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Commercial Ion Exchange Resin Vitrification in Borosilicate Glass

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COMMERCIAL ION EXCHANGE RESIN VITRIFICATION IN BOROSILICATE GLASS

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ABSTRACT

Bench-scale studies were performed to determine the feasibility of vitrification treatment of six resins representative of those used in the commercial nuclear industry. Each resin was successfully immobilized using the same proprietary borosilicate glass formulation. Waste loadings varied from 38 to 70 g of resin/100 g of glass produced depending on the particular resin, with volume reductions of 28% to 68%. The bench-scale results were used to perform a melter demonstration with one of the resins at the Clemson Environmental Technologies Laboratory (CETL). The resin used was a weakly acidic methacrylic cation exchange resin. The vitrification process utilized represented a ~64% volume reduction. Glass characterization, radionuclide retention, offgas analyses, and system compatibility results will be discussed in this paper.

INTRODUCTION AND BACKGROUND

The commercial nuclear industry utilizes ion exchange resins to clarify their process and storage waters. The resins are used to remove unwanted impurities, such as radioactive, hazardous, or other contaminants that could potentially harm the equipment or corrode reactor fuel rods. Over time, these resins have to be re-generated or replaced. When this happens, the spent resins have to be disposed. Disposal often becomes an economic problem because of the large volumes of resin produced and the relatively few technologies capable of economically stabilizing this waste.

Resins for removing Cs from Department of Energy (DOE) High Level Waste (HLW) are being investigated by DOE. The DOE also uses divinylbenzene/styrene resins in their reactor facilities to purify fuel rod storage basin water and in other nuclear facilities. Thus, a viable and economic treatment option would benefit both the commercial nuclear industry and DOE.

Vitrification is a preferred technology because it is capable of consistently producing a durable, leach resistant wasteform, while simultaneously minimizing disposal volumes through organic destruction, moisture evaporation, and porosity reduction. Vitrification of organic ion exchange resins presents a challenge because of the high organic content and the volatile Cs-137 that is usually present. High organics tend to induce reducing environments in melters, which can result in metals reduction and melter materials corrosion. The presence of organics can also result in reduced glasses, which has been shown to affect durability.[1] Cs-137 has been shown to be extremely volatile at high temperatures; thus, stabilization of this contaminant in the glass matrix presents a challenge which must be met if the waste is to be successfully stabilized.

Vitrification has been shown to be a feasible treatment method for organic ion exchange resins. Vitrification studies have been performed at the Savannah River Technology Center (SRTC) with both a resorcinol based organic ion exchange resin and divinylbenzene/styrene copolymer resins, but met with only limited success from a volume reduction standpoint.[2,3] Recent vitrification studies with the divinylbenzene/styrene resin matrices have met with more success through the use of a SRTC borosilicate glass formulation. Bench and pilot-scale studies with this formulation have produced homogeneous and durable glass products when tested on ion exchange resins from DOE reactor facilities and Argentina's commercial nuclear industry.

For this study, samples of six organic resins were obtained that are considered representative of commercial nuclear industry resins. The resins will be identified as A-550, IRC-50, 21H, 900-OH, 200-H, and 650-C, and a brief description of the structure and properties is given in Table I.

Table I. Structure and Properties of Commercial Ion Exchange Resins

Resin	Ion Form	Active Group	Matrix	Structure	Type
A-550	Cl	Quaternary Amine Type II Alkyl	Polystyrene	Gellular	Strong Anion
IRC-50	H	Carboxylic Acid	Methacrylic	Beads	Weak Cation
21H	H/OH	N/A	Polystyrene/ Cellulose	Powdered	2 part Cation/ 1 part Anion
900-OH	OH	Quaternary Amine Type I Alkyl	Polystyrene	Macroreticular	Strong Cation
200-H	H	Sulphonic Acid	Polystyrene	Macroreticular	Strong Cation
650-C	H	Sulphonic Acid	Polystyrene	Macroreticular	Strong Cation

Typical commercial nuclear industry radioactive contaminants include Mn, Co, Sr, C, and Cs. Based on data available, typical contaminant concentrations are 5.8×10^{-4} μCi/g of Co-60, 7.9×10^{-4} μCi/g of Cs-137, 6.2×10^{-4} μCi/g of Cs-134, and 5.4×10^{-4} μCi/g of Mn-54.[4] Other known non-radioactive contaminants include iron, borate, and lithium depending on the type of reactor. In this instance, the non-radioactive contaminants don't present a vitrification problem because these elements can easily be incorporated into glass.

