AZTECH SYSTEMS AND TESTING

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ABSTRACT

The General Electric Company, Nuclear Energy Business Operations, has developed an advanced technology radwaste system known as the AZTECH volume reduction and solidification system. This system will be used for the treatment of low-level waste streams typically encountered in BWR and PWR plants. This paper discusses the systems and approach used for development of the AZTECH process, as well as waste form qualification testing performed by GE to satisfy the 10CFR61 licensing requirements. The AZTECH process development equipment included bench scale, pilot plant and full scale demonstration systems. The qualification testing program follows the specific 10CFR61 requirements guidance, including test standards, provided in the NRC Branch Technical Position (BTP) on waste form. The basic premise of this unique testing plan for AZTECH qualification (NRC approval) was to prepare samples for analysis using actual (representative) processing in a pilot plant and a demonstration plant for full-scale (55-gallon drum) correlation. Samples were analyzed by an independent laboratory and the results were provided to the NRC in a Licensing Topical Report (LTR). Simulated waste forms of sodium sulfate, boric acid, powdered resin, bead resin and a typical decontamination solution were tested. Simulated waste samples containing non-radioactive tracers (cobalt, cesium and strontium) were used for leachability and immersion testing. A unique advantage of the approach used by GE in developing this test plan is representative and full-scale correlated testing which will allow future testing of simulated customer waste streams using the AZTECH pilot plant.

BACKGROUND

The name AZTECH (trademark of General Electric Company) is derived from the unique General Electric patented process which uses low temperature AZeotropic vacuum distillation TECHnology to remove water from the various radioactive waste feed streams and encapsulates the resulting dried solids into a dense, plastic monolithic solid which meets or exceeds all U.S. Nuclear Regulatory Commission (NRC) requirements for long term storage at low-level waste sites. The AZTECH system is suitable for applications to treatment of low-level waste streams typically encountered in BWR and PWR plants. Following conceptualization and patenting, development of the AZTECH process using simulated waste streams was initiated. The AZTECH process represents four years of development by General Electric wherein the process has proceeded stepwise from laboratory phase, pilot plant-scale phase and then through a full-scale demonstration plant phase. The relative product capacities of these systems were 1 gallon, 5 gallons, and 220 gallons (four 55-gal drums), respectively. This effort has provided substantial confidence in the process and in the quality of the final products. Throughout this development, emphasis was placed on safety, attainment of a superior product quality, and simplicity of operation.

The essential portions of the AZTECH process are shown in Fig. 1.

Integrated bench-scale testing was initiated in 1981 at the General Electric Vallecitos Nuclear Center (VNC) to evaluate fundamental process parameters for the AZTECH process. Previous work at General Electric's Plastics Applications Laboratory in Louisville, Kentucky focused on polyester selection and polymerization while work at General Electric's Pittsfield, Massachusettes Plastics Facility was targeted toward development of azeotropic technology.

The initial basis for the AZTECH process was the formation of a vapor-phase, water-styrene azeotrope over a boiling (distilling) mixture of a styrene-containing resin and an aqueous solution or slurry of waste materials. These waste materials included sodium sulfate, boric acid, powdered and bead resins, decontamination solutions, ash, oils, etc. The azeotropic vapor was condensed and separated into impurity-free water and styrene. The water was discarded and the styrene was returned to the distillation vessel.

For the initial integrated testing wherein AZTECH product solids were produced, toluene was substituted for the styrene-containing polyester.

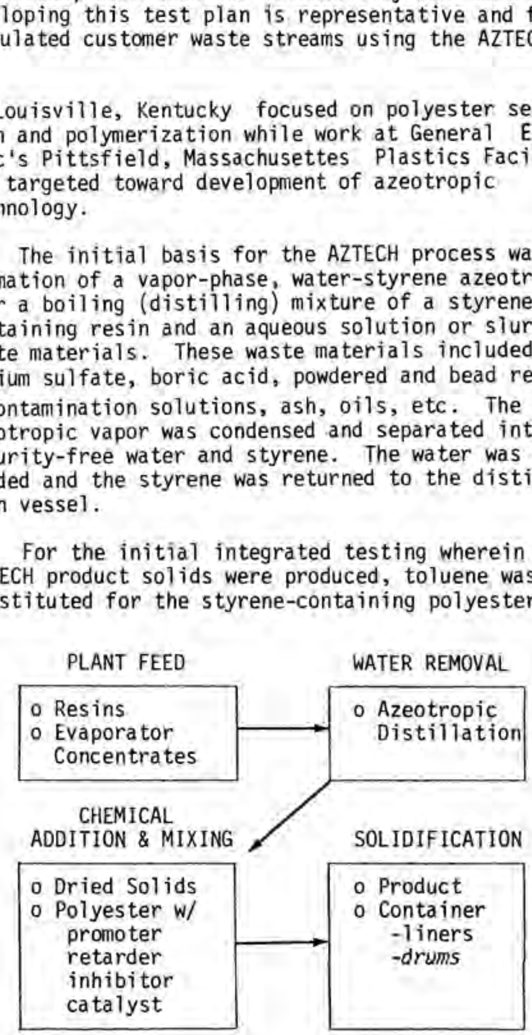

Fig. 1. Laboratory scale development.

