STUDENT SUMMER INTERNSHIP TECHNICAL REPORT

Modeling of Chemical Slurry Rheology in DWPF Sludge Batch (SB) 10 Simulants

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EXECUTIVE SUMMARY

The Defense Waste Processing Facility (DWPF) treats high-activity radionuclides from sludge through a process called vitrification. This process converts radioactive liquid waste currently stored in tank farms into a solid glass form that is suitable for long-term storage and disposal. Due to the complexities involved in vitrifying this waste within each operation of the Chemical Processing Cell (CPC), waste rheology is studied to characterize the fluid-mechanical properties as it passes through the CPC and into the Melter. To better understand the waste and validate flow behavior, slurry rheology of simulants that represents the waste was studied at various acid stoichiometry percentages and solids concentrations to determine the simulant's yield stress and viscosity.

This research work has been supported by the DOE-FIU Science & Technology Workforce Development Initiative, an innovative program developed by the U.S. Department of Energy's Office of Environmental Management (DOE-EM) and Florida International University's Applied Research Center (FIU-ARC). During the spring of 2022, a DOE Fellow intern, Brendon Cintas, spent 10 weeks doing a summer internship at Savannah River National Laboratory (SRS) under the supervision and guidance of Dan Lambert, Chemical Flowsheet Development. The intern's project was initiated on June 6, 2022, and continued through August 11, 2022 with the objective of assisting scientists at SRNL's Rheology and Grout Laboratory at Aiken Country Technology Lab (ACTL) better understand the effect of sludge composition on the rheology of a simulant slurry using a HAAKE RheoStress 6000 rheometer and extrapolate the results to the real-waste data.

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LIST OF ACRONYMS

ACTL	Aiken County Technology Laboratory
CPC	Chemical Processing Cell
DWPF	Defense Waste Processing Facility
MFT	Melter Feed Tank
MST/SS	Monosodium Titanate
PRFT	Precipitate Reactor Feed Tank
SB	Sludge Batch
SEFT	Strip Effluent Feed Tank
SME	Slurry Mix Evaporator
SRAT	Sludge Receipt and Adjustment
SRMC	Savannah River Mission Completion
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
SS	Sludge Solids
SWPF	Salt Waste Processing Facility

1. INTRODUCTION

At the Savannah River Site (SRS), radioactive liquid waste exists in sludge (liquid) forms and salt (solid) forms that is treated for long-term storage. The Defense Waste Processing Facility (DWPF) at the site is designed to treat high-activity radionuclides from the waste. At DWPF, the waste undergoes a process known as vitrification [1], in which the liquid waste is converted into a solid glass form suitable for long-term storage and disposal in a federal repository once available, while the solid-form waste is transferred to the Salt Waste Processing Facility (SWPF) to be converted into saltstone after decontamination. During the process of liquid-waste vitrification, the sludge is processed via the Chemical Processing Cell (CPC) in which chemical treatments and solids additions are conducted to target operation parameters within DWPF's acceptable safety criteria.

The process of vitrification begins in the high-volume underground storage tanks in which more than 1 million gallons of high-level legacy waste is stored. This waste in these containers exists in two forms: a liquid sludge form and salt form [2]. From a selection of storage tanks, the liquid waste enters Tank 51 (Tk51), which batches the waste from the tank streams. This batch is then transferred to Tank 40 (Tk40) and blended with the remnants of the previous Sludge Batch (SB) – this process generates the new SB that enters DWPF. Then, the blended batch is transferred to DWPF in a smaller batch of approximately 6000 gallons. It enters the first of three vessels of the CPC: the Sludge Receipt and Adjustment Tank (SRAT). Here, the sludge is treated with nitric-glycolic acid (previously nitric-formic acid); next, the slurry is transferred to the Slurry Mix Evaporator (SME) vessel in which a sand-like borosilicate ("frit") is added and mixed into the sludge; after addition of the frit within SME, the batch is transferred to the Melter feed tank (MFT). The MFT operates as a holding tank for the sludge before finally being sent to the Melter. Due to the complexities of the process, the slurry must be within an acceptable yield stress and viscosity before it enters the Melter, and the slurry's properties must not be below (too thin) or above (too thick). If the slurry is thicker than the maximum acceptable yield stress and viscosity, then it can neither be mixed nor pumped, resulting in potential plugging of the feed piping to the Melter. Conversely, if the slurry is too thin, then the solids settle too quickly.

In the case of DWPF, knowing the properties that yield a certain rheology is critical due to the potential increase in size of equipment needed to transport, mix, or pump the fluid, and improper equipment could lead to process upsets, equipment damage, or even total failure [3]; however, Conducting rheological studies of high-activity waste is laborious, costly, and it could potentially expose personnel to risk [3]. Thus, to better determine operations at DWPF without analyzing real waste, Savannah River Mission Completion (SRMC), the current Liquid Waste Operator (LWO) at SRS requested that Savannah River National Laboratory (SRNL) conduct laboratory-scale simulations of the DWPF CPC operations using non-radioactive, chemically similar simulants to develop a flowsheet in support of operations of Sludge Batch 10 [4]. During testing, fourteen CPC simulant tests were performed at the Aiken County Technology Laboratory (ACTL) in Aiken, SC to simulate the processes within SRAT and SME. At the end of both the SRAT and SME cycles for the CPC operation simulations, a sample of the slurry was collected. These samples were studied at various acid stoichiometry percentages and solids loading to determine the simulant's yield stress in Pascals (Pa) and viscosity in centipoise (cP) to evaluate the slurry's rheological properties as it moves through the DWPF.