BENCH-SCALE STUDIES

These studies were funded by the DOE - Office of Science and Technology Development, and the focus was to determine optimum vitrification processing parameters. Since the commercial nuclear industry ion exchange resins were very similar to other ion exchange resins used in previous SRTC research, attempts were made to vitrify these resins using the same borosilicate glass composition. Previous testing with this base composition and divinylbenzene/styrene ion exchange resins had indicated that a resin loading of approximately 42 g of resin/100 g of glass produced was optimal. Therefore, this loading was used as the starting point.

Experimental

The resins were mixed with the reagent grade chemicals to produce the borosilicate glass composition. All glasses were melted at 1150°C for four hours. The melt temperature was limited to 1150°C to help avoid volatilizing the Cs normally found on spent resins. All glasses were examined for completeness of reaction, visual homogeneity, and presence of metal sulfides. Testing with the resins involved slowly increasing or decreasing the amount of resin until the limit was found for producing acceptable glass. In some cases, the glasses were made by allowing the batch materials to react before melting to help with oxidation of the resin.

The optimum compositions were used to melt surrogate doped resin glasses. Dopants used were based on the representative radioactive contaminant concentrations given earlier. Higher levels of dopants (six to twelve orders of magnitude) had to be used because the actual amounts of the isotopes only equated to very small gram quantities of the elements, which would have been difficult to detect in the glasses when they were analyzed. Non-radioactive compounds were used as the dopants. These compounds were dissolved in water and mixed with the appropriate amounts of resin. The borosilicate glasses were analyzed for Co, Cs, and Mn content so the radioactive retention could be determined.

Results

Most of the resins produced homogeneous glasses. However, in a few instances, small pellets were found in the glass matrix that were determined by Scanning Electron Microscopy coupled with Energy Dispersive Spectroscopy to be metal sulfides. These have been shown to form when glasses become too reduced. Therefore, glasses which produced these pellets were not considered acceptable, and either the waste loading was lowered or the batch reaction time was increased.

After visual examination, several of the resin glasses were analyzed by the SRTC Mobile Laboratory to determine the glass redox ratio by the colorimetric method. Since it is likely that the resins would be treated in a joule-heated vitrification unit, acceptable $Fe^{2+}/\Sigma Fe$ was considered to be less than 0.33, which is consistent with the operational limits established for the Defense Waste Processing Facility melter[5]. If the redox ratio was found to be acceptable, the glass was further characterized for chemical composition, crystalline content, and durability. The $Fe^{2+}/\Sigma Fe$ ratios are given in Table II. The base glass (without resin added) redox ratio was previously determined to be 0.054. The Glass IDs given in Table II denote the resin type and the resin loading per 100 g of glass produced.

Table II. Measured $Fe^{2+}/\Sigma Fe$ Ratios for Bench-Scale Studies

Glass ID	Ratio	Glass ID	Ratio
550-50	0.259	900-70	0.124
50-50	0.119	200-38	0.269
21-62	0.054	650-38	0.330

For the 21H and 900-OH resin glasses, the redox ratio was not strongly affected by the resin loading. The redox ratios of the 200-H and 650-C resin glasses, on the other hand, were very dependent on the amount of resin in the glass. These glasses had the most problems with metal sulfides forming and incorporating high waste loadings. For the A-550 and IRC-50 resin glasses, the redox ratio gradually increased and became a limiting factor above 50 g of resin/100 g of glass.

The glasses were analyzed for chemical composition by the Analytical Development Section (ADS) of the SRTC. The glass compositions should be fairly consistent because the resin solids contribute only a minor amount to the glass composition. Overall, the glass compositions were very similar, and the type of resin did not seem to greatly impact the glass composition.

