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Rheology of Settled Solids in the Small Column Ion Exchange (SCIX) Process

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Savannah River National Laboratory
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REVIEWS AND APPROVALS

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LIST OF ACRONYMS

ACTL	Aiken County Technology Laboratory
ARP	Actinide Removal Process
CST	Crystalline Silicotitanate
DWPF	Defense Waste Processing Facility
MST	Monosodium Titanate
NIST	National Institute of Standards and Technology
ORNL	Oak Ridge National Laboratory
RMF	Rotary Microfilter
SCIX	Small Column Ion Exchange
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
SRS	Savannah River Site

1.0 SUMMARY

The Small Column Ion Exchange (SCIX) process is being developed to remove cesium, strontium, and actinides from Savannah River Site (SRS) Liquid Waste using an existing waste tank (i.e., Tank 41H) to house the process. This process adds monosodium titanate (MST) to a waste tank containing salt solution (and entrained sludge solids). While the process is operating, the solid particles will begin to settle at temperatures up to 45 °C. Previous testing has shown that sludge-MST slurries that sit for extended periods (i.e., 1 – 61 days) at elevated temperatures (i.e., 23 – 80 °C) can develop large shear strengths which could make them difficult to resuspend and remove from the tank.[1,2] The authors are conducting rheological testing of mixtures containing various concentrations of sludge, MST, and crystalline silicotitanate (CST, ground and unground) that have been aged at different times (i.e., 0 to 13 weeks) and isothermally heated to 30, 45, or 60 °C. Additional tests are being conducted that will allow the solid particles to settle at 45 °C for 6, 12, and 24 months.

The objectives of this task are to determine the impact of settling time and temperature on the shear strength, yield stress, and consistency of the slurries and to determine the impact of radiation on slurry rheology. The testing will determine the relative impact of these parameters rather than predict the shear strength, yield stress, and consistency as a function of feed and operating conditions. This document describes the rheology of slurries containing MST and simulated sludge that sat at elevated temperatures (i.e., up to 60 °C) for up to 13 weeks. Rheology of CST-containing slurries, as well as results of the long term settling (6, 12, and 24 months) and irradiation tests (10 and 100 MRad), will be reported later.

The conclusions from this analysis follow:

- MST only slurries that sat at elevated temperatures had larger shear strength, yield stress, and consistency than MST plus sludge slurries that settled at elevated temperatures.
- The addition of sludge to an MST slurry reduces the shear strength, yield stress, and consistency.
- The impact of settling time and temperature on slurry rheology is inconclusive at this time. The authors are collecting additional data to attempt to determine the impact of settling time and temperature on slurry shear strength, yield stress, and consistency.

2.0 INTRODUCTION

Savannah River Remediation (SRR) is working to deploy the SCIX process to remove cesium, strontium, and select actinides from SRS Liquid Waste using an existing waste tank (i.e., Tank 41H) to house the process. The process adds MST to the waste tank (i.e., Tank 41H) to sorb the strontium and select actinides, removes the MST and entrained sludge with an in-riser rotary microfilter (RMF), and removes cesium with ion-exchange columns containing CST. The RMF returns the concentrated solids (i.e., MST and entrained sludge) to the waste tank. After being loaded with cesium, the CST is ground to reduce its size and transferred into a waste tank. The process baseline is to transfer the ground CST to Tank 40H, the Defense Waste Processing Facility (DWPF) feed tank. Efforts are underway to determine the feasibility of transferring the ground CST to Tank 41H. The MST, sludge, and CST (if transferred to Tank 41H) in the waste tank will be periodically transported to a sludge batch preparation tank (i.e., Tank 42H or Tank 51H), and ultimately transported to DWPF.

Previous Savannah River National Laboratory (SRNL) and Oak Ridge National Laboratory (ORNL) testing showed that allowing slurries containing simulated sludge and MST to sit for extended times at elevated temperature led to large increases in the slurry shear strength.[1,2] In the SRNL tests, the sludge and MST settled for 1, 14, 28, and 42 days at 25, 35, and 50 °C. In the ORNL tests, the sludge and MST settled for 0 – 61 days at 23 and 80 °C.

To assist SRR in designing the SCIX process, SRNL is conducting bench-scale rheology tests to determine the impact of changes in solids composition and process conditions on the solids in SCIX. The authors are conducting rheological testing of mixtures of sludge, MST, and CST (ground and unground) that have been aged at different times (i.e., 0 to 13 weeks), while being held at 30, 45, and 60 °C.[3] Additional tests are being conducted to examine the impact of long term settling (up to 24 months) and to examine the impact of irradiation on slurry rheology. Results of the CST containing slurry rheology, long term settling, and irradiation tests will be documented in future reports.

The minimum ratio of MST to CST in Tank 41H will be ~ 1:17, and the minimum ratio of MST to entrained sludge will be 1:1.5. These ratios are based on the SCIX flowsheet and the historical SRS baseline for entrained sludge composition of SRS salt solution (600 mg/L). Initially, the MST to CST ratio will be much larger than 1:17. Qualification of feed to the Actinide Removal Process (ARP) has shown the MST to sludge ratio to be much greater than 1:1.5. The maximum ratio of MST to CST and the maximum ratio of MST to sludge will be bounded by the tests containing only MST. In a sludge tank, the estimated ratio of MST to CST to sludge will be 1:17:660.

The objectives of this task are to determine which slurries have the largest shear strength, yield stress, and consistency[a], to determine the impact of settling time and temperature on the shear strength, yield stress, and consistency, and to determine the impact of radiation on slurry rheology. The shear strength data will provide an indication of the relative ease with which a slurry can be suspended in a waste tank. The yield stress and consistency data will provide an indication of the relative ease with which a slurry can be transferred between processes in the SRS Tank Farm.

This document describes the initial results obtained for slurries containing MST or MST plus simulated sludge. After the rheology of CST-containing slurries and irradiated slurries has been measured, the authors will prepare another report documenting those results. The results of the long term settling tests will be documented in a separate report after they are completed.

