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Saltstone Studies Using the Scaled Continuous Processing Facility

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August 2015

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EXECUTIVE SUMMARY

The Savannah River National Laboratory (SRNL) has supported the Saltstone Facility since its conception with bench-scale laboratory experiments, mid-scale testing at vendor facilities, and consultations and testing at the Saltstone Facility. There have been minimal opportunities for the measurement of rheological properties of the grout slurry at the Saltstone Production Facility (SPF); thus, the Scaled Continuous Processing Facility (SCPF), constructed to provide processing data related to mixing, transfer, and other operations conducted in the SPF, is the most representative process data for determining the expected rheological properties in the SPF. These results can be used to verify the laboratory scale experiments that support the SPF using conventional mixing processes that appropriately represent the shear imparted to the slurry in the SPF.

Defense Waste Processing Facility (DWPF)/Saltstone Facility Engineering (DF-E) requested that SRNL utilize the SCPF to determine the capabilities of the Readco-Kurimoto (RK) mixer for two different studies:

- Study 1: RK mixer shaft speed study. Determine the RK mixer speed operating range for processing an equivalent 25, 35, and 45 tons/hr premix using a salt solution simulant. 35 tons/hr is the nominal case.
- Study 2: Scaled system rheology study. Assess the physical properties of the grout at the RK mixer discharge, immediately downstream of the hose pump, and at the exit of the hose for an equivalent 35 ton/hr premix using a simulant salt solution for a range of RK mixer speeds.

These activities assessed the capabilities of the RK mixer and determined how reduced RK mixer speeds impacted the rheological properties. By reducing the RK mixer shaft speed, it may be advantageous in reducing paddle wear and improved wetting of the premix (10 wt% cement, 45 wt% slag, 45 wt% fly ash) (P/M). The rheologies of the SCPF samples were then compared to each other and to the rheology of grout samples using SRNL's conventional benchtop mixing techniques.

The RK mixer shaft speed study showed that the RK mixer has a greater capacity than it is presently processing, easily processing the 45 tons/hr. If the present throughput of 35 tons/hr is maintained at a water to premix ratio of 0.59, the RK mixer can operate at a greatly reduced shaft speed, from 220 RPM to approximately 110 RPM. Reduction of shaft speed has a potential to increase the life of the wetted parts (paddles) by decreasing the shear rate or velocity at the tips of the paddles, areas which have the highest erosion rates due to the highest shear rates and gaps. As the paddles wear over time, the RK mixer speed can be increased to maintain the targeted P/M feed rate until the maximum RK mixer speed is achieved, at which point the RK mixer paddles will need to be replaced.

The SCPF rheology study showed that additional mixing in the transfer line was beneficial in providing a consistent product rheologically and with no clumps upon discharge from the hose for RK mixing speeds from 115 to 220 RPM. The soft clumps that were present at the discharge of the RK mixer were incorporated in the grout hopper and transfer systems and provided a product that had little to no thixotropic properties. As the RK mixer speed was reduced, more clumps were observed leaving the RK mixer discharge, indicating lowering the speed did not improve the wetting of the premix with the salt solution. In fact, the samples after the Watson Bredel SPX20 hose pump yielded consistent rheological data with the hose data and were not clumping as much as the RK mixer discharge samples. The SCPF transfer line was scaled based on maintaining the same shear rate at the wall. This scaling basis results in the SCPF being in a more laminar flow condition that in the SDU, resulting in less rigorous mixing. Additionally, due to the pressure capabilities of the SCPF hose pump, a short piece of hose was used, resulting in a shorter transfer line residence time as compared to the future SPF to SDUs transfer times. It is expected that the product entering the SDU vaults would be as or more consistent as compared to what was measured in the SCPF.

The conventional bench scale mixing utilized by SRNL to prepare grout samples when compared to the SCPF hose discharge samples are conservative with respect to the plastic viscosity, which was almost twice as large as large as the SCPF plastic viscosities. The Bingham Plastic yield stresses were comparable to each other. SRNL's conventional mixing method will require a higher shear rate process to better represent the discharge properties.

In both of these studies, it was visually observed that as the RK mixer speed increased, the level of dusting increased.

It is recommended that additional tests be performed to determine the effect of reducing the RK agitator speed with respect to wear rate. It is expected that reducing the RK mixer speed will reduce wear, hence increasing the life of the mixer. Testing at three different speeds is recommended and to be determined at a future date. Three points may be sufficient to provide an operating curve for erosion based on the normal, average, and minimum RK mixer speeds shown in Table 4-1 for 35 tons/hr premix feedrate. Additional, bench scale testing such as the Miller test[16], would complement this test where such test methods could be used to assess other potential materials in assessing the wear rates due to erosion/corrosion.

It is also recommended that the SCPF be upgraded such that the dry material system is automated to permit continuous operations of the SCPF with minimal operator interference. This will reduce overall cost of operating the SCPF.

It is also recommended that the present hose pump be replaced with a higher pressure hose pump or to use two of the existing hose pumps in series that would allow for the SCPF have the same residence time as that observed in the SPF transfer line.

Additional bench scale work is also recommended to determine a mixing method that would result in similar rheological properties as those observed in the SCPF.

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LIST OF ABBREVIATIONS

DAS	Data Acquisition System
DF-E	DWPF/Saltstone Facility Engineering
DWPF	Defense Waste Processing Facility
FS	Full Scale
GPM	Gallons Per Minute
HDPE	High Density Polyethylene
LLW	Low Level Waste
M	Molar, Molarity
M&TE	Measurement and Test Equipment
RK	Readco-Kurimoto
SCPF	Scaled Continuous Processing Facility
SDU	Saltstone Disposal Unit
SFT	Salt Feed Tank
SPF	Saltstone Production Facility
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
T/C	Thermocouple
WAC	Waste Acceptance Criteria
WMB	Watson Marlow Bredel
W/P	Water to Premix Ratio

1.0 Introduction

The Savannah River National Laboratory (SRNL) has supported the Saltstone Production Facility (SPF) since its conception with bench-scale laboratory experiments. To complement the traditional laboratory testing, the Scaled Continuous Processing Facility (SCPF) was constructed to provide processing data related to mixing, transfer, and other operations conducted in the SPF.[1] At the SPF, the dry premix is weighed, mixed, and transferred to a Readco-Kurimoto (RK) continuous mixer where it is mixed with the low level waste (LLW) salt solution from the Salt Feed Tank (SFT) to produce fresh saltstone grout slurry. The grout slurry is discharged from the RK mixer into the grout hopper that uses a conventional mixer and has a residence time of approximately two minutes when operating in the full condition. The grout pump hopper feeds the Watson Marlow Bredel (WMB) hose pump that pumps the grout slurry through piping and discharges it into the Saltstone Disposal Units (SDU) for permanent disposal. The SCPF has been designed to mimic these operations with appropriate scaling factors for research and facility support studies. Defense Waste Processing Facility DWPF/Saltstone Facility Engineering (DF-E) requested that SRNL utilize the SCPF to determine the expected impacts of reduced mixer shaft speeds on SPF operations. Reducing the mixer shaft speed at the SPF may be advantageous in reducing paddle wear and improved wetting of the dry materials. DF-E also requested that SRNL characterize changes in slurry rheology as a function of progression through the SCPF while changing the RK mixer speed and maintaining a constant premix feed rate. There are minimal opportunities for the measurement of rheological properties of the grout slurry at the SPF; thus, the SCPF is the most representative, available process for determining the actual rheological properties expected in the SPF. Finally, bench scaled prepared grout samples using conventional mixing techniques at SRNL were compared to the SCPF results to determine if the conventional mixing technique provided the same level of shearing and hence rheological properties.

This work was initiated via Technical Task Request HLW-SSF-TTR-2013-0010, Revision 1.[2] The work was performed following Task Technical and Quality Assurance Plan SRNL-RP-2013-00821, Revision 0.[3] The scope of the test was revised to a mixer shaft speed study and rheology study in memorandum SRNL-L3100-2014-00001, Revision 0.[4] Requirements for performing reviews of technical reports and the extent of review are established in manual E7 procedure 2.60.[5] SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.[6]

2.0 System Description

The SCPF process and instrument diagram is shown in Figure 2-1. The SCPF uses a Schenck AccuRate Mechatron® gravimetric feeder to supply premix to a RK 2-inch continuous processor mixer. The RK mixer in SPF is a 10-inch continuous processor mixer. Scaling methods provided by RK are based on residence time (volume), power, or heat transfer (area).[A] For SPF applications, RK recommended using volumetric scaling.[B] In this case, SRNL used the cube of the diameter ratios ($(2/10)^3$) and multiplied this value with the rate (tons/hr or gallons/min) in the SPF to determine the rate (lbm/hr or gallons/min) in the SCPF. Using this scaling for tons/hr is permitted, because the density of the premix or grout in both scales is the same. If using the actual working volumes of the 10-inch and 2-inch RK mixers, a smaller volumetric scaling ratio is the result hence the processing rates used in this SCPF are conservative. Given the water to premix mass ratio (W/P), liquid density, and mass fraction solids in the liquid, the salt solution feed rate to the RK mixer was determined and provided by a variable speed gear pump. The gear

[A] Readco-Kurimoto LLC Presentation, 26 August 2010
[B] Per phone and email communications with Stan Myers of Readco-Kurimoto, 3/18/2011

pump is adjusted to obtain the flow rate required to maintain the targeted W/P of 0.59, which was constant for all tests. The liquid feed consisted of either process water or a salt solution simulant (see Section 2.4). The liquid feed is initiated prior to starting the premix (see Section 2.5). Fresh grout is discharged from the RK mixer into the grout hopper fitted with an agitator. The SCPF grout hopper is a linearly geometrically scaled version of the SPF hopper. The primary difference is the agitator; the SCPF agitator does not have the top impeller used in the SPF. A WMB SPX20 high performance hose pump was used to pump the grout from the grout hopper through a 5/8 inch inside diameter tube to the grout receipt containers, which in these tests are 55-gallon drums. The WMB SPX20 has a maximum operating pressure of 100 psig. The 5/8 inch tubing (or hose) was selected based on obtaining approximately the same shear rate at the pipe wall in SPF transfer lines at the nominal SPF grout flowrate of 150 gallons per minute (GPM), assuming the shear rate in either the SPF and SCPF is determined using 8V/D, where V is the average line velocity and D is the inside pipe/hose diameter. As scale increases, from the SCPF to the SPF, and given constant velocity, the condition of the flow will tend towards turbulence sooner given the physical properties of the grout are the same in the two scales. Hence the level of mixing would be greater in the SPF. Additionally, given the maximum operating pressure of the WMB SPX20, the transfer pipe (tube) length is the SCPF is shorter than that in the SPF, resulting in a shorter residence time as compared to the distances to the new vaults in SPF. Hence additional mixing is expected in the SPF piping.