This substitution was done so that pure sodium sulfate crystals produced by the AZTECH process could be isolated for further study without dissolution of unpolymerized polyester. Previous research had shown that water and toluene also formed an azeotrope similar to water and styrene, so sodium sulfate crystal production was feasible. The crystals produced using toluene were primarily used for physical property and polymerization parameter development studies. Only limited process testing was conducted using toluene and the bench-scale testing was predominantly accomplished with styrene-containing polyester. The products produced were crystals or other dehydrated solids (depending on the waste form) thoroughly mixed and coated with polyester resin. The resin was then polymerized and the encapsulated waste (greatly reduced in volume) was formed into the final monolithic solid.

The bench-scale tests demonstrated the AZTECH process to be very feasible and safe to operate. The fundamental azeotropic process was demonstrated with various materials. Process parameters such as temperatures, pressures, and compositions were investigated. Also, waste feedstock particle size, solids content and various mixes were tested. Compression tests of all typical waste products far exceeded NRC criteria (>50 psi) and ranged from 330 to 18,300 psi with the majority of anticipated waste products in the range of 3000 to 9000 psi. Although leach indices were not routinely calculated, leach test results for anticipated waste products by the weight loss method were good.

PILOT PLANT DEVELOPMENT

Pilot plant testing was conducted at VNC between October, 1981 and February, 1983. Most of the anticipated simulated nuclear power plant waste forms were tested under actual process conditions.

Pilot plant testing in process equipment scaled up from bench testing was conducted to further develop and demonstrate the AZTECH process, including various process parameters, and to evaluate the resultant waste products. The pilot plant was designed based upon knowledge gained during evolution of the bench-scale process equipment system. Operation of the pilot plant proved to be less complex than bench-scale processing and was essentially trouble-free due to its design configuration and to the presence of more sophisticated automatic controls.

One major process change from the bench-scale process was made during pilot plant testing: the styrene-containing polyester resin was replaced with vinyltoluene-containing polyester and finally with pure vinyltoluene (VT) for the dehydration(azeotropic) portion of the process. When waste dehydration is complete, the VT is removed by evaporation and VT-containing polyester is mixed with the dry solids to form the final solid product. This change was made to improve processing and to assure added safety for the operation, since the flammable region for VT is further removed from the normal operating limits than the corresponding region for styrene.

Sodium sulfate, boric acid, ion exchange resins, decontamination solution, incinerator ash, detergent waste feeds and several combination mixtures were tested using at least 17 different process variations in terms of temperature, pressure, and operational sequence. A total of 80 actual process test runs were conducted. Highlights of the knowledge acquired during testing include the following:

a. The composition of the water-VT azeotrope was found to be fairly consistent at pressures ranging from 130 to 748 mm Hg for the pure components. Somewhat more variability can be expected during full-scale processing, depending upon the degree of water removal from the waste.

b. The water distilled from the waste was saturated with approximately 90 ppm of VT. This concentration could be reduced to 2 ppm by simple aeration.

c. VT was found to thicken and to spontaneously become partially polymerized upon prolonged exposure to elevated temperatures. An empirical equation was developed to predict this occurrence and to set time-temperature inhibitor limits for processing.

d. Some waste compositions such as boric acid and incinerator ash foamed during processing at low pressure. This discovery led to the design of increased mixer-evaporator (M-E) freeboard in the demonstration plant and commercial modular designs.

e. Polymerization of the final product could be successfully carried out with oven heating, microwave heating, internal (in-product) heat sources, and chemically-activated exothermic heating. Demonstration plant testing has shown the latter to be the most practical for commercial application.

The final products of this testing were generally very good. As in bench-scale testing, compression tests of all typical waste products ranged from 370 to 21,600 psi. Leach test results were good for all anticipated waste products produced by typical processes.

The pilot plant equipment system consists primarily of: (1) an Atlantic Research Model SC 5-gal M-E; (2) a 9 kW water heater for M-E heating; and (3) a vacuum pump for generation of a low-pressure M-E environment. Other associated equipment items include a condenser, a decanter vessel for separation of condensed aqueous and organic phases, collection tanks for water and organic liquids, and feed makeup tanks. A photograph of the pilot plant system is shown in Fig. 2. This system with minor modifications, including an M-E configured to the modular design, is scheduled to remain operational for several years. Future pilot plant testing is planned for product and process improvement, alternate waste forms and combinations and innovative equipment testing.

