

**Process Enhancements to Improve Overall Throughput in the Defense  
Waste Processing Facility at the Savannah River Site - 11458**

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**ABSTRACT**

Savannah River Remediation (SRR) LLC is currently pursuing an aggressive program to empty its High Level Waste (HLW) tanks at the Savannah River Site (SRS). To accommodate this program, downstream processes, such as the Defense Waste Processing Facility (DWPF) and the Saltstone Production and Disposal Facilities, must improve overall throughput. To coincide with initiatives addressing improvements in salt processing, sludge processing enhancements have commenced which aim to improve the overall throughput in the DWPF. Specifically, three major enhancements have been proposed including the incorporation of an alternative reductant in the Chemical Process Cell (CPC), as well as two separate tasks addressing the reduction in water currently introduced during CPC processing. These enhancements promise to improve overall cycle time associated with melter feed preparation and reduce facility downtime.

**INTRODUCTION**

Commitments for bulk waste removal and subsequent waste tank closure at the Savannah River Site (SRS) require fundamental flowsheet changes to downstream processes to accommodate expeditious treatment and disposal of salt and sludge waste. Increased throughput is necessary to address waste tank space management while in operation as well as to decrease the overall mission life of the SRS. Specifically, initiatives have been identified to improve salt waste processing throughput at SRS [1]. Similarly, enhancements have been proposed which address increased sludge waste processing. In general, an increase in sludge waste processing requires improvements in bulk waste removal, sludge batch preparation and qualification, and final disposal. Though a number of initiatives are currently underway to address each of these areas, this paper will focus on three specific enhancements related to improvements in final disposal at the DWPF.

**Facility Overview**

The current DWPF flowsheet is shown in Figure 1. Nominally, 22,500 liters of sludge is received on a batch-by-batch basis in the Sludge Receipt and Adjustment Tank (SRAT) from a 3.7 million liter feed tank. The sludge is chemically adjusted in the SRAT via addition of concentrated nitric and formic acids. The purpose of the chemical adjustment is to acidify the incoming sludge to adjust the rheological properties to improve processing, remove mercury from the sludge feed,

and to prepare the sludge feed for melter operation (e.g. maintain control over the oxidation state of the glass). The SRAT also receives and processes by-products from salt processing, namely an actinide-rich stream containing primarily monosodium titanate solids as well as a cesium-rich dilute nitric stream. Following chemical adjustment and concentration in the SRAT, the sludge material is transferred to the Slurry Mix Evaporator (SME) where the material is blended with frit (glass former). There are two sources of frit additions made during the SME process including a bulk process frit addition, and frit recycled from the canister decontamination process. The SME represents a hold point in the process to ensure the contents will produce acceptable glass (based on statistical process control rather than statistical quality control). If the blended SME material is deemed acceptable, the material is transferred to the Melter Feed Tank (MFT). The MFT represents a transition in the process from a batch to continuous process, as the MFT continuously feeds the Melter.