[a] The term shear strength is sometimes referred to as a vane yield stress, settled solids yield stress, or vane shear strength. The term yield stress is sometimes referred to as a flow curve yield stress. The term consistency is sometimes referred to as a flow curve consistency, plastic viscosity, or infinite viscosity.

3.0 TESTING

Measurements of the rheological properties of the aged sludge simulant, CST, and MST mixtures include measuring the shear strength of the settled solids using the vane method and flow curves using a Searle type bob/cup configuration. The rheological measurements were obtained at the Aiken County Technical Laboratory (ACTL) using the Haake Rheostress rheometers. The flow curves were analyzed using a Bingham Plastic rheological model. Vane and flow curve measurements were performed in duplicate with duplicate samples.

3.1 SAMPLE PREPARATION

Personnel prepared the rheology samples in the following manner. The MST used is from the same lot (Harrell Industries Lot# 102209, pail 32 of 66) as the MST currently being used at the ARP. It had an insoluble solids concentration of 15 wt % and a median particle size of ~ 16 micron. Prior to preparing samples, the MST was allowed to settle and the clear supernate decanted to produce a 22.3 wt % slurry. The concentrated slurry was mixed with an impeller to produce a homogeneous MST slurry. Samples were collected and the needed amount of MST was added to 2.5-inch diameter by 4-inch tall snap lid poly bottles (see Figure 1).

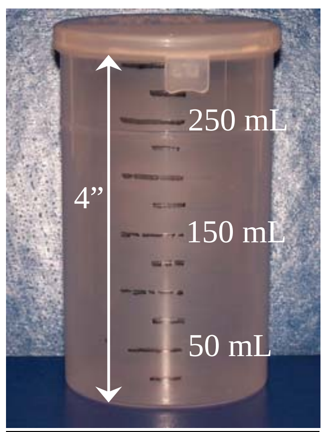

Figure 1. 300-mL poly bottle with 25-mL graduations

The researchers selected simulated Sludge Batch 6 for the rheology tests.[4] Simulated Sludge Batch 6 was chosen, because it is slow settling, viscous, and representative of sludge that will be contained in Tanks 51H and 40H. Because it is slow settling, it will also mimic the physical properties of the entrained sludge expected in Tank 41H. The simulated sludge slurry had an insoluble solids concentration of 11.35 wt %, a soluble solids concentration of 6.3 wt % (primarily sodium nitrate and sodium nitrite), a median particle size of ~ 23 micron, a trimodal particle size distribution, and a yield stress of ~ 50 Pa at 11.35 wt % insoluble solids. Since the simulated Sludge Batch 6 material did not settle, no settling and decanting was necessary. The material was mixed to produce a homogeneous slurry and appropriate amounts added to the sample bottles.

Table 1 in section 3.3 details the relative amounts (by mass) of MST and simulated sludge added to the samples. The MST and sludge were added to the bottles by weight to produce a nominal volume of 140 mL[b]. An additional 70 mL of simulated Tank 37H salt solution (6.44 M sodium, 2.57 M free hydroxide, 0.35 M aluminate, and 0.004 M silicate) was added to each of the bottles. Personnel mixed the bottle contents to create a homogeneous slurry, and then placed the bottles into ovens at 30, 45, or 60 °C for the desired duration.

The CST used is from a lot produced by UOP, the CST manufacturer (median particle size ~ 400 micron). The CST was ground by Hockmeyer (grinder vendor), using the same grinding technology proposed for the SCIX process. The ground CST slurry contained 20 – 40 wt % insoluble solids with a median particle size of 1 - 10 micron (measured by Microtrac). The rheology of CST-containing slurries will be described in a subsequent report.

Samples were removed from the ovens, packed to minimize disturbance, and brought to ACTL for the characterization work. It was understood that small disturbances during shipping might impact the less densely packed settled solids, and that the primary focus of the study was on identifying samples which had undergone a significant transformation into a more compacted and difficult to suspend slurry rather than on identifying subtle differences between the various sample matrices. Settled solids volumes generally exceeded 125 mL.

3.2 RHEOLOGY

Rheological measurements of yield strength and flow curves were performed using the Haake RheoStress 600 rheometer at ACTL. Yield strength was measured with the FL22 vane sensor, shown in Figure 2.

The four vane section has an effective diameter in rotation of 22 mm which is the basis for the name. The vane is lowered into the sample matrix until the point marked "solid interface" by the white arrow is aligned with the top of the settled solids bed. This puts the four vanes 15.5-31 mm into the settled solids (top to bottom of vane). Ideally the bottom of the vane sensor is also at least 15.5 mm above the bottom of the settled solid layer (bottom of the snap lid jar).

Once the vane is positioned, it is rotated at 0.30 revolutions per minute for up to 90 seconds, or 45% of a full single revolution. Solids in the spaces between the four vanes are forced to move relative to the solids above and below the vanes, and also further out from the tips of the vanes. The friction between the moving and stationary particles creates a torque which is recorded by the rheometer. The maximum torque is typically observed in the first 15 seconds. This value is combined with the effective shearing area of the vanes to calculate the yield strength of the settled solids in Pascals (force/unit area). An example of vane data is given in Figure 3.

Figure 2. FL22 vane rotor used for yield strength measurements

[b] Samples containing MST and sludge in a 1:1.5 ratio are referred to as "MST plus low sludge". Samples containing MST and sludge in a 1:660 ratio are referred to as "MST plus high sludge".

