Vitrification of Simulated Fernald K-65 Silo Waste at Low Temperature

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VITRIFICATION OF SIMULATED FERNALD K-65 SILO WASTE AT LOW TEMPERATURE

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ABSTRACT

Vitrification is one of the technologies that has been chosen to solidify ~18,000 tons of geologic mill tailings, designated as K-65 wastes, at the Fernald Environmental Management Project (FEMP) in Fernald, Ohio. The glass formula developed in this study for the FEMP wastes is a lithia substituted soda-lime-lithia-silica (SLLS) composition which melts at 1050°C¹. Low melting formulations minimize volatilization of hazardous species such as arsenic, selenium, chromium, and lead during vitrification. Formulation in the SLLS system avoids problematic phase separation known to occur in the MO-B₂O₃-SiO₂ glass forming system (where MO = CaO, MgO, BaO, and PbO which are all constituents of the FEMP wastes). The SLLS glass passed the Environmental Protection Agency (EPA) Toxic Characteristic Leach Procedure (TCLP) for all the hazardous constituents of concern under the previous characteristic waste regulations. The SLLS glass is as durable as high melting soda-lime-silica glasses and is more durable than the borosilicate glasses previously developed for the K-65 wastes. Optimization of glass formulations in the SLLS glass forming system should provide glasses which will pass the newly promulgated Universal Treatment Standards (UTS) which take effect on August 28, 1998.

INTRODUCTION

Vitrification is one of the technologies that has been chosen to solidify geological mill tailings at the FEMP facility in Fernald, Ohio. The geologic mill tailings are residues from the processing of pitchblende ores during 1949-1958. These residues are contained in silos in Operable Unit 4 (OU4) at the FEMP facility. OU4 is one of five operable units at the FEMP. OU4 consists of four concrete storage silos

¹ patent pending on SRTC Lithia Additive Melting Process (LAMPTM) technology

and their contents. Silos 1 and 2 contain K-65 mill tailing residues, Silo 3 contains non-radioactive metal oxides, and Silo 4 is empty. The 14,262 tons (12.9 million kilograms) of K-65 residues contain radium (Ra^{226}), uranium ($\text{U}^{234,235,236,238}$), uranium daughter products (Th^{230}), and heavy metals such as lead and barium plus 1,185 tons of bentonite which was added to the silo headspace in 1991 to lower the radon concentration in the silo domes in 1991 [1].

Vitrification was chosen as the preferred technology for the Silo 1, 2, and 3 wastes [1,2]. Glasses that melted at high temperatures (1289-1595°C) were developed by Pacific Northwest National Laboratory (PNNL) [1,2] and glasses that melted at lower temperatures (1150-1350°C) were developed by the Vitreous State Laboratory (VSL) for the K-65 silo wastes. The PNNL formulated glasses primarily in the soda-lime-silica (SLS) system since the waste is high in BaO which readily substitutes for CaO (lime) in SLS glass. The VSL formulated glasses primarily in the borosilicate glass forming system. The PNNL and the VSL conducted both crucible and pilot scale vitrification studies.

A Vitrification Pilot Plant (VPP) was constructed at FEMP to operate at temperatures up to 1450°C. The VPP began operation at FEMP on June 19, 1996. During the initial startup testing, 12.5 tons of benign startup "frit" were melted. Silo 1, 2, and 3 surrogate melter tests were completed in September 1996 and produced 11 tons of glass. Another campaign using Silo 1 and 2 surrogate with bentonite was subsequently performed. All of the VPP surrogate testing was completed at melt temperatures between 1130°C and 1350°C. A final transition to lower temperature operation at ~1150°C was planned when the VPP failed on December 26, 1996.

This study documents SRTC glass formulation and development for K-65 waste that was completed in April, 1993. The SLS based glass developed in this study takes advantage of the glass forming ability of the waste, e.g. has an 84% waste loading and a volume reduction of ~ 60%, but melts at 1050°C. The low vitrification temperature minimizes volatilization of the RCRA hazardous constituents (As, Pb, Se, Cr) and minimizes corrosion of melter materials of construction.

