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Evaluation Of Wetting Agents To Mitigate Dusting When Transferring Dry Glass Former Chemicals

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ABSTRACT

Plant design support for the US Department of Energy (DOE) River Protection Project (RPP) – Waste Treatment Plant (WTP) required pilot scale testing of the High Level Waste (HLW) glass former chemical (GFC) delivery system. A pilot facility was assembled at the Clemson Environmental Technology Laboratory (CETL) under the direction of the Savannah River National Laboratory (SRNL). Tests were performed using a representative HLW GFC blend to determine the behavior of the dry chemicals when transported through a chute and discharged into the enclosed head space of an agitated tank. The use of chute purge air, injected upstream of the point where the GFCs were added to the chute, was investigated. The pilot scale testing showed purge air was effective in reducing GFC holdup in the chute and that when the GFCs were discharged into the tank head space, dusting was evident during all transport conditions. This dusting lead to additional bench scale and laboratory scale tests that showed the addition of wetting agents to HLW and Low Activity Waste (LAW) GFC blends effectively mitigated dusting at the bench and pilot scales.

INTRODUCTION

A challenge that must be addressed when conveying chemicals in powder form is the control of dust that may be generated due to process conditions. The SRNL was initially tasked with investigating potential dusting issues related to the dry GFCs used in the vitrification of HLW at the RPP WTP HLW vitrification plant, where the GFCs are discharged from a hopper, gravity transported down a pipe, and are finally discharged into the enclosed head space of a mixing tank. Dust from the GFCs was very evident in the tank head space for all test conditions and determined to be an operational and safety issue.

The selection of wetting or agglomerating agents that can be added to the dry GFC blends to effectively mitigate dusting was demonstrated by laboratory scale and bench scale tests. The maximum permitted amount of wetting agent that can be added to the dry GFC blend is 10 wt%_G (water basis) of the mass of GFC blend to be processed. Both qualitative and quantitative measurements of the dust reduction were made on the un-wetted and wetted GFC blends.

The flowability of the resulting un-wetted and wetted GFC blends was evaluated by a “chute angle” test, which demonstrated the ability of the GFC blends to flow down a chute and discharge into an agitated tank of water. Generated dust in the enclosed tank vapor space was quantitatively measured to determine the effectiveness of the wetting agent used to mitigate dust generation as compared to the un-wetted GFC blends.

EXPERIMENTAL ASSEMBLY

Three separate tests were performed to determine the propensity of the GFC blends to generate dust. The first test was a pilot scale testing facility constructed at the CETL under the direction of SRNL. This pilot scale facility was sized on preliminary design input data and the scaling methods used for sizing the pilot facility are provided in reference 2. All scaling parameters were met, other than the elevation drop, which was limited at CETL due to the physical layout of the CETL facility. The objective of the 2nd and 3rd tests was to determine the effectiveness of added wetting agents to mitigate dust generation. The 2nd tests were bench scale tests, performed by SRNL and consisting of a counter-top food processor/mixer with auxiliary lighting and situated within a fume hood. The bench scale tests were used to determine the quantity of selected wetting agents required to mitigate dusting by visually observing for dusting reduction and elimination. The 3rd and final test was a laboratory scale testing facility constructed at the SRNL and was similar in design (although not to relative scale) to the pilot scale facility. The laboratory scale tested the selected wetting agents from the bench scale test to demonstrate the effectiveness of the wetting agent to mitigate dust as compared to an un-wetted GFC blend.

Pilot Scale Layout

The layout of the pilot facility at CETL is shown in Figure 1. The tank was constructed of reinforced fiberglass containing three plexiglass view ports; one on the top of the tank and two on the sides of the tank. The off-gas line and the GFC addition chute were located geometrically in the same location as that of the HLW Melter Feed Process Vessel (MFPV). The vertical sections of the GFC chute and vessel off-gas line were constructed of clear PVC pipe, allowing for observation of the flow behavior of the GFCs. The sloped section of the GFC chute was constructed from stainless steel pipe. Dry purge air was introduced to the tank vapor space and to the agitated liquid (via bubblers). The off-gas line contained two 0.2 μ m cartridge filters, a flow indicator, a vacuum pump, and a control valve.