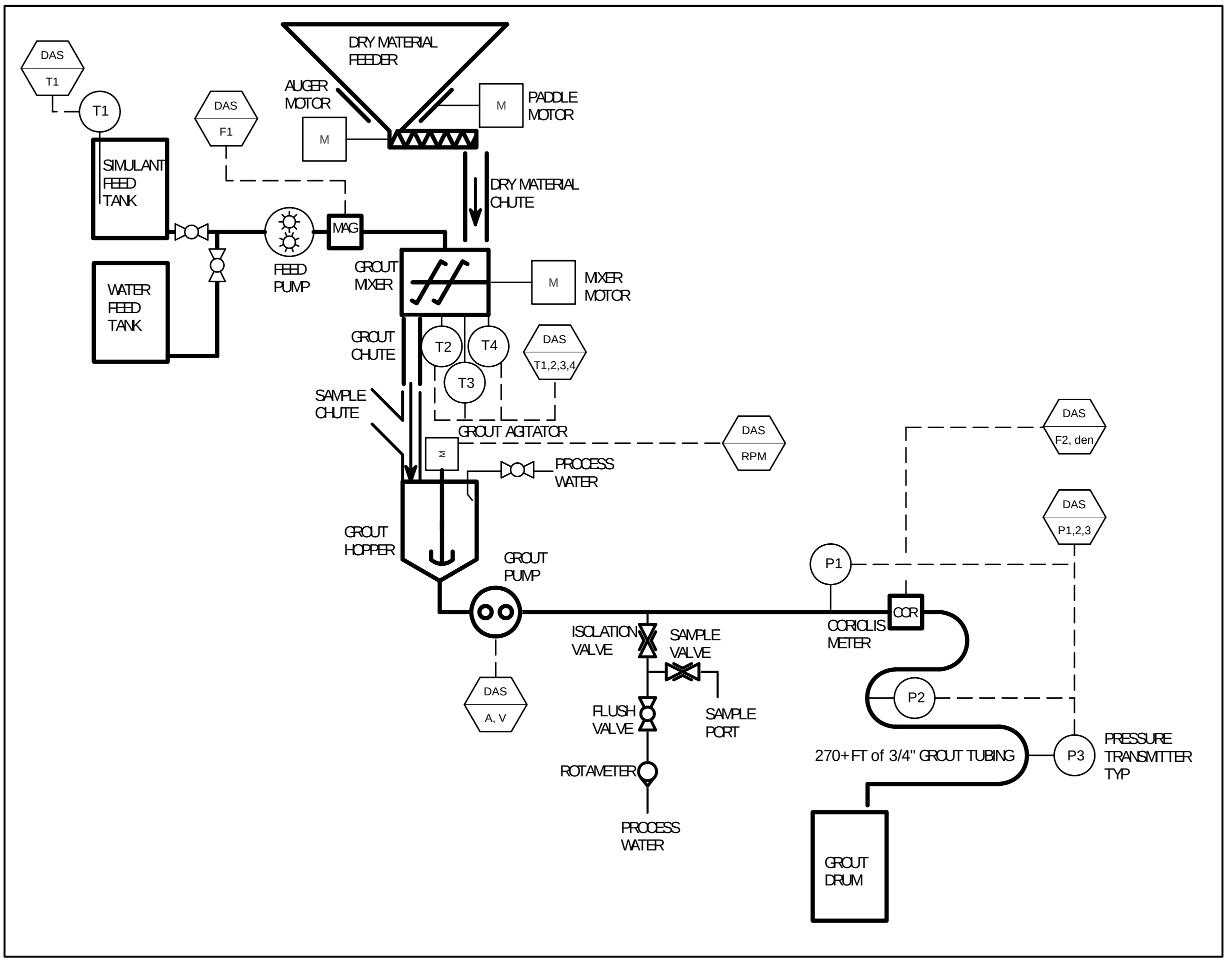


Figure 2-1. Process and Instrument Diagram

For this testing, the grout line consisted of approximately 270 feet of both stainless steel and high density polyethylene (HDPE) three-quarters inch tubing. The grout line was primarily HDPE tubing with short stainless steel sections directly downstream of the grout pump and at locations where a flowmeter, pressure instrumentation or valves were installed. The 270 feet of hose was determined by performing hydraulic calculations using the methods proposed by Darby[7] by limiting the maximum pressure loss in the hose to approximately 90% of the maximum operating pressure of the WMB SPX20 hose pump, the velocity based on the nominal flowrate, and using the following physical properties: density – 1.75 g/cm^3, plastic viscosity – 122 centipoise, and Bingham Plastic yield stress – 7.96 Pa.[8] The original intent was to equate the residence time or total applied shear (at the wall) in the transfer line between the two scales. This could not be achieved due to the maximum operating pressure of the WMB SPX20.

2.1 Mixer Configuration

The 2-inch RK mixer uses co-rotating, twin shafts configured with intermeshing paddles and augers. Clearance between intermeshing paddles and between the paddles and the barrels are approximately one-eighth inch. This clearance between the paddle and barrels produces a self-wiping action that minimizes material buildup, but results in the highest region of shear rates. The RK mixer motor is connected to a variable speed drive and controlled through an integral touch-screen panel. The premix is delivered into the auger section of the RK mixer.

The paddles were configured as recommended by SRNL to the SPF.[9,10] Forward conveying helical paddles were placed in stages 1-6 and non-conveying (flat) paddles were placed in stages 7-25. Reverse conveying helical paddles were placed in stage 26, to direct grout away from the thrust shaft seals. The initial helical paddles in stage 1 were aligned with the spiral outlet of the premix augers, resulting in a smooth transition between the spiral and helical paddles. The subsequent paddles were each offset 45° from the previous paddle. Liquid feed was introduced in the center of the mixing chamber between stages 5 and 6. Figure 2-2 shows the actual shafts, augers, and paddle arrangement of the RK mixer and specifies the location of liquid injection. The grout mixer discharge opening was in the full open position for all tests.

