Task 3 - Pyrolysis of Plastic Waste

Semi-Annual Report

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TASK 3 - PYROLYSIS OF PLASTIC WASTE

1.0 INTRODUCTION/BACKGROUND

Over the last 50 years, the U.S. Department of Energy (DOE) has produced a wide variety of radioactive wastes from activities associated with nuclear defense and nuclear power generation. These wastes include low-level radioactive solid wastes, mixed wastes, and transuranic (TRU) wastes. A portion of these wastes consists of high-organic-content materials, such as resins, plastics, and other polymers; synthetic and natural rubbers; and cellulosic-based materials; and oils, organic solvents, and chlorinated organic solvents. Many of these wastes contain hazardous and/or pyrophoric materials in addition to radioactive species. Physical forms of the waste include ion-exchange resins used to remove radioactive elements from nuclear reactor cooling water, lab equipment and tools (e.g., measurement and containment vessels, hoses, wrappings, equipment coverings and components, and countertops), oil products (e.g., vacuum pump and lubrication oils), bags and other storage containers (for liquids, solids, and gases), solvents, gloves, lab coats and anticontamination clothing, and other items. Major polymer and chemical groups found in high-organic-content radioactive wastes include polyvinyl chloride (PVC), low-density polyethylene (LDPE), polypropylene (PP), Teflon™, polystyrene (PS), nylon, latex, polyethylene terephthalate (PET), vinyl, high-density polyethylene (HDPE), polycarbonate, nitriles, Tygon®, butyl, and Tyvec®.

Costs associated with the accumulation, storage, and disposal of high-organic-content radioactive wastes are high because of irregularities in shape, volume, and composition. Storage of combinations of these contaminated materials in sealed barrels and other containers can cause degradation reactions that yield a wide range of radioactive products (many of which have some degree of volatility and/or environmental mobility) and other hazardous materials, including hydrogen gas. Options for dealing with high-organic-content radioactive waste include volume reduction and storage. Large quantities of these wastes are currently being stored in barrels and casks. One waste reduction option involves separating wastes into combustibles and noncombustibles and then incinerating the combustibles to yield ash and gas. The radioactive component of the waste is reduced in volume and can be stored more easily. Difficulties associated with this approach include the potential for entrainment of radioactive species in the product gas stream and volatilization of radioactive species during the high-temperature combustion process. On-line and full-stream gas analysis systems are being developed to monitor emissions more accurately, but controlling volatile emissions is limited by physical constraints and statistical probabilities.

The University of North Dakota Energy & Environmental Research Center (EERC) is developing a process for efficient, complete separation of radionuclides from high-organic-content radioactive waste. The process is a low-temperature thermal decomposition-separation technology that will yield a small volume of particulate solids product containing radioactive species, a nonradioactive organic condensable gas product, and a nonradioactive hydrocarbon-rich gas product. By controlling process conditions, the yield of condensable gas product can be varied from 20 to 80 wt% of the feed material, with the remaining organic content of the feed material converted to gas. Processing at a low reactor temperature (600°C or below) ensures against

radionuclide volatilization and results in a high condensable versus a high noncondensable gas yield.

The EERC thermal decomposition-separation process for radionuclide separation is not a combustion process. The process will accomplish the following:

- Decompose, volatilize, and recover for (disposal) the condensable organic content of a waste
- Volatilize and capture chlorine (which may be present in the waste as either organic or inorganic materials)
- Concentrate radionuclide species in a dry particulate solids product

The process will be applicable to the separation of radionuclides from the following waste streams:

- Low-level radioactive solid waste, defined as solid radioactive waste that is not classified as high-level waste, TRU waste, or spent nuclear fuel as defined in DOE Order 5820.2A. This category is generally used to refer to wastes that are radioactive but do not contain components classified as hazardous.
- Mixed wastes, defined as wastes that contain both radioactive and hazardous components as defined by the Atomic Energy Act and the Resource Conservation and Recovery Act (RCRA), including solvents, pyrophoric substances, and other chemically contaminated items. Wastes under this category are of special importance 1) because no current plan for their treatment has been accepted and 2) because of their chemical diversity, these wastes could react during containment to yield a wide variety of products, many of which may be volatile, reactive, ignitable, toxic, or otherwise hazardous.
- TRU waste, defined as waste that has a radioactivity greater than 100 nanocuries per gram (nCi/g) and is contaminated with alpha-emitting radionuclides that have an atomic number greater than 92 and a half-life of greater than 20 years.

