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MELT RATE ASSESSMENT OF SB2/3 WITH FRIT 418 – EFFECTS OF WASTE LOADING AND ACID ADDITION

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February 2004

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EXECUTIVE SUMMARY

Preparations are being made by the Defense Waste Processing Facility (DWPF) to blend Sludge Batch 2 (SB2) with Sludge Batch 3 (SB3) (blend referred to as SB2/3), and implement a frit change from Frit 320 to Frit 418. A series of dry-fed tests have been performed to investigate the effect of waste loading (WL) on the melt rate of the SB2/3 – Frit 418 system. As was observed in the SB2 – Frit 320 system, dry-fed melt rate furnace (MRF) tests of the SB2/3 – Frit 418 system showed that melt rate decreased as the waste loading increased (WL range of 31-43% tested). In addition, waste throughput of the SB2/3 – Frit 418 system reached a peak (at approximately 34% WL) with increased waste loading before steadily decreasing (based on MRF tests). Based on this testing, the Immobilization Technology Section (ITS) recommends that the waste loading starting point for DWPF should be approximately 34% when the processing of the SB2/3 begins with Frit 418. The current target waste loading for DWPF is 34% with SB2 – Frit 320, and the results from this testing do not indicate any reasons to reduce this target. Then after a period of steady processing, incrementally higher waste loadings may be evaluated in order to determine the actual waste throughput peak for radioactive vitrification of SB2/3 – Frit 418 feed at DWPF.

The MRF data indicate that the SB2 – Frit 320 and SB2/3 – Frit 418 feed systems will have about the same waste throughputs at DWPF for waste loadings in the mid-30's, but SB2/3 – Frit 418 will have greater waste throughputs at higher waste loadings (≥38% WL). However, previous SB2 – Frit 320 SMRF tests with feed at 31, 35, and 40% waste loadings showed that the feed was thick and did not flow well over the melt pool. A separate SMRF test with SB2/3 – Frit 418 feed at 40% waste loading did not exhibit this problematic cold cap behavior. Since the MRF results are not impacted by this type of feed/cold cap behavior, there is a distinct possibility that melt rates/waste throughputs obtained in the processing of actual SB2/3 – Frit 418 feed in the DWPF will be higher than those for SB2 – Frit 320 feed at similar waste loadings.

In addition, MRF tests were also performed to investigate the impact of acid stoichiometry on melt rate of the SB2/3 – Frit 432 system. Frit 432 was used to evaluate the impact of acid stoichiometry since Frit 418 was not readily available at the time, and Frit 432 is compositionally similar to Frit 418. In MRF tests of the SB2/3 – Frit 432 system, as acid stoichiometry increased, melt rate also increased (all tests performed at 35% WL). The recommendation of this testing is to investigate acid addition strategy effects on melt rate for SB2/3 – Frit 418 in the MRF and the slurry-fed melt furnace (SMRF) since the amount of acid and frit composition were both changed. Because all of this testing was performed on small-scale equipment with a dried, non-radioactive simulant, melter feed rheology impacts on cold cap behavior could not be assessed. Therefore, the exact behavior of the radioactive sludge in a DWPF-sized melter could not be quantified. Any gains or waste throughput peaks observed in the DWPF melter may be somewhat different due to the non-quantified effects of feed rheology or cold cap behavior.

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LIST OF ACRONYMS

ACTL	Aiken County Technology Laboratory
DWPF	Defense Waste Processing Facility
ITS	Immobilization Technology Section
LMR	Linear Melt Rate
MRF	Melt Rate Furnace
PCCS	Product Composition Control System
REDOX	REDuction/OXidation
SB1A	Sludge batch 1A
SB1B	Sludge batch 1B
SB2	Sludge batch 2
SB3	Sludge batch 3
SME	Slurry Mix Evaporator
SMRF	Slurry-fed Melt Rate Furnace
SRAT	Sludge Receipt and Adjustment Tank
SRS	Savannah River Site
SRTC	Savannah River Technology Center
WL	Waste Loading
WSRC	Westinghouse Savannah River Company

1.0 INTRODUCTION

Sludge Batch 2 (SB2) is currently being processed with Frit 320 by the Defense Waste Processing Facility (DWPF) at the Savannah River Site (SRS). Preparations are also being made to blend SB2 with Sludge Batch 3 (SB3) (blend referred to as SB2/3 throughout this report) and to implement a frit change. The frit recommended for the processing of SB2/3 is Frit 418 (Smith et al. 2003; Peeler and Edwards, 2003). This recommendation came from extensive dry-fed and slurry-fed melt rate testing of several frits (Smith et al. 2003) and model-based assessments of projected operating windows (Peeler and Edwards, 2003).

