

**VITRIFICATION OF MUNICIPAL SOLID WASTE
COMBUSTION AIR POLLUTION CONTROL
RESIDUES
USING CORNING, INC. PROCESS**

Teresa Kosson, David Kosson, and Ben Stuart
Rutgers, The State University of New Jersey
Department of Chemical and Biochemical Engineering
P.O. Box 909
Piscataway, NJ 08855-0990

Dale Wexell and John Stempin
Advanced Materials Research
Corning Inc.
SP-FR-5-1
Corning, NY 14831

ABSTRACT

A demonstration was conducted to vitrify municipal solid waste (MSW) combustor air pollution control residue (APC) under the USEPA Municipal Waste Innovative Technology Evaluation Program. A duplicate demonstration was conducted using a process developed by Corning Inc. in a cold crown melter. The resulting vitrified product was a monophasic, homogeneous, and physically durable glass. Cadmium, chromium, copper, lead, and zinc did not volatilize during the vitrification process. These contaminants did not leach from the vitrified product based on the Toxicity Characteristic Leaching Procedure, the Availability Leaching Test, the Monolithic Leaching Test, or the Accelerated Strong Acid Durability Test. All data were very similar indicating the vitrification process is reproducible.

INTRODUCTION

The US Environmental Protection Agency is evaluating the treatment of Municipal Solid Waste (MSW) residues under the Municipal Innovative Technology Evaluation (MITE) Program. Vitrification is among the treatment technologies being evaluated. Rutgers, The State University of New Jersey, was funded by the USEPA Risk Reduction Engineering Laboratory (RREL) to provide oversight and to evaluate a laboratory scale vitrification process for municipal solid waste combustion (MWC) air pollution control (APC) residues. Corning Inc. funded all costs associated with process demonstration. Duplicate melting demonstrations were conducted at Corning Inc. under observation.

The MSW combustor APC residue evaluated in this study was collected from a modern mass burn facility with a nominal capacity rating of 2,100 tons/day. The service area of the facility was primarily household waste with some commercial and industrial contributions. The APC residue consisted of mixed residuals from the lime slurry drier acid gas scrubber and the baghouses. An elemental analysis of the APC residue on a weight basis is presented in Table 1.

A cold crown glass melter is used in the Corning Inc. vitrification process. A cold crown melter is a melter in which a blanket of unmelted feed is maintained on top of the molten glass throughout the process. Additives mixed with the APC residue are fed into the cold crown melter where high temperatures are sustained producing a homogeneous, single phase glass.

Objectives

The objectives of this evaluation were to:

1. Demonstrate that cadmium, chromium, copper, lead and zinc are retained in the vitrified product.
2. Demonstrate that a monophasic, homogeneous, physically durable, vitrified product is produced from vitrification of APC residue, and;
3. Assess the leachability of metals (cadmium, chromium, copper, lead and zinc) from the vitrified product.

This paper investigates the retention of metals in the vitrified product by performing mass balances around the processes involved in the vitrification. The physical tests data were evaluated to determine if a monophasic, homogeneous, physically durable glass could be produced. Leaching test data were evaluated to assess the leachability of metals.

VITRIFICATION PROCESS

The vitrification process was conducted in a bench-scale cold crown glass melter with an offgas collection system. The feed materials were fed into the top of the cold crown melter so that a blanket of unmelted feed was maintained on top of the molten glass. This blanket of unmelted feed served as a cooling layer for gases formed during the melting process and refluxes condensed volatile constituents back into the reactor. The temperature of the molten glass in the melter was between 1200 - 1550°C with the exact temperature being proprietary.

The Corning Inc. vitrification process for APC residue consisted of three stages including: dechlorination, blending with proprietary additives, and melting. Figure 1 presents the process flow diagram for the vitrification process. The design of the melter and the offgas collection system and details of each step for the demonstration are discussed in the following sections.

Melter and Offgas Collection System

The cold crown glass melting unit consisted of a silica crucible placed in a radiant electric heating furnace. The top of the crucible extended through the top of the furnace to allow for continuous batch feeding. The bottom of the crucible contained a resealable orifice through which molten glass was periodically removed.