2. RESEARCH DESCRIPTION

A. Theory

A Newtonian fluid is a type of fluid that deforms linearly when applied a shearing stress τ at a controlled rate $\dot{\gamma}$. This behavior can be written as the shear stress as a function of the speed at which stress is applied:

$$\tau(\dot{\gamma}) = \mu\dot{\gamma} \tag{1}$$

When a fluid undergoes an applied force, the fluid resists the flow. This is defined as *dynamic viscosity* μ, which is the ratio of the shearing stress τ to the velocity gradient du/dy in a fluid [5]:

$$\mu = -\tau\left(\frac{du}{dy}\right)^{-1} \tag{2}$$

Figure 1 represents the flow behavior of a Newtonian, National Institute of Standards and Technology (NIST) traceable viscosity reference oil standard in which the published viscosity of the fluid is ~92 cP at 20°C. In the figure, the slope, which is the ratio of change in shear stress against change in shear rate, is the fluid's viscosity. As shown, the change in the stress versus the change in shear rate is constant across the entire domain. This indicates that the fluid's dynamic viscosity is independent from the rate of flow, and the viscosity is constant across the entire flow regime. This is also shown in the lighter colored curve, which shows the viscosity as a function of the strain rate $\mu(\dot{\gamma})$. There is some noise from 0-100 inverse seconds of shear rate, but as the shear rate increases, it is mostly constant. Averaging the values of the viscosity across the entire range returns a near-identical value of 92-8 cP. Another characteristic of a Newtonian fluid is that, when the curve is extrapolated from zero inverse seconds of shear rate, the shear stress is also zero. This implies that the fluid yields immediately, as the shear stress crosses through the origin.

Non-Newtonian fluids, however, behave non-linearly with respect to an applied stress across the range of rate of shear. These are referred to as power-law fluids, which are mathematically represented by *K* – the flow consistency index, multiplied by the shear rate raised to an exponent *n,* which represents the flow behavior index:

$$\tau(\dot{\gamma}) = K\dot{\gamma}^n \tag{3}$$

K, the flow consistency index, determines the consistency of the fluid. Larger values of *K* indicate thicker fluids. The flow behavior index *n* indicates linearity of the fluid. For an index $n = 1$, there is a linear relationship between shear stress and shear rate. For values of $n \neq 1$, however, a shear-dependent viscosity – *apparent* viscosity, can be determined, in which viscosity varies across the entire range of shear [6].

$$\mu = K(\dot{\gamma})^{n-1} \tag{4}$$

Another notable attribute that distinguishes a Newtonian from non-Newtonian fluid is the yield stress of a fluid. Yield stress is a parameter of a non-Newtonian fluid which characterizes a maximum allowable load before the fluid begins to deform.

$$\tau(\dot{\gamma}) = K\dot{\gamma}^n + \tau_0 \tag{5}$$

Like solid mechanics, when a fluid is applied a shear stress that is below the maximum allowable load, the fluid retains its original shape once the stress is removed from it; this is known as elastic deformation. However, if the stress is beyond the maximum allowable load of the fluid, the fluid will no longer be able to retain its original shape when the stress is removed; this is plastic deformation. In the case of fluids, plastic deformation is defined as the deformation in which the fluid begins to flow. Figure 2 represents the flow behavior of a non-Newtonian slurry. In the figure, the viscosity is shear dependent as shown by the curve's non-linearity. As well, the fluid's shear stress at near-zero shear rate is not also zero, which demonstrates that the fluid has a yield stress.