The assessments of melt rate and waste throughput for this study were performed with the melt rate furnace (MRF), which uses dry feed. Tests were performed with non-radioactive, simulated SB2/3 material. Since Frit 418 was chosen as the “frit of choice” for the processing of SB2/3, the objectives of this study were to investigate the impact of waste loading on melt rate and waste throughput. In addition, the impact of acid stoichiometry on melt rate was investigated (with Frit 432). Due to the small-scale of the test equipment and the design of the equipment, as well as the use of dried simulant sludge, the behavior of the actual radioactive feed in the DWPF melter cannot be fully proven. This includes rheological effects, cold cap behavior, and off-gas surges.

2.0 EXPERIMENTAL

2.1 22-L SRAT/SME Feed Preparation

All Sludge Receipt and Adjustment Tank (SRAT) products were fabricated at the Aiken County Technology Laboratory (ACTL). Each SRAT run was performed at a targeted acid stoichiometry and a REDuction/OXidation (REDOX) (expressed as $Fe^{2+}/\Sigma Fe$) target of 0.2, and Frit 418 or 432 (nominal and measured compositions listed in Table 2-1) were later added for the targeted waste loading (WL). Frits 418 and 432 are compositionally similar – Frit 432 has 2% less sodium (difference added to SiO_2).

Table 2-1. Nominal and Measured Compositions of Frits 418 and 432 Used for Melt Rate Tests

	Frit 418		Frit 432	
	Nominal	Measured*	Nominal	Measured**
Oxide	(wt%)	(wt%)	(wt%)	(wt%)
B_2O_3	8	7.72	8	8.54
Li_2O	8	7.99	8	7.62
Na_2O	8	8.01	6	5.86
SiO_2	76	74.51	78	76.5
Trace	---	0.89	---	0.04
Total	100	99.12	100	98.56

* Average of three samples, measured in duplicate. **Average of one sample, measured in duplicate.

For the tests investigating the impact of WL on melt rate, the SRAT product fabricated for the MRF tests targeted an acid stoichiometry of 135%. For the MRF tests to determine the effect of the amount of acid on melt rate, four 4-L SRAT runs were performed with targeted acid stoichiometries of 140%, 155%, 170%, and 185%. Additional details of the various feed preparation runs are given in Savannah River Technology Center (SRTC) notebooks WSRC-NB-2003-00121 and WSRC-NB-2003-00212.

2.2 Melt Rate Testing

The dry-fed MRF utilized has a cylindrical inner chamber approximately 0.5 cubic feet in size, with heating coils winding around the chamber walls. The diameter of the chamber is ~7", and an insulating sleeve and a 1200 mL stainless steel beaker were inserted from the top. The tests were conducted with 6" deep stainless steel beakers inserted with the sleeve so that the beaker bottom was approximately flush with the top of the uppermost chamber coil. An insulating block was used to cover the beaker. The furnace was heated to approximately 1150°C with the top opening covered. Once the furnace reached the setpoint, the cover was removed and the beaker containing sufficient dried, sieved material to produce 500 grams of glass was inserted. After 50 minutes, the beaker was removed from the furnace and allowed to slowly cool to room temperature. This residence time in the furnace was determined during testing in 2002 to establish a standard test time for melt rate comparison for this dry-fed furnace (Lorier et al. 2002).