BACKGROUND

In 1991 PNNL analyzed the composition of K-65 residues from Fernald on a dry wt% calcine (oxide) basis [1]. The waste analyses were comparable to (a) analyses performed by Controls for Environmental Pollution, Inc. (CEP) in 1990 and (b) analyses performed by National Lead of Ohio (NLO) prior to 1989 [1]. In 1993, PNNL published [2] an average composition for the K-65 residues in Silos 1 and 2, an average composition of the Silo 3 waste, and an average composition for the BentoGrout™ added to the tank headspace (Table I). Note that the PNNL analyses are normalized and contain significant contributions of unspecified components designated as "other." The K-65 Silo 1 and 2 residues

were reported to contain between 1-3 wt% sulfate and the Silo 3 material is reported to contain up to 18 wt% sulfate on a dry solids basis [2].

Analyses performed by FEMP in 1993 indicate that the sulfate in Silo 1 varies between 0.04 and 0.35 wt% on a wet solids basis while the sulfate in Silo 2 varies between 0.26-1.93 wt% on a wet basis [3]. The total organic carbon (TOC) for Silo 1 wastes varies from 0.52 to 3.48 wt% on a wet basis while the TOC for Silo 2 varies from 0.01 to 2.44 wt% on a wet solids basis [3]. In February, 1998 FEMP developed revised surrogate waste compositions for Silo 1 and 2 wastes and BentoGrout™ [3]. For comparison, the 1993 PNNL average compositions are compared to the 1998 FEMP surrogate waste compositions in Table I.

Due to the high concentrations of silica (SiO₂), lime (CaO), baria (BaO), and PbO in the K-65 Fernald wastes, PNNL chose the use of soda-lime-silica (SLS) glass for solidification [1, 2]. PNNL only formulated glasses in the borosilicate system for Silo 3 wastes which contained no PbO. For the K-65 wastes, the initial PNNL formulations required only one simple additive, Na₂O as NaOH or Na₂CO₃, rather than formulation of a complex glass making frit.

Table I. Chemical Analyses and Oxide Surrogates for K-65 Residues (Silos 1 and 2), Silo 3 FEMP Wastes, and BentoGrout™

	1993 Average K-65 Silos 1 & 2	K65 Silo 1 Surrogate with 9 wt% Bento- Grout™*	K65 Silo 2 Surrogate with 9 wt% Bento- Grout™*	1993 FEMP Silo 3	1993 Bento- Grout™	1998 Bento- Grout™
	[2]	[4]	[4]	[2]	[2]	[4]
Al ₂ O ₃	3.7	4.77	5.77	8.3	15.8	16.27
BaO	6.3	6.32	3.48	0.1	---	---
CaO	1.5	0.18	3.73	7.3	1.8	1.85
Fe ₂ O ₃	4.8	3.56	6.69	12.6	3.6	3.71
K ₂ O	0.9	0.81	1.50	2.8	0.8	0.82
MgO	1.7	2.18	2.11	15.8	6.3	6.49
Na ₂ O	1.7	1.41	1.34	9.3	3.4	3.50
P ₂ O ₅	0.8	0.58	0.68	14.5	1.4	---
PbO	12.3	13.81	6.92	0.3	---	---
SiO ₂	62.9	62.08	62.38	22.0	65.4	67.35
SO ₄	---	4.30	5.41	---	---	---
Other	3.5	---	---	7.2	1.5	---
SUM	100.00	100.0	100.0	100.0	100.0	100.0

*2-3 grams kerosene added to the surrogate to simulate the Total Organic Carbon

PNNL developed four series of glass compositions for various combinations of actual FEMP wastes [1, 2]. Series A glass was a SLS formulation containing 88-89 wt% K-65 waste and 11-12 wt% Na₂O glass formers. Series B glass was an SLS formulation containing equal amounts of ~45% K-65 and ~45% BentoGrout™. Series C glass was a borosilicate formulation containing ~74% Silo 3 waste. Series D glass was an SLS formulation containing ~37% K-65 and ~37% Silo 3 wastes. Carbon was added to PNNL-A, B and D melts to reduce the sulfate (SO₄) to SO₃ or SO₂ vapor [2]. However, the final glass redox was not measured and several of the glasses exhibited the formation of metallic nodules indicating that too much reduction had occurred in the glass causing sulfides and metallic species to form. For the Series D glasses even significant quantities of carbon (9g of carbon/100g of glass) did not mitigate sulfate formation on the melt surface.

All of the PNNL glasses contained between 74-89 wt% waste on a dry oxide basis which corresponded to volume reductions of 50-68% [1, 2]. All of the PNNL glasses melted at elevated temperatures between 1289° and 1595°C. Glasses PNNL-A, C and D contained unidentified white crystalline inclusions while PNNL-C (a borosilicate glass) and PNNL-D (a high sulfate containing glass) were reported to be potentially phase separated.