Inventories of low-level, mixed, and TRU wastes accumulated throughout the United States as of 1993 have been compiled and are detailed in a report prepared by Oak Ridge National Laboratory entitled "Integrated Data Base Report - 1993: U.S. Spent Nuclear Fuel and Radioactive Waste Inventories, Projections, and Characteristics" (DOE/RW-0006, Rev. 10, December, 1994). A significant portion of annually generated radioactive organic waste is made up of spent (radionuclide-loaded) ion-exchange resins from nuclear power facilities, and in addition to nuclear defense and power generation activities, large volumes of low-level radioactive wastes are generated by hospitals and medical research facilities. The EERC technology is being developed for application to these and other waste streams.

2.0 OBJECTIVES

2.1 General Project Objectives

The EERC is developing a technology for the thermal decomposition of high-organic-content, radionuclide-contaminated mixed wastes that will enable the separation and concentration of radionuclides as dry particulate solids and the generation of nonradioactive condensable and noncondensable gas products. Successful application of the technology will enable a significant volume reduction of radioactive waste and the production of an inexpensively disposable nonradioactive organic product. The objective of the proposed effort is to develop and demonstrate the commercial viability of a continuous thermal decomposition process that can fulfill the following requirements:

- Separate radionuclides from radioactive waste streams containing a variety of types and levels of polymers, chlorinated species, and other organics, including rubber, oils, resins, and cellulosic-based materials.
- Concentrate radionuclides in a homogeneous, dry particulate product that can be recovered, handled, and disposed of efficiently and safely.
- Separate and recover any chlorine present (as PVC, chlorinated solvents, or inorganic chlorine) in the contaminated mixed-waste stream.
- Yield a nonradioactive, low-chlorine-content, condensable organic product that can be economically disposed.

2.2 Focused Objective – Volume Reduction of Ion-Exchange Resins

As a result of ongoing discussions with personnel at commercial nuclear waste-processing facilities and Stone & Webster Engineering Corporation (subcontracted by the EERC to provide guidance on process optimization and radioactive waste stream targeting), the EERC decided to target application of the thermal decomposition process to volume reduction of spent (radioactive) ion-exchange resin. Commercial application of the technology to spent ion-exchange resin, which accounts for about 50,000 to 200,000 cubic feet per year of the total annual U.S. nuclear waste volume of 500,000 to 700,000 cubic feet, would establish the viability of the process and help in its application to other less homogeneous waste streams. Economic motivation is provided by the cost of landfill disposal, which is about \$400 per cubic foot, since contaminated resin is categorized as a Class A nuclear waste. A primary project objective is to effectively reduce this cost to \$200 per cubic foot by economic optimization of the volume reduction process.

3.0 ACCOMPLISHMENTS/WORK PERFORMED

3.1 Mixed-Waste Plastics Test Results

Using the continuous fluid-bed reactor (CFBR) system described in a prior (April–October 1995) semiannual report, a series of tests were performed at a reactor feed rate of about 1 kg/hr of

mixed-waste plastics with added calcium oxide (for catalytic activity and chlorine capture), virgin polymer resins (to more closely simulate potential organic radioactive waste streams), and radionuclide surrogates. Three reactant mixtures were prepared. Tables 1 and 2 provide composition data on the plastics-plus-calcium oxide portion and the radionuclide surrogate portion, respectively, of each reactant mixture. With each feedstock mix, three tests were performed at three different temperatures: 450°, 500°, and 600°C. Table 3 lists reaction parameters and sampling procedures for the nine-test series. In Table 1, "Addition Polymer" refers to the fact that Tyvec® and ABS (acrylonitrile butadiene styrene) are addition polymers, and "Condensation Polymer" refers to the fact that nylon and polycarbonate are condensation polymers. Water addition is often advantageous to reactor operation when processing reactant mixtures with high levels of condensation polymer, because the presence of water enables monomer formation through hydrolysis reactions. Without water, decomposing condensation polymers have a greater tendency to form reactor-plugging coke.