3.0 RESULTS AND DISCUSSION

3.1 Effect of Waste Loading

Tests in the MRF were conducted with SB2/3 SRAT product plus Frit 418 in the WL range of 31-43%. Linear melt rates were determined for each run via the same method stated in Lorier and McGrier (2002) – measure the glass height at ¼" intervals across the beaker, and then average the values to obtain an average glass pool height. Then the average of the glass pool height is divided by the run time to obtain the linear melt rate result in inches per hour. The relative linear melt rates (LMR) as a function of waste loading are listed in Table 3-1.

Table 3-1. Linear Melt Rates (inches/hour) of MRF "Effect of Waste Loading" Tests

Targeted* waste loading	LMR (in/hr)
31%	0.95
33%	0.92
35%	0.84
37%	0.78
38%	0.75
40%	0.71
43%	0.59

* Material prepared for the MRF incorporates a calcine factor of the SRAT product. Once the calcine factor is determined, the amounts of SRAT product and frit are calculated to obtain this "targeted" waste loading.

The data indicate a trend of decreased melt rate as WL increased. This same trend for melt rate was also observed with the SB2 – Frit 320 system (Lorier and McGrier, 2002). Figure 3-1 plots linear melt rate versus waste loading of SB2/3 – Frit 418 and SB2 – Frit 320 for comparison. At lower waste loadings (30-37%), the two systems basically yield the same melt rates. Though there is greater variation in the SB2 – Frit 320 system (possibly due to limited experience with the test method at the time), the trend is the same – decreased melt rate as WL increased. However, previous SB2 – Frit 320 SMRF tests with feed at 31, 35, and 40% waste loadings showed that the feed was thick and did not flow well over the melt pool. A separate SMRF test with SB2/3 – Frit 418 feed at 40% waste loading did not exhibit this problematic cold cap behavior. Since the MRF may not account for feed rheology and cold cap behavior, there is a distinct possibility that melt rates obtained in the processing of actual SB2/3 – Frit 418 feed in the DWPF will be higher than those for SB2 – Frit 320 feed at similar waste loadings.

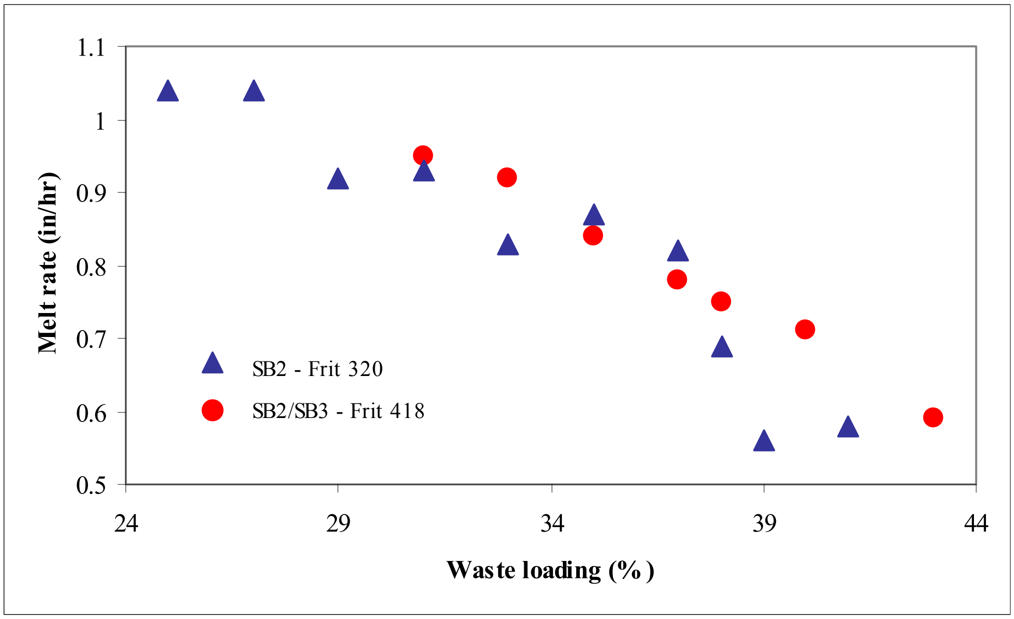

Figure 3-1. Melt Rate vs. Waste Loading: Comparison of SB2/3 – Frit 418 and SB2 – Frit 320

