INNOVATIVE TECHNOLOGY
Summary Report

DOE/EM-0480

Mixed Waste Encapsulation in Polyester Resins

Treatment for Mixed Wastes Containing Salts

Mixed Waste Focus Area

Prepared for
U.S. Department of Energy
Office of Environmental Management
Office of Science and Technology

September 1999

INNOVATIVE
TECHNOLOGY
Summary Report

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Treatment for Mixed Wastes Containing Salts

OST Reference #1685

Mixed Waste Focus Area

DEPARTMENT OF ENERGY · UNITED STATES OF AMERICA

Demonstrated at
Hanford Nuclear Site
through SGN Eurisys Services Corporation
Richland, WA

INNOVATIVE TECHNOLOGY
Summary Report

Purpose of this document

Innovative Technology Summary Reports are designed to provide potential users with the information they need to quickly determine if a technology would apply to a particular environmental management problem. They are also designed for readers who may recommend that a technology be considered by prospective users.

Each report describes a technology, system, or process that has been developed and tested with funding from DOE's Office of Science and Technology (OST). A report presents the full range of problems that a technology, system, or process will address and its advantages to the DOE cleanup in terms of system performance, cost, and cleanup effectiveness. Most reports include comparisons to baseline technologies as well as other competing technologies. Information about commercial availability and technology readiness for implementation is also included. Innovative Technology Summary Reports are intended to provide summary information. References for more detailed information are provided in an appendix.

Efforts have been made to provide key data describing the performance, cost, and regulatory acceptance of the technology. If this information was not available at the time of publication, the omission is noted.

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TABLE OF CONTENTS

APPENDICES

SECTION 1

SUMMARY

Technology Summary

Throughout the Department of Energy (DOE) complex there are large inventories of homogeneous solid mixed wastes, such as treatment residues, fly ashes, and sludges that contain relatively high concentrations (greater than 15% by weight) of salts. The inherent solubility of nitrate, sulfate, and chloride salts makes traditional cement stabilization of these waste streams difficult, expensive, and challenging. Salts can effect the setting rate of cements and can react with cement hydration products to form expansive and cement damaging compounds. Many of these salt wastes are in a dry granular form and are the by-product of treating spent acidic and metal solutions used to recover and reformulate nuclear weapons materials over the past 50 years. At the Idaho National Engineering and Environmental Laboratory (INEEL) alone, there is approximately 8,000 cubic meters of nitrate salts (potassium and sodium nitrate) stored aboveground with an earthen cover. Current estimates indicate that over 200 million kg of contaminated salt wastes exist at various DOE sites. Continued primary treatment of wastewater coupled with the use of mixed waste incinerators may generate an additional 5 million-kg of salt-containing, mixed waste residues each year.

One of the obvious treatment solutions for these salt-containing wastes is to immobilize the hazardous components to meet Environmental Protection Agency/Resource Conservation and Recovery Act (EPA/RCRA) Land Disposal Restrictions (LDR), thus rendering the mixed waste to a radioactive waste only classification. One proposed solution is to use thermal treatment via vitrification to immobilize the hazardous component and thereby substantially reduce the volume, as well as provide exceptional durability. However, these melter systems involve expensive capital apparatus with complicated offgas systems. In addition, the vitrification of high salt waste may cause foaming and usually requires extensive development to specify glass formulation recipes. As an alternative to thermal treatments, low-temperature stabilization of these materials in basic cementitious grouts has also been widely employed. However, salts interfere with the basic hydration reactions of Portland cement, leading to an inadequate set or deterioration of the waste form over time. Sufficient and compliant stabilization in cement can be achieved by lowering waste loadings, but this involves a large and costly increase in the volume of material requiring handling, transporting, and disposal.

As a consequence of these stabilization deficiencies associated with salt containing mixed wastes, the Mixed Waste Focus Area (MWFA), a DOE Environmental Management (EM)-50 program, sponsored the development of five low-temperature stabilization methods as an alternative to cement grouting. One of these developed alternatives is microencapsulation by polyester resins. Polyester resins, cross-linked, with styrene have been employed as a waste solidification method for over 20 years in Japan, Europe, and the United States. The process has routinely been applied to low-level waste (LLW) streams generated by the nuclear power industry. However, very little waste form performance data for applications to mixed waste are available; especially those waste streams containing relatively large concentrations of RCRA heavy metals and troublesome salt components. Based on the results of surrogate testing and treatability studies provided in this ITSR, polyester resins can be an effective, economical, and simple encapsulation process for salt-containing mixed wastes. In some applications, they provide higher waste loadings and a more durable waste form than the baseline method of cement grouting. However, the regulatory and/or the chosen disposal site’s acceptance of the method used to prepare waste form specimens for hazardous metal leachability determinations can influence the selection of polyester as a waste form. The high flammability of the styrene cross-linking agent is also a safety concern that must be considered for any large-scale deployments of the polyester microencapsulation process.

Demonstration Summary

Under MWFA sponsorship, investigators employed by the COGEMA Engineering Corporation at the DOE Hanford site performed a series of development tests and treatability studies involving polyester microencapsulation. Specifically, the studies involved the encapsulation of both surrogate and actual salt-containing mixed wastes with the following resins: Orthophthalic polyester, Isophthalic polyester, vinyl ester, and a water-extendible polyester (W.E.P.).

Surrogate waste tested included nonradioactive simulants containing nitrate or chloride/sulfate salts, as well as RCRA hazardous metals at nominal concentrations of 1,000 ppm. The surrogates represented typical dry salt, mixed waste streams currently residing in the DOE complex, including that of unconcentrated spent offgas scrub solutions (blowdown) from a mixed waste incinerator. These surrogate tests were performed over a range of conditions and with waste loadings up to 50%. Polyester waste forms containing up to 70-wt % of only the nitrate or chloride salts of sodium were also evaluated. These additional surrogate tests were performed in an attempt to identify the salt loading limit of the polyesters. Polyester waste forms using an actual mixed waste stream were also prepared and evaluated. This treatability study consisted of microencapsulating a wet, low-level radioactive, calcium sulfate brine stream with only the W.E.P. Surrogates of the actual waste stream were stabilized before the treatability study to estimate acceptable waste loading limits, emulsion mixing times, and solution pH conditions. This particular mixed waste stream is an evaporator residue generated from the treatment of various wastewaters at Hanford's Effluent Treatment Facility (ETF). In the near future, the ETF anticipates treating wastewaters that will generate RCRA defined hazardous waste requiring stabilization before land disposal.

All of the resins tested have thermosetting properties and are cross-linked with styrene during the waste encapsulation process. The cross-linking polymerization process is initiated by adding a catalyst to the liquid polyester –styrene mix, followed by immediate addition of the waste. The blending process can take place in any type of mixing vessel where, if adequate mixing is achieved, the cross-linking resin coats each waste particle (i.e., microencapsulates) and begins to cure. Before hardening, the waste form is poured into a mold, capped, and placed in a curing chamber. Data collected during the formation of each different waste form consisted of both mixing effectiveness and temperature increase. The performance of cured waste forms was then evaluated against both established RCRA and Nuclear Regulatory Commission (NRC) disposal criteria. Specific tests included percent free liquid determination, leachability, and compressive strength. Surface morphological observations were also made, along with determinations of waste form density, waste loading, and volume reduction.