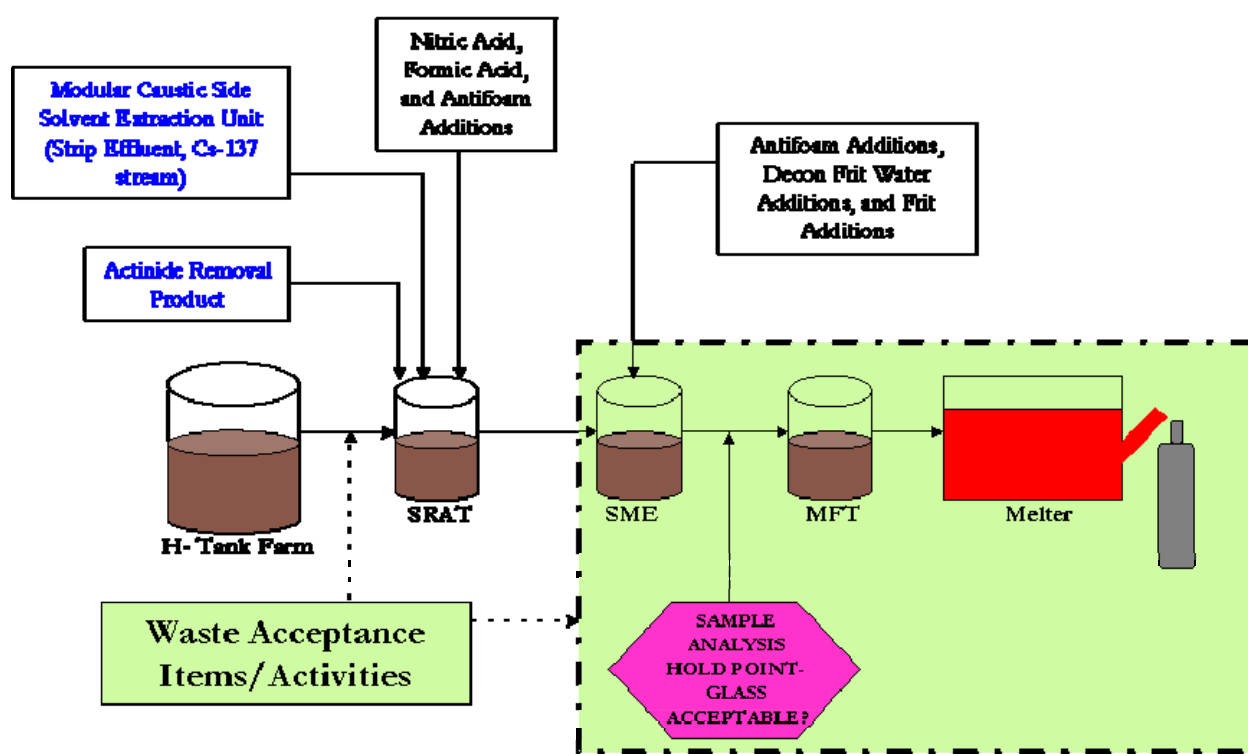


Fig. 1. Schematic providing an overview of the current Defense Waste Processing Facility flowsheet.

Overall waste throughput at the DWPF is limited by the slowest portion of the process. Prior to the installation of bubblers in the Melter in September of 2010, waste throughput at the DWPF was limited by Melter operations. However, the CPC (i.e. SRAT and SME operations) currently represents the rate-limiting step in the overall process. Thus, process enhancements discussed in this paper specifically address CPC operations. Each of the process enhancements is discussed in more detail below, including the purpose of the enhancement, the benefits each has on the current DWPF flowsheet, as well as an update on the status of each enhancement.

## **ALTERNATE REDUCTANT**

The purpose of the alternate reductant task is to eliminate or minimize the amount of hydrogen generated from the catalytic decomposition of formic acid by either directly replacing formic acid with an alternative reductant, or minimizing the amount of formic acid in the current flowsheet. The primary driver for introducing an alternate reductant is to decrease CPC cycle time as well as facility downtime. The formic acid induced hydrogen generation requires a purge source on the affected CPC vessels, which in turn limits the boil-up rate. Fundamentally, if the purge volume can be decreased in response to a decrease in flammable vapor present in the CPC vessels (i.e. hydrogen), the steam rate capacity in the CPC vessels can increase. Higher overall steam rates reduce the total amount of time required for boiling off excess water introduced to the SRAT and SME, including concentration of sludge feed and processing large volumes of by-products from salt processing. Furthermore, the current flowsheet requires operation of a safety class on-line gas chromatograph system to monitor off-gas conditions in the CPC. Implementation of a reductant alternative to formic acid in the current flowsheet may allow relaxation of the safety classification of the gas chromatographs, decoupling operation of the gas chromatographs with SRAT and SME processing and potentially reducing overall facility downtime.

### **Preliminary Evaluation and Selection of Alternative Reductants**

Aside from the benefits anticipated from implementation of an alternate reductant, there are a number of processing objectives which provide additional selection criteria for alternate reductant candidates. Primarily, an alternate reductant must exemplify the ability to maintain control of the glass oxidation state. Melter feed which is too oxidizing can result in excessive foaming in the Melter, potentially resulting in several significant problems such as blockage of critical Melter off-gas components, inadvertent glass pouring, and formation of an insulating foam layer on the melt pool surface. Tests have shown that foaming is mitigated when reducing agents, such as formic acid, are added to the melter feed stream. However, melter feed which is too reducing could cause precipitation of copper and noble metals out of solution, potentially providing an alternate current path for the Melter electrodes and severely reducing the ability to maintain melt pool temperature. Additionally, an alternative reductant must maintain feed processability. Specifically, the reductant must maintain acceptable rheology of the feed and must not introduce degradation products or additives which unnecessarily complicate CPC or Melter operation. Lastly, the original design basis of the facility was to address mercury removal in the CPC as opposed to the Melter. As such, the Melter off-gas system is currently unable to accommodate removal of large quantities of mercury. The preference is thus to maintain removal of mercury during CPC processing.