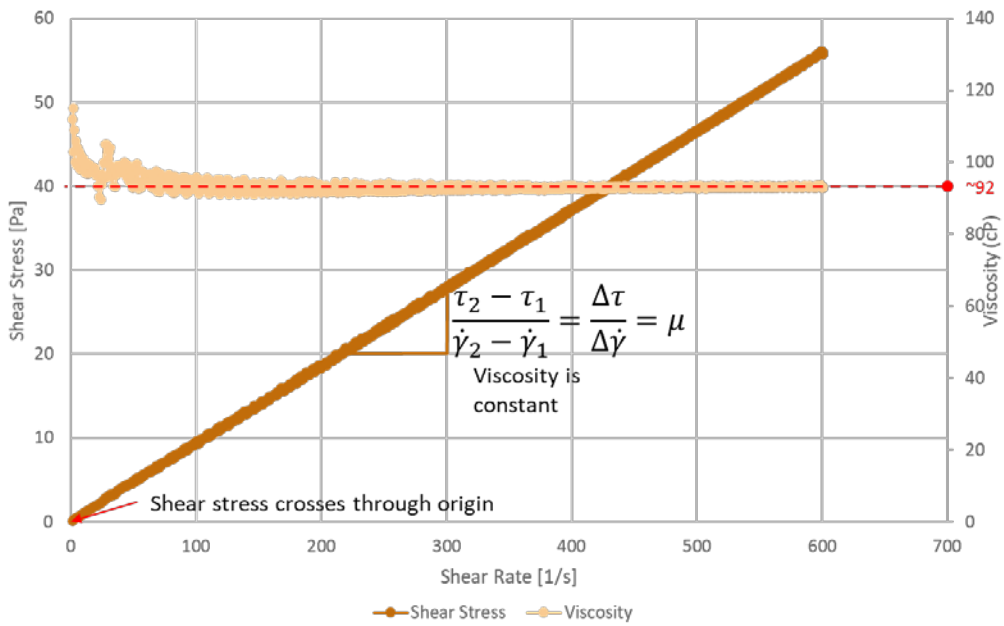

Figure 1. NIST Traceable Viscosity Reference Standard N44 at 20°C

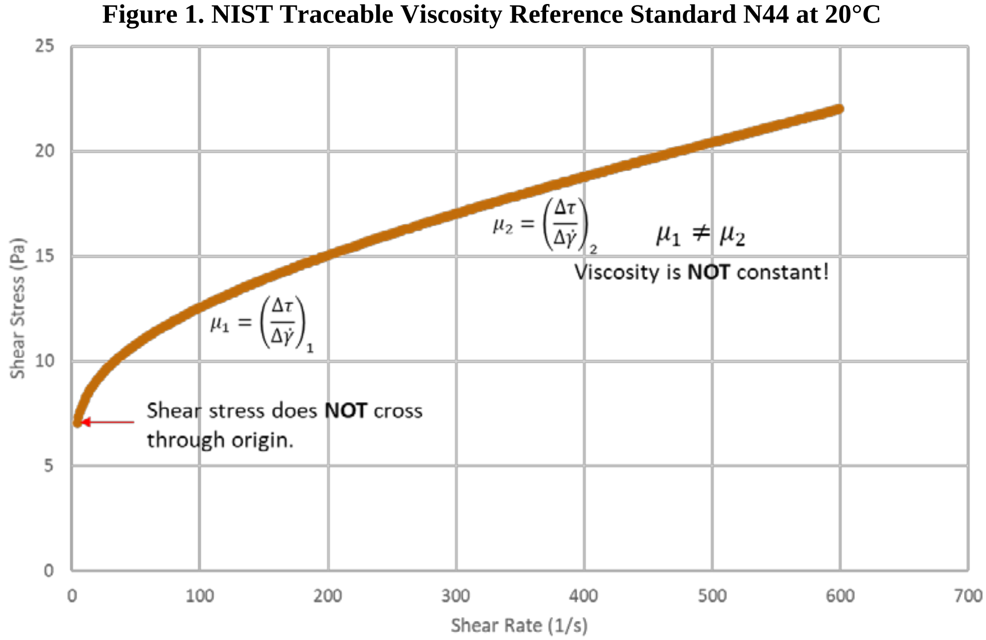

Figure 2. Tk40-1 SRAT Slurry Simulant at 20°C

B. Curve-Fitting and Interpolation

In each experiment, a data set composed of the shear stress, and shear rate is generated. Using this data, the flow curves shown in either Figure 1 (for a Newtonian fluid) or Figure 2 (for a non-Newtonian fluid) can be generated. Then, from the flow curve, rheological properties can be extrapolated via curve-fitting – a numerical modeling process that constructs a mathematical function ("curve") that best fits a dataset. In modeling the flow curve, the rheological properties are predicted based on known models derived from analytical solutions. For both Newtonian and non-Newtonian fluids, a linear fit is often used. For Newtonian fluids, this is shown in Equation (1). For non-Newtonian fluids, however, the flow can be modeled also as a Newtonian fluid, only after the stress applied is beyond the yield stress. This is known as "Bingham Plastic", in which a fluid is elastic until yielded, but its plastic behavior can be approximated to Newtonian flow behavior, as shown in Equation (6):

$$\tau(\dot{\gamma}) = \mu\dot{\gamma} + \tau_0 \tag{6}$$

In the Bingham Plastic model, because it is modeled after a Newtonian fluid, a constant viscosity and yield stress can be obtained. However, the original flow curve shown in Figure 2, when approximated using the Bingham Plastic model, is non-linear, and its non-linearity contributes to a predicted yield stress that is larger than its true yield stress, as shown in Figure 3. Thus, another model to determine the yield stress to a much greater accuracy is the Herschel-Bulkley model, which is shown in Equation (5). In the Herschel-Bulkley model, non-linearity is accounted for through the flow index *n* and consistency index *K*; however, there is no way to interpolate a constant viscosity as the viscosity is no longer constant across the entire flow regime. Figure 3 represents the comparison between the two models, and the data that can be extracted from them.

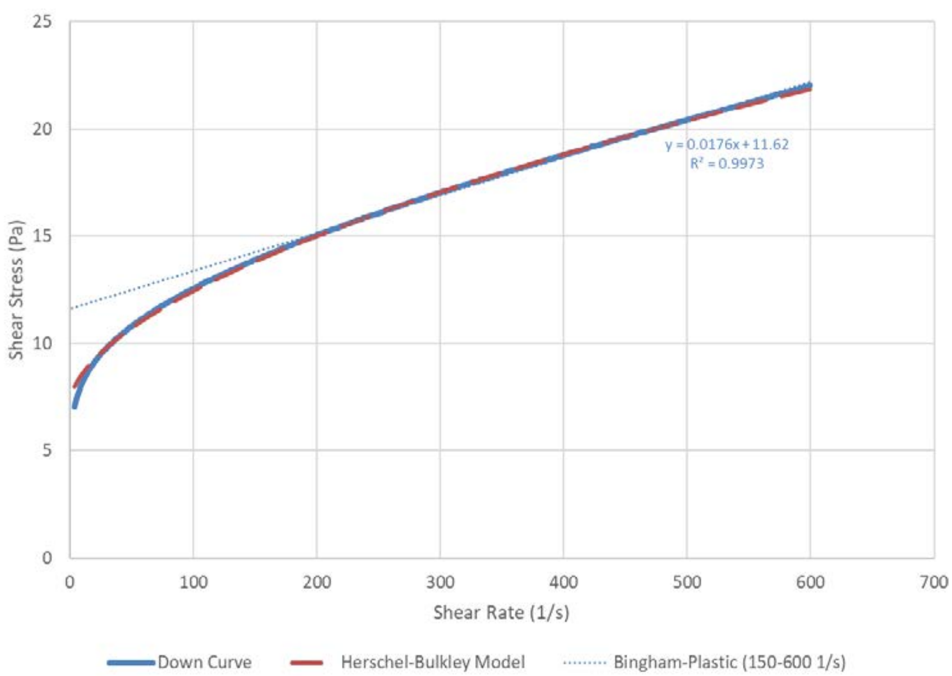

Figure 3. Comparison between the Herschel-Bulkley and Bingham Plastic models for Tk40-1 SRAT Slurry Simulant at 20°C.