The offgas collection system consisted of an inverted glass funnel connected with silicone tubing to a series of three impingers terminated by a carbon trap. The first impinger was filled with distilled water and kept in an ice bath. The second impinger also was filled with distilled water. The third impinger was filled with 1.2 N NaOH and phenol red indicator. A slight negative pressure was maintained throughout the off gas collection system with a flow of approximately 775 ft/min through use of a vacuum pump. At completion of the demonstration, the vacuum was maintained until the melter had cooled to a minimum temperature of 400°C.

Dechlorination

The Corning Inc. vitrification process limited the feed chloride content to a maximum of 5 wt %. The APC residue as supplied contained 16 wt % chloride and this required dechlorination as a first process step. Dechlorination was performed by extracting the APC residue with deionized (DI) water using two sequential extractions. In the first extraction, five liters of DI water per kg of APC residue was combined and mixed for 15 minutes, then allowed to settle for 16 hours. Clear supernatant was decanted and filtered with Whatman 41 filter paper. The solids retained on the filter paper were scraped off and returned to the extraction vessel for an identical second extraction procedure. The moist, dechlorinated APC residue then was dried at 500°C for 16 hours. Total recovery from the extraction process was 87 % of the total water added as filtrate and 99.7 % of the APC residue. Each sequential dechlorination was conducted in six separate batches due to laboratory scale limitations. Table 2 presents chemical analysis of the dechlorinated APC residues on a weight basis after drying at 500°C.

Blending With Proprietary Additives

The APC residue was blended in three batches, subdivided, recombined, and remixed twice using a turbula mixer. The batch mix consisted of 18 kg of dechlorinated APC residue and 16.7 kg of glass forming additives. Exact composition of the process additives was proprietary. The batch additives were analyzed separately and did not contribute any cadmium, chromium, lead or zinc to the batch mix.

Melting

The cold crown melter was heated continuously during operation. Batch mix was added intermittently to the top of the melter as required to maintain the cold crown on top of the molten material. The melter was operated with batch mix feed but without vitrified product withdrawal (delivery through the bottom orifice) for a period of one hour. This period was followed by operation with both intermittent batch mix feed and continuous vitrified product delivery for 20 minutes to complete one operating cycle. This mode of operation was maintained for five cycles. Mix was added to the top of the crucible at typically 10 minute intervals to maintain the layer of unmelted material on top of the molten glass. The 34 kg of mix yielded approximately 15 kg of glass during the melt process. Both unmelted batch mix and molten product remained in the melter at the conclusion of operation. This material was quantified after the melter cooled. The molten glass was collected in preheated graphite molds and annealed at approximately 1000°F.

CHEMICAL ANALYSIS

Solid samples were prepared for total elemental analysis by mixing with an alkali borate and fusing at 1000°C. The fused samples were then dissolved in distilled water and analyzed for elemental composition using inductively coupled plasma (ICP). Alkali earth elements were analyzed using flame emission spectroscopy after preparing samples by acid digestion. These analysis were conducted in the Corning Inc. Analytical Laboratories.

Anion analysis were conducted using ion chromatography at the Rutgers University, Department of Chemical and Biochemical Engineering. Duplicate metals analysis also were conducted on selected samples using atomic absorption spectroscopy. Low level metal concentrations in leaching test extracts were analyzed by ICP mass spectroscopy by the Rutgers University Department of Chemistry. Duplicate analysis for total elemental composition were conducted by neutron activation at the University of Illinois in Urbana.

PHYSICAL PROPERTIES AND LEACHING PROPERTIES TESTS

Physical Properties Tests

Several physical properties of the vitrified product were determined for evaluation. These included: softening point, annealing point, strain point, thermal expansion, density and the Accelerated Strong Acid Durability Test. Each are briefly discussed in the following sections.

The viscosity of glass varies continuously from the molten state to the lowest temperature at which structural adjustments are perceptible, provided the glass is maintained in a stabilized condition. The softening, annealing, and strain points are common viscosity fix points. Viscosity fix points are the temperatures corresponding to approximate viscosity levels that have been found useful for control studies.

Softening Point. The softening point is the temperature at which a fiber of uniform diameter elongates under its own weight at a rate of 1mm/min while the upper 100 mm of length is heated at a rate of 5°C/min. ASTM C-338 Method for the Determination of Fiber Elongation Softening Point was used to determine the softening point of the vitrified product.

