Polyethylene Encapsulation of Simulated Blowdown Waste for SEG Treatability Study

**Letter Report on Phase I Screening: Waste Treatment and
Specimen Preparation**

Paul D. Kalb

August 17, 1993

**Work Conducted in Support of the
Scientific Ecology Group Treatability Study for the
Savannah River Consolidated Incineration Facility**

**Work Conducted by the
Environmental & Waste Technology Center
Dept. of Advanced Technology
Brookhaven National Laboratory
Upton, NY 11973**

MASTER

ep

1. Introduction

The Environmental and Waste Technology Center is a participating subcontractor in the Scientific Ecology Group (SEG) Treatability Study for Westinghouse Savannah River Co.'s Blowdown Waste. This waste will be generated at the Consolidated Incinerator Facility and will consist of the neutralized aqueous scrubber solution from the incinerator. Since the facility is designed to burn low-level radioactive, hazardous, and mixed wastes, the blowdown waste will likely be a mixed waste.

Polyethylene encapsulation is an improved treatment method that has been developed at BNL over the last 10 years. Polyethylene is an inert, thermoplastic polymer with a melt temperature of 120°C. The BNL process is a modification of standard plastics extrusion technology that has been utilized successfully by the plastics industry for over 50 years. Polyethylene binder and dry waste material are fed through separate calibrated feeders to the extruder, where the materials are thoroughly mixed, heated to a molten condition, and then extruded into a suitable mold. A monolithic solid waste form results on cooling. Compared with conventional hydraulic cement and thermosetting polymer process, polyethylene has several distinct advantages:

- solidification is assured on cooling since no chemical reactions are required,
- a wide range of waste types can be accommodated,
- higher waste loadings can be achieved,
- improved waste form properties are attained (e.g., durability, leachability)
- despite higher binder costs, the process can provide overall cost savings.

Previous development work has shown the process to be effective for a wide range of waste types including nitrates, sulfates, borates and other soluble salt concentrates, sludges, incinerator ash, and ion exchange resins [1,2,3,4].

The objective of the Phase I screening effort was to prepare test specimens of CIF surrogate waste encapsulated in polyethylene for leach testing using EPA's Toxicity Characteristic Leaching Procedure (TCLP). BNL received aqueous CIF surrogate from SEG, pretreated the simulant for processing, and fabricated TCLP test specimens for analysis at an independent laboratory. Laboratory and processing procedures are described in this letter report.

2. Pre-Treatment of the CIF Surrogate

BNL received two batches of CIF surrogate, identified as Batch SM-CIF-006 (approx. 7 liters packaged in glass bottles and 4 liters packaged in plastic bottles) and Batch SM-CIF-007 (approx. 5.2 liters packaged in glass bottles). According to the SEG data sheets (see Simulant Batch Tickets enclosed in the Appendix) the 006 surrogate waste contained 35.3% solids with an specific gravity of 1.265 and the 007 surrogate contained 32.7% solids with an specific gravity of 1.267. Each surrogate contained metal tracers including silver, arsenic, barium, cadmium, chromium, mercury, nickel, lead, antimony, selenium, thallium, and zinc. The concentrations of these metals are shown in Table 1.

Table 1. Metal Concentrations for CIF Surrogate

Compound	Symbol	Concentration, ppm
Silver	Ag	500
Arsenic	As	250
Barium	Ba	500
Cadmium	Cd	125
Chromium	Cr	500
Mercury	Hg	250
Nickel	Ni	500
Lead	Pb	2250
Antimony	Sb	500
Selenium	Se	500
Thallium	Tl	500
Zinc	Zn	500

In order to make the aqueous surrogate compatible with polyethylene extrusion (at process temperatures of approx. 150°C), the solution must first be pre-treated to a dry powder. This was accomplished using a bench-scale stirred vacuum dryer. The dryer has a 2-gallon stainless steel mixing vessel which is heated by a microprocessor controlled oil bath circulation heater. The oil bath was set to 80°C. Vessel temperature was monitored with a flush mounted thermocouple at the mid-section. Liquid temperature was maintained between 68 and 78°C during boiling. Vacuum is maintained by a roughing pump at 28 - 30 in. Hg. At these temperatures and vacuum, vigorous boiling is maintained. Vapor is carried off through stainless steel tubing to a series of three glass condenser columns, cooled by a single pass of cool water. Condensate is collected in glass flasks at the base of each condenser column. The dryer is stirred by two steel and teflon mixing blades that are operated by an orbital planetary drive. The blades rotate on their own axis and around the circumference of the vessel at about 20 rpm. In order to reduce splash and unnecessary carry-over of liquid (especially when the vessel is almost full), the mixing blades were set to cycle for 5 seconds on and 60 seconds off. During the final phase of drying, once a thick paste has been achieved, the blades are operated continuously.

