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WSRC-TR-2005-00294
Revision 0

IMPACT OF SIMULANT PRODUCTION METHODS ON SRAT PRODUCT

D. P. Lambert
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March 2006

Process Science & Engineering Section
Savannah River National Laboratory
Aiken, SC 29808

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EXECUTIVE SUMMARY

Four 2-liter Sludge Receipt and Adjustment Tank (SRAT) cycles were performed: Tests ISPM-T1, ISPM-T6, ISPM-T7, and ISPM-T8. The purpose of these tests was to determine whether sludge simulant production methods have an impact on SRAT processing or SRAT product chemical and physical properties. All four runs used the same Sludge Batch 3 (SB3) chemical composition target but used different production methods. The important process parameters are as follows:

ISPM-T1 Baseline Feed, 155% acid, No Hg, SRAT cycle
ISPM-T6 Coprecipitation of all metals except Al, 155% acid, No Hg, SRAT cycle
ISPM-T7 Coprecipitation of all metals, 155% acid, No Hg, SRAT cycle
ISPM-T8 Coprecipitation of all metals with thermal treatment, 155% acid, No Hg, SRAT cycle

This work is a continuation of a task to assess the impact of simulant production methods on the physical properties of Sludge Batch 3 simulant.[2] In the earlier task, eight batches of sludge were produced and characterized. In this study, four of the eight sludge batches were used to assess the impact of simulant production methods on the physical properties of DWPF Batch 3 SRAT product. The three batches which were closest to the physical properties of actual Batch 3 sludge[3] were chosen along with the baseline sludge for comparison to earlier testing.

Results from processing the four sludge batches were as follows:

- The chemistry of the SRAT process does not mitigate the differences in rheology and particle size distribution between differently prepared sludges of the same nominal composition. Before testing began, it was hypothesized that the SRAT process would eliminate the physical property disparities on the differently prepared test sludges. If this had occurred, all of the products would have similar rheological properties. Instead, the conclusion from this study is that the SRAT does not eliminate physical property disparities between sludges prepared by different methods.
- No foaming or processing issues such as air entrainment were identified. The amount of antifoam used was within the current DWPF antifoam strategy.
- Visually, the sludge slurry and SRAT product appeared to be very thin, and slight problems were experienced with rapid solids settling when the material was not being mixed. No problems with mixing or heating were encountered.
- The chemical composition of the four starting sludge simulants and the four resulting SRAT products were very similar.
- The four pH profiles and resulting final pH of the SRAT products were very similar. As expected, the minimum SRAT pH occurred at the end of acid addition. The measured minimum pH ranged from 4.04-4.14. All runs had a SRAT product pH in the range of pH 4.62 - 4.94.
- The formate destruction was very similar, the destruction efficiency varied from 11% to 22%.
- The four SRAT products were concentrated by removing supernate to perform a rheology study. The more concentrated samples had the highest yield stress values. The average yield stress for the sheared SRAT product at 15 wt% insoluble solids ranged from 2.96-6.64 Pa or 29.6-66.4 dynes/cm^2. The highest yield stress was for the 15 wt % SRAT product from ISMP-T8.
- The plastic viscosity for the sheared SRAT product at 15 wt% insoluble solids ranged from 19.4-28.8 cp. The highest plastic viscosity was for the 15 wt % SRAT product from ISMP-T8.
- The yield stress of each of the four SRAT products was within the operating window for DWPF rheology. The sharp increase in yield stress of the 15 wt % insoluble solids ISMP-T8 SRAT product suggests that processing of these simulants above 15 wt % insoluble solids should be avoided.

- The particle size distributions of the sludge from the three new fabrication methods and their SRAT products were very similar before and after the SRAT cycles. The particle size distribution of the baseline sludge changed considerably during processing with fewer small and large particles in the SRAT product. Each particle size analysis was completed without sonication of the sample.
- ISMP-T1 SRAT product had the smallest average particle size by volume while ISMP-T6 SRAT product had the largest particle size by volume. ISMP-T1 SRAT product also had the smallest average particle size by number, while ISMP-T8 SRAT product had the largest particle size by number.
- SRAT processing had minimal impact on the sludge particle size and particle distribution for ISMP-T8. The sludge used in ISMP-T8 had two peaks both before and after processing. The heat treatment and coprecipitation produced insoluble solids that were stable throughout the SRAT process. In contrast, the sludge for ISMP-T1 had a significant change in particle size as the result of the SRAT processing with the large particles becoming significantly smaller and the smallest particles becoming larger. The SRAT product had a single broad peak at approximately 10 • m, while the starting sludge had two peaks. Also, the distribution of the two peaks was very different from the two peaks seen in the other tests

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF ACRONYMS

ACTL	Aiken County Technologies Laboratory
ADS	Analytical Development Section
ASP	Analytical Study Plan
CBU	Closure Business Unit
CETL	Clemson Environmental Technologies Laboratory
cP	Centipoise
CPC	Chemical Process Cell
DWPF	Defense Waste Processing Facility
FAVC	Formic Acid Vent Condenser
GC	Gas Chromatograph
HLW	High Level Waste
IC	Ion Chromatography
ICP-AES	Inductively Coupled Plasma – Atomic Emission Spectroscopy
MCU	Modular Caustic Side Solvent Extraction Unit
MST	Monosodium Titanate
MWWT	Mercury Water Wash Tank
Pa	Pascals
PSE	Process Science & Engineering Section
SB3	Sludge Batch 3
SB4	Sludge Batch 4
SME	Slurry Mix Evaporator
SMECT	Slurry Mix Evaporator Condensate Tank
SRAT	Sludge Receipt and Adjustment Tank
SRNL	Savannah River National Laboratory
TIC	Total Inorganic Carbon
TTR	Task Technical Request

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1.0 INTRODUCTION AND BACKGROUND

The research and development programs in support of the Defense Waste Processing Facility (DWPF) and other high level waste vitrification processes require the use of both nonradioactive waste simulants and actual waste samples. The nonradioactive waste simulants have been used for laboratory testing, pilot-scale testing and full-scale integrated facility testing. Recent efforts have focused on matching the physical properties of actual sludge. These waste simulants were designed to reproduce the chemical and, if possible, the physical properties of the actual high level waste. This technical report documents a study of simulant production methods for high level waste simulated sludge and their impact on the physical properties of the resultant SRAT product.

The sludge simulants used in support of DWPF have been based on average waste compositions and on expected or actual batch compositions. These sludge simulants were created to primarily match the chemical properties of the actual waste. These sludges were produced by generating manganese dioxide, MnO_2, from permanganate ion (MnO_4^-) and manganous nitrate, precipitating ferric nitrate and nickel nitrate with sodium hydroxide, washing with inhibited water and then addition of other waste species.[1] While these simulated sludges provided a good match for chemical reaction studies, they did not adequately match the physical properties (primarily rheology) measured on the actual waste.

A study was completed in FY04 to determine the impact of simulant production methods on the physical properties of Sludge Batch 3 simulant.[2] This study produced eight batches of sludge simulant, all prepared to the same chemical target, by varying the sludge production methods. The sludge batch, which most closely duplicated the actual SB3 sludge physical properties, was Test 8. Test 8 sludge was prepared by coprecipitating all of the major metals (including Al). After the sludge was washed to meet the target, the sludge simulant was heat treated at 98 °C for eight hours.

Before testing began, it was hypothesized that the SRAT process would eliminate the rheology disparities on the differently prepared test sludges due to the chemistry of the SRAT process. If this hypothesis was true, all of the products would have similar rheological properties.

The objective of the project documented in this report was to determine the best method for producing a DWPF simulated sludge based on the physical properties of the SRAT product. In order to determine the best processing method, four SRAT cycles were completed using the four best sludge simulants.

The objectives of the testing were:

1. Produce four batches of SRAT product to be used primarily in a rheology study.
2. Develop an improved understanding of the impact of various methods of simulated sludge production on the physical properties of the SRAT product.

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2.0 APPROACH

This section describes the approach used to perform the initial Phase I testing. It is divided into four subsections. The first, Section 2.1, describes the sludge simulant composition and the preparation methods. Section 2.2 describes the procedures and equipment utilized in the testing. Section 2.3 describes the analytical methods and procedures used to characterize the sludge and SRAT products. Finally, Section 2.4 describes the preparation of SRAT product samples for rheology analysis.

2.1 Sludge Simulant Preparation

The targeted SB3 simulant composition is given in Table 2-1. The compositional basis is derived from the composition of two dip samples collected from Tank 40H in March 2003.[3] The targeted slurry composition is given in Table 2-2. Table A - 1 shows the typical recipe for the baseline sludge. No uranium was added to the simulant, and no other materials were substituted for the uranium. The recipe was not adjusted for the lack of uranium, but instead the ratios of the various elements to iron were held constant. The preparation of the SB3 sludge simulants are described in a FY04 report.[2] The recipe was followed for fabrication, and the resulting sludge was analyzed before testing was initiated.

One significant difference in composition between the actual high level SB3 sludge waste and the SB3 simulants is the concentration of insoluble solids. The insoluble solids measured in SB3 was 14.8 wt%. The insoluble solids in the four sludges produced by gravity settling varied from 6.85 wt % to 7.99 wt %. This is the maximum concentration of insoluble solids sludge that could be produced using the gravity settling method with a limited settling time. As a result, there is more supernate and less insoluble solids by mass in the simulant than the actual waste.

Before each run, noble metals were added to the sludge in the SRAT vessel to ensure that the small quantities were present at the targeted concentrations. Table 2-3 shows the targeted levels of noble metals. The sludges were not re-analyzed after the noble metals were added since they have a minimal impact on the overall composition.

Table 2-1: Targeted SB3 Simulated Sludge Dried Solids Composition, wt %

Sludge Feed ID	Target
Al	6.04
Ba	0.05
Ca	1.66
Cr	0.25
Cu	0.03
Fe	19.44
K	0.33
Mg	1.67
Mn	3.95
Na	13.84
Ni	1.09
P	0.43
Pb	0.06
S	0.29
Si	0.4
Ti	0.02
Zn	0.03
Zr	0.01

Table 2-2: Projected SB3 Simulated Slurry Composition

PARAMETER	BASELINE
SpGr (kg/L)	1.19
Na (M)	1.43E+00
NO_2 (M)	4.0E-01
NO_3 (M)	2.02E-01
OH (M)	4.30E-01
Cl (M)	6.71E-03
SO_4 (M)	4.18E-02
F (M)	1.47E-02
CO_3 (M)	2.44E-01
AlO_2^{-2} (M)	NA
$C_2O_4^{-2}$ (M)	2.79E-02
PO_4^{-3} (M)	9.91E-02
K (M)	2.01E-02
Insoluble Solids (wt %)	14.8
Total Solids (wt %)	20

Table 2-3: Target Levels of Noble Metals Used in Testing

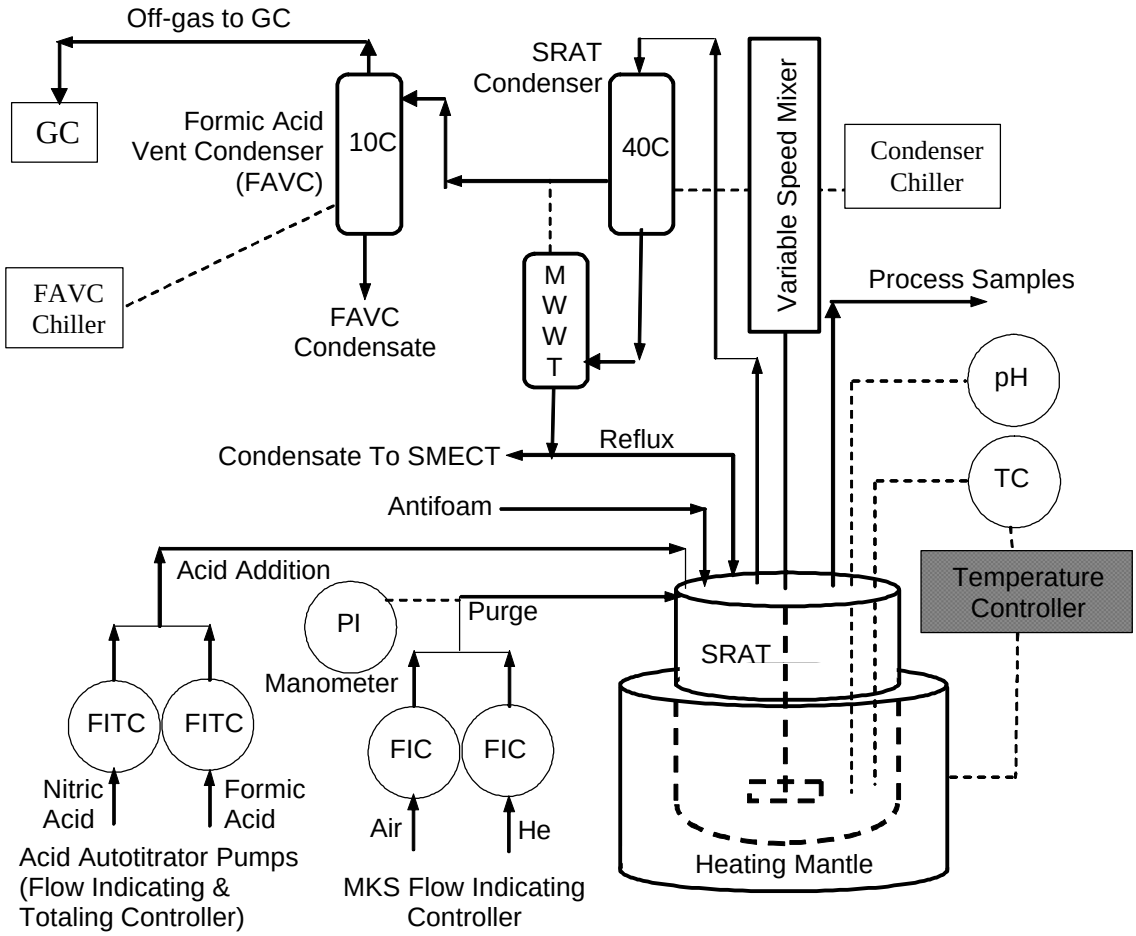
Noble Metal	Wt% in Total Solids
Ag	0.0300*
Pd	0.0014
Rh	0.0076
Ru	0.0360

* Ag added with original sludge preparation

2.2 Procedures and Equipment Used in Testing

The testing was performed at the Aiken County Technology Laboratory (ACTL) using 2-liter kettles with glassware fabricated to functionally replicate the DWPF processing vessels. The 2-L glass kettle is used to replicate the SRAT, and it is connected to the SRAT Condenser, the Mercury Water Wash Tank (MWWT), the Slurry Mix Evaporator Condensate Tank (SMECT), and the Formic Acid Vent Condenser (FAVC). For the purposes of this paper, the condensers and wash tank are referred to as the offgas components. No offgas analysis was completed as part of this testing. A sketch of the experimental setup is given as Figure 2-1.

Figure 2-1: Schematic of SRAT Equipment Set-Up



SRAT processing parameters for the four SRAT cycles are summarized in Appendix A, Table A - 2. Each SRAT test followed the run plans written for ISPM-T1[4], ISPM-T6[5], ISPM-T7[6], ISPM-T8[7], and the memo numbers are given in Table A - 2. The runs were performed in accordance with Procedure ITS-0094 ("Laboratory Scale Chemical Process Cell Simulations") of Manual L29. One significant deviation from normal processing was that dewatering was completed after the twelve-hour reflux period was complete. Slurry pH and temperature were measured during these experiments using in-line instrumentation. During the runs, the kettle was monitored to observe reactions that were occurring during each run, and to observe foaming, air entrainment, rheology changes, loss of heat transfer capabilities, and offgas carryover. Observations were recorded in laboratory notebook WSRC-NB-2005-00032 and are discussed in Section 3.0.

Concentrated nitric acid (50-wt%, 10.53 M) and formic acid (90-wt%, 23.60 M) were used to acidify the sludge and perform neutralization and reduction reactions during processing. The amounts of acid to add for each run were determined using the existing DWPF acid addition equation. The split of the acid was determined using the redox equation currently being used in DWPF processing[8]. The redox target (Fe^{2+}/• Fe) was 0.2. To account for the reactions and anion destructions that occur during processing, assumptions about nitrite destruction, nitrite to nitrate conversion, and formate destruction were made for each run. The values used for each run are provided in Section 3.0.

To prevent foaming during processing, 200 ppm IIT 747 antifoam was added during heat-up at 40°C and 500 ppm was added at the completion of acid addition. SRAT processing included 12 hours of reflux plus the dewater time at boiling to remove the appropriate amount of waster to produce a SRAT product at 22.7 wt % total solids. The dewater time for all of the runs is given in Table A - 2 of Appendix A.

