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## UNIFORMITY OF MATERIAL IN THE SME AND MFT (U)

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UNIFORMITY OF MATERIAL IN THE SME AND MFTINTRODUCTION AND SUMMARY

The DWPF will satisfy the product consistency specification in the Waste Acceptance Preliminary Specifications[1] through control of the chemical composition of the glass product.[2] This control will be achieved by ensuring that each batch of feed in the Slurry Mix Evaporator (SME) will produce glass which satisfies the specification.

To this end, a sampling system (including equipment and administrative controls) has been designed which will provide representative samples of each process batch. An analytical system (including instruments, analytical methods, and laboratory procedures) has been developed which will provide accurate and reproducible determinations of the chemical compositions of each process batch. A Product Composition Control System (PCCS) has been developed which will combine the analytical data with other process information to determine whether each SME batch will produce glass which satisfies the specification. However, these systems will fail if the SME or the Melter Feed Tank (MFT) does not provide a uniform process batch.

The purpose of this report is to determine what degree of uniformity can be expected of material in the SME and MFT. These vessels were designed based on a design development process[3] which had proven successful for similar hard-to-mix feeds in the past. This process resulted in a design of the SME and MFT agitation systems which was intended to provide highly uniform melter feed material. Based on the results of extensive tests in prototypic equipment, the SME and MFT designs have met this design goal.

- Under normal operating conditions (agitation of a 50 wt% solids melter feed slurry at 130 rpm), there is no discernible dependence of slurry composition on level within the vessel.
- Even at somewhat dilute conditions (40 wt% solids slurry), the chemical composition of process batches in the SME and MFT will vary by < 4 percent (on a relative basis).

DESIGN REQUIREMENTSSlurry Mix Evaporator (SME)

The objective of SME operations is to complete preparation of feed for the melter. This includes addition of frit (both spent frit from canister decontamination, and fresh frit) to the sludge/PHA

slurry, evaporation of excess water from the SME batch, cooling and sampling of the SME batch, evaluation of the SME batch, and transfer of the SME batch to the Melter Feed Tank.

The SME has been designed to satisfy the following requirements:

- **Agitation.** The SME must be able to produce a process batch with a uniform chemical composition throughout the vessel. If the process batch is not uniform, then samples from the SME are unlikely to be representative of the vessel's contents. This, in turn, may render meaningless the evaluation of the SME batch (based on the PCCS). It may also result in production of a glass product very different from that which would be produced by a uniform process batch. Two situations of great importance are:

- (1) Mixing of a relatively fluid suspension (e.g., frit in formic acid solution) with a more viscous, non-Newtonian, waste slurry (e.g., sludge and the precipitate hydrolysis aqueous (PHA) product).
- (2) Resuspension of a settled frit/waste slurry (e.g., after an extended outage).

- **Heating.** Materials are added to the SME as aqueous slurries. The SME must be able to evaporate excess water to provide a process batch with consistent physical properties and water content.

- **Cooling.** The SME must be able to cool the process batch for sampling, and to control the rates of chemical reactions.

The mixing capability of the SME is very important in satisfying the product consistency specification. Neither the heating nor the cooling capabilities of the SME are of direct importance in meeting the product consistency specification. However, they have affected the design of the agitation system, and thus have played an indirect role. Very early in the design process, the decision was made to place a series of coils in the vessel for heating and cooling (see Figure 1). The agitation system for the vessel was then designed based on the presence of these coils, and other vessel internal pieces (e.g., sample line and transfer line). Slurry property data used for design are listed in Table 1.

**TABLE 1**  
**SLURRY PROPERTIES FOR DESIGN**  
 (from Reference 4)

<u>Property</u>	<u>Maximum</u>	<u>Minimum</u>
Yield stress, dynes/cm <sup>2</sup>	250	25
Consistency, cp	60	10
Density, g/cm <sup>3</sup>	1.46	1.29
Solids content, wt%	50	40
Transfer velocity, ft/sec	10	3

#### Melter Feed Tank (MFT)

The objective of the MFT is simply to receive the process batch from the SME, and feed it to the melter. The MFT has the same design requirements for agitation and cooling as the SME. However, for design purposes, it has been assumed that there is no significant source of water addition to the MFT; thus, there is no need for heating capability. Thus, the coil assembly for the MFT has no capability for heating the vessel's contents. Other than this minor difference, the design considerations for the two vessels have been the same.