Batch 007 was treated first. Following vigorous shaking, the contents of each of the containers was emptied into the dryer vessel and residual material in the bottles was rinsed using 50 - 100 ml of distilled water. The vessel was sealed, brought to temperature and vacuum conditions, and was processed as described above. After about 5 hours, a thick slurry was observed through the glass viewing port. After another 30 minutes of drying, the mixture appeared dry and a sample was taken for moisture analysis. Percent moisture was determined to be 3.16% using a Sartorius Automatic Moisture Analyzer. Following an additional 40 minutes of drying, the surrogate contained 1.04% moisture and the process was shut down.

Approximately 6 liters (6,761 g) of Batch 006 were then processed in a similar manner, except that 108.5 g of a soluble pre-treatment additive mixture was added prior to heating. This technique was used to reduce leachability of the metal contaminants by reducing their solubility. After drying for approximately 5.5 hours, the surrogate was tested for moisture content and contained 0.74% moisture. Vacuum was maintained, the heat turned off and the stirrer set to mix for 2 seconds every minute to keep the mixture dry while extruder process equipment was being set up. After 2.5 hours, the mixture was removed (1598.7 g were recovered) and sieved through a 1.4 mm screen prior to extrusion processing to eliminate any large particles. This yielded 912.1g of material < 1.4mm in size which was transferred to the metering hopper for extrusion. Complete particle size analyses were not conducted due to schedule constraints, but these data will be determined following pre-treatment of future surrogate batches.

3. Extrusion Processing

The pre-treated CIF surrogate was encapsulated in low-density polyethylene using a 1.25 in. diameter bench-scale extruder with a maximum output of 16 kg/hr. Dry surrogate and polyethylene pellets were fed separately through two loss-in-weight feeders. The feeders are controlled by microprocessors that monitor the instantaneous weight loss as material is delivered. The actual mass feed rate is compared with the pre-set rate and adjustments in delivery speed are made automatically to keep the feed rate within about 1% of the set rate. The two individual controllers are regulated, in turn, by a master controller which monitors and adjusts the delivery of each feeder so that the waste/binder ratio is maintained. The feeders were set to maintain a recipe of 61.5 wt% surrogate waste and 38.5 wt% polyethylene. The master controller maintained an overall delivery rate of 25 g/min (15.4g/min surrogate, 9.6 g/min polyethylene).

The extruder has three separately controlled heating zones and a die zone. Zone temperatures, melt temperature, melt pressure, current draw, and screw speed are monitored during processing. Data for the extrusion of CIF surrogate are given in Table 2.

Table 2. Extruder Parameters for Processing CIF Surrogate

Zone 1, ° F	Zone 2, ° F	Zone 3, ° F	Die Zone, ° F	Melt Temp, ° F	Melt Press., psi	Current Draw, amps	Screw Speed, rpm
280	280	320	331	329	1400- 2500	2 - 3	8 - 20

The molten mixture of CIF surrogate and polyethylene was extruded into teflon pellet molds. The pellet size is approximately 0.25" diameter x 0.25" height. On cooling, the pellets were removed from the molds and weighed. A set of pellets (101.2 g) was sent to SEG for Toxicity Characteristic Leaching Procedure (TCLP) analysis. Results, as supplied by SEG, are presented in Table 3.

Table 3. TCLP Data for 61.5 wt% CIF Surrogate Encapsulated in Polyethylene

Metal	TCLP Concentration, ppm	Allowable Concentration, ppm
Silver	< 0.002	0.072
Arsenic	0.234	5.0
Barium	0.573	100
Cadmium	0.694	0.066
Chromium	< 0.004	0.094
Mercury	0.0008	0.025
Nickel	2.76	0.32
Lead	7.32	0.51
Antimony	1.19	0.15
Selenium	0.121	5.7
Thallium	1.12	0.15
Zinc	6.11	0.05

4. Summary and Conclusions

The target waste loading of about 62 wt% CIF surrogate waste represents a nearly maximum waste loading from the standpoint of processability. At this waste loading, TCLP leachability was above allowable limits (as presented by SEG) for six of the twelve metals. According to SEG, the polyethylene waste loading was considerably higher than those of other potential binders. Therefore, they recommended reducing the waste loading and repeating the TCLP screening tests. For Phase II, (4) replicate sets of polyethylene TCLP specimens will be fabricated at waste loadings of 20, 30, and 40 wt% CIF surrogate waste.

REFERENCES

1. Kalb, P.D., and P. Colombo, "Polyethylene Solidification of Low-Level Wastes," BNL-51867, Brookhaven National Laboratory, Upton, NY, October 1984.
2. Kalb, P.D., J.H. Heiser, and P. Colombo, "Polyethylene Encapsulation of Nitrate Salt Wastes: Waste Form Stability, Process Scale-Up, and Economics," BNL-52293, Brookhaven National Laboratory, Upton, NY, July 1991.
3. Kalb, P.D., and M. Fuhrmann, "Polyethylene Encapsulation of Single-Shell Tank Wastes," BNL-52365, Brookhaven National Laboratory, Upton, NY, September, 1992.
4. Kalb, P.D., J.H. Heiser, and P. Colombo, "Long-Term Durability of Polyethylene for Encapsulation of Low-Level Radioactive, Hazardous, and Mixed Wastes," Emerging Technologies in Hazardous Waste Management III, D.W. Tedder and F. G. Pohland, eds., American Chemical Society Symposium Series 518, Washington, DC, 1993.