The Vitreous State Laboratory (VSL) formulated K-65 waste glasses in the sodium-borosilicate (SBS)² system at various boron concentrations (5.88-12.8 wt%). During minimelter tests, urea was added to control foaming attributed to sulfate volatilization. These SBS glasses had waste loadings varying between 60-74 wt% and melted at temperatures between 1000-1050°C.

The K-65 Silo 1 and 2 wastes contain 7-12.3 wt% PbO, 3.5-6.3 wt% BaO, 0.2-3.7 wt% CaO, and 1.7 to 2.2 wt% MgO (Table I). The PbO-SiO₂-B₂O₃, BaO-SiO₂-B₂O₃, CaO-SiO₂-B₂O₃, and MgO-SiO₂-B₂O₃ glass forming systems all contain large regions of phase separation [5]. At concentrations greater than 40 wt% SiO₂, the phase separation is metastable in the PbO-SiO₂-B₂O₃ system [6] and causes the separation of an SiO₂ rich glass from a much denser PbO•2B₂O₃ rich glass [6]. Therefore, formulation of borosilicate waste glasses for the K-65 wastes may readily produce phase separated glasses, as observed in the PNNL-C series glasses, with the PbO being stabilized in a borate rich phase. The borate rich phase, being more soluble than the silica rich phase, would be prone to release more Pb than a homogeneous glass during durability testing.

The low melting borosilicate glasses developed by the VSL and those subsequently developed by FEMP had total MO (where MO = PbO, BaO, CaO and MgO) concentrations within the known regions of phase separation in the PbO-SiO₂-B₂O₃ and BaO-SiO₂-B₂O₃, CaO-SiO₂-B₂O₃, and MgO-SiO₂-B₂O₃ systems [7].

²This report distinguishes between borosilicate and soda-lime-silic (SLS) based on the ASTM C-162 definition of borosilicate glass containing >5 wt% B₂O₃.

MAY 1998 ENVIRONMENTAL PROTECTION AGENCY (EPA) REGULATIONS

The K-65 Silo 1 and 2 wastes are a characteristically hazardous waste, as they leach lead at a rate greater than 100 times [1] the allowable Environmental Protection Agency (EPA) Resource, Conservation, and Recovery Act (RCRA) concentration limits [8], (the limits given in Table II, Column A). Therefore, the K-65 Silo 1 and 2 wastes are RCRA hazardous and radioactive (mixed) wastes. The Silo 3 wastes are a dry powdery mixture of metal oxides with a much lower concentration of radionuclides (thorium, uranium and Pb^{210}) than the Silo 1 and 2 wastes. There are approximately 4000 tons of Silo 3 wastes for disposal. The Silo 3 wastes leach arsenic, chromium, cadmium, and selenium at concentrations exceeding characteristically hazardous RCRA limits. Therefore, the K-65 Silo 3 wastes are also RCRA characteristically hazardous and radioactive (mixed) wastes.

Prior to August, 1998, the FEMP wastes would have had to be treated to meet the EPA Characteristically Hazardous Limits given in Table II, Column A. However in August, 1995, the EPA proposed a new rule (Phase IV of the Land Disposal Restrictions) which would require Characteristically Hazardous Waste to be treated to the same Universal Treatment Standards (UTS) as those for listed mixed wastes [9]. The Phase IV Rule was finalized on May 26, 1998, and will take effect August 26, 1998 [10]. Any mixed wastes not stabilized by that date will be required to be treated to the UTS limits, rather than to the Characteristically Hazardous Limits shown in Table II. The final Phase IV UTS limits for the RCRA metals are given in Table II, Column B. This lowers the acceptable Pb TCLP concentration for K-65 waste forms from the previous 5 mg/L to 0.75 mg/L.

The final rule requires that a treated waste form be tested against the UTS standards not only for the constituents which fail the TCLP characteristically hazardous limit, but for all "hazardous constituents which are present in the waste, i.e., underlying hazardous constituents" [10]. The Fernald Silo 1 and 2 wastes exhibit TCLP leaching concentrations for lead which exceed the Toxicity Limit, and leachant levels for nickel (Ni) and selenium (Se) which exceed the UTS in some of the analysis. Therefore, under the final regulations the final waste form will have to meet the UTS for lead, and probably for Ni and Se, as given in Table II, Column B. Similarly, the Silo 3 wastes contain As, Cd, Cr, Pb, and Se as underlying hazardous constituents. The final waste form for the Silo 3 waste would also have to meet the UTS standards for all of these constituents.