Annealing and Strain Point. Annealing and Strain point determinations were made using the beam bending viscometer according to ASTM C598 Standard Test Method for Annealing Point and Strain Point of Glass Beam Bending. At the annealing point, internal stresses are substantially relieved in a matter of minutes. At the strain point, internal stresses are substantially relieved in a matter of hours.

Thermal Expansion. Thermal expansion is the change in length caused by a change in temperature. Glass expansion is an important property in many applications and is frequently set for operating limits. The thermal expansion was determined according to ASTM E-228.

Density. The density (weight per unit volume) was determined according to ASTM-693.

Accelerated Strong Acid Durability Test. The Accelerated Strong Acid Durability Test (ASAD) was selected to determine the durability of the vitrified product in an aggressive acidic pH regime (pH < 1). This test is carried out on a sample crushed to less than 9.5 mm. The crushed sample is challenged with 5% HCl (pH < 1) for 24 hours at a 20:1 liquid to solid ratio. A constant temperature of 95°C was maintained during the extraction .

Leaching Properties Tests

Four leaching test were performed on the vitrified product. Resulting extracts were analyzed for a range of metals and ions. This paper will limit the evaluation of leachability to cadmium, chromium, copper, lead, and zinc. The leaching tests included the Toxicity Characteristic Leaching Procedure (TCLP), the Availability Leaching Test (ALT), the Monolithic Leaching Test (MLT), and the Accelerated Strong Acid Durability Test (ASAD). Each are discussed in the following sections.

Toxicity Characteristic Leaching Test (TCLP). The TCLP was selected to provide a framework of comparison only. The TCLP was carried out in accordance with the method outlined in the 29 Jun 1990 Federal Register, Volume 55, No. 126. This test is carried out on a sample crushed to less than 9.5 mm. Extraction is carried out at a 20:1 liquid to solid ratio using dilute acetic acid.

Availability Leach Test (ALT). The ALT was selected to assess the maximum amount of specific elements or species which could be released under an assumed "worst case" environmental scenario. This test was originally developed by the Netherlands Energy Research Center (ECN). The test is carried out on a sample crushed and size reduced to less than 300 mm. Two serial extractions are carried out, each at a 100:1 liquid to solid ratio, using distilled water. The pH is controlled to pH 7 during the first extraction and to pH 4 during the second extraction, using an automatic pH controller which delivers dilute nitric acid. The first and second extracts are combined for analysis. This test generally extracts all species which are not tightly bound in a mineral or glassy matrix. The test does not provide information on the rate of contaminant release.

Accelerated Strong Acid Durability Test(ASAD). The Accelerated Strong Acid Durability Test was selected to assess the effect of an aggressive acidic pH regime on the mobility metals inherent to the vitrified glass. This test is described in the Physical Properties Tests section.

Monolith Leach Test (MLT). The Monolith Leach Test was selected to assess the release rate of specific elements and species from the vitrified product under diffusion controlled conditions. The MLT was carried out based on a modification of the American Nuclear Society (ANS) American National Standard Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-Term Test Procedure.(ANSI-16.1-1986). The test was carried out using a 2-in. diameter by 2-in. long cylindrical, vitrified sample instead of the specified size test specimen. Samples were extracted by contacting with a specified volume of distilled water for up to 64 days. Contacting water was replaced with fresh distilled water at 1, 2, 4, 8, 16, 32 and 64 days and analyzed for metals and other species.

DISCUSSION OF RESULTS

Mass Balance

A process mass balance was performed to determine the amount of specific elements retained in the product and the fate of elements not retained. Separate mass balances were made around the dechlorination process and around the vitrification process. This paper will limit the mass balance

discussion to cadmium, chromium, copper, lead, zinc, and chloride. The mass balance data for each demonstration are presented in Table 4.

Dechlorination Mass Balance. The percent change of the mass of each element in the APC residue during dechlorination were calculated from the difference in the elemental content before dechlorination and after dechlorination. Negative percent changes indicate removal during dechlorination. No change or small changes indicate that dechlorination had no effect and the element remained in the APC residue.