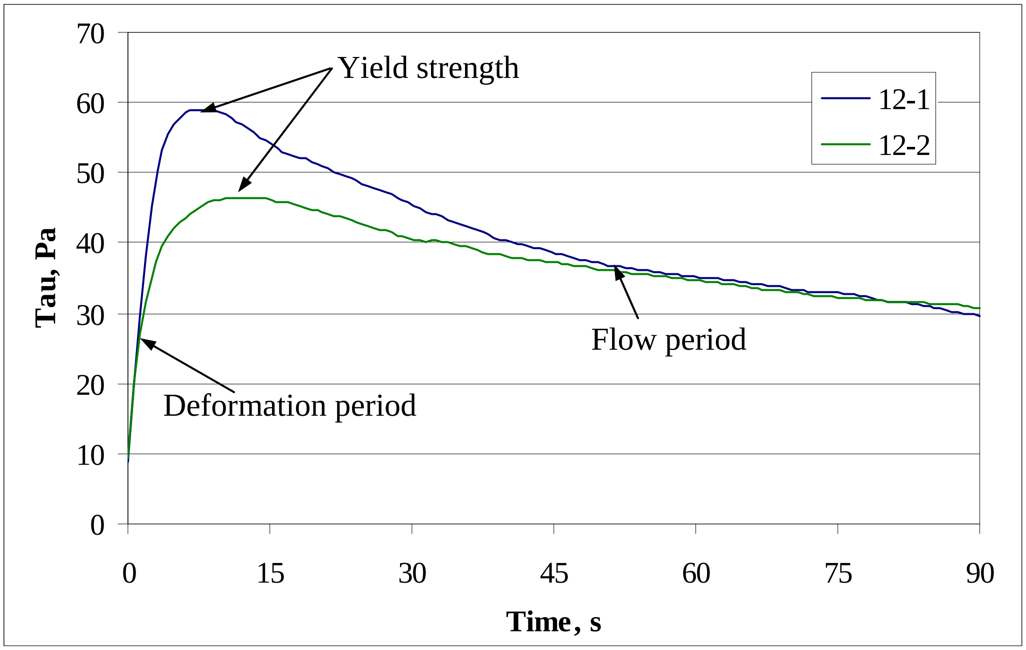

Figure 3. Example of vane data for sample #12

A vane measurement disturbs the settled solids such that the measurement can not be repeated in a vessel as small as the 300-mL sample bottles used. Therefore, each sample was provided in duplicate so that two values of the yield strength could be obtained. Nevertheless, the measurement is delicate and the act of mounting the sample under the vane can cause some disturbance to the settled solids resulting in loss of reproducibility. It was also noted during this study that the replicate samples were not always identical with respect to settled solids volume, even though they presumably contained nearly identical masses of the matrix components. The majority of sample pairs with this feature had a higher yield strength on the more compacted bed of solids as would be intuitively expected. Vane measurements were performed at room temperature. A few samples were still warm when measured, but this was not believed to impact the results significantly.

The flow curve measurements provide rheological properties of mixed slurries and are applicable to the transport of slurries between facilities. Flow curve measurements were made using previously documented methods and the standard DWPF slurry sample protocols.[5] Performance of the RS600 was checked regularly using National Institute of Standards and Technology (NIST) traceable viscosity standards. Flow curve measurements were made using a 25 °C constant temperature bath surrounding the measurement cup.

Each sample was either diluted with a salt solution provided with the samples (one solution for all the diluted samples) or decanted to remove 25-mL in order to obtain a second wt % insoluble solids data point. The provided salt solution was measured to be approximately 29.9 wt % dissolved solids by drying the sample with a halogen lamp. The three sludge-rich types, L5, L6, and L7 were decanted, while the other types were diluted. Decanting required centrifuging, since the remixed solids of the three sludge-rich types did not form sufficient supernate in a practical time span for any significant decanting.

3.3 STATISTICAL DESIGN

These tests will examine the effect of temperature, settling time, and different combinations of MST and sludge solids on the rheological properties of the settled solids. Because of the large number of parameters that need to be investigated, a statistical design of the experimental conditions was conducted to reduce the number of samples needed and to examine parameter interactions. Table 1 shows the combination of treatments to be applied to the respective sludge, CST, and MST solid mixtures. Results of the CST-containing samples will be evaluated in a separate report. The resulting rheology data is fitted to a polynomial function such as the one listed in equation 1, where Y is the slurry shear strength, yield stress, or consistency. In addition, statistical analyses were performed to determine which parameters affect the shear strength, yield stress, and consistency.

$$Y = a + b\,(\text{feed slurry factor}) + c\,(\text{time factor}) + d\,(\text{temperature factor}) \qquad [1]$$

Table 1. Design of Test Matrix

Test #	Time (weeks)	Temp (°C)	Material
1	1	60	L4
2	13	60	L7
3	13	60	L4
4	1	60	L5
5	13	60	L2
6	1	60	L1
7	4	60	L8
8	10	60	L6
9	1	60	L3
10	13	60	L3
11	13	30	L4
12	1	30	L7
13	1	30	L1
14	1	30	L2
15	10	30	L5
16	4	30	L3
17	13	30	L2
18	13	30	L6
19	1	30	L8
20	13	30	L1
21	1	30	L6
22	1	30	L4
23	13	30	L7
24	13	30	L5
25	10	30	L2
26	1	30	L3
27	1	30	L5
28	13	30	L8

Table 1. Design of Test Matrix (cont.)

Test #	Time (weeks)	Temp (°C)	Material
29	4	30	L1
30	13	30	L3
31	1	60	L8
32	13	60	L6
33	4	60	L3
34	1	60	L2
35	1	60	L7
36	13	60	L5
37	10	60	L4
38	13	60	L8
39	13	60	L1
40	1	60	L6
41	4	45	L7
42	1	45	L2
43	4	45	L6
44	10	45	L3
45	1	45	L5
46	13	45	L7
47	13	45	L1
48	10	45	L8
49	13	45	L4
50	1	45	L4

L1= MST
L2= MST + ground CST (1:17 wt ratio)
L3= MST + unground CST (1:17 wt ratio)
L4 = MST + Sludge (1:1.5 wt ratio) [*MST-Low Sludge*]
L5 = MST + Sludge + ground CST (1:660:17 wt ratio)
L6 = MST + Sludge + unground CST (1:660:17 wt ratio)
L7 = MST + Sludge (1:660) [*MST-High Sludge*]
L8 = MST + Sludge + ground CST (1:1.5:17 ratio)

4.0 RESULTS

4.1 VANE MEASUREMENTS

Table 2 illustrates the measured shear strength as a function of feed and settling conditions. The MST plus sludge samples were prepared using two ratios. One ratio (1:1.5) corresponds to the expected ratio in Tank 41H, and is called “MST-Low Sludge”. The other ratio (1:660) corresponds to the expected ratio in Tank 40H, and is called “MST-High Sludge”. Replicate samples, in general, showed good agreement. Figure 4 shows the shear strength as a function of feed slurry. The highest shear strength samples appear to be those containing MST only. The average shear strength of the MST only samples is 765 Pa. The average shear strength of the MST plus sludge samples is 65 Pa for the low sludge case and 122 Pa for the high sludge case.