Based on well-defined facility constraints and project goals, the Savannah River National Laboratory (SRNL) identified and evaluated potential acidic and non-acidic reductant candidates [2]. Each reductant was evaluated by performing simplified bench-scale tests with supernatant (resembling SRAT supernatant following nitric acid addition) spiked with nominal amounts of mercury as a pre-cursor to more formal testing. The only direct alternative that successfully reduced mercury in the supernatant system was acrylic acid. However, significant material handling issues (i.e. self-polymerization) in excess of those associated with formic acid prevent acrylic acid from providing a viable direct replacement.

The inability to identify a direct replacement for formic acid in the current DWPF flowsheet led SRNL to conduct a series of informal small-scale SRAT runs using varying formic/nitric ratios in an attempt to determine the minimum required formic acid addition for mercury reduction. Results from the small-scale testing indicated that reduction of mercury was possible with significantly less formic acid (on a molar basis) than is added with the current flowsheet. In principle, a strategy may be available to add minimum formic acid to reduce mercury, while an alternative reductant is utilized to balance the oxidation state of the melter feed, thus achieving project goals related to minimizing hydrogen generation while maintaining facility constraints related to balancing the oxidation state of the melter feed. Based upon previous testing at the Pacific Northwest National Laboratory (PNNL) [3] as well as testing on the supernatant system, glycolic acid was utilized in conjunction with nitric acid and the minimum formic acid amount to maintain control of the oxidation state of the glass. Preliminary test results based upon small-scale testing indicated successful mercury removal of a balanced oxidation state system using an 80:20 blend of glycolic and formic acids (on a molar basis).

In addition to the success of the glycolic acid based flowsheet, studies have previously been conducted exemplifying sugar as a successful melter feed reductant for the DWPF process as well as other vitrification programs [2]. Though successful as a reductant for high level waste vitrification, the use of sugar as a direct replacement for formic acid does not result in the reduction and subsequent removal of mercury during the CPC process, requiring significant facility modifications to the Melter off-gas system to accommodate collection and removal. As such, direct replacement of formic acid with sugar was eliminated as a feasible option [4]. However, driven by facility constraints, Vitreous State Laboratory (VSL) and EnergySolutions identified an additional sugar-based flowsheet for consideration. To appropriately address mercury removal in the CPC, minimum formic acid was proposed in combination with sugar to adjust the oxidation state of the melter feed.

Upon identifying the alternate reductant flowsheets most conducive to achieving project goals and facility constraints, testing was initiated to further define each flowsheet. To accommodate schedule commitments associated with implementation of the alternate reductant and to take maximum advantage of resources, SRNL undertook developing the glycolic-based flowsheet while VSL undertook the sugar-based flowsheet. To ensure consistency between the laboratories, sludge simulant with bounding quantities of noble metals and mercury was procured from an off-site vendor and provided to both laboratories for testing. In addition to the respective flowsheets, simulant testing utilizing the current DWPF flowsheet (i.e. nitric/formic) was performed by both laboratories to provide a basis for comparison for each laboratory, and more importantly, to compare the alternative flowsheets. The simulant make-up was chosen to specifically challenge the alternative flowsheets in terms of hydrogen generation and processability. A summary of experimental results are provided for each flowsheet in the next sections.

### **Status of the Glycolic-based Flowsheet**

Details surrounding development of the glycolic-based flowsheet are provided in a summary report [5]. Testing with the baseline nitric/formic flowsheet proved difficult with respect to meeting existing DWPF processing criteria. While nitrite was successfully destroyed and mercury was removed, the rheology of the SRAT and SME products were well above design basis limits (for existing pumps and agitators) and hydrogen generation far exceeded the DWPF

limit (0.295 kg/hr, or 0.65 lbs/hr) at nearly 0.73 kg/hr [1.6 lbs/hr]. For comparison, subsequent runs with the nitric/glycolic/formic flowsheet (at a nominal 80:20 molar blend of glycolic and formic acids) successfully removed mercury with almost no hydrogen generation (<0.02 kg/hr) and SRAT and SME product rheology which fell within current DWPF processing criteria. Thus, though experience is limited, the nitric/glycolic/formic flowsheet appears to outperform the current flowsheet in many regards, including off-gas generation, mercury removal, and general processability [5].