Dechlorination did not alter the APC residue content of cadmium or chromium. Small quantities of copper and zinc were removed during dechlorination, but only in one of the two demonstrations. This decrease can likely be attributed to analytical error and it can be assumed copper and zinc were retained in the APC residue. The dechlorination process removed approximately 25% of the lead and 97% of the chloride.

Vitrification Mass Balance. The percent change of the mass of each element in the APC residue during vitrification was calculated from the difference in the elemental content before vitrification and after vitrification. Negative percent changes indicate mass losses during vitrification. Positive percent changes indicate increases in mass during vitrification and are attributable to analytical and sampling error. No change or small percent changes indicate that the element was retained in the vitrified product.

Percent changes for chromium and copper was very small indicating they were retained in the vitrified product. Cadmium, lead and zinc exhibited positive percent changes for both demonstrations averaging 34 %, 10 %, and 14 % respectively. These mass increases are attributable to analytical and sampling error and it is assumed that cadmium, lead, and zinc were retained in the vitrified product. The vitrification process resulted in loss of chloride with an average negative percent change of 53 %. This loss can be attributed to volatilization and, or, uncontrolled dusting.

Physical Testing Results.

The vitrified product was a clear glass, emerald green in color. It was seedy but free of any stones or crystalline inclusions. Table 5 list the physical properties of the vitrified products. The physical properties were very similar between demonstrations indicating reproducibility of the Coring Inc. vitrification process.

The vitrified products demonstrated excellent acid durabilities with $<1\text{mg/cm}^2$ weight loss. Volume reduction calculations were made. The Corning Inc. vitrification process produced greater than 40% reduction in the volume of the original APC residue.

Assessment Of Leachability

The leaching tests conducted were the Toxicity Characteristic Leaching Procedure (TCLP), the Availability Leaching Test (ALT), the Monolithic Leaching Test (MLT), and the Accelerated Strong Acid Durability Test (ASAD). This paper will limit the leachability discussion to cadmium, chromium, copper, lead, and zinc. The leaching test data are presented in Table 6.

Toxicity Characteristic Leaching Procedure. The TCLP is a USEPA regulatory leaching test and has established maximum allowable leachate concentrations for selected elements. This test procedure was used as a benchmark for comparison purposes only and is not currently regulatory applicable to either the untreated APC residue or the vitrified product. Table 6A lists the TCLP results for the vitrified product on a release basis presented as a percent of the total composition. Cadmium, chromium, and lead release was below detection limit. The copper release was 0.1 % of the total copper present in the vitrified product. The zinc release also was 0.1 % of the total zinc present in the vitrified product. Lead, cadmium, and chromium were retained in the vitrified product while negligible amounts copper and zinc were released.

Availability Leach Test (ALT). The ALT was selected to assess the maximum amount of specific elements or species which could be released under an assumed "worst case" environmental scenario of pH 7 and then pH 4. The extract generated in each pH regime were combined and analyzed. The ALT data for the vitrified product are presented on a release basis in Table 6A. For all elements of concern except zinc, the release was below detection limit. Zinc release was 0.35 % of the total zinc in the vitrified product. This indicates that the contaminants of concern are tightly bound by the vitrified matrix and rendered immobile.

Accelerated Strong Acid Durability Test(ASAD). The ASAD was selected to assess the effect of an aggressive acidic pH regime (pH <1) on the mobility of metals inherent to the vitrified

product. The cadmium, chromium and lead are below detection limit in the vitrified product leachate. Copper concentrations were the same order of magnitude as the detection limit. Zinc concentrations were three orders of magnitude higher than the detection limit.

Monolith Leach Test (MLT). The MLT was selected to assess the release rate of specific elements and species from the vitrified product under diffusion controlled conditions. The MLT data evaluation is in the preliminary stages, but in general, excluding zinc, most contaminant concentrations were near or below detection limit. For zinc, only the initial challenges produced leachate with elevated levels and the following challenges resulted very low to nonexistent concentrations of zinc. Figure 2 illustrates this behavior for zinc. The 1 day challenge leachates had zinc concentrations four times that of the blank. The following leachates had cumulative zinc release parallel to that of the blank which is primarily background.

