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Real Waste Testing of Sludge Batch 5 Melter Feed Rheology

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EXECUTIVE SUMMARY

Clogging of the melter feed loop at the Defense Waste Processing Facility (DWPF) has reduced the throughput of Sludge Batch 5 (SB5) processing. After completing a data review, DWPF attributed the clogging to the rheological properties of the Slurry Mix Evaporator (SME) product. The yield stress of the SB5 melter feed material was expected to be high, based on the relatively high pH of the SME product and the rheological results of a previous Chemical Process Cell (CPC) demonstration performed at the Savannah River National Laboratory (SRNL).

DWPF submitted real waste SB5 SME and Melter Feed Tank (MFT) samples to SRNL for physical and chemical characterization, including rheology and solids distribution determinations. Analysis of the SME and MFT samples indicated that the yield stress of the melter feed material was low – sufficiently low to limit the solid phase suspension capacity of the fluid, a characteristic which is necessary to prevent solid-liquid phase separation and subsequent solid phase deposition.

Concentrating the solids content of the melter feed material via settling/decanting and boiling/evaporation was shown to provide a practical means of raising the yield stress of the material to meet the DWPF design basis criteria. Based on the data, increasing the total solids concentration of the SME product by 3-6 wt% on an absolute basis (from ~42 wt% to between 45 and 48 wt%) is expected to reduce or eliminate the clogging problem under the assumption that low yield stress is the primary cause of the problem.

Several differences between the DWPF SB5 melter feed material and the SB5 SME product generated in the CPC qualification demonstration have been identified. Included among these are boiling time, pH, total solids, and waste loading. Some or all of these differences have an impact on yield stress. Differences in the total solids content and the waste loading are likely the primary drivers of the differences in yield stress.

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LIST OF ABBREVIATIONS

SRNL	Savannah River National Laboratory
DWPF	Defense Waste Processing Facility
SB5	Sludge Batch 5
MFT	Melter Feed Tank
SME	Slurry Mix Evaporator
CPC	Chemical Process Cell
TTQAP	Task Technical and Quality Assurance Plan
HDPE	High Density Polyethylene
ICP-AES	Inductively Coupled Plasma-Atomic Emission Spectroscopy
SRAT	Sludge Receipt and Adjustment Tank

1.0 INTRODUCTION

The Defense Waste Processing Facility (DWPF) has been experiencing problems with loss of feed to the melter feed loop during Sludge Batch 5 (SB5) processing. The feed behavior has been such that the transfer lines of the Melter Feed Tank (MFT) have become clogged, halting melter feed and requiring remedial measures (including addition of prime water) to restart the melter feed pump. This has resulted in a decrease in melter throughput.

The pH of the SB5 Slurry Mix Evaporator (SME) product is significantly higher than that of previous batches (pH ~9 for SB5 versus pH ~7 for SB4, for example). High pH can affect the rheology as it partitions greater proportions of metals to the solid phase, leading to an increased insoluble solids concentration and an increased yield stress. After considering the elevated pH of the SB5 SME product and reviewing the available process data for potential trends, DWPF Engineering developed a path forward for resolving the melter feed clog issue.[1] The path forward focused on chemical changes targeting reduced pH conditions as a potential means of lowering the yield stress of the SB5 SME product and making it more conducive to effective pumping and transfer. As identified in the path forward, the two primary methods of lowering pH included increasing the acid additions and decreasing the redox conditions.

SRNL's original plan[2,3] was to perform real waste tests in which the rheological properties of SB5 SME product and MFT slurries were monitored as a function of: a) nitric acid additions; b) formic acid additions; c) total solids contents; d) extent of pH-adjustment; e) amount of time following pH-adjustment; and f) duration of caustic boiling. However, upon performing the initial rheology measurements, the yield stresses of the real waste SME and MFT samples were found to be unusually low, despite the solids concentrations being in the normal range. These results changed the assumptions regarding the cause of the melter feed loop clogs and provided the impetus for revising the scope of the testing. The new scope focused on the extent that the melter feed slurries could be concentrated without producing unwieldy yield stresses and/or plastic viscosities.[4]

This study was performed at the request of the Waste Solidification Engineering group of Savannah River Remediation.[2] Note that a portion of the requested work was performed on simulated SB5 SME-MFT slurries, as opposed to real waste slurries. Results of the simulated slurry tests have been reported in a separate document.[5]

2.0 OBJECTIVES

The overall goal of the study was to provide data supporting understanding and resolution of the SB5 melter feed loop clog problems. The specific objectives were:

1) Characterize the SB5 melter feed material with respect to: a) solids distribution; b) pH; c) rheology (yield stress and plastic viscosity); d) appearance; and e) frit dissolution;

2) Determine the extent that the slurry rheology changes as solids are concentrated via settling/decanting and boiling/evaporation;

3) Determine the hydrogen generation rate during slurry boiling/evaporation; and

4) Identify similarities and differences between the DWPF SME product and the SME product generated in the Shielded Cells during the 2009 CPC Np qualification demonstration.[6]

3.0 QUALITY ASSURANCE

This study was conducted in accordance with the quality assurance protocols identified in the Task Technical and Quality Assurance Plan (TTQAP).[3] All of the raw data and ancillary information related to this study have been recorded in laboratory notebook SRNL-NB-2010-00010.[7]

4.0 METHODOLOGY

4.1 Sample Description

Five 200-mL SME slurry samples and one 200-mL MFT slurry sample (from Batch 503) were provided by DWPF for use in this study. The samples were received at the SRNL Shielded Cells Facility on October 28, 2009. The five SME samples were composited in a volume-calibrated 2-L wide mouth high density polyethylene (HDPE) container. The total mass of the SME composite sample was 1366 g – the total volume was 1032 mL. The MFT sample was placed into a 250-mL wide mouth HDPE container. The total mass of the MFT sample was 281 g. Prior to transferring the slurries out of the sampling vessels, each sampling vessel was agitated/inverted for a minimum of one minute to facilitate suspension of settled insoluble particles. Minimal residual material remained in the sampling vessels after transferring the samples to the HDPE labware. Before removing aliquots of the samples for laboratory use, the material in the HDPE containers was similarly agitated/inverted.

4.2 Sample Characterization

Measurements of the solids distribution, pH, and rheology of the SME and MFT slurries were performed in the SRNL Shielded Cells. In contrast, metals analysis of the SME supernatant was performed outside of the cells by Analytical Development, after separating the supernatant from the solid phase in the cells. The appearances of the as-received SME and MFT slurries and the undiluted boiled/evaporated SME slurry were recorded to provide a qualitative basis of comparison between unconcentrated and concentrated SME slurries. The density of the as-received SME slurry was quantified, despite its absence from the list of planned measurements in the TTQAP. The relative ease of determining density using the available information made this determination worthwhile. Summaries of the various preparation and measurement methods are given below.

Solids distribution: Total solids and dissolved solids contents were determined by performing wet and dry weight measurements of slurry and supernatant aliquots, respectively, and quantifying the ratios of dry weight to wet weight. Dry weights were measured after driving water from the samples at a nominal temperature of 100-110 ºC. For each type of sample, the supernatant aliquots were generated by passing the slurry through a 0.45 μm filtration membrane. Four slurry aliquots and four supernatant aliquots of each sample were utilized for the measurements, along with a sodium chloride standard solution. The mass of each aliquot was ~3.0 g, and the dryings were performed in alumina crucibles. Insoluble solids content and soluble solids content were then calculated based on the total solids and dissolved solids measurements. Calcined solids contents were subsequently determined by heating the dried slurry aliquots (the ones used for determining total solids contents) to a nominal temperature of 1100 ºC, producing metal oxides in a glass matrix, and then ratioing the cooled glass weights to the original wet weights. The full procedure for quantifying the solids distribution is given in the L29 Manual.[8]

pH: pH measurements of the SME and MFT samples were conducted using a conventional pH probe system calibrated with buffer solutions standardized to pH 4.0, 7.0, and 10. Just prior to the measurement, the sample was agitated/inverted to facilitate suspension of insoluble particles. The probe was then immersed into the slurry and used to gently stir the slurry for a couple of seconds. The probe was then held stationary and the pH measurement was allowed to stabilize. Between measurements, the pH probe was rinsed with de-ionized water, and then shaken gently to remove free water droplets.

Rheology: Yield stresses and plastic viscosities were determined by generating "flow curves"of shear stress as a function of shear rate. The flow curve data were acquired using a Haake RV-30 viscometer equipped with the MVII rotor, at a temperature of 25 ºC. The shear rate was increased from 0-300/s over a five minute period, held at 300/s for one minute, and then reduced from 300-0/s over a five minute period. The yield stress was determined by extrapolating the linear portion of the "UP" flow curve back to the Y-axis. The plastic viscosity was determined by calculating the slope of the linear portion

of the "UP" curve. Note that the data analysis was performed on the "UP" flow curves (as opposed to the subsequent "DOWN" flow curves) so that the impacts of particle settling would be minimized. Two rheology measurements were performed on a single 60 mL aliquot of each slurry. Before performing each rheology measurement, the slurry aliquot was mixed to facilitate suspension of the insoluble particles (this was done between replicate measurements, as well as with initial measurements). The full procedure for performing the rheology measurements is given in the L29 Manual.[9]

Metals Analysis: Two supernatant aliquots from a settled, as-received SME composite sub-sample were collected using a slurry pipet. These supernatant aliquots were submitted undiluted to Analytical Development, for analysis of frit-containing metals by inductively coupled plasma-atomic emission (ICP-AES) spectroscopy. One aliquot of de-ionized water and one aliquot of multi-element standard solution were also submitted, for quality assurance purposes. The mass of each of the submitted aliquots was approximately five grams.

Density: The density of the SME composite sample was quantified by dividing the entire mass of sample received (1366 g) by the volume indicated on the pre-calibrated HDPE sample container (1032 mL).

4.3 Settling/Decanting of SME Material

The first method used to concentrate the SME solids was settling/decanting. A 259 g aliquot of the SME composite material was allowed to settle for three days, then a 39 g aliquot of supernatant was decanted using a slurry pipet. Rheological measurements of the resulting slurry were performed after agitating the material to suspend insoluble particles. Following the measurements, the aliquots of the concentrated slurry were combined in a sample container and allowed to settle for 7 days. An additional 22 g aliquot of supernatant was decanted to further concentrate the slurry, and then rheological measurements of the resulting slurry were performed. The solids distributions of the concentrated slurries were calculated based on the fractions of supernatant removed and the original solids distribution. The rheology measurements were performed using the method outlined in Section 4.2.

4.4 Boiling/Evaporation of SME Material

The second method used to concentrate the SME solids was evaporation/boiling. (This method mimicked the DWPF processing approach). A special heating/mixing apparatus[10] was developed to allow: a) temperature-controlled boiling of the SME material; b) sufficient mixing to ensure suspension of insoluble particles during boiling (this was necessary to provide uniform heating and prevent settling/deposition of insoluble solids); c) isolation of off-gases for accurate hydrogen monitoring; and d) identification of the volume of condensed evaporated water. A schematic of the heating/mixing apparatus is given in Figure 4-1.