Table 2. Measured Slurry Rheological Properties

Feed	Time (weeks)	Temp (°C)	Shear Strength (Pa)	Yield Stress (Pa)	Consistency (cp.)
MST	1	30	199	5.0	16
MST	1	30	236	10.3	24
MST	1	60	106	5.3	14
MST	1	60	119	10	24
MST	4	30	489	7.5	18
MST	4	30	533	11.3	27
MST	8	45	3592	12.4	26
MST	8	45	2036	11.8	25
MST	13	30	484	8.6	19
MST	13	30	352	8.7	19
MST	13	45	458	12	31
MST	13	45	421	7.8	21
MST	13	60	762	37	115
MST	13	60	927	36	113
MST-Low Sludge	1	30	32	2.7	7.3
MST-Low Sludge	1	30	36	3.4	7.3
MST-Low Sludge	1	45	19	1.85	6.4
MST-Low Sludge	1	45	44	2.4	6.3
MST-Low Sludge	1	60	38	2.1	6.5
MST-Low Sludge	1	60	35	1.9	6.4
MST-Low Sludge	8	60	86	1.8	6.2
MST-Low Sludge	8	60	100	2.5	7.2
MST-Low Sludge	10	60	77	3.3	7.7
MST-Low Sludge	10	60	156	4.4	9.4
MST-Low Sludge	13	30	35	3.2	7.7
MST-Low Sludge	13	30	20	4.0	7.8
MST-Low Sludge	13	45	53	3.5	7.2
MST-Low Sludge	13	45	63	2.8	6.9
MST-Low Sludge	13	60	118	3.0	7.5
MST-Low Sludge	13	60	130	2.9	7.2
MST-High Sludge	1	30	59	5.1	12.9
MST-High Sludge	1	30	47	5.1	12.9
MST-High Sludge	1	60	79	2.0	6.7
MST-High Sludge	1	60	68	1.9	6.5
MST-High Sludge	4	45	74	2.63	7.39
MST-High Sludge	4	45	95	2.75	7.36
MST-High Sludge	13	30	71	2.4	6.6
MST-High Sludge	13	30	80	4.5	8.1
MST-High Sludge	13	45	75	3.5	7.4
MST-High Sludge	13	45	90	3.4	7.3
MST-High Sludge	13	60	601	3.7	7.9

The table shows a shortage of data points at intermediate settling times (4, 8, and 10 weeks) and intermediate temperatures (45 °C). The lack of data points at these conditions makes determining the time and temperature effects more difficult. The data also shows a few points that appear to be outliers with the remaining data points. These data points (e.g., MST, 8 weeks, 45 °C; MST, 1 week, 60 °C) suggest higher shear strength at lower temperatures and shorter settling times, which is inconsistent with previous test data.[1,2] The authors are working to fill these gaps and determine whether some of the data points are outliers by collecting additional data at intermediate times and temperatures, and repeating tests at conditions where the data points appear to be outliers.

The 8 week, 45°C, MST test was repeated, and the measured shear strength was 350.5 and 193.5 Pa. Rigorous analysis of the results including that data has not been performed. However, if the data in Table 2 are replaced with those data points, the maximum shear strength, yield stress, and consistency for the MST slurries occur at the longest settling time (13 weeks) and the highest temperature (60 °C), which agrees with previous data.[1,2] The maximum shear strength with the MST-High Sludge slurry was measured at 13 weeks and 60 °C. These results will be described, along with the rheology of CST-containing slurries, in a future report.

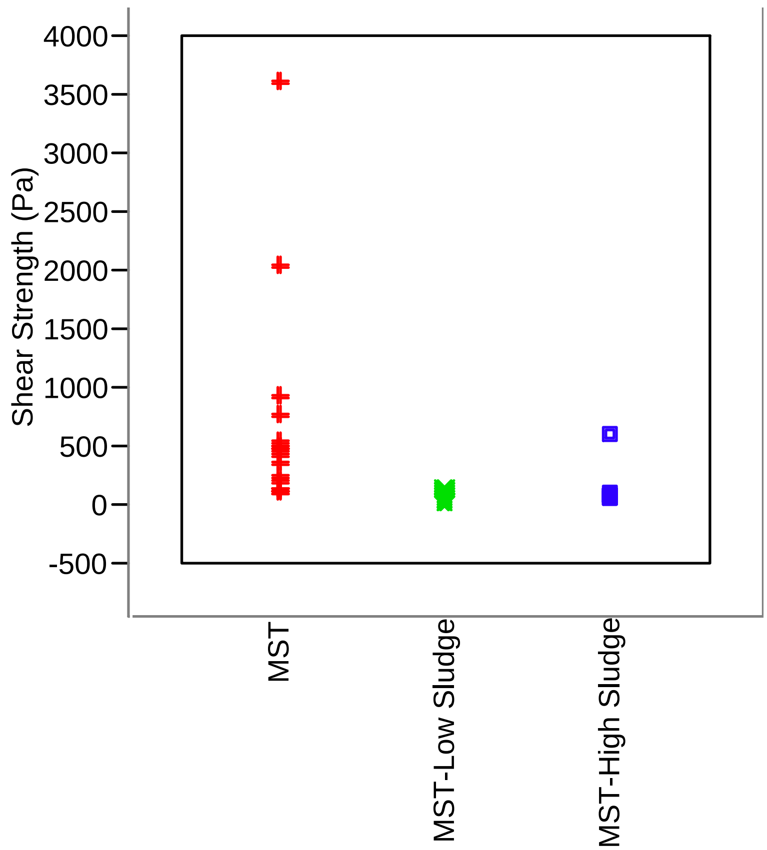

Figure 4. Shear Strength (in Pa) as a Function of Feed Slurry

The authors performed a statistical analysis on the rheology data to determine which parameters (feed slurry, settling time, settling temperature) had a significant effect on the slurry shear strength. They performed the analysis (with JMP® software) by developing a model to calculate the slurry shear strength as a function of feed slurry, settling time, and settling temperature (see equation [1]).