Discussion of Leaching Results. The leaching behavior of the elements was very systematic based on the results of the leaching tests conducted. Cadmium, chromium, and lead were immobile, and trace amounts of copper were mobile or released, in each of the leaching tests conducted. Vitrification yields a product that tightly binds cadmium, chromium, lead, and copper to extreme environmental conditions such as low pH and high liquid-to-solid solubility ratios.

Zinc also exhibited systematic behavior according to the TCLP and ALT release and extraction concentration in the ASAD. Examination of the MLT results show that only the initial challenge of the vitrified products to the leachate resulted in elevated concentrations of zinc. (See Figure 2). After the initial washoff, zinc released per unit area was no higher than that of the leachate blanks of subsequent challenges. The mobility of zinc can likely be attributed to surficial dissolution phenomena.

SUMMARY AND CONCLUSIONS

A bench scale demonstration of cold crown vitrification of MWC combustor APC residue was conducted at Corning Inc. under observation. Prior to vitrification, dechlorination of the APC residue was successfully conducted. Duplicate batches consisting of 52 wt. % dechlorinated APC residue and 48 wt. % glass forming additives were vitrified in the cold crown melter. A clear, green amorphous, monophasic, glass was produced. The chemical and physical data for the two demonstrations were very similar.

A mass balance performed around the dechlorination process revealed 97 % of the chlorine and approximately 30 % of the lead was removed. It was determined that lead could be retained in the APC residue during dechlorination by maintaining a pH of 9.8 subsequent to the demonstration. Future project demonstrations will be conducted using dechlorination processes that retain lead.

A mass balance also was performed around the vitrification process. Cadmium, chromium, copper, lead, and zinc were retained in the vitrified product. Greater than half of the chloride was not retained. Losses during vitrification can be attributed to volatilization of the elements and to dusting of the APC residue.

The physical properties of the vitrified product were within the range of those for a soda lime glass system. The vitrified product was very durable in an acidic regime (pH <1) and results of all physical testing were very similar for both demonstrations.

The Toxicity Characteristic Leaching Procedure (TCLP) leachate concentrations for cadmium, chromium, copper, and lead were near or below detection limits. Zinc had substantially larger release rates and extract concentrations than the other elements evaluated. Similar behavior of the cadmium, chromium, copper, lead and zinc was observed for the Toxicity Characteristic Leaching Procedure (TCLP), Availability Leach Test (ALT) and the Accelerated Strong Acid Durability Test (ASAD). For the Monolithic Leach Test (MLT) cumulative release over the 64 day period did not exceed that of the methods blanks for cadmium, chromium, copper, and lead. However, for zinc the initial challenge leachate had concentrations two orders of magnitude higher than the blank. After the initial challenge, zinc concentrations were not distinguishable from the methods blanks. This behavior of zinc can be attributed to surficial washoff.

The following conclusions were made based on the data generated during the vitrification of MSW combustor APC residues using the Corning Inc. process.

1. Vitrification of MSW combustor APC residue yielded a monophasic, homogeneous, physically durable vitrified product.
2. Cadmium, chromium, copper, lead, and zinc can be retained during cold crown vitrification.

3. Cadmium, chromium, copper, lead and zinc show systematic behavior for the TCLP , ALT, MLT and the ASAD. The cadmium, chromium copper and lead concentrations in the leachate of each leaching test were very near or below detection limit. Based on MLT data, elevated zinc concentrations can be attributed to surficial dissolution phenomena.
4. The vitrified product TCLP leachate concentrations for cadmium, chromium copper, lead and zinc were near or below detection limit and well below the maximum allowable limit for regulated materials.
5. Vitrified products from both demonstrations were similar in chemical composition, had similar physical and durability properties and exhibited the same systematic leaching behavior.

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Table 1. Elemental Analysis % of APC Residue Prior to Treatment (wt %).

Element	Weight %	Element	Weight %
Si	7.4	Cd	0.024
Al	3.17	Cu	0.0445
Ca	25.66	Cr	0.023
Na	2.13	As	0.00674
K	2.45	Ni	0.0038
Zn	1.50	Mo	0.0067
Fe	0.79	C	1.49
Ti	0.71	S	2.89
Ba	0.047	Cl	16.1
Pb	0.41		

Table 2. Chemical Analysis of Dechlorinated APC Residue (wt %).