A 200 mL aliquot of the original SME composite material was transferred to the vessel of the heating/mixing apparatus and then heating/mixing was initiated. The condenser temperature was 20 ºC, the chiller temperature was 20 ºC below the ambient temperature, the heater temperature was slightly higher than 100 ºC, and the air purge rate was 25 standard cubic centimeters per minute. The total period of heating/mixing was 3.5 hours. This period produced an evaporation of 67 mL of water, which targeted a final total solids concentration that was approximately ten percent higher than that of the most settled/decanted slurry described in Section 4.3.

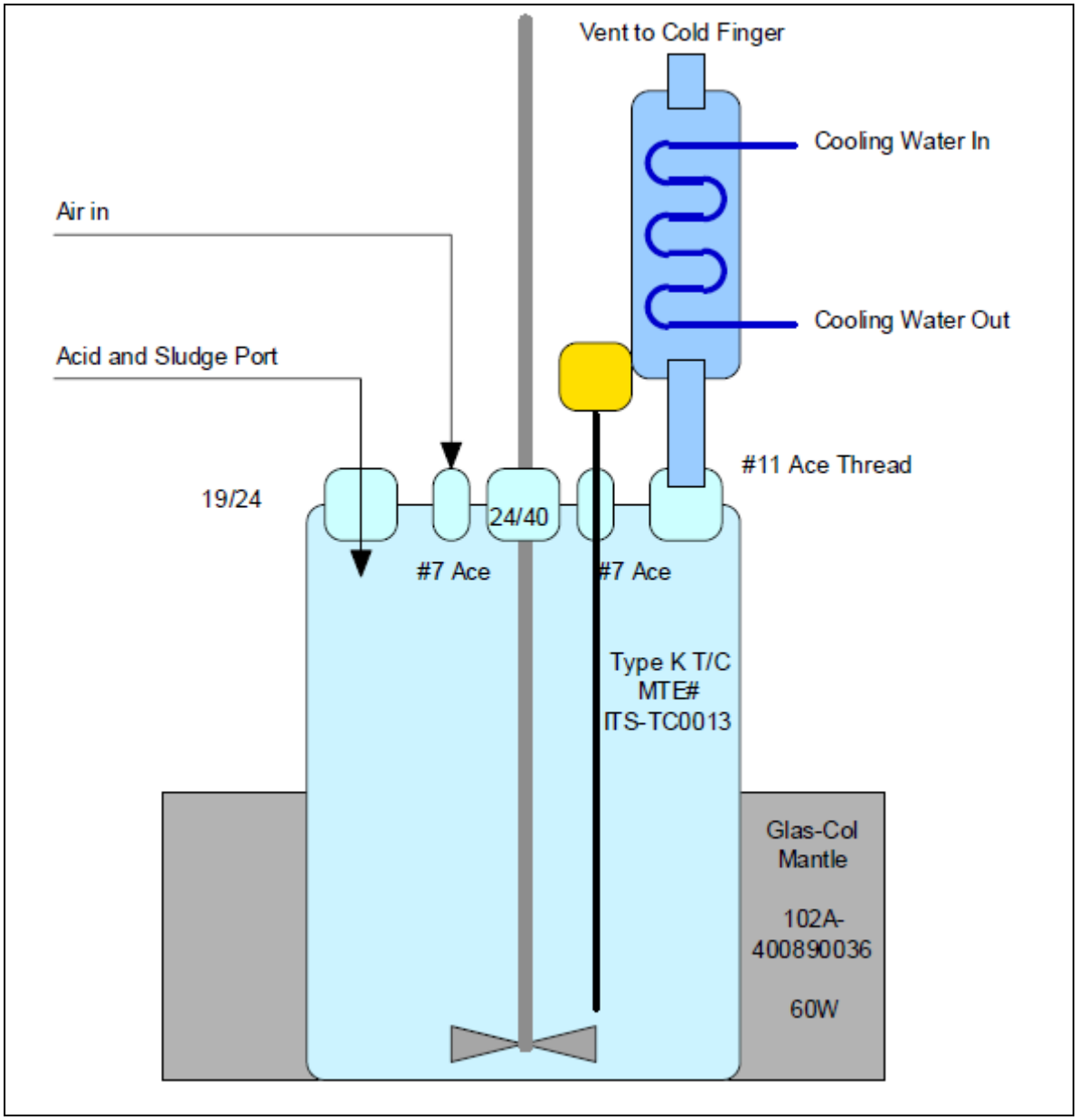

Figure 4-1. Schematic of SME Heating/Mixing Apparatus

During the boiling period, the hydrogen generation rate was measured using an in-line Agilent M200 gas chromatography unit. Helium was introduced as an inert tracer at a concentration of 0.5% of the total air purge. Use of the helium tracer supported quantification of the peak hydrogen generation rates and converting the results to a DWPF basis. A 6000 gallon batch volume was assumed for DWPF-scaling purposes.

The rheology and total solids content of the boiled/evaporated SME material (after removing 67 mL of water) were measured using the methods outlined in Section 4.2. A 137 g aliquot of the boiled/evaporated SME material was diluted with approximately 15 g de-ionized water, and then measured for rheology. Subsequently, a 132 g aliquot of the diluted boiled/evaporated SME material was further diluted with approximately 13 g of deionized water. The resulting slurry was measured for rheology. Total solids contents of the two diluted boiled/evaporated SME slurries were calculated based on the total solids content of the undiluted boiled/evaporated SME slurry and the dilution specifications.

4.5 Analysis Summary

Analyses performed on the various slurries are summarized in Table 4-1. Note that the letter "X" identifies analyses that were executed.

Table 4-1. Summary of SME and MFT Slurry Analyses

Analysis	As-Rec'd SME Slurry	As-Rec'd MFT Slurry	Settled/Decanted SME Slurries	Boiled/Evaporated SME Slurries
Calcined solids	X	X		
Total solids	X	X	X	X
Insoluble solids	X	X	X	
Soluble solids	X	X	X	
Dissolved solids	X	X	X	
pH	X	X		
Yield stress	X	X	X	X
Plastic viscosity	X	X	X	X
Density	X	X		
Dissolved metals	X			

4.6 Comparison of DWPF SME/MFT and CPC Qualification Demo SME Products

Characteristics associated with the DWPF SB5 SME/MFT products were compared to those of the SB5 SME product generated in the 2009 Shielded Cells CPC Np qualification demonstration. Specifically, a comparison was made between key physical and chemical characteristics including solids content, density, pH, yield stress, and plastic viscosity – plus operational characteristics[11] including acid addition quantities and ratios, redox targets, boiling time targets, and waste loading targets. The purpose of the comparison was twofold – to identify similarities and differences associated with the field- and lab-generated products – and to identify potential drivers of the differences.

5.0 RESULTS AND DISCUSSION

5.1 Characterization Results

Characterization results for the various SB5 SME and MFT slurries are given in Table 5-1. Concentrations of frit-containing metals in the SB5 SME supernatant are given in Table 5-2. Plots of the yield stresses and the plastic viscosities as functions of the total solids contents are given in Figures 5-1 and 5-2, respectively, and the flow curves from the rheological measurements are given in Appendix B.

Based on the results of the as-received SME and MFT slurries, the melter feed material contained 36-38 wt% calcined solids, 42-43 wt% total solids, 34-36 wt% insoluble solids, and 7-8 wt% soluble solids. Despite the relatively high pH of the melter feed material (pH=9.6), the yield stress was very low (1-2 Pa). In contrast, the plastic viscosity was moderate (~10 cP). The thin appearance of the slurry material (like chicken broth) was reflective of the low yield stress. The density of the material was 1.32 g/mL.

Available data for separate SB5 melter feed samples analyzed at DWPF[12,13] were similar to those shown in Table 5-1. Specifically, the DWPF data indicated that the melter feed slurries contained 35 wt% calcined solids and 41 wt% total solids, with a pH of 9.3 and a density of 1.32 g/mL (note that DWPF did not determine the insoluble solids content, soluble solids content, yield stress, or plastic viscosity). Given the inherent uncertainties associated with sampling and analysis, the DWPF results were considered to be experimentally equivalent to the SRNL results. This similarity provides support that the samples submitted to SRNL were sufficiently representative.

As illustrated in Figure 5-1, the yield stress of the feed material increased by a factor of approximately three as the total solids were concentrated from ~42 to 50 wt%. At the highest solids concentration (56.5 wt% total solids), the yield stress was an order of magnitude higher (13 Pa) than it was for the as-received SME slurry and a factor of about three higher than it was at ~50 wt% total solids. Boiling/evaporation seemed to increase the yield stress to a greater extent than settling/decanting.

As illustrated in Figure 5-2, the plastic viscosity also increased with the solids concentration. At a total solids content of ~50 wt%, the plastic viscosity was 3-4 times higher than it was at ~42 wt%. As in the case of the yield stresses, the increase in plastic viscosities seemed greater for slurries concentrated by boiling/evaporation versus settling/decanting.

Rheological differences between the two types of concentrated slurries are attributed to differences in the relative amounts of soluble and insoluble solids. At a given total solids content, the soluble-to-insoluble solids ratio for a boiled/evaporated slurry is assumed to be higher than for a settled/decanted slurry, due to the removal of soluble solids during decanting. Under this assumption, the results suggest that higher soluble-to-insoluble ratios lead to higher yield stresses and plastic viscosities. On a relative basis, the impact of the solids ratio appears small compared to the impact of the total solids concentration.