Table 3 shows the basic statistical calculations for a linear model. The software calculated the probability of the variance being due to chance alone. The term "Prob > F" is the probability that the null hypothesis is true (i.e., the variance measured is due to random error). Values less than 0.05 indicate the effect is statistically significant (with 95% confidence). Since the probability is 0.0136, the model contains at least one significant factor.

Table 3 shows the feed slurry has a significant effect on the slurry shear strength (i.e., the "Prob > F" is less than 0.05), with MST-only slurries having the largest shear strength. The table shows that the settling time and temperature are not statistically significant effects. This result is somewhat surprising considering previous testing has shown that MST plus sludge slurries that settle for extended times show a significant increase in shear strength. [1,2] The lack of a measureable effect could be from the shortage of data points, a few outlier points, or because the feed slurry has such a significant effect.

Table 3. Response Shear Strength

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	4673882	1168471	3.6474
Error	36	11532840	320357	Prob > F
C. Total	40	16206722		0.0136

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Feed	2	2	4343358.5	6.7789	0.0032
Time	1	1	262698.8	0.8200	0.3712
Temp	1	1	146308.9	0.4567	0.5035

Feed

Least Squares Means Table

Level	Least Sq Mean	Std Error	Mean
MST	774.66624	152.38399	765.207
MST-Low Sludge	46.93164	143.57339	65.013
MST-High Sludge	135.76967	171.21119	121.508

Researchers looked at the effects of settling time and temperature on the shear strength of each of the feed slurries. Figure 5, Figure 6, and Figure 7 show the shear strength as a function of settling time and temperature. The plots do not show a linear correlation between shear strength and settling time or temperature for these slurries. The plots show a few data points that appear to be outliers. The authors performed a statistical analysis of the effect of settling time and temperature on the shear strength of each feed slurry. Neither time nor temperature had a significant effect on the shear strength of MST-only slurries or the MST plus high sludge slurries (MST:Sludge 1:660). Time and temperature did have a significant effect on the shear strength of the MST plus low sludge slurries (MST:Sludge 1:1.5). This result is consistent with previous testing, and this slurry had an MST:sludge ratio that was similar to the ratio in those tests.[1,2] The authors are collecting more data at intermediate settling times and temperatures, as well as repeating some of the tests that had outlier points, to better determine the impact of settling time and temperature on slurry shear strength. These results will be described in the report describing the rheology of CST-containing slurries.

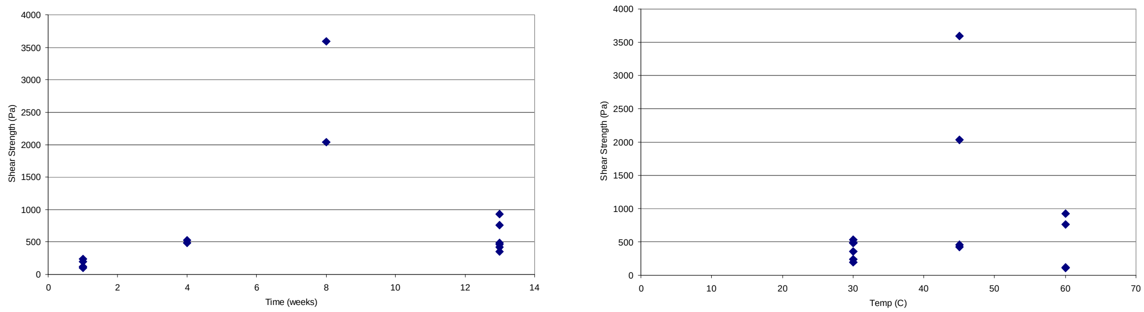

Figure 5. Impact of Settling Time and Temperature on Shear Strength of MST Slurries

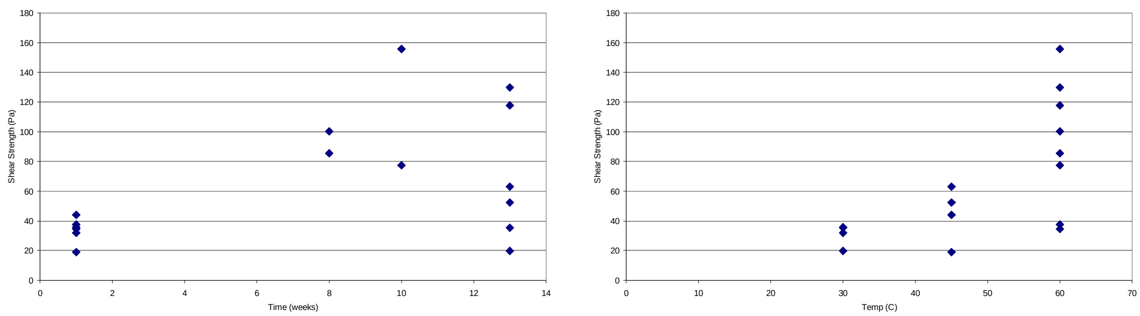

Figure 6. Impact of Settling Time and Temperature on Shear Strength of MST Plus Low Sludge Slurries

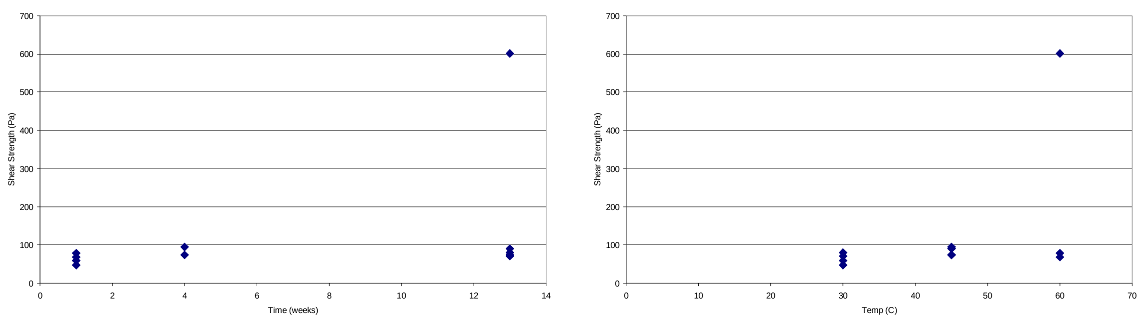

Figure 7. Impact of Settling Time and Temperature on Shear Strength of MST Plus High Sludge Slurries