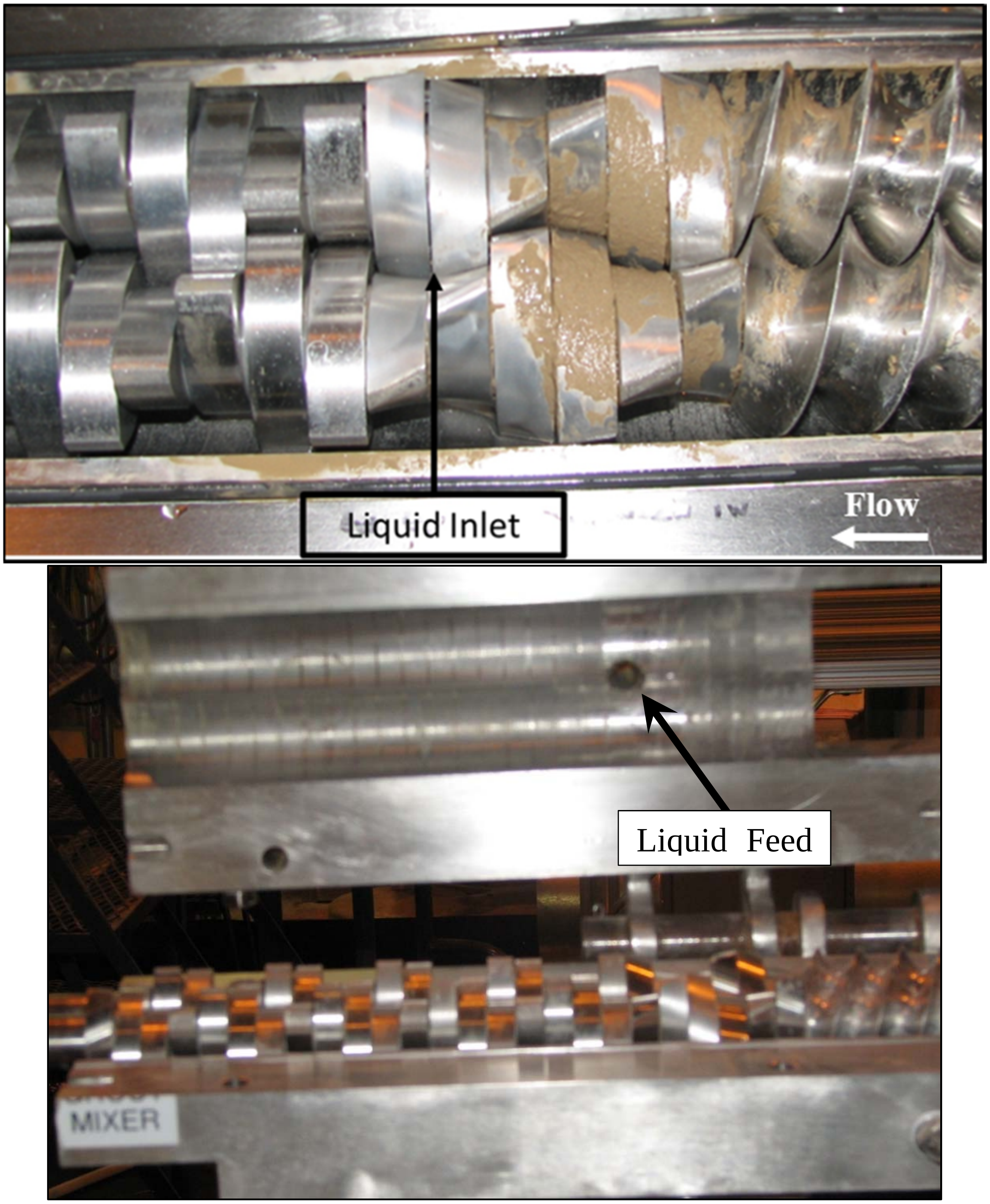


Figure 2-2. RK Mixer Shaft, Auger and Paddle Configuration

2.2 Sampling and Characterization

During processing, fresh grout is sampled at three locations; at the discharge of the PK mixer, approximately five feet downstream of the WMB SPX20 hose pump, and at the exit of the discharge tubing (~270 feet from the pump). A sampling chute located above the grout hopper allowed sampling of fresh grout from the discharge of the RK mixer (Figure 2-3). A combination of diaphragm valves five feet downstream of the WMB SPX20 hose pump were used for sampling while the pump was operating and flushed upon completion of sampling. Using a 500 mL beaker, approximately 350 mL of grout was pulled at all sampling locations for physical characterization.

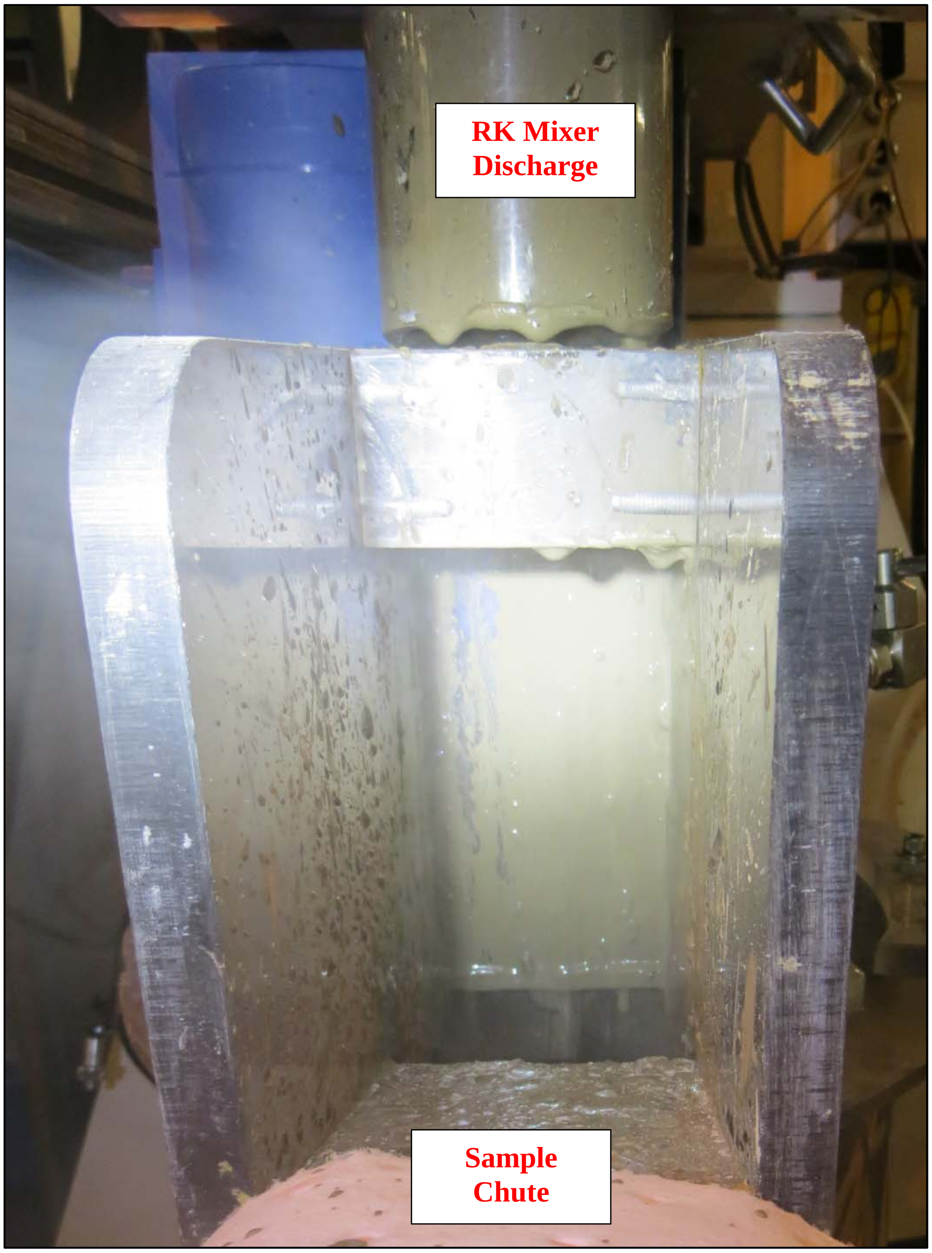


Figure 2-3. RK Mixer Discharge

The sampled fresh grout was immediately analyzed for rheological properties using the Haake VT-550 roto-viscometer. The flow curve method and analysis are provided in reference 8 and the grout was analyzed as a Bingham Plastic Fluid. Fresh grout was also poured into a 100 mL cylindrical sample bottles to at least three-quarters full, capped and allowed to cure for at least 30 days prior to measuring the diameter (d), height (h) and mass (m) of the sample to determine its density (ρ) using equation (1). Visual observations of the fresh grout samples were recorded in a notebook.

$$\rho = \frac{m}{\frac{1}{4}\pi d^2 h} \tag{1}$$

2.3 Instrumentation and Data Acquisition System (DAS)

Pressure, temperature and flow instrumentation were installed in the SCPF process to monitor test conditions. Instrument outputs were recorded electronically on a DAS with the exception of the dry material (Premix) feeder operating conditions, which were recorded manually. Pressure was recorded and monitored at three locations on the grout discharge tubing; at approximately 5 feet, 110 feet and 182 feet downstream of the Bredel SPX20 hose pump (see Figure 2-1). The rotational speed and torque of the agitator in the grout hopper were recorded on the DAS. The list of instruments utilized in the SCPF is provided in Table 2-1. For this testing, a camera was located in the dry material discharge chute to record the conditions of the how the premix flowed into the RK augers and to provide an indication of when the premix started to backup into the chute.

Table 2-1. Instrumentation List for the SCPF

System	Designation	Description	Instrument	Range	Tolerance
Material Feed	LF	Liquid feed flow rate	Magnetic flow meter	0-5 gpm	+/- 0.4 gpm
	DF	Dry material feed rate	Gravimetric feeder	0 – 1000 lb/hr saltstone premix	N/A
	Temp4	Liquid feed temp, simulant	Type K T/C	-200 to 1259 °C	+/- 2.2 °C
PK Mixer	Temp1	Mixer housing temp, inlet	Type J T/C	0 to 750 °C	+/- 2.2 °C
	Temp2	Mixer housing temp, mid	Type J T/C	0 to 750 °C	+/- 2.2 °C
	Temp3	Mixer housing temp, exit	Type J T/C	0 to 750 °C	+/- 2.2 °C
Grout Hopper and Discharge Hose	Spd	Grout agitator speed	Laboratory mixer	50-2000 rpm	N/A
	Tor	Grout agitator torque	Laboratory mixer	0-60 N-cm	N/A
	GF	Grout flow rate	Coriolis meter	0-8 gpm	+/- 0.7 % FS
	GD	Grout density	Coriolis meter	0-200 lb/ft^3	+/-0.6 lb/ft^3
	P1	Grout line press, location 1	Pressure transmitter	0-900 psig	+/- 1% FS
	P2	Grout line press, location 2	Pressure transmitter	0-100 psig	+/- 1% FS
	P3	Grout line press, location 3	Pressure transmitter	0-100 psig	+/- 1% FS

2.4 Salt Simulant

The salt solution for these tests was remediated from an existing salt simulant used at SRNL to approximate a Tank 50 Waste Acceptance Criteria (WAC) analysis.[11] Using sodium hydroxide and sodium nitrate, the total solids content was adjusted to a target 5 to 5.5 M Na and the nitrite plus nitrate to hydroxide ratio was also adjusted to approximate the salt solution in Reference 11. SRNL made 260 gallons of this salt solution simulant for this testing. Table 2-2 is the targeted, calculated blend, and measured composition of the salt solution used in this testing.