All glasses appeared to be visually homogeneous upon cooling. In order to ensure that no crystalline phases were present, X-Ray Diffraction (XRD) analyses were performed, and all glasses were found to be amorphous.

To determine the final product durability, the Product Consistency Test (PCT), ASTM C1285-94[6], was performed. Glass samples were run in triplicate, and the results were averaged and normalized. The normalized PCT results are given in Table III. The measured leachate pH is also listed, since this provides a secondary indication of durability. The PCT for the base glass composition is given for comparison. Since no acceptance criteria have been established for waste glasses other than HLW glasses, the durabilities of the glasses produced were compared against the Environmental Assessment (EA) glass[7], which is the benchmark for HLW.

The PCT results were fairly consistent between the different glasses, with the 200-H and 650-C glasses having slightly higher PCT releases. Most of the glasses had similar releases to the base glass composition for B and Si, with Na releases that were slightly higher. These results indicate that the presence of the resin had little affect on the glass durability performance. The pH results also indicate that the glasses had similar durabilities, with the 200-H and 650-C glasses once again having the slightly poorer durabilities. No apparent trend was observed between the amount of

Table III. Normalized PCT Results (g/l)

Glass ID	Base	550-50	50-50	21-62	900-70	200-38	650-38	EA[7]
B	0.16	0.22	0.19	0.17	0.19	0.25	0.23	16.695
Si	0.07	0.07	0.07	0.07	0.06	0.07	0.08	3.922
Na	0.27	0.50	0.41	0.31	0.48	0.99	0.90	13.346
pH	9.66	10.16	10.05	9.87	10.11	10.59	10.64	11.85

resin in the glass and the PCT release; however a slight increase in B and Na release was seen for the glasses with the higher redox ratios. All glasses had excellent durability compared to the EA glass based both on the normalized releases and on the measured pH.

In order to determine the potential volume reduction of using vitrification treatment on each resin, bulk densities were measured and volume reduction calculations were performed. A measured glass density of 2.854 g/mL was used. The calculated volume reductions are shown in Table IV.

Table IV. Calculated Volume Reductions

Resin	Waste Loading	Resin Density (g/mL)	Volume Reduction
A-550	50 g/100 g or 33.3 wt%	0.6365	55.9%
IRC-50	50 g/100 g or 33.3 wt%	0.6235	55.9%
21-H	62 g/100 g or 33.3 wt%	0.6410	63.5%
900-OH	70 g/100 g or 41.2 wt%	0.6305	68.3%
200-H	38 g/100 g or 27.5 wt%	0.7230	34.0%
650-C	38 g/100 g or 27.5 wt%	0.7790	28.0%

No information on the water associated with these resins when they are in use or storage was found. When the resins are disposed of, they usually have an associated volume of water that also has to be disposed. In many cases, the resins also have a large volume of water that is adsorbed in the resin matrix. Volume reductions for the slurried spent resins will be higher because of this extra water that is evaporated during the vitrification process.

For the radioactive dopants studies, no apparent effect on melt behavior or glass formation was seen. The calculated retentions are shown in Table V. Calculated retentions were fairly consistent from glass to glass with the exception of the Cs retention for the glass made from the 900-OH resin. The calculated retentions were lower than anticipated, but were consistent with other vitrification processes. One explanation is that the radioactive surrogates were not processed through a resin column as would be done in practice, so the materials were not adsorbed as well as they would be in a resin column. With the radioactive materials strongly bonded to the resin, the potential for higher retentions exists.

Table V. Calculated Retentions for Glasses

Glass ID	550-50	50-50	21-62	900-70	200-38	650-38
Cs	73.1%	73.5%	76.9%	100%	70.3%	72.2%
Mn	89.1%	81.1%	83.5%	84.8%	83.3%	87.1%
Co	83.3%	76.9%	81.1%	84.2%	86.4%	79.0%

Based on all of the analytical results, the glasses with the optimal waste loading were homogeneous and very durable glasses. The SRTC borosilicate glass formulation seemed well suited for incorporating these wastes.