4.2 FLOW CURVE MEASUREMENTS

This section covers rheological results on free flowing, well-mixed slurries where the solids from the vane tests in Section 4.1 were fully resuspended in the supernate. Concentric cylinder flow curve data were fit to a Bingham plastic equation to give a yield stress (intercept) and consistency (slope). Figure 8 shows the slurry yield stress as a function of feed. The plot, as well as Table 2, shows that the yield stress appears to be a function of feed, with the MST-only slurries having the largest yield stress. MST only slurries also had about twice the wt% insoluble solids content of both the low and high sludge slurries which likely explains much of the difference between the three types. A comparable statistical analysis was performed to determine whether any of the test parameters has a statistically significant effect on the slurry yield stress. Table 4 shows the results. Since the "Prob>F" is < 0.0001, the model contains at least one significant factor. Table 4 shows the feed slurry has a significant effect on the slurry yield stress, with MST only slurries having the largest yield stress. The table shows settling time and temperature are not statistically significant effects. This result is again surprising considering previous testing has shown that MST plus sludge slurries that settle for extended times show a significant increase in yield stress.[2,1] The lack of a measureable effect could be from the shortage of data points, a few outlier points, or because the feed slurry has such a significant effect.

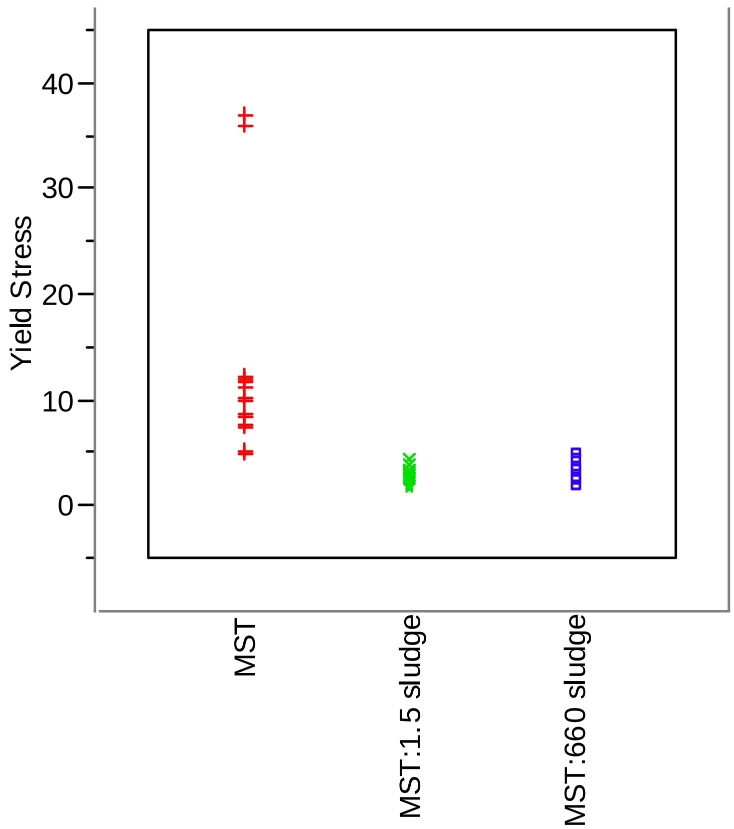

Figure 8. Yield Stress (in Pa) as a Function of Feed Slurry

Table 4 Response Yield Stress

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	1154.8574	288.714	9.0651
Error	36	1146.5643	31.849	Prob > F
C. Total	40	2301.4218		<.0001

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Feed	2	2	990.95338	15.5571	<.0001
Time	1	1	114.43730	3.5931	0.0661
Temp	1	1	97.45924	3.0600	0.0888

Feed

Least Squares Means Table

Level	Least Sq Mean	Std Error	Mean
MST	13.379004	1.5193941	13.1214
MST-Low Sludge	2.400848	1.4315452	2.8594
MST-High Sludge	3.700944	1.7071168	3.3618

Researchers again looked at the effects of settling time and temperature on each of the feed slurries. Figure 9, Figure 10, and Figure 11 show the yield stress as a function of settling time and temperature. The plots do not show a direct correlation between yield stress and settling time or temperature for these slurries. The authors performed a statistical analysis of the effect of settling time and temperature on the yield stress of each feed slurry. The data show temperature has a significant effect on the yield stress of MST only slurries and MST plus high sludge slurries, while time has a significant effect on MST plus low sludge slurries. However, the effect of time on MST plus high sludge and the effect of temperature on MST plus low sludge are negative, which contradicts previous test data.[1,2] The authors are collecting additional data to better quantify these effects. These results will be included in the report describing the rheology of CST-containing slurries.

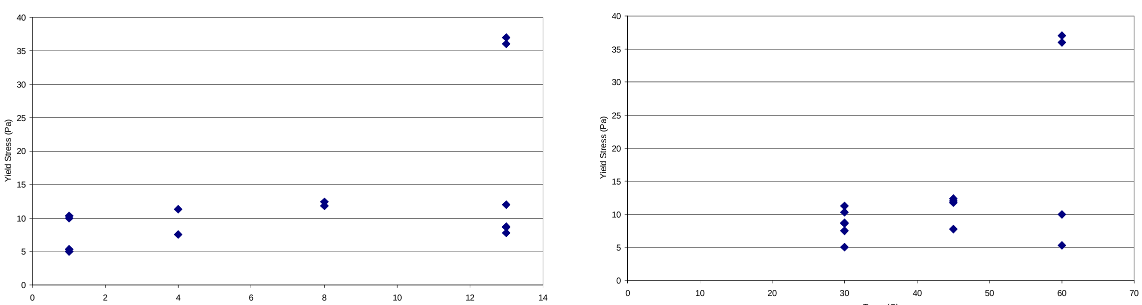

Figure 9. Impact of Settling Time and Temperature on Yield Stress of MST Slurries