In addition, waste throughput factors were calculated for the SB2/3 – Frit 418 and SB2 – Frit 320 systems. The waste throughput factors, developed for comparison purposes, were calculated via Equation 1. The waste throughput factor is *WTfactor*, the waste loading is *WL*, and the linear melt rate is *LMR*.

$$WTfactor = (WL)(LMR) \tag{1}$$

For example, the *WTfactor* at 31% WL of the SB2/3 – Frit 418 system is 29.5, which was calculated as follows:

$$WTfactor = (31)(0.95)$$

The waste throughput factor allows for a comparison of relative waste throughputs for different feed systems. The waste throughput factors of the SB2/3 – Frit 418 and SB2 – Frit 320 systems are listed in Table 3-2, and are plotted for comparison in Figure 3-2.

Table 3-2. Waste Throughput Factors of MRF Tests: SB2/3 – Frit 418 and SB2 – Frit 320 Systems

	WTfactor	
Targeted waste loading	**SB2/3 – Frit 418**	**SB2 – Frit 320**
25%	---	26.0
27%	---	28.1
29%	---	26.7
31%	29.5	28.8
33%	30.4	27.4
35%	29.4	30.5
37%	28.9	30.3
38%	28.5	26.2
39%	---	21.8
40%	28.4	---
41%	---	23.8
43%	25.4	---

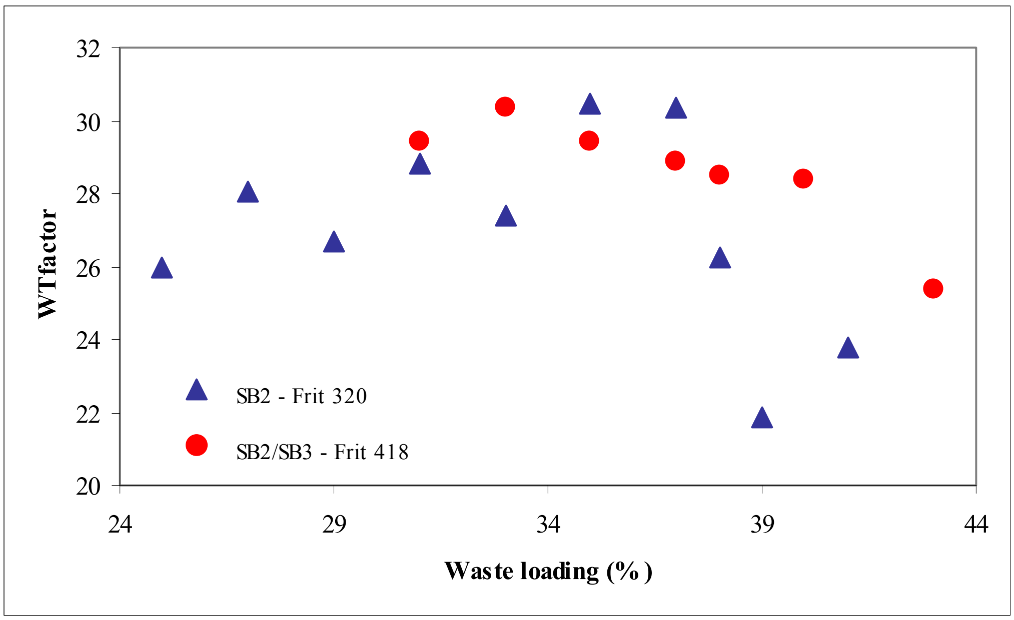

Figure 3-2. WTfactor vs. Waste Loading: Comparison of SB2/3 – Frit 418 and SB2 – Frit 320

The waste throughput factor, and hence waste throughput, for the SB2/3 – Frit 418 system increased before decreasing at higher waste loadings (see Figure 3-2). The data indicate that the waste throughput factor "peaks" at approximately 34% WL for the SB2/3 – Frit 418 system. As was the case with melt rate, the same trend for the waste throughput was observed with the SB2 – Frit 320 system (Lorier and McGrier, 2002). The waste throughput factors of the SB2/3 – Frit 418 system are greater than the SB2 – Frit 320 system at the higher waste loadings (≥38% WL). These data are solely based on dry-fed, non-radioactive, small-scale testing and any gains or waste throughput peaks observed in a melter may be different due to slurry feeding and the interactions of cold cap and feed rheologies.