Subsequent to the initial comparisons, SRNL performed tests to examine the sensitivity of the process to variations in acid stoichiometry (total acid amount). Specifically, the total acid amount added was varied from the stoichiometric amount to quantities in excess of the stoichiometric amount (up to 200% of the calculated amount). In all cases, mercury removal exceeded benchmarks set by the baseline run. As expected, hydrogen generation increased with increasing total acid, though always in quantities at least an order of magnitude less than the baseline (i.e. nitric/formic) run. Additionally, tests were performed with various molar blends of glycolic and formic acids ranging from 40% to 100% glycolic (all at constant total acid). Surprisingly, all blend ratios including the test with 100% glycolic were successful in reducing mercury, with hydrogen generation increasing as the blend ratio shifted towards formic acid. Preliminary test results from varying total acid amount and blend ratio indicate the possibility of altering parameters to optimize the glycolic-based flowsheet.

Observations during testing indicated several outstanding issues which require further attention to fully develop the nitric/glycolic/formic flowsheet. Particularly, additional work is required to fully understand the impact of the new flowsheet on controlling the glass oxidation state. Though oxidation state targets were established for each run, none of the preliminary runs produced glass which accomplished this target. Improvements on glass reduction/oxidation state control is likely to occur as understanding of the flowsheet chemistry improves. Additionally, refinement of the analytical method used to evaluate glycolate (including an overall improvement in the accuracy) would be beneficial to controlling the oxidation state of the glass. An additional observation made during flowsheet testing was dissolution of iron, which increased with increasing glycolic/formic blend ratios. Iron solubility during DWPF processing is a potential concern because it is presently credited for criticality control since it is assumed (under the current flowsheet) that both iron and plutonium remain relatively insoluble during processing. Thus, evaluating the extent of metal solubility (particularly iron) during processing becomes important to fully develop and ultimately implement the nitric/glycolic/formic flowsheet. A summary of the hydrogen generation and iron solubility as a function of the glycolic/formic blend ratio is provided in Figure 2. Once again, it is apparent that parameters (in this case, the glycolic/formic molar ratio) are available to optimize the flowsheet for DWPF processing. For example, a higher percentage of glycolic acid in a glycolic/formic blend results in higher iron solubility, which compromises the presence of iron as a neutron poison in the CPC, but produces less peak hydrogen generation.

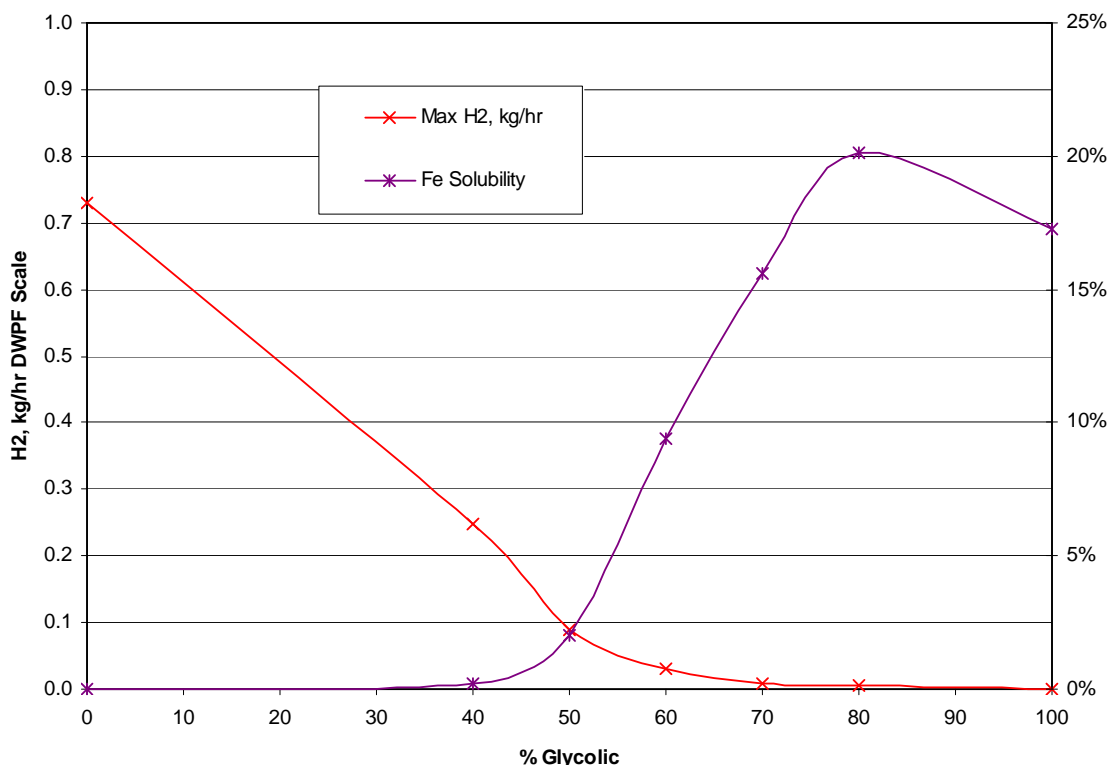


Fig. 2. Peak hydrogen generation (in kg/hr) and iron solubility (mass basis) plotted as a function of the percentage of glycolic acid (molar basis) in a glycolic/formic blend.