Some of the waste forms prepared and tested met or exceeded the requirements established by the RCRA and NRC criteria that were imposed on the investigators. Included among the successes were polyester waste forms containing a 50 wt% loading of surrogate salt waste consisting of 60% nitrate salts, as well as waste forms containing over 60 wt% of an actual mixed waste from Hanford's ETF. Measured compressive strengths were up to 100 fold greater than that recommended by the NRC (i.e., 60 psi), and leaching indices for nonhazardous species were above the criteria (i.e., 6), including those calculated for the salt anions. The retention of compression strength after water immersion was also excellent. Leachability of the RCRA hazardous components from the polyester waste form was determined using EPA's toxicity characteristic leaching procedure (TCLP). Of the waste forms specially prepared for leach testing, all met the LDR levels that were required before the promulgation of the newer and more restrictive Universal Treatment Standards (UTS).

Contacts

Technical

Principal Investigator
Rabindra K. Biyani
COGEMA Engineering Corporation
An SGN/Cogema, Inc. Company
P.O. Box 840
Richland, WA 99352
Mail Stop-H3-26
509-376-1004
E-MAIL: biyani@COGEMA-Engineering.com

Management

DOE-ID Program Director
William Owca
Mixed Waste Focus Area
U.S. Department of Energy
Idaho Operations Office
785 DOE Place
Idaho Falls, Idaho 83401-1563
(208)-526-1983
FAX: (208)-526-5964
E-MAIL: owcawa@inel.gov

MWFA Product Line Manager
Vince Maio, Advisory Engineer
Mixed Waste Focus Area
Lockheed Martin Idaho Technologies Company/LMITCO
Idaho National Engineering and Environmental Laboratory/INEEL
P.O. Box 1625
Idaho Falls, ID 83415-3875
(208) 526-3696
FAX: (208) 526-1061
EMAIL: vmaio@inel.gov

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SECTION 2

TECHNOLOGY DESCRIPTION

Overall Process Definition

For over 20 years, various organic polymers have been used to encapsulate waste materials since their superior mechanical properties, such as compressive strength, usually allow for higher waste loadings than that achievable with cements. Polymers in general also have moderate to excellent resistance to the acids, bases, and organics present in most waste streams. They are normally formed through either chain or condensation type polymerization reactions involving one type or several types of monomers. In most waste solidification operations, the waste media is mixed with a melted preformed polymer or gets microencapsulated in the waste during a controlled polymerization reaction. The inherent resistance of organic polymers to water favors the low leachability potential of a final waste form, but also presents difficulties in encapsulating wastes with high water contents. However, the development and use of water-extendible polymers and emulsifiers can adequately encapsulate some aqueous wastes.

For mixed waste solidification applications, polymers can broadly be classified as either having thermoplastic or thermosetting properties. Thermoplastics, such as low and high-density polyethylene, are noncross-linked linear polymers that melt and become flowable at a specific transition temperature. They can be reworked and returned to their original form upon cooling. They require no reaction to solidify, and the waste material does not interact with the polymer chemistry. Basic microencapsulation operations involve simply mixing the waste with the polymer in the melting phase of an extruder, and pouring the homogenous polymer- waste mix in a disposal drum for curing, cooling, and hardening. Thermosetting polymers, on the other hand, cannot be reworked, reformed or remelted, since they decompose upon over heating. They are usually cross-linked and rely on polymerization reactions to solidify. Unlike thermoplastic resins, the waste can react and interfere with the thermosetting resin reactions. Polyester resins, as well as epoxys, are classified as thermosetting.

A simple ester, R’C (O) OR, is usually formed via a condensation reaction between an organic alcohol, R’OH, and an organic acid, RC (O) OH. During the reaction, water is given off - hence the term “condensation.” The symbols R and R’ can represent any unsaturated or saturated aliphatic or cyclic hydrocarbon group, C_XH_Y. To form a chain-like or “poly” ester compound, diols (i.e., dialcohols and glycols), HO-R’-OH, and diacids (i.e., anhydrides), HO (O) C-R-C (O) OH, are frequently used as starting monomers to provide condensation- type polymerization reactions on both sides of the monomer compound.

For three of the polyesters studied by COGEMA in the development effort documented in this ITSR, R and R’ either represent hydrocarbon cyclic/ring (phthalic) compounds, - C_6H_4 -, or the double bond vinyl group, -HC=CH-. Upon completion of the condensation polymerization reactions, each phthalic or vinyl compound is attached to two ester groups for each link in the polymer chain. The position of the ester group on the cyclic compound further classifies two of the polymers studied. Orthophthalic polymers have ester groups attached to the ring adjacent to each other, whereas iso (or meta) phthalic polymers have an unsubstituted ring carbon between the two attached ester groups.

The fourth polymer studied by COGEMA involved a specific water-extendible polyester (W.E.P.). Details of its specific makeup are proprietary, but it allows aqueous waste to be stabilized and solidified from an emulsion mixed at very high shear rates. To the contrary, the other three polymers can only be used for wastes that are sufficiently dried. All four polymers were provided, in a preformed condition, by the Ashland Chemical Co. (Columbus Ohio) under the trade names or codes listed below.

POLYMER	TRADE NAME/CODE
Orthophthalic	S2293
Isophthalic	Aropol ^{TM} 7334
Vinyl Ester	Hetron^{R} 922-L25
Water Extendible	Aropol^{TM} WEP 662P

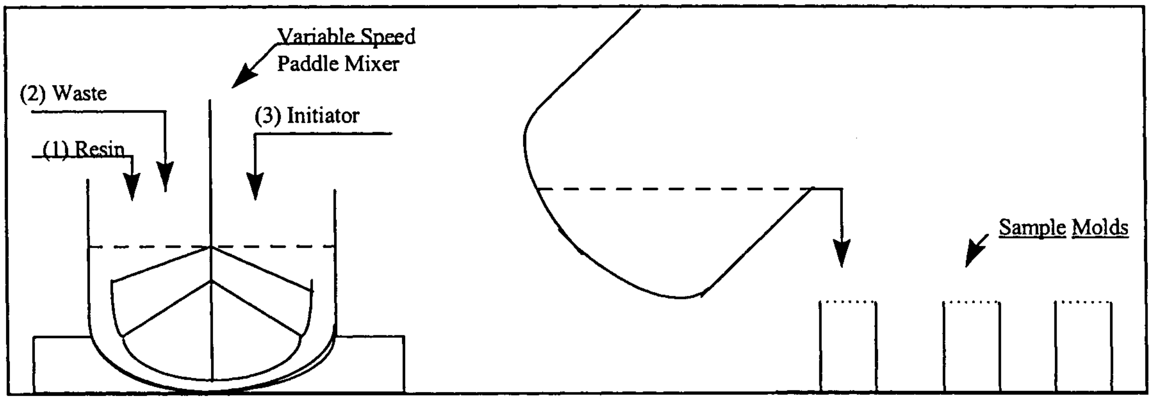
A common practice is to use basic polyester chains that are preformed by the above process before the waste solidification step. The polymeric thermosetting reaction that occurs during waste encapsulation is one of cross-linking the basic preformed polyester chains. It is this cross-linking that provides the enhanced microencapsulation of the waste, the resistance to chemical and organic solutions, and the superior structural strength of the waste form. Cross-linking is initiated using another polymer and another type of polymerization reaction. For the polyester resins evaluated in this development effort, styrene, because of its high radiation resistance, was used as a cross-linking polymer for all four of the polyesters tested. Unlike condensation polymerization, cross-linking of the polyester with styrene was achieved via a reaction referred to as *free radical polymerization*, since it involves the breaking of double bonds and the formation of oxidized and reactive carbon species. These species react in a chain like manner to form single bonds that connect the various polyester chains to the styrene chains, which in turn link to different polyester chains.