The data indicates that the two feed systems will have about the same waste throughputs at DWPF for waste loadings in the mid-30's. However, previous SB2 – Frit 320 slurry-fed (utilizing the SMRF) tests with feed at 31, 35, and 40% waste loadings showed that the feed was thick and did not flow well over the melt pool. At times the cold cap even piled up to the point that the SMRF feed tube was plugged (Smith and Jones, 2003). Due to these problems, a melt rate could not be determined. A separate SMRF test with SB2/3 – Frit 418 feed at 40% waste loading did not exhibit this problematic cold cap behavior (Smith et al. 2003). Since the MRF results are not impacted by this type of feed/cold cap behavior, there is a distinct possibility that melt rates/waste throughputs obtained in the processing of actual SB2/3 – Frit 418 feed in the DWPF will be higher than those for SB2 – Frit 320 feed at similar waste loadings.

3.2 Effect of Acid Addition

Melt rate tests investigating the effect of acid addition on the SB2/3 – Frit 432 system were performed in the MRF. Frit 432 was used because Frit 418 was not available at the time of this suite of tests and because the two frits are compositionally similar (see Table 2-1). Frit 432 has 2% less Na_2O and 2% more SiO_2 than Frit 418 and has a lower melt rate, per MRF results (Smith et al. 2003). Therefore, the melt rates at the various acid stoichiometries for these tests should be lower than if Frit 418 had been used. The linear melt rates of these tests are listed in Table 3-3. Each of these tests was performed at a waste loading of 35%.

Table 3-3. Linear Melt Rates (inches/hour) of MRF "Effect of Acid Stoichiometry" Tests

Acid Stoichiometry	LMR (in/hr)
140%	*
155%	0.70
170%	0.74
185%	0.87

* Test aborted before 50-minute runtime due to furnace controller malfunction (no feed remaining to rerun).

These data indicate that the amount of total acid present (predetermined mixture of formic and nitric acids to keep a REDOX of 0.2) has an effect on melt rate. Over the acid stoichiometry range of 155-185% (test at 140% cannot be used due to a malfunction of the furnace controller, causing the test to be aborted before the 50-minute runtime), melt rate increased as acid stoichiometry increased for SB2/3 – Frit 432, based on dry-feed testing. As a comparison, the linear melt rate of SB2/3 – Frit 418 at 35% WL (135% acid stoichiometry) was 0.84 in/hr. The results may be confounded since more than one variable changed – frit composition (amount of alkali) and acid stoichiometry. Since the SB2/3 – Frit 418 system showed a greater melt rate at a lower acid stoichiometry (same WL), the frit's higher alkali content may be a more dominant effect than the acid amount. Because melt rate differences were observed with different acid amounts on a dry-feed basis in the SB2/3 – Frit 432 system, MRF and SMRF tests of the SB2/3 – Frit 418 system should also be performed to assist in qualifying any impacts of the acid addition strategy for DWPF. Flowsheet testing for SB2/3 has recommended a starting acid stoichiometry of 155% (Baich et al., 2004); however, the behavior of the feed in DWPF may necessitate a change in the stoichiometry. The SMRF could help determine if the variations in melt rate associated with changes in the acid addition strategy shown on the dry-fed testing holds in a slurry-fed system.