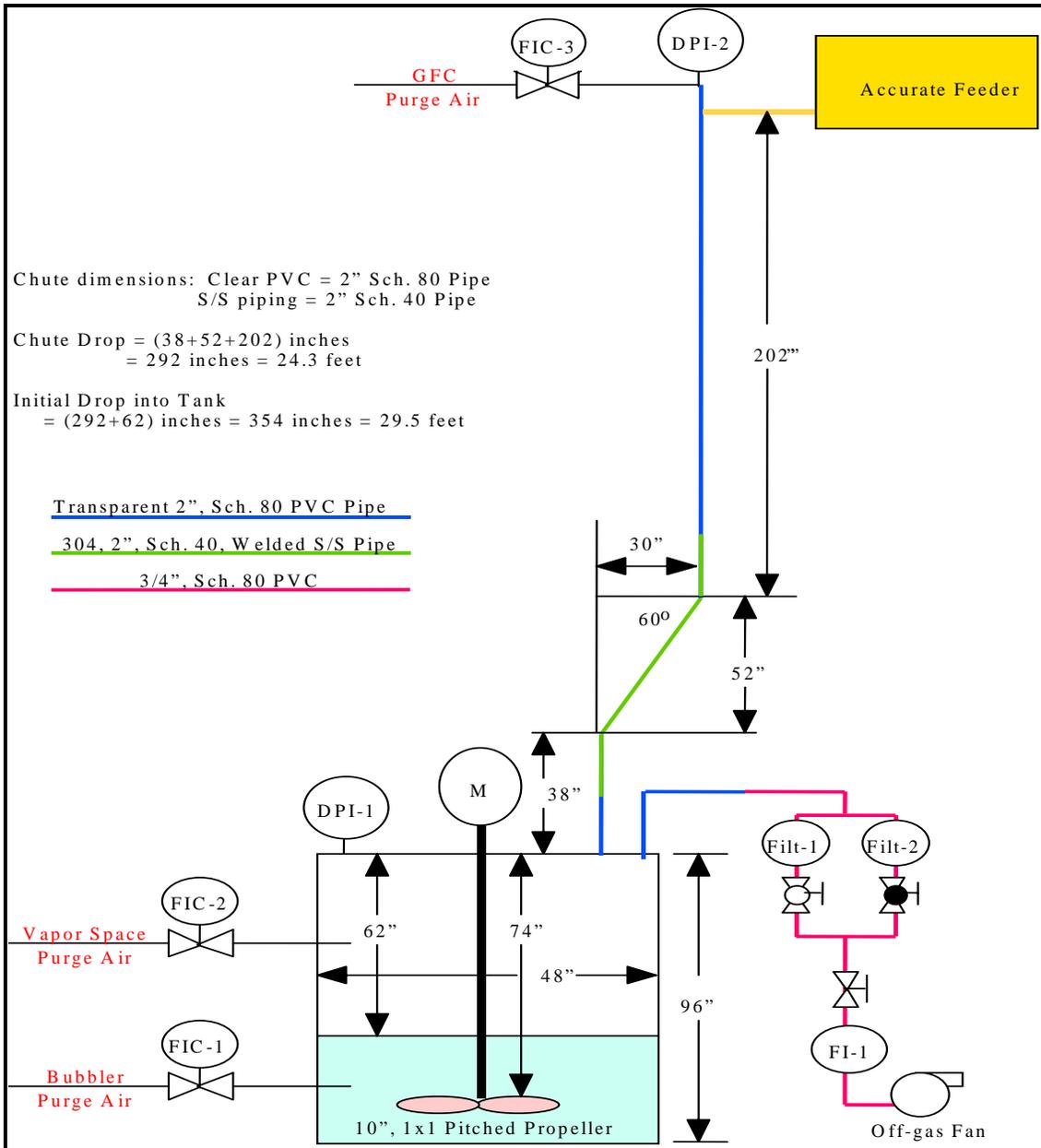
The instrumentation used in this pilot facility included a differential pressure gauge between the mixing tank vapor space and environment, air flow control regulators / indicators, and an off-gas line flow meter. A differential pressure gauge was also located upstream of where the GFCs were added to the GFC chute and the mixing tank vapor space to control the rate at which GFC purge air was added. The chute purge air, when used, was controlled to maintain the differential pressure between the mixing tank vapor space and GFC addition point into the chute close to zero. Details of the equipment and instrumentation utilized in the pilot scale facility are described in reference 2.