2.3 Analytical

Analyses for this task used the guidance of Analytical Study Plan[9]. Sample request forms were used for samples to be analyzed, and analyses followed the guidelines and means of sample control stated in the Analytical Study Plan for the task. A unique ITS, Immobilization Technology Section - Mobile Lab (Mobile Lab), and/or Analytical Development Section (ADS) lab identification number was assigned to each sample for tracking purposes. Analyses were performed using approved analytical and QA procedures.

2.3.1 Chemical Composition Measurements

Samples were taken of each batch of sludge simulant before the runs were initiated and of each batch of SRAT product at the end of the cycles for analyses. The samples were analyzed by the Mobile Lab, the ITS, and the ADS. The Mobile Lab performed analyses on the sludge slurries to determine the chemical composition, total and dissolved solids, density, and pH. Samples for anion analyses were prepared using weighted dilutions and were analyzed using Ion Chromatography (IC). The chemical composition was determined in duplicate by calcining the samples at 1100 °C and then dissolving the product using Na_2O_2/NaOH fusion and lithium metaborate fusion. The preparations were then analyzed using Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES) to measure the cations present. The total and dissolved solids were measured on two aliquots and the insoluble and soluble solids fractions were calculated from the results. Density and pH measurements of the samples were also performed on the initial and product samples. ITS performed the titration on the starting sludge samples to provide the necessary input for the acid calculation. A manual titration was performed at ACTL using a 1M HNO_3 solution and 10:1 dilution of the sample. The calibration curve was performed to a pH of 4 and was performed in duplicate at a minimum. Finally, the ADS measured the total inorganic carbon (TIC) of the sludge simulant using the ITS Acid Demand TIC method. The total inorganic carbon information was needed as an input in the acid calculation.

2.3.2 Weight Percent Solids and Density Measurements

The weight percent solids were determined using a Mettler Toledo HR73P Halogen Moisture Analyzer. The HR73P is programmed to heat the sample to 105 °C and monitor the mass of the sample until the change in mass is less than or equal to 1 mg over a period of 130 seconds. The advantage of this method is that a weight percent solids analysis can be performed in less than 20 minutes, while a complete analysis of total solids in the sludge and dissolved solids in the supernate can take less than an hour. The homogenous sample (slurry or liquid) is placed on a glass fiber pad and the pad placed in the HR73P. The HR73P weighs the sample. The initial mass of the sample is the total mass (m_{tt}). The sample is then heated by the infrared radiation from a Halogen lamp to 105 °C (controlled by a thermocouple) to drive off all the water (assuming mass loss is only from water) and the resulting remaining mass is the total solids (m_{ts}) in the sample. The weight percent (wt %) total solids (TS) of the sludge was determined using equation [1].

$$wt\%_{ts} = \frac{m_{ts}}{m_{tt}} \times 100\% \qquad [1]$$

A sample of the slurry was centrifuged (at 4332 gravities) to obtain the supernate. The resulting supernate was then processed through a 0.45 μm filter. A sample of the filtered supernate was then placed on a glass fiber pad, placed in the HR73P, and weighed. The mass of sample used was considered as the total mass of the supernate (m_{st}). The sample was then heated by the Halogen lamp to 105 °C to drive off all the water and the resulting remaining mass was the total dissolved solids (m_{ds}) in the supernate. The weight percent of total dissolved solids (DS) in the supernate was determined using equation [2]. This analysis assumes that all the solids in the resulting supernate were dissolved.

$$wt\%_{ds} = \frac{m_{ds}}{m_{st}} \times 100\% \qquad [2]$$

The weight percent of insoluble solids (IS) and soluble solids (SS) of the slurry are then calculated by the following conservation of mass relationships, equations [3] and [4] respectively.

$$wt\%_{is} = \frac{wt\%_{ts} - wt\%_{ds}}{100\% - wt\%_{ds}} \times 100\% \qquad [3]$$

$$wt\%_{ss} = wt\%_{ts} - wt\%_{is} \qquad [4]$$

Density was determined using an Anton Paar DMA 4500 density meter. The density meter determines the density of a sample by measuring the resonant frequency of a sample-filled U tube at a specified temperature.

2.3.3 Rheology Measurements

Slurry rheology measurements were performed using a Haake RS600 rheometer at 25 °C. The rheometer uses a Searle type measuring system, where both speed and torque are measured at the rotating shaft. The rheometer was operated in the controlled rate mode for all of the data reported in this report. A few measurements were also made in the controlled stress mode when additional clarification of a rheology result was needed but are not reported. The measuring geometries used were the cylindrical sensor and cup (Z41 Ti) for the less viscous slurries (when sufficient sample was available) and the cone and plate (60 mm Ti/2 degree) for the slurries that were too thick for loading into the cylindrical geometry or for small samples (which were the majority of samples), which had insufficient material for the cup geometry. The thick slurries were produced by concentrating the SRAT product through supernate removal.

Flow curves were obtained by linearly varying the shear rate from 0 to 600 seconds^{-1} over a given time period. The program details for the flow curves are listed in Table 2-4 and Table 2-5 for the cylindrical and cone geometries respectively. The measured shear stresses for the linear portion of the up and down flow curves were fitted to the Bingham Plastic rheology model (equation 5) over the shear rate range of 50 to 600 seconds^{-1}.

$$\tau = \tau_0 + \eta_0 \dot{\gamma} \qquad [5]$$

τ = shear stress, Pa

τ_0 = Bingham Yield Stress, Pa

$\dot{\gamma}$ = Shear rate, 1/seconds

η_0 = Bingham consistency, mPa.sec (or cP)

The upper limit for the fitted shear rate region was adjusted to a lower value of shear rate when necessary to avoid nonlaminar flow conditions. The lower limit for the fitted shear rate region was adjusted to the start of the linear portion of the curve after any initial hump as long as the shear stress increased with increasing shear rate (positive slope). For any flow curve that did not show a positive slope to the flow curve, the yield stress was taken as the maximum observed in the flow curve.

Table 2-4: Cylindrical Geometry Rheology Program

Program Section	Shear rate, seconds^{-1}	Time, minutes
Up Curve	0 to 600	5
Hold Period	600	1
Down Curve	600 to 0	5

Table 2-5: Cone and Plate Rheology Program

Program Section	Shear rate, seconds^{-1}	Time, minutes
Up Curve	0 to 600	5
Hold Period	600	1
Down Curve	600 to 0	5

2.3.4 Particle Size Measurements

Particle size analysis was obtained by submitting samples to the Analytical Development Section for analysis. Samples were analyzed with a Microtrac S3000 Tri-laser Particle Size Analyzer. This instrument uses angular light scattering techniques to measure the particle size distribution. Preparation of the samples for analysis by the Microtrac consists of dilution of the slurry with water. The particle size distribution can be expressed in terms of a volume distribution, number distribution or area distribution. In this report, the graphical display of particle size data will use the volume distribution. The calculated mean of the volume, number and area distributions will also be reported. It should be noted that the mean for a volume distribution is weighted toward the larger particles while the mean for the number distribution is weighted toward the smaller particles.[2] The calculated specific surface area in meters^2/cm^3 is based on an assumption of smooth, solid spherical particles and does not reflect porosity or topology of the particles.

2.4 Preparation of Samples for Rheological Analysis

The SRAT product samples were concentrated by the removal of supernate to prepare four samples from each run for rheological analysis. The four concentrations were as received, 10 wt %, 12 wt % and 15 wt% insoluble solids. The concentration targets were calculated knowing the total solids of the slurry and filtrate from each run. The calculated quantity of supernate to be removed for each test is summarized in Table 2-6.

Table 2-6: Preparation of SRAT Products for Rheological Analyses

Sample # 1, as is, need 5 mL (6 g) per sample cone and plate, analyze in duplicate

	Initital Mass, g	Final Mass, g	Removed Supernate	Insoluble Solids Mass, g	Soluble Solids Mass, g	Total Solids, g	Water Mass, g	Insoluble Solids, wt %	Total Solids, wt %
SBT1	100.0	100.0	0.0	6.59	14.55	21.14	78.86	6.59%	21.14%
SBT6	100.0	100.0	0.0	7.47	14.35	21.82	78.18	7.47%	21.82%
SBT7	100.0	100.0	0.0	5.95	14.38	20.33	79.67	5.95%	20.33%
SBT8	100.0	100.0	0.0	5.78	14.98	20.76	79.24	5.78%	20.76%

Sample #2, remove supernate until 10 % insoluble solids

	Initital Mass, g	Final Mass, g	Removed Supernate	Insoluble Solids Mass, g	Soluble Solids Mass, g	Total Solids, g	Water Mass, g	Insoluble Solids, wt %	Total Solids, wt %
SBT1	100.0	65.9	34.1	6.59	9.24	15.83	50.07	10.0%	24.0%
SBT6	100.0	74.7	25.3	7.47	10.43	17.90	56.80	10.0%	24.0%
SBT7	100.0	59.5	40.5	5.95	8.19	14.14	45.36	10.0%	23.8%
SBT8	100.0	57.8	42.2	5.78	8.27	14.05	43.75	10.0%	24.3%

Sample #3, remove supernate until 12 % insoluble solids

	Initital Mass, g	Final Mass, g	Removed Supernate	Insoluble Solids Mass, g	Soluble Solids Mass, g	Total Solids, g	Water Mass, g	Insoluble Solids, wt %	Total Solids, wt %
SBT1	100.0	54.92	45.08	6.59	7.53	14.12	40.80	12.0%	25.7%
SBT6	100.0	62.25	37.75	7.47	8.50	15.97	46.28	12.0%	25.6%
SBT7	100.0	49.58	50.42	5.95	6.67	12.62	36.96	12.0%	25.5%
SBT8	100.0	48.17	51.83	5.78	6.74	12.52	35.65	12.0%	26.0%

Sample #4, remove supernate to 15% insoluble solids

	Initital Mass, g	Final Mass, g	Removed Supernate	Insoluble Solids Mass, g	Soluble Solids Mass, g	Total Solids, g	Water Mass, g	Insoluble Solids, wt %	Total Solids, wt %
SBT1	100.0	43.93	56.07	6.59	5.82	12.41	31.53	15.0%	28.2%
SBT6	100.0	49.80	50.20	7.47	6.57	14.03	35.77	15.0%	28.2%
SBT7	100.0	39.67	60.33	5.95	5.16	11.11	28.56	15.0%	28.0%
SBT8	100.0	38.53	61.47	5.78	5.21	10.99	27.55	15.0%	28.5%

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3.0 RESULTS AND DISCUSSION

The data from the testing and any observations will be discussed in this section. This section has been divided into five subsections. Section 3.1 discusses the analyses of the starting sludges and the necessary inputs for the acid calculation. Section 3.2 discusses the general observations about processing and the pH profiles. Section 3.3 discusses the SRAT product characterization. Section 3.4 discusses the SRAT product rheology. Finally, section 3.5 discusses the SRAT product particle size.

3.1 Starting Sludge Composition

Eight sludge batches were produced in FY04 in an attempt to produce a simulant that more closely matched the rheological properties of the actual sludge.[2] Note that these sludges were significantly lower in insoluble solids than the actual waste. This was the result of the slow settling of the insoluble solids using gravity settling. The actual waste was 14.8 wt% insoluble solids and the simulants were 6.9 to 8.0 wt %.

The baseline sludge (Test 1) and the three best sludges (Test 6, 7, and 8) were chosen for this testing (best sludges were chosen based on having similar rheology to actual SB3 sludge). Table 3-1 presents the analysis of the feeds used in this study. Noble metals are routinely added directly to the SRAT vessel rather than in the sludge makeup because they are present in small quantities. When they are added with the sludge, rinse water is also used to ensure that all chemicals and the sludge have been transferred. No samples of the sludge simulant after trimming (i.e., "receipt" samples) were taken in this study, since the only difference between the "sludge" and "receipt" sample was the addition of noble metals, mercury, and water, very little difference was expected in the analytical results and the changes could be calculated based on the known addition amounts. Rinse water would later be removed with the dewater amount during concentration.

Table 3-1: Sludge Simulant Analyzed Compositions

Sludge Feed ID	ISPM-T1 Baseline	ISPM-T6	ISPM-T7	ISPM-T8
Elemental (wt% in calcined solids)				
Al	7.89	7.77	6.24	6.63
Ba	0.050	0.050	0.044	0.045
Ca	2.31	2.21	2.24	2.08
Cr	0.194	0.112	0.1015	0.108
Cu	0.032	0.031	0.038	0.031
Fe	20.3	20.4	19.6	19.5
K	0.266	0.264	0.316	0.220
Mg	1.84	1.78	1.725	1.705
Mn	4.37	4.31	4.07	4.17
Na	23.4	23.4	24.3	24.0
Ni	1.12	1.10	1.06	1.04
P	0.693	0.698	0.731	0.773
Pb	<0.010	0.014	0.019	0.022
S	0.452	0.454	0.445	0.256
Si	0.478	0.443	0.411	0.415
Ti	0.022	0.021	0.015	0.013
Zn	0.037	0.035	0.0315	0.0315
Zr	<0.010	<0.010	<0.010	<0.010
Anions (mg/kg in slurry)				
NO_2^-	16400	16500	16500	16600
NO_3^-	11450	11650	11500	12350
Cl^-	205	207	208	206
SO_4^{2-}	1650	1670	1680	949
$C_2O_4^{2-}$	970	985	990	992
Physical Properties				
ACTL Total Solids (wt %)	15.37%	15.11%	15.23%	15.13%
ACTL Insoluble Solids (wt %)	7.99%	7.56%	7.32%	6.85%
ACTL Soluble Solids (wt %)	7.39%	7.56%	7.91%	8.28%
Calcined Solids (wt %)	11.02%	10.81%	9.85%	9.86%
ACTL Density (g/ml)	1.12	1.12	1.12	1.12
pH	12.09	11.96	11.83	12.1
ADS TIC (mg/kg)	1640	2180	1770	2170
Base Equivalents at pH 7 (Eq/L)	0.714	0.601	0.700	0.649
Calcine Factor	0.733	0.723	0.697	0.695

The acid calculation was performed using the "sludge" analysis data given in Table 3-2. The acid calculation used the average value of nitrite and nitrate for the four sludges. In addition, the Mn result was divided by the calcine factor in each of the acid equation spreadsheets. For example, the measured Mn for Run ISPM-T1 was 4.370 wt% Mn on a total solids basis (or 5.965 wt% Mn on a calcined solids basis) but 5.965 wt % Mn on a total solids basis was used in the acid spreadsheet. This led to the addition of 161.3-162.0% acid, approximately 4% higher than the target.

Table 3-2: Pre-Run Measured Inputs and Assumptions for Acid Calculation

Input Parameter	ISPM-T1 Baseline	ISPM-T6	ISPM-T7	ISPM-T8
Nitrite (mg/kg)	16,500	16,500	16,500	16,500
Nitrate (mg/kg)	11,738	11,738	11,738	11,738
Oxalate (mg/kg)	970	985	990	992
TIC (mg/kg)*	1640	2180	1770	2170
Base Eqv. (M)	0.714	0.601	0.684	0.647
Mn used in acid calc (wt% in total solids)	5.965	5.959	5.843	6.003
Actual Mn (wt% in total solids)	4.370	4.310	4.070	4.170
Total Solids (wt %)	15.37	15.11	15.23	15.12
Density (g/ml)	1.122	1.120	1.121	1.122
Calcine Factor	0.733	0.723	0.697	0.695
Hg (% in Total Solids)	0.00%	0.00%	0.00%	0.00%
Nitrite to Nitrate Conversion	30.00	30.00	30.00	30.00
Formate Destruction	13.00	13.00	13.00	13.00
Sludge Simulant Mass (g)	785.40	785.40	785.40	785.40
Acid Stoichiometry	155%	155%	155%	155%
Recalculated Acid Stoichiometry	161.3%	161.5%	162.0%	162.0%
Redox Target	0.200	0.200	0.200	0.200
Ratio of Formic to Nitric	0.8564	0.8573	0.8570	0.8544
Mol Acid/Liter of Slurry	2.2801	2.2543	2.2604	2.3253

3.2 SRAT Processing

The SRAT runs were performed simultaneously in two different hoods at the ACTL in the 2-liter vessels. The SRAT cycles were initiated after the trim chemicals were added. Nitric acid was added first and then formic acid. After the completion of acid addition, the vessel was ramped to boiling. Once boiling was initiated, the SRAT was refluxed for 12 hours. Dewatering of the SRAT contents was completed after the 12 hour reflux was complete to bring the sludge to the target solids concentration. The dewater time was very short, as reflected in Table A - 2.