Three-dimensional cross-linking via free radical polymerization requires an initiator (catalyst) to generate the free radicals that break the initial double bonds needed to set off the chain reactions described above. Initiators are usually relatively unstable compounds that form free radicals upon decomposition. Examples include peroxides and other substances containing double bonded oxygens [e.g., R-C (O)-O-O- (O) C-R]. The catalyst used for all the polyesters studied in this development effort was methyl ethyl ketone peroxide (MEKP) and its selected amount affects the gel and cure time of the waste form. Furthermore, a promoter is routinely used to accelerate the effects of the catalyst and facilitate curing at room temperature. A promoter basically induces the decomposition of the initiator by breaking its O-O bond. The use of a compatible initiator-promoter combination is critical in ensuring a reasonable working time before the polyester-waste mixture sets. All of the polyesters evaluated by COGEMA were received from Ashland prepromoted with cobalt naphthenate. In addition to being prepromoted, each of the polyesters provided by Ashland were in a liquid state dissolved in the styrene monomer. The styrene monomer making up 40 to 60% of the received solution. Therefore, addition of the catalyst is all that is required to initiate the gelling of the polyester-styrene solution after the waste is added. To avoid any unwanted cross-linking of the polyester in shipment or storage (before catalyst addition and waste encapsulation), Ashland ships the resins with an inhibitor. The inhibitor employed was of hydroquinone, which acts as a free-radical scavenger during transportation.

System Operation

As depicted in Figure 1, the basic flowsheet needed to fabricate polyester mixed waste forms consists of a simple mixer, as well as containers for curing the specimens. A 55-gallon capacity planetary type mixer is more than likely the preferred method for larger operational scale systems, but any vessel containing at least a variable speed impeller-type mixer will suffice. Industrial scale systems currently in use for the polyester microencapsulation of nuclear power industry wastes vary. Some consist of blend tanks, feed hoppers, premixers, and drum conveyor components. In these systems, the waste polymer blend is transported from the mixer to the disposal drum before hardening. Other arrangements consist of emulsion tanks and planetary mixers. For these systems the drum serves as both the mixing vessel and the final disposal container.

Figure 1. Laboratory Polyester Microencapsulation Flow Diagram



Nominal operations for dry mixed wastes consist of adding predetermined amounts of the initiator to one of three of the Ashland polyester-styrene blends (i.e., orthophthalic-, isophthalic-, or vinyl ester) previously batched to the mixing vessel. Free-flowing solid powder waste is then added in amounts to achieve the desired waste loading, while intimate mixing is initiated. However, for aqueous wastes that require the W.E.P. resins, the waste is slurried to the mixer as a steady stream into a prebatched amount of resin. This blend is then mixed at a low rate for 5 to 10 minutes until a homogeneous emulsion is formed. The desired amount of initiator is then added and mixing continues for another 2 to 5 minutes at a high rate until a temperature rise signals the onset of curing. Before setting, the still viscous waste forms were then transferred to molds for complete hardening.

For testing purposes, the curing molds were placed in adiabatic chambers. Use of the chamber allows for simulating larger scale operations, where the dissipation of heat from the polymerization reaction is considerably less than that of the smaller test specimen waste forms. This procedure was necessary to determine the maximum temperature rise that could be expected from the exothermic polymerization and cross-linking reactions occurring under operational conditions.

SECTION 3

PERFORMANCE

Demonstration Plan

The polyester microencapsulation process was developed using both surrogate and actual salt-containing, mixed waste streams. In all, three surrogate tests were completed as well as one treatability study involving an actual Hanford waste stream. Two of the three surrogate waste formulations are detailed in Table 1 and were provided by the MWFA. One of the surrogates waste streams contained a high quantity of nitrate salts and represented a waste stream that had previously been unsuccessfully stabilized with simple Portland cement. Such waste streams are not uncommon in the DOE complex. The other surrogate waste stream contained salts of chloride and sulfate at levels representative of a dried, but not concentrated, spent incinerator offgas scrub solution (i.e., blowdown). Both of the surrogates were free of moisture, contained oxide forms of RCRA heavy metals in the 1,000-ppm range, and contained trichloroethylene as a trace organic contaminant. To obtain comparable waste form performance data, the same surrogate formulations were provided to other investigators involved in parallel efforts to also develop salt waste stabilization alternatives. The two MWFA salt waste surrogates were employed in evaluating the performance of only the vinyl ester, orthophthalic, and isophthalic resins. A third surrogate, representing the actual mixed waste stream to be studied later, was used to evaluate the performance of the W.E.P. resin. The four polyester resins' properties, as well as costs, are provided in Table 2. Table 2 also summarizes some of the resins' test performance results (i.e., highest temperature achieved, time to peak temperature, and compressive strength) and conditions (initiator amounts).

For the surrogate testing involving the MWFA formulations and the three resins, a factorial experimental design was used to generate forms over a range of waste loadings and initiator (catalyst) amounts. As a result, 16 waste forms were prepared for performance testing. Eight of these waste forms were prepared with the nitrate salt- containing surrogate and the remaining eight with the chloride/sulfate surrogate. Waste loadings for each surrogate type included 40, 50, and 60-wt %. MEKP initiator amounts, as a weight percentage of the resin mass, were varied over the following six levels: 1.0, 1.25, 1.75, 2.25, 2.5, and 3.0%. Surrogate testing involving the W.E.P. resins only involved the preparation of two waste forms for performance testing. Both of the waste forms were prepared using 1.25 wt-% initiator (as measured as a percentage of the resin mass), but were varied in surrogate waste loadings (i.e., 60 and 70-wt%). All waste form preparations were initiated at ambient conditions.

Table 2. Physical data for the resins and average test results and conditions by resin type.