C. Affecting Rheology within DWPF

Rheology is the most important processing parameter in defining the CPC operating window for the nitric-glycolic flowsheet. During operations within the CPC, the receipt slurry and the slurry that enters the SRAT, SME, and Melter are significantly different from a rheological perspective. This is due to the changes that the slurry undergoes during each phase of the CPC: In the SRAT, the acid treatment causes the insoluble solids dissolve, reducing the mass of insoluble solids occupied within the sample. The SME, however, causes an increase in rheology, due to the addition of frit. Table 1 shows DWPF's design basis rheology and target slurry rheology at each phase of the CPC. As seen in the table, the receipt sludge is thinned considerably in SRAT, and slightly more concentrated in the SME. Varying the acid addition with SRAT or waste loading by combining PRFT/SEFT and addition of frit can affect the rheology of the slurry as it passes through DWPF. In a sense, the rheology can then be parametrized, where rheology can be trimmed to ensure that the slurry can be fed into the Melter without complication.

Table 1. Slurry Rheology Design Criteria in each phase of DWPF CPC

Property	Sludge	SRAT	SME
Total Solids (wt.%)	13-19	18-25	40-50
Yield Stress	25-100	1.5-5	2.5-15
Consistency (cP)	4-12	5-12	10-40

3. RESULTS AND ANALYSIS

In ACTL, fourteen CPC simulant tests were performed to simulate the chemical and physical effects on the slurry simulant as it goes through DWPF. At the end of each phase, a sample was collected and labeled as Tk40 or Tk51 followed by a number 1-10 for Tk40, and 1-4 for Tk51. Tk51 were sludge-only experiments at the design basis boilup rate that supports the SRNL Shielded Cells qualification test. Ultimately, they were performed to understand the acid stoichiometry and acid loadings that produced sludge capable of being processed. Tk40, however, supported SB10 implementation within DWPF, and were extensively tested. Tk40-1 through Tk40-7 test SRAT-only experiments to understand acid stoichiometry impact on CPC processing (reported as a percentage of Koopman Minimum Acid [%KMA]). Tk40-8 extends the knowledge gained from Tk40-1 through Tk40-7 into the SME cycle. Tk40-9 analyzes lower boilup rates within DWPF, and Tk40-10 quantifies changes associated with introduction of SWPF waste streams [4].

All slurry simulant rheology data was generated using a HAAKE Rheostress 6000 rheometer and Z41 concentric cylinder spindle and cup. The rheometer was operated at a controlled rate of shear that started from zero and terminated at a high frequency, but not high enough to require consideration of wall effects. The rheometer increased the rate of shear linearly, until the destination frequency is attained. The rheometer rotated at the set speed for a fixed length of time before decreasing back to zero in the same linear manner. The result of each trial is one up-curve in which the shear increases, and one down-curve in which the shear decreases. Figure 4 shows a sample of Tk40-10 SME Product simulant, and the effect settling solids (such as frit) has on the flow curve for the same sample operating at the same frequencies.

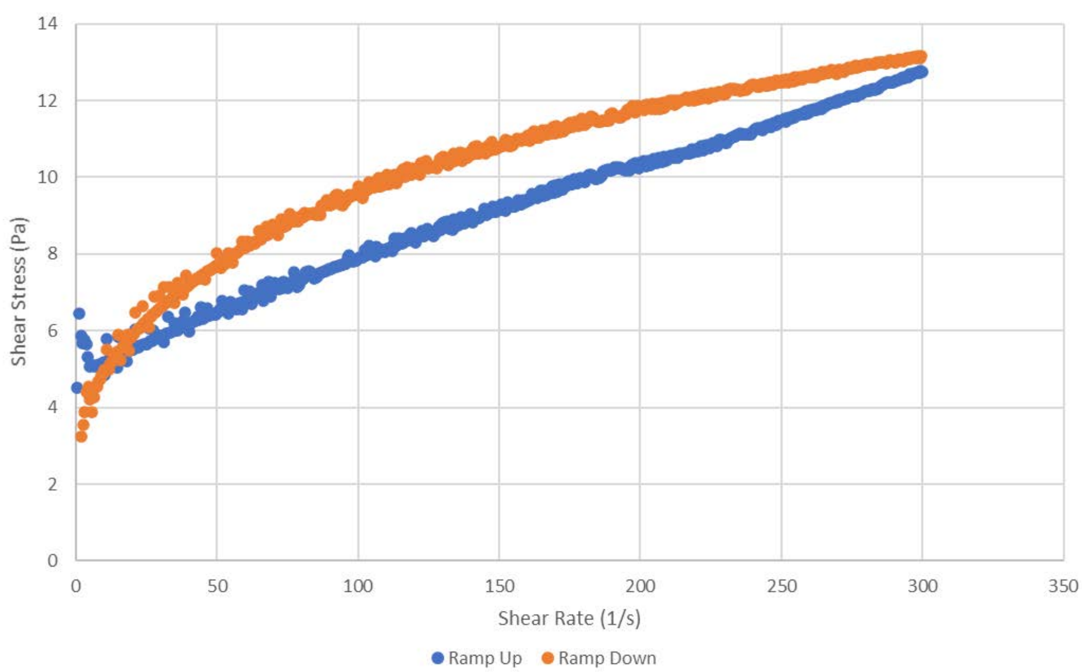

Figure 4. Tk40-10 SME Product Flow Curves for Ramp-up and Ramp-down rheology operations.