### Status of the Sugar-based Flowsheet

Data generated from the development of the sugar-based alternate flowsheet is provided in a summary report generated by VSL/EnergySolutions [6]. Testing of the sugar-based alternative flowsheet was established based upon results from a series of bench-scale screening tests performed at VSL on a simplified waste simulant. These tasks were directed primarily at assessing the qualitative variation of mercury reduction as a function of acid addition strategy. The bench-scale testing evaluated both 3-cycle (in which nitric acid was added until a pH value of ~10, followed by addition of formic acid, followed by a subsequent nitric acid addition to a prescribed final pH value) and 2-cycle (in which all of the nitric is added first, followed by the formic acid addition) acid addition approaches, a range of final pH values, and a range of formic acid additions. The bench-scale screening tests identified the 3-cycle acid addition approach as more effective for removal and recovery of mercury. Thus, the 3-cycle acid addition was chosen as the primary approach for small scale SRAT and SME testing, with key variables including the pH endpoint as well as the amount of formic added (relative to the baseline flowsheet).

Similar to SRNL, the baseline flowsheet was found to be difficult to process, particularly with respect to rheology during SRAT and SME processing. Hydrogen generation during the baseline run peaked at 0.42 kg/hr [0.92 lbs/hr], with off-gas characteristics similar to those observed during baseline testing performed at SRNL. Testing with the proposed 3-cycle acid addition

strategy with a target pH endpoint of 5 resulted in significantly less peak hydrogen generation (<0.01 kg/hr) compared to the baseline as well as improved mercury reduction. Subsequent runs utilizing the 3-cycle approach at different pH endpoints resulted in insufficient mercury removal at lower pH values, and difficulty processing due to unacceptable slurry rheology at higher pH values. Regardless of the pH endpoint value, the maximum hydrogen generation observed never exceeded 0.01 kg/hr. As such, a pH endpoint value of 5 was determined to be optimal in terms of facility constraints (i.e. hydrogen generation and rheological limitations). Testing was also performed to determine the effect of varying amounts of formic acid. The amounts evaluated ranged from 20-40% of the total amount of formic added during the baseline run. As expected, as the total amount of formic acid increased, the peak hydrogen generation increased (though still significantly below the baseline value), but yielded no apparent improvements in mercury removal.

In all runs, sugar was added to the SME product to control the oxidation state of the glass. Sugar additions were made based upon a preliminary convention in which the total moles of organic carbon balance the total moles of nitrate. This convention was used since the precise stoichiometry of the reactions involved was neither known nor constant under the conditions prevailing in the Melter. Nonetheless, using this convention, a sugar ratio was established such that a ratio of 1.0 corresponded to one mole of sucrose per eight moles of nitrate. Sugar was added to SME product samples to achieve a range of sugar ratios from 0.5 to 1.2 based upon the analyzed nitrate, nitrite and formate content. These samples were then melted using the SRNL closed crucible procedure and the resulting glass products analyzed for the oxidation state using Mossbauer spectroscopy. The results of the oxidation state testing are shown in Figure 3.