Table 5-1. Characterization of SB5 SME and MFT Slurries

Analysis or Observation	As-Rec'd SME Slurry	As-Rec'd MFT Slurry	Settled/Decanted SME Slurry		Boiled/Evaporated SME Slurry		
			Least Concentrated	Most Concentrated	Most Diluted	Least Diluted	Not Diluted
Calcined solids, wt% of slurry	36.1	38.2					
Total solids, wt% of slurry	41.7	43.2	46.9	50.9	43.2	49.0	56.5
Insoluble solids, wt% of slurry	33.7	36.0	39.7	44.2			
Soluble solids, wt% of slurry	8.0	7.2	7.2	6.7			
Dissolved solids, wt% of supernatant	12.0	11.3	12.0	12.0			
pH	9.6	9.6					
Yield stress, Pa	1.0	1.7	2.2	2.8	2.6	4.1	13
Plastic viscosity, cP	10	11	14	33	19	44	50
Appearance of slurry consistency	Chicken broth	Chicken broth					Thin catsup
Density, g/mL	1.32						

Table 5-2. Frit-Containing Metals in SB5 SME Supernatant

Constituent	Concentration, mg/L
Boron	6.2E+2
Lithium	1.5E+3
Silicon	5.1E+2
Sodium	3.6E+4

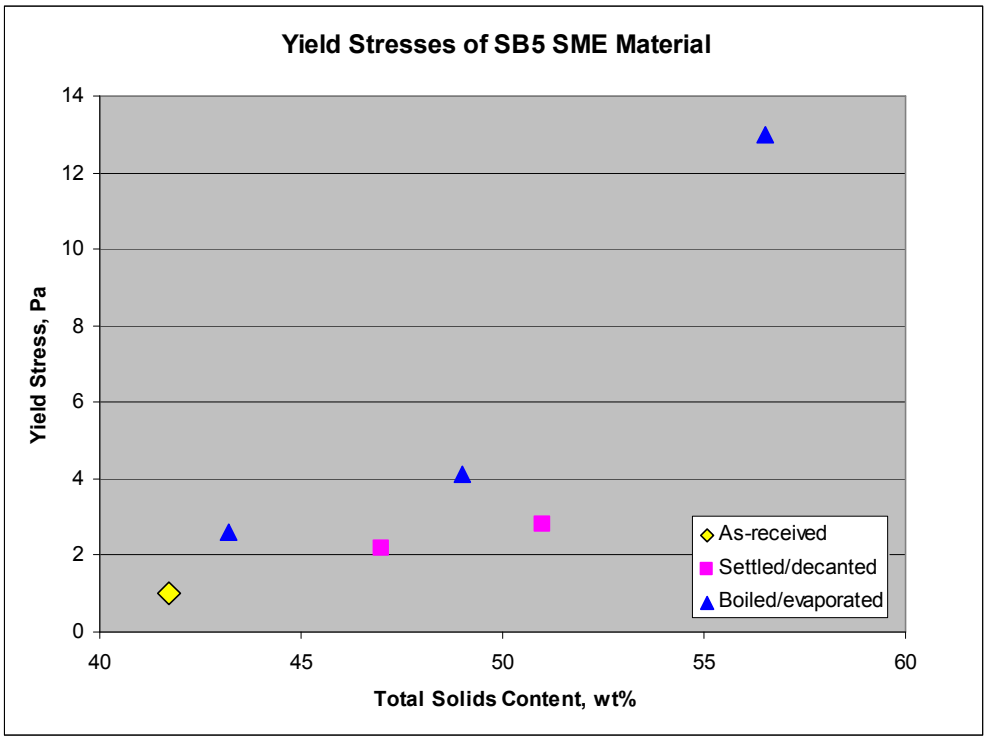

Figure 5-1. Yield Stresses as a Function of Total Solids Content

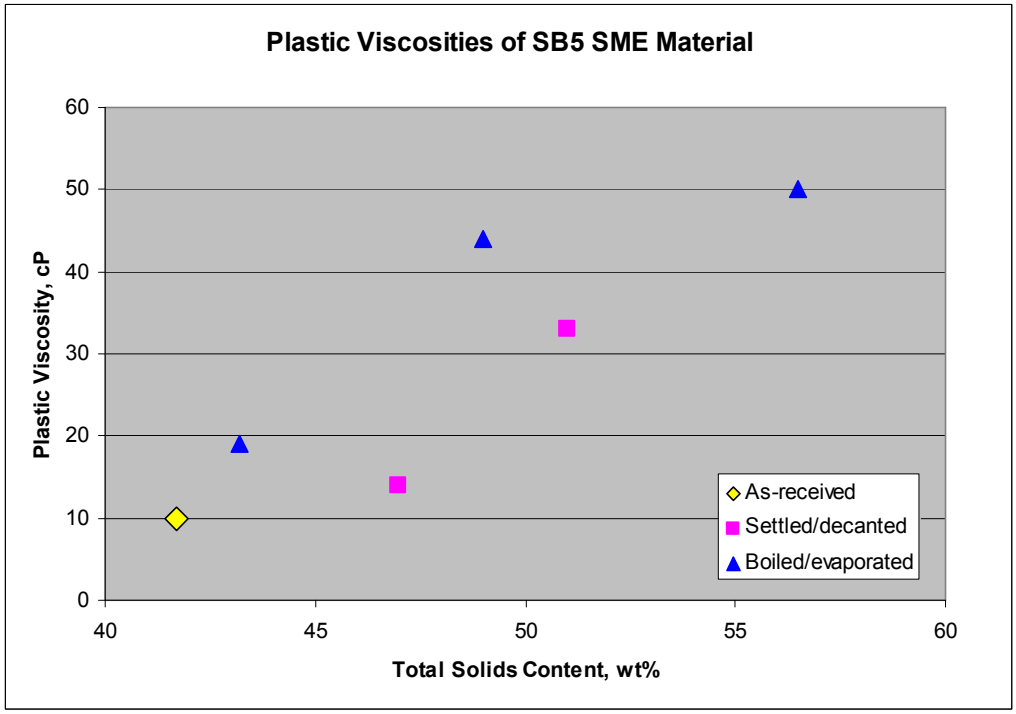

Figure 5-2. Plastic Viscosities as a Function of Total Solids Content

The low yield stress of the SME material (Batch 503) is thought to facilitate clogging of the melter feed loop, as the solid phase suspension capacity of a low yield stress fluid is limited and promotes solid-liquid phase separation.[14] Such separation can lead to deposition of insoluble particles. Observations from working with the SME material in the Shielded Cells confirmed the rapid settling and deposition of insoluble particles. Given such behavior, increasing the yield stress of the slurry by concentrating the solids is expected to impede solid-liquid phase separation and therefore reduce clogging of the melter feed loop.

The DWPF design basis identifies a yield stress ranging from 2.5-15 Pa and a plastic viscosity ranging from 10-40 cP.[15] Based on the data shown in Figures 5-1 and 5-2, boiling/evaporation of the slurry to attain a total solids content between about 45 and 48 wt% should be sufficient to produce rheological properties meeting the design basis specification. In doing so, the goal would be to concentrate the total solids enough to raise the yield stress above 2.5 Pa without raising the plastic viscosity above 40 cP. Because of the inherent experimental uncertainties, the solids target range of 45-48 wt% should be considered somewhat tentative.

Note that the aforementioned design basis identifies the requirements for effective mixing and sampling of the tank contents – not for preventing clogging of the melter feed loop. Although there may be similarities between the rheological requirements necessary for effective tank mixing/sampling and for maintaining suspension of solid particles during feeding through the melter loop, it is clear that identification of the requirements in the melter feed loop would ultimately be necessary to assure understanding of the melter feed clogging issue. However, in the absence of specific melter feed requirements, it is assumed that targeting the mixing/sampling requirements will be beneficial.

Concentrations of frit-containing metals in the SME product supernatant provide a measure of the portions of frit components that were soluble. As shown in Table 5-2, the measured concentrations of boron, lithium, silicon, and sodium were approximately 600, 1500, 500, and 36000 mg/L, respectively. Based on the nominal frit content of the SME slurry, the dissolved portions of B_2O_3, Li_2O, and SiO_2 were approximately 6, 10, and 0.4%, respectively. Note that the dissolved portion of the Na_2O was not determined, since data quantifying the soluble sodium contributed by the sludge was not available.

The dissolved portions of B_2O_3, Li_2O, and SiO_2 in the real waste tests were 10-60 times those seen in the recent SB5 SME simulant tests (pH 10).[5] The differences are probably due to the absence of a true SME cycle in the simulant tests, providing less potential for frit-leaching. In the simulant tests, the SME product was generated by adding frit to the simulated Sludge Receipt and Adjustment Tank (SRAT) product. Investigation of the effect of frit-leaching during SME simulant preparation should be considered in future laboratory tests.

The DWPF-scaled hydrogen generation rate during boiling of the SME material is plotted as function of time in Figure 5-3. Over the period extending from ~60 minutes to ~230

minutes, the hydrogen generation rate increased steadily from ~0.002 lbs/hr to a maximum of ~0.012 lbs/hr. Note that the boiling was terminated at ~210 minutes, but the hydrogen generation rate continued to rise for an additional 20 minutes. Regardless, the maximum generation rate was more than an order of magnitude below the DWPF limit of 0.223 lbs/hr.

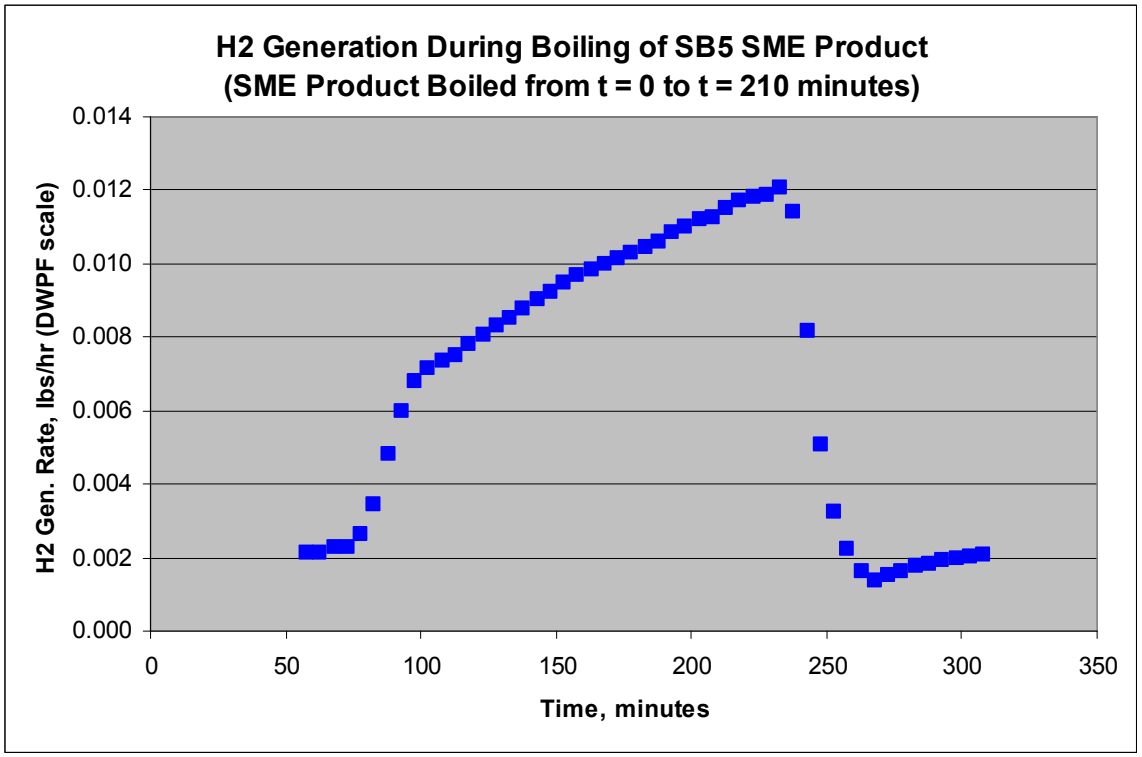

Figure 5-3. DWPF-Scaled Hydrogen Generation Rates

5.2 Comparison of DWPF SME/MFT and CPC Demonstration SME Products

Characteristics of the DWPF SME/MFT material (Batch 503) and the SME product generated in the 2009 Shielded Cells CPC Np qualification demonstration are compared in Table 5-3. With respect to rheology, the yield stress of the DWPF material (1-2 Pa) is significantly lower than that of the 2009 CPC demonstration (38 Pa). However, the plastic viscosities of the two materials are almost identical (both ~10 cP).