TABLE 1

Postconsumer Plastics-Based Reactant Mixture Compositions			
Reactant Mixture, wt%	Postconsumer (Mix D)	Addition Polymer (Mix E)	Condensation Polymer (Mix F)
Postconsumer Plastics Content	95	85	85
Calcium Oxide Content	5	5	5
Tyvec Content		5	
ABS Content		5	
Nylon Content			5
Polycarbonate Content			5

TABLE 2

Radionuclide Surrogate Concentrations in Reactant Mixtures D, E, and F		
Radionuclide Surrogate	Added as:	Surrogate-Only Concentration, $\mu\text{g/g}$
Cesium (Cs)	Cesium Chloride (CsCl)	2810
Strontium (Sr)	Strontium Nitrate ($\text{Sr}(\text{NO}_3)_2$)	1810
Zirconium (Zr)	Zirconium Chloride (ZrCl_4)	1710
Cerium (Ce)	Cerium Chloride Hydrate	1630
Titanium (Ti)	Titanium Chloride (TiCl_3)	740
Vanadium (V)	Vanadium Pentoxide (V_2O_5)	2000
Tin (Sn)	Sn	2740
Antimony (Sb)	Sb	3580
Copper (Cu)	Cu	2740

TABLE 3

Postconsumer/Surrogate Test Reaction Parameters and Sample Procedures	
Reaction Temperature	450°, 500°, and 600°C
Reaction Pressure	30 psig
Reactant Mixture Feed Rate	1.0 kg/hr
Fluidization Gas Velocity	0.75 linear feet per second
Water Injection Rate	No water injection
Condenser Exit Temperature	25°–30°C
Postcondenser Gas Scrubber Solution	1.0 M potassium hydroxide
Pretest Stabilization Period Duration	2 hours
Balance Period Duration	4 hours
Solids Residual Sample Collection	Cumulative, sample collected over duration of balance period
Condensable Gas Sample Collection	Cumulative, sample collected over duration of balance period
Noncondensable Gas Sample Collection	Gas bag samples collected at midpoint and end of balance period

Following each test, each solids residual was weighed, ground (to ensure homogeneity), and sampled for ash and surrogate quantitation. Each condensable gas product was weighed, blended (to ensure homogeneity), and sampled for surrogate quantitation. Each noncondensable gas sample was analyzed for quantitation (mole percent basis) of hydrogen, methane, acetylene, C_2 – C_4 paraffins and olefins, carbon monoxide, carbon hydroxide, oxygen, nitrogen, carbonyl sulfide, and hydrogen sulfide. Based on the mole percent data, an average molecular weight is calculated for each gas sample, which is used in conjunction with total balance period gas volume data to calculate a total weight of product gas generated during the balance period. The total balance period weights of solids residual, condensate, and product gas are used to calculate material balances for each test.

The objective of these tests was to investigate the effectiveness of the EERC thermal decomposition process in concentrating radionuclide surrogates in a solids residual while yielding a surrogate-free condensate product. Tables 4 and 5 show preliminary surrogate quantitation data for 450°C tests performed with Mix E (with added addition polymers) and Mix F (with added condensation polymers), respectively.

The data in Tables 4 and 5 indicate that, with the exception of tin (which has a boiling point of about 232°C), all of the surrogates were concentrated in the solids residual, but that some carryover occurred, as noted by the presence of detectable levels of antimony, titanium, and cesium. The problem of solids carryover and how it is being addressed are discussed in Section 3.2.

TABLE 4

Surrogate Concentrations in Test M549 (450°C) Products¹

Surrogate	Feedstock	Solids Residual ²	Condensate	Postcondenser Scrubber Solution ³
V	2000	9030	<2	NA ⁴
Sn	2740	1410	6	NA
Sb	3580	15,300	2	<60
Cu	2740	21,800	<7	NA
Ti	740	1400	6	NA
Sr	1810	11,500	0.3	NA
Ce	1630	5800	<50	NA
Zr	1710	3520	<1	NA
Cs	2810	9640	0.4	15

¹ All concentration values are averages of duplicate analyses, and unless otherwise noted, all values are in micrograms per gram ($\mu\text{g/g}$).