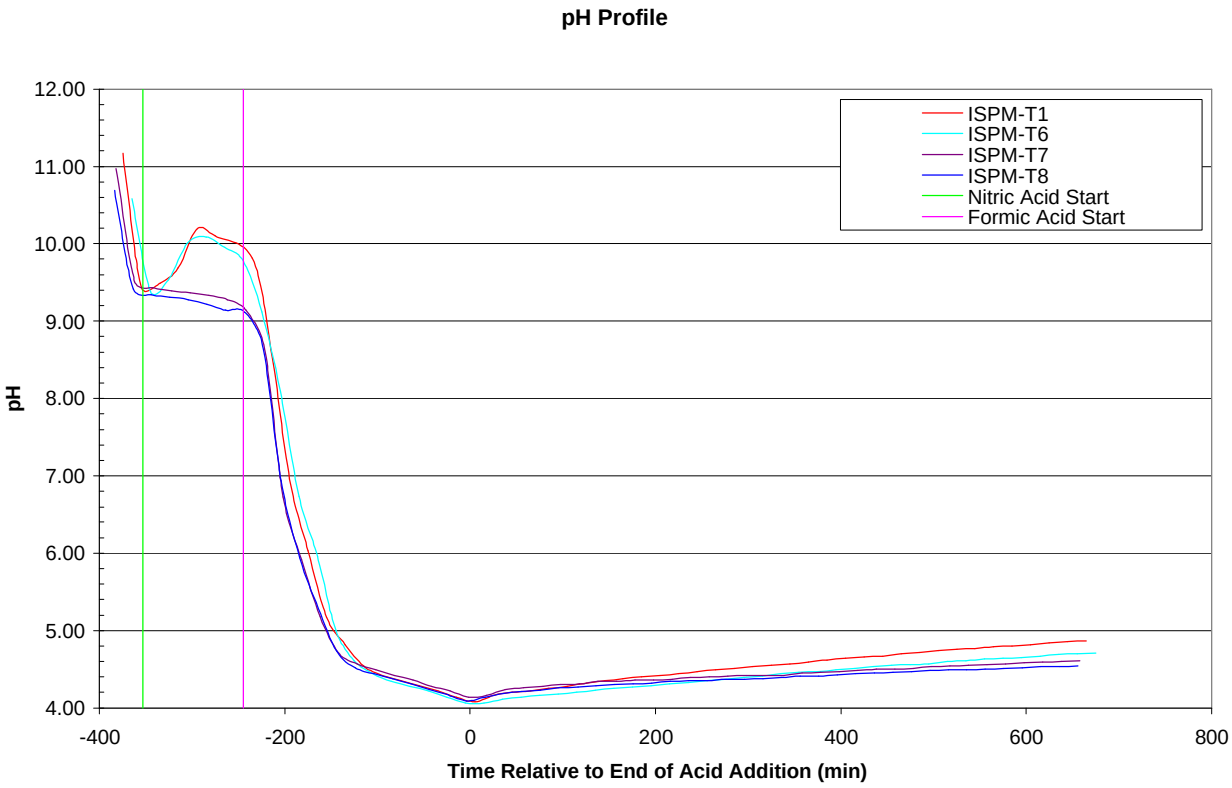
Overall mass balance closure was poor for the runs (within 200 g on a mass basis) compared with other runs at this scale. The bulk of the material balance deficit was probably contained in lost water vapor, solid

deposits on the SRAT vessel and in the offgas non-condensable species (O_2, CO_2, NO, NO_2, N_2O, and H_2). Larger mass losses tend to invalidate calculations on nitrite to nitrate conversion and formate loss.

Mixing and heating of the slurries during the SRAT cycles were not an issue. No problems with foaming or processing of the slurries were evident. No additional antifoam was added to cover the DWPF amount added between acid additions. No other problems were seen during the runs.

The pH was measured throughout the runs. Figure 3-1 is a plot of the measured pH during the SRAT cycle. There was excellent agreement between all four runs from pH 9 to 4. However, two of the SRAT runs had a temporary reversal in the pH trend between pH 11 and pH 9. The only consistent difference between these tests was that the tests with the reversal (Test 1 and Test 6) had aluminum added as the oxide after washing while the tests without the reversal had the aluminum co-precipitated at the start of sludge preparation.

Figure 3-1: pH Plots for All Runs



3.3 SRAT Product Chemical Analyses

The SRAT product from each run was characterized for the anion concentration, cation concentration, solids content, density, and pH. The product anion concentration for each run is given in Table 3-3.

Table 3-3: SRAT Product Anion Concentration (mg/kg)

Anion	Nitrite	Nitrate	Formate
ISPM-T1	<100	37,100	72,950
ISPM-T6	<100	40,950	69,550
ISPM-T7	<100	36,850	78,450
ISPM-T8	<100	38,900	77,700

Note: Analyses performed on weighted dilution of samples.
Results represent an average of two measurements.

The percent conversion of nitrite to nitrate and percent destruction of formate during the SRAT process is given in Table 3-4. The conversions/destructions are calculated based on the amount of nitrite, nitrate, and formate in the simulant and added during processing versus the amount that is present in the SRAT product. The calculation of the formate destruction and especially the nitrate conversion is strongly impacted by the mass balance closure for each run. For example, if the mass balance indicates there is more final product than was added based on known additions, the nitrate conversion will be higher than is reasonable. As a result, the nitrite to nitrate conversion is much higher than the prediction for runs T6 and T8 and the formate conversion is lower than predicted for run T8. As a result, for runs T6 and T8 the nitrite to nitrate conversion is less than the predictions and the formate conversion may be more than the prediction. The concentrations of the actual nitrate, nitrite, and formate indicate that the formate destruction and nitrite to nitrate conversion is similar in all four runs.

Table 3-4: Formate Destruction, Nitrate Conversion – SRAT Receipt Relative to SRAT Product

Run ID	% Acid	Hg Present	% Formate Destruction		% Nitrite to Nitrate Conversion	
			Predicted	Measured	Predicted	Measured
ISPM-T1	161.3	No	13	12.0	30	32.0
ISPM-T6	161.5	No	13	11.1	30	59.2
ISPM-T7	162.0	No	13	13.3	30	15.7
ISPM-T8	162.0	No	13	0.8	30	58.5

As mentioned in Section 2.3, the SRAT products were calcined at 1100°C in order to prepare them for cation analyses. The elements detected in the calcined solids are given in Table 3-5.

Table 3-5: SRAT Product Results (Calcined Solids Wt %)

Sludge Feed ID	ISPM-T1 Baseline	ISPM-T6	ISPM-T7	ISPM-T8
Al	5.46	6.53	5.77	6.14
Ba	0.05	0.04	0.04	0.04
Ca	2.11	1.80	1.84	1.86
Cr	0.20	0.12	0.13	0.13
Cu	0.01	0.01	0.02	0.01
Fe	21.15	21.30	18.90	18.95
Gd	0.06	0.05	0.06	0.05
K	0.50	0.46	0.55	0.46
Mg	1.82	1.62	1.58	1.52
Mn	4.65	4.70	4.02	3.95
Na	23.70	22.80	24.90	24.80
Ni	1.15	1.22	1.00	0.94
P	0.71	0.69	0.76	0.76
Pb	0.03	0.03	0.03	0.03
S	0.48	0.47	0.52	0.27
Si	0.56	0.49	0.42	0.45
Ti	0.05	0.04	0.04	0.04
Zn	0.02	0.02	0.02	0.02
Zr	0.04	0.05	0.04	0.04

Note: Two aliquots are removed from the product sample. Each aliquot is then calcined, dissolved, and analyzed. Results represent an average of the two measurements. The sum of oxides for these analyses was outside the 95-105% target expected. The reason for the low sum of oxides (89.3-92.8% is likely due to incomplete calcination or digestion. Insufficient sample was available to repeat the sample preparation and measurement.

When the SRAT product compositions are compared with the simulant compositions given in Table 3-1, most of the oxides are very similar. Overall, the compositions represented a reasonable estimation of the SB3 simulant major components.

The total and dissolved solids were measured on the SRAT products, and the insoluble and soluble solids were then calculated. As mentioned above, the calcined solids were also measured. To complete the physical property analyses, the slurry density and pH were measured. The results are given in Table 3-6.

Table 3-6: Physical Property Data on SRAT Products

Analysis	ISPM-T1	ISPM-T6	ISPM-T7	ISPM-T8
Slurry Total Solids, wt %	21.14	21.83	20.33	20.76
Insoluble Solids, wt %	6.59	7.47	5.95	5.78
Soluble Solids, wt %	14.55	14.35	14.38	14.98
Density, g/mL	1.156	1.162	1.151	1.154
Filtrate Solids, wt %	15.58	15.51	15.29	15.90
pH	4.94	4.81	4.71	4.62

Note: Measured on two aliquots from the same sample. Data reported is an average. Total and dissolved solids were actually measured and insoluble and soluble solids were calculated.

3.4 SRAT Product Rheology

SRAT product samples at four levels of insoluble solids (as received, 10%, 12% and 15%) were produced by removing supernate from 100 g SRAT product samples. The as received SRAT products were rheologically thin, so no dilution of the samples was warranted. The target for each of the samples is discussed in section 2.4. The results of the preparation of these samples relative to the target are reported in section 3.4.1. The rheological results are reported in section 3.4.2 and Appendix A. Note that several of the targets could not be reached due to inadequate gravity settling of the samples.

3.4.1 Rheology Sample Preparation

The concentrated SRAT samples were prepared by removing supernate (soluble solids plus water) from the original slurry after concentrating through gravity settling. Two of the samples required additional concentration by centrifuging for three minutes (Test 1 and Test 8 SRAT Products targeting 15 wt % insoluble solids) at 500 rpm using an IEC Centra GP8 centrifuge. The resultant concentrated slurry had the same mass of insoluble solids as the original slurry, assuming no insoluble solids were removed. The concentrated samples were analyzed for total solids. The insoluble solids concentration was calculated from the previously analyzed supernate solids analysis. The calculated removal of supernate from the original SRAT products to produce the sixteen concentrated rheology samples is summarized in Table 3-7. Note that the measured total solids were within 5% of the target for all samples.

Table 3-7: Preparation of Concentrated SRAT Products

Rheology Sample	Predicted Total Solids, wt%	Measured Total Solids, wt%	Initial Slurry, g	Removed Supernate, g	Final Slurry, g
ISPM-T1-as is*	21.14%	21.14%	100.00	0.00	100.00
ISPM-T1-10%	24.02%	24.49%	100.00	34.10	65.90
ISPM-T1-12%	25.31%	25.81%	100.00	42.85	57.15
ISPM-T1-15%	26.96%	28.28%	100.00	51.15	48.86
ISPM-T6-as is	21.82%	21.82%	100.00	0.00	100.00
ISPM-T6-10%	23.96%	23.68%	100.00	25.31	74.69
ISPM-T6-12%	25.65%	25.41%	100.00	37.75	62.25
ISPM-T6-15%	28.18%	28.25%	100.00	50.20	49.80
ISPM-T7-as is	20.33%	20.33%	100.00	0.00	100.00
ISPM-T7-10%	23.76%	24.06%	100.00	40.50	59.50
ISPM-T7-12%	25.45%	25.58%	100.00	50.42	49.59
ISPM-T7-15%	27.89%	28.38%	100.01	59.99	40.01
ISPM-T8-as is	20.76%	20.76%	100.00	0.00	100.00
ISPM-T8-10%	24.31%	24.42%	100.00	42.20	57.80
ISPM-T8-12%	25.99%	26.31%	100.00	51.82	48.18
ISPM-T8-15%	28.52%	29.01%	100.00	61.47	38.53

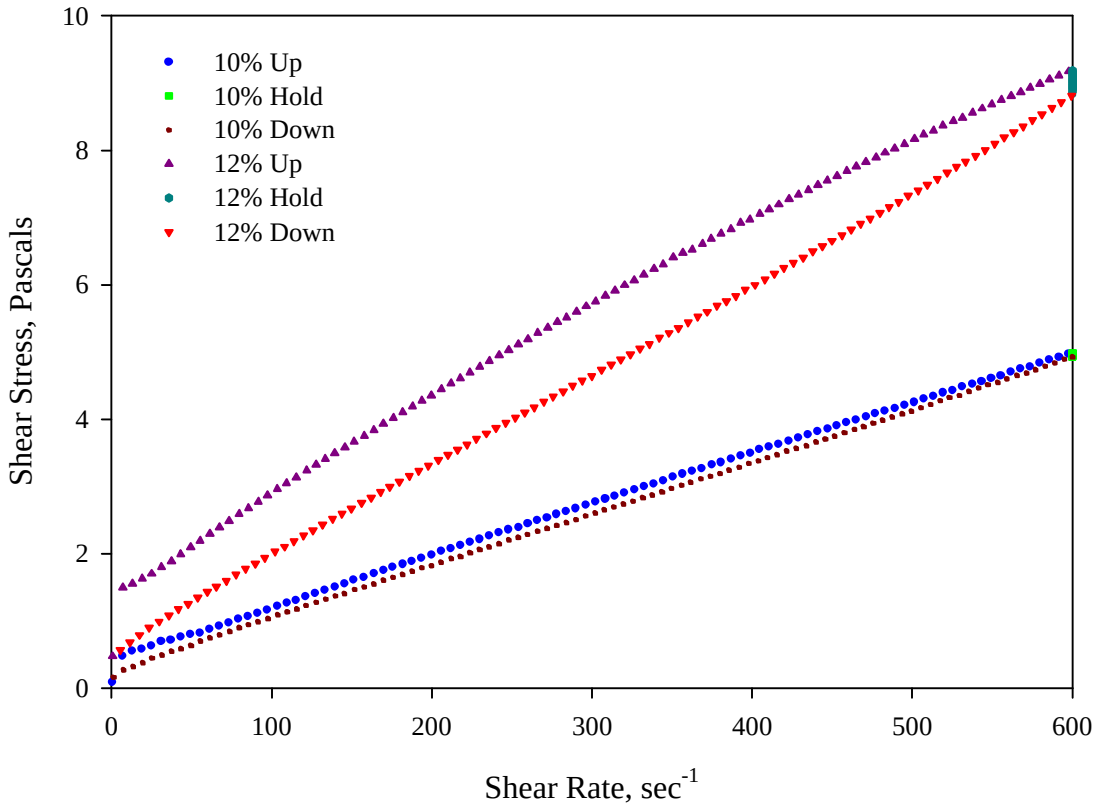
"as is" is equivalent to as received

3.4.2 Rheology Results

Rheological analyses of all of the SRAT products were performed at a minimum in duplicate. Appendix B presents the flow curves and the individual and averaged Bingham Plastic yield stress and plastic viscosity determined using the Bingham Plastic rheological model. All of the products at low wt % insoluble solids content were visually and rheologically thin, and only minor differences were observed for product and feed slurries.

The flow curves for the low wt % insoluble solids products showed that the material was initially nearly Newtonian in fluid properties shifting to increasingly non-Newtonian as the solids loading increased. At a solids loading of 12 wt % and higher, the samples began to show thixotropic fluid properties (thinning with time under shear). Figure 3-2 shows an example of flow curves for nominally 10 and 12 wt % insoluble solids SRAT products produced from the Test 7 sludge. The up and down portion of the flow curve for the 10 wt % insoluble solids sample show a very small separation while the 12 wt % insoluble solids sample shows substantial shear thinning. All of the SRAT products at the highest insoluble solids loading showed an even greater thixotropic behavior.

Figure 3-2: ISPM-T7 SRAT Product Flow Curves



In order to properly represent the properties before and after shearing, the results for the linear regression of the Bingham equation on the up and down flow curves are reported separately in Appendix B. The down flow curve represents the sheared SRAT material and is the normal operating regime for the SRAT product (continuous agitation). The up flow curve represents the unsheared SRAT product and would be typical of an abnormal (such as restart after layup) condition for the SRAT process.

The as received and concentrated rheological analyses were performed by PSE technicians at ACTL. The rheology analyses are summarized in Table 3-9 and Table 3-10. The Bingham Plastic yield stress data (individual data points and average line) are graphed in Figure 3-3 and Figure 3-4. The plastic viscosity data (individual and average) are graphed in Figure 3-5 and Figure 3-6. The more concentrated samples had the highest yield stress values. The average yield stress at 15 wt% insoluble solids ranged from 2.96-6.64 Pa or 29.6-66.4 dynes/cm^2 for the sheared samples (down flow curves) and 9.11-21.45 Pa or 91.1-214.5 dynes/cm^2 for the unsheared samples (up flow curves). The highest yield stress for a sheared sample was for the 15 wt

% SRAT product from Test 8. The plastic viscosity at 15 wt% insoluble solids ranged from 19.4-28.8 cp. The highest plastic viscosity was for the 15 wt % SRAT product from Test 8. Note that ISPM-T8 had a very sharp increase in yield stress from 12% to 15% for the sheared data, while that magnitude of increase was not seen in the other three experiments for the sheared data. All of the unsheared samples showed a sharp increase in yield stress at the highest solids loading.

Flow curve phenomena similar to those seen with Sludge Batch 2 simulant sludge slurry were seen in many of these SRAT products. The earlier behavior was examined in detail by Koopman[10]. These included a complex behavior (structural breakdown) during the initial ramp of shear rate from zero to the maximum shear rate. The down curve data was linearly regressed using the Bingham Plastic model, since the down curves were more nearly linear and would be the appropriate parameters for slurry in a fully-developed steady shear flow (normal SRAT operation).