In what follows, the discussion will center around the SME. Since the requirements for agitation of the MFT are the same as those for the SME, the agitation systems are virtually identical. Thus, the conclusions about the uniformity of process batches in the SME should be equally true of those in the MFT.

#### DESIGN PROCESS

The agitation system for the SME (and the MFT) was designed by the Du Pont Engineering Department. The design process used was based on extensive corporate experience with slurries whose properties were similar to those in Table 1.[3] Starting with the Basic Data,[4] an initial design concept for the vessel and the agitation system was proposed. This design concept was then tested on a small scale, with both model fluids and a simulated melter feed. The small-scale tests were used to confirm the adequacy of the design concept, and to scale it up to the actual design.

#### Design Concept[5]

As noted above, the basic vessel geometry (including the position of the coil assembly) was part of the initial design concept.

Since the agitator in the SME must operate over a wide range of fluid levels, a dual impeller design was selected. Corporate experience indicated that starting a flat-bladed impeller in a layer of settled solids would require much less power than starting a pitched blade impeller.[3],[5] Corporate experience also indicated that a flat-bladed impeller would be less likely to undergo a mechanical failure under such conditions.[5] For this reason, the lower impeller was a four-bladed flat blade impeller. A 45° pitched blade impeller was provided near the top of the coils. A two-speed motor was specified, to allow a slow start. The initial design concept also included four baffles on the vessel wall, 90° apart.

#### Small-scale Tests[6]

The initial design concept was then discussed with vendors of agitation equipment (Lightnin and Chemineer).[7] These experts in agitation advised that there were potential agitation problems with the design concept (e.g., relative positions of coil assemblies and impeller blades). They unanimously recommended scale model testing of the design concept. The experts from Lightnin and Chemineer also agreed that any actual problems uncovered during testing could most likely be solved by modifying the design of the agitator, rather than the vessel's dimensions or internals. They also expressed an opinion that baffling of the vessel was most likely unnecessary.

Based on these discussions, Du Pont requested that Ekato (Schopfheim, FRG) perform small-scale tests of the design concept. Ekato was selected because of their strong technology base, and their superior laboratory facilities for the studies required, and their openness in sharing design information.

Ekato performed tests in two vessels. Both vessels were fabricated so that vessel internals (including the coil assembly) were simulated. Two fluids were used: a transparent model fluid (77% polyisobutylene, 18% benzene, and 5% finely dispersed silica), and a simulated melter feed prepared by the Savannah River Laboratory (SRL). The simulated melter feed was a rather accurate simulation of the major chemical components in the feed, and was prepared in a manner which mimicked the chemistry expected in the waste tanks. The rheological properties were in the design basis range (Table 1), which were based on the properties observed in actual waste slurries. As can be seen in Table 2, the model fluid represented the rheological properties of the simulated melter feed quite well.

**TABLE 2**  
**COMPARISON OF MODEL FLUID AND SIMULATED MELTER FEED**

<u>Model Fluid</u>	<u>Simulated Melter Feed</u>
Yield stress (dynes/cm <sup>2</sup> )	70
Consistency (cp)	25
Density (g/cm <sup>3</sup> )	0.834
Solids content (wt%)	5.0
	80
	22
	1.54
	57.3

The criterion used for evaluating agitation was a very important feature of these tests. Ekato and Du Pont representatives agreed that motion of material throughout the vessel would be the basis for determining whether agitation was effective. Since separation of frit from frit/waste slurries occurs only under stagnant conditions, any motion will apparently overcome such separation. This criterion is based on theoretical work by Nienow, and is discussed further in reference 3. This criterion does not provide a quantitative estimate of the uniformity of the slurry's chemical composition. However, it does provide assurance that variations will be limited.

The major conclusions from the Ekato studies were:

- Surface penetrations of vessel internals near the vessel wall were the last areas to show motion.
- Baffles were not needed for effective agitation, and in fact interfered with mixing.
- Scaling up on tip speed of the agitator, Ekato concluded that the full-scale DWPF agitator should normally operate at 130 rpm. For startup, operation at half this speed was recommended.
- A test with the transparent fluid indicated that even with the most viscous slurry in Table 1 (yield stress of 250 dyne/cm<sup>2</sup>, consistency of 60 centipoise), the agitation system would be able to mix water into the slurry and achieve a uniform material.