The specific cause of the yield stress difference is not clear, but is hypothesized to be due to a combination of chemical and physical differences. Only a couple of similarities exist between the two products, and those include quantity of acid added (as well as excess acid), ratio of formic acid to total acid, and redox target. Otherwise, the characteristics of the two products are different. The solids content of the DWPF material is clearly less than that of the CPC Np qualification demonstration material. Consistent with the solids difference is the lower density of the DWPF material. Other differences include the higher pH, lower targeted boiling time, and lower targeted waste loading associated with the DWPF material.

Table 5-3. Comparison of SME Characteristics (DWPF vs. 2009 CPC Demo)

Characteristic	DWPF SME/MFT Material (Batch 503)	2009 Shielded Cells CPC Demo SME Product [6]
Total solids, wt% of slurry	42-43	48-49
Insoluble solids, wt% of slurry	34-36	40
Soluble solids, wt% of slurry	7-8	~9
Calcined solids, wt% of slurry	36-38	43
Slurry density, g/mL	1.32	1.44
pH	9.6	8.4
Yield stress, Pa	1-2	38
Plastic viscosity, cP	10-11	10
Acid addition, moles/L	1.5*	1.4
Acid stoichiometry, %	135	144
Excess acid, moles/L	0.4	0.4
Ratio of formic acid to total acid	0.88	0.88
Redox target	0.12	0.12
SRAT boiling time target, hours	27	44
Waste loading target, wt% of glass	30	34

*Assumes 85 gallons 50% nitric acid and 280 gallons 90% formic acid added to a 7173 gallon batch containing 1500 gallons of heel (already acidified) and 600 gallons of flush water.[11]

Insight into the effects of solids content and waste loading on yield stress can be found in the results of other SRNL studies. The effect of total solids was illustrated during the 2009 CPC Np qualification demonstration, when the yield stress was measured at four different solids concentrations obtained via settling/decanting (40.0, 42.9, 47.5, and 48.5 wt%).[6] At the lower two concentrations (40.0 and 42.9 wt%), the yield stresses were relatively similar (9 and 10 Pa). However, at the next highest concentration (47.5 wt%), the yield stress was twice as high (~20 Pa), and at the highest concentration (48.5 wt%), the yield stress was four times as high (~40 Pa). Clearly the increases in yield stress were much more significant as the solids concentration approached 48 wt%. Based on the data, the expectation is that the yield stress of the material would continue to increase significantly if solids concentrations were raised above 48 wt%.

The effect of waste loading on yield stress was illustrated during 2004 SB2/3 SME simulant testing, when rheology measurements were performed on slurries with similar solids contents but different loading conditions (31, 35, and 40% waste loading).[16] At the two lower waste loading conditions (31 and 35%), the yield stresses were approximately 3 and 5 Pa, respectively. However, at the highest waste loading condition (40%), the yield stress was approximately 20 Pa, a factor of four to seven times higher. As in the case of the increasing solids contents, the increased waste loadings had a much more significant impact on yield stress once a "threshold" waste loading value was exceeded.

Due to the differences between the DWPF SB5 melter feed material and the SME materials utilized in the aforementioned studies, a direct comparison can not be made between the yield stresses of the various products. Nonetheless, the results of the studies provide a basis for expecting that slurries with solids contents and waste loadings below a certain threshold will have relatively low yield stresses, while slurries with solids contents and waste loadings above the threshold will have relatively high yield stresses.

6.0 CONCLUSIONS

1) The low yield stress of the DWPF SB5 melter feed material seems to be responsible for the solid-liquid phase separation problems which lead to clogging of the melter feed loop.

2) Concentrating the solids content of the SB5 SME material by 3-6% (on an absolute basis) is expected to produce a rheology which will meet the DWPF design basis and reduce clogging of the melter feed loop.

3) Differences between the rheology of the DWPF melter feed material and the SME product generated in the Shielded Cells CPC Np qualification demonstration are due to a combination of chemical and physical differences. The lower solids content and the lower waste loading associated with the DWPF material appear to be two of the drivers responsible for the reduced yield stress. The effects of other differences such as boiling time are currently unclear.

7.0 RECOMMENDATIONS

1) Further concentration of the SB5 SME material in the DWPF, via boiling/evaporation, is recommended. Increasing the total solids content of the slurry to between 45 and 48 wt% is expected to produce a rheology which meets the DWPF design basis and reduces clogging of the melter feed loop.

2) Further testing focusing on the effects of the other operational parameters (such as boiling time) is recommended if additional concentration of the melter feed material does not adequately resolve the clogging problem.

3) Determination of the ranges of SME product yield stresses and plastic viscosities necessary to minimize or prevent clogging of the melter feed loop is recommended.

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4 Bricker, J. M. *Email message to S. H. Reboul with subject heading of "Re: Change in scope for SME rheology testing*," Savannah River Site, Aiken, SC, 12:33 p.m. on November 21, 2009 (see Appendix A).

5 Fernandez, A. I. *Rheological and Elemental Analyses of Simulant SB5 Slurry Mix Evaporator-Melter Feed Tank Slurries*, Savannah River National Laboratory, Aiken, SC, SRNL-STI-2009-00751, February 2010.

6 Pareizs, J. M., B. R. Pickenheim, C. J. Bannochie, and M. E. Stone. *Demonstration of the DWPF Flowsheet in the SRNL Shielded Cells with Tank 40 and H Canyon Neptunium*, Savannah River National Laboratory, Aiken, SC, SRNS-STI-2009-00233, April 2009.

7 Reboul, S. H. *Laboratory Notebook: Rheology of SB5 SME/MFT Material*, Savannah River National Laboratory, Aiken, SC, SRNL-NB-2010-00010, Date Opened: February 1, 2010.

8 Pareizs, J. M. *Weight Percent Solids Distribution Using a Furnace or Oven*, Savannah River National Laboratory, Aiken, SC, SRNL L29 Manual, ITS-0078, March 30, 2007.

9 Hansen, E. K. *Setup and Operation of the Shielded Cells Viscometer*, Savannah River National Laboratory, Aiken, SC, SRNL L29 Manual, ITS-0086, August 31, 2007.

10 Stone, M. E. *R&D Directions for SRAT Mock-up Testing – Apparatus for SME-MFT Mixing Test*, Savannah River National Laboratory, Aiken, SC, SRNL-L3100-2009-00267, October 20, 2009.

11 Clark, M. C. *SRAT Batch 503 Acid Requirements Spreadsheet*, Savannah River Site, Aiken, SC, DWPF Process Engineering, December 31, 2008.

12 *Analytical Data Report: DWPF 221-S Laboratory, SME-1, Batch 503, Sample ID 200016777*, Savannah River Site, Aiken, SC, October 26, 2009.

13 *Analytical Data Report: DWPF 221-S Laboratory, MFT-3, Batch 503, Sample ID 200016820*, Savannah River Site, Aiken, SC, October 24, 2009.

14 Heywood, N. *Slurry Mixing, Rheology, and Handling*, Slurry Retrieval, Pipeline Transport, Plugging, and Mixing Workshop, Marriot Village, Orlando, FL, January 14-18, 2008.

15 *Basic Data Report: Defense Waste Processing Facility Sludge Plant, Savannah River Plant 200-S Area,* Savannah River Site, Aiken, SC, DPSP-80-1033, Revision 10, July 1992.

16 Koopman, D. C. and E. K. Hansen. *A Summary of Rheology Data for SB3 and SB2/3 Blend Simulant Savannah River Site Wastes*, Savannah River National Laboratory, Aiken, SC, WSRC-TR-2004-00116, March 2004.

APPENDIX A: CHANGE IN WORK SCOPE

Jonathan Bricker/SRR/Srs
11/21/2009 12:33 PM

To Scott Reboul/SRNL/Srs@Srs
cc Brandon Hodges/SRR/Srs@Srs, Connie Herman/SRNL/Srs@Srs, Erich Hansen/SRNL/Srs@Srs, Helen Pittman/SRR/Srs@Srs, John
bcc
Subject Re: Change in scope for SME rheology testing

History: This message has been replied to and forwarded.

Scott,

You have accurately captured everything discussed in Thurdays meeting. I concur with the change in work scope. I am in all next week, so feel free to contact me if you need anything.

Thanks,
Jonathan Bricker
DWPF-Engineering
8.7162

Scott Reboul/SRNL/Srs

Scott Reboul/SRNL/Srs
11/19/2009 04:13 PM

To Jonathan Bricker/SRR/Srs@Srs
cc Terri Fellinger/SRR/Srs@srs, Ryan Mcnew/SRR/Srs@Srs, Helen Pittman/SRR/Srs@Srs, Brandon Hodges/SRR/Srs@Srs, Sean Cassidy/SRR/Srs@Srs, Richard Odriscoll/SRR/Srs@Srs, Erich Hansen/SRNL/Srs@Srs, Michael Stone/SRNL/Srs@Srs, Connie Herman/SRNL/Srs@Srs, John Occhipinti/SRR/Srs@Srs
Subject Change in scope for SME rheology testing

Jon:

Based on today's SME Rheology Testing meeting, I believe we agreed that the following test activities are the next tasks SRNL should pursue:

1) Perform rheology measurements on settled SME composite material that has had the maximum practical amount of supernatant removed via decanting (which will result in a total solids content higher than 47 wt%);

2) Perform rheology measurements on SME composite material that has been concentrated via boiling/evaporation to solids contents comparable to those of the decanted SME material; and

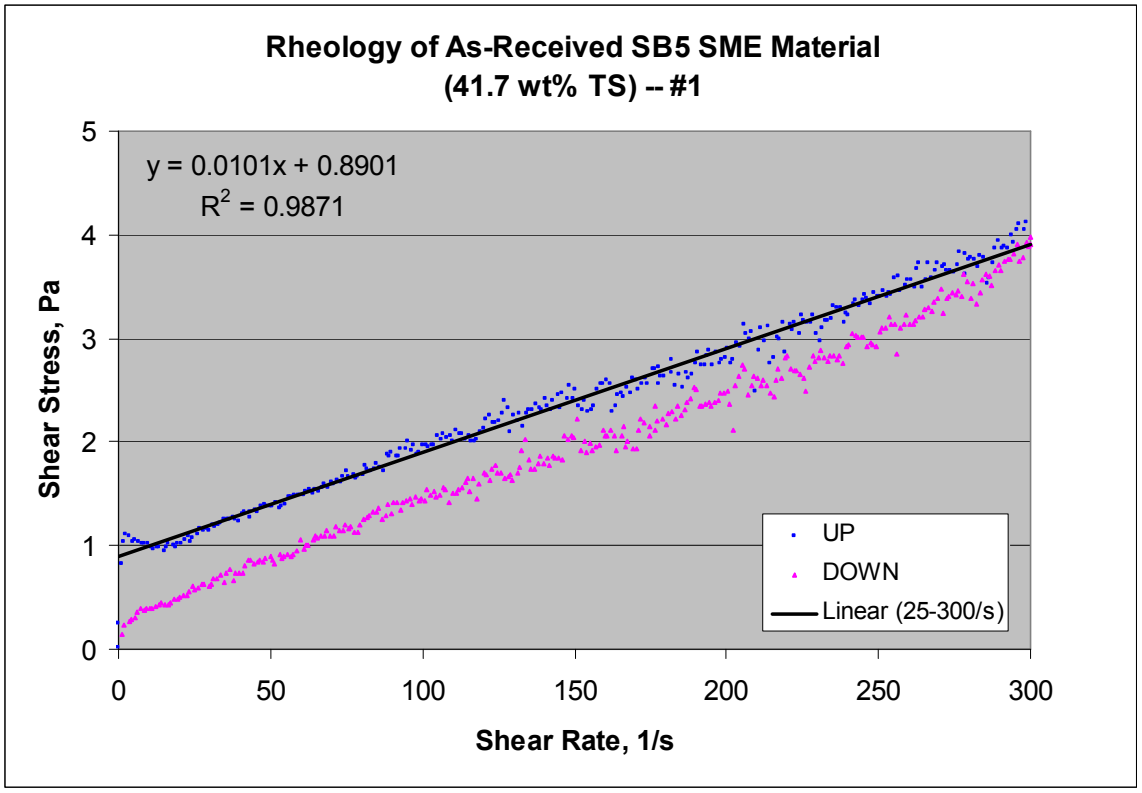
3) Perform ICP-AES analysis of supernatant from the as received SME composite sample to determine the soluble concentrations of frit-related constituents including Si, Li, and B.