	Vinyl Ester	Orthophthalic	Isophthalic	W.E.P.
Resin I.D.	922L-25	2293-T15	7334-T30	WEP-662
Resin Viscosity, centipoise (cp)	275	425	450	130
Resin Specific Gravity	1.03	1.07	1.09	1.03
wt% Styrene in Resin	48	45	46	60
Cost,$/lb	1.62	0.72	1.07	1.42
Avg. T_{max}, ˚C	112.5	112.0	127.0	80.6
Avg. Time to Peak Temp (hr)	2.3	2.2	2.4	1.78
Avg. C.S. (psi)	6,120	4,893	5,217	2,441
wt% Initiator for NO_3 /Cl wastes	2.5 and 3	1 and 1.25	1.75 and 2.25	1.25

Table 1. Composition of mixed waste salt surrogates

Constituent	ETF Liquid (wt%)	High-Chloride (wt%)	High-Nitrate (wt%)
Fe_2O_3	0.0	12.1	5.6
$Al(OH)_3$	0.0	8.0	3.9
$Mg(OH)_2$	0.0	8.0	3.9
Na_3PO_4	0.0	3.9	2.0
$MgSO_4$	1.2	0.0	0.0
MicroCel E	0.0	16.0	7.8
Portland cement (Type I-II)	0.0	4.1	2.0
H_2O	75.2	32.6	15.4
SiO_2	0.7	0.0	0.0
Na_2SO_4	12.7	0.0	0.0
$NaNO_3$	5.9	0.0	58.4
$CaSO_4$	3.8	4.3	0.0
NaCl	0.5	9.5	0.0
Hazardous Components[1]	**mg/kg**	**mg/kg**	**mg/kg**
PbO	0.0	956	992
CrO_3	0.0	949	974
HgO	0.0	955	982
CdO	0.0	959	997
NiO	0.0	959	981
Trichloroethylene (TCE)	0.0	948	976
Radionuclide Surrogates[1]	**mg/kg**	**mg/kg**	**mg/kg**
$Sr(NO_3)_2$	0.0	953	982
$CsNO_3$	0.0	947	979
$Co(NO_3)_2$	0.0	948	980
Total wt%:	100.0	100.0	100.0

Notes: MicroCel E is a registered trademark of the Celite Corporation, Lompoc, CA

1. The radiological surrogates and hazardous components make up ~ 1 wt % of the nitrate and chloride surrogates.

An actual mixed waste was encapsulated using only the W.E.P. resin. The waste chosen for this treatability study consisted of unevaporated salt residues from the treatment of Hanford contaminated ground water in their ETF complex. The ETF is a centralized wastewater treatment complex consisting of filters, ion exchange columns, organic oxidation units, and dryers for conditioning a variety of Hanford's current and projected mixed wastewaters. Presently, the residues are nonhazardous and are usually dried to a concentrated salt before disposal as a LLW only. However, future operations will generate mixed waste residues that will require further treatment before land disposal, and a process to encapsulate these wet residues may eliminate use of the dryer. The salt slurry residues chosen for the treatability study contained both dissolved and suspended solids of sodium nitrate and calcium sulfate respectively at a total salt concentration of ~25 %. The waste was also spiked with solutions of RCRA heavy metals and radionuclides to test the durability of the waste form and improve the detectability of any contaminants during later waste form leach testing. Final waste heavy metal and radionuclide concentrations are provided in Tables 3 and 4, respectively.

Based on previous ETF surrogate waste studies and other trial runs, the treatability study was carried out by preparing a stable W.E.P. emulsion at a pH of 9 and a waste loading of 62 wt.%. A MEKP initiator/catalyst amount of 1.25-wt%, measured as a percent of the resin mass, was used.

Sufficient polyester waste form specimens, for each surrogate and treatability trial run, were prepared to ensure that the following waste form performance tests could be completed:

- free liquids evaluation,
- compressive strength via ASTM C-39,
- maximum temperature (exotherm) achieved during polymerization and curing,
- leachability of RCRA hazardous heavy metals via the TCLP test,
- leachability (over time) of radionuclides, salt anions, and other species via ANSI 16.1,
- waste density, volume expansion factors, and waste loading.

Many of the above waste form performance criteria tests and evaluations are based on established EPA (RCRA) and NRC tests required for the land disposal of both low-level and mixed wastes. They were imposed on all investigators developing low-temperature, stabilized waste forms for salt-containing mixed waste. Use of identical surrogate and performance testing allows the enduser to compare and select among alternative stabilization and microencapsulation methods.

Table 3. RCRA metal levels in ETF treatability study waste.

Contaminant		As	Ba	Cd	Cr	Pb	Se
As received waste	mg/L	<0.07	0.79	0.40	1.98	<0.02	<0.03
Spiked waste	mg/L	159.3	154.1	119	151.3	132.7	140.9

Table 4. Radionuclide levels in ETF treatability study waste.

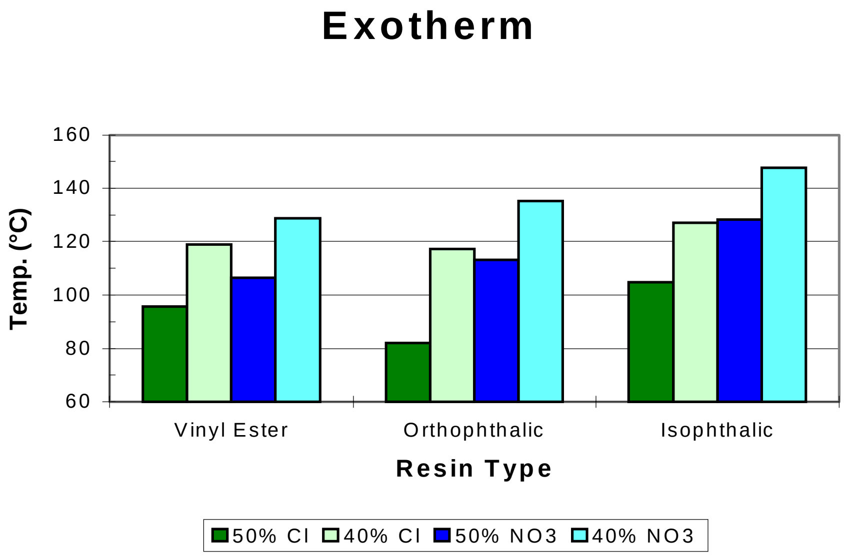
Contaminant		^{137}Cs	^{60}C0	^{99}Tc	^{90}Sr
As received waste	pCi/L	326	21	6.4E+04	340
Spiked solution	pCi/L	2.4E+06	2.7E+06	2.2E+06	2.3E+06
Spiked waste	pCi/L	1.2E+05	1.1E+05	1.3E+05	1.1E+05

Results

All of the polyester surrogate and treatability waste forms tested contained less than 5 vol% liquids as recommended by the NRC criteria, and greatly exceeded the 60 psi compressive strength criteria (refer back to Table 2). The retention of this compression strength after 90 days of water immersion was also superb, ranging from 74 to 93% of the original value.

Figure 2 provides the maximum temperatures (exotherms) achieved in the surrogate waste forms as a result of the polymerization cross-linking reactions occurring during the microencapsulation and subsequent curing steps. The figure graphically presents the maximum temperature for three of the resins tested, under varying conditions of waste surrogate type and waste loading. For comparison, the maximum temperature achieved during the treatability study to encapsulate actual waste in the W.E.P. resin was ~ 95 degrees C. The time to reach the maximum temperature during curing averaged just under 2 hours.