Element	Demo 1	Demo 2
Al	4.5	4.5
As	<0.01	<0.01
Ba	0.06	0.06
Ca	25.5	26
Cd	0.03	0.04
Cr	0.03	0.03
Cu	0.06	0.07
Fe	1.15	1.0
K	1.4	1.4
Mo	<0.01	<0.01
Na	1.1	1.5
Ni	<0.01	<0.01
Pb	0.42	0.42
Si	7.5	7.0
Ti	1.0	0.95
Zn	2.15	2.25
Cl	0.75	0.41
S	3.45	3.55

Table 3. Chemical Analysis based on weight % of total mass % of vitrified product.

	Demonstration 1 (wt %)	Demonstration 2 (wt %)
Al	3.3	3.4
As	<0.01	<0.01
Ba	0.03	0.03
Ca	16	16.7
Cd	0.03	0.03
Cr	0.02	0.02
Cu	0.04	0.04
Fe	0.73	0.78
K	0.8	0.8
Mo	<0.01	<0.01
Na	4.3	4.3
Ni	<0.01	<0.01
Pb	0.24	0.28
Si	27.3	27.5
Ti	0.73	0.73
Zn	1.53	1.58
Cl	0.55	0.51
S	0.66	0.6
C	<0.01	<0.01

Table 4. Mass Balance Data for Dechlorination Process and Vitrification Process.

DEMO 1				DEMO 2			
Dechlorination Process				Dechlorination Process			
Element	APC Residue (g)	After Dechlorination (g)	Percent Change (%)	Element	APC Residue (g)	After Dechlorination (g)	Percent Change (%)
Qd	6.7	6.7	0	Qd	7.0	7.0	0
Cr	6.4	6.4	0	Cr	6.2	6.2	0
Cu	13	12	-8**	Cu	14	14	0
Pb	117	83	-29	Pb	110	84	-24*
Zn	430	430	0	Zn	460	450	-2**
Cl	4600	150	-97	Cl	4600	82	-98*

DEMO 1				DEMO 2			
Dechlorination Process				Vitrification Process			
Element	APC Residue (g)	After Dechlorination (g)	Percent Change (%)	Batch Mix (g)	Material Remaining in Melter (g)	Vitrified Product (g)	Percent Change (%)
Qd	6.7	6.7	0	5.0	2.9	3.9	36**
Cr	6.4	6.4	0	5.1	2.3	3.0	4**
Cu	13	12	-8**	11	-4.6	6.0	-4**
Pb	117	83	-29	66	38	36	12**
Zn	430	430	0	370	170	230	8**
Cl	4600	150	-97	390	71	83	-61*

- * Attributed to removal during process, most likely due to volatilization
- ** Attributed to analytical error

Table 5. Physical Properties of Vitrified Product.

	Demo 1	Demo 2
Quality	Amorphous, single phase, clear	Amorphous, single phase, clear
Color	Emerald green	Emerald green
Softening point	804°C	808°C
Annealing point	632°C	637°C
Strain point	595°C	598°C
Thermal expansion	79.9 x 10 ⁻⁷ /°C	80.3 x 10 ⁻⁷ /°C
Density	2.728 g/cc	2.719 g/cc
Acid durability	<1 mg/cm ² weight loss	<1 mg/cm ² weight loss

Table 6. Leaching Test Data.

Table 6A. Percent of Total Composition Released During TCLP and ALT on Vitrified APC Residue.

Element	Total Concentration (mg/kg)	TCLP		ALT	
		mg/kg	% of total	mg/kg	% of total
Cadmium	258	BDL(1)	NA(2)	BDL	NA
Chromium	195	BDL	NA	BDL	NA
Copper	388	0.4	0.10%	BDL	NA
Lead	2506	BDL	NA	BDL	NA
Zinc	15016	15.10	0.10%	53.00	0.35%

Table 6B. ASAD Reported on an Extraction Concentration Basis (mg/l).

