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MIXED AND CHELATED WASTE TEST PROGRAMS WITH BITUMEN
SOLIDIFICATION

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ABSTRACT

Mixed and Chelated Waste Test Programs With Bitumen Solidification

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INTRODUCTION

This paper will present the results of bitumen solidification tests on mixed wastes and chelated wastes. The French Atomic Energy Commission (CEA) performed demonstration tests on radioactive wastes contaminated with chelating agents for Associated Technologies, Inc. (ATI). The chelated wastes were produced and concentrated by Commonwealth Edison Co. as a result of reactor decontamination at Dresden Nuclear Station, Unit 1.

Law Engineering in Charlotte, N. C. produced samples and performed tests on simulated heavy metal laden radioactive waste (mixed) to demonstrate the quality of the bituminous product. The simulation is intended to represent waste produced at Oak Ridge National Labs operated by Martin-Marietta.

MIXED WASTE TEST PROGRAM OBJECTIVES

ATI wants to demonstrate that the bituminization of a mixed waste stream representative of waste generated at the Oak Ridge National Lab produces a product that satisfies hazardous waste regulations as promulgated by EPA.

The objective of the program is also to perform testing to demonstrate that the solidified waste test specimens meet the NRC guidelines for stability for low-level radioactive wastes and do not exhibit any of the characteristics of a RCRA hazardous waste.

CHELATE TEST PLAN OBJECTIVES

ATI wants to demonstrate that a bituminous product containing chelating agents can be produced to meet the stability requirements as set forth in 10CFR61 and the accompanying waste form Branch Technical Position (BTP). To demonstrate this ATI first needed to produce samples that were certain to pass the battery of tests required to prove stability. SQN, ATI's licensor for bitumenization, and the OEA first demonstrated that a product could be produced that was homogeneous with no free water. To do this the OEA developed a chemical pretreatment plan that would eliminate product gassing.

Bitumen's most vulnerable characteristic for meeting waste form stability requirements is its mechanical strength. Once the pretreatment technology was established samples were produced using oxidized bitumens of varying viscosities and solid contents. In bitumen, the higher the solids content in the product the higher the mechanical strength. Also, in bitumen, the greater the bitumen viscosity the higher the mechanical strength. The optimum combination of bitumen viscosity and solids loadings was established and many samples were produced.

The samples were sent to various labs for waste form testing. The irradiation tests and biodegradation tests were initiated first. Later the freeze-thaw and leaching tests were begun.

DESCRIPTION OF MIXED WASTE

The test program involves preparation and evaluation of solidified specimens for two liquid waste streams: 1) Wastewater Treatment (Biodenitrification) Sludge; and 2) Wastewater Treatment (Metal Plating) Sludge.

1. Wastewater Treatment (Biodenitrification) Sludge

This waste stream is approximately 40 to 45 wt.% solids which consists primarily of calcium carbonate. Prior to biodenitrification, the liquid waste is high in nitrates at a concentration of 30,000 to 40,000 ppm. Calcium acetate is added to the waste as a source of carbon for the denitrifying bacteria. The carbon is oxidized to carbonate while the nitrate is reduced to nitrogen gas. A large variety of metal salts at relatively low levels are present as well as about 1000 ppm of uranium in the sludge. Approximately 5% of the sludge mass comes from diatomaceous earth used in a filtration operation to concentrate the solids. The sludge and supernate contain primarily alpha emitters.

Phenolics are present in the sludge in the range of 0.03 wt.% . Soaps, detergents, and many other solvents are present at relatively low concentrations. It was not practical to identify all of these compounds for inclusion in the surrogate formula for the test program.

It is estimated that 300,000 gallons of sludge at between 40 to 45 wt.% solids will be generated each year.

2. Wastewater Treatment (Metal Plating) Sludge

This sludge is approximately 50 wt.% total solids and contains as much as 5% aluminum, 20% calcium, 4% phosphorus, and many metal hydroxides. Complex phenolics are present from the degradation and hydrolysis of hydraulic fluids, oils, and greases. The viscosity of the sludge is about 700,000 centipoise at 25°C. The activity of the sludge is mostly from alpha emitters.

The waste comes from metal plating operations, laboratory sink drains, metal preparation cleanup operations, decontamination processes and mop waters. The liquid wastes contain nitrate, cyanide, nickel, cadmium. The waste is treated to precipitate solids and centrifuged to 50 wt.% solids. The sludge is classified as a RCRA sludge because of the electroplating operations.