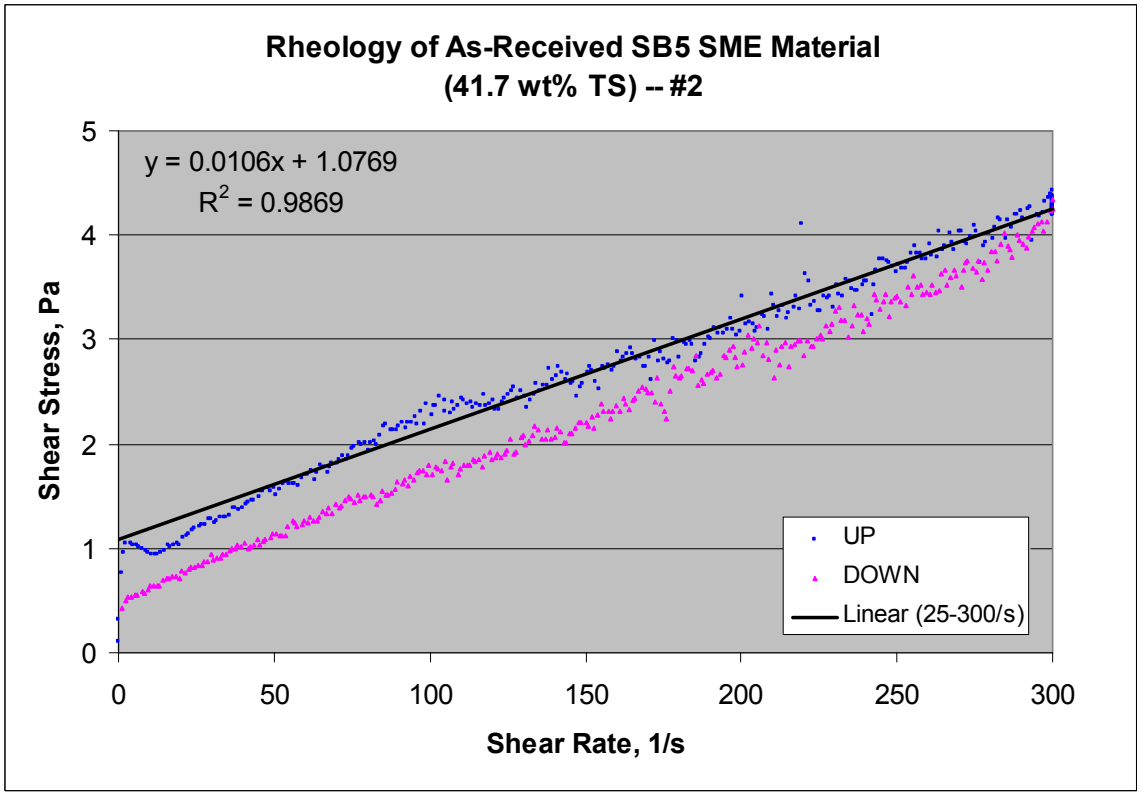
Since Tasks #1 and 2 deviate somewhat from the scope identified in the TTQAP (SRNL-RP-2009-01199), I want to make sure you agree with the changes before I begin the work. Please confirm that the activities identified above are consistent with your needs.

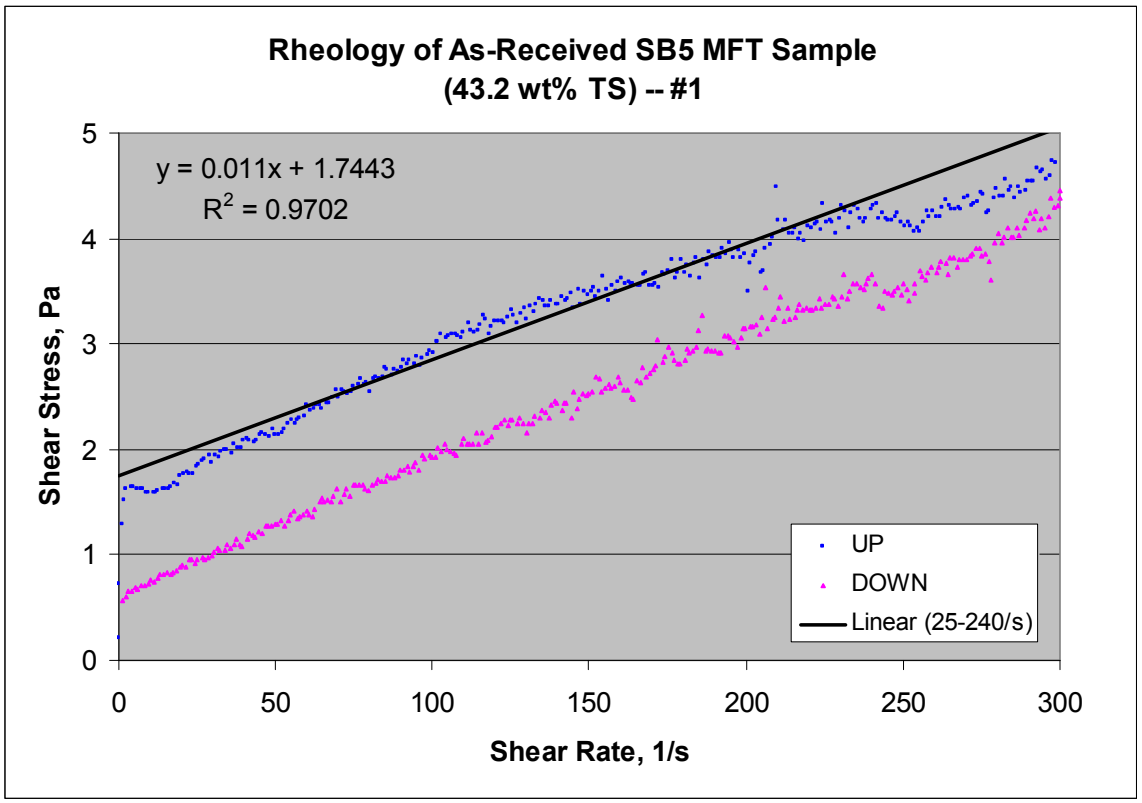
Thanks,

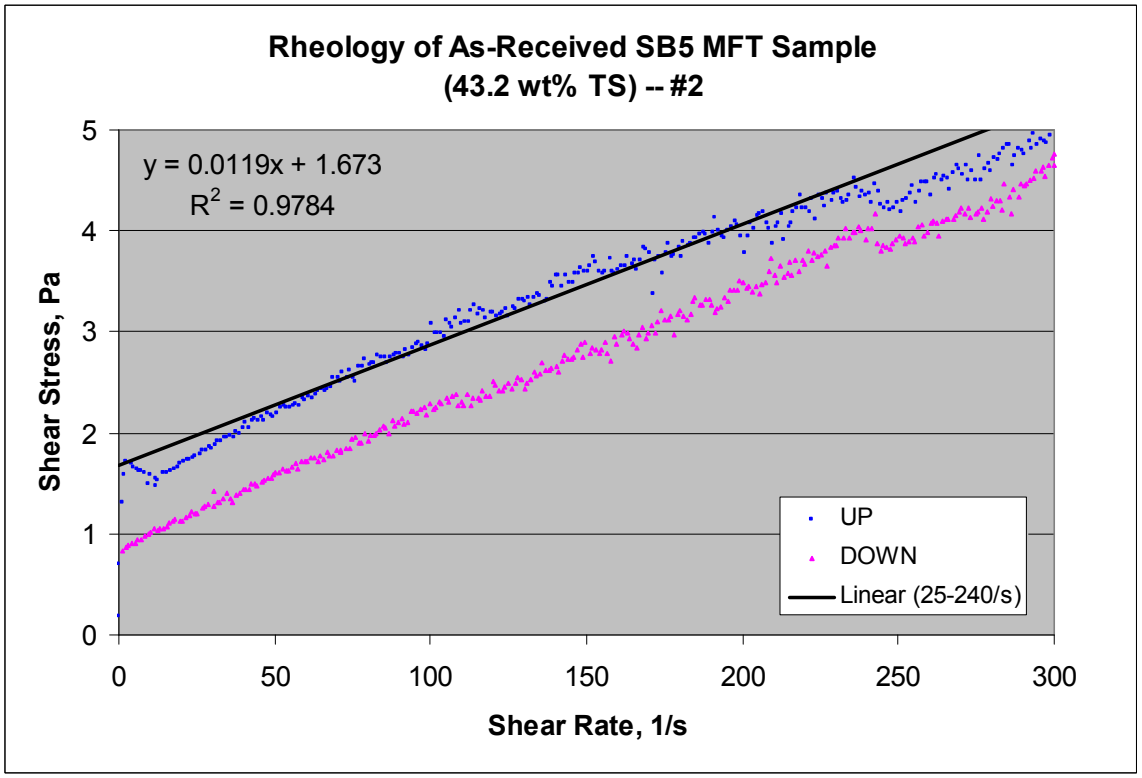
Scott
SRNL Environmental & Chemical Process Technology
5-3737/19369