² Samples consist of all solids recovered from reactor overflow and the cyclone particulate removal system over the balance period; a portion of the solids generated during each balance period is unrecovered and remains as reactor bed material.

³ Concentration values given as micrograms per liter ($\mu\text{g/L}$).

⁴ Not analyzed.

TABLE 5

Surrogate Concentrations in Test M550 (450°C) Products¹

Surrogate	Feedstock	Solids Residual ²	Condensate	Postcondenser Scrubber Solution ³
V	2000	8500	<2	NA ⁴
Sn	2740	1270	12	NA
Sb	3580	23,500	1	<60
Cu	2740	15,000	<7	NA
Ti	740	1460	6	NA
Sr	1810	17,300	<0.3	NA
Ce	1630	6610	<10	NA
Zr	1710	4280	<1	NA
Cs	2810	13,800	0.4	50

¹ All concentration values are averages of duplicate analyses, and unless otherwise noted, all values given are in $\mu\text{g/g}$.

² Samples consist of all solids recovered from reactor overflow and the cyclone particulate removal system over the balance period; a portion of the solids generated during each balance period is unrecovered and remains as reactor bed material.

³ Concentration values given as $\mu\text{g/L}$.

⁴ Not analyzed.

3.2 Ion-Exchange Resin Test Results

A fluid-bed test was conducted using Rohm and Haas Amberlite® IRN-150 nuclear-grade resin, into which approximately 30 grams per cubic foot of cesium was ion-exchanged, according to procedures provided by Stone & Webster, as feedstock. The 1.0-kg/hr test was performed at a reactor temperature of 550°C and a pressure of 30 psig. Steady-state conditions were maintained for an approximate 2-hour balance period, during which solids residual and condensed vapor samples were accumulated. Product gas samples were collected at the midpoint and the end of the balance period. Analytical results from the resin test, provided in Table 6, show that the cesium level in the condensed, filtered (through a 0.45-micron filter) offgas material was less than the current EERC analytical detection limit of 400 nanograms per gram (ng/g). However, the cesium concentration in unfiltered condensate was determined to be about 4 to 20 $\mu\text{g/g}$, which indicates that fine particulate material passed through the reactor cyclone. This finding led to consideration of reconfiguring the reactor to reduce the probability of reactor carryover.

TABLE 6

Cesium Separation from Ion Exchange Resin – M553 CFBR Test Results	
Feedstock Cesium Concentration, g/ft ³	25
Overall Mass Balance, %	106
Normalized Product Yields, wt%	
Solids	3
Condensable Organic Vapors	60
Noncondensable Gas	37
Cesium Separation ¹	
Ion-Exchange Resin Volume Reduction, %	97
Cesium Balance, wt%	78
Calculated wt% of Cesium Recovered in Solids Residual	99.998
Cesium Concentration in Condensed Offgas, ng/g	< 400

¹ All cesium analyses performed in duplicate using acid digestion followed by quantitation with atomic absorption spectroscopy, and verified at the Radiation Center at Oregon State University using neutron activation analysis.

In all tests with radionuclide surrogates, the small quantities of surrogates observed in product gas streams appeared to be present as particulates, since the surrogates were removable with filtration. While the small particulate loadings observed would be acceptable for most

industries, the presence of radioactive species makes any amount of particulate emissions unacceptable. It should be noted that probably any system will produce a limited amount of reactor carryover and/or will require some form of emission control to provide protection against a system upset.