Fig. 2. AZTECH Pilot plant system.

DEMONSTRATION PLANT DEVELOPMENT

The demonstration plant was designed, installed and operated primarily for the purpose of process and equipment evaluation on a full-scale (multiple 55-gal drum capacity) basis. This evaluation provided data and a basis for design of the (commercial) modular plant unit. In addition to the modular plant design support, testing was performed to provide samples from a full-scale solid for independent laboratory analysis of one waste product (Na_2SO_4). The demonstration plant is similar in design to the pilot plant but is scaled up to produce 55-gal quantities of waste product. Also, process controls are provided by a sophisticated process control system similar to the commercial modular plant unit control system. A photograph of the demonstration plant is shown in Fig. 3. Highlights of the demonstration testing program are as follows:

a. Suitability of the equipment to the process and operating parameters was evaluated during each test. In addition, tests were run to specifically evaluate certain pieces of equipment. It was observed during several runs that an emulsion of water and VT was delivered to the condensate receiver. The decanter size was increased and its location was changed to just downstream of the vacuum pump. These changes stabilized the system and VT losses to the condensate receiver were eliminated.

b. Process parameters were evaluated at full-scale during production runs. The effects of scaling up the process were determined, and operating temperature and pressure ranges have been adjusted as a result of this information.

c. Several process changes were made as a result of the information gained from the demonstration plant. Early sodium sulfate production runs produced large crystals and agglomerates. To eliminate these effects, the liquid feed was sprayed into an organic (VT) pool. This has eliminated the problem of large crystal formation and agglomeration. Also, the catalyst is no longer added to the polyester in the M-E. Instead, the catalyst and polyester-solid product are mixed in a static mixer outside the M-E. This eliminates the potential for premature polymerization of the polyester mix.

d. Design data and process flow rates for the modular unit were determined from production runs. Also, the VT-water stripper operation was evaluated.

e. The ease and effectiveness of cleaning the M-E was evaluated. The holdup in the vessel for successive runs and the amount of material washed out after a polyester addition without additional feed was determined from this information. The contaminants in the overhead vapor were also determined.

f. During the production runs the control system was evaluated. The goal was to determine the accuracy of the control instruments for optimum control. The ranges required in the specifications and for calibration were determined as well as the operability of selected instruments.

g. In conjunction with these tests an evaluation was made as to the magnitude and nature of required operator training. The length of training for operator effectiveness was also studied. To shorten the training period, as well as to portray process operation, the information from the control system was presented in a flow sheet format on the CRT.

Fig. 3. AZTECH demonstration plant system.

AZTECH PRODUCT QUALIFICATION

This section describes the AZTECH product qualification requirements for low level waste storage and presents test results to meet these requirements. The results discussed were obtained by processing non-radioactive waste solutions or slurries comparable to typical nuclear power plant low level wastes. Representative test samples were prepared from these actual AZTECH process/product test runs and were tested according to the NRC approved test plan for NRC approval.

Preliminary and supportive compressive, leachability, immersion and homogeneity testing was performed by a GE laboratory, independent of AZTECH processing. Also, absence of product "free liquid" documentation was prepared by GE for NRC approval. The AZTECH product qualification testing was performed by Brookhaven National Laboratory (BNL).

Experimentation was facilitated and personnel exposure to radiation was avoided by performing product qualification testing with simulated non-radioactive wastes. A very important aspect of this product qualification testing approach was the preparation of representative samples from product produced during actual process runs. This approach is considered superior to exclusive bench scale preparation of samples using non-processed materials because it provides more representative product and sample preparation. The specific waste forms tested for NRC approval consisted of the following:

a. Sodium Sulfate (Na_2SO_4)
b. Boric Acid (H_3BO_3)
c. Mixed-Bed Powdered Resin (Powdex) - anion (hydroxyl form) and cation (hydrogen form) typically loaded with 20 w/o iron oxide
d. Mixed-Bed Bead Resin - anion (hydroxyl form) and cation (hydrogen form) typically loaded with 20 w/o iron oxide
e. Decontamination Solution (CANDECON[a])

The decontamination solution is typical bead resin (maximum of 60 gms/liter CANDECON) in a mixture of a 6:1 ratio of anion to cation bead resin. Mixed-bed bead resin was also prepared and tested to simulate typical chemical resin depletion conditions expected at power plants. The bead resin was loaded

[a]London Nuclear Trademark

with 20 w/o Fe_2O_3 and approximately 25% ion loading (4 w/o of the dry bead resin weight) of Na_2SO_4. The resin also contained approximately 1.5 w/o total metal ion (cobalt, cesium, and strontium) loading.