Figure 2. Maximum temperatures achieved during MWFA surrogate waste polyester microencapsulation



Leachability of the RCRA hazardous heavy metals in the polyester forms containing the surrogate wastes were determined using EPA’s toxicity characterization leaching procedure (TCLP). Since this single test validates the waste form’s exit from RCRA hazardous status, it is usually of greatest interest to end users and waste managers. All existing LLW disposal sites require that waste forms demonstrate leach levels below the established TCLP limits before land disposal. Table 5 provides the results of these leach tests for selected trial run specimens, and also provides the limits that were acceptable for land disposal at the time of the testing. Table 6 provides the conditions of the trial run number listed in Table 5.

As indicated in Table 5, all specimens for test runs 1 through 6 passed (i.e., were below the allowable) or were below detectectable limits for all of the RCRA metals, except that of cadmium. Failure is more than likely attributed to how the TCLP specimen sample was prepared from the waste form. The procedure requires that the specimen be sized to at least a 9-mm size fraction. If grinding or cutting achieves this size, the polyester coating on the surface of the waste form is probably destroyed and the hazardous metal contaminants are highly exposed for leaching. To counter this effect and eliminate grinding and cutting, fully coated polyester waste form molds of less than 9 mm were specifically prepared for TCLP testing. This was the case for Specimen 1A, which passed the TCLP leachability for all tested metals, including cadmium.

Recently the leach limits of RCRA hazardous metals allowable for land disposal have been lowered to the more stringent UTS. Comparison of UTS levels to the polyester (surrogate) waste form leach performances of Table 5, show failures for many of the test specimens-- including that of chromium for Run 1A.

Leaching as a function of time was also evaluated using a modified version of the ANSI 16.I test. This leach test is not required by RCRA for land disposal, but is recommended by the NRC. The test determines a leachability index for any chemical or radiochemical anion or cation in the waste form, and is an excellent indication of the waste form's ability to stay intact during its disposal life. Technically, the determined index is the negative base 10 log of the particular leaching species' diffusion coefficient. Thus the higher the value the better. Values of 6 or more are desired. For the polyester (MWFA salt surrogate) forms, indices for 17 different species (including those for Hg, Cd, Cr, Pd and the salt anions Cl, NO, SO_4) were determined. Values ranged form 6.3 to 12.6.

Table 5. TCLP leach results for polyester forms of MWFA salt surrogates.

	Cd	**Cr(+6)**	**Pb**	**Hg**	**TCE**
Max. allowable concentration	1.0	5.0	5.0	0.2	0.5
Run #	mg/L	mg/L	mg/L	mg/L	mg/L
1	1.29	1.35	0.147	0.1	<0.005
2	1.36	2.20	<0.03	0.1	<0.005
3	1.30	2.85	0.03	0.2	<0.005
4	1.61	3.05	0.22	<0.01	<0.005
5	2.34	2.95	0.39	<0.01	<0.005
6	2.34	3.00	0.34	<0.01	<0.005
1A	0.53	2.25	.15	N/R	N/R

Table 6. Conditions of test run specimens of Table 5

Run #	Resin Type	Initiator Amount (wt% of resin amount)	MWFA Surrogate Waste Type	Waste Loading (wt%)
1	orthophthalic	1.25	Chloride	50
2	isophthalic	2.25	Chloride	50
3	vinyl ester	3.0	Chloride	50
4	orthophthalic	1.0	Nitrate	50
5	isophthalic	1.75	Nitrate	50
6	vinyl ester	2.5	Nitrate	50
1A	orthophthalic	1.25	Chloride	50

Waste form testing with the MWFA salt surrogates validated polyester microencapsulation for salt loadings up to only 30-wt%. As such, surrogate type waste form testing was extended in an attempt to find the salt loading limit of polyester. Five additional tests were performed using pure sodium chloride or sodium nitrate as a waste. Only the orthophthalic resin was employed and polyester forms with pure salt waste loadings of 30, 50, and 70% were prepared. As indicated by the exotherm data of Table 7, these polyester forms properly cured without any visible physical defects. However, further leach testing with surrogates containing hazardous RCRA metals is required to verify the durability of these forms.

Results of leach tests performed on the W.E.P. waste forms made with actual mixed waste from Hanford's ETF are provided in Tables 8 and 9. Results of both the ANSI 16.1 and RCRA required TCLP leach tests indicate acceptable performance for land disposal of the waste form. The adequate TCLP leach performance can be partly attributed to the TCLP sample preparation method, where 8-mm molds were prepared, as opposed to cutting or grinding from a larger waste form. However, comparison of the TCLP leach performance data to the newer UTS, indicates a failure for cadmium.

Table 7 Results of higher salt only waste loadings for the orthophthalic resin

Run #	Waste type and loading	Wt.% Initiator (MEKP 925)	Peak exotherm (° C)	Time to peak exotherm, hours
1	50 wt% NaCl	2.5	135.3	0.57
2	30 wt% NaCl	2.5	152.8	0.55
3	70 wt% NaCl	2.5	97.2	0.80
4	50 wt% $NaNO_3$	1.0	126.0	0.67
5	70 wt% $NaNO_3$	1.0	91.6	1.00

Table 8. Leachability indices for an ETF mixed waste form made with the W.E.P.

Analyte	As	Ba	Cd	Cr	Pb	Se	^{137}Cs	^{60}Co	^{99}Tc	^{90}Sr
L.I	8.0	10.4	9.4	7.5	9.8	8.6	10.1	10.1	10.2	10.8

Table 9. TCLP results for an ETF mixed waste form made with the W.E.P.

Toxic Metal	Units	Max. RCRA allowable concentration	TCLP leachate concentration
As	mg/L	5.0	0.15
Ba	mg/L	100.0	0.51
Cd	mg/L	1.0	0.51
Cr	mg/L	5.0	0.42
Pb	mg/L	5.0	0.35
Se	mg/L	1.0	0.35

SECTION 4

TECHNOLOGY APPLICABILITY AND ALTERNATIVES

Competing Technologies

Over the last 50 years of DOE operations, large quantities of mixed low-level wastes (MLLW) were generated from past nuclear weapons based industries and more recently from waste treatment processes. Stabilization has been and still is an effective, inexpensive, and simple treatment alternative for many types of these mixed wastes. This alternative is also accepted as safe and environmentally sound by both the regulators and the concerned public. A review of the technical literature and of past DOE operating experience shows that low-temperature hydraulic cement and bitumen grout based stabilization methods produce waste forms that meet or exceed final disposal requirements. The success level of the waste form is dependent on the original waste medium and the type and amount of hazardous and/or troublesome components in the untreated waste. Unfortunately, these current stabilization techniques have had limited success in accommodating homogeneous solid and sludge wastes containing relatively high concentrations of salts in addition to RCRA hazardous heavy metals.

Salts (defined as the reaction product generated when a metal ion replaces the hydrogen ion of an acid) are generally highly soluble and reactive. As a consequence of these characteristics, low- temperature cement stabilized forms of MLLW containing salts do not adequately cure. Salts also interfere with the basic hydration reactions of cements and easily undergo mineral growth that can cause deteriorating expansions. This deterioration may lower the durability and strength of the stabilized waste form and create pathways for the hazardous and radiological constituents to be released from the immobilized waste.