Pilot Scale Testing

During the operation of the pilot scale, two different tests were performed. The 1st test involved the delivery of the GFCs through the chute without the addition of purge air to the chute. The 2nd test was a duplication of the first, except chute purge air was injected immediately upstream of where the GFCs were added to the chute. The off gas system was operated such that the air additions to the enclosed tank (via bubblers and chute purge air) was removed to maintain a zero differential pressure between the tank head space and the environment.

In both tests, the pilot facility was maintained as a dry system. The tank walls, GFC chute, off-gas system, and other items that were not in contact with the agitated liquid contained little to no moisture on their exposed surfaces. The water used in these tests was allowed to come to thermal equilibrium with the environment prior to being transferred into the mixing tank. The bubbler purge air was not initiated until two minutes prior to the start of the individual tests. A representative AZ-101 HLW GFC blend (shown in Table 1) was used in both tests. No wetting agent was used.

The differential pressure gauge used between the point where the GFCs were added to the chute and tank vapor space was used to help regulate the GFC purge air that would provide a differential pressure of zero. Due to the limitation of the off-gas fan and the rotometers used to control the purge air additions, the liquid bubbler purge rate was limited to 1 SCFM and a maximum measurable chute purge air rate of 4 SCFM was achieved. With the GFC purge rate of 4 SCFM, there was still a slight negative pressure at the point where the GFCs were added to the GFC chute during the 2nd test.



The GFCs used in this task are the baseline raw materials approved by RPP-WTP Research and Technology (R&T) division [2]. The raw chemicals were procured and blended at ACTL using a mechanical tumbler. The GFCs were fed into the chute using a spiral auger Accurate® feeder.

Table 1: AZ-101 HLW GFCs Utilization and Blending Composition

Chemical	Vendor	Name – Grade	Wt. % of GFC in Blend	Moisture Content (wt. %) at 105°C
$\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$	U.S. Borax	10 Mole Borax – Technical	32.51%	12.02%
Li_2CO_3	Chemettal-Foote	Lithium Carbonate Technical, min 99%	8.88%	0.09%
Na_2CO_3	Solvay Mineral	Soda Ash Dense Anhydrous	9.10%	0.80%
SiO_2	U.S. Silica	Silica Sil-Co-Sil 75	47.48%	0.01%
ZnO	Zinc Corporation of America	Zinc Oxide Kadox-920	2.02%	0.01%

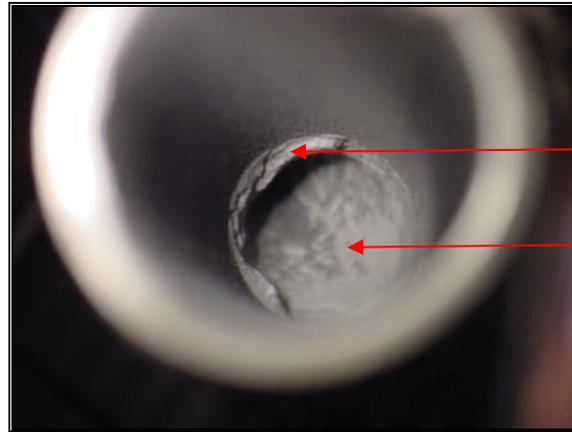
During each test, the GFC particles collected in the off-gas line and filter were analyzed for particle size distribution (PSD) and chemical composition at SRNL. The PSDs were obtained using MICROTRAC X100 and MICROTRAC SRA150 particle size analyzers, without sonification. The chemical composition was determined via inductively coupled plasma emission spectroscopy (ICP-ES) for the specific GFCs used in these tests.