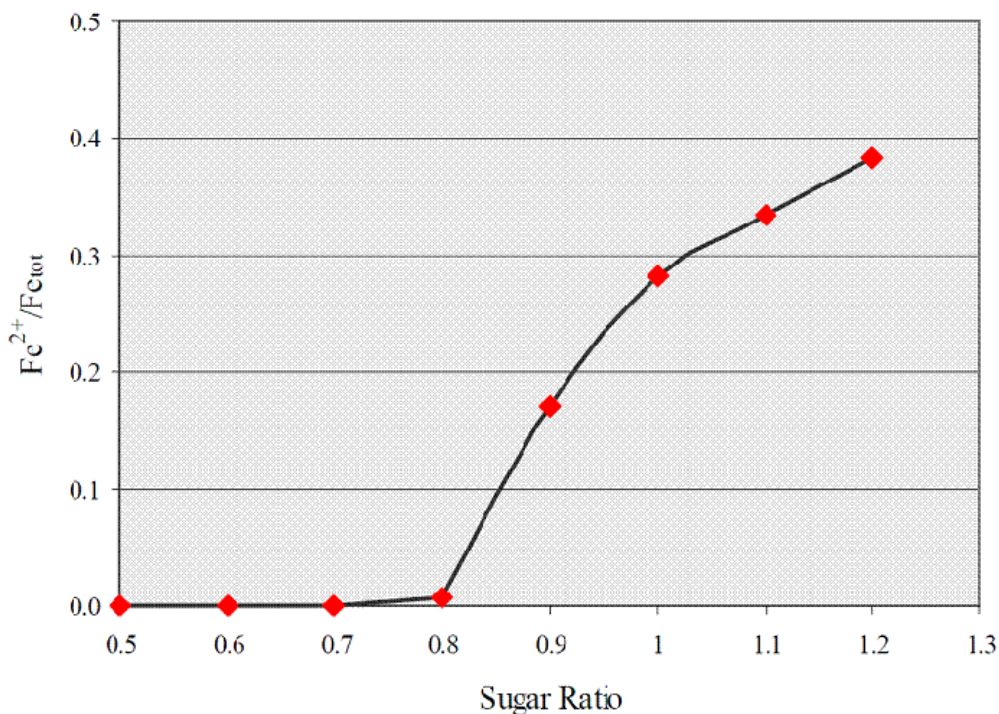


Fig. 3. Measured glass oxidation state (defined as the ratio of the concentration of iron (II) to total iron (Fe) in the feed stream) as a function of the sugar ratio for the nitric/formic/sugar flowsheet.

Outstanding issues related to the sugar-based flowsheet include control of the oxidation state of the glass as well as the impact of sugar additions on the rheological properties of the melter feed. Control of the glass oxidation state may prove difficult based upon the observed relationship between the sugar ratio and glass oxidation state (see Figure 3). The typical DWPF target for the glass reduction/oxidation state is  $Fe^{2+}/Fe_{tot}=0.2$ , which appears to lie on the steepest portion of the curve, potentially making accurate control difficult as small errors in measurement of nitrate concentration, feed volume, or sugar addition amount could lead to significant change in the glass oxidation state. Furthermore, the impact of sugar on melter feed is unknown. Of particular concern is the impact of vessel heels, though preliminary testing indicates that the impact of sugar on feed rheology is minimal.

### **Path Forward for Alternate Reductant**

Based upon information received from the flowsheet development efforts described above, primary flowsheet parameters will be identified for both the glycolic-based and sugar-based flowsheets to facilitate generation of melter feed. Melter feed will be prepared for each of the flowsheets (including the baseline flowsheets) using larger scale reactors at the SRNL. Melter feed will then be shipped to VSL to perform testing on the DM10 melter system. The goal of the scaled melter testing is to obtain melter off-gas data for evaluation. The melter off-gas data from each flowsheet will be coupled with information gained from the CPC simulations to enable a down-select between the available options. Down-select criteria have been established considering technical feasibility as well as the related business impacts. The flowsheet down-select will be a joint effort consisting of representatives from SRNL, VSL, *EnergySolutions*, and DWPF. Following down-select, the selected flowsheet will undergo further development for facility implementation.

### **REDUCTION IN WATER**

Two separate tasks are currently on-going to reduce the total amount of water introduced to the CPC, including 1) a task to replace the current slurry-fed bulk frit transfer design with a dense phase (dry) conveying system, and 2) implementation of a water separation technology to remove water from the decontamination frit slurry. The benefits of these tasks include a reduction in the overall water returned to the Tank Farm, thus reducing burden on the Tank Farm evaporators, and an overall reduction in the SME cycle time related to a reduction in the total time required to boil off water from these streams. In addition to these benefits, the water reduction tasks provide the ability to accommodate future by-products from the Salt Waste Processing Facility (SWPF). Each of the tasks is described briefly in the following sections.

#### **Dry Bulk Frit Addition**

The current flowsheet relies upon a slurry-fed bulk frit addition to transfer frit material from outside the 221-S facility directly to the SME. The resultant slurry is approximately 35wt% frit prior to introduction to the SME vessel, resulting in approximately 7600 liters [2000 gallons] of water added to the SME process on a batch by batch basis. This water is subsequently boiled off during the SME process and eventually returned to the Tank Farm as recycle water. A dry frit feed system has been proposed to reduce overall recycle water volume and to reduce SME cycle time. A number of commercially available granular feed technologies were considered based upon facility constraints as well as restrictions imposed by the system itself. For example, in the



absence of a carrier fluid (such as water), the frit utilized in the DWPF process has been shown to be conducive to line pluggage and premature component erosion due to the irregular morphology of the frit particles. Thus, options such as dilute phase conveyance, which rely upon large volumes of air at high velocities were disregarded based upon concerns with erosion. Instead, dense phase conveyance (in which slugs of material are transferred using a pressurized vessel) was chosen as an appropriate technology for the DWPF application.