For Tk40-1 through Tk40-7, each product was subsampled for further analysis in solids concentration. Each subsample varied in their weight percentages due to frit addition, insoluble solids, and dissolved solids due to acid addition, and the summation of all three weight fractions yields the weight fraction of total solids – one of the two parameters for analyzing slurry rheology.

- *Insoluble Solids*

$$(wt.\ \%)_{IS} = \frac{m_{IS}}{m_{SRAT} + m_F - m_{wr} + m_{wa}}$$

- *Frit*

$$(wt.\ \%)_F = \frac{m_F}{m_{SRAT} + m_F - m_{wr} + m_{wa}}$$

- *Dissolved Solids*

$$(wt.\ \%)_{DS} = \frac{m_{TS} - m_{IS}}{m_{SN} - m_{wr} + m_{wa}}$$

- *Total Solids*

$$(wt.\%)_{TS} = (wt.\%)_{IS} + (wt.\%)_F + (wt.\%)_{DS}$$

Figure 5 represents the effect of solids loading on Tk40-1 subsamples and their corresponding yield stresses as a function of the weight fraction of total solids. As shown, as solids concentration increases, yield stress increases exponentially. Further analysis in Figure 5 also shows this trend in varying degrees of intensity for each type of solid loading within the simulant. In Figure 6, this exponential behavior is clearly defined in the dissolved solids, but the same cannot be said for frit addition – which shows a largely-scattered dataset, and insoluble solids – which does not seem to change yield stress significantly in varying its weight fraction. Though not apparent, insoluble solids and frit addition do have an impact on the yield stress and would better be represented in tandem with the effect dissolved solids has on yield stress.

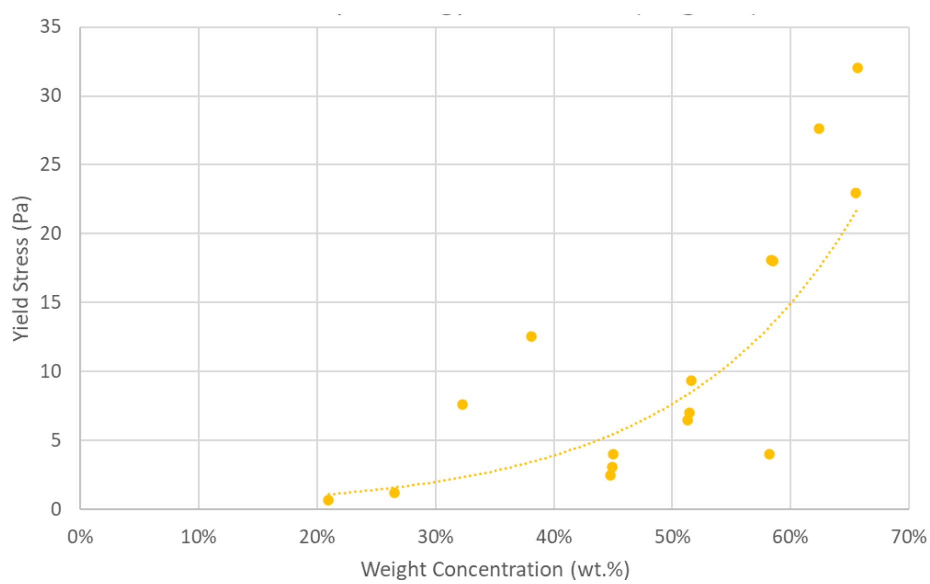

Figure 5. Tk40-1 Yield Stress as a function of the total weight fraction.

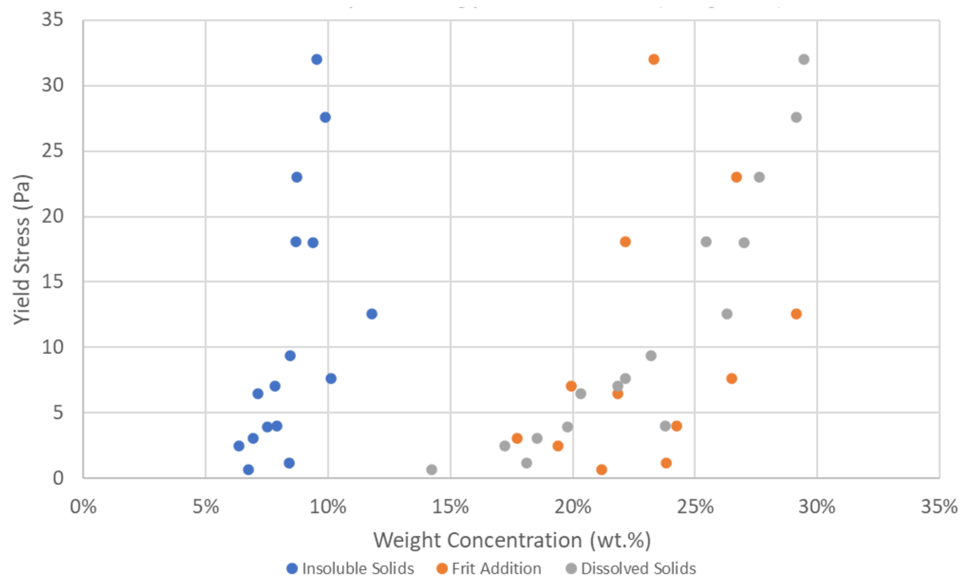

Figure 6. Tk40-1 Yield Stress as a function of (a) Insoluble Solids (b) Frit Addition (c) Dissolved Solids.