Table 3-8: Physical Property Data on Sheared SRAT Products

Run	Total Mass, g	Water Mass, g	Total Solids, g	Soluble Solids, g	Insoluble Solids, g	% Insoluble Solids
ISPM-T1-asis	100.00	78.86	21.14	14.55	6.59	6.59%
ISPM-T1-10%	65.90	49.76	16.14	9.18	6.96	10.56%
ISPM-T1-12%	57.15	42.40	14.75	7.82	6.93	12.12%
ISPM-T1-15%	48.86	35.04	13.82	6.47	7.35	15.05%
ISPM-T6-asis	100.00	78.18	21.82	14.35	7.47	7.47%
ISPM-T6-10%	74.69	57.01	17.69	10.46	7.22	9.67%
ISPM-T6-12%	62.25	46.43	15.82	8.52	7.29	11.72%
ISPM-T6-15%	49.80	35.73	14.07	6.56	7.51	15.08%
ISPM-T7-asis	100.00	79.67	20.33	14.38	5.95	5.95%
ISPM-T7-10%	59.50	45.19	14.32	8.16	6.16	10.35%
ISPM-T7-12%	49.59	36.90	12.68	6.66	6.02	12.15%
ISPM-T7-15%	40.01	28.66	11.36	5.17	6.18	15.45%
ISPM-T8-asis	100.00	79.24	20.76	14.98	5.78	5.78%
ISPM-T8-10%	57.80	43.68	14.11	8.26	5.86	10.13%
ISPM-T8-12%	48.18	35.50	12.68	6.71	5.96	12.38%
ISPM-T8-15%	38.53	27.35	11.18	5.17	6.01	15.53%

"as is" is equivalent to as received

Table 3-9: Rheology Data on Sheared SRAT Products

Sample	Average Plastic Viscosity (cP)	Average Yield Stress (Pa)	Predicted Insoluble Solids, wt%	Measured Insoluble Solids, wt%	Predicted Total Solids, wt%	Measured Total Solids, wt%
ISPM-T1-asis	4.3	0.56	6.59%	6.59%	21.14%	21.14%
ISPM-T1-10%	10.6	2.47	10.00%	10.56%	24.47%	24.49%
ISPM-T1-12%	14.3	3.55	11.53%	12.12%	25.97%	25.81%
ISPM-T1-15%	21.5	4.58	13.49%	15.05%	27.88%	28.28%
ISPM-T6-asis	4.1	0.29	7.47%	7.47%	21.82%	21.82%
ISPM-T6-10%	6.4	0.78	10.00%	9.67%	24.03%	23.68%
ISPM-T6-12%	10.3	1.53	12.00%	11.72%	25.77%	25.41%
ISPM-T6-15%	19.4	2.97	15.00%	15.08%	28.39%	28.25%
ISPM-T7-asis	3.2	0.02	5.95%	5.95%	20.33%	20.33%
ISPM-T7-10%	7.6	0.29	10.00%	10.35%	23.76%	24.06%
ISPM-T7-12%	13.6	0.60	12.00%	12.15%	25.45%	25.58%
ISPM-T7-15%	25.7	3.32	14.87%	15.45%	27.89%	28.38%
ISPM-T8-asis	3.4	0.04	5.78%	5.78%	20.76%	20.76
ISPM-T8-10%	9.0	0.47	10.00%	10.13%	24.31%	24.42%
ISPM-T8-12%	14.9	1.33	12.00%	12.38%	25.99%	26.31%
ISPM-T8-15%	28.8	6.64	15.00%	15.53%	28.52%	28.96%

"as is" is equivalent to as initially produced

Rheological data were obtained on the SB2/3 blend SRAT cycle product made in the Shielded Cells in 2004 (C. J. Bannochie, J. M. Pareizs, and D. C. Koopman, Sludge Batch 2/3 Blend SRAT Cycle in the SRNL Shielded Cells, WSRC-TR-2004-00097, May 2004, etc.). The starting sludge for this test was very close in composition to that which formed the basis for the simulants in this study. The SRAT product was at 11.8 wt. % insoluble solids and 27.3 wt. % total solids, i.e. fairly similar to the -12% simulant compositions above. The stoichiometric acid factor for the Shielded Cells run has been recomputed to correct for an error in one of the base equivalents titration results and for an improved value of the formic acid molarity. The revised value is 150.5%, which is fairly close to the 155% target in the simulant runs.

Two flow curves were made on this sample using the RV30 rheometer in the Shielded Cells. The MV1 concentric cylinder geometry was used, which is generally similar to the Z41 geometry used in the RS600 simulant measurements. These produced a yield stress of 1.17 Pa and a plastic viscosity of 4.9 cP. The yield stress is essentially the same as the average of the ISPM-T6, ISPM-T7, and ISPM-T8 results. The plastic viscosity is about half as large as the ISPM-T6, ISPM-T7, and ISPM-T8 results. This is still fairly good agreement between simulant and radioactive sample. The Shielded Cells sample, however, was not thixotropic, therefore, the distinction between sheared and unsheared results for the Shielded Cells sample did not exist.

Table 3-10: Rheology Results for Unsheared SRAT Samples

Run	% Total Solids	% Insoluble Solids	Average Plastic Viscosity (cP)	Average Yield Stress (Pa)
ISPM-T1-asis	21.14%	6.59%	4.3	0.60
ISPM-T1-10%	24.49%	10.56%	9.2	4.75
ISPM-T1-12%	25.81%	12.12%	8.0	8.64
ISPM-T1-15%	28.28%	15.05%	NA*	21.47*
ISPM-T6-asis	21.82%	7.47%	4.0	0.37
ISPM-T6-10%	23.68%	9.67%	6.3	0.91
ISPM-T6-12%	25.41%	11.72%	9.5	2.54
ISPM-T6-15%	28.25%	15.08%	10.1	10.64
ISPM-T7-asis	20.33%	5.95%	2.5	0.39
ISPM-T7-10%	24.06%	10.35%	7.6	0.46
ISPM-T7-12%	25.58%	12.15%	13.0	1.76
ISPM-T7-15%	28.38%	15.45%	20.7	9.13
ISPM-T8-asis	20.76%	5.78%	3.4	0.06
ISPM-T8-10%	24.42%	10.13%	8.8	0.79
ISPM-T8-12%	26.31%	12.38%	13.8	3.07
ISPM-T8-15%	28.96%	15.53%	15.9	18.34

*Yield Stress was taken as the maximum in the curve between 0 and 100 sec^{-1} since the curve did not properly reflect a Bingham material before shearing (i.e. the shear stress declined with increasing shear rate). Plastic viscosity could not be determined for the same reason.
"as is" is equivalent to as initially produced.

Figure 3-3: Yield Stress as a Function of Insoluble Solids for Sheared SRAT Product

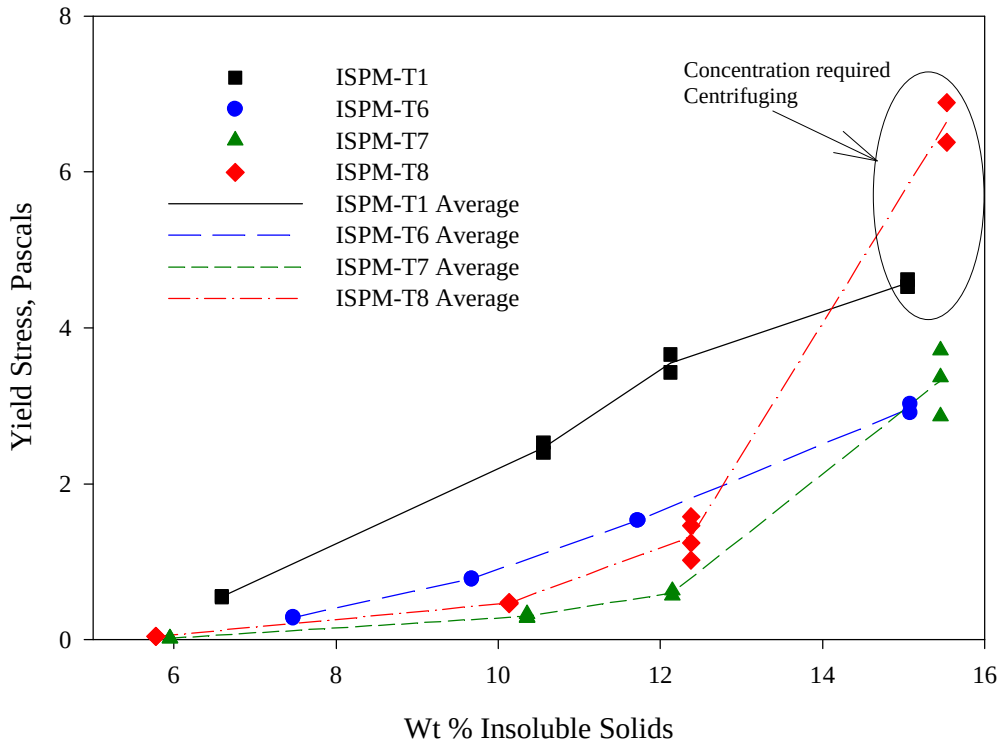


Figure 3-4: Yield Stress as a Function of Insoluble Solids for Unsheared SRAT Slurries

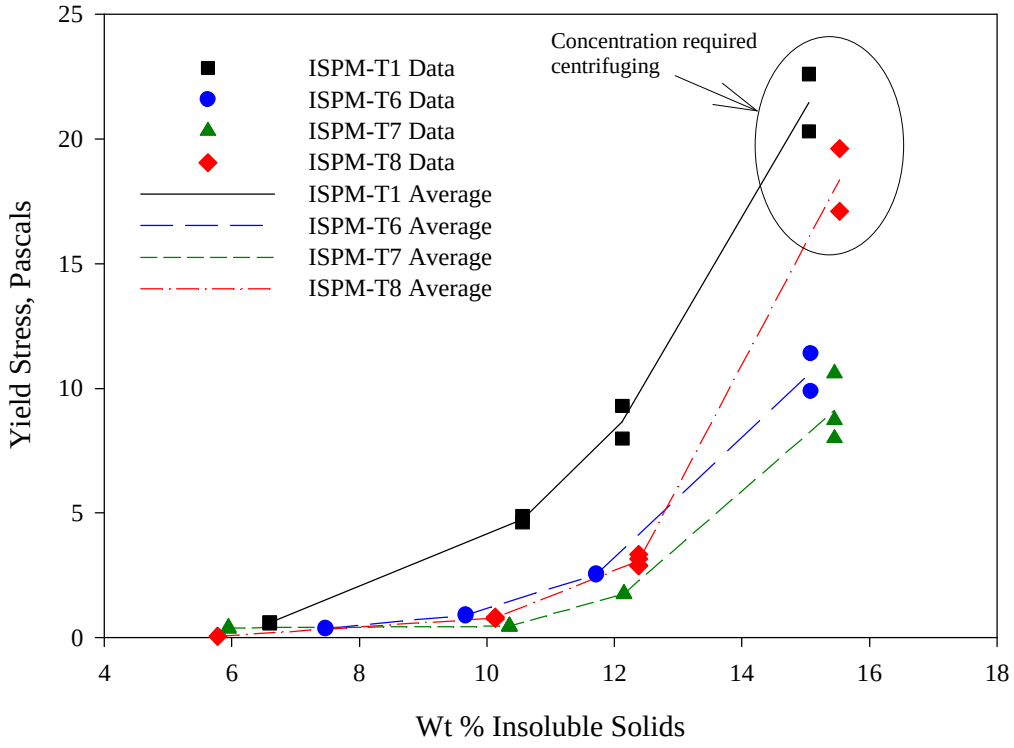


Figure 3-5: Plastic Viscosity of Sheared SRAT Product Slurries

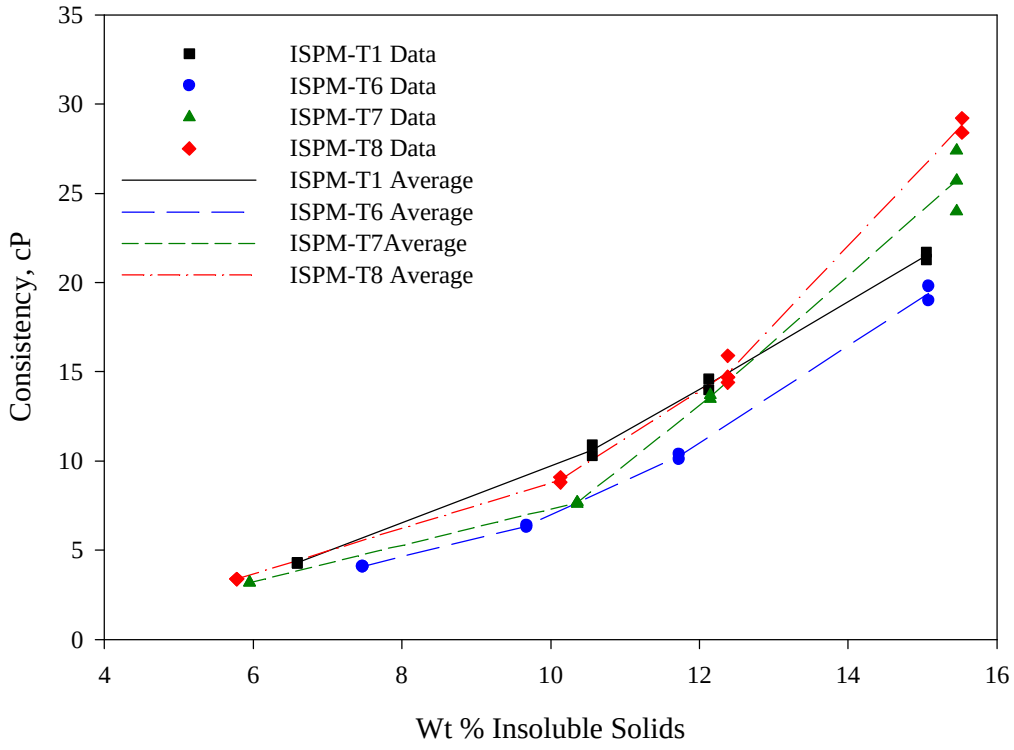
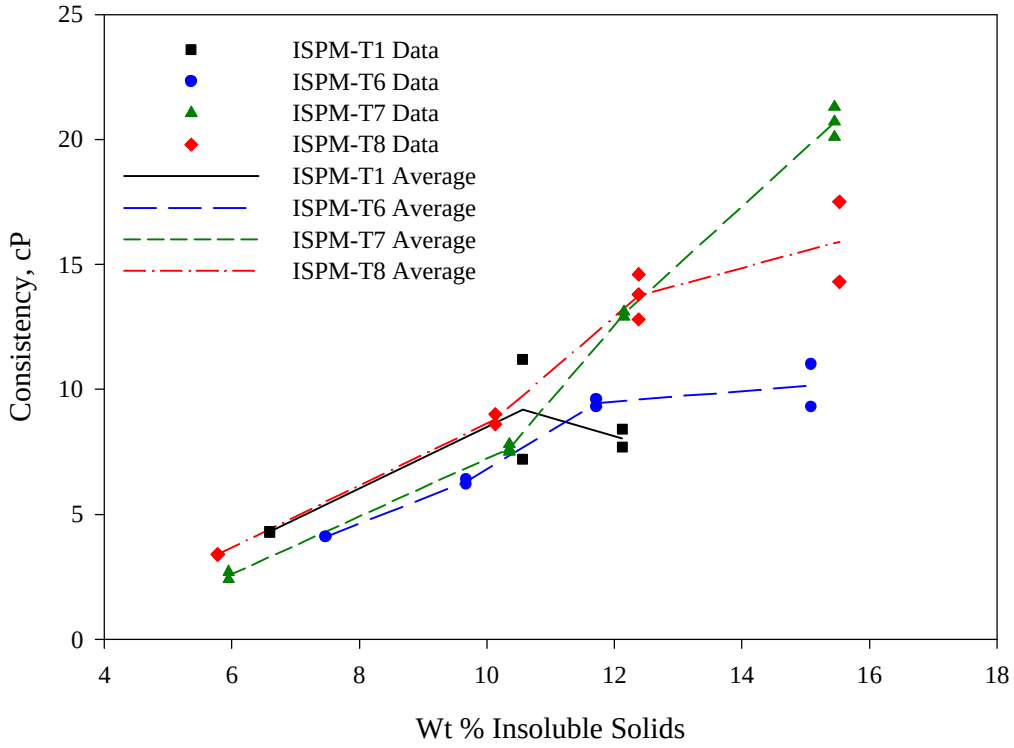


Figure 3-6: Plastic Viscosity for Unsheared SRAT Slurries



The rapid increase in yield stress observed in the SRAT product samples is typical of the rheological response of slurries to the increase of solids within those slurries.[11] This rapid rise can be expressed as an exponential function of the form:[12],[13]

$$Y = \frac{e^{AX}}{(1 - X/B)} \qquad (6)$$

where A and B are independent parameters, Y is the viscosity and X is the volume fraction of the solids in the slurry. Parameter B represents the limiting amount of solids that yields a solid material instead of a fluid. Equation (6) has been extended successfully to yield stress and consistency in previous rheology studies of DPWF simulants.[14],[15] Instead of the solids volume fraction, X would be the weight % insoluble solids. Note that the yield stress and consistency could also be fitted to similar equations expressed in terms of weight % total solids. Application of this equation does not adequately describe the very low solids concentration region since at no solids the aqueous liquid would be expected to not have a yield stress. Therefore, the model equation was modified (eguation 7) by adding a third parameter, C (units in Pascals), which allows a closer match to the expected low solids rheology regime.

$$Y = \frac{e^{AX}}{(1 - X/B)} - C \qquad (7)$$

An alternative approach was considered that replaced the variable C with a constant whose value would be one Pascal. This is analogous to forcing a fit through the origin for a linear function. Using either form produced similar results so all of the data was modeled using equation (7). The curve fitted equation is only applicable in the range in which the data was obtained. Using the nonlinear equation (7) as the model, the data in Table 3-9 and Table 3-10 (along with the assumption that at zero wt % insoluble solids the yield stress would be zero Pa) was fit to the model using TableCurve® 2D software. The resulting curves are shown in Figure 3-7 and Figure 3-8. The parameters for equation 7 and the quality of the fit are listed in Table 3-11 and Table 3-12. An examination of the B parameter for the ISPM-T6, ISPM-T7, and ISPM-T8 sludges reveals that all three are very similar, suggesting that the maximum insoluble solids parameter is about 17 wt %. There does seem to be a difference based upon the yield stress results for the ISPM-T1 SRAT product. In general, the different methods of sludge production did not necessarily lead to different SRAT products based upon their rheological properties.