4.0 CONCLUSIONS

Preparations are being made by DWPF to blend SB2 with SB3 (blend referred to as SB2/3) and to implement a frit change from Frit 320 to Frit 418. A series of dry-fed tests have been performed to investigate the effect of waste loading on the melt rate of the SB2/3 – Frit 418 system. In addition, MRF tests were also performed to investigate the impact of acid stoichiometry on melt rate of the SB2/3 – Frit 432 system. The conclusions of these melt rate tests are as follows:

- As was observed in the SB2 – Frit 320 system, dry-fed (MRF) tests of the SB2/3 – Frit 418 system showed that melt rate decreased as the waste loading increased (WL range of 31-43% tested).
- As was observed in the SB2 – Frit 320 system, the waste throughput factors of the SB2/3 – Frit 418 system reached a peak with increased waste loading before steadily decreasing (based on MRF tests). The peak was at approximately 34% WL for the SB2/3 – Frit 418 system.
- The MRF data indicates that the SB2 – Frit 320 and SB2/3 – Frit 418 feed systems will have about the same waste throughputs at DWPF for waste loadings in the mid-30's. However, previous SB2 – Frit 320 SMRF tests with feed at 31, 35, and 40% waste loadings showed that the feed was thick and did not flow well over the melt pool. A separate SMRF test with SB2/3 – Frit 418 feed at 40% waste loading did not exhibit this problematic cold cap behavior. Since the MRF results are not impacted by this type of feed/cold cap behavior, there is a distinct possibility that melt rates/waste throughputs obtained in the processing of actual SB2/3 – Frit 418 feed in the DWPF will be higher than those for SB2 – Frit 320 feed at similar waste loadings.
- The waste throughput factors of the SB2/3 – Frit 418 system were greater than the SB2 – Frit 320 system at higher waste loadings (≥38%).
- In MRF tests of the SB2/3 – Frit 432 system (Frit 418 was not available at the time of the tests), as acid stoichiometry increased, melt rate also increased (all tests performed at 35% WL). Since the variables of acid stoichiometry and frit composition were changed, the results may be confounded and the effect of acid stoichiometry on melt rate should be investigated further with the SB2/3 – Frit 418 system.

5.0 RECOMMENDATIONS/PATH FORWARD

Based on the results of this study, the following recommendations are made:

- When the processing of the SB2/3 begins with Frit 418, the targeted, starting waste loading for DWPF should be 34%. This is the current target waste loading for DWPF for SB2 – Frit 320 processing, and the results from this testing do not indicate any reasons to reduce this target. After a period of steady processing (assuming no issues at 34% WL), incrementally higher waste loadings may be evaluated in order to determine the actual waste throughput peak for DWPF, radioactive vitrification of SB2/3 – Frit 418 feed.
- Perform SMRF tests to further investigate acid addition strategy effects on melt rate for SB2/3 – Frit 418. Also, perform further testing (e.g., thermal analysis) to investigate why melt rate increased with increased total acid.

6.0 REFERENCES

Baich, MA, DR Best, TK Snyder, and MF Williams. 2004. *Sludge Batch 2-3 Blend Flowsheet Simulations: Determination of Acid Addition Window*, WSRC-TR-2004-00042, Revision 0, Westinghouse Savannah River Company, Aiken, South Carolina.

Lorier, TH, TM Jones, and DC Witt. 2002. *Melt Rate Testing for the DWPF: Summary of FY02 Testing*, WSRC-TR-2002-00545, Revision 0, Westinghouse Savannah River Company, Aiken, South Carolina.

Lorier, TH and PL McGrier. 2002. *Melt Rate Improvement for the DWPF: Higher Waste Loading Testing*, WSRC-TR-2002-00344, Revision 0, Westinghouse Savannah River Company, Aiken, South Carolina.

Peeler, DK and TB Edwards. 2003. *Projected Operating Windows for Various Sludge Batch 2/3 Blends: A Progression for a PAR to a MAR Assessment*, WSRC-TR-2003-00509, Revision 0, Westinghouse Savannah River Company, Aiken, South Carolina.

Smith, ME and TM Jones. 2003. *DWPF Melt Rate Testing for SB2/Frit 320 (31, 35, and 40% Waste Loaded*, WSRC-TR-2003-00512, Westinghouse Savannah River Company, Aiken, South Carolina.

Smith, ME, TH Lorier, and TM Jones. 2003. *SMRF and MRF DWPF Melt Rate Testing for SB2/3 (Case 6b-250 Canisters)*, WSRC-TR-2003-00466, Revision 0, Westinghouse Savannah River Company, Aiken, South Carolina.