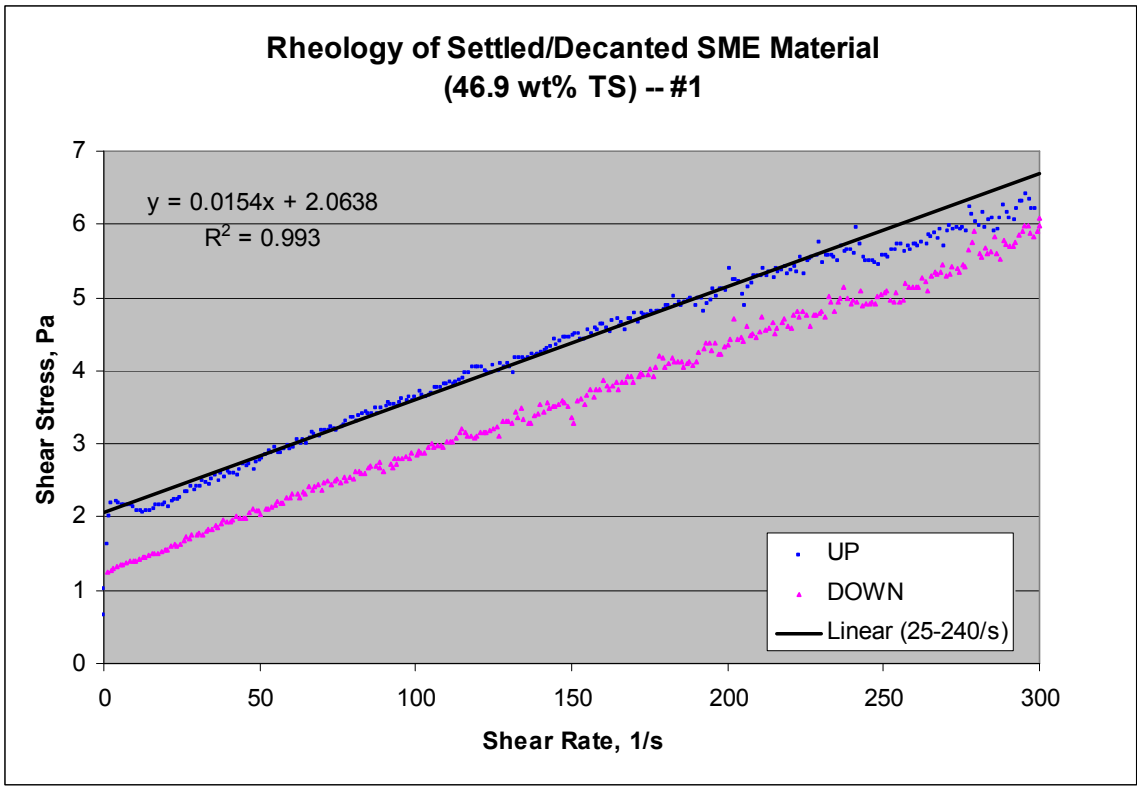
APPENDIX B: FLOW CURVES FROM RHEOLOGY MEASUREMENTS

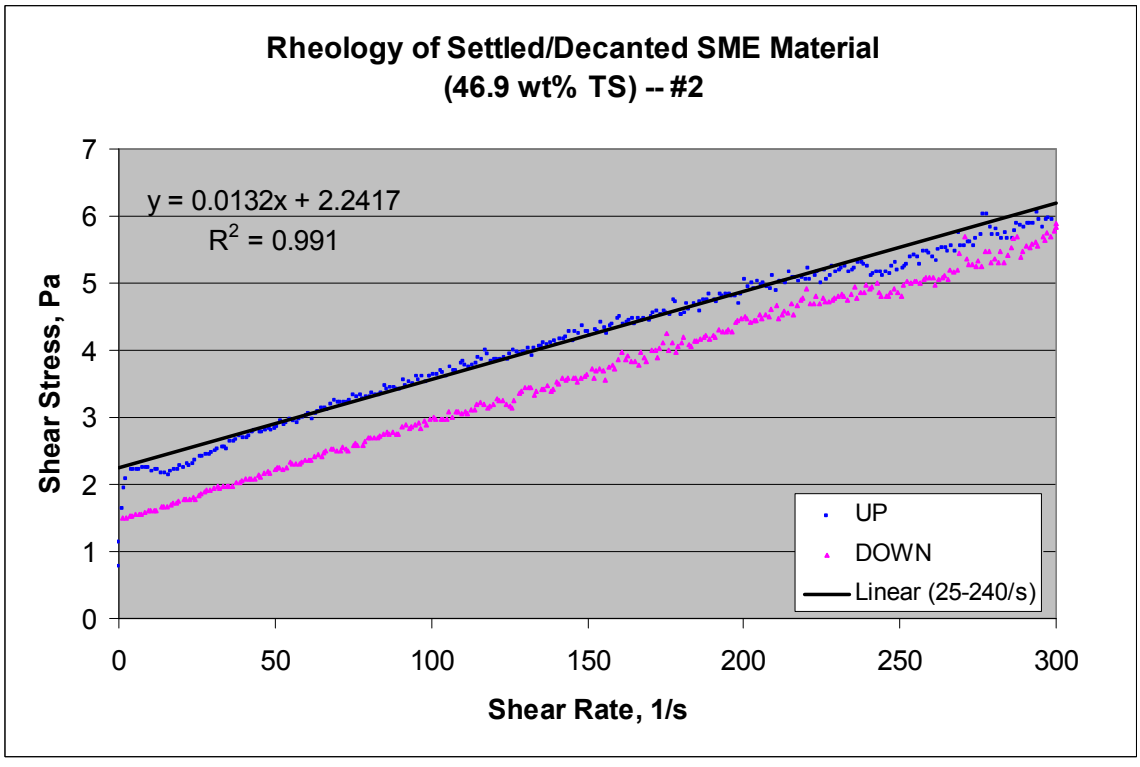


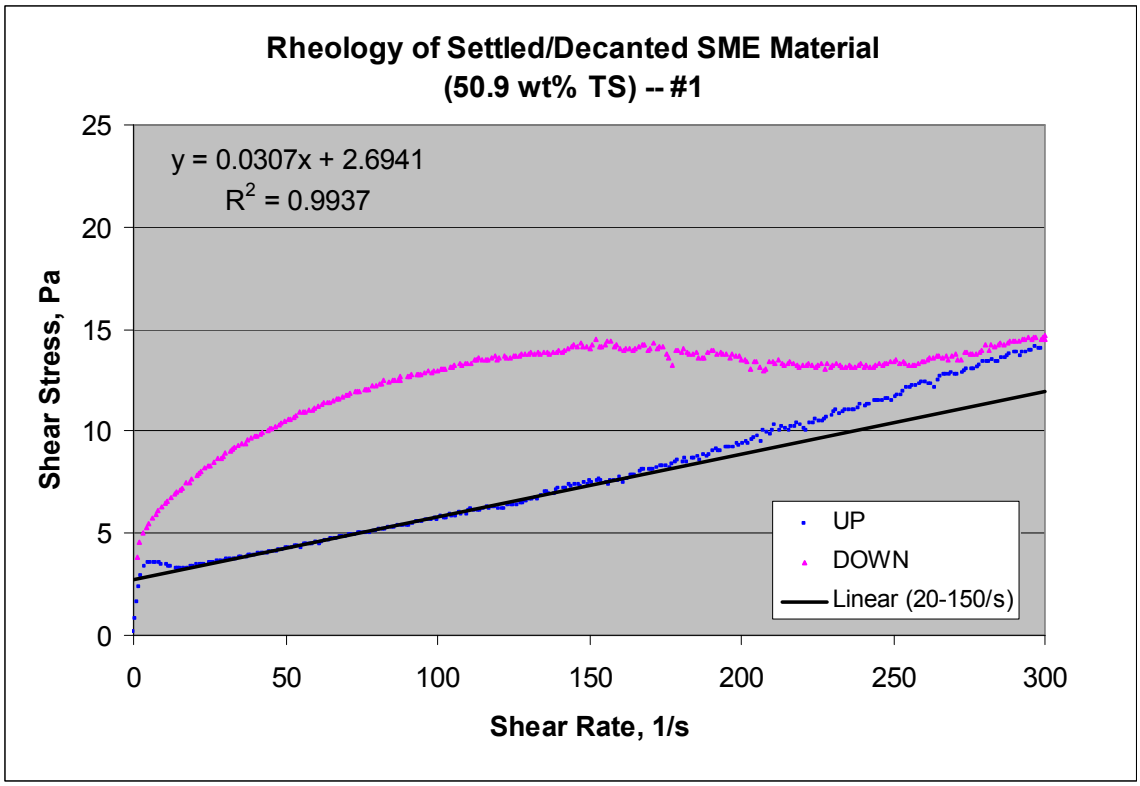


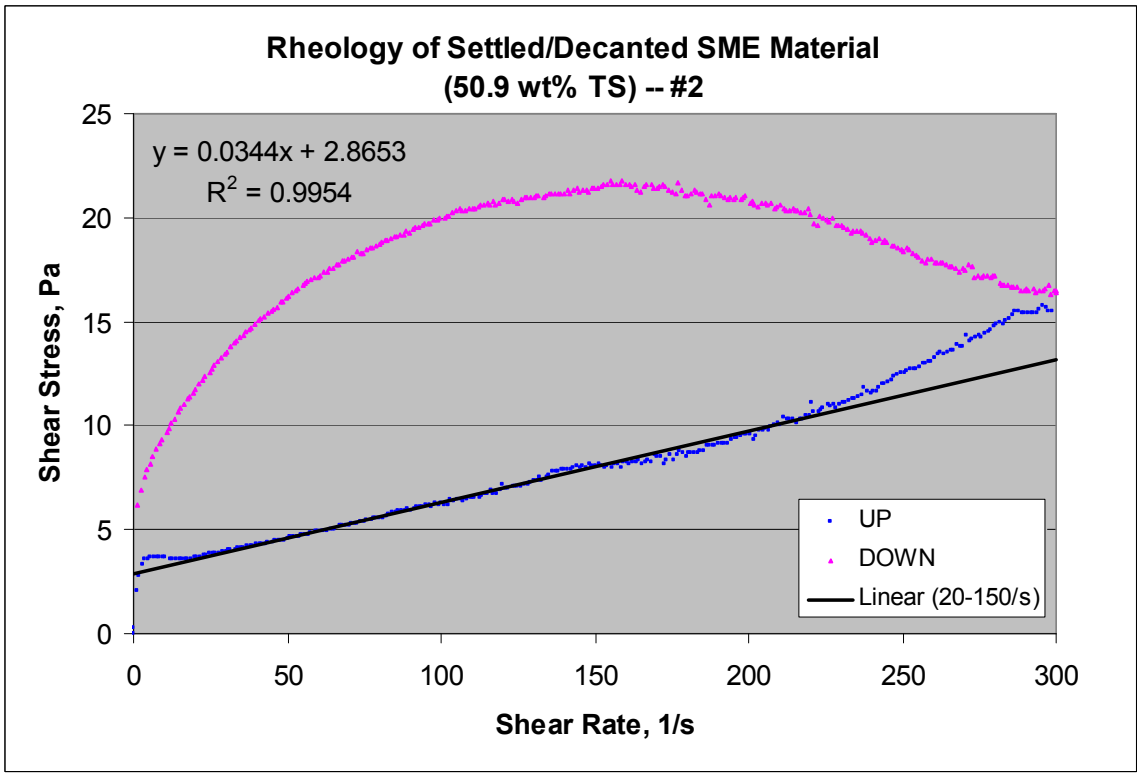

Rheology of As-Received SB5 MFT Sample
(43.2 wt% TS) -- #1
y = 0.011x + 1.7443
R2 = 0.9702
Shear Stress, Pa
Shear Rate, 1/s
UP
DOWN
Linear (25-240/s)
0
1
2
3
4
5
0
50
100
150
200
250
300