Table 2-2. Properties and Composition of the Simulated Salt Solution.

pH	13.8			
Total Solids (wt%)	30.1			
Density (g/ml)	1.262			
Element/Compound		Concentration (g/L)		
		Target	Calculated Blend	Measured
Aluminum	Al	4.06	4.53	4.95
Chloride	Cl	0.50	2.34	2.51
Hydroxide	OH	32.90	30.14	30.14
Nitrate	NO_3	136.00	126.64	144.00
Nitrite	NO_2	21.30	17.36	20.85
Phosphorus	P	0.14	0.64	0.65
Potassium	K	0.23	3.03	3.72
Sodium	Na	119.32	126.17	139.00
Sodium	(Molar)	5.19	5.49	6.05
Sulfate	SO_4	5.12	8.31	9.10
Ratio	$(NO_2+NO_3)/OH$	4.78	4.78	5.47

2.5 Premix

The premix contained 45 wt % slag, 45 wt % fly ash, and 10 wt % portland cement. The premix was blended either by a third party vendor or using the SPF premix system and obtained from SRR. X-ray diffraction analysis was performed on samples from each of these sources to confirm the approximate blending ratio was met. The spectrum labeled "Fox Premix A" is the blended material in a super sack from the SPF. The spectrum labeled "Fox Premix 50" is the blended material in 50 pound bags from the vendor in a previous task.[12] The same phases were identified in both spectra. The density of the portland cement, slag and fly ash are 3.07, 2.75 and 2.26 g/cm^3 respectively. The calculated density of the premix using volume additivity was 2.53 cm^3.

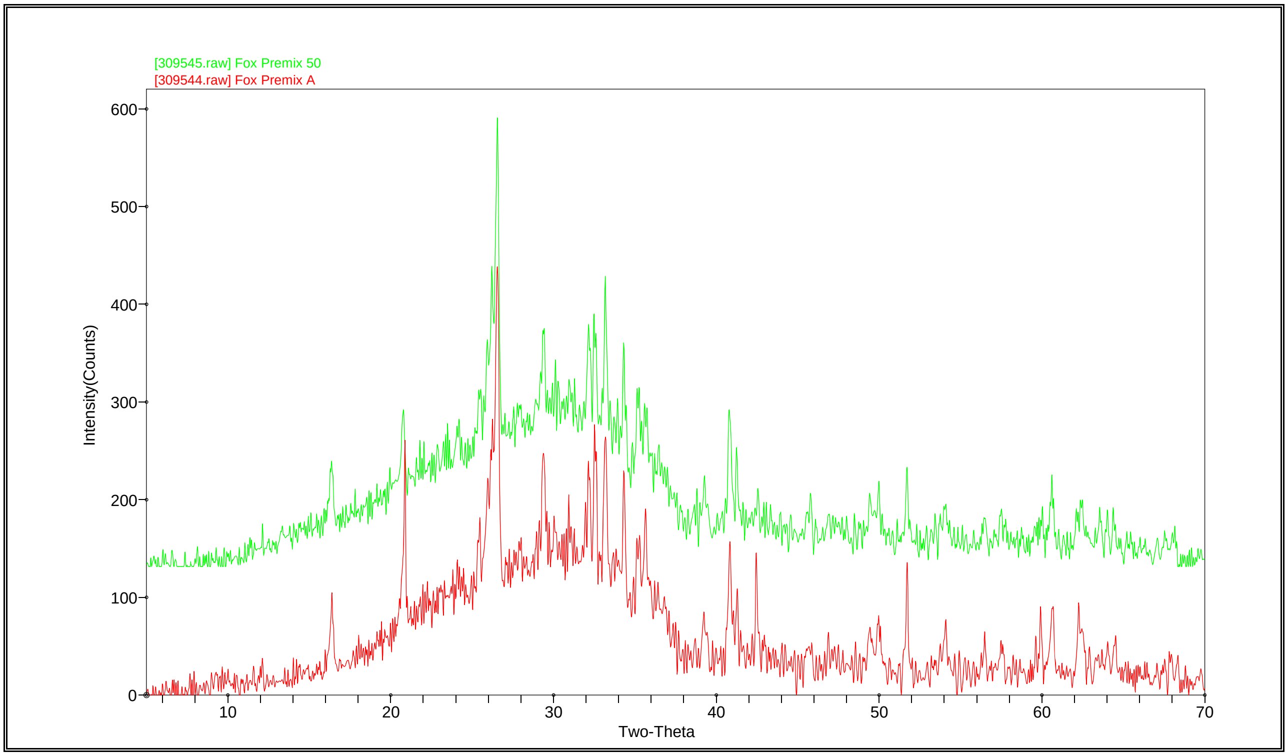


Figure 2-4. X-Ray Diffraction Patterns for Premix Sources.

3.0 Experimental Procedure

Two tests were performed. First, the RK Mixer Shaft Speed Study was performed with the objective of demonstrating operation with reduced mixer shaft speeds at three feed rates. The second test, the Scaled System Rheology Study was performed with the objectives of measuring rheology at multiple locations along the SCPF and demonstrating operation with reduced RK mixer shaft speed as determined in the Mixer Shaft Speed Study.

3.1 Mixer Shaft Speed Study

The Mixer Shaft Speed Study was performed to determine the lowest achievable RK mixer shaft speeds for targeted premix feed rates (25, 35, 45 tons/hr SPF equivalent). The 35 tons/hr was the baseline condition, the lower limit of 25 tons/hr as a lower limit to reflect long transfer lines, and 45 tons/hr to reflect short transfer lines. It is not expected the SPF will operate at these limiting bounds. The lowest achievable speed was quantified as the point where the dry premix backed up into the dry material chute and remained at a stable level. The height of the dry material chute is approximately 8 inches (from the dry feeder discharge to the top of the RK mixer augers). A camera was mounted at the top of the chute to assess the stability of the feed level and auger section of the RK mixer. The slurry hopper, WMB SPX20 pump, and transfer line were not part of this study, the grout leaving the RK mixer was discharged directly into 55 gallon drums.

The SPF premix feed rates (25, 35, 45 tons/hr equivalent) in the SCPF and the corresponding salt solution flow rates were determined using equations (2) and (3) respectively. The results are provided in Table 3-1 for a W/P of 0.59 and the salt solution properties provided in Table 2-2.

$$PM_{SCPF} = \frac{PM_{SPF}}{125} \cdot 2000 \quad (2)$$

$$Q_{L,SCPF} = PM_{SCPF} \cdot \left(1 - \frac{1}{1 + \frac{W/P}{1 - f_{TS}}}\right) \cdot \left(1 + \frac{W/P}{1 - f_{TS}}\right) \cdot \frac{7.4805}{\rho_{SS} \cdot 60 \cdot 62.4} \quad (3)$$

Where: PM_{SPF} = SPF premix feed rate (ton/hr)
PM_{SCPF} = SCPF premix feed rate (lbm/hr)
W/P = water to premix ratio (lbm-water/lbm-premix)
f_{TS} = fraction total solids in salt solution, Table 2-2, (lbm-total solids/lbm-salt solution)
ρ_{SS} = Density of salt solution, Table 2-2, (g/ml) or specific gravity
$Q_{L,SCPF}$ = SCPF salt solution feed rate (gpm)

Table 3-1. SPF Premix Feed Rate and Resulting SCPF Premix and Salt Solution Rates

Flow Parameter	Units	Rates		
SPF Premix Feed Rate:	tons/hr	45	35	25
Targeted SCPF Premix Feed Rate	lbm/hr	720	560	400
Targeted SCPF Salt solution feed rate	gpm	0.96	0.75	0.54

This study consisted of testing and data collection at the three feed rates:

- Start at feed rates of 45 tons per hour equivalent
  - Set SCPF RK mixer shaft speed to 220 RPM. Shaft speed scales one to one between the SPF and SCPF.[13,C]
  - Set salt solution flow rate of 0.96 gpm and premix of 720 lbm/hr.
  - Allow SCPF RK mixer to operate for one minute after each speed change and monitor dry feed behavior and record behavior in notebook
  - Reduce SCPF RK mixer shaft speed in 10 RPM increments
  - Continue to reduce SCPF RK mixer shaft speed in increments until premix begins to back up into the premix chute
  - Identify minimum SCPF RK mixer shaft speed.

- Reduce feed rates to 35 tons per hour equivalent
  - Perform same procedure used for 45 ton per hour equivalent.
  - Set salt solution flow rate of 0.75 gpm and premix of 560 lbm/hr
  - Identify minimum SCPF RK mixer shaft speed.

- Reduce feed rates to 25 tons per hour equivalent
  - Perform same procedure used for 45 ton per hour equivalent.
  - Set salt solution flow rate of 0.54 gpm and premix of 400 lbm/hr
  - Identify minimum SCPF RK mixer shaft speed.

[C] Discussions with READCO staff stated speed is scaled one to one when comparing one scale to another using their equipment.