#### **DESIGN DESCRIPTION**

The SME design in Figure 1 reflects the results of these studies. The agitator has a three-bladed hydrofoil as the upper impeller, and a four-bladed vertical flat blade turbine as the lower. The agitator can operate at either 130 rpm (motor operating at 100

horsepower), or at 65 rpm (50 horsepower). The lower impeller blades are ten inches from the vessel floor, and are 9.75 inches tall. The lower impeller is 36 inches in diameter. The centerline of the upper impeller is 58.5 inches from the vessel floor. The bottom of the coil assembly is twelve inches above the vessel floor, and is 70 inches high. It contains both heating and cooling coils (The MFT has only cooling coils). There are no baffles in the vessel.

#### DESIGN CONFIRMATION STUDIES

As noted above, the studies used for development of the design did not provide quantitative estimates of the uniformity of the chemical composition of the process batch. Tests have been performed at the Savannah River Technology Center's TNX facility to validate the small-scale studies, and to develop quantitative estimates of uniformity.

##### Caplan's Study

In the first of these, Caplan[8] used a prototypical SME vessel, and used solids content (in wt percent) to determine process batch uniformity at the higher agitator speed. Solids content was used because the technique was believed to be more reproducible than chemical analyses, and because it was rapid.

Caplan used an air-operated diaphragm pump arrangement to sample material at three levels (17, 92, and 107 inches from the vessel floor). The nominal solids content was 41 wt%. Caplan also compared his results at the three different levels to those of samples taken using the reference sampling equipment (sample inlet point nine inches above the melter floor).

The average solids content for each level in the vessel, the number of samples, are compared to each other and to those taken with the reference sampler in Table 3. The results show a maximum difference of in the means of 1.0 wt%, or a 2.5 relative % difference. There was no statistically discernible difference (based on Scheffé's method) between the two higher level means, or between the lower level mean and the mean of samples from the reference sample inlet point. However, the differences between the upper level means and the lower level means were statistically significant. The overall variance in solids content due to level was 0.5 wt%. Caplan also confirmed that the higher agitator speed was preferred for material uniformity.

**TABLE 3**  
**SOLIDS CONTENT AS A FUNCTION OF LEVEL**

<u>Distance</u> <u>From Vessel Floor</u>	<u>Number of</u> <u>Observations</u>	<u>Mean Solids</u> <u>Content (wt%)</u>
9 inches*	106	41.095
17 inches	109	40.802
92 inches	111	41.856
107 inches	109	41.705

\*Sampled using reference DWPF sample system.

#### Voogd's Study

Voogd, [9] while preparing feed for large-scale melter tests, examined both slurry settling and the ability of the SME to resuspend settled slurries. The agitator was turned off in the prototypical SME, and slurry (at a nominal 48 wt% solids) was allowed to settle for 24 hours. Samples were taken at the reference sampler location (9 inches above the vessel floor), and at a location 84 inches from the vessel floor, using the same sampling method used by Caplan. The data are summarized in Table 4.

Voogd concluded that there were no statistically discernible differences in the two locations, as a function of time (However, the data clearly indicate settling of solids in the vessel, albeit slowly). Comparing the initial data (0 hours) to that four hours later indicates that loss of agitation for up to four hours does not lead to significant settling of the slurry. Voogd also found that agitation speed did not affect the uniformity of the slurry in the range of 65 to 130 rpm. Voogd had no difficulty in resuspending the solids after the extended period after agitation was stopped.

**TABLE 4**  
**SOLIDS CONTENT AS A FUNCTION OF LEVEL**

<u>Time after Agitation Stopped (hours)</u>	<u>Solids Content (wt%)</u>	
	<u>9 inches from floor</u>	<u>84 inches from floor</u>
0	48.07	48.54
4	48.39	48.44
8	48.92	49.07
12	48.90	49.33
16	50.41	49.82
20	50.33	49.89
24	50.69	50.56

Jenkins' Study

The studies of Caplan and Voogd, while very useful, had the following limitations:

- Solids content was used. While probably closely related to the chemical composition, no quantitative relationship between the two was developed.
- Samples were not taken directly at different levels, but instead were pumped out of the vessel. While great care was taken to minimize line length, it is not known to what extent line velocities were controlled. Thus, this may have introduced error into the results.