Alternatively, a delisting petition could be prepared per 40 CFR 260.22 "Petitions to Exclude a Waste Produced at a Particular Facility," for the K-65 treated waste glass. The potential delisting limits (Table II, Columns C and D) required for the K-65 waste glass are calculated as follows:

- assume 1.8 million gallons waste x 50% waste loading x 10 yr treatment = 180,000 gallons disposed/yr [7]

This allows a Dilution/Attenuation Factor (DAF) of 100 and generates the Delisting Limits given in Table II, Column C, assuming a 10 year treatment period. If a 5 year treatment time is used, the volume of glass produced per year is increased, and the DAF is reduced to 80 - yielding the delisting limits in Table II, Column D. If the waste form is delisted it will have to meet the delisting limits for all the constituents given in Table II, including fluoride and cyanide. Delisting of the K-65 waste glass product is attractive because it raises the Pb TCLP release limit from 0.75 mg/L to 1.2 or 1.5 mg/L depending on the treatment times.

Table II. Final 1998 EPA TCLP Land Disposal Restrictions, Phase IV [10].

	Column A	Column B	Column C	Column D
Element	Characteristically Hazardous TCLP Limits (mg/L) [8]	Final Phase IV TCLP UTS Limits (mg/L) [10]	Delisting Limit TCLP, mg/L 10 Year Treatment DAF =100 [7]	Delisting Limit TCLP, mg/L 5 Year Treatment DAF =80 [7]
Ag	5	0.14	20	16
As	5	5.0	5.0	4.0
Ba	100	21	200	160
Be	---	1.22	0.40	0.32
Cd	1	0.11	0.50	0.40
Cr	5	0.60	10	18.0
Hg	0.2	0.025	0.20	0.16
Ni	---	11	10	8.0
Pb	5	0.75	1.5	1.2
Sb	---	1.15	0.60	0.48
Se	1	5.7*	5.0	4.0
Tl	---	0.20	0.20	0.16
V**	---	1.6	20	16
Zn**	---	4.3	---	---
F	---	400	400	320
CN	---	30 (amenable)	590 total mg/kg	590 total mg/kg

* Se must be treated to the characteristic limit (1.0 mg/L) to be non-hazardous, although it may be land disposed as a hazardous waste if <5.7 mg/L TCLP

** V and Zn are not underlying hazardous constituents

This would necessitate less reformulation of the Fernald waste glass product than the alternative of meeting the Phase IV UTS limits.

EXPERIMENTAL

Due to the potential for stable and/or metastable phase separation in the MO-SiO₂-B₂O₃ glass forming systems, this study concentrated on methods to lower

the melt temperature of SLS glasses. In the SLS glass forming system PbO, MgO and BaO can all substitute for CaO in the glass structure without separation of a second phase.

Only one SLS glass was formulated by SRTC as an initial scouting test for developing a low melting SLS glass. The glass had a targeted waste loading of ~82% based on an the average Silo 1 and Silo 2 waste composition given in Table I. Lithia (Li_2O) was partially substituted for the Na_2O so that about 8 wt% Li_2O and 8 wt% Na_2O were the only two additives. This surrogate waste glass composition melted at 1050°C. The target and analyzed composition of this glass is given in Table III. The final waste loading in the as analyzed glass was 84%.

The TCLP results for the SRTC glass formulation are given in Table IV and compared with published TCLP results of the raw K-65 waste and the glasses formulated by PNNL. The SRTC formulation melts 300°C lower than the PNNL formulation but is of equivalent durability and waste loading to the high temperature K-65 glass and more durable than the low melting VSL borosilicate glass. The PNNL, VSL and SRTC glasses all pass the EPA TCLP concentration limits before the Phase IV UTS limits went into effect, e.g. pre-August, 1998 (Table IV). The PNNL and SRTC glasses pass at or below the delisting limits while the VSL glasses do not. The SLLS glass formulations require further optimization based on the 1998 waste compositions in reference 4 (Table I) now that the final Phase IV UTS limits (Table II) have been adopted.

The glass attributes of the SRTC SLLS glass are compared to the glasses formulated for FEMP by PNNL and VSL in Table V.