Figure 3-7: Rheology Model Equations for the Sheared Yield Stress Results

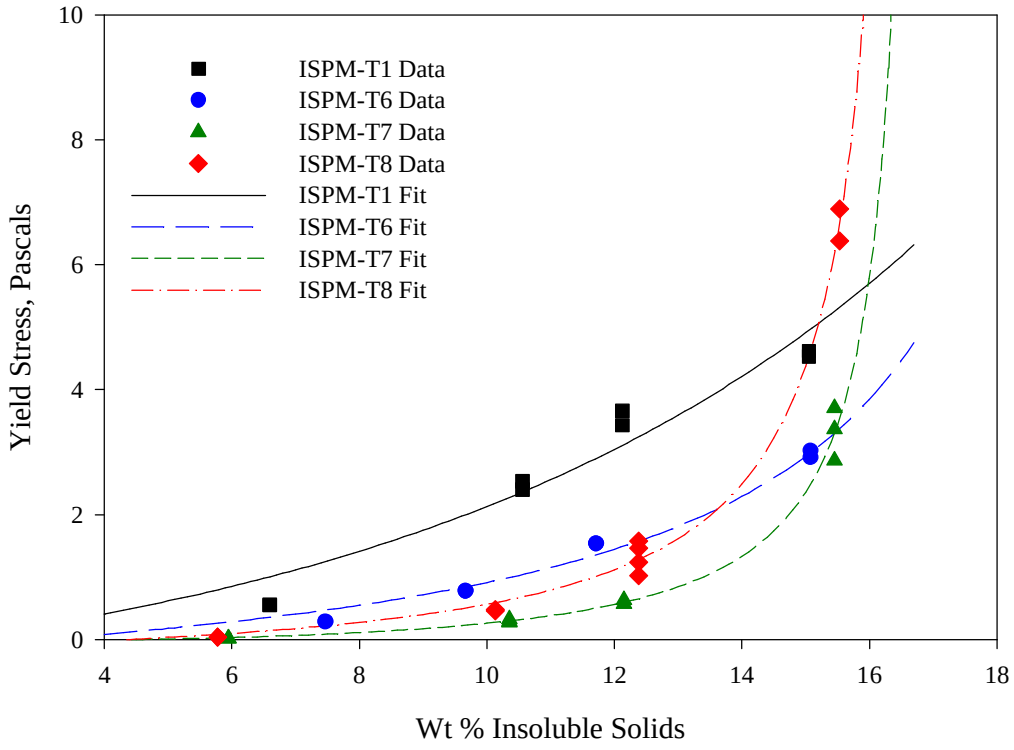


Table 3-11: Rheology Model Parameters for Sheared SRAT Products

SRAT Product	Factor A	Factor B	Factor C, Pa	Fit (R^2)
ISPM-T1	0.11	99.99	1.21	0.95
ISPM-T6	0.0093	20.78	1.2	0.98
ISPM-T7	-0.066	16.87	1.01	0.98
ISPM-T8	-0.037	16.74	1.15	0.99

Figure 3-8: Rheology Model Equations for the Unsheared Yield Stress Results

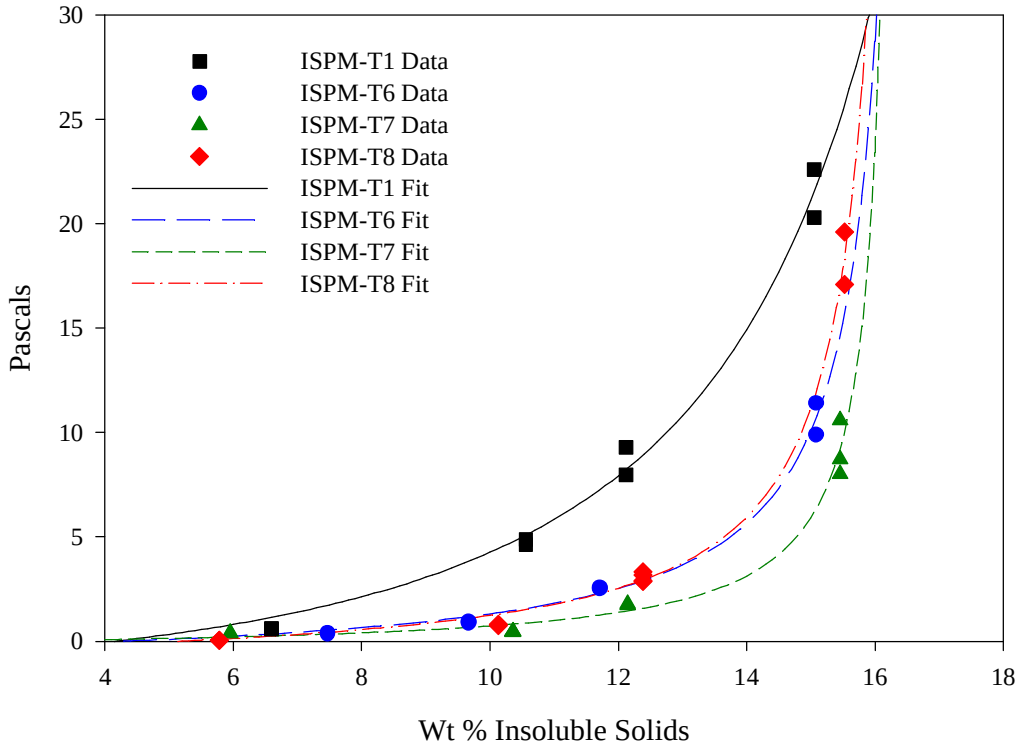
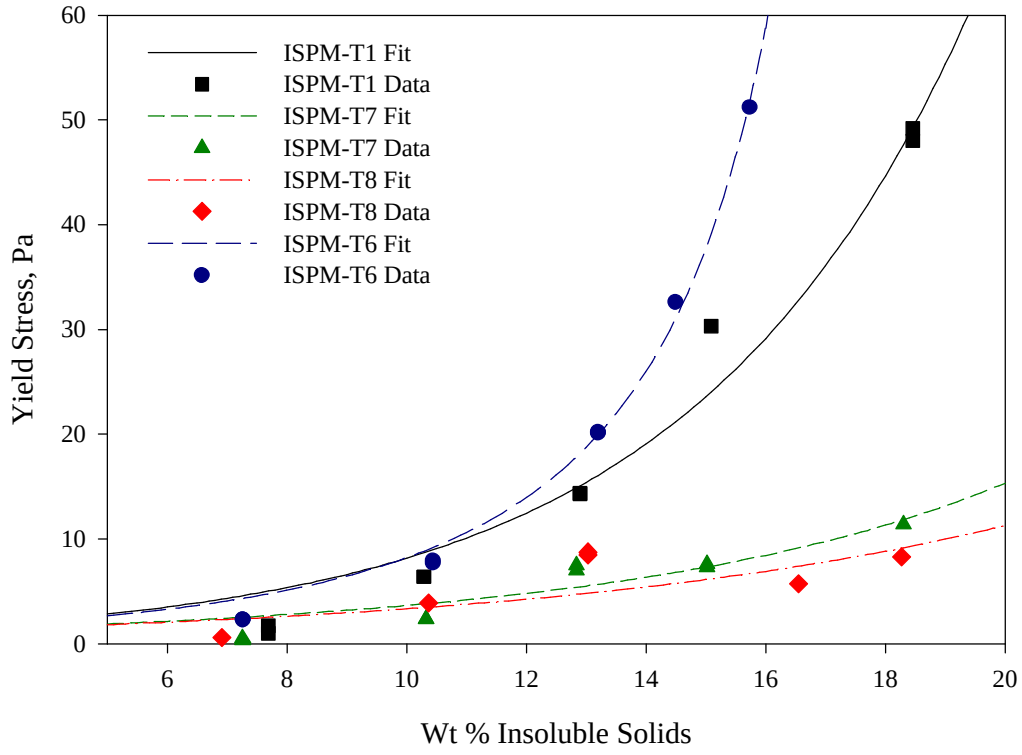


Table 3-12: Rheology Model Parameters for Unsheared SRAT Products

SRAT Product	Factor A	Factor B	Factor C, Pa	Fit (R^2)
ISPM-T1	0.112	19.54	2.02	0.99
ISPM-T6	0.0073	16.62	1.39	0.99
ISPM-T7	-0.035	16.38	1.07	0.97
ISPM-T8	0.0077	16.46	1.51	0.99

Before testing began, it was hypothesized that the SRAT process would eliminate the physical property disparities on the differently prepared test sludges due to the chemistry of the SRAT process. If this had occurred, all of the products would have similar rheological properties. The rheology of the sheared sludge feeds to the SRAT runs as a function of insoluble solids is shown in Figure 3-9.[2] Before the SRAT run, the Test 6 and the Test 1 sludge were thicker than the Test 7 and 8 sludges, which were similar. A comparison of Figure 3-9 and Figure 3-7 reveals that while all of the sludges became more fluid for a given wt % insoluble solids, not all of the feeds were modified to produce a similar product based on rheology. The ISPM-T6 sludge was altered the most while the ISPM-1 sludge remained thicker. Therefore, the conclusion from this study is that the SRAT does not eliminate physical property differences between sludges prepared by different methods.

Figure 3-9: Yield Stress Curves for the Starting SRAT Feed



Note that similar model equations can be produced using equation 7 by applying the total solids values instead of insoluble solids. The plastic viscosity (consistency) results did not fit as well to equation 7, since the data was more nearly linear with respect to insoluble or total solids loading. The results of the consistency fits will not be given in this report.

3.5 SRAT Product Particle Size

SRAT product samples were analyzed for particle size after dilution with water. Table 3-13 and Figure 3-10 summarize the data from this analysis. The particle size distribution of ISPM-T6, ISPM-T7 and ISPM-T8 SRAT Products were very similar with a major peak at approximately 25 • m and a smaller peak at 8 • m. The ISPM-T1 SRAT Product has a single peak at approximately 10 • m. ISPM-T1 also appeared to be the most viscous. ISPM-T1 SRAT product had the smallest average particle size by volume while ISPM-T6 SRAT product had the largest particle size by volume. ISPM-T1 SRAT product had the smallest average particle size by number while ISPM-T8 SRAT product had the largest particle size by number.

SRAT processing had minimal impact on the ISPM-T8 Sludge particle size and particle distribution as can be seen in Figure 3-11. The ISPM-T8 Sludge had two peaks both before and after processing. The heat treatment and coprecipitation produced insoluble solids that were stable throughout the SRAT process. In contrast, ISPM-T1 sludge had a significant change in particle size as the result of the SRAT processing with the large particles becoming significantly smaller and the smallest particles becoming larger. The SRAT product had a single broad peak at approximately 10 • m while the starting sludge had two peaks. Also, the distribution of the two peaks was very different from the two peaks seen in ISPM-T6, ISPM-T7 and ISPM-T8.

This data should be compared with actual sludge particle size distribution when available. In addition, testing of the particle size should be repeated using supernate, not water to dilute the sample as the large change in ionic strength due to the dilution by water may have dissolved some samples or broken up weak agglomerates into smaller particles.

Table 3-13: Particle Size of Sludge and SRAT Products

Analysis	ISPM-T1	ISPM-T6	ISPM-T7	ISPM-T8
Sludge Mean Particle Size, Volume Basis, • m	33.8	22.4	19.7	20.1
SRAT Product Mean Particle Size, Volume Basis, • m	15.78	29.12	19.18	18.81
Sludge Mean Particle Size, Number Basis, • m	0.75	1.7	2	2.1
SRAT Product Mean Particle Size, Number Basis, • m	1.191	1.294	1.52	3.052
Sludge Mean Particle Size, Area Basis, • m	10.4	9	9.1	8.8
SRAT Product Mean Particle Size, Area Basis, • m	6.484	10.75	9.382	10.47

Note: Measured on a single sample diluted with water.

Figure 3-10: Particle Size, volume basis, for All Runs

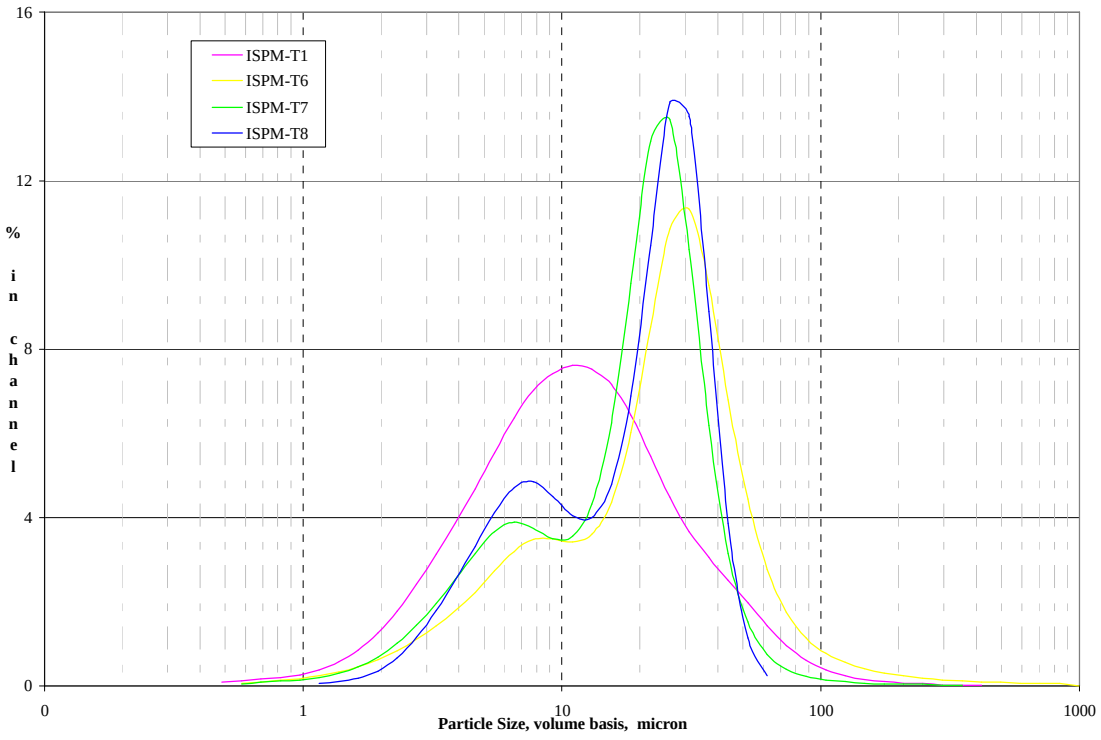
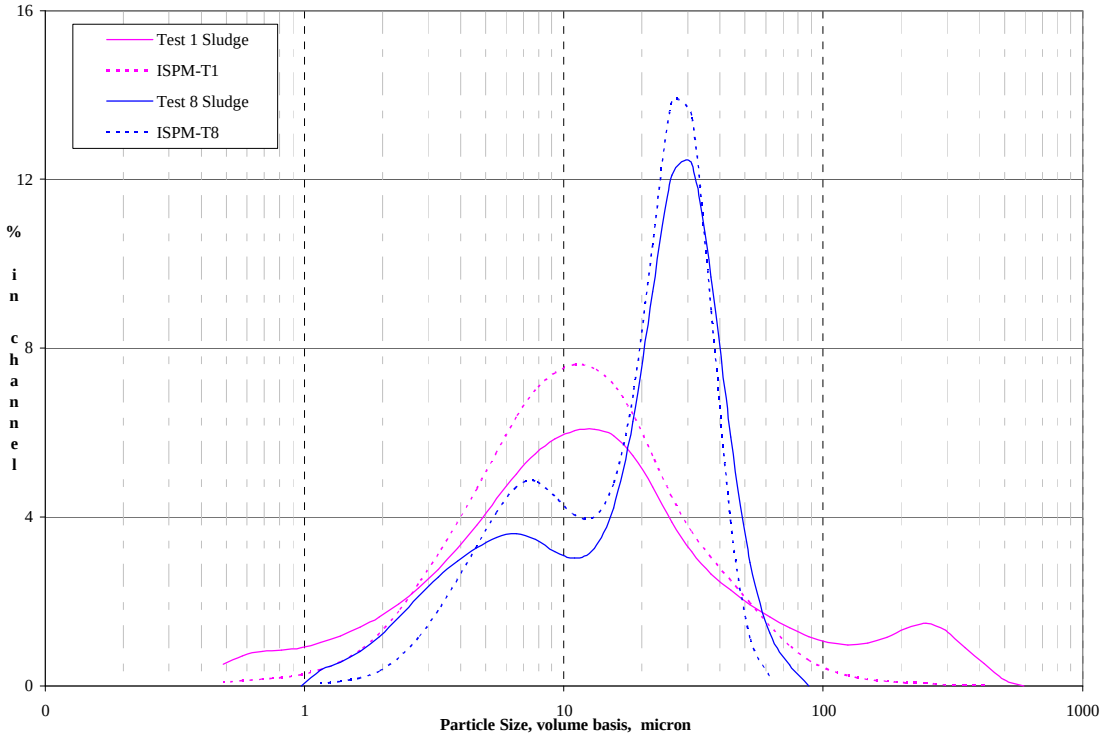