MELTER DEMONSTRATION

The primary objective of the melter test was to verify the feasibility of stabilizing commercial industry organic ion exchange resins using vitrification technology. From the six commercial resins studied, IRC-50 was selected because its matrix was the most different from the divinylbenzene/styrene resin material already demonstrated. This resin was shown to produce acceptable glasses in a crucible at a loading of 50 g of resin/100 g of glass or 33 wt%. Visual observations of the operating behavior of the melter was very important, including the presence of foam or salt layers. Corrosion of the melter components was also an important consideration.

Melter System Description

The melter used for the demonstration was a 1/4 square foot stirred tank melter. The melter utilizes an Inconel® melt chamber, which serves as one of the electrodes. The impeller is also fabricated from Inconel® 690 and serves as the other electrode. It provides a stirring action that results in much higher mixing and production rates than can be achieved by convective mixing alone. The maximum temperature obtainable in the melter is ~1050°C because of the temperature limitations of the Inconel® components. Pouring of the glass occurs through an overflow drain. Flow through the drain is controlled by controlling the temperature of the spout.

Slurry feeding is accomplished through the use of a slurry feed port that disperses the feed over the melter surface, so it can be rapidly mixed in with the molten glass through the impeller stirring action. The melter can also operate in a cold-top mode by using slower stirring motion to incorporate the new feed into the molten pool. For this demonstration, the slurry was fed from a continuously stirred feed vessel (~19 L capacity). A peristaltic pump in the feed line directed the feed to the melter at a constant rate, which could be adjusted to accommodate the feed melt rate.

For this demonstration, an oxygen sparger was located at the bottom of the melt tank. Oxygen could be metered into the melt at a rate of 0.6 - 6.10 standard liters per minute to help maintain oxidizing conditions in the melt or to help break down organics in the feed.

The melter was connected to an offgas treatment system that included a filter system as a prefilter to the laboratory exhaust system. A sampling manifold was installed between the melter and the laboratory exhaust system, which enabled isokinetic sampling for EPA Method 29.

Feed Fabrication

Three different batches were made for the demonstration, equaling approximately one melter volume of feed. The Stir-Melter can be started with very little glass in the melter tank, so very little turn-over of the melter contents is required before reaching steady-state. The glass in the melter was of the same base composition. Cobalt was not included in feed batches #2 and #3 because it was not available. After initial feeding to the melter, the glass appeared to be viscous, so additional Na_2O was added directly to the melter vessel through the dry batch feed port and the pour riser. The remaining batches were adjusted to this Na_2O level.

Since no information was available on water content of resin slurries, the resin used at the CETL was only mixed with minimal water to help feeding and adsorption of radionuclide surrogates. The radioactive surrogates were dissolved in the water and then the resin was well mixed with the radioactive surrogate containing water. Once again, the actual adsorption onto the resins was not believed to be optimized. Therefore, the radioactive surrogates may have been more volatile during the demonstration because they were not bound to a matrix.

The representative radiolytical characterization given earlier was used as the basis for spiking the resins with surrogate contaminants for the demonstration. The amounts actually used were 9.7×10^7 µCi/g for Co, 5.0×10^5 µCi/g for Cs, and 5.4×10^4 µCi/g for Mn. Higher amounts were used because of the small mass associated with the radiation dose. Cs was only spiked for the Cs-134 portion since the corresponding mass of Cs-134 compared to Cs-137 was much greater for their particular activities.

Melter Operation

During the demonstration, an optimum feed rate was determined based on the slurry properties and feed melt rate. The optimum feed rate was defined to ensure that the melt surface was evenly covered and that no unnecessary build-up of glass or cold-cap material occurred. Slurry was initially added at a rate of 80 mL/min. This rate was maintained for the first day of feeding. However, as more of the feed built up in the melter, the feed rate had to be decreased because of cold-cap build-up and foaming. The foam layer was controlled by adjusting the impeller height, turning off the sidewall resistance heaters, or adjusting spindle speed. The optimal feed rate was determined to be 30 mL/min.

In addition to monitoring the feed rate, system temperatures were also monitored to obtain the desired temperatures and melt rates. The drain temperature was set at 1025 or 1030°C when pouring, and the melt temperature was set at 1050°C. The actual temperature readings were a little lower for the pour temperature at first then hit the set point during pouring. Melt temperature was a few degrees higher than the target. Both sets of resistance heaters were kept on to maintain the melt temperature and glass viscosity. They were at 940 to 970°C during pouring. No problems with drain pluggage or glass exit viscosity were seen during the demonstration.