The EERC objective is to establish a reactor system configuration that provides the best radioactive volume reduction and the most safety at the least capital, operational, and maintenance costs. After analyzing the results of the CFBR resin test, a decision was made to evaluate the use of an auger reactor in place of the fluid-bed reactor. This decision was based on performance and cost considerations. It is likely that significant capital and operational cost savings could be achieved by reducing the required capacity of postreactor particulate and sulfur control devices, which is achievable provided that the amount of fluidization gas (or, in the case of an auger reactor, purge gas) flowing through the reactor is reduced. This would reduce the amount of gas requiring treatment and the velocity of product gas leaving the reactor, which would result in a reduction in particle entrainment. The auger reactor was selected as an alternative to the fluid bed because it can be operated with a reduced amount of purge gas, since reactant fluidization is not required.

Figures 1 and 2 are schematics of the EERC thermal depolymerization process configured with a fluid-bed and an auger reactor, respectively. Upcoming tests (planned for early November 1996) will be performed using the recently completed auger reactor. Throughout construction and

Figure 1. EERC thermal decomposition process application to volume reduction of radioactive ion-exchange resin—fluid-bed reactor-based configuration.

Figure 2. EERC thermal decomposition process application to volume reduction of radioactive ion-exchange resin—auger reactor-based configuration.

installation of the auger reactor, EERC staff have maintained contact with personnel at GTS Duratek (a commercial nuclear waste-processing company) who are interested in application of the EERC process to ion-exchange resin. Duratek has supplied resin feedstocks to the EERC for demonstration testing and provided information regarding desired process throughput and related work on resin processing. It is anticipated that a Duratek staff member will visit the EERC to observe operation of the reactor system during the current reporting period.

4.0 WORK PLANNED FOR THE NEXT SIX MONTHS

Although systems analysis indicates that the auger reactor should be superior to the fluid bed in processing spent ion-exchange resin, it must be proven technically. EERC personnel are familiar with several projects in which rotary reactors for soil treatment, coal gasification, or tire pyrolysis have been modified to treat high-polymer-content streams. The success rate has been low to medium, primarily because of lack of knowledge regarding the effects of thermal depolymerization on specific polymers and polymer combinations.

Over the last two years the EERC has demonstrated with a variety of feedstocks and radionuclide surrogates that thermal decomposition is a viable approach to separating radioactive materials from an organic matrix. The next ongoing step is to optimize the process as applied to ion-exchange resin based on a performance evaluation and a cost/benefit analysis comparing an auger reactor-based system with a fluid-bed-based system. Issues that need to be addressed include the following:

- Ensuring that the resin polymer does not form a sticky liquid that reduces heat-transfer capacity and plugs up the solids removal system
- Ensuring that feed material flows freely through the feed system
- Ensuring that liquids do not collect in the offgas-sampling system
- Deciding whether inert material should be added to the feedstock to enhance reactor performance
- Determining whether the process will generate a dioxin problem
- Ensuring process safety

The fluid-bed option has been demonstrated with ion-exchange resins at the bench scale and has been demonstrated at near-commercial scale with postconsumer plastics. The viability of the auger reactor, which may provide performance and cost advantages, has not yet been demonstrated. Table 7 provides a description of the three tests that are planned. Since this is a shakedown series, parameters may be adjusted as results warrant. The primary objective of the test series is to determine whether the auger reactor will perform better than the fluid-bed system, as measured by the ability of the auger-based process to yield a cesium-free, condensable gas product. Tests will be conducted with and without a postreactor packed-bed filter for chlorine- and sulfur-species removal.

TABLE 7

Planned Ion-Exchange Test Series for Auger Reactor Evaluation

Test Number	M554	M555	M556
Temperature, °C	600	550	500
Pressure	Ambient	Ambient	Ambient
Gas Residence Time, seconds	6	6	6
Solids Residence Time, minutes	30	30	30
Feed Rate, kg per hour	1	1	1

**U.S. DEPARTMENT OF ENERGY
FEDERAL ASSISTANCE MANAGEMENT SUMMARY REPORT**

1. Program/Project Identification No. DE-FC21-94MC31388	2. Program/Project Title EM Task 3 - Pyrolysis of Plastic Waste	3. Reporting Period 7/1/98 through 9/30/98
4. Name and Address Energy & Environmental Research Center University of North Dakota PO Box 9018, Grand Forks, ND 58202-9018 (701) 777-5000		5. Program Start Date 9/30/94 6. Completion Date 9/29/99