The composition of the sludge was also capable of determining an acid requirement [4]. An effective acid stoichiometry metric was used to determine the acid stoichiometry of each slurry simulant:

$$[\eta_{acid}] = [OH_{equiv}] + [Hg] + [CO_3]_{dis} + [NO_2] + 1.5([Ca] + [Mg] + [Mn])$$

Where:

$[\eta_{acid}]$ is required amount of acid, $[OH_{equiv}]$ is the base equivalents concentration, $[Hg]$ is the concentration of mercury, $[CO_3]_{dis}$ is concentration of soluble carbonate, $[NO_2]$ is concentration of nitrite, $[Ca]$ is concentration of calcium, $[Mg]$ is concentration of magnesium, and $[Mn]$ is the concentration of manganese [7]. Table 2 represents the acid stoichiometries and their corresponding rheology as a function of the acid stoichiometries are presented in Figure 6. As shown in the figure, both samples follow a similar trend whereas acid stoichiometry increases, the fluid is thinned, indicated by a reduction in viscosity Figure 7) and yield stress (Figure 8). From the data and the design requirements shown in Table 1, it can be indicated that for SRAT, stoichiometries above 110% KMA fall below DWPF's minimum of 1.5 Pa of yield stress and 5 cP of viscosity; conversely, stoichiometries below 100% produced a SRAT product higher than DWPF's maximum of 5 Pa of yield stress, even though no SRAT product that exceeded DWPF's viscosity requirement was produced.

Table 2. Acid Stoichiometry for Tank 40 and Tank 51 Slurry Simulants

Experiment	%KMA	Yield Stress (Pa)	Viscosity (cP)
Tk40-1	107.9	3.30	7.80
Tk40-2	127.1	0.10	3.20
Tk40-3	107.9	2.10	7.30
Tk40-4	128.6	0.40	3.90
Tk40-5	118.3	0.40	3.80
Tk40-6	92.3	10.80	11.00
Tk40-7	137.7	0.10	3.20
Tk40-8	105.7	2.40	7.50
Tk40-9	103.6	2.70	8.00
Tk40-10	106.2	0.30	3.50
Tk51-2	116.6	0.90	8.60
Tk51-3	106.1	0.80	5.30
Tk51-4	107.3	4.60	9.40

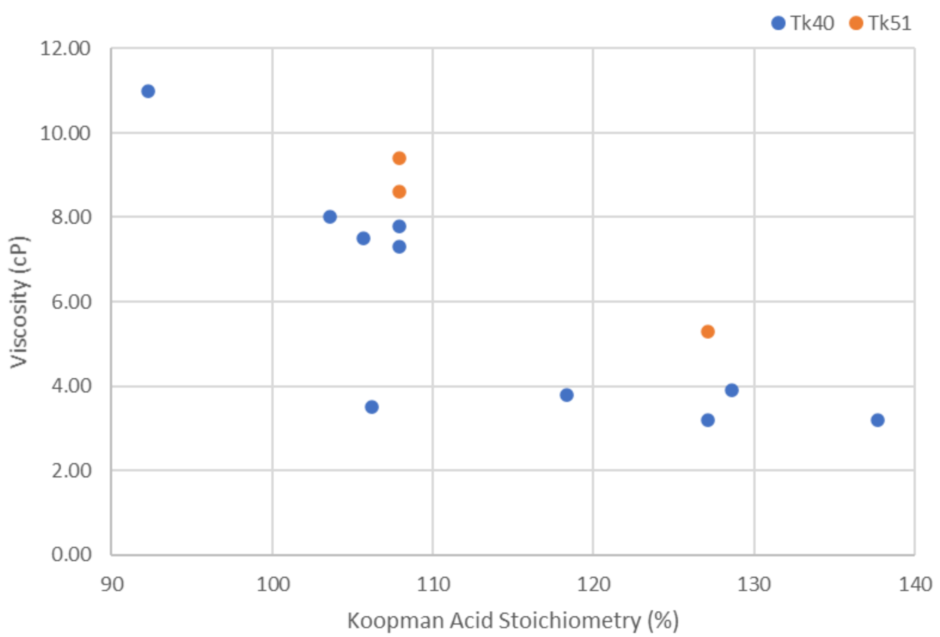

Figure 7. Viscosity versus Acid Stoichiometry of Tk40 and Tk51 simulants.

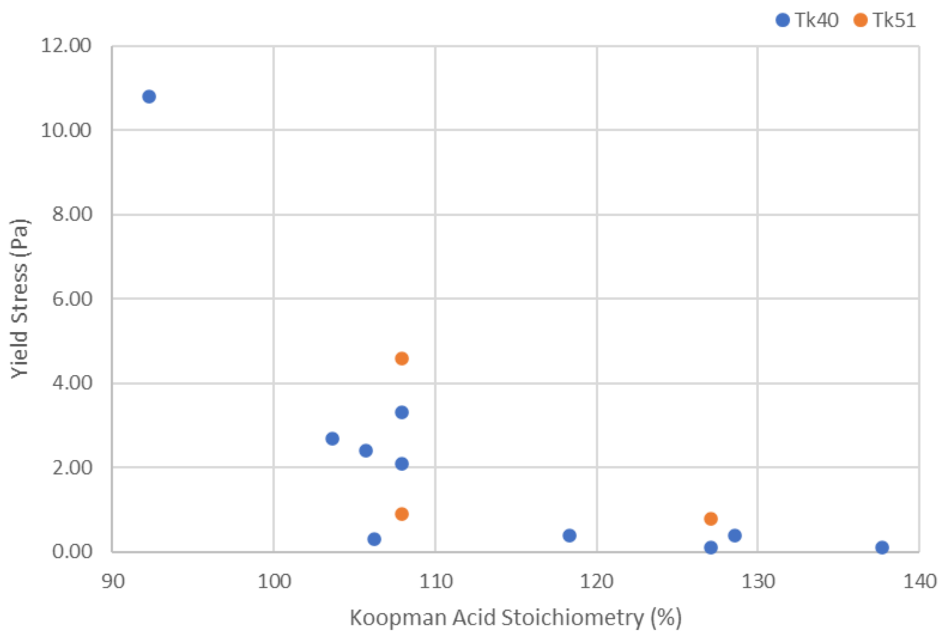

Figure 8. Yield Stress versus Acid Stoichiometry of Tk40 and Tk51 simulants.