Pilot Scale Results

GFC airborne particles (dust) were generated in both pilot scale tests. More dust was generated in the 2nd test that employed chute purge air as compared to the test without chute purge air and was confirmed both visually and by the amount of GFCs collected in the off-gas filters. GFC airborne particles were deposited in the off-gas line and off-gas line filter(s), adhering to internal tank surfaces and the agitator shaft, and were entrained in the water base agitated slurry.

The mean volume GFC particle size collected in the off-gas lines was 9.8 μm and 9.7 μm with chute purge air added and without purge air, respectively. The mean volume GFC particle size collected in the off-gas line filter was 3.5 μm for the test conducted with chute purge air added. The largest particles were between 50 to 60 μm . There was not sufficient material in the off-gas filter following the test without purge air to permit PSD or chemical analysis.

Counter flow of air and GFCs with that of falling GFCs were observed in the vertical section of the transparent Poly Vinyl Chloride (PVC) piping during both tests, and was much more severe in the case of no purge air due to the larger negative pressure at the point where the GFCs were added to the chute as compared to the case with purge air. Visual observation of the vertical section of the off-gas line indicated that some of the GFC particles were agglomerating and these agglomerated particles were falling back into the mix tank. Visual observation of the sloped GFC chute section (see Figure 2) revealed a build up of GFCs upstream of the first elbow during both tests. In the test without purge air, GFC build up was also present in the second elbow. The transparent PVC piping used in the vertical section of the chute showed no buildup of GFCs, other than where the GFCs were added to the chute. GFCs did accumulate on the transparent PVC in the vertical section of the off-gas line. Mass, PSD and chemical composition of GFC collected in the off-gas line is shown in Table 2.



Sloped bottom section of pipe discharging into vertical section of piping

Top of sloped section of pipe

Figure 2: Pilot Chute GFC Dust Accumulation

Table 2: Mass, Particle Size and Chemical Composition of Collected GFC in the Off-Gas System

		First Test		Second Test		
		Off-gas Line	Filter	Off-gas Line	Filter	
Mass of GFC collected in (grams)		6.03**	0.52	17.36	8.40	
Wt.% GFC	Na ₂ B ₄ O ₇ -10H ₂ O	5.1	*	5.5	3.5	
	Li ₂ CO ₃	4.6	*	5.1	3.6	
	Na ₂ CO ₃	1.6	*	1.5	0.7	
	SiO ₂	83.8	*	83.0	75.0	
	ZnO	4.9	*	4.9	17.2	
Particles size distribution (µm)	SRA150	Volume Range (>0.01%)	0.972 – 62.23	*	0.972 – 62.23	0.972 – 44.00
		Mean volume	9.682	*	9.834	3.465
		Number Range (>0.01%)	0.972 – 31.11	*	0.972 – 31.11	0.972 – 11.00
		Mean number	1.528	*	1.508	1.286
	X100	Volume Range (>0.01%)	0.972 – 62.23	*	0.972 – 52.33	0.043 – 26.16
		Mean volume	8.505	*	8.277	2.345
		Number Range (>0.01%)	0.972 – 22.00	*	0.972 – 22.00	0.043 – 3.889
		Mean number	2.079	*	1.856	0.162

* Sample could not be obtained from filter

** Value may be a little understated. Off-gas line was disturbed during testing.

Bench Scale Testing

Further testing was required to determine if the application of wetting agents would mitigate dust generation. An upper limit of water that could be added to the blended GFCs was set at 10% (by weight) of the GFC blend. The weight percent of wetting agent added to the mass of GFCs is denoted as wt%_G.