Preliminary vendor testing was conducted to provide proof of concept for three different dense phase conveying systems. Vendors were provided facility drawings to facilitate mock-up of the proposed system and were supplied with frit to evaluate the transport ability of the systems. Though proposing slightly different equipment setup, each vendor successfully demonstrated transport of dry frit material using dense phase conveyance. No significant transport issues were identified during testing. A Request for Proposal (RFP) has been submitted to each vendor for cost and schedule associated with facility implementation of the respective equipment setups.

In addition to identifying the dense phase conveying technology, several technical considerations were addressed related to facility implementation. Piping and system component erosion is a concern based upon facility experience with frit handling. Thus, SRNL performed testing to determine materials compatibility with pneumatic transfer. A sand blasting cabinet was utilized to evaluate various materials, including alumina, basalt, and vulcanized rubber, to determine a material most resistant to piping erosion. Based on results from the scoping tests, alumina was determined to be the best erosion resistant material for pipe lining when considering pneumatic frit transfer [7]. In addition to component erosion, the use of water to mitigate the potential for excessive dust generation in the SME vapor space introduced from the dry frit addition was evaluated. Dust associated with the frit can cause problems such as matting to the wetter interior surfaces of the SME and associated support systems, such as the off-gas system. This can cause long term operational issues, such as line plugging or hardened cake material falling back into the slurry causing downstream pumping/plugging problems. Specifically, six different frits with various quantities of water were tested by visually observing the outlet of a bench scale mixer used to mix the water with the frit. Figure 4 shows visual observations of dusting in a Frit 320/water system. Note that the particle sizes for Frit 320 range from 38 to 177 microns. Clearly, no airborne debris is observed for water concentrations  $>0.7\text{wt}\%$ . However, based on testing investigating the flowability of wetted frit, SRNL recommended the frit be blended with a slightly higher water concentration (1.2 wt%) prior to its addition in the SME to mitigate dusting [8].