The offgas system was operational throughout the demonstration. Samples of the offgas were taken near the end of the campaign when the melter was at steady-state operating conditions. An EPA Method 29 Multiple Metals Sampling Train (MMST) was performed to determine the distribution of metals within the offgas system. The train can also be used to determine particulate emissions according to EPA Method 5. For the demonstration, an isokinetic source sampler was used to perform the Method 29 test. Due to past problems experienced with standard EPA organic sampling methods, the sampling and analysis approach used was to primarily identify the constituents and, thus, determine the efficiency of combustion within the melt chamber.

At test completion, the melter was shut-down according to CETL procedures. The melter system was visually inspected and examined for wear or corrosion.

Results

Samples of each feed batch were taken during the demonstration to determine the physical properties and chemical composition. The physical properties of each feed sample are shown in Table VI. Weight percent solids was determined at 105°C, while calcined solids were determined at 1050°C, which was the melting temperature.

Table VI. Physical Properties of Feed Samples

Sample ID	Wt% Solids	Calcined Solids (Wt%)	Density (g/mL)
#1	51.8	46.95	N/A
#2	29.0	28.62	1.66
#3	31.0	29.16	1.35

Sample #1 had a much higher solids content than the rest of the materials. Part of this batch of feed was used to start-up the melter, so less water was added. The density of sample #1 could not be measured because the material was so thick that it was hard to get a representative sample.

The chemical compositions of the material fed to the melter and the glass produced were determined by the Mobile Laboratory of SRTC and ADS. Only feed sample #3 was analyzed for Cs concentration, and the concentrations in the other feed batches were calculated based on the proportions batched to this feed. It was assumed that no batching errors occurred and proportional quantities of Cs would be found in the other batches.

The melter feed composition varied from sample to sample. It is believed that some heterogeneity or settling of glass additives may have occurred. Additional problems may have been experienced with getting a representative feed sample for analyses because of the rheology of the feed. The entire feed sample would likely have to be vitrified to get a truly representative sample because of the feed settling and unmixing that occurs. Compared to the target compositions, the feed samples were lower in B_2O_3, Co_2O_3 and Cs_2O and higher in SiO_2 than targeted.

For the analyzed glass, some deviation from the target composition existed. This deviation may have resulted from incomplete mixing when Na_2O was added directly to the melter. If the melt contents were not completely homogenized, portions of the glass could contain excess levels of Na_2O. It also appears that excess SiO_2 was added, consistent with the batch sheet calculations.

The glass composition was compared to the Sample #3 feed composition. Theoretically, the feed and glass compositions should be roughly the same with minor differences due to volatility during vitrification. Most oxides were comparable except Na_2O and B_2O_3 which were lower than found in the feed, which was expected since they are volatile components.

In order to understand the radioactive retention in the glass matrix, retention calculations were performed. Percent retention was calculated based on the total glass output and total feed input. These results indicated that most of the feed was incorporated in the glass matrix. Approximately 1% Cs_2O and 7% Co_2O_3 were emitted or not accounted for, while all MnO was retained. Two possible explanations exist for the low Co_2O_3 number. One is that the Co_2O_3 was not evenly mixed in the glass because it was only added in one batch, while the other is that the Co_2O_3 was not completely sorbed onto the resin and so it volatilized with the water in the resin slurry.

The $Fe^{2+}/\Sigma Fe$ ratios for glass samples taken throughout the demonstration were measured in duplicate by the Mobile Laboratory. Some samples were taken from the melter itself, while other samples were taken from the pour spout. Redox ratios for the glass samples varied from 0.000 to 0.007, while a sample of the foam layer had an average ratio of 0.026. All glasses were very oxidized. The measured ratios were lower than limits established at SRTC for joule-heated melters.[5] For joule-heated melters, very oxidizing conditions can result in foaming in the melter. Foaming was seen during the latter part of the demonstration. It is interesting to note that the foam layer was less oxidized than the melt pool based on the redox results. This may have been the result of the oxygen sparging to the melt pool.