APPENDIX

WSRC-P-CIF-002, SAVANNAH RIVER CIF TREATABILITY TEST PLAN

ENCLOSURE 9.1 Simulant Batch Ticket

Date: 6/23/93

Batch # SM-CIF-0060

- 7.3.1 Quantity of simulant required 14 kg
 7.3.2 Quantity demin. water start 5600 ml
5600 gm
 7.3.3 Quantity Sodium Acetate added 2478 gm
 7.3.4 Quantities of metal compounds:

Constituent	Lot #	Manuf.	Wt.	Constituent	Lot #	Manuf.	Wt.	Constituent	Lot #	Manuf.	Wt.
Silver	T1	GFS	9.328	Chromium	T1	GFS	35.856	Antimony	X1	GFS	13.125
Arsenic	H1	GFS	10.750	Mercury	T1	GFS	4.726	Selenium	H1	GFS	9.946
Barium	X2	GFS	12.412	Nickel	X1	GFS	28.332	Thallium	0310 KXSY	Aldrich	9.832
Cadmium	09102N	Reagents	2.873	Lead	X2	GFS	47.098	Zinc	R1	GFS	15.595

- 7.3.5 Simulant pH 6.24
 7.3.6 HCl added 99 gm
 7.3.7 New pH 5.78
 7.3.8 Quantity of NaCl added 1637 gm
 7.3.10 Solution pH 5.72
 7.3.11 Quantity NaOH added N/A gms; Final pH 5.73
 7.3.12 Quantity perlite added 1260 gm
 7.3.13 Specific gravity 1.2165
Volume 8157 ml
 7.3.15 Radioactive sources added
 Cs-137 N/A pCi
 U-235 N/A mg
 7.3.16 Non-radioactive tracers added:
 Cesium Chloride 18.4 mg
 Cerium Chloride 23.8 mg
 7.3.17 Water added 2464 gm
 7.3.18 Percent solids 35.3 %
 7.3.19 TSS 9.59 %

Remarks: N/A

Tim Hall 6/24/93 Tom Miller 6/23/93
 SEG Treatability Man. date Technician date
Carl J. Ross 7/6/93
 SEG QA date

WSRC-P-CIF-002, SAVANNAH RIVER CIF TREATABILITY TEST PLAN

ENCLOSURE 9.1 Simulant Batch Ticket

Date: 6/24/93

Batch # SM-CIF-007

- 7.3.1 Quantity of simulant required 6 kg
 7.3.2 Quantity demin. water start 2400 ml
2400 gm
 7.3.3 Quantity Sodium Acetate added 1178 gm
 7.3.4 Quantities of metal compounds:

Constituent	Lot #	Manuf.	Wt.	Constituent	Lot #	Manuf.	Wt.	Constituent	Lot #	Manuf.	Wt.
Silver	T1	GFS	3910	Chromium	T1	GFS	15.340	Antimony	X1	GFS	5.621
Arsenic	H1	GFS	4.616	Mercury	T1	GFS	2.014	Selenium	H1	GFS	4.227
Barium	X2	GFS	5.350	Nickel	X1	GFS	12.143	Thallium	OS10 KX2Y	Aldrich	3.525
Cadmium	09102N	TN Reagents	1.223	Lead	T2	GFS	18.111	Zinc	R1	GFS	6.228

- 7.3.5 Simulant pH 6.32
 7.3.6 HCl added 53 gm
 7.3.7 New pH 5.70
 7.3.8 Quantity of NaCl added 701 gm
 7.3.10 Solution pH 5.70
 7.3.11 Quantity NaOH added N/A gms; Final pH 5.70
 7.3.12 Quantity perlite added 540 gm
 7.3.13 Specific gravity 1.267
 Volume 3850 ml
 7.3.15 Radioactive sources added
 Cs-137 N/A pCi
 U-235 N/A mg
 7.3.16 Non-radioactive tracers added:
 Cesium Chloride 8.3 mg
 Cerium Chloride 12.3 mg
 7.3.17 Water added 1046 gm
 7.3.18 Percent solids 32.7 %
 7.3.19 TSS 12.29 %

Remarks: Due to the decreasing time it takes to perform the study the
amount of water being evaporated is not as great as previously seen.
Therefore, this would decrease the % solids in solution

Tim Hall / 6/24/93 - Tom Nicks / 6/24/93
 SEG Treatability Man. date Technician date
Carol Riser / 7/6/93
 SEG QA date

**DATE
FILMED**

4 / 11 / 94

END