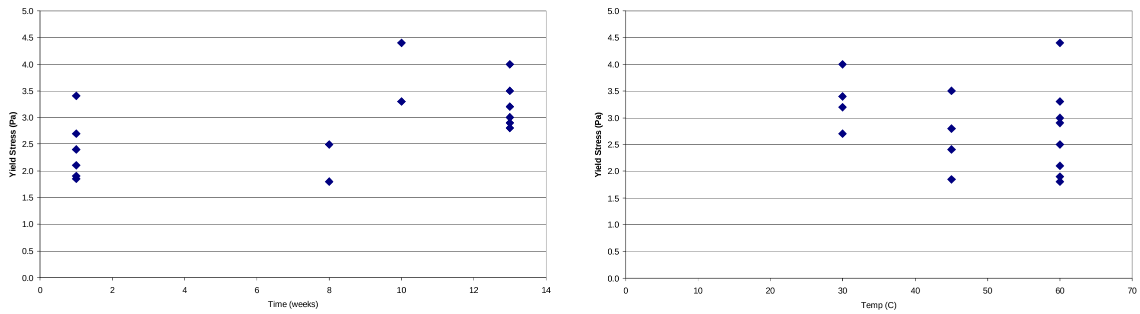

Figure 10. Impact of Settling Time and Temperature on Yield Stress of MST Plus Low Sludge Slurries

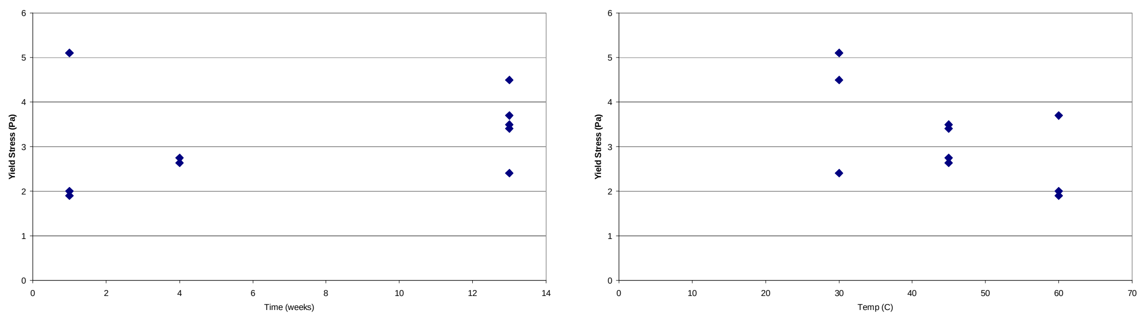

Figure 11. Impact of Settling Time and Temperature on Yield Stress of MST Plus High Sludge Slurries

Figure 12 shows the consistency data plotted as a function of feed slurry. A comparable statistical analysis was performed to determine whether any of the test parameters has a statistically significant effect on the slurry consistency. Table 5 shows the results. Since the "Prob>F" is 0.0004, the model contains at least one significant factor. Table 5 shows the feed slurry has a significant effect on the slurry consistency, with MST only slurries having the largest consistency. The table shows settling time and temperature are not statistically significant effects.

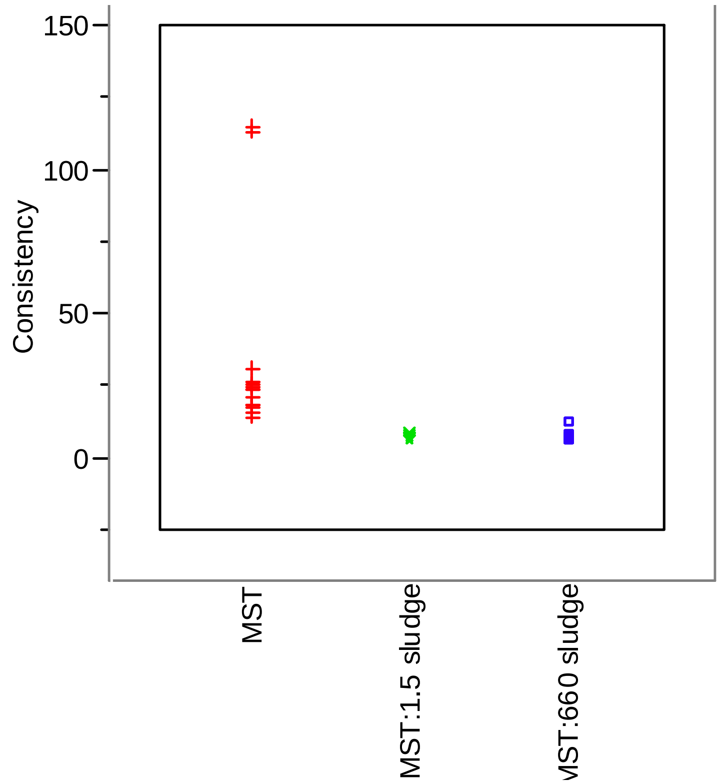

Figure 12. Consistency (in Centipoise) as a Function of Feed Slurry

Table 5. Response Consistency

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	9313.981	2328.50	6.6944
Error	36	12521.704	347.83	Prob > F
C. Total	40	21835.685		0.0004

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Feed	2	2	7682.6644	11.0439	0.0002
Time	1	1	859.6316	2.4714	0.1247
Temp	1	1	1378.2283	3.9624	0.0542

Feed

Least Squares Means Table

Level	Least Sq Mean	Std Error	Mean
MST	36.169420	5.0211472	35.1429
MST-Low Sludge	5.498295	4.7308323	7.1875
MST-High Sludge	9.427764	5.6415150	8.2773

Figure 13, Figure 14, and Figure 15 show the effect of settling time and temperature on the consistency of each feed slurry. Examination of Figures 13 – 15 does not show an obvious correlation between consistency and settling time or temperature. Statistical analysis of the data shows that temperature has a significant effect on the consistency of MST only slurries.