Visual examination of the resulting glass indicated that the glass appeared amorphous. The glass had no visible signs of crystallinity. No devitrification was noticed in the glass containers from the demonstration. XRD analysis confirmed that the glass was amorphous.

The PCT was performed on glass from the end of the demonstration. The standard procedure was performed and the triplicate results were averaged and normalized for the glass elemental content. The normalized PCT results were 0.70 g/L for B, 0.21 g/L for Si, and 0.81 g/L for Na, while the leachate pH was 11.80. The normalized releases for B, Si, and Na were substantially less than the EA glass. The leachate pH was very high for this glass, which sometimes indicates poor durability. However, the elemental releases indicated that the glass was acceptable. The radionuclide surrogates were not detected in the leachates at any appreciable amounts.

Glass viscosity was measured as a function of temperature by CETL. Viscosity at 1150°C was 45.5P, while it was ~135P at 1050°C. The measured viscosities indicate that the glass would be considered acceptable for processing in typical joule-heated melters that are maintained above ~1075°C. For the Stir-Melter, the viscosity was near the upper processing limit.

The samples from the MMST were analyzed according to EPA methods. The front half of the impactor used in the sampling was analyzed separately from the back half. The front half represents the particulate matter captured during the test, while the back half represents the condensable species that were in a volatilized state.

The glass matrix components were the biggest contributors to the offgas pollutants. Cs was found only on the front end of the sampler, indicating it was in the particulate form. The Mn concentration was relatively low and was found in both the particulate and volatile forms, while Cs concentration was a little higher. Co was not measured. The emissions are typical of emissions found during vitrification of waste glasses of similar compositions. The mass of particulate metals captured was orders of magnitudes greater than the mass of volatile metals, which allows for easier capture in an offgas system.

Organic samples were drawn through a condenser and 100 mL of water in the sampling manifold. The samples were analyzed by Gas Chromatography/Mass Spectroscopy (GC/MS). No peaks found by the GC/MS had greater than 90% correspondence. The compounds identified were believed to be heterocyclic ring compounds with nitrile side groups and high molecular weight cyclic and heterocyclic nitriles and cynates. These compounds are expected from pyrolysis of organics. These compounds would present environmental problems (e.g., potential carcinogens) and could cause tarring in an offgas system. Therefore, possible means of breaking down these compounds should be explored.

Total feed to the melter was 32.8 kg. Of this total amount, the resin waste was 7.36 kg with an approximate density of 0.6235 g/mL. The total mass of glass produced was 11.95 kg. Density was determined to be 2.844 g/mL using the buoyancy method. Therefore, the volume reduction associated with using vitrification treatment was 64.4%. Given that resins to be stabilized would have an associated water content, the volume reduction would be higher.

Minimal wear or corrosion was seen to the melt vessel and impeller due to the vitrification of the commercial ion exchange resin.

CONCLUSIONS

Based on the studies performed with the six resins representative of commercial industry resins, it appears that using the SRTC borosilicate glass composition will produce very durable and homogeneous glasses. For the resins, different optimum waste loadings were determined, and they were strongly dependent on the type of resin being vitrified. The waste loadings ranged from 38 to 70 g of resin/100 g of glass produced, with associated volume reductions of 28.0-68.3%. These waste loadings and associated volume reductions are higher than what has been seen in other studies with organic resin, but are similar to volume reductions seen with this glass composition. The resins that were the most difficult to vitrify were the strong cation sulfonated styrene resins.

The melter demonstration showed that vitrification of commercial organic ion exchange material is viable. Processing of this feed did not result in any significant problems. The resulting glass product was homogeneous and durable. The most significant particulate and volatile offgas pollutants were attributed to the glass matrix components. The radionuclide surrogate releases were relatively small compared to the particulate releases of the major glass components. The radioactive material retention (demonstrated with surrogate chemical compounds) was ~93% for Co, ~99% for Cs, and 100% for Mn. For this demonstration, a 64.4% volume reduction was demonstrated based on a 33.3 wt% waste loading.

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