These waste forms are normally received in the power plant radwaste storage systems as solutions or slurries and these forms were used for the AZTECH product qualification program. AZTECH processing removes the liquid and leaves the waste solids which are mixed with polyester to form a dry, dense plastic monolithic solid.

The regulation "Licensing Requirements for Land Disposal of Radioactive Waste." 10CFR61 requires Class B and C waste to be stabilized. As defined in Section 61.56(b) of the rule, stability requires that the waste form maintain its structural integrity under the expected disposal conditions. To ensure that Class B and C wastes or their containers will maintain stability, a technical position on waste forms has been developed by the NRC to provide guidance to waste generators on waste form test methods and results acceptable to the NRC staff for implementing the 10CFR61 waste form requirements. The objective of this program was to determine by sample chemical analysis that the AZTECH solidified wastes met the requirements of 10CFR61 and the recommendations of the "Final Waste Classification and Waste Form Technical Position Paper", dated May 11, 1983. General Electric and independent laboratory tests were conducted using waste form samples to demonstrate compliance with 10CFR61. The test methods used were in compliance with applicable ASTM/ANS Test procedures as set forth in the NRC Technical Position on Waste Forms (May 1983).

AZTECH product testing and sampling for product qualification was as follows:

a. Laboratory scale testing was performed to refine product and component parameters based on previous experience. For example, alternate catalyst/promoter/retarder testing was performed to establish optimum polymerization of the AZTECH product. General Electric analyzed samples with varying ratios of solids, catalyst/promoter/retarder and VT. Results of the sample analysis were used to provide guidance for pilot and demonstration scale test tuns.

b. Pilot scale testing with selected process parameters was performed to produce final samples for testing by BNL which has proven experience in performing product qualification testing to meet NRC 10CFR61 requirements.

c. Full-scale demonstration testing was performed to correlate pilot scale results. Samples were also tested by BNL.

Based on the extensive development work already completed, the acceptable range of wt% solids concentration over which a satisfactory waste product could be produced by the AZTECH process was known for each of the five waste forms selected for qualification testing.

This development testing also indicated that below any selected wt% solids value, the product integrity (e.g., leachability) improves or does not fall below NRC criteria. Qualification testing was performed over the range from essentially zero to the upper level of wt% solids for one waste form (Na_2SO_4) to demonstrate this fact. All other waste form samples were tested at the maximum concentration of solids in the current test run final products. The test plan waste forms tested and test runs are indicated in Table I.

Samples for analysis were nominally 1 inch in diameter by 2 inches long and were prepared by casting in plastic containers. However, free liquid and homogeneity determination used a large full-scale 55-gal drum casting, requiring "cut" samples. A minimum of two samples per analysis per waste form were taken. Three non-radioactive tracers (0.1 to 0.2% cobalt, cesium and strontium) were used for leachability analysis. These tracers were present in a solution added to each process feed batch of simulated waste. This waste was processed to dry solids, i.e., to the point where polyester would normally be added. A sample of these dry solids was removed for tracer contents analysis.

TABLE I

Test Plan

	Waste Form	Pilot Plant Runs	Demo Plant Runs
a.	Na_2SO_4	1-4[b]	1
b.	H_3BO_3	1[b]	--
c.	Powdered Resin (Typical Loading)	1	--
d.	Bead Resin (Typical Loading)	1	--
e.	Decon Solution	1	--
	Total Runs	5	1

Final sample testing (90 day tests) is not complete as of this date. However, test results to date indicate the AZTECH process produces very stable waste product. For example, the ranges of current test results on the five waste forms tested are as follows:

Test	Range of Test Data	NRC Acceptance Criteria
Compression (psi)	2900-7900	>50
Leachability (DI water)	8.7-15.8	L > 6
Leachability (Sea water)	8.6-11.2	L > 6
Thermal Cycling, Compression (psi)	4800-8700	>50
Irradiation, Compression (psi)	3800-8700	>50

Conclusions based on the data presented in this report are as follows:

1. The AZTECH process is capable of successfully solidifying all of the various low level radioactive wastes normally encountered at BWR and PWR power plants. The demonstration plant has demonstrated the capability to operate reliably and to produce consistent results.

[b]Product for test sampling was actually provided by the demo plant rather than the pilot plant. Sample preparation was accomplished by mixing catalyst into product from the process runs and casting the mixture in vials where polymerization occurred.

2. The AZTECH process can be used in any plant to supplement, replace or improve existing radwaste systems.

3. The final product in all instances is free of liquid and meets or exceeds existing regulations and guidelines established by the NRC, the U.S. Department of Transportation (DOT), and the states in which low-level radioactive wastes will be buried.

4. Based on the Product Qualification tests completed to date, the solidified product generated by the AZTECH system will satisfactorily withstand the effects of long-term exposure to the environmental conditions anticipated at any of the proposed burial locations.