Bench scale tests were performed to screen potential wetting agent candidates [1]. The test was to determine the quantity of wetting agent required to mitigate dusting by visually observing for the reduction and elimination of dust generation. The bench scale test assembly consisted of a Braun™ K 1000 food processor, Model 3210, which was used as a dust generator and mixer to screen the wetting agents. This unit was set up with the mixing/kneading bowl and the internal drive arm mixing blade as shown in Figure 3. The mixing blade rotates around the periphery of the mixing bowl. A hole was drilled through the matching lid to facilitate application of the wetting

agent and the filling tube was left open to allow for observation of the dust created during the mixer operation. A weighed amount of blended GFCs was placed in the mixing bowl, the mixing blade inserted, and the lid installed. The speed of the Braun mixer was set and the power turned on. A Mikron™ fiber optic light source (150 watt – quartz halogen lamp) was placed perpendicular to the filling tube opening to illuminate a portion of the dust stream exiting the opening. A Sony™ Mini-DV digital video camera was placed 90° to the light beam and any dust generated during the test was observed onto a black background. The different GFC blends used are shown in Table 3, and the wetting agents tested are shown in Table 4. The HLW D, AZ101 MOD GFC blend replaced the borax used in the HLW D AZ101 blend with boric acid and sodium carbonate. The GFCs used in the bench scale test were batched into and blended using a rotary V blender (see Figure 4).

During each test, the wetting agent was added to the injection port (see Figure 3) in incremental amounts until there was no observed dust leaving the dust exhaust port. Incremental amount of wetting agent were also added during tests to determine the affects of over-wetting the GFC blend. At the completion of each wetting test, two 5 grams samples of the wetted GFC blend was obtained from the plastic bag and the agglomeration effectiveness quantitatively determined by sieve screening with an ATM Ultra Sonic Sifter. This technique utilized a stack of progressively finer sieves and ultra sonic vibration to drive the separation. An intermittent pulse to the screens was included to increase the rate of sifting. The sieve stack can include up to six ASTM E-11 sieves with the sieves becoming progressively finer towards the bottom of the stack. Each sieve and section, including the top latex diaphragm, the plastic cone, and the fines collector are weighed prior to the screening test. After the screening test was complete the sieves and sections were re-weighed and the difference in mass is that captured on the sieve or section. The mass collected in the top latex diaphragm, the plastic cone, and the fines collector were summed and are reported as less than 25 microns in size and considered as dust.



Figure 3: Bench Scale Assembly at SRNL



Figure 4: Rotary V-Blender

Table 3: LAW and HLW Blend Compositions for Bench Scale Testing

Glass Former Chemical	Weight Percent Distribution in GFC Blends		
	LAW A44, AN105	HLW D, AZ101	HLW D, AZ101 MOD
Kyanite, -325 Mesh Raw	4.26	0	0
Boric Acid, Tech. Grade Granular	18.35	0	21.60
Wollastonite, NYAD325	4.77	0	0
Iron Oxide, 5001	7.69	0	0
Lithium Carbonate, Tech. Grade	0	8.88	9.10
Olivine, #180 Grade	4.75	0	0
Silica, SiCoSil 75	42.65	47.48	48.65
Sodium Carbonate, Dense	0	9.10	18.58
Air Floated Rutile Ore, "94"	2.42	0	0
Zinc Oxide, Kadox 920	3.37	2.02	2.07
Zircon Flour, 325 Mesh	5.18	0	0
Sugar Granular	6.57	0	0
Boraxdecahydrate, Ten Mole	0	32.51	0

Table 4: Wetting Agents Evaluated in Bench Scale Tests

Wetting Agent Solution	Solution Components
Deionized Water	100% DI water
9.1 wt% Sodium Metasilicate	50 gr. SMS / 500 ml. DI water
28.6 wt% Sodium Metasilicate	200 gr. SMS / 500 ml. DI water
33.3 wt% Sodium Metasilicate	500 gr. SMS / 1000 ml. DI water
1.85 wt% Van Gel B	18.8 gr. Van Gel B / 1000 ml. DI water
2.91 wt% Van Gel B	15 gr. Van Gel B / 500 ml. DI water
3.38 wt% Van Gel B	17.5 gr. Van Gel B / 500 ml. DI water
16.7 wt% Sugar	100 gr. Sugar / 500 ml. DI water
4.76 wt% Boric Acid	25 gr. Boric Acid / 500 ml. DI water