Jenkins, [10] in the course of characterization of sampling errors, also examined the uniformity of the chemical composition of a prototypical SME vessel. In these tests, he addressed both of the issues identified above. Both the chemical composition and the solids content was measured for each sample. Samples were removed from the vessel by a "grab" sampler, which did not require pumping material out of the vessel.

Jenkins sampled the vessel at two locations - one 20 inches from the vessel floor, and one about 6 inches below the liquid level of a normal SME batch (~100 inches from vessel floor). Two solids contents were studied (41 and 49 wt%). The higher solids loading

is that expected during normal operation of the SME and MFT. The agitator was operated at the higher speed setting, as will be done for normal operations.

Jenkins' data are summarized in Table 5. At the higher solids content, no statistically discernible difference was found between the two levels in the vessel, for any of the elements or for solids content. Thus, under normal operating conditions (agitation of a 50 wt% solids melter feed slurry at 130 rpm), there is no discernible dependence of slurry composition on level within the vessel.

Even for the more dilute slurry (41 wt% solids slurry), Jenkins found differences of only 1 - 3.2 relative %, depending on the element. The higher relative differences generally were seen for the more dilute elements. In general, samples at the top of the vessel were somewhat lower in frit elements (1 - 2 %), and somewhat higher in waste components (2 - 3 %). The relative difference in the solids content values was 2.9 %, which was a greater difference than for any of the elemental analyses with the exception of Cu (which was present at a low concentration).

This latter point is important, because it puts both Caplan's and Voogd's data in better perspective. It implies that solids content data are, in fact, indicative of the differences which are observed in elemental analyses.

### **CONCLUSIONS**

An important goal of the design of the SME and MFT systems has been to ensure that these vessels would provide a uniform material (in terms of chemical composition). The studies cited here indicate that this goal has been met.

- Under normal operating conditions (agitation of a ~50 wt% solids melter feed slurry at 130 rpm), there is no discernible dependence of slurry composition on level within the vessel. This conclusion rests on Jenkins' elemental data, supported by both Voogd's and Caplan's solids content data.
- Even at somewhat dilute conditions (~40 wt% solids slurry), the chemical composition of process batches in the SME and MFT will vary by < 4 percent (on a relative basis). This conclusion rests on Jenkins' elemental data, supported by Caplan's solids content data.

The studies reported here also indicate that solids content is a good indicator of the uniformity of chemical composition.

**TABLE 5**  
**SME UNIFORMITY - SOLIDS CONTENT AND SAMPLE LOCATION**

<u>Species*</u>	<u>High Solids Content</u>		<u>Relative Difference</u> (Percent)
	<u>Low Level</u>	<u>High Level</u>	
Al	3.57	3.56	0.0
Fe	11.34	11.46	0.5
Mn	2.19	2.18	0.4
Cu	0.277	0.277	0.1
K	2.38	2.36	0.4
Ti	0.186	0.183	0.1
B	6.74	6.70	0.8
Li	4.14	4.13	0.5
Si	50.95	50.64	0.5
Solids Content	49.30	49.14	0.4
Content			