Additional research into vane rheology was also conducted. Vane rheology measures yield stress using a rotating vane that is submerged into the medium and deformed at a constant, controlled rate of shear. The shear stress as a function of time is plotted, and the maximum value of shear stress of that function is the yield stress of the fluid. Three SME products were selected for analysis using the vane: Tk40-10, Tk51-3, and Tk51-4, due to the availability of the slurry for subsampling and thicker consistency, which reduces risk of noise within the rheology curve. Each simulant was tested once using a Z38 concentric cylinder and cup to generate a flow curve that plots shear stress against shear rate. Then, after the flow curve was generated, more simulant was added and stirred for approximately one minute before replacing the concentric cylinder for an FL22 vane. The vane was then immersed within the fluid and sheared a low, constant controlled rate. Figure 9 shows curve modeling shear stress as a function time for Tank 51-3 SME product at a controlled rate of 0.03 s^{-1}. As shown, a clearly defined maximum of 16.1 can be visualized, which infers a yield stress of approximately 16 Pascals for the Tk51-3 sample. Two additional tests were conducted in this manner for each SME product to determine a mean yield stress. This average yield stress can then be defined as an initial value in the Herschel-Bulkey model to generate a second flow curve that more accurately predicts the flow behavior. This is achieved through numerical modeling in which a computational tool attempts to maximize a goodness of fit (R^2) value onto the existing data set by varying the consistency index and flow index, whilst keeping the mean yield stress fixed. Results shown in Figure 10 (Tk51-3 SME Product Flow Curve) and Table 3 (results for Tk40-10, Tk51-3, and Tk51-4) all show a goodness of fit within 10% of error, indicating validity in the proof of concept for measuring yield stress of future SB slurries using a vane as opposed to the concentric cylinder. Testing a similar method of setting the vane yield stress to the Bingham Plastic model was also done; however, because the yield stress from the vane tends to be less than the approximate yield stress from a standard Bingham Plastic curve, setting the yield stress to be the lower value on the Bingham Plastic curve results in a significant difference in viscosity. Though this method of fit would be feasible for thin fluids that generally act Newtonian after shear such as Tk40-10 SME Product, it is not recommended for thicker fluids with a higher yield stress and consistency

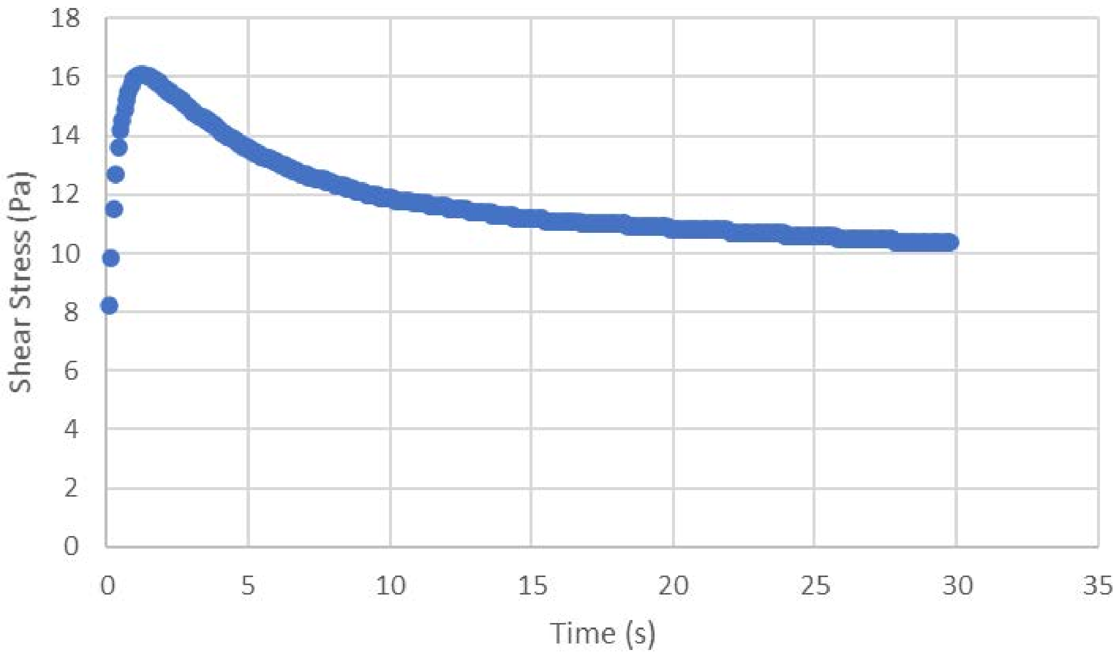

Figure 9. Tk51-3 SME Product, Controlled Shear Rate using FL22 Vane, Trial 2.