Element	Vitrified Product		
	Demonstration 1	Demonstration 2	Detection Limit
Cadmium	BDL	BDL	0.01
Chromium	BDL	BDL	0.001
Copper	0.05	0.03	0.01
Lead	BDL	BDL	0.01
Zinc	1.12	1.19	0.006

- (1) BDL - Below Detection Limit
- (2) NA - Not Applicable

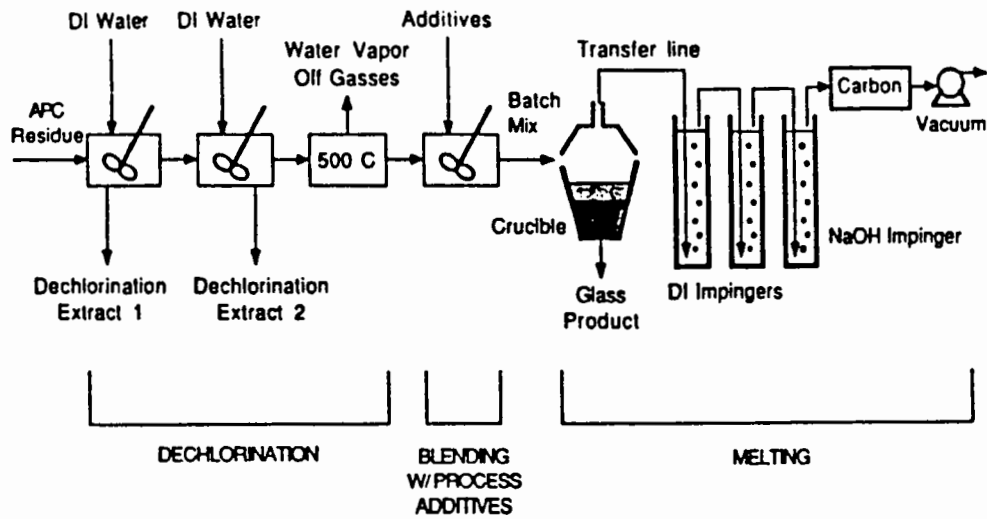


Figure 1. Process Flow Diagram for the Corning Vitrification Demonstration.

Zinc Released per Unit Area

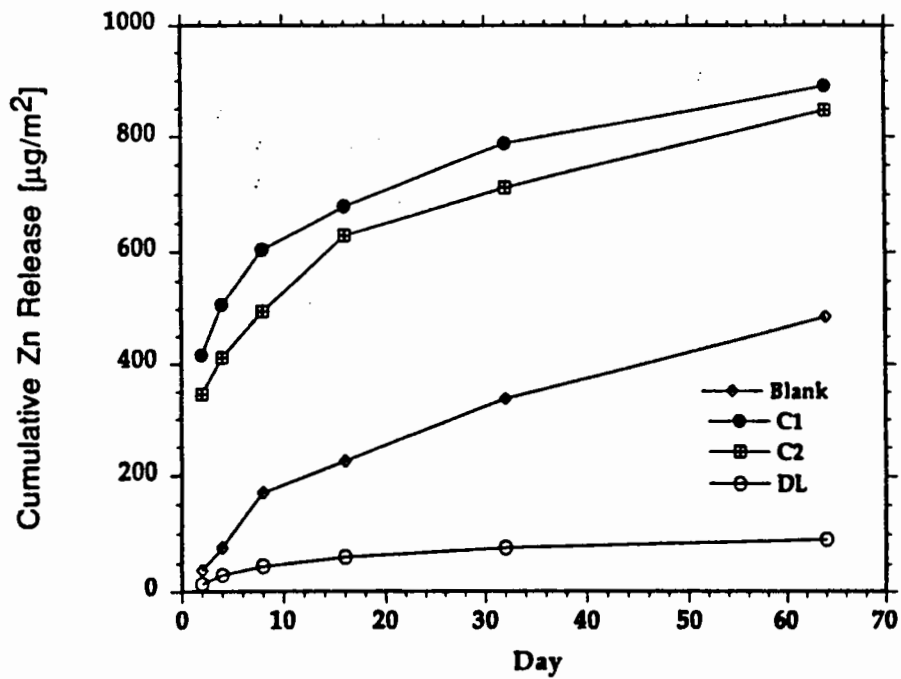


Figure 2. Zinc released per unit area.

TECHNICAL REPORT DATA

(Please read Instructions on the reverse before completing)

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