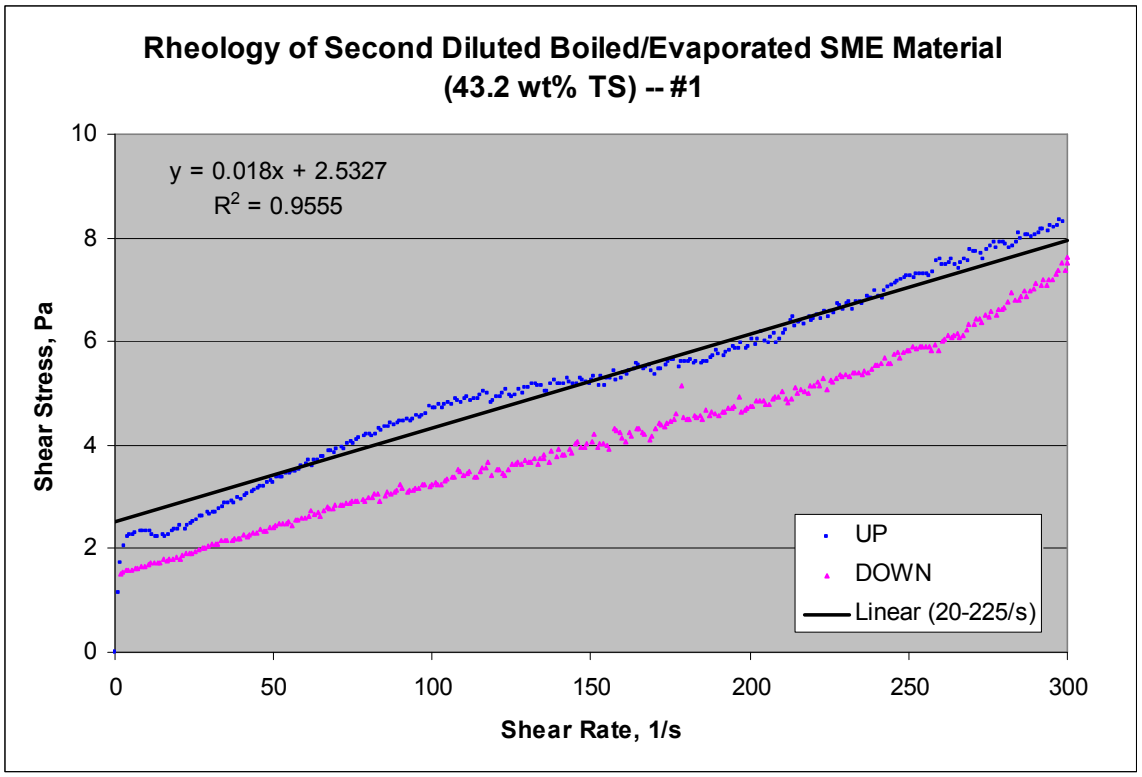

Rheology of As-Received SB5 MFT Sample
(43.2 wt% TS) -- #2
y = 0.0119x + 1.673
R2 = 0.9784
Shear Stress, Pa
Shear Rate, 1/s
UP
DOWN
Linear (25-240/s)
0
1
2
3
4
5
0
50
100
150
200
250
300

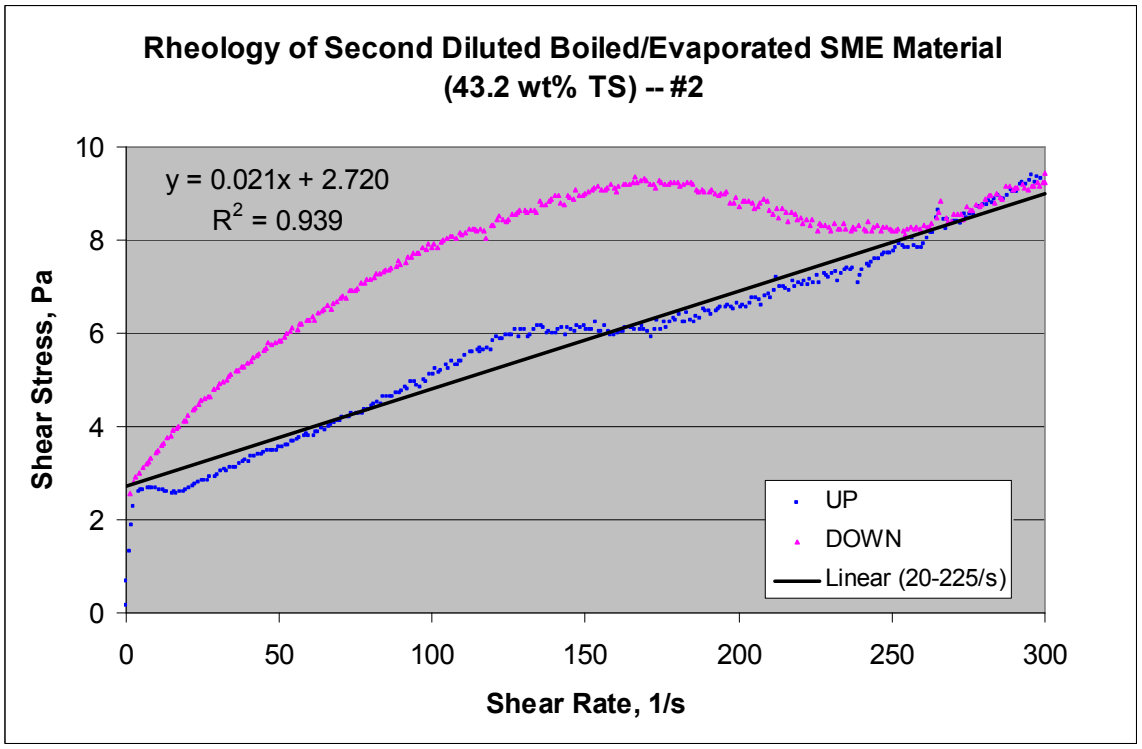

Rheology of Settled/Decanted SME Material
(46.9 wt% TS) -- #1
y = 0.0154x + 2.0638
R2 = 0.993
Shear Stress, Pa
Shear Rate, 1/s
UP
DOWN
Linear (25-240/s)
0
1
2
3
4
5
6
7
0
50
100
150
200
250
300

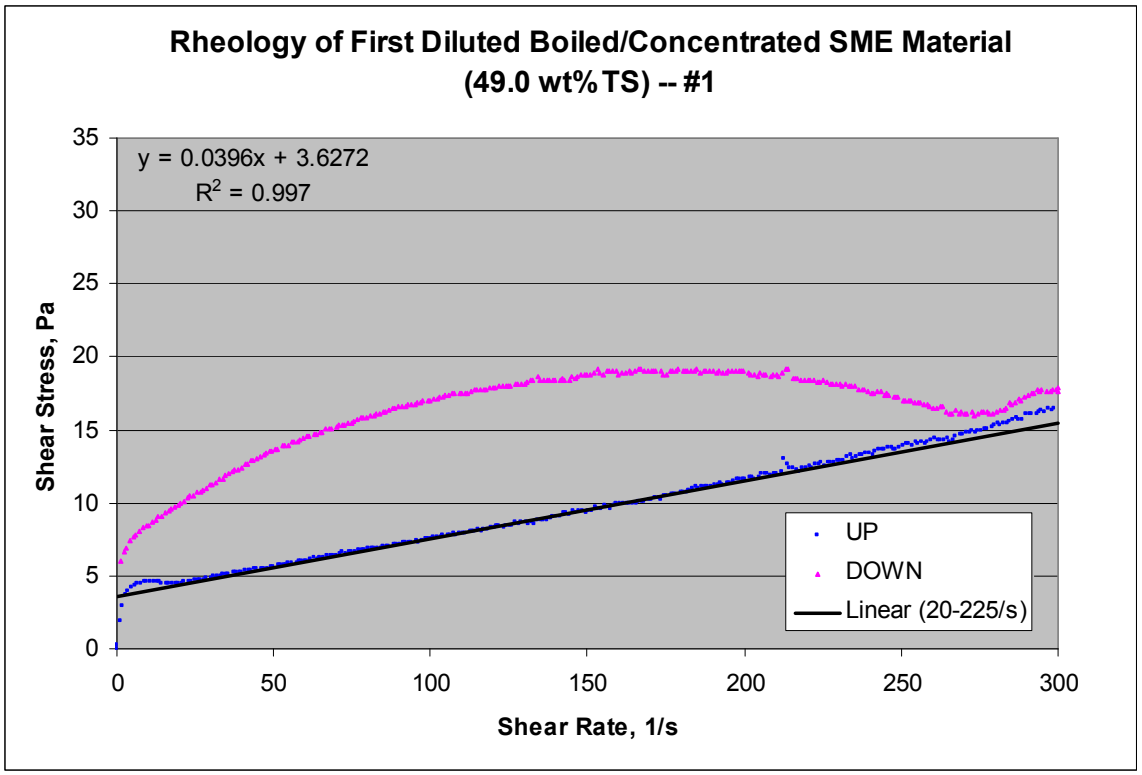

Rheology of Settled/Decanted SME Material
(46.9 wt% TS) -- #2
y = 0.0132x + 2.2417
R2 = 0.991
Shear Stress, Pa
Shear Rate, 1/s
UP
DOWN
Linear (25-240/s)
0
1
2
3
4
5
6
7
0
50
100
150
200
250
300