The current volume of this sludge is approximately 35,000 gallons and is being generated at a rate of about 50 gallons per day.

DESCRIPTION of CHELATED WASTES

Dow Chemical licensed IT Corp. to use a proprietary solvent for removing radioactive scale build-up on metal surfaces. That solvent is called NS-1 and contains chelating agents. Commonwealth Edison Co. awarded the unit 1 reactor decontamination contract to IT Corp. The contract was successfully completed leaving behind a large volume of NS-1 contaminated waste water. The waste was subsequently volume reduced through evaporation resulting in 11,000 gallons of concentrated NS-1 waste and 600 ft³ of slightly contaminated resins. The TRU content in concentrated NS-1 waste drove the waste into a Class C category.

TEST PROGRAM FOR MIXED WASTE

Surrogate wastes representative of waste streams on the Oak Ridge Reservation were prepared and solidified to demonstrate ATI technology in immobilization of low-level radioactive wastes. The ATI volume reduction/solidification process involves waste pretreatment, evaporation of water, and solidification of remaining solids in bitumen.

The ATI production scale process is a continuous process which utilizes a LONA Thin-Film Evaporator for evaporation of water and simultaneous mixing of the solids and bitumen. The bench scale evaluation described herein utilized laboratory scale equipment to prepare bituminized waste product samples which were representative of the end product which would be produced by the ATI process.

Surrogate wastes were prepared in accordance with the procedures provided by Martin-Marietta Energy Systems , Inc. Waste pretreatment was performed as appropriate in order to enhance processing performances and optimize product quality.

Water was evaporated from the waste by application of heat to the vessel containing the waste. This proceeded until a wet sludge "cake" was obtained. The wet cake was then dried in an oven until no more than trace amounts of water remained.

Bitumen was melted and then heated to a specified process temperature in a mixing vessel. The heat input was controlled to maintain the temperature within process specifications.

The dried waste solids were then added and mixed into the bitumen at a rate sufficiently low so as to maintain acceptable constant temperature levels. Mixing continued for a minimum, predetermined time after the dried solids addition to ensure a homogeneous end product. The solids to bitumen weight ratio was one to one.

The molten and continuously mixed bituminized product was poured into molds and allowed to cool to produce specimens required for testing by Martin-Marietta Energy Systems, Inc. and ATI. Test specimens and blanks required for Waste Form Tests by Martin-Marietta were delivered to Oak Ridge, Tennessee. Waste Form Tests which were the responsibility of ATI were performed, and test results were provided to Martin-Marietta in the Test Report.

TEST PROGRAM FOR CHELATED WASTE

Inactive, simulated waste was prepared, utilizing a proprietary recipe provided by International Technology Corporation, the manufacturer of the NS-1 solvent. An attempt was made to bituminize the waste in a pilot-scale LUWA thin-film evaporator with no pretreatment, but the product exhibited a high degree of off-gassing, which yielded a porous product when cool. A proprietary pretreatment, developed by the French CEA, was added to the simulated waste, and the pretreated waste was processed in the LUWA evaporator. The product no longer exhibited off-gassing when pretreated properly.

Because the actual chelated waste is a Class C waste, the bitumenized product is required to meet the stability requirements of 10CFR61 and the associated Technical Position on Waste Form. These stability requirements include a minimum compressive strength of 60 psi, when measured in accordance with ASTM D1074.

In order to meet this compressive strength requirement, specimens of bitumenized waste were prepared in a bench-scale evaporator/mixer, utilizing simulated waste and a number of asphalt types, including roofing asphalts Type I, Type II, and Type III, as defined in ASTM D312. In addition, some specimens were made utilizing a small addition of diatomaceous earth, which increases the strength of the matrix.

Based upon the results of the compressive strength testing of the various specimens, the most appropriate combination of asphalt and additives was selected for the full range of stability tests, including leach, immersion, irradiation, biodegradation and thermal degradation testing. In addition, a 55-gallon specimen was produced by processing the simulated waste with a pilot-scale thin-film evaporator. This 55-gallon specimen was tested for free liquid content in accordance with ANS 55.1, and core specimens were tested for compressive strength to verify the homogeneity of the 55-gallon specimen.

TEST RESULTS

Most of the test results will be available by April 1988. By the time this paper is presented, all results will be available.