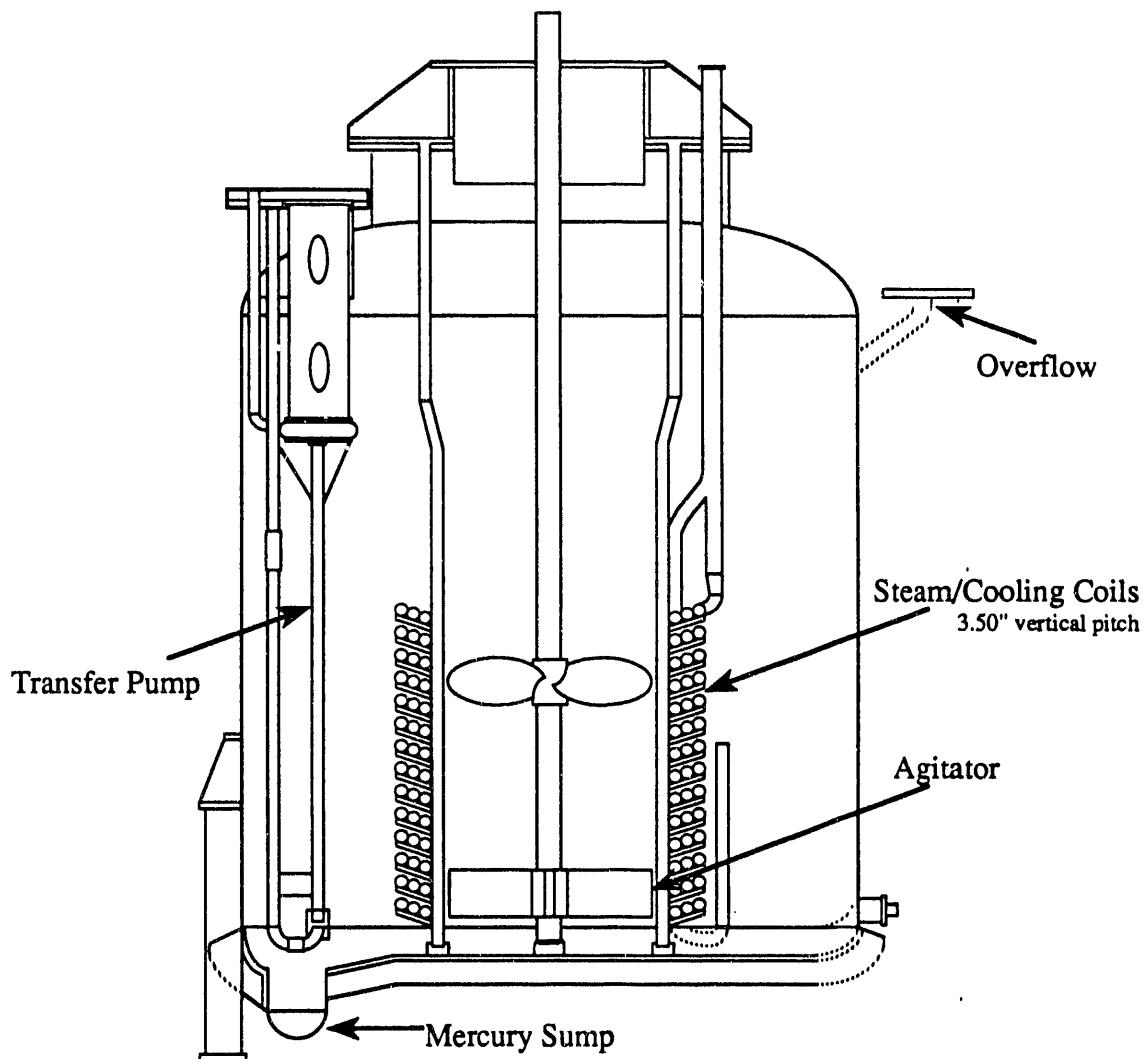
<u>Species*</u>	<u>Low Solids Content</u>		<u>Relative Difference</u> (Percent)
	<u>Low Level</u>	<u>High Level</u>	
Al	3.32	3.36	1.8
Fe	10.54	10.66	1.9
Mn	2.02	2.06	1.7
Cu	0.252	0.262	3.2
K	2.12	2.14	1.7
Ti	0.185	0.187	0.8
B	6.86	6.84	0.3
Li	4.27	4.19	1.4
Si	52.78	52.17	0.9
Solids Content	41.14	39.88	2.9
Content			

\*Elements are reported as wt% oxide in vitrified slurry sample; solids content is in wt% dried (100°C) solids.

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FIGURE 1  
ARRANGEMENT OF SME  
(MFT is similar, except for coils)



Vessel capacity	42000 L (11000 gal)
Vessel height	550 cm (216 in)
Vessel inner diameter	366 cm (144 in)
Sample pump inlet	28 cm (11 in)
Transfer pump inlet	20 cm (8 in)
Impeller diameter	91 cm (36 in)

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