3.2 Scaled System Rheology Study

The full SCPF process (Figure 2-1) was used in this study and the baseline condition of 35 tons/hr was used throughout as the baseline for comparison. This study consisted of testing and data collection at three mixer shaft speeds and collecting data from the laboratory and comparing these results to each other:

- Start test with SCPF RK mixer shaft speed to match SPF RK mixer shaft operating speed.
  - Set salt solution flow rate of 0.75 gpm
  - Scale hopper agitator shaft speed to match full scale vortex shape, visually
  - Set premix feed rate to 35 tons per hour equivalent (560 lbm/hr)
  - Operate until steady state conditions are obtained
  - Collect samples at RK mixer exit, WMB SPX20 hose pump exit, and from the discharge of the transfer line and measure physical properties.

- Reduce SCPF RK mixer shaft speed to the halfway point between SPF RK mixer shaft operating speed and minimum SCPF RK mixer speed determined earlier for 35 tons per hour equivalent feed rate.
  - Collect samples at each of the three locations as described above and measure physical properties.

- Reduce SCPF RK mixer shaft speed to the SCPF RK mixer minimum speed determined earlier for 35 tons per hour equivalent feed rate
  - Collect samples at each of the three locations described above and measure physical properties

- Perform bench top mixing of the premix and salt solution used in the SCPF mixing studies. The conventional SRNL mixing method used is described in reference 8 and rheological measurements were obtained up completion of mixing. The minimum mixing time to equal the calculated processing time for grout in the SCPF for an equivalent 35 tons per hour was used for the first measurement. A subsequent rheological measurement on the remaining sample was made 11 minutes later to determine the impact of extended mixing time for this grout. The comparison between the bench top and SCPF samples was to determine if bench top mixing is capturing the applied stresses and/or shear rates observed by the grout in the SCPF process, specifically the point of comparison is at the discharge.

3.3 Preparations

Operation of the SCPF was controlled by work instruction ITS-WI-0053.[14] Prior to operation, the equipment was prepared by loading the dry material feeder with premix, programming the feeder to the targeted discharge rate, and filling the water and simulant supply tanks. The dry material feed hopper has an extension that holds 8.7 ft^3 of dry material, which is approximately 400 lbm of premix. The grout mixer hopper was filled with water to submerge the agitator impeller and the WMB SPX20 hose pump was operated to fill the discharge line.

3.4 Startup

The SCPF process was first started by adjusting the grout mixer hopper agitator speed; the level of agitation was judged by the depth of the vortex. The surface motion in the SCPF grout mixer hopper was targeted to match the surface motion observed in the SPF grout mixer hopper video provided by SRR. The liquid feed pump was started and water was introduced into the RK mixer. After visual confirmation of water exiting the RK mixer and into the grout hopper, the WMB SPX20 hose pump was started and its speed adjusted to obtain a stable level in the grout hopper. The RK mixer was started and set to the targeted rotational speed and then the dry material feeder was started. Once grout was observed discharging in to the grout hopper, the liquid feed was transferred from water to the salt solution simulant. The operating parameters (mass flow rate of premix and GPM of simulant) were verified and adjustments made as necessary to maintain stable operation throughout testing. The rotational speed of the RK mixer was verified using a tachometer.

3.5 Operations

Once initiated, the process ran continuously until the study was complete. When the dry material feeder inventoried mass was below 70 lbm, the feeder switched from gravimetric mode (controlled weight loss) to volumetric mode. The dry material feed hopper was reloaded manually during the volumetric mode where bags of premix were poured into the feeder hopper extension. The dry material feeder reverted to gravimetric mode when the feed hopper inventoried mass exceeded 400 lb. The reloading task was completed within 5 minutes.

During operations the feed rate of the premix, liquid, grout tank level, and WMB SPX20 hose pump speed were monitored and maintained. Grout samples were collected from the three sampling location at each of the test conditions mentioned in Section 3.2. When the sample was collected from the discharge of the WMB SPX20 hose pump, the sample valve/piping was flushed with process water. Agitation in the grout hopper was monitored and adjusted to provide movement of the grout targeted during startup. In previous tests, it was noted that inadequate movement of the grout in the grout mixer hopper allowed grout to collect and build on the hopper walls. However, if excessive agitation is used, air is more easily entrained into the grout slurry and it affected pressure, density, and flow measurements.

3.6 Shut-down

At the completion of a study, the system flushing operations began by stopping the dry material feeder and switching the liquid feed from simulant to water. Flushing and equipment cleaning was mostly a manual operation and continued until water coming from the mixer and from the grout discharge line ran clear. The details of the flushing and cleaning operations are described in Appendix A.

4.0 Results and Discussion

4.1 Mixer Shaft Speed Study

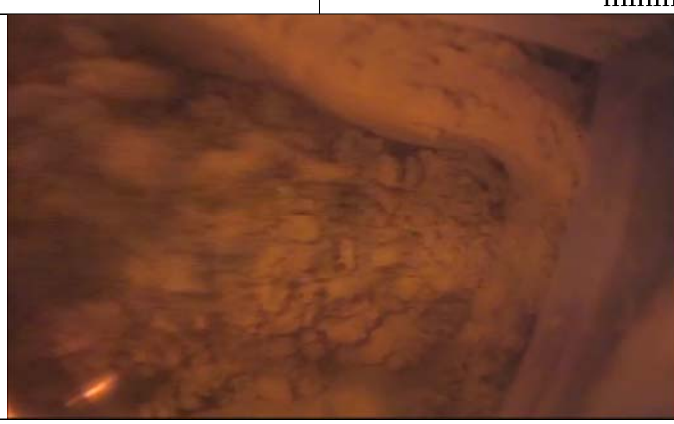
The normal, medium, minimum and backup RK mixer speeds determined in the mixer shaft speed study are provided in Table 4-1. The normal speed is the same in all cases, given it is the maximum and fixed speed used for the SPF RK grout mixer. The minimum speed is based on the point where the RK augers are just covered with premix; prior to reaching this point the premix was observed to be backing up in the auger section closest to the auger/paddle interface first and then slowly filling up the auger from this interface to the beginning of the auger section as the RK mixer speed was further reduced. The medium is the average speed between the normal and minimum speeds. The backup speed is when the premix started to backup into the dry materials feeder chute. The results provided are for the premix, salt solution, and w/p ratio used in this study. If different materials or w/p ratios are used, the minimum/backup speeds could potentially be different. This is due to the different dry powders having different flow characteristics that could impact how the materials flow and are wetted in the RK mixer with the salt solutions. Such variability was not performed in this study and such studies are not recommended unless such changes are to be implemented in SPF. Figure 4-1 shows three pictures from the camera located in the dry materials feeder chute during testing; the first shows the RK augers prior to the addition of dry feed, the second shows dry material being added and the speed greater than the minimum, and the third shows the premix backed up into the chute. During this test, it was visually observed that as the RK mixer speed was reduced, the level of dusting exiting the discharge of the RK mixer decreased for all tonnage rates. The level of dusting was not quantified.

Table 4-1. Test Conditions for the Rheology Test

Dry Feed Rate (lb/hr)	RK Mixer Speed (rpm)	
720 (45 tons/hr eq.)	Nominal	220
	Medium	180
	Minimum	140
	Backup	120
560 (35 tons/hr eq.)	Nominal	220
	Medium	165
	Minimum	110
	Backup	90
400 (25 tons/hr eq.)	Nominal	220
	Medium	150
	Minimum	80
	Backup	70

Empty

Condition when auger speed is greater than minimum



Dry Material Auger Chute Filling up

Figure 4-1. Plan view of the mixer premix feed augers, empty, above minimum speed and "backed up."

4.2 Scaled System Rheology Study

This study started at the maximum RK mixer speed and the speed was reduced as the study proceeded. Additional samples were obtained on top of those originally projected, given there was additional feed materials that could be used and in this case, samples were obtained covering additional operating speeds. The samples were collected in chronological order, starting with 1 and ending with 19 from the SCPF sampling points as shown in Table 4-2. Table 4-2 provides the sample location, sample number, RK mixer speed, and plastic viscosity, Bingham Plastic yield stress and density of the grout. The samples were poured first for the rheology measurement and then for density and the remaining sample poured out at the 55 gallon drums. Bench scale samples were also prepared to compare the rheological data to bench prepared samples. A single 575 mL bench scale sample was processed and analyzed based on the time a slug is processed through the SCPF (approximately 6.2 minutes), otherwise known as the total residence time. The remaining mass not used for the initial rheological measurement was mixed for an additional 11 minutes prior to a final rheological measurement. The flow curve for each sample is provided in Appendix B.

Table 4-2. Physical Property Results for Scaled System Rheology Study

Sample Location	Sample Number	RK Mixer Speed (RPM)	Plastic Viscosity (cP)	Bingham Plastic Yield Stress (Pa)	Density (g/ml)
RK Mixer Discharge	1	230	104.0	6.26	1.686
	5		99.6	5.78	1.673
	18		94.3	6.01	1.806
	6	165	96.9	6.08	1.690
	9		94.3	5.69	1.708
	10	115	80.9	5.34	1.755
	13		76.2	5.07	1.802
	15		98.5	3.78	1.760
	16	140	69.3	4.55	1.800
	17		93.3	6.26	1.660
	19	85	86.3	5.56	N/M*
WMB SPX20 Pump Discharge	4	230	99.4	6.14	N/M
	8	165	93.2	5.54	1.676
	12	115	82.1	6.12	1.680
	14		89.9	5.87	1.783
Hose Discharge	2	230	86.3	5.28	1.694
	3		102.3	6.62	1.656
	7	165	103.8	6.64	1.661
	11	115	92.8	6.67	1.657
Laboratory	20	-	165.9	6.57	N/M
	21	-	157.5	7.08	N/M

*N/M = not measured

The following observations and assessments from the testing from this study are summarized:

- It was visually observed that as the mixer speed decreased, the level of dusting at the exit of the RK mixer discharge decreased. This was consistent with the previous study. The level of dusting was not quantified.