In some cases, leach resistant salt waste forms of sufficient durability are possible with the current basic cement techniques. However, these techniques usually result in forms with excessive increases in waste volume due to low waste loadings. Basic Portland cement formulations can only accommodate ~15 wt% of salts. These process inefficiencies and subsequent high disposal costs offset any benefits. The limitations of these current methods are of immediate concern since future volumes of salt wastes are anticipated as other MLLW treatment processes are implemented. Future effluents from MLLW wastewater treatment systems and scrubber blowdown from future and present MLLW thermal systems (i.e., incinerators and melters) will significantly add to the MLLW salt inventory.

There are many mixed waste stabilization/solidification technologies at various stages of development that could be considered as competing with the polyester resins microencapsulation process. Numerous tests with low-temperature stabilization techniques involving ceramics, enhanced cements, and other polymers indicate that greater waste loadings (than those achievable with conventional Portland cement) are possible with even the troublesome salt-containing wastes. In addition, alternatives involving thermal-sintering techniques also may lead to acceptable waste forms with considerably more volume reduction compared to that achievable with grouts or ceramics. Mixed waste stabilization methods currently in the later stages of development include phosphate-bonded ceramics, enhanced concretes using proprietary additives, and several methods provided by commercial vendors. Low-temperature methods like phosphate bonded ceramics not only provide a low porosity ceramic barrier, but render the RCRA metal less hazardous by converting it to the insoluble phosphate salt. Microencapsulation techniques involving, polyethylene, infiltrated polyceramic sol-gels, and polysiloxane have also been demonstrated on surrogate and/or actual salt containing mixed wastes.

Sintering differs from vitrification in that only melting at grain phase boundaries occurs without the complete amorphous restructuring that takes place in glass formation. Like vitrification, sintering occurs at temperatures over $1,000^0$C and can emit volatile hazardous metals. Even though densification is possible for some additional volume reduction, slight volume increases usually occur. However, waste loadings as high as 80% are possible. The equipment for sintering is less complex than vitrification, but more complex than grouting. For a typical sintering process, grinding, mixing, and extruding equipment are required, as well as ovens, calciners, and offgas treatment systems. For most waste streams, sintering methods will require an extensive process development effort involving statistically designed experiments.

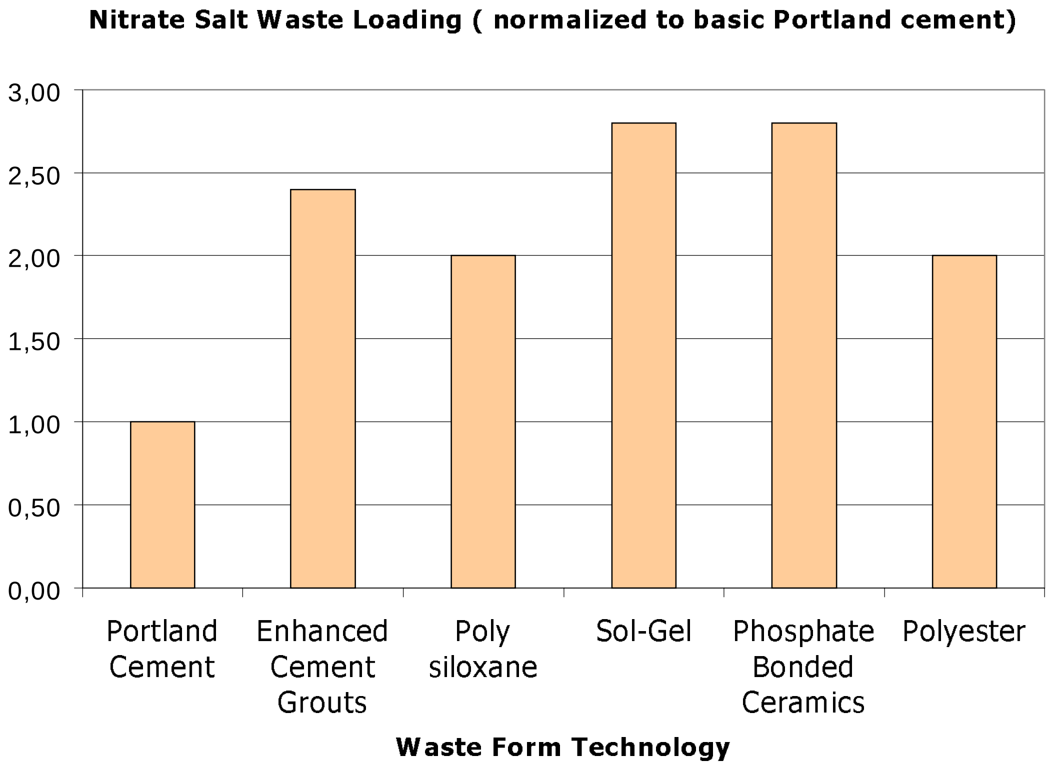
Recently developed alternative polymeric methods using batch mixers or extruder systems are currently available. Like polyester, these low-temperature microencapsulation techniques do not chemically incorporate the waste, but create an impermeable barrier between the hazardous components in the waste and the environment. Waste loadings in these organic media are usually on the order of 50% for many troublesome wastes, such as incinerator fly ash or those containing appreciable salts. This value is at least twice that achievable with conventional cement grout methods.

Table 10 and Figure 3 give ‘head –to –head’ comparisons of the waste form performance between that of polyester microencapsulation and some of the other more recently developed salt-containing, mixed waste stabilization methods as described above. Table 10 provides waste loading, strength, and leachability results for five low-temperature technologies stabilizing the same two types of salt surrogates. The pass criteria for TCLP leachability in Table 10 are the less stringent non-UTS values. Figure 3 graphically represents the waste loading benefit achieved over simple Portland cement grouting when using polyester microencapsulation or any of the other recently developed methods. The values have been normalized to the approximate salt-waste loading limit (i.e., 15-wt%) of basic Portland cement. Therefore, the value of 2 for polyester represents a 30-wt% (i.e., 2 X 15-wt%) salt loading (e.g., a 50-wt% loading of a surrogate waste containing 60-wt% nitrate salts).

Table 10. Selected salt waste stabilization results and waste performance data with identical MWFA surrogates.

Waste form performance measure	Waste loading wt%	Salt loading wt%	Compressive strength psi	Leachability TCLP	Leachability ANSI 16.1 index
Surrogate type	*Nitrate/ Chloride*	*Nitrate/ Chloride*	*Nitrate/ Chloride*	*Nitrate/Chloride*	*Nitrate/ Chloride*
Technology					
Enhanced concrete	69/55	36.3/8.2	1,032/535	Passed for Cd, Cr, Ni, and Pb	NA
Polyester (vinyl ester resin only)	**50/50**	**30/7.5**	**6,200/5,100**	**Passed for Cr, Cd, Pb, Hg**	**7.7/7.6 for Na ions**
Polysiloxane	50/50	30/7.5	420/>637	Passed for Cd, Cr, Hg	NA
Phosphate bonded ceramics	70/58	42/9.0	3,500/1,800	Passed for Cd, Cr, Pb, Hg, Ni	7.1 for Cl 9.0 for NO_3
Sol-gel	70/60	43.5/11.5	1,513/1,050	Passed for Cd, Cr, and Hg	7.6/7.5 for Na ions

Figure 3. Salt Waste loading comparisons for five low temperature stabilization methods and Portland cement grouting.