Figure 3-11: Particle Size, volume basis, for ISPM-T1 and ISPM-T8



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4.0 CONCLUSIONS

Four SRAT cycles were completed to determine the impact of the sludge preparation method on SRAT processing, SRAT product chemistry, rheology and particle size. The four sludge batches were processed through SRAT cycles, and the resulting SRAT product was analyzed to determine the impact of SRAT processing on chemical and physical properties. The following conclusions result from this study:

- The chemistry of the SRAT process does not mitigate the differences in rheology and particle size distribution between differently prepared sludges of the same nominal composition. Before testing began, it was hypothesized that the impact of the SRAT process on the differently prepared test sludges would be the elimination of physical property differences due to the chemistry of the SRAT process. If this had occurred, all of the products would have similar rheological properties. Instead, the conclusion from this study is that the SRAT does not eliminate physical property differences between sludges prepared by different methods.
- The chemical composition of the four starting sludge simulants and the four resulting SRAT products were very similar as desired and/or expected.
- The rheological and particle size distribution properties of the SRAT products from Tests 6, 7, and 8 starting sludges were very similar, but clearly different from those of the baseline simulant used in Test 1.
- The pH profile and resulting final pH were very similar. As expected, the minimum SRAT pH occurred at end of acid addition. The measured minimum pH ranged from 4.04-4.14. All runs had a SRAT product pH in the range of pH 4.62 - 4.94.
- The formate destruction was similar, the destruction efficiency varied from 11% to 22%.
- The more concentrated samples had the highest yield stress values. The average yield stress for the sheared SRAT product at 15 wt% insoluble solids ranged from 2.96-6.64 Pa or 29.6-66.4 dynes/cm^2. The highest yield stress was for the 15 wt % SRAT product prepared from Test 8 sludge.
- The plastic viscosity for the sheared SRAT product at 15 wt% insoluble solids ranged from 19.4-28.8 cp. The highest plastic viscosity was for the 15 wt % SRAT product prepared from Test 8 sludge.
- The yield stress of each of the four SRAT products was within the operating window for DWPF rheology. The sharp increase in yield stress of the 15 wt % insoluble solids ISPM-T8 SRAT product suggests that simulant processing above 15 wt % insoluble solids should be avoided.
- The particle size distributions of the sludges and SRAT products from the three new sludge preparation methods were very similar before and after the SRAT cycles. The particle size distribution of the baseline sludge changed considerably during processing with fewer small and large particles in the SRAT product.
- ISPM-T1 SRAT product had the smallest average particle size by volume and number. ISPM-T6, ISPM-T7, and ISPM-T8 are nearly identical in volume mean particle size.
- SRAT processing had minimal impact on the ISPM-T8 Sludge particle size and particle distribution. The ISPM-T8 Sludge had two peaks both before and after processing. The heat treatment and coprecipitation produced insoluble solids that were stable throughout the SRAT process. In contrast, Test 1 sludge had a significant change in particle size as the result of the SRAT processing with the large particles becoming significantly smaller and the smallest particles becoming larger. The SRAT product had a single broad peak at approximately 10 • m while the starting sludge had two peaks. Also, the distribution of the two peaks was very different from the two peaks seen in ISPM-T6, ISPM-T7, and ISPM-T8.
- The chemistry of the SRAT process does not mitigate the differences between differently prepared sludges of the same nominal composition.

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5.0 RECOMMENDATIONS

- The particle size data should be compared with actual sludge particle size distribution when available.
- Testing of the particle size should be repeated using supernate, not water to dilute the sample as the large change in ionic strength due to the dilution by water may have dissolved some samples or broken up weak agglomerates into smaller particles.
- Research on the processes that control the physical properties of simulants should continue and include those that are impacted by the scale of the production method and by the chemical composition of the sludge.
- When new simulants or new methods of producing simulants are developed, these simulants should be tested for their impact on the physical properties of the SRAT product.

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APPENDIX A. SRAT RUN PARAMETERS

Table A - 1: Sludge Batch 3 Compositional Basis[3]

Basis	Measured Solids	Measured Slurry	Slurry	Concentration
Analyte	microgram/gram	microgram/gram	g/L slurry	moles/Liter
Ag	300		0.0714	6.62E-04
Al	60400		14.3752	5.33E-01
B	100		0.0238	2.20E-03
Ba	500		0.1190	8.67E-04
$C_2O_4^{-2}$		1033	1.2293	1.40E-02
Ca	16600		3.9508	9.86E-02
Cd	1900		0.4522	4.02E-03
Ce	1100		0.2618	1.87E-03
Cl		200	0.2380	6.71E-03
Cr(TOTAL)	2500		0.5950	1.14E-02
Cu	300		0.0714	1.12E-03
F	235	235	0.2797	1.47E-02
Fe	194400		46.2672	8.28E-01
Gd	500		0.1190	7.57E-04
K	3300		0.7854	2.01E-02
La	400		0.0952	6.85E-04
Li	400		0.0952	1.37E-02
Mg	16700		3.9746	1.64E-01
Mn	39500		9.4010	1.71E-01
Mo	500		0.1190	1.24E-03
Na	120600		28.7028	1.25E+00
Ni	10900		2.5942	4.42E-02
NO_2^-		15462	18.3998	4.00E-01
NO_3^-		10536	12.5378	2.02E-01
OH^-			7.3132	4.30E-01
P (by ICP-ES)	4300		1.0234	3.30E-02
Pb	600		0.1428	6.89E-04
PO_4^{-3} (by IC)		940	1.1186	1.18E-02
S	2900		0.6902	2.15E-02
Sb	600		0.1428	1.17E-03
Si	4000		0.9520	3.39E-02
Sn	500		0.1190	1.00E-03
SO_4^{-2}		1689	2.0099	2.09E-02
Sr	4200		0.9996	1.14E-02
Ti	200		0.0476	9.94E-04
U	69900		16.6362	6.99E-02
V	100		0.0238	4.67E-04
Zn	300		0.0714	1.09E-03
Zr	100		0.0238	2.61E-04

Table A - 2: SRAT Run Parameters

Parameter	ISPM-T1	ISPM-T6	ISPM-T7	ISPM-T8
Initial Sludge Mass (g)	785.4	785.4	785.4	785.4
Hg Target (wt% in total solids)	0	0	0	0
HgO Added (g)	0	0	0	0
$AgNO_3$ Added (g)	0	0	0	0
$Pd(NO_3)_2$*H_2O Added (g) – 15.27% Solution	0.0109	0.0113	0.0117	0.0111
$Rh(NO_3)_3$*$2H_2O$ Added (g) – 4.93% Solution	0.1863	0.1865	0.1877	0.1865
$RuCl_3$ Added (d)	0.1043	0.1042	0.1059	0.1045
Rinse Water for Trim Chemicals (g)	40	40	44.99	40
DWPF SRAT Scale Factor (6,000 gallon basis)	1:32,400	1:32,400	1:32,400	1:32,400
Acid Stoichiometry	155%	155%	155%	155%
Nitric Acid Amount Added (ml)	22.894	22.526	22.618	22.618
Nitric Acid Addition Rate (ml/min)	0.241	0.240	0.241	0.228
Nitric Acid Moles	0.241	0.237	0.238	0.238
Formic Acid Amount Added (ml)	60.901	60.360	60.461	61.297
Formic Acid Addition Rate (ml/min)	0.249	0.248	0.240	0.248
Formic Acid Moles	1.442	1.420	1.424	1.456
Total SRAT Dewater Amount (g)	175.500	188.580	182.300	183.690
Dewater Amount after Reflux (g)	55.35	73.80	61.50	54.90
Concentration/Dewater Time after Boiling (hrs)	0.75	1.00	0.83	0.75
SRAT Target Boil-up Rate (g/min)	1.23	1.23	1.23	1.22
SRAT Air Purge on System (sccm)	211.3	211.3	211.3	211.3
SRAT Helium Purge on System (sccm)	0	0	0	0
Initial Sludge pH with Trim Chemicals	12.09	11.96	11.83	12.10
Minimum pH during SRAT	4.08	4.04	4.14	4.09
pH at End of SRAT (at boiling)	4.94	4.81	4.71	4.62
Antifoam Addition (g)	5.81	5.81	5.81	5.81
Run Plan Document Number	SRNL-ITS-2005-00080	SRNL-ITS-2005-00089	SRNL-ITS-2005-00090	SRNL-ITS-2005-00087

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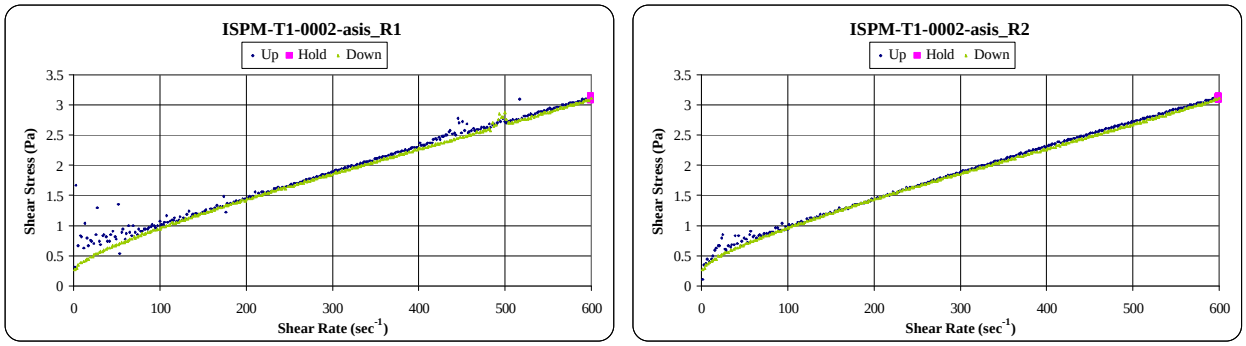
APPENDIX B. RHEOLOGY DATA

All of the rheogram measurements in Appendix B were made using the 60 mm/ 2 degree Ti cone and plate.

Figure B - 1: Rheology of ISPM-T1-asis SRAT Product



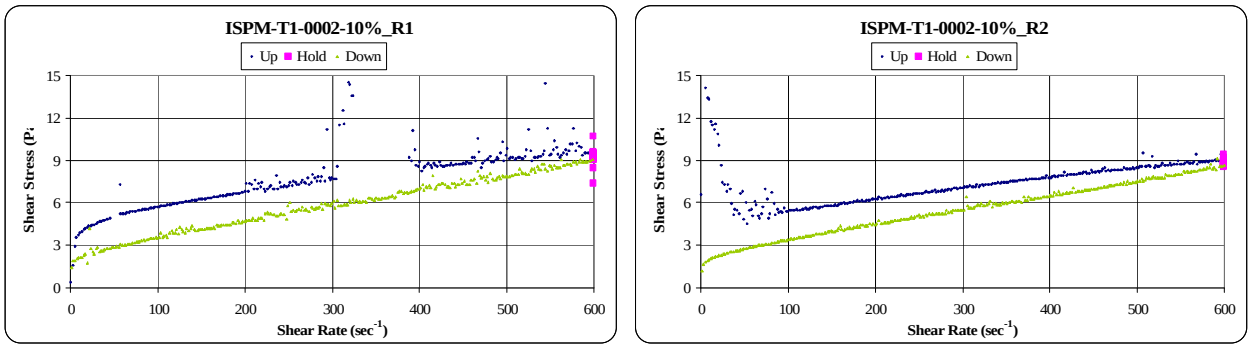
Unsheared Results (Up Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T1-0002-asis_R1	4.25	0.61	0.9932	4/27/2005	50-600
ISPM-T1-0002-asis_R2	4.30	0.58	0.9990	4/27/2005	50-600
Average	4.28	0.60			
Stdev	0.04	0.02			
%Stdev	0.8%	3.6%			

Sheared Flow Curve Results (Down Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T1-0002-asis_R1	4.28	0.55	0.9980	4/27/2005	50-600
ISPM-T1-0002-asis_R2	4.26	0.56	0.9988	4/27/2005	50-600
Average	4.27	0.56			
Stdev	0.01	0.01			
%Stdev	0.3%	1.3%			

Figure B - 2: Rheology of ISPM-T1-10% SRAT Product



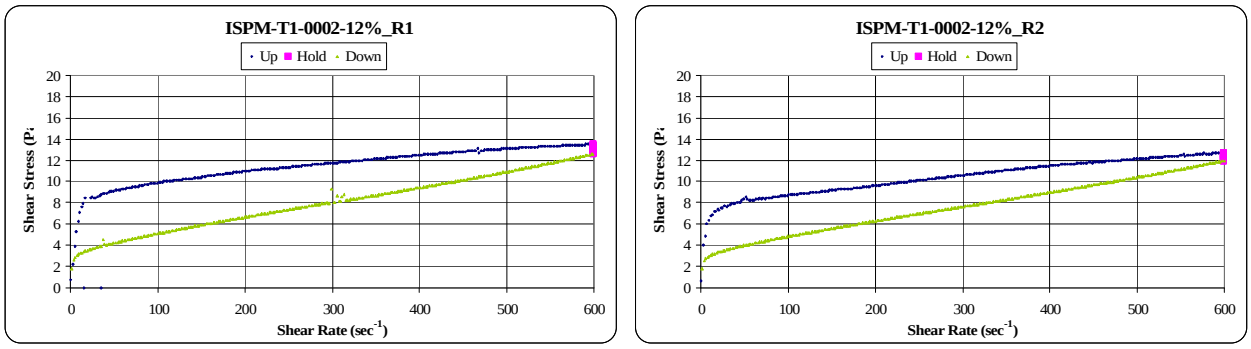
Unsheared Results (Up Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T1-0002-10%_R1	11.19	4.63	0.8057	4/28/2005	50-300
ISPM-T1-0002-10%_R2	7.25	4.87	0.9675	4/28/2005	50-600
Average	9.22	4.75			
Stdev	2.8	0.17			
%Stdev	30.2%	3.6%			

Sheared Results (Down Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T1-0002-10%_R1	10.91	2.53	0.9912	4/28/2005	50-600
ISPM-T1-0002-10%_R2	10.34	2.40	0.9962	4/28/2005	50-600
Average	10.63	2.47			
Stdev	0.4	0.09			
%Stdev	3.8%	3.7%			