Bench Scale Results

The initial LAW wetting agent tested was DI water, where 2.91 wt%_G of DI water mitigated dusting and is below the maximum of 10 wt%_G. Water impacts downstream processes by diluting the waste streams and increasing the water load on the glass melter. DI water was selected as the first wetting agent for laboratory scale test. The success of the DI water result allowed the selection of wetting agents to be limited to those that are expect not to impact downstream processes, specifically the rheological properties of the blended waste that will contain the GFCs. Of the additional wetting agents tested, the 1.85 wt% wetting agent in solution (wt%_S) Van Gel B solution performed well, was one of the more viscous (non-Newtonian) solutions tested, and was selected as the second wetting agent for the laboratory scale test. Both the DI water and 1.85 wt%_S Van Gel B solution drastically increased the size of the agglomerated particles, such that 98% of the agglomerated particles were greater than 53µm. The baseline LAW GFC blend PSD showed it comprised of 33% by volume of particles less than 53µm.

Additional testing of the 1.85 wt%_S Van Gel B solution and DI water at different wt%_G concentrations of the LAW batch mass were performed to determine the effect of applying excess wetting and to optimize the target wt% of wetting agent to mitigate dusting. As the wt% of the applied wetting solution was increased, the size of the agglomerated particles increased and the dusting decreased or was totally eliminated. Comparing the change in the agglomerated PSD for each wetting agent application against the baseline GFC PSD provides an indication of the effectiveness of the wetting agent. These tests helped determine the minimum wt%_G of the DI water and 1.85 wt%_S Van Gel B solutions to use in laboratory scale tests. The addition of excess wetting solution past the point of mitigating dusting did not provide additional benefit and could potentially cause processing problems, resulting in a sticky or cohesive blend (more cohesive).

The initial HLW wetting agent tested was DI water, where 6.0 wt%_G of DI water mitigated dusting and was selected as the first wetting agent for additional bench scale tests. Of the additional wetting agents tested, the 28.6 wt%_S sodium metasilicate (SMS) solution was much more effective in creating larger agglomerates and minimizing dusting than DI water at the same quantity (approximately 3 wt%_G) of applied solution. Testing of the HLW modified GFC blend showed that it reacted with water, generating heat, and over time seemed to dry out the GFC blend, making it more dust prone. This HLW modified GFC blend also tended to harden as it dried, thus making it unacceptable for processing and was not used for laboratory scale testing.

Testing of DI water and 33.3 wt%_S SMS solution at other wt%_G concentrations of the HLW batch mass were conducted. As the wt%_G of the applied wetting solution increased, the size of the agglomerated particles tended to increase. The use of excess wetting agent above the minimum required to mitigate dust did not provide any appreciable additional benefit in removing the dust.

Additional details from the bench scale testing are provided in reference 1.

Laboratory Scale Testing

A laboratory scale mixing and GFC delivery system was constructed to determine if dusting was mitigated and the improvement in the amount of dust mitigation between the baseline (un-wetted) and wetted GFC blends. This equipment was located in similar geometrical location, but not sized to scale, as that of the HLW MFPV design parameters. The layout of the laboratory scale test facility is shown in Figure 4. The addition of un-wetted and wetted GFCs into the chute was performed manually. The equipment and instrumentation used in the laboratory scale test facility at SRNL is described in detail in Reference 1.