Table III. SRTC Soda-Lithia-Lime-Silica (SLLS) Glass

GLASS COMPONENT	OXIDE WT%	OXIDE WT%
	Target	As Analyzed
Al_2O_3	3.14	4.32
BaO	5.35	4.95
CaO	1.27	1.44
Cr_2O_3	---	0.14
Fe_2O_3	4.08	4.51
K_2O	0.76	0.73
Li_2O	8.50	7.92
MgO	1.44	1.63
Na_2O	10.88	9.85
NiO	-	0.09
P_2O_5	0.68	0.96
PbO	10.45	10.30
SiO_2	53.44	53.10
SUM	99.99	100.03

Table IV. Comparative EPA TCLP Testing for K-65 Waste and for K-65 Waste Glasses Formulated by PNNL, VSL, and SRTC

RCRA Metal	1997 Proposed UTS Limits (mg/L)	Raw K-65 Residue [1] (mg/L)	PNNL-A SLS K-65 Waste Glass [2] (mg/L)	VSL Borosilicate K-65 Waste Glass [2] (mg/L)	SRTC SLLS K-65 Waste Glass Replicate A (mg/L)	SRTC SLLS K-65 Waste Glass Replicate B (mg/L)
Ag	0.11	<0.1	<0.01	---	0.026	0.020
As	5.0	<1.0	0.004- 0.005	---	<0.002	<0.002
Ba	21	0.76	0.69-0.87	---	2.62	2.73
Cd	0.20	0.1	<0.005	---	<0.010	<0.010
Cr	0.85	<0.2	<0.01	---	<0.04	<0.04
Hg	0.025	<0.03	<0.0002	---	<0.0096	<0.0096
Ni	13.6	3.2	---	---	0.062	<0.050
Pb	0.75	630	0.81-1.2	1.9-2.76	1.272	1.619
Se	5.7	<0.1	<0.002	---	0.158	0.297

Table V. Comparison of Attributes of Glasses Formulated for FEMP K-65 Silo Wastes.

GLASS ATTRIBUTES	SRTC Formulation	PNNL Formulations	PNNL Formulations	VSL Formulations
Glass Forming System	Soda-Lithia-Lime-Silica	Soda-Lime-Silica and Borosilicate	Borosilicate	Borosilicate
Phase Separation	No Potential	No Potential	Observed	Potential
Melt Temperature (°C)	1050	1289-1595	1150	1000-1050
Waste Loading (%)	84	80-84	<50	60-74
Volume Reduction (%)	~68	55-68	Not Determined	Not Determined
Density (g/cm ³)	2.86	3.1	Not Determined	Not Determined
Inconel® 690 Compatible	Demonstrated	Not Determined	Not Determined	Not Determined

CONCLUSIONS

Glasses for the FEMP K-65 silo wastes should be formulated in the Soda-Lime-Lithia-Silica (SLLS) system or the to avoid problematic phase separation known to occur in borosilicate glass forming systems, e.g. the $\text{MO-B}_2\text{O}_3\text{-SiO}_2$ glass forming systems where $\text{MO} = \text{CaO}, \text{MgO}, \text{BaO}, \text{and PbO}$. The K-65 wastes contain all of these MO oxides which makes phase separation likely. Phase separation is known to compromise glass durability.

Glasses for the FEMP K-65 silo wastes should be formulated to melt at the lowest temperatures reasonably achievable to minimize volatilization of radioactive radon and hazardous species such as lead, arsenic, selenium, and chromium. The one scouting test performed in this study indicates that acceptable low melting SLS glasses can be formulated which melt at temperatures as low as 1050°C . High waste loading, e.g. 84 wt%, can still be maintained by the addition of only two glass forming additives, Li_2O and Na_2O using the SRTC Lithia Additive Stabilization Process (LAMPTM).^{*} Further optimization of SLLS glasses using the LAMP technology needs to be investigated for FEMP.

The SRTC SLLS glass passes the Environmental Protection Agency (EPA) Toxic Characteristic Leach Procedure (TCLP) for all the hazardous constituents of concern under the pre August, 1998 regulations and at the delisting limits. The SLLS glass is as durable as the high melting SLS glasses formulated by Pacific Northwest Laboratory (PNNL) and is more durable than the borosilicate glasses formulated by the Vitreous State Laboratory (VSL) for the K-65 wastes.

SRTC successes in late 1997 with co-vitrification of two highly variable mixed wastes at the Oak Ridge Reservation (ORR) into SLLS glass indicate that high sulfate and high organic containing (including wood chips) wastes can successfully be vitrified [11] using the LAMPTM technology. These vitrification successes were completed in a field scale Transportable Vitrification System (TVS) at the ORR.

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^{*} Patent pending.

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