However, the analysis indicates that increasing temperature decreases the consistency, which contradicts previous testing.[1,2] This result could be from the temperature having a small effect, the few outlier points, or from the consistency being confounded with the yield stress. Statistical analysis of the MST plus high sludge slurries shows that settling time has a significant effect on consistency, with increasing time yielding larger consistency. Neither time nor temperature has an impact on the MST plus low sludge slurries. The authors are collecting additional data to better quantify these effects. These results will be included in the report describing the rheology of CST-containing slurries.

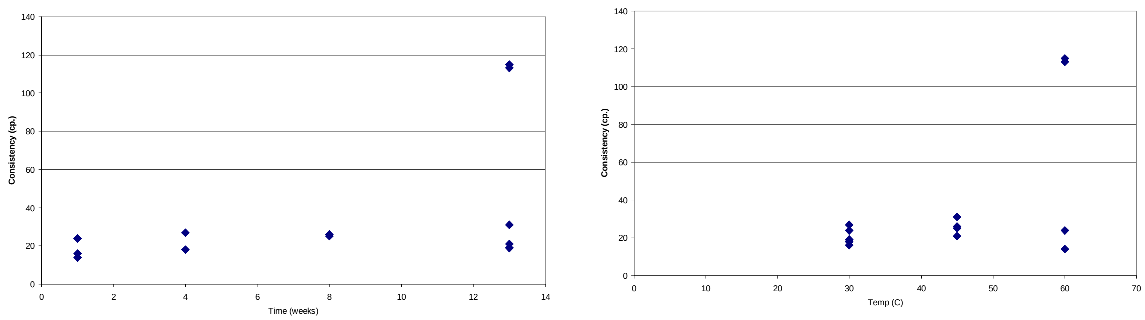

Figure 13 Impact of Settling Time and Temperature on the Consistency of MST Slurries

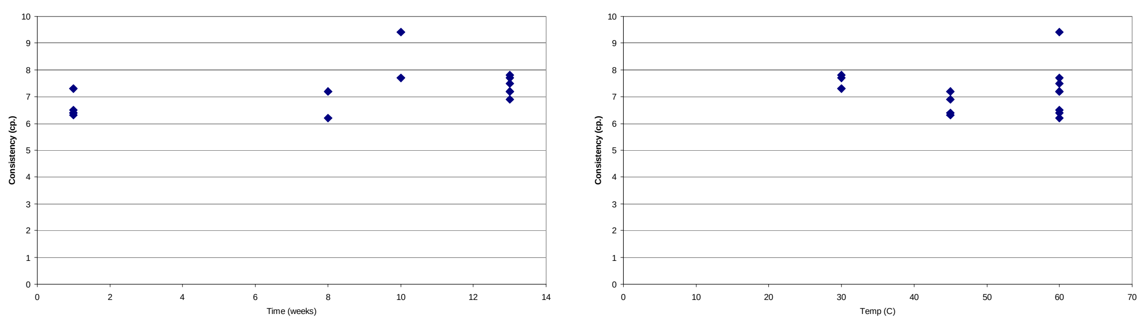

Figure 14. Impact of Settling Time and Temperature on the Consistency of MST Plus Low Sludge Slurries

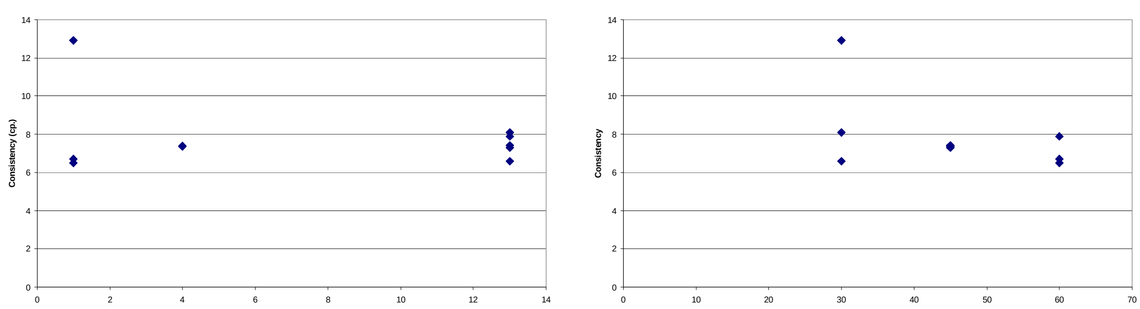

Figure 15. Impact of Settling Time and Temperature on the Consistency of MST Plus High Sludge Slurries

4.3 OBSERVATIONS

After preparing the samples, SRNL personnel mixed them so all of the solid materials would be dispersed throughout the solid phase. However, after sitting for several weeks at elevated temperature, the solid particles appear to have segregated (see Figure 16). SRNL is currently investigating this phenomenon and will continue to monitor the rheology samples to better understand it.

Figure 16. Rheology Samples after Sitting

5.0 CONCLUSIONS

The conclusions from this analysis follow:

- MST only slurries that sat at elevated temperatures had larger shear strength, yield stress, and consistency than MST plus sludge slurries that settled at elevated temperatures.
- The addition of sludge to an MST slurry reduces the shear strength, yield stress, and consistency.
- The impact of settling time and temperature on slurry rheology is inconclusive at this time. The authors are collecting additional data to attempt to determine the impact of settling time and temperature on slurry shear strength, yield stress, and consistency.

6.0 PATH FORWARD

The authors plan to complete the rheology tests with the CST-containing slurries. In addition, they plan to repeat tests with MST and sludge-containing slurries that produced data points that appeared to be outliers. To better determine the effects of settling time and temperature on slurry rheological properties, they will conduct additional tests at intermediate settling times and temperatures. These results will be documented in a future report.

7.0 REFERENCES

[1] Poirier, M. R.; Herman, D. T.; Fondeur, F. F.; Hansen, E.; and Fink, S. D. "MST/Sludge Agitation Studies for Actinide Removal Process and DWPF", WSRC TR-2003-00471, October 10, 2003.

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[5] D. C. Koopman, Rheology Protocols for DWPF Samples, WSRC-RP-2004-00470, October, 2004, Savannah River Site, Aiken, SC, 29808.