As indicated by the comparison data in Table 10 and Figure 3, no cement alternative, low-temperature stabilization technology for salt waste clearly out performs the others. Potential end users may need to consider other factors outside of waste form performance in choosing an alternative. These factors include, but are not limited to, the issue of 'stabilization versus encapsulation' technology, the availability of equipment, previous operating experience, the applicability of the technology to other types of waste media, and issues involving both safety and stakeholder concerns.

Technology Applicability

As long as adequate mixing is achievable, polyester resins are applicable to relatively inorganic, homogeneous low-level and low-level mixed waste solids, such as soils, evaporator residues, and fly ashes. Testing has shown that since polyester resins are available in a variety of forms and structures (e.g., W.E.P.s), they are applicable to both dry and wet solids. As indicated by the test results reported in this ITSR, they are applicable to mixed wastes containing high levels of salts and RCRA hazardous metals. Care must be taken when the salt is also an oxidizer, such as sodium or potassium nitrate. The mix of an organic waste form like polyester with an oxidizing waste can be unstable if temperature increases become excessive as a result of the exothermic polymerization reactions.

Polyester resin microencapsulation is not applicable for large debris, organic waste, energetic, or highly reactive, and explosive wastes. They are not applicable for transuranic (TRU) wastes since alpha radiolysis of the organic polyester will result in hydrogen gas generation, which may in turn create an explosive and/or pressure hazard in the waste drum.

A long history of successful LLW encapsulation validates polyester's treatment applications to reprocessing plant wastes, ion exchange residues, and reactor evaporator concentrates containing sodium sulfates.

Patents/Commercialization/Sponsor

Since polyester resins have been in use as a waste binder for over 25 years, there are no existing patents protecting its exclusive use or limiting its deployment toward DOE mixed waste. Presently, no commercial entity is actively promoting the technology as a mixed waste treatment route, however several have available processes for the low-level wastes generated by the nuclear power industry. The more notable organizations that have used and/or are using the polyester resin waste encapsulation method include:

- Japanese Gasoline Corporation (JGC),
- General Electric Company,
- Dow Chemical,
- Ontario Hydro,
- Technicatome, France,
- Diversified Technologies

SECTION 5

COST

Methodology

Cost estimates for deploying an operational-scale system for the polyester microencapsulation of mixed wastes are based on previously established systems for cementitious stabilization methods, particularly if they include planetary type mixers. Capital, labor, and material cost estimates assumed a batch 55-gallon drum capacity system with a waste form production rate of three barrels per shift, --- or nine barrels a day for an 'around the clock' operation. This rate corresponds to a waste throughput of ~1.87 cubic meters per day, assuming an average waste-to-waste form volume expansion of ~ 0%. The mixed waste throughput is based on waste form performance data collected during the development of the polyester process as reported in this ITSR. These data indicate that the polyester waste forms can consistently and reliably maintain a 50 wt-% loading of dry mixed waste containing an appreciable salt component.

Labor costs assume four Full -Time Equivalents (FTEs) at the fully loaded rate of $70.00 per hour. Labor costs in the methodology assume two full-time operational technicians, as well as part-time management, engineering, safety, maintenance, health physics, manifesting, and transportation personnel. Polyester material costs are assumed at the supplier (i.e., Ashland Chemical Company) bulk rate of $1.22/lb and are based on an average of the four costs provided in Table 2. New standard DOT 7A barrels are estimated at $100.00 each and the assumed operation consists of using the same drum for the mixing, curing, and final disposal of each 55-gallon batch.

Disposal costs vary depending on which available LLW, Subtitle D disposal site is used. At the INEEL's Radioactive Waste Management Complex (RWMC), a relatively low cost of $7.08 per cubic feet of waste form is available, whereas at the Barnwell Site in South Carolina costs can run as high as $1500 per cubic foot of waste form. For this particular analysis, a value of $60 per cubic foot of waste form was chosen, since it is an intermediate number frequently used in cost and system-engineering studies on mixed waste management throughout the DOE complex. Assuming that the volume expansion of a cementitious waste form is approximately 75%, the cost of disposing an equal amount of waste in a polyester matrix is ~57% of that of cement, since the waste loading in polyester can be over twice that of cement. This assumption and its related calculation is based on a polyester waste from density of ~ 1400 kg/M^3 compared to a cement waste form density of over 2650 kg/M^3.

Cost Analysis

Available cost documentation for existing or planned stabilization systems at Argonne National Laboratory-East (ANL-E) and INEEL indicate capital costs, including equipment design and development, for a total of ~$2000K. Because standard and similar equipment is deployed, this cost is valid for either stabilization (i.e., cement) or nonextruder microencapsulation (i.e., polymeric) systems. The capital cost estimate assumes a 55-gallon batch system and also assumes available infrastructure and facility housing to support the system. Operating costs, which include labor and material costs, are estimated at ~ $5940 per cubic meter of waste form based on the data provided in the above methodology. A corresponding cost for the baseline cement process is lower at ~$4430 per cubic meter of waste form because of its inexpensive materials costs. However, the disposal cost of the polyester waste form (~$2100 per cubic meter of waste) is less than that for cement (~$3700) as a result of its accommodation of higher waste loadings. All estimates are based on immobilizing a sodium nitrate sludge waste with a stabilized Specific Gravity (Sp. Gr.) of 2.0, and bulk dry waste Sp. Gr. of 0.7.

Cost Conclusions

The cost effectiveness of a polyester, microencapsulation waste treatment process is best determined by comparing it to the baseline process, basic Portland cement stabilization. A summary of the costs and associated data calculated for the two waste forms is provided in Table 11. The assumption that labor and capital costs for the two processes are the same is a valid one considering that for a given waste form throughput, the processing times, batch capacities, and types of equipment are similar, if not identical. Therefore, the cost benefit of polyester over that of the baseline is based exclusively on material cost and disposal costs. Cost estimates for treating waste volumes over 30 cubic meters indicate that with the high waste loadings achievable with polyester, the process more than recovers the higher initial material and development costs. For small waste volumes, higher waste loading becomes less of a factor and cement stabilization may be just as cost-effective.