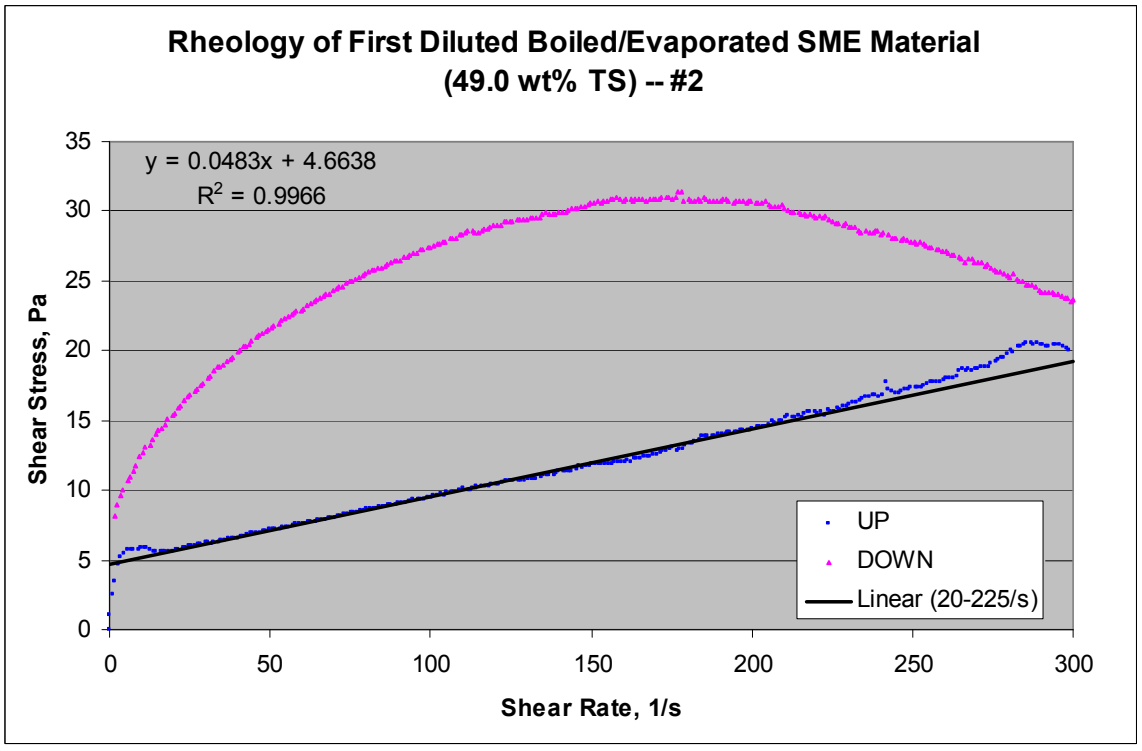

Rheology of Settled/Decanted SME Material
(50.9 wt% TS) -- #1
y = 0.0307x + 2.6941
R2 = 0.9937
Shear Stress, Pa
Shear Rate, 1/s
UP
DOWN
Linear (20-150/s)
0
5
10
15
20
25
0
50
100
150
200
250
300

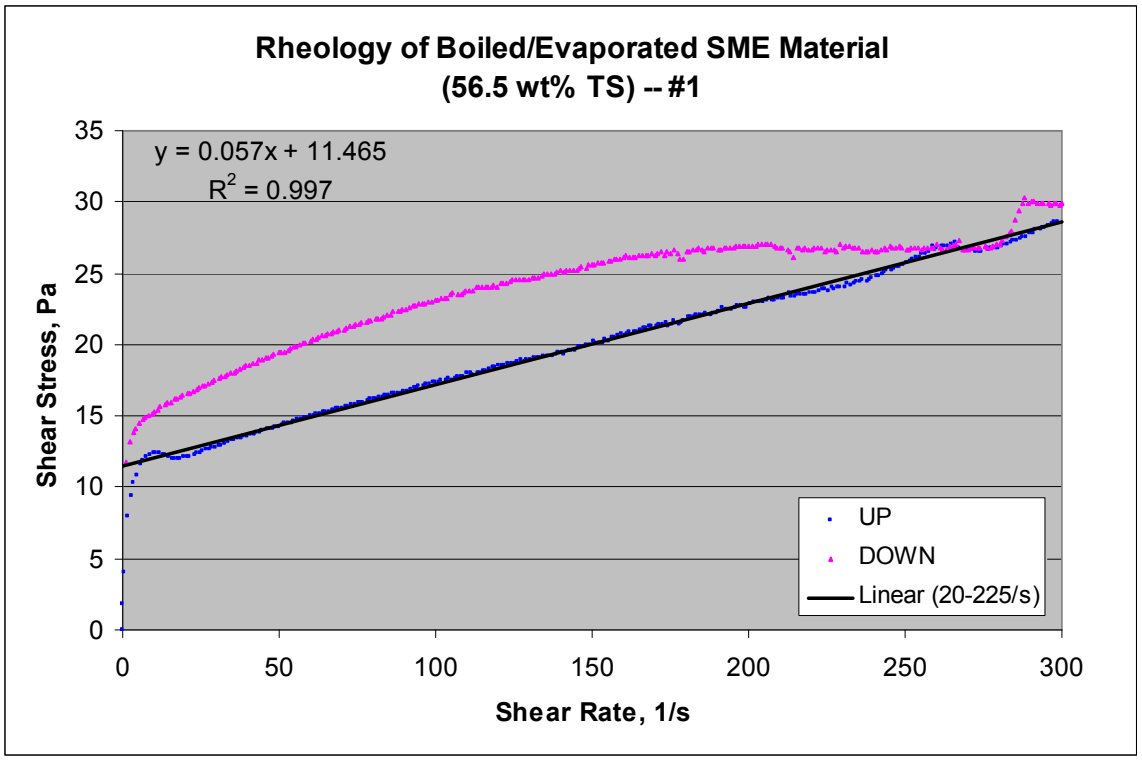

Rheology of Settled/Decanted SME Material
(50.9 wt% TS) -- #2
y = 0.0344x + 2.8653
R2 = 0.9954
Shear Stress, Pa
Shear Rate, 1/s
UP
DOWN
Linear (20-150/s)
0
5
10
15
20
25
0
50
100
150
200
250
300

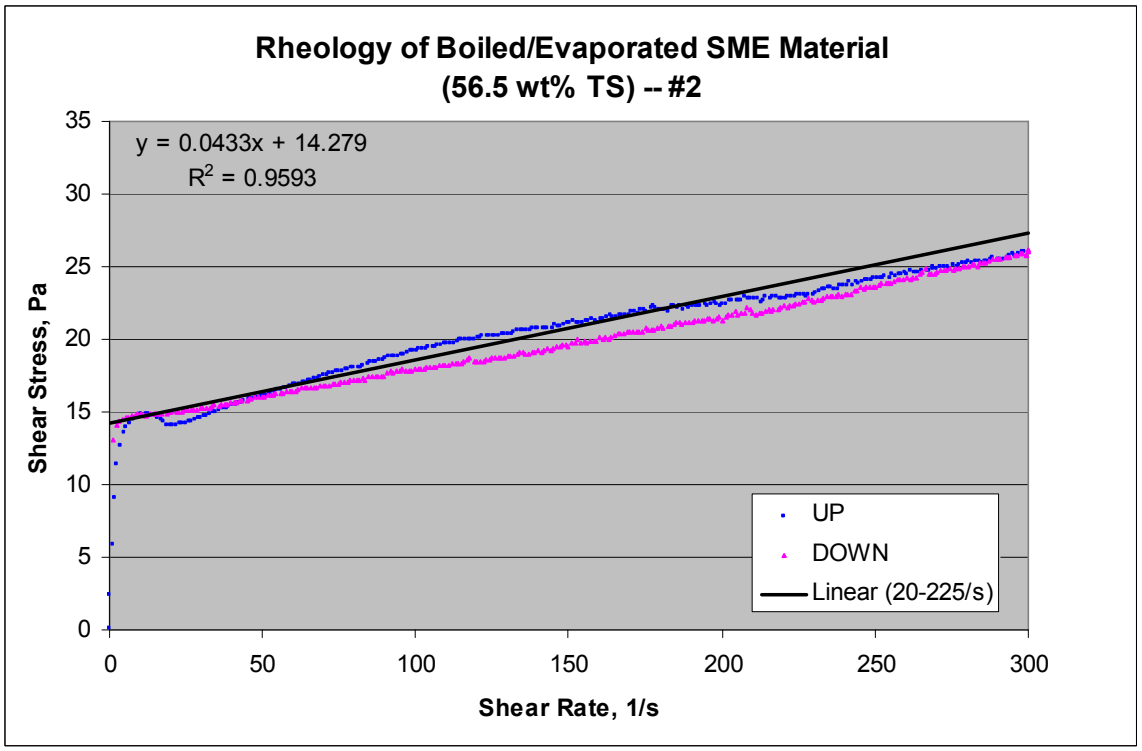

Rheology of Second Diluted Boiled/Evaporated SME Material
(43.2 wt% TS) -- #1
y = 0.018x + 2.5327
R2 = 0.9555
Shear Stress, Pa
Shear Rate, 1/s
UP
DOWN
Linear (20-225/s)
0
2
4
6
8
10
0
50
100
150
200
250
300


Rheology of Second Diluted Boiled/Evaporated SME Material
(43.2 wt% TS) -- #2
y = 0.021x + 2.720
R2 = 0.939
Shear Stress, Pa
Shear Rate, 1/s
UP
DOWN
Linear (20-225/s)
0
2
4
6
8
10
0
50
100
150
200
250
300


Rheology of First Diluted Boiled/Concentrated SME Material
(49.0 wt% TS) -- #1
y = 0.0396x + 3.6272
R2 = 0.997
Shear Stress, Pa
Shear Rate, 1/s
UP
DOWN
Linear (20-225/s)
0
5
10
15
20
25
30
35
0
50
100
150
200
250
300


Rheology of First Diluted Boiled/Evaporated SME Material
(49.0 wt% TS) -- #2
y = 0.0483x + 4.6638
R2 = 0.9966
Shear Stress, Pa
Shear Rate, 1/s
UP
DOWN
Linear (20-225/s)
0
5
10
15
20
25
30
35
0
50
100
150
200
250
300


Rheology of Boiled/Evaporated SME Material
(56.5 wt% TS) -- #1
y = 0.057x + 11.465
R2 = 0.997
Shear Stress, Pa
Shear Rate, 1/s
UP
DOWN
Linear (20-225/s)
0
5
10
15
20
25
30
35
0
50
100
150
200
250
300


Rheology of Boiled/Evaporated SME Material
(56.5 wt% TS) -- #2
y = 0.0433x + 14.279
R2 = 0.9593
Shear Stress, Pa
Shear Rate, 1/s
UP
DOWN
Linear (20-225/s)
0
5
10
15
20
25
30
35
0
50
100
150
200
250
300

Distribution:

B. N. Attaway, 773-A
C. J. Bannochie, 773-42A
A. B. Barnes, 999-W
N. E. Bibler, 773-A
R. W. Blessing, 773-A
J. M. Bricker, 704-27S
D. A. Crowley, 773-43A
T. L. Fellinger, 704-26S
A. I. Fernandez, 999-W
S. D. Fink, 773-A
R. H. Galloway, 773-A
B. J. Giddings, 786-5A
J. M. Gillam, 766-H
B. A. Hamm, 766-H
E. K. Hansen, 999-W
C. C. Herman, 999-W
E. W. Holtzscheiter, 704-15S
S. J. Howard, 773-A
J. F. Iaukea, 704-30S
M. L. Jenkins, 773-A
D. P. Lambert, 999-W
M. T. Keefer, 766-H
D. C. Koopman, 999-W
D. D. Larsen, 766-H
S. L. Marra, 773-A
R. T. McNew, 704-27S
J. E. Occhipinti, 704-S
J. M. Pareizs, 773-A
D. K. Peeler, 999-W
F. M. Pennebaker, 773-42A
H. M. Pittman, 704-27S
J. W. Ray, 704-S
S. H. Reboul, 773-A
H. B. Shah, 766-H
D. C. Sherburne, 704-S
M. E. Stone, 999-W
R. C. Sullivan, 773-A