A Ross™ blender (see Figure 5) was used to pre-blend the LAW and HLW batches. The Ross blender has a blending screw auger that orbits the conical vessel wall while it turns and gently lifts material from the bottom of the vessel and discharges the material to the surface. The Ross blender is not considered a high shear blender, such as the Braun blender used for bench scale testing. The individual GFCs for a given LAW or HLW batch were bagged separately given the wt. % distribution in Table 3, resulting in a total batch size of 28,350 grams for each laboratory scale test. The individual GFCs were added to the Ross blender, allowed to blend for approximately 20 minutes and then discharged while both the auger and orbital were moving.



Figure 5: Ross Vertical Mixer

Deionized (DI) water was proven in the bench scale testing to be effective in dust mitigation for both the LAW and HLW blends. 2.25 weight percent (wt%_G) DI water was applied to the LAW GFC blend to eliminate dusting, while the HLW GFC blend required only 5.0 wt%_G DI water addition. This quantity was shown to be effective in increasing the PSD to greater than 53 microns, which is the selected minimum particle size required based on the pilot scale tests. PSD comparison between the Ross mixer and Braun blender indicated that the Ross mixer was more efficient in creating larger particles given the same wt%_G of wetting agent used. This could be attributed to the fact the Braun is a higher shearing piece of equipment, hence reducing the size of some of the agglomerated GFC particles.

The batched (28,850 grams) GFCs were then placed back into the Ross mixer. The auger and orbital were turned on at maximum speed. The required mass of wetting agent was then applied to the GFCs using a garden spray nozzle set on a wide spray distribution. The wetting agent was applied to the top of the GFCs in intervals so as to allow the material to mix. The wetting agent was also added such that there were no puddles created that would result in larger agglomerations.

Table 5 provides the test conditions and measured results of the selected wetting agents.

The laboratory scale chute tests for the LAW GFC blend showed little difference in the flowability of the dry and wetted GFC blends through the stainless steel (S/S) chute. The wetted GFC blends were more cohesive than the dry GFC blends. The quantities of GFC material remaining in the chute for the dry LAW blend (76.1 grams) was at least an order of magnitude greater than for the wetted LAW GFC blends (8.2 grams). A similar reduction in material holdup was exhibited by the HLW GFC blends in the laboratory scale chute tests, where 16 grams of dry material remaining in the chute as compared to 8 grams of wetted HLW GFC material.

No dust was collected on the off-gas filters for the wetted GFC blends. For the un-wetted HLW GFC blend, 0.102 grams of dust was collected on the off-gas filters, and 0.485 grams of dust was collected on the off-gas filters for the un-wetted LAW GFC blend.

Table 5: Laboratory Operational Conditions and Measured Results

Detail	HLW			LAW		
	Baseline	5 wt% _G DI water	3 wt% _G of 33.3 wt% _S SMS	Baseline	2.25 wt% _G DI water	1.75 wt% _G DI of 1.85 wt% _S VG
Mass of wetting agent (g)	N/A	1417.5	852	N/A	638.4	496.1
GFC addition time (min)	N/A	15	10	N/A	10	*
Agitator speed (400 to 450 RPM)	Yes	Yes	Yes	Yes	Yes	Yes
Off-gas flow rate (2 L/min)	Yes	Yes	Yes	Yes	Yes	Yes
Off-gas pressure downstream of filter (psig)	0.28 to 0.40	0.32	0.32	0.34 to 0.52**	0.28	0.12
Time of GFC feeding (minutes)	9.48	8.42	8.28	7	7.42	8.0
Feed rate (lb _m /hr)	253	285	290	342	323	276
Mass of GFCs collected in chute (g)	16	13.1	8	76.1	3.1	8.2
Mass of GFCs collected in filters (g)	0.102	0	0	0.485	0	0
Concentration (g collected / L of gas)	0.0054	0	0	0.026	0	0