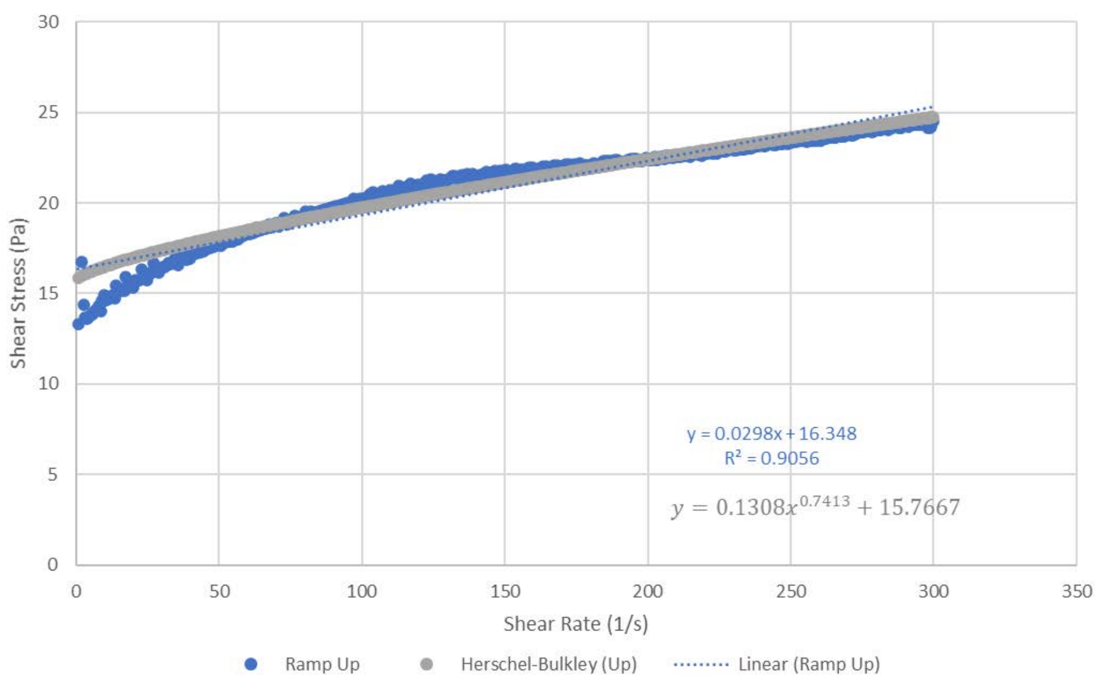

Figure 10. Tk51-3 SME Product, Flow Curve using Z38 Concentric Cylinder

Table 3. Baseline Z38 Concentric Cylinder and Cup using Bingham Plastic Model

	Z38 CC and Cup		
Sample	Viscosity (cP)	Yield Stress (Pa)	R^2
N44	76.2	-	0.9999
Tk40-10	25.1	5.2862	0.9942
Tk51-3	29.8	16.348	0.9056
Tk51-4	39.9	22.388	0.8718

Table 4. Bingham Plastic Curve Fit with Adjusted Yield Stress from FL22 Vane Measurements.

	Vane - Bingham Plastic Model		
Sample	Vane Yield Stress (Pa)	Viscosity (cP)	R^2
N44			
Tk40-10	4.5600	28.3	0.9942
Tk51-3	14.0000	32.7	0.9056
Tk51-4	17.8000	56.2	0.8718

Table 5. Herschel-Bulkley Curve Fit with Adjusted Yield Stress from FL22 Vane Measurements.

	Vane - Herschel-Bulkley Model		
Sample	Consistency Factor	Flow Index	R^2
N44			
Tk40-10	4.98728	5.01207	5.0669
Tk51-3	0.13080	0.74132	0.9501
Tk51-4	0.69391	0.52992	0.9638

4. CONCLUSION AND INTERN REFLECTION

During the ten-week internship experience, several lessons were learned from assisting data-analysis and experimental work for Sludge Batch 10 qualification. These lessons were:

1. *Don't be afraid to ask questions about a topic unfamiliar to you.*
   During this internship, there were many topics that were previously unfamiliar to me from a rheological perspective and from a chemical engineering and chemistry perspective. In order to better understand the processes of DWPF, such as its purpose, how it prepares the slurries, and the chemistry involved in it, requires extensive foreknowledge in chemistry and radiochemistry. With a background in mechanical engineering, the concepts of the chemistry involved in the DWPF process were not entirely familiar. Thankfully, within the Chemical Flowsheet Development group, there is no shortage of people who can explain these concepts in a manner that is easy to understand.

2. *Networking is key.*
   In previous internships, it has been said that networking is critical to making connections that would potentially open doors to future internship opportunities or even employment. However, due to COVID and teleworking, networking became a challenge. This was not the case whilst working with the Chemical Flowsheet Development group. Instead, I had multiple opportunities to connect with administration, mentorship, and colleagues that allowed me to share my knowledge on rheology and learn more from the subject matter experts in my own field. Therefore, I've considered coming back to SRNL next summer doing more targeted research in rheology or computational fluid modeling – two topics that align closely with my Flushing research at FIU.

3. *Safety is paramount.*
   Though this was already known beforehand, the emphasis on safety transitioning from a university environment to a National Laboratory environment is not just impressive, but inspiring. Though intimidating at first – especially for someone who does not normally work in a laboratory – it was easy to follow along with the safety culture that SRNL promotes. Additionally, the SRNL safety culture is a part of a larger initiative of safety site-wide, often offering safety reminders when accessing the Site intranet or walking in designated walkways for pedestrians. I intend on replicating some of the safety culture in my own work at FIU such as the labeling scheme used for labeling samples.

In conclusion, efforts were presented regarding qualification of Sludge Batch 10 from a rheological perspective. Studies were done using chemical slurry simulants that replicate conditions at DWPF CPC to determine what parameters can be adjusted to optimize slurry rheology that is acceptable according to DWPF's design criteria. Additional rheology methods using a vane and constant, controlled rate of shear for accurate yield stress measurements that could be applied to the Herschel-Bulkley fluid model and recommendations on when to use the vane were made.

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