- It was visually observed that as the mixer speed decreased, the size/mass of clumps increased in the RK mixer samples. This suggests that the lower mixer speeds (shear rates) did not do an effective job in blending all the premix with the salt solution simulant into a homogeneous product leaving the RK mixer. The size/masses of the clumps were not quantified.
- It was visually observed that the clumps were present in some of the Bredel SPX20 pump discharge samples, though not as large or in the quantity as those observed in the RK mixer discharge samples.
- Clumps were not identified in the hose discharge samples.
- The clumps were not hard, but seemed to lack more liquid than the surrounding grout. Clumps were not pulled and analyzed for moisture or total solids content to assess this assumption.
- A reason why clumps increased as the RK mixer speed decreased could be due to the lower shear rates applied at the lower mixer speeds not blending the premix with the salt solution.
- The calculated density of the grout was 1.73 g/mL using volume additivity for a w/p ratio of 0.59, this assumes no air entrainment. Small bubbles were observed in all the cured density samples.
    - For the RK mixer and WMB SPX hose pump samples, the data indicates that large clumps were present in some of the cases and in other, less clumps or bubbles were present.
    - For the hose samples, the data suggests clumps were not present, but entrainment is present and reflected in this data.
    - The laboratory sample densities were not measured given previous experience has shown that samples prepared in such a manner were similar (slightly less than) to the calculated densities due to the SRNL conventional mixing method vents air entrained due to the dry premix.[8] Such is not the case for the SCPF or SPF and if air is entrained into the process, the mixing in the piping has the capabilities to reduce the size of the bubbles and to provide a more uniform and smaller distribution upon discharge into the vaults.
    - The low densities of the hose samples suggest air entrainment will be present in the grout that will be discharged into the SPF vault.
- The quantity of entrained air was not calculated for any of the samples.
- The flow curves (see Appendix B) for the RK mixer and WMB SPX20 hose pump samples were more thixotropic (a time dependent rheological condition where the up curve is above the down curve) than those of the hose samples, indicating the additional mixing in the hose removed the thixotropic properties.
- The RK mixer samplers were generally more thixotropic than the WMB SPX20 hose pump samples (see Appendix B).
- In the case of the laboratory prepared samples, the original sample (#20) was very thixotropic and when sheared for an additional 11 minutes, it was less thixotropic and on about the same order as those of the WMB SPX20 hose pump.
- The laboratory samples bound the SCPF samples with respect to the rheological properties. The plastic viscosity is much greater in the laboratory samples. What this means is that the laboratory samples if used for transport analysis in the SPF would yield a higher velocity in which transition between laminar and turbulent flow would occur. These data have not been assessed for pressure drop.
- The shear rate applied to the laboratory samples indicate they are lower than that applied by the SCPF given the rheological data.
- At higher RK mixer speeds, the rheological data is comparable to the hose data. As the mixer speed is reduced, the differences become larger and this could be due to the clumps.
- The WMB SPX20 hose pump rheology results are similar to the hose data results. This suggests that the mixing in the hopper and pump provide blending of the clumps, providing a more uniform feed. This data also suggests that sampling from a similar point in the SPF process could be used to assess the rheological properties of the actual process.

- Increased shear rate on grout samples have been shown to decreases the rheological properties, specifically the plastic viscosity if assessed as a Bingham Plastic fluid.[15]

5.0 Conclusions

The RK mixer shaft speed study showed that the RK mixer has a greater capacity than it is presently processing, easily processing the 45 tons/hr. If the present throughput of 35 tons/hr is maintained at a water to premix ratio of 0.59, the RK mixer can operate at a greatly reduced shaft speed, from 220 RPM to approximately 110 RPM. Reduction of shaft speed has a potential to increase the life of the wetted parts (paddles) by decreasing the shear rate or velocity at the tips of the paddles, areas which have the highest erosion rates due to the highest shear rates and gaps. As the paddles wear over time, the RK mixer speed can be increased to maintain the targeted P/M feed rate until the maximum RK mixer speed is achieved, at which point the RK mixer paddles will need to be replaced. Given the present limitations of the hose pump at SPF, processing at the higher premix throughput is not possible, particularly when pumping to the furthest SDUs.

The SCPF rheology study showed that additional mixing in the transfer line was beneficial in providing a consistent product rheologically and with no clumps upon discharge from the hose for RK mixing speeds from 115 to 220 RPM. The soft clumps that were present at the discharge of the RK mixer were incorporated in the grout hopper and transfer systems and provided a product that had little to no thixotropic properties. As the RK mixer speed was reduced, more clumps were observed leaving the RK mixer discharge, indicating lowering the speed did not improve the wetting of the premix with the salt solution. In fact, the samples after the Watson Bredel SPX20 hose pump yielded consistent rheological data with the hose data and were not as clumping as the RK mixer discharge samples. The SCPF transfer line was scaled based on maintaining the same shear rate at the wall. This scaling basis results in the SCPF being in a more laminar flow condition that in the SDU, resulting in less rigorous mixing. Additionally, due to the pressure capabilities of the SCPF hose pump, a short piece of hose was used, resulting in a shorter transfer line residence time as compared to the future SPF to SDUs transfer times. It is expected that the product entering the SDU vaults would be as or more consistent as compared to what was measured in the SCPF.

The conventional bench scale mixing utilized by SRNL to prepare grout samples when compared to the SCPF hose discharge samples are conservative with respect to the plastic viscosity, which was almost twice as large as large as the SCPF plastic viscosities. The Bingham Plastic yield stresses were comparable to each other. SRNL’s conventional mixing method will require a higher shear rate process to better represent the discharge properties.

In both of these studies, it was visually observed that as the RK mixer speed increased, the level of dusting increased.

6.0 Recommendations, Path Forward or Future Work

It is recommended that additional tests be performed to determine the effect of reducing the Readco-Kurimoto agitator speed with respect to wear rate. It is expected that reducing the Readco-Kurimoto mixer speed will reduce wear, hence increasing the life of the mixer. Testing at three different speeds is recommended and to be determined at a future date. Three points may be sufficient to provide an operating curve for erosion based on the normal, average, and minimum RK mixer speeds shown in Table 4-1 for 35 tons/hr premix feedrate. Additional, bench scale testing such as the Miller test[16], would complement this test where such test methods could be used to assess other potential materials in assessing the wear rates due to erosion/corrosion.

It is also recommended that the SCPF be upgraded such that the dry material system is automated to permit continuous operations of the SCPF with minimal operator interference. This will reduce overall cost of operating the SCPF.

It is also recommended that the present hose pump be replaced with a higher pressure hose pump or to use two of the existing hose pumps in series that would allow for the SCPF have the same residence time as that observed in the SPF transfer line.

Additional bench scale work is also recommended to determine a mixing method that would result in similar rheological properties as those observed in the SCPF.

7.0 Reference

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Appendix A. Flushing and Cleaning

Flushing the system was initiated at the end of a run or test by stopping the dry material feed, switching the liquid feed to water and allowing the system to run for a while with water only. However, flushing simply with the liquid feed was not sufficient to clean the entire system in preparation for an extended idle period. Leaving grout residue anywhere in the system would impede future operation if the grout was allowed to cure.

As a first step in flushing the mixer the dry material chute was removed and water introduces onto the top of the augers while the mixer was running. In addition, the grout hopper was flushed using a lid-mounted spray nozzle and a hand-held hose to direct spray onto areas not affected by the nozzle. A scrub brush was necessary for stubborn residue in the grout hopper. Intermittently the speed of the grout pump was increased to help push residue out of the discharge line. The sample valve downstream of the grout pump was flushed using a 3-valve system connected to process water (Figure 2-1). Flushing continued until liquid in the grout hopper and liquid discharging from the transfer line ran clear.

However, it was discovered during pretest checkout that the relatively unobtrusive methods described above were not sufficient in removing all grout residue from the system. Areas of low velocity or dead zones collected grout. Two notable areas where this occurred were at the exit of the mixer (Figure D-D) and in the elbow and reducer below the grout hopper (Figure E-E). Other areas where grout collected were in between the paddles and at minor obstructions in the flow path such as the inlets of fittings and valves. These areas were cleared of grout only through disassembly.

Figure A- 1. Scalled Mixer Discharge Outlet

As shown in Figure A- 1 grout collected and built up on the edges of the rectangular discharge of the mixer. This behavior has occurred on all previous tests with the SPCF but never to the point where the grout flow was inhibited.

Figure A- 2 shows the exit piping of the grout hopper. The elbow was 1-1/2” schedule 40 pipe and the reducer transitioned from that size to ¾” schedule 40 pipe. As mentioned previously, during the flushing operation the grout pump speed would be increased to help remove residue. Data show that the max flow rate during the flush operation was 2.3 gpm. Given the elbow ID was 2.61 inches the velocity was only 0.36 ft/s, which proved to be insufficient at removing residue.

Figure A- 2. Elbow and Reducer below SCPF Grout Hopper

One problem with grout curing in the system occurs when it eventually sloughs off and travels through the system. These chunks of grout can become lodged in congested areas and impede the flow. This occurred several times during the pretest checkout of the system. Chunks of grout leftover from previous tests were lifted into the flow stream and lodged in the tubing upstream of the coriolis meter. The ID of the coriolis meter was smaller than the discharge line a proved to be an ideal place to collect chunks of grout. When this occurred the pressure in the discharge line in front of the clog rose above safe levels and precipitated the shutdown of the system. Once the origin of the chunks of grout was discovered the piping was disassembled and cleaned. A high reading alarm was applied to the P1 pressure reading (ref. Figure 2-1) and the reading was closely monitored during testing to detect clogs.