* Addition time was not recorded

** Ran on one filter until pressure was at 0.52 psi and then switched to the other filter.

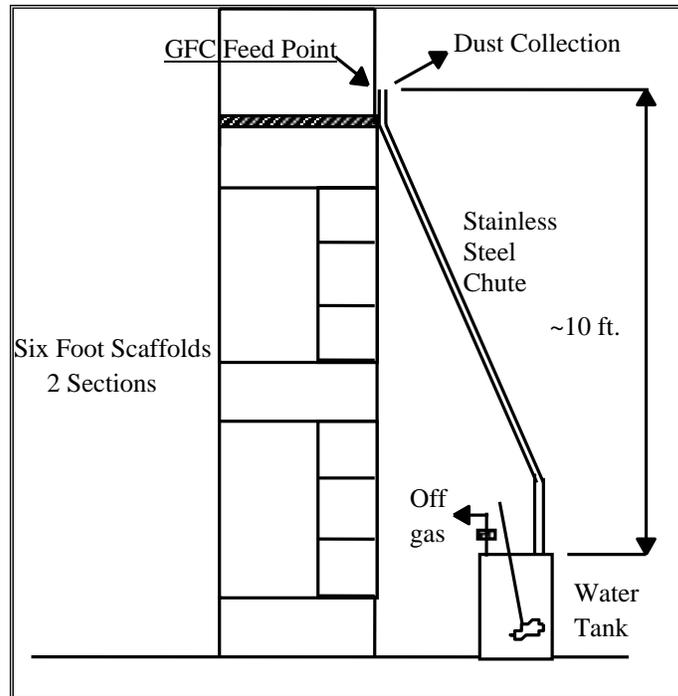


Figure 4: Laboratory Scale Facility Layout at SRNL

CONCLUSION

Counter flow of air and GFCs to that of falling GFCs was observed in the vertical section of the transparent PVC piping during pilot scale testing, and was more severe in the case where no purge air was added. The addition of purge air reduced the build up of GFCs at the elbows at both ends of the sloped piping section. In the test without purge air, GFC build up was also present in the second elbow. The transparent PVC piping used in the vertical section of the chute showed no buildup of GFCs, other than where the GFCs were added to the chute. GFCs did accumulate on the transparent PVC in the vertical section of the off-gas line. There was more dust generated when using purge air. As the purge air increased (from the 1st to 2nd test), the amount of GFCs captured in the off-gas line increased.

Bench scale testing as described in this paper is an effective means in determining the amount of wetting agent required to mitigate dusting, where dust is considered to be a particle with a geometric diameter less than 50 microns. Water addition is effective in dust mitigation when applied to the LAW GFC blend (2.25 wt%_G) and to the HLW GFC blend (5.0 wt%_G). The wetted GFC blends were more cohesive than the unwetted (dry) GFC blends.

The comparison of the LAW and HLW blends wetting tests indicates that different wetting agents and concentrations are required for the different GFC blends. Van Gel B solution was effective in reducing the quantity of wetting agent needed for dust mitigation on the LAW GFC blend. Dusting was effectively eliminated in the LAW GFC blend with the addition of 1.75 wt%_G of 1.85 wt%_S Van Gel B solution, as compared to 2.25 wt%_G deionized water needed for the same GFC blend. Likewise, SMS solution was effective in reducing the quantity of wetting agent needed for dust mitigation on the HLW GFC blend. 3.0 wt%_G of 33.3 wt%_S SMS solution eliminated dusting in the HLW GFC blend, as compared to 5.0 wt%_G deionized water needed for the same GFC blend. The impact using the Van Gel B and SMS on simulant or actual feed is unknown.

Laboratory scale chute tests for both the LAW and HLW GFC blends showed little difference in the flowability of the dry and wetted GFC blends going down the stainless steel (S/S) chute. The quantities of GFC material left in the chute for the dry LAW GFC blend was at least an order of magnitude greater than for the wetted GFC blends. However, the quantities of HLW GFC material left in the chute were comparable between the dry and wetted HLW GFC blends. Based upon visual observations (both bench and laboratory scale) and weighing the filters used for the laboratory scale tests, no measurable dust was measured for the wetted GFC blends. However, dust was collected on the filters for both of the dry GFC blends. Approximately 4 times the mass of dry LAW GFC blend was collected on the filter as compared to the dry HLW GFC blend given essentially the same operating conditions.

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