7. FY 95/96	8. Months or Quarters Quarters	b. Dollar Scale	1st OCT	2nd NOV	3rd DEC	4th JAN	5th FEB	6th MAR	7th APR	8th MAY	9th JUN	10th JUL	11th AUG	12th SEP
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9. Cost Status **a. Dollars Expressed In Thousands**

10. Cost Chart

Fund Source		Quarter				Cum. to Date	Tot. Plan
		1st	2nd	3rd	4th		
DOE	P	3	7	100	80	190	590
	A	3	7	19	80	109	
	P						
	A						
	P						
	A						
	P						
	A						
Total P		3	7	100	80	190	590
Total A		3	7	19	80	109	
Variance		0	0	81	50	81

P = Planned A = Actual		c. Cumulative Accrued Costs											
Total Planned Costs for Program/Project \$590	Planned			3			10			110			190
	Actual			3			10			29			109
	Variance			0			0			81			81

11. Major Milestone Status	Units Planned	
	Units Complete	
3.1 Literature Review and Test Matrix Development	P	
	C	
3.2 Single-Component CFBR Testing	P	
	C	
	P	
	C	
	P	
	C	
	P	
	C	
	P	
	C	
	P	
	C	
	P	
	C	

12. Remarks

13. Signature of Recipient and Date 1-11-96	14. Signature of DOE Reviewing Representative and Date
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Total A		53	82	117	74	435																																																																																																			
Variance		2	(54)	103	23	155																																																																																																			
		c. Cumulative Accrued Costs																																																																																																							
Total Planned Costs for Program/Project \$590		Planned		245		273		493		590																																																																																															
		Actual		162		244		361		435																																																																																															
		Variance		83		29		132		155																																																																																															

11. Major Milestone Status	Units Planned	
	Units Complete	
3.3a - CFBR Radionuclide Test Program	P	
	C	
3.3b - Surrogate Sample Transfer and Analysis Verification	P	
	C	
3.4a - Lab-Scale Radionuclide Verification Program	P	
	C	
3.4b - Lab-Scale Radionuclide Verification Test Program and Technology Transfer	P	
	C	
3.5a - Pilot Plant Test Matrix and Technology Transfer Development	P	
	C	
3.5b - Pilot Plant Test Program	P	
	C	
3.6 - A&E Process Technological and Economic Assessment	P	
	C	
3.7 Final Report	P	
	C	

12. Remarks

13. Signature of Recipient and Date 1-11-96	14. Signature of DOE Reviewing Representative and Date
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**U.S. DEPARTMENT OF ENERGY
FEDERAL ASSISTANCE MANAGEMENT SUMMARY REPORT**

1. Program/Project Identification No. DE-FC21-94MC31388	2. Program/Project Title EM Task 3 - Pyrolysis of Plastic Waste	3. Reporting Period 7/1/96 through 9/30/96
4. Name and Address Energy & Environmental Research Center University of North Dakota PO Box 9018, Grand Forks, ND 58202-9018 (701) 777-5000		5. Program Start Date 9/30/94
		6. Completion Date 9/29/99

Milestone ID. No.	Description	Planned Completion Date	Actual Completion Date	Comments
Year 1				
3.0	Pyrolysis of Plastic Waste			
3.1	Literature Review and Test Matrix Development	3/95		100%
3.2	Single-Component CFBR Testing	9/95		100%
Year 2				
3.3a	CFBR Radionuclide Surrogate Test Program	5/96	8/96	100%
3.3b	Surrogate Sample Transfer and Analysis Verification	6/96		50%
3.4a	Lab Scale Radionuclide Verification Program	7/96		
3.4b	Lab-Scale Radionuclide Verification Test Program and Technology Transfer	7/96		50%
3.5a	Pilot Plant Test Matrix and Technology Transfer Development	7/96		20%
3.5b	Pilot Plant Test Program	7/96		20%
3.6	A&E Process Technological and Economic Assessment	7/96		70%
3.7	Pilot Plant Test Matrix and Technology Transfer Development	7/96		20%