Appendix B. Flow Curves

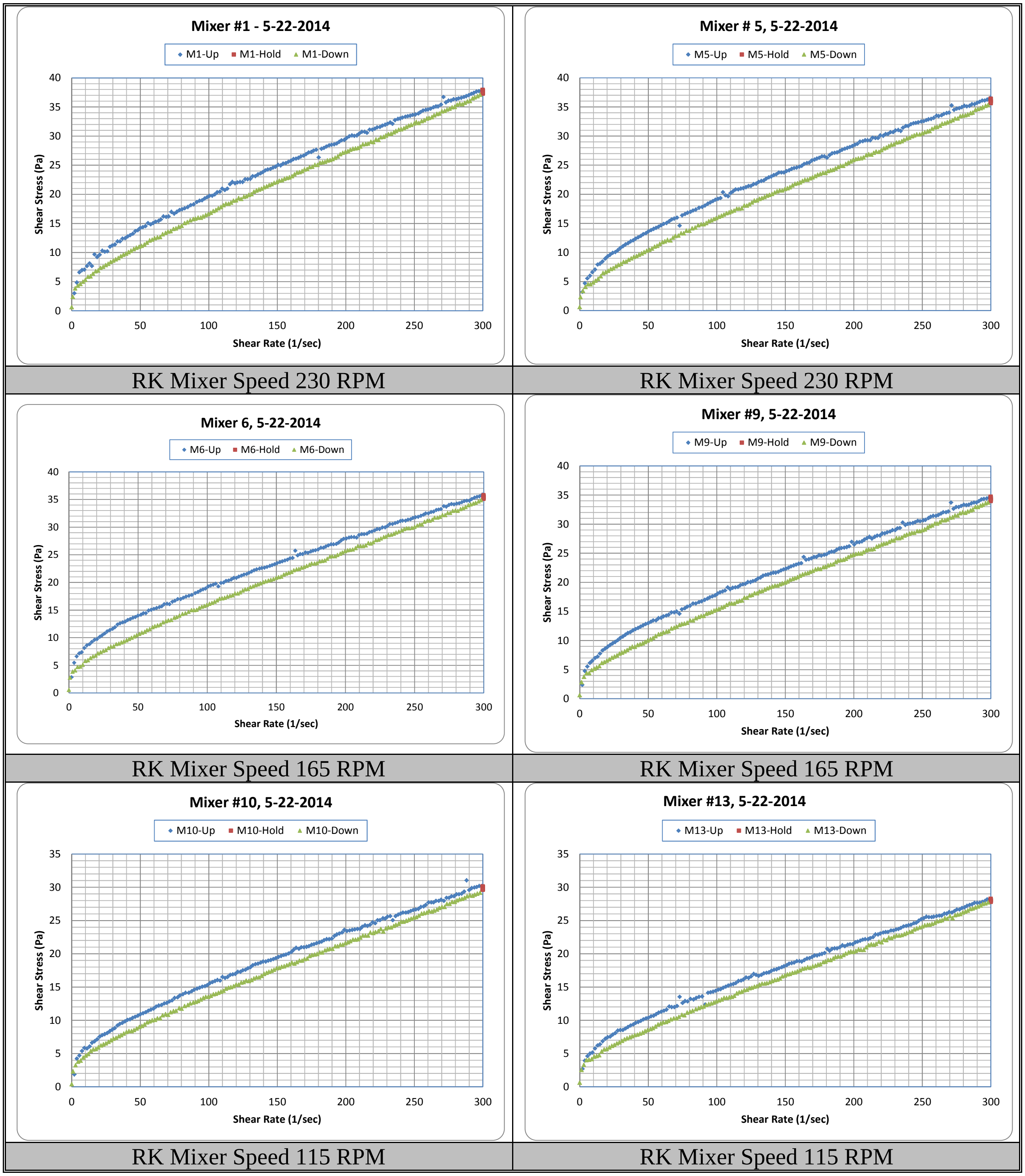


Figure B- 1. Flow Curves for RK Discharge: Samples 1, 5, 6, 9, 10, and 13

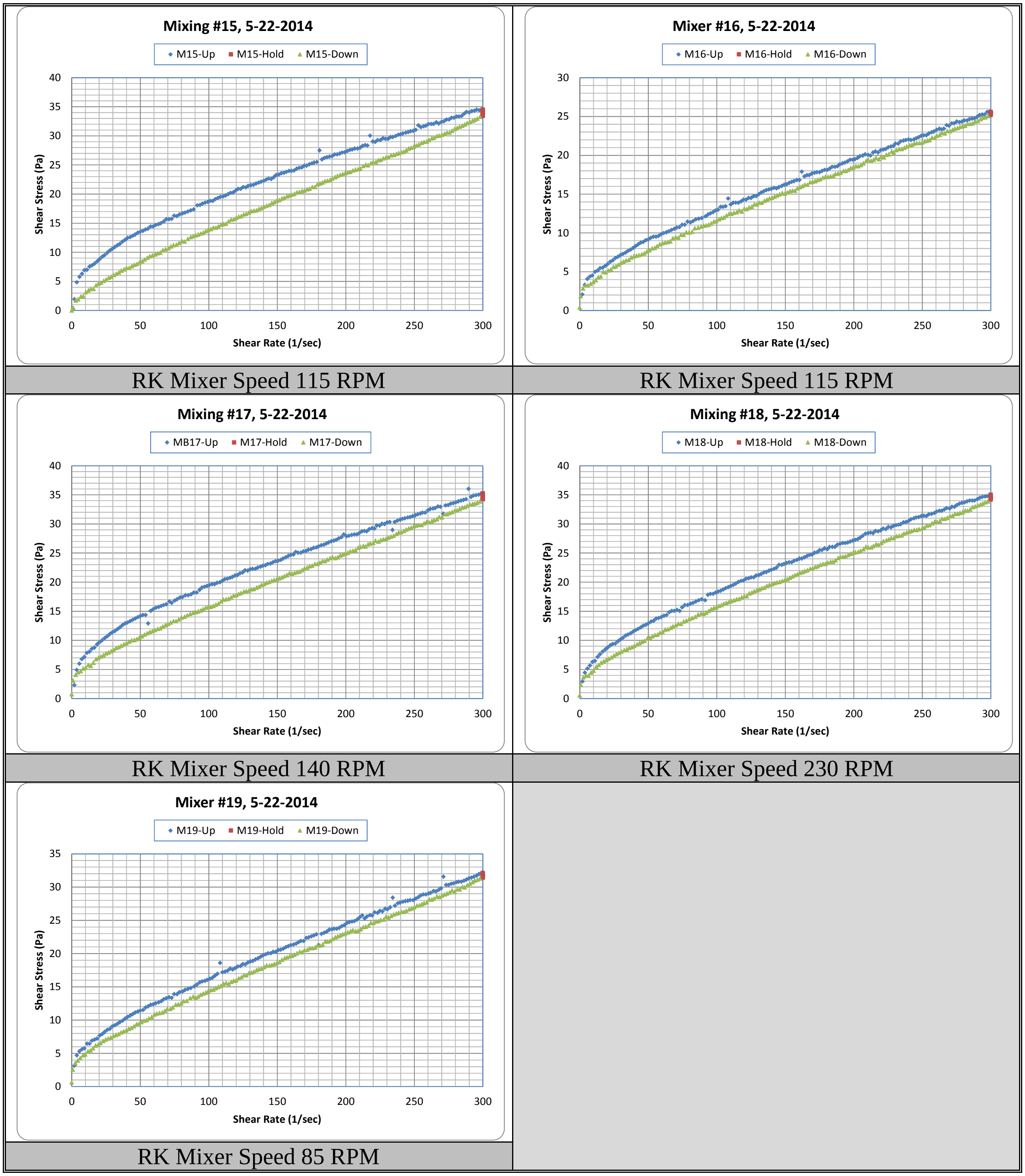


Figure B- 2. Flow Curves for RK Discharge: Samples 15, 16, 17, 18, and 19

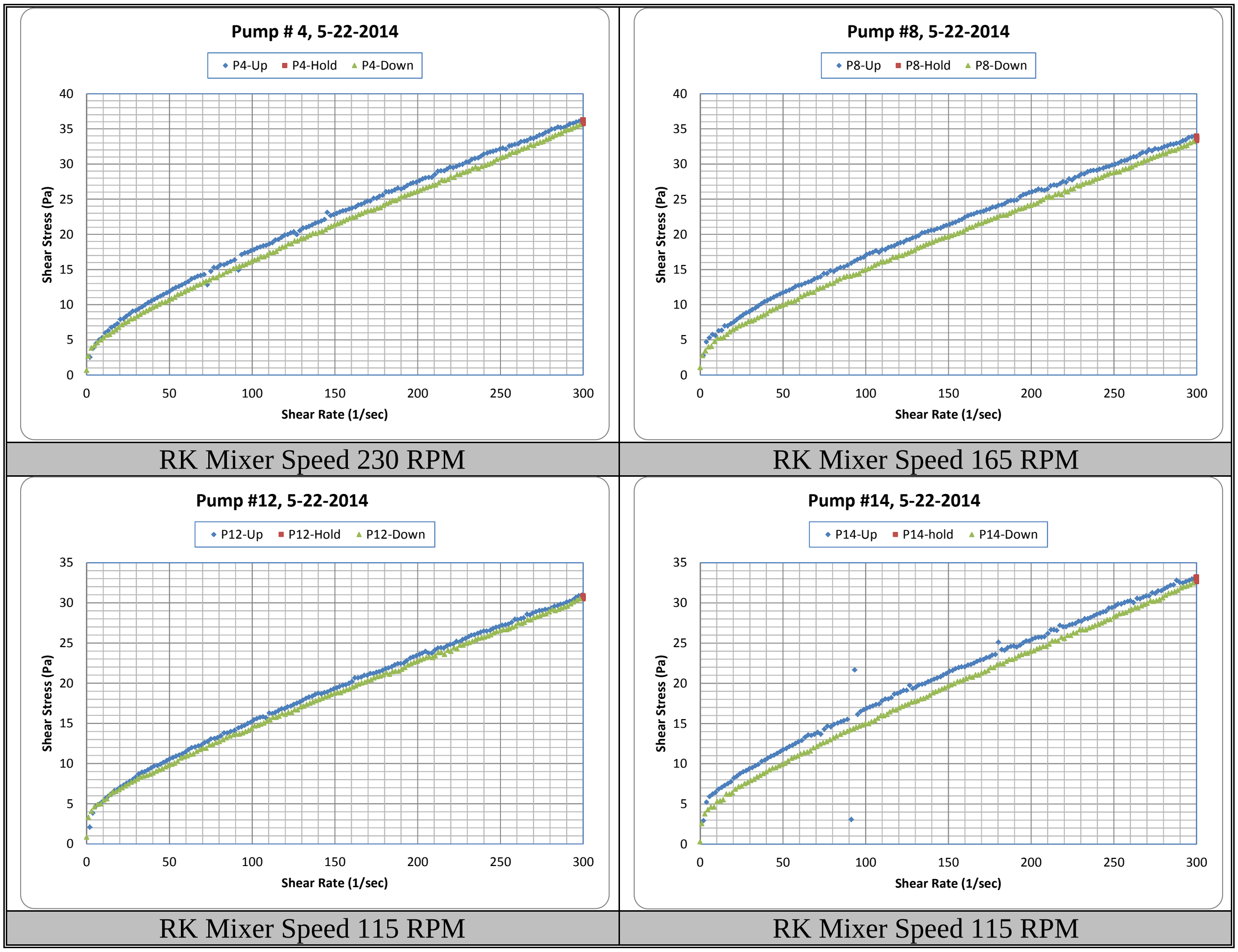
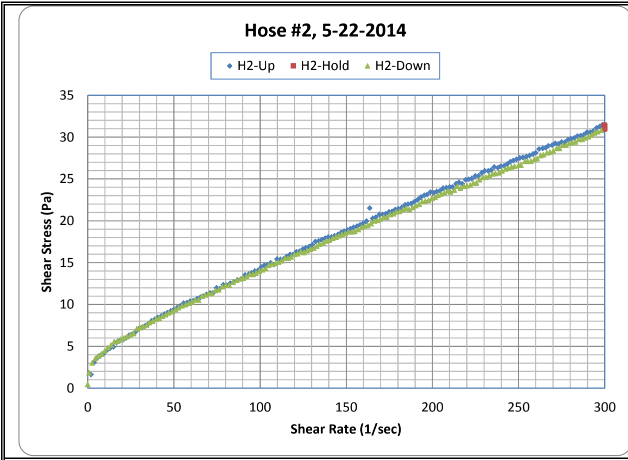
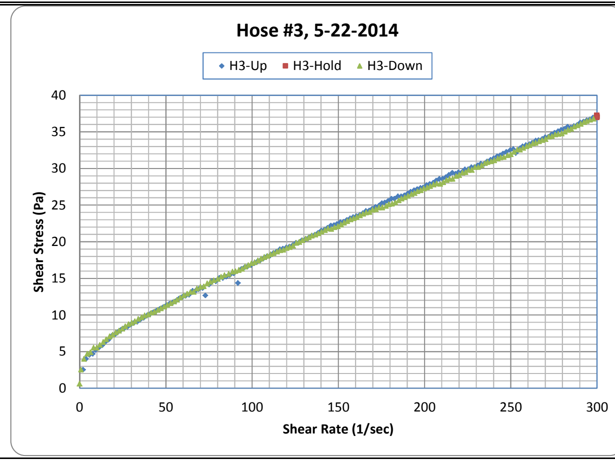


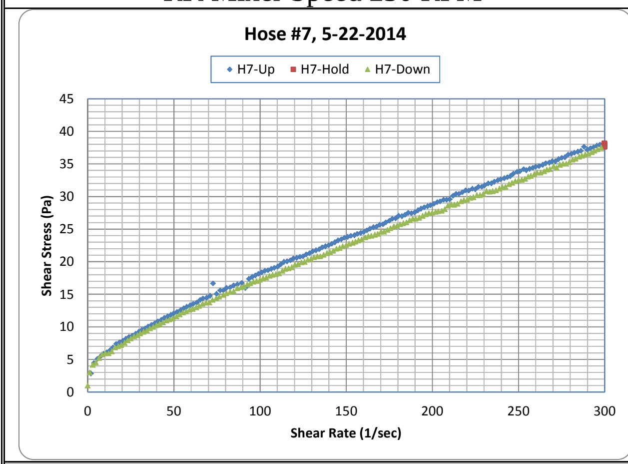
Figure B- 3. Flow Curves for Bredel SPX20 Hose Pump Discharge: Samples 4, 8, 12, and 14



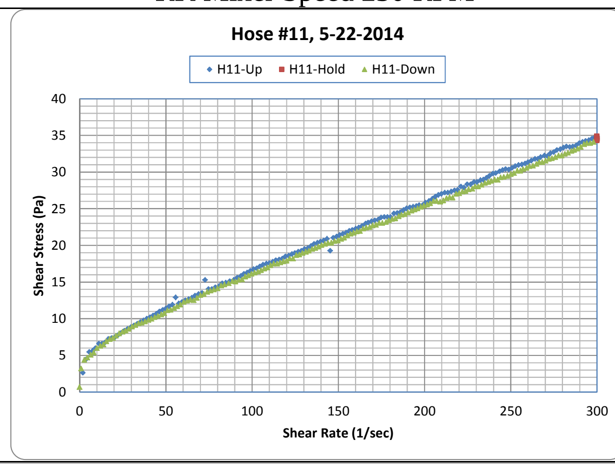
RK Mixer Speed 230 RPM



RK Mixer Speed 230 RPM

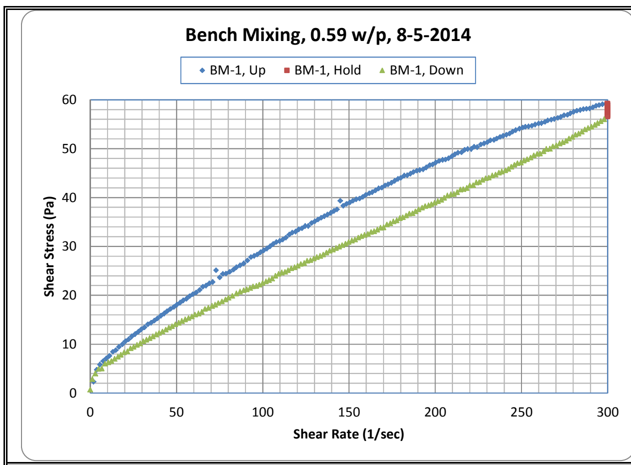