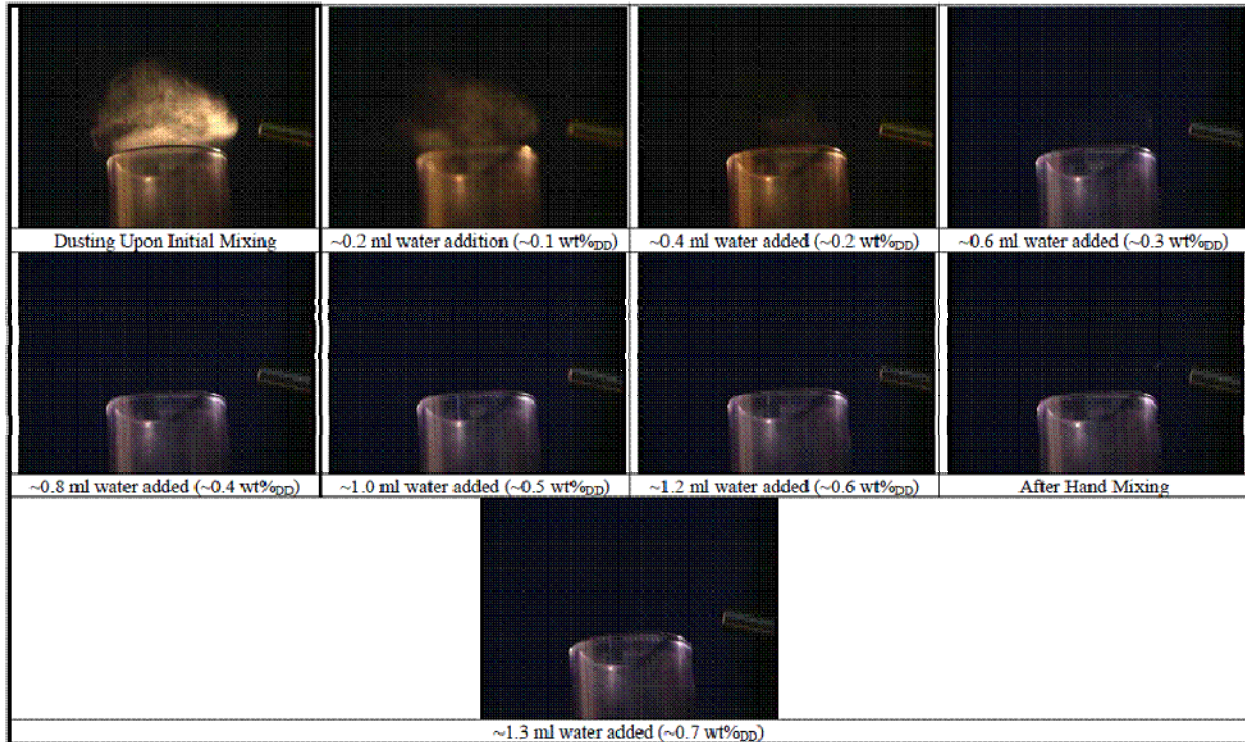


Fig. 4. Qualitative results from testing of Frit 320 with additions of de-ionized water ranging from 0 to 0.7 wt%.

#### Path Forward for Dry Bulk Frit Addition

Responses to the RFP will be used in conjunction with data accumulated during vendor testing to down-select a dense phase conveying system for implementation at the DWPF. In parallel with the technology down-select, construction on a butler building external to 221-S will begin to house the frit feed system components (i.e. frit bin inverter, receipt hopper, and dense phase conveyer). Design of facility modifications includes an external through-wall penetration as well as jumper designs to accommodate transfer of the dry frit stream to directly to the SME.

#### Water Separation Technology for Decontamination Frit Addition

Glass filled stainless steel canisters are currently decontaminated using a pressurized stream of frit and water. The frit slurry (nominally 8wt% frit) is collected and transported directly to the SME. Though the process takes advantage of recycling the used frit, approximately 19000 liters [5000 gallons] of water per SME batch accompanies the frit slurry transfers, resulting in significant amounts of water returned to the CPC process. To reduce the volume of water, a separation technology is planned to remove water from the decontamination frit slurry. The frit-rich stream will be routed to the SME to continue recycle of frit, while the water-rich stream will be sent to an alternate on-site disposition facility (e.g. Effluent Treatment Plant).

A literature review of commercially available separation technologies was performed by EnergySolutions, including consideration of

- Rotary microfiltration
- Backpulse filtration

- Crossflow filtration
- Hydrocyclone
- Clarification
- Decanting Centrifugation

Based upon facility needs including reliability, efficiency, and flexibility, the hydrocyclone technology (which relies upon centrifugal forces to drive separation) was chosen along with backpulse filtration (which relies upon differential pressure to drive the feed stream through sintered metal filter tubes) as viable candidates for separation of the frit slurry. Subsequent testing was performed by *EnergySolutions* on each technology to provide proof of concept. Testing with a prototypic hydrocyclone yielded less efficient separation of frit solids from the feed stream as compared to backpulse filtration, likely requiring some additional downstream separation to accommodate waste facility acceptance criteria. However, as a consequence of the periodic flushing sequence required to remove solids from the backpulse filter, significantly more water is returned to the SME during operation of the backpulse filter (33% of the incoming volume versus 10% for the hydrocyclone). Additionally, the backpulse filter is significantly more complex (i.e. multiple valves, additional jumpers) than the hydrocyclone, significantly complicating remote facility operation. Details of the testing are provided in a separate report [9].

### **Path Forward for Water Separation Technology**

A down-select will be considered addressing 1) the separation technology most applicable in terms of operation and ease of implementation, and 2) the final disposition pathway for the water-rich stream. Pending consideration of the two options and the disposition pathway, further testing may commence regarding technical baseline testing for facility implementation.

### **CONCLUSIONS**

Commitments for accelerated bulk waste removal and subsequent waste tank closure at the Savannah River Site require fundamental flowsheet changes to accommodate increased waste throughput at the Defense Waste Processing Facility. Specifically, three major enhancements have been proposed including the incorporation of an alternative reductant in the Chemical Process Cell, as well as two separate tasks addressing the reduction in water currently introduced during processing, each of which are currently in various phases of design and development. The alternate reductant task aims to improve process flexibility and increase facility attainment by reducing the role of the gas chromatographs. The water reduction tasks aim to reduce the total volume of water returned to the Tank Farm and decrease the Slurry Mix Evaporator cycle time. These enhancements will ultimately lead to a significant reduction in the overall mission life of the facility.

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