Table 11. Calculated cost, material, and performance data for comparing polyester and cement waste forms

	Polyester Waste Form	Cement Waste Form
Waste Form Production Rate bbls/day	9	9
gals/day	495	495
cubic meters/day	1.874	1.874
kgs/day	2623	4994
Waste-to-waste form volume expansion	0%	75%
Waste Throughput cubic meters / day	1.25	1.07
kgs/day	1312	749
Waste Loading	50-wt%	15-wt%
Polyester Use kgs/day	1312	0
cubic meters/ day	.937	0
Polyester: waste weight ratio	1	0
Cement : waste weight ratio	0	5.66
Polyester: waste form weight ratio	0.5	0
Cement :waste form weight ratio	0	0.85
Labor $/day	6720	6720
$/ kg of waste	5.12	8.97
Barrels $/day	900	900
$/kg of waste	.686	1.20
Materials-Polyester $/day	3520	0
$/kg of waste	2.68	0
$/kg of waste form	1.34	0
Materials-Cement $/day	0	425
$/kg of waste	0	0.57
$/kg of waste form	0	.09
Disposal $/M^3 of waste form	2118	2118
$/kg of waste	3.03	5.29
$/kg of waste form	1.51	0.80
Total (Labor+Material+Disposal)		
$/day	15,109	12,261
$/kg of waste	11.52	16.37

SECTION 6

REGULATORY AND POLICY ISSUES

Regulatory Considerations

The regulatory goal of any end user deploying the polyester microencapsulation process is to produce waste forms that meet LDRs for land burial. In most applications, this means that the RCRA 40 CFR 268.40 treatment standards must be met at the lower limits proposed by the UTS, if the waste form is to be placed in a Subtitle D landfill. As a result, any full-scale, treatment facility will require a Part B RCRA permit or a modification to an existing permit.

Validation that the treatment standards are met requires leach testing using the EPA established TCLP method. In accordance with the method, TCLP test specimens must be of a certain size. As a consequence, the polyester waste form will be cut or ground to meet the size requirement. This sizing technique will more than likely damage the outside layer of the polyester waste form, causing unacceptable leaching of RCRA contaminants. To combat this undesired result, many waste treatment managers chose to make polyester leach specimens with small molds that meet the TCLP specimen size requirement. The acceptance of this approach by both regulators and various disposal sites vary, and potential end-users of the polyester process should fully understand their local acceptance of this approach before deployment.

Nuclear Regulatory Commission (NRC) 10 CFR 61 waste form testing will also be necessary if disposal is to be in an NRC licensed facility. Additional requirements for applying encapsulation using polyester resins at a federal facility include a National Environmental Policy Act (NEPA) review (a categorical exclusion is most likely to be applied), and any air emission considerations and/or permits as required under the National Environmental Standards for Hazardous Air Pollutants (NESHAPS). Any commercial facility treating radiological waste must secure an NRC permit.

If future development using polyester resins requires testing with actual waste streams, a NEPA approval through a categorical treatability study exclusion must be obtained. The state cognizant environmental agency in which the treatability study is to be performed must be notified 45 days before receiving archived samples for testing. In addition, the regional EPA must be notified.

Safety, Risks, Benefits, and Community Reaction

Polyester microencapsulation is a relatively simple and safe process. Excessively high temperatures are not achieved (e.g., less than 150 degrees C), pressures are ambient, and with the exception of the mixing impeller, there are few complex or moving parts. However, safety precautions in regard to the high flammability of styrene need to be implemented. This may involve the grounding of certain equipment to avoid sparking. Any styrene vapors generated could be removed however, with the use of a carbon absorption bed. The lesser concern of stabilizing oxidizing salts in an organic polyester matrix must also be mitigated. Administrative and engineering controls are required to avoid temperature increases that could lead to unstable conditions when stabilizing oxidizing salts (e.g., nitrates).

The polyester process generates no potentially hazardous offgases or secondary wastes, and therefore should bring little or no resistance from community stakeholder groups. These groups may even favor the technology, since it will generate less disposal volume when compared to the baseline Portland cement method.

SECTION 7

LESSONS LEARNED

Implementation Considerations

Considering the vast experience available on the use of polyester resins as a LLW encapsulant in the nuclear power industry, implementation of the technology for DOE mixed wastes should be fairly straightforward. However, there are a few factors that require consideration before its deployment:

1. A decision needs to be made as to which type of two basic systems to employ. For one type of system, the equipment can be integrated to pour uncured polyester –waste mixes from a dedicated mixing vessel to the disposal drum for final setting and curing. Another system involves planetary, hydraulic-type mixer systems to encapsulate and cure the polyester – waste mix in the same drum to be used for disposal. The latter systems are usually less complex and costly, but do not easily handle process upsets (e.g., feeding improper amounts of resin, waste, or initiator) or the reworking of waste form batches that fail disposal requirement specifications.

2. The potential end user must put in place proper administrative and engineering safety controls to address and mitigate the two identified safety issues. The grounding of various equipment to avoid sparking may be necessary since styrene is highly flammable. Cooling capability may be necessary for the mixer-reactor system to ensure temperatures are limited to below critical values. The high exotherms generated may result in unstable conditions for situations involving the polyester encapsulation of wastes containing oxidizing salts.

3. The potential end user must also establish and maintain development facilities to support the operational-scale systems. Testing will be required to identify operating parameters and exotherms for new waste streams to be polyester encapsulated. Key operating parameters to be determined through development include waste loading, resin type, initiator amounts, cure time, and the extent of temperature control.

Technology Limitations and Needs for Future Development

As previously mentioned, the polyester waste treatment process is an encapsulating method, and as such the hazardous and radiological components of the salt waste stream are not truly stabilized. The process simply provides a water-resistant barrier between the waste contaminants and the environment. Further development to identify chemical additives to the polyester process that may increase its ability to reduce the solubility and toxicity of the RCRA hazardous metals in the mixed waste is of value. The addition of additives (i.e., sulfide and phosphorous compounds) that will react with the RCRA metals to convert them to less soluble forms may avoid contamination of the environment if the polyester resin barrier is ever compromised.

Past documentation on various polymeric (organic) based methods indicates a potential for creating an unstable mixture when wastes containing oxidizing salts, such as nitrates are encapsulated. Flammability testing is recommended before deploying the polyester process in these applications. This testing should identify the type of polymer resin to be used as well as safe waste loading amounts and temperature limits. Development of polyester resins that use alternative cross-linking polymers is also justified in light of the high flammability of styrene.

Technology Selection Considerations

Obviously, DOE complex wide end users with the responsibility of mixed waste management need to consider multiple factors when selecting a low temperature stabilization technology, like polyesters. The most important factors are usually total waste volume, waste characteristics and constituents, technology simplicity and stakeholder concerns.

Waste Volume

The greater the volume of a relatively homogeneous waste inventory, the greater the benefits of a cement grout alternative, like polyesters. The more waste treated, the greater the savings in handling, transportation, and disposal costs as a result of the greater waste loadings polyesters can provide. If there is sufficient waste volume, these greater savings can more than recover any high up- front material and development costs.

Waste Characteristics and Constituents

Since the polyester method is primarily an encapsulation process, wastes containing high (i.e., >5000 ppm) concentrations of RCRA TCLP methods may not be sufficiently immobilized. Depending on how the TCLP samples are prepared from the waste form, these samples may fail, especially at the current UTS levels. Such wastes may require pretreatment steps to lower the solubility of troublesome constituents before the polyester process. Size reduction of the waste feed may also be required to ensure thorough mixing and adequate microencapsulation. Depending on the particular troublesome component and its concentration, these pretreatment steps may be too expensive to implement. As a consequence, investigations into other inorganic based techniques, such as modified cement grouts and low temperature ceramics may be justified.

Technology Simplicity

The primary reason that polyester encapsulation