RK Mixer Speed 165 RPM

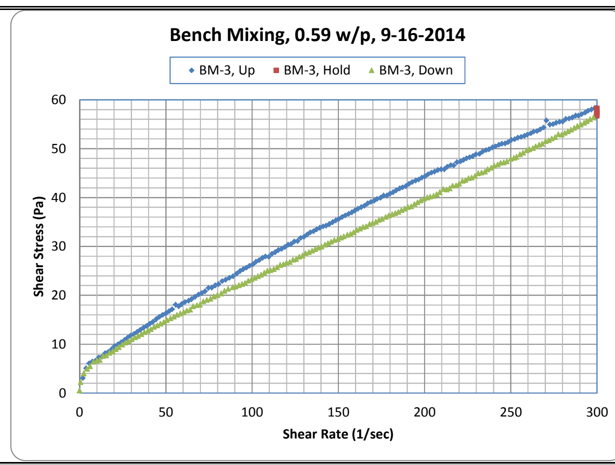


RK Mixer Speed 115 RPM

Figure B- 4. Flow Curve for Hose Discharge: Samples 2, 3, 7, and 11



Benchtop #1 Mixing Time = Cycle Time
(Sample 20)



Benchtop #2, Extended Mixing
(Sample 21)

Figure B- 5. Flow Curves for Laboratory Measurement: Samples 20 and 21

Distribution:

A. P. Fellinger, 773-41A
T. B. Brown, 773-A
D. H. McGuire, 999-W
S. D. Fink, 773-A
C. C. Herman, 773-A
E. N. Hoffman, 999-W
F. M. Pennebaker, 773-42A
W. R. Wilmarth, 773-A
Records Administration (EDWS)

J. S. Contardi, 766-H
V. Jain, 704-Z
J. N. Leita, 704-Z
S. P. Simner, 249-8H