Figure B - 3: Rheology of ISPM-T1-12% SRAT Product



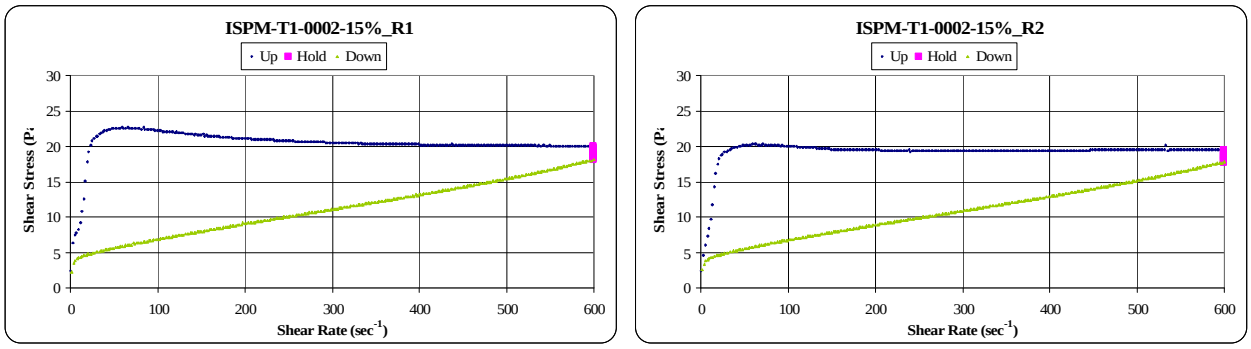
Unsheared Results (Up Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T1-0002-12%_R1	7.67	9.30	0.9799	5/3/2005	50-600
ISPM-T1-0002-12%_R2	8.39	7.98	0.9916	5/3/2005	50-600
Average	8.03	8.64			
Stdev	0.51	0.93			
%Stdev	6.3%	10.8%			

Sheared Results (Down Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T1-0002-12%_R1	14.62	3.66	0.9976	5/3/2005	50-600
ISPM-T1-0002-12%_R2	14.01	3.43	0.9996	5/3/2005	50-600
Average	14.32	3.55			
Stdev	0.43	0.16			
%Stdev	3.0%	4.6%			

Figure B - 4: Rheology of ISPM-T1-15% SRAT Product



Unsheared Results (Up Flow Curve)

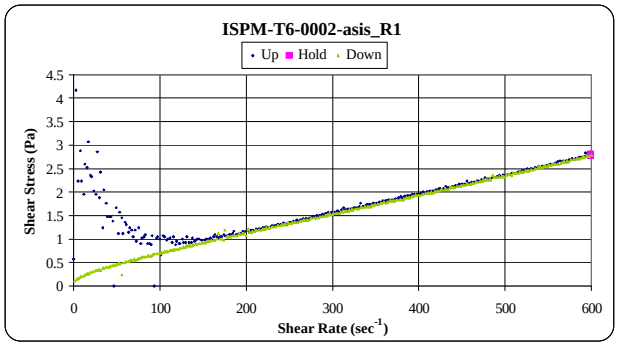
Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T1-0002-15%_R1	NA	22.64*	NA	5/4/2005	NA
ISPM-T1-0002-15%_R2	NA	20.29*	NA	5/4/2005	NA
Average	NA	21.47			
Stdev	NA	1.66			
%Stdev	NA	7.7%			

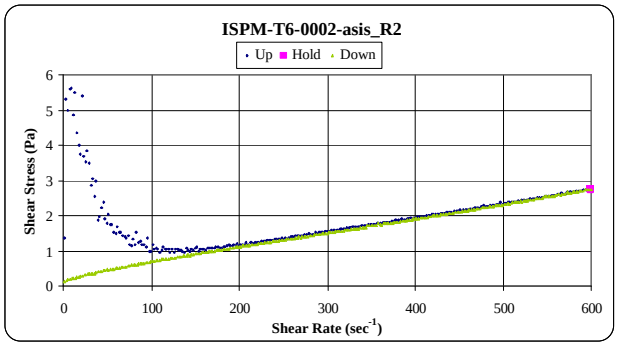
*Yield Stress was taken as the maximum in the curve between 0 and 100 sec^{-1} since the curve did not properly reflect a Bingham material before shearing. Plastic viscosity could not be determined for the same reason.

Sheared Results (Down Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T1-0002-15%_R1	21.70	4.62	0.9986	5/4/2005	50-600
ISPM-T1-0002-15%_R2	21.34	4.53	0.9986	5/4/2005	50-600
Average	21.52	4.58			
Stdev	0.25	0.06			
%Stdev	1.2%	1.4%			

Figure B - 5: Rheology of ISPM-T6-asis SRAT Product





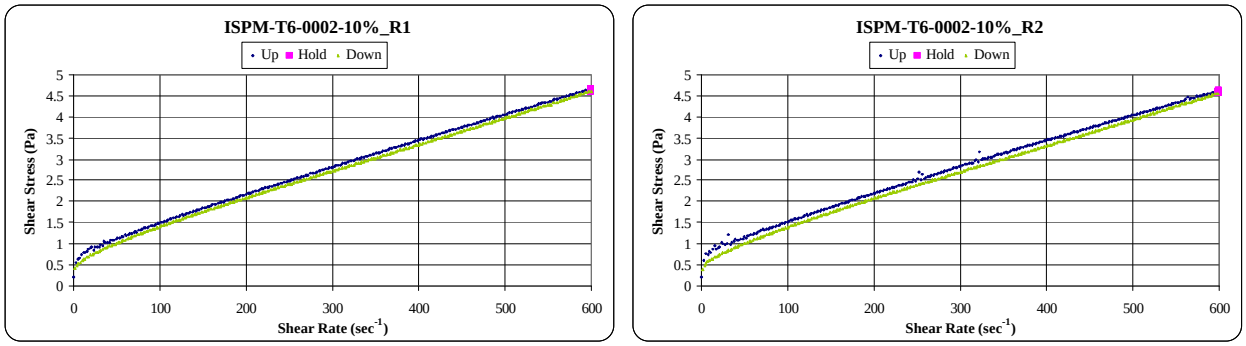
Unsheared Results (Up Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R2	Date	Range, sec^{-1}
ISPM-T6-0002-asis_R1	4.05	0.35	0.9990	4/27/2005	150-600
ISPM-T6-0002-asis_R2	3.90	0.39	0.9988	4/27/2005	150-600
Average	3.98	0.37			
Stdev	0.11	0.03			
%Stdev	2.7%	7.6%			

Sheared Results (Down Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R2	Date	Range, sec^{-1}
ISPM-T6-0002-asis_R1	4.13	0.29	0.9984	4/27/2005	50-600
ISPM-T6-0002-asis_R2	4.06	0.28	0.9996	4/27/2005	50-600
Average	4.10	0.29			
Stdev	0.05	0.01			
%Stdev	1.2%	2.5%			

Figure B - 6: Rheology of ISPM-T6-10% SRAT Product



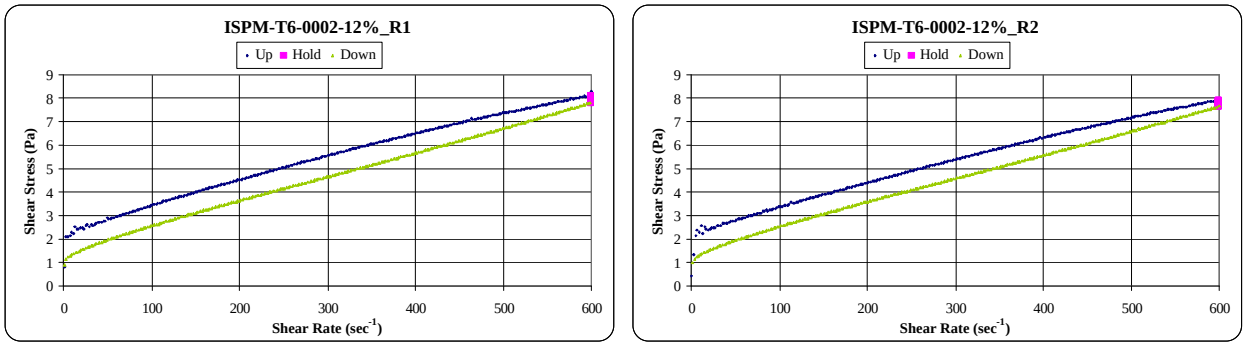
Unsheared Results (Up Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T6-0002-10%_R1	6.37	0.88	0.9994	4/27/2005	50-600
ISPM-T6-0002-10%_R2	6.24	0.93	0.9988	4/27/2005	50-600
Average	6.31	0.91			
Stdev	0.09	0.04			
%Stdev	1.5%	3.9%			

Sheared Results (Down Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T6-0002-10%_R1	6.40	0.78	0.9996	4/27/2005	50-600
ISPM-T6-0002-10%_R2	6.33	0.78	0.9994	4/27/2005	50-600
Average	6.37	0.78			
Stdev	0.05	0.00			
%Stdev	0.8%	0.0%			

Figure B - 7: Rheology of ISPM-T6-12% SRAT Product



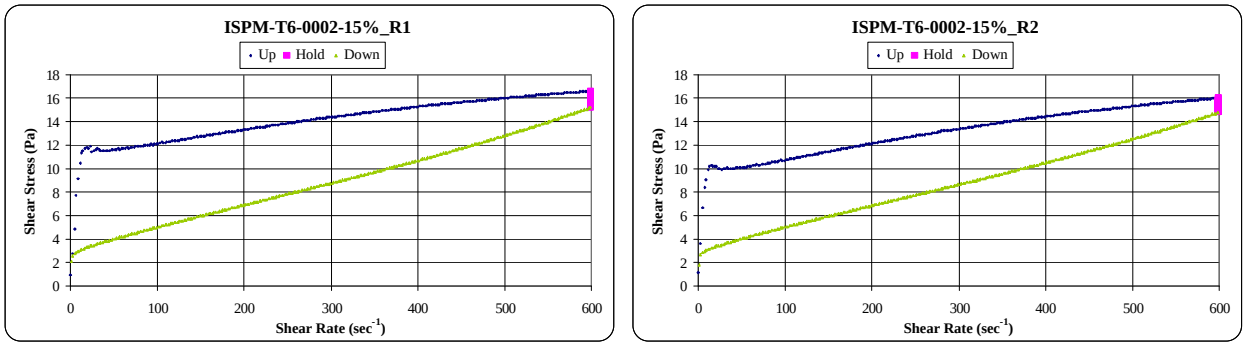
Unsheared Results (Up Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T6-0002-12%_R1	9.61	2.57	0.9960	4/27/2005	50-600
ISPM-T6-0002-12%_R2	9.34	2.51	0.9972	4/27/2005	50-600
Average	9.48	2.54			
Stdev	0.19	0.04			
%Stdev	2.0%	1.7%			

Sheared Results (Down Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T6-0002-12%_R1	10.39	1.53	0.9998	4/27/2005	50-600
ISPM-T6-0002-12%_R2	10.14	1.53	0.9998	4/27/2005	50-600
Average	10.27	1.53			
Stdev	0.18	0.00			
%Stdev	1.7%	0.0%			

Figure B - 8: Rheology of ISPM-T6-15% SRAT Product



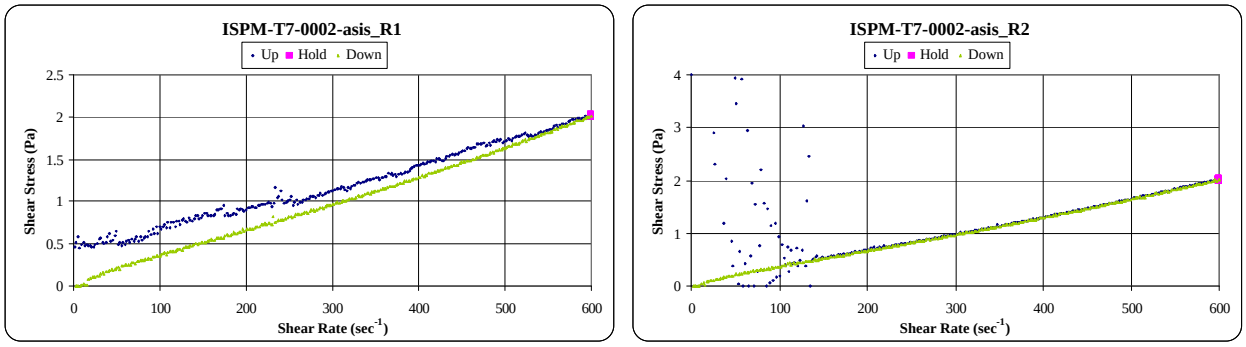
Unsheared Results (Up Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T6-0002-15%_R1	9.28	11.40	0.9874	5/3/2005	50-600
ISPM-T6-0002-15%_R2	10.95	9.88	0.9878	5/3/2005	50-600
Average	10.12	10.64			
Stdev	1.18	1.07			
%Stdev	11.7%	10.1%			

Sheared Results (Down Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T6-0002-15%_R1	19.83	2.91	0.9982	5/3/2005	50-600
ISPM-T6-0002-15%_R2	19.04	3.02	0.9988	5/3/2005	50-600
Average	19.44	2.97			
Stdev	0.56	0.08			
%Stdev	2.9%	2.6%			

Figure B - 9: Rheology of ISPM-T7-asis SRAT Product



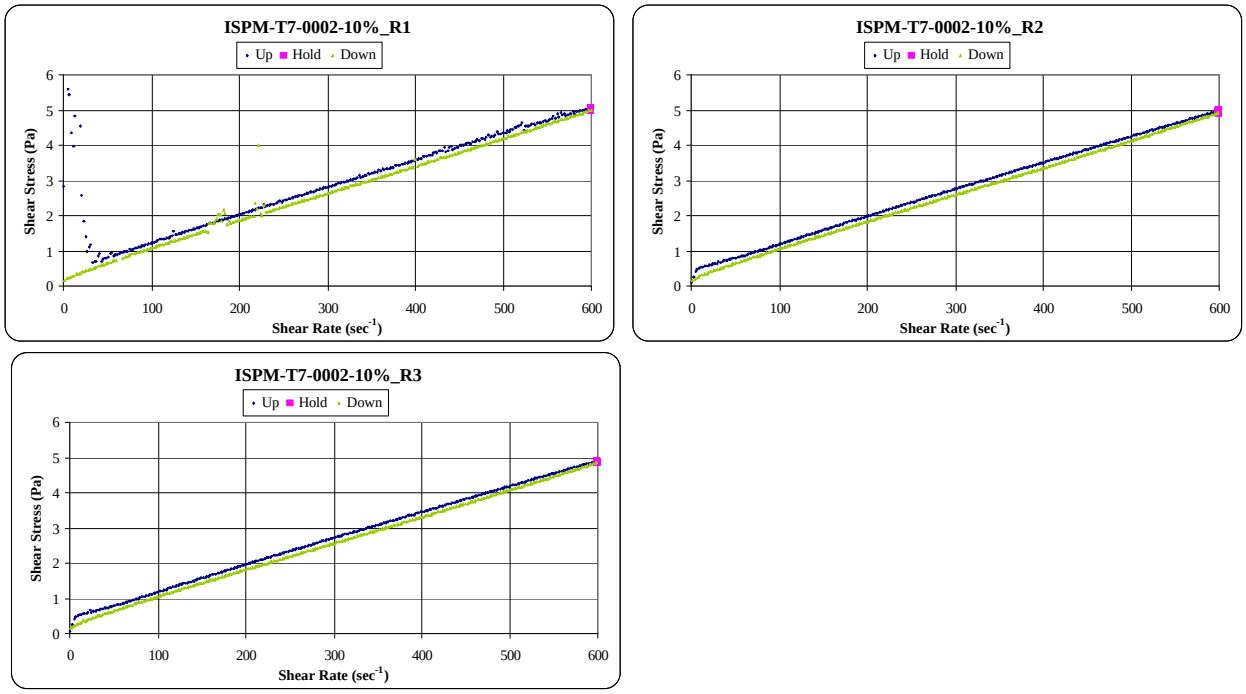
Unsheared Results (Up Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T7-0002-asis_R1	2.67	0.37	0.9900	4/27/2005	50-600
ISPM-T7-0002-asis_R2	2.36	0.41	0.4306	4/27/2005	50-600
Average	2.52	0.39			
Stdev	0.22	0.03			
%Stdev	8.7%	7.3%			

Sheared Results (Down Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T7-0002-asis_R1	3.22	0.02	0.9978	4/27/2005	50-600
ISPM-T7-0002-asis_R2	3.22	0.02	0.9980	4/27/2005	50-600
Average	3.22	0.02			
Stdev	0.00	0.00			
%Stdev	0.0%	0.0%			

Figure B - 10: Rheology of ISPM-T7-10% SRAT Product



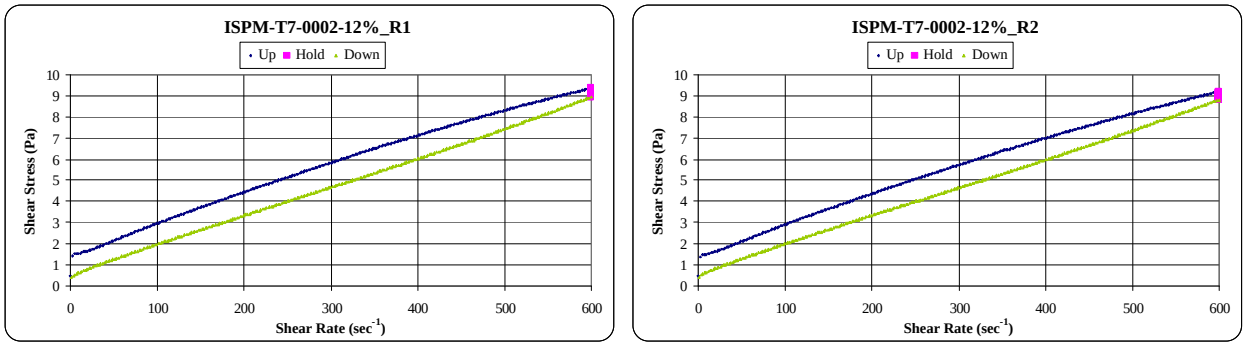
Unsheared Results (Up Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T7-0002-10%_R1	7.75	0.47	0.9994	4/27/2005	50-600
ISPM-T7-0002-10%_R2	7.61	0.45	0.9998	4/27/2005	50-600
ISPM-T7-0002-10%_R3	7.47	0.46	0.9998	4/27/2005	50-600
Average	7.61	0.46			
Stdev	0.14	0.01			
%Stdev	1.8%	2.2%			

