settled layer would be determined by the liquid level height and by gas chromatography.

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