Sheared Results (Down Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T7-0002-10%_R1	7.72	0.33	0.9883	4/27/2005	50-600
ISPM-T7-0002-10%_R2	7.70	0.28	0.9999	4/27/2005	50-600
ISPM-T7-0002-10%_R3	7.57	0.29	0.9999	4/27/2005	50-600
Average	7.64	0.29			
Stdev	0.09	0.01			
%Stdev	1.2%	2.5%			

Figure B - 11: Rheology of ISPM-T7-12% SRAT Product



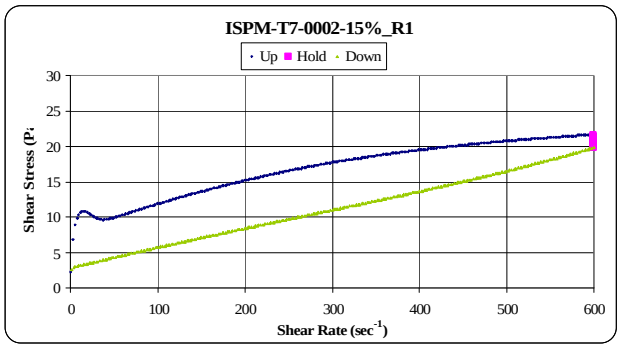
Unsheared Results (Up Flow Curve)

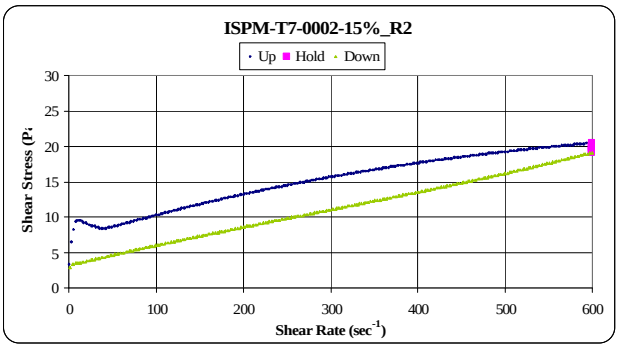
Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T7-0002-12%_R1	13.10	1.78	0.9966	4/28/2005	50-600
ISPM-T7-0002-12%_R2	12.86	1.74	0.9966	4/28/2005	50-600
Average	12.98	1.76			
Stdev	0.17	0.03			
%Stdev	1.3%	1.6%			

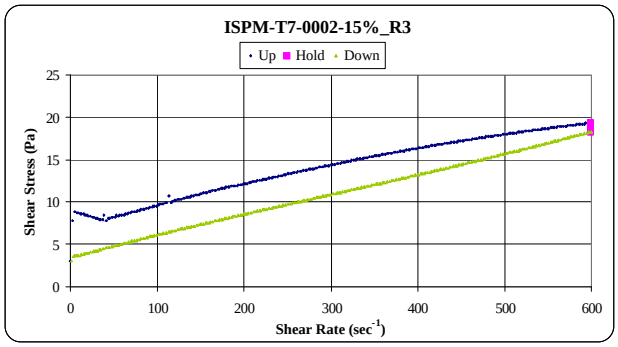
Sheared Results (Down Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T7-0002-12%_R1	13.74	0.57	0.9996	5/03/2005	50-600
ISPM-T7-0002-12%_R2	13.47	0.63	0.9998	5/03/2005	50-600
Average	13.61	0.60			
Stdev	0.19	0.04			
%Stdev	1.4%	7.1%			

Figure B - 12: Rheology of ISPM-T7-15% SRAT Product







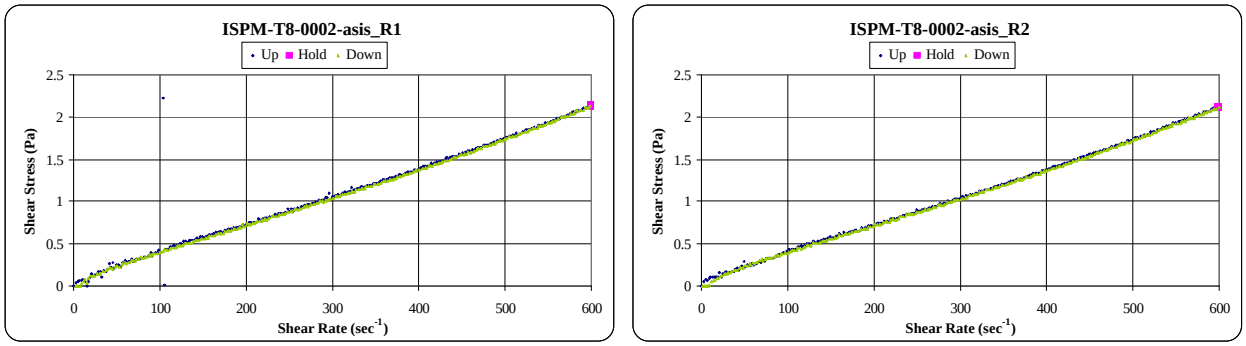
Unsheared Results (Up Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T7-0002-15%_R1	20.67	10.65	0.9528	5/3/2005	50-600
ISPM-T7-0002-15%_R2	21.30	8.73	0.9787	5/3/2005	50-600
ISPM-T7-0002-15%_R3	20.11	8.00	0.9886	5/3/2005	50-600
Average	20.69	9.13			
Stdev	0.60	1.37			
%Stdev	2.9%	15.0%			

Sheared Results (Down Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T7-0002-15%_R1	27.35	2.87	0.9988	5/3/2005	50-600
ISPM-T7-0002-15%_R2	25.68	3.37	0.9994	5/3/2005	50-600
ISPM-T7-0002-15%_R3	23.99	3.71	0.9998	5/3/2005	50-600
Average	25.67	3.32			
Stdev	1.68	0.42			
%Stdev	6.5%	12.7%			

Figure B - 13: Rheology of ISPM-T8-asis SRAT Product



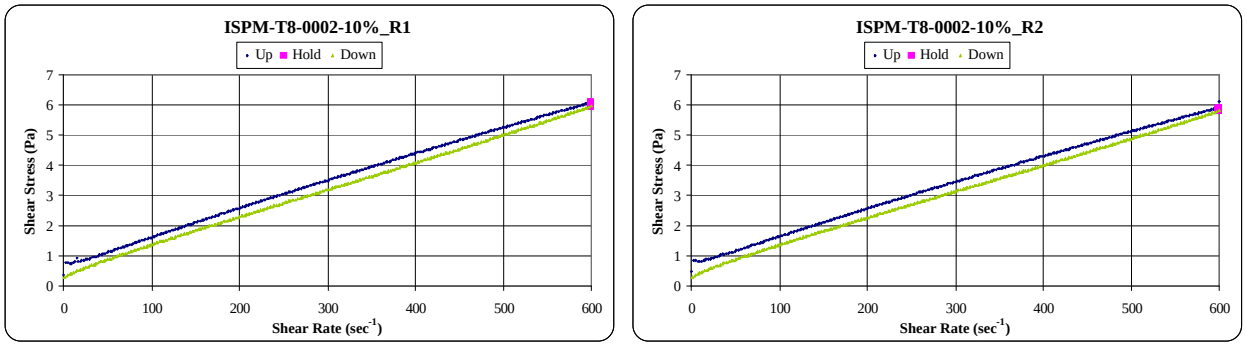
Unsheared Results (Up Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T8-0002-asis_R1	3.38	0.06	0.9994	4/27/2005	50-600
ISPM-T8-0002-asis_R2	3.37	0.05	0.9992	4/27/2005	50-600
Average	3.38	0.06			
Stdev	0.01	0.01			
%Stdev	0.2%	12.9%			

Sheared Results (Down Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T8-0002-asis_R1	3.38	0.04	0.9984	4/27/2005	50-600
ISPM-T8-0002-asis_R2	3.36	0.04	0.9984	4/27/2005	50-600
Average	3.37	0.04			
Stdev	0.01	0.0			
%Stdev	0.4%	0.0%			

Figure B - 14: Rheology of ISPM-T8-10% SRAT Product



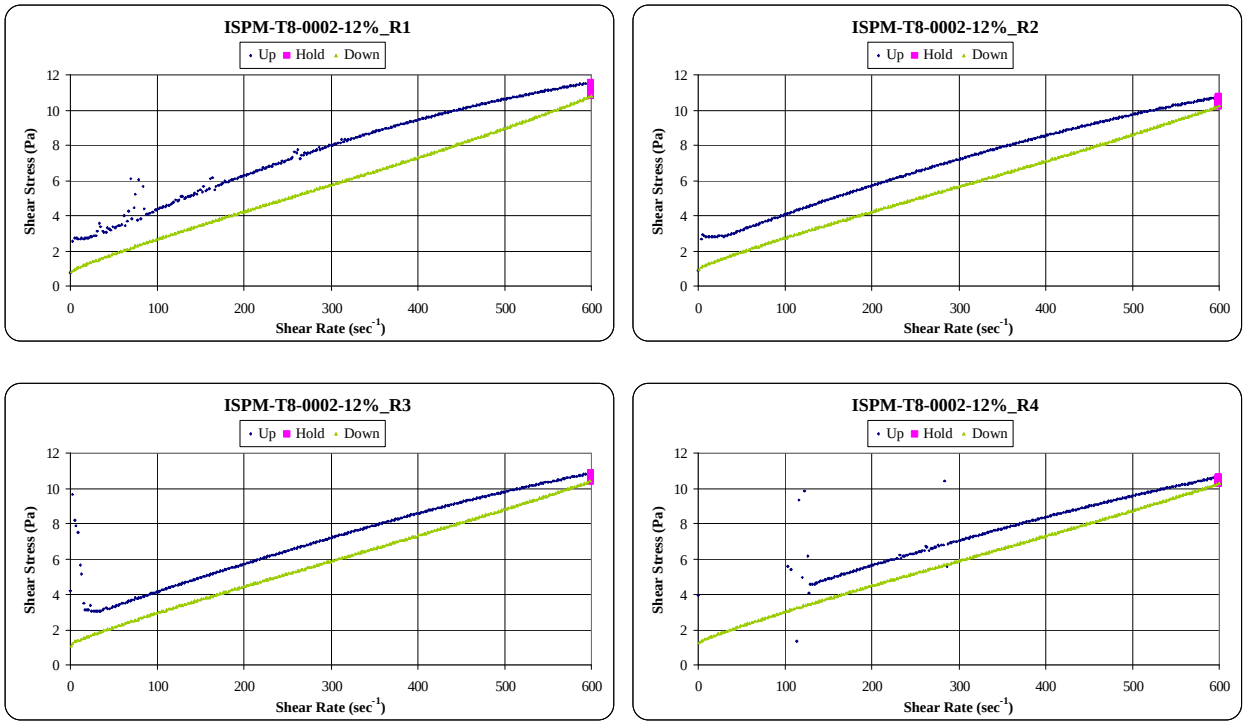
Unsheared Results (Up Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T8-0002-10%_R1	9.00	0.76	0.9992	4/28/2005	50-600
ISPM-T8-0002-10%_R2	8.63	0.82	0.9992	4/28/2005	50-600
Average	8.82	0.79			
Stdev	0.26	0.04			
%Stdev	3.0%	5.4%			

Sheared Results (Down Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T8-0002-10%_R1	9.10	0.46	1.0000	4/28/2005	50-600
ISPM-T8-0002-10%_R2	8.81	0.48	1.0000	4/28/2005	50-600
Average	8.96	0.47			
Stdev	0.21	0.01			
%Stdev	2.3%	3.0%			

Figure B - 15: Rheology of ISPM-T8-12% SRAT Product



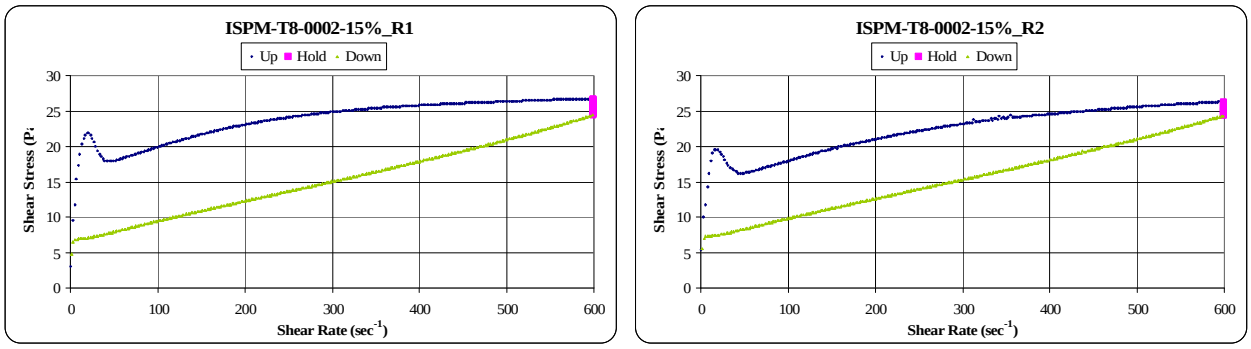
Unsheared Results (Up Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T8-0002-12%_R1	14.63	3.33	0.9803	4/28/2005	50-600
ISPM-T8-0002-12%_R2	13.78	2.88	0.9934	4/28/2005	50-600
ISPM-T8-0002-12%_R3	13.82	2.90	0.9960	5/09/2005	50-600
ISPM-T8-0002-12%_R4	12.77	3.17	0.9742	5/09/2005	150-600
Average	13.75	3.07			
Stdev	0.76	0.22			
%Stdev	5.5%	7.1%			

Sheared Results (Down Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T8-0002-12%_R1	15.90	1.02	0.9994	4/28/2005	50-600
ISPM-T8-0002-12%_R2	14.74	1.24	0.9998	4/28/2005	50-600
ISPM-T8-0002-12%_R3	14.72	1.46	0.9998	5/09/2005	50-600
ISPM-T8-0002-12%_R4	14.35	1.58	0.9998	5/09/2005	50-600
Average	14.93	1.33			
Stdev	0.67	0.25			
%Stdev	4.5%	18.7%			

Figure B - 16: Rheology of ISPM-T8-15% SRAT Product



Unsheared Results (Up Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T8-0002-15%_R1	14.27	19.57	0.8681	5/4/2005	50-600
ISPM-T8-0002-15%_R2	17.48	17.11	0.9347	5/4/2005	50-600
Average	15.88	18.34			
Stdev	2.27	1.74			
%Stdev	14.3%	9.5%			

Sheared Results (Down Flow Curve)

Sample I.D.	Plastic Viscosity (cP)	Yield Stress (Pa)	R^2	Date	Range, sec^{-1}
ISPM-T8-0002-15%_R1	29.18	6.38	0.9988	5/4/2005	50-600
ISPM-T8-0002-15%_R2	28.36	6.89	0.9992	5/4/2005	50-600
Average	28.77	6.64			
Stdev	0.58	0.36			
%Stdev	2.0%	5.4			

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Distribution:

C. J. Bannochie, 773-42A
M. J. Barnes, 999-W
D. R. Best, 786-1A
N. E. Bibler, 773-A
D. B. Burns, 786-5A
T. B. Calloway, 999-W
D. A. Crowley, 773-A
R. E. Edwards, 773-A
R. E. Eibling, 999-W
T. L. Fellinger, 704-27S
J. M. Gillam, 766-H
J. R. Harbour, 773-42A
E. K. Hansen, 999-W
C. C. Herman, 773-42A
P. J. Hill, 766-H
J. F. Iaukea, 704-30S
C. M. Jantzen, 773-A
D. C. Koopman, 773-42A
D. P. Lambert, 773-A
S. L. Marra, 999-W
D. H. Miller, 999-W
M. S. Miller, 704-S
J. E. Occhipinti, 704-S
J. M. Pareizs, 773-A
P. M. Patel, 704-27S
D. K. Peeler, 999-W
J. W. Ray, 704-S
M. A. Rios-Armstrong, 766-H
H. B. Shah, 766-H
M. E. Smith, 999-W
M. E. Stone, 999-W
W. B. Van-Pelt, 704-S
G. G. Wicks, 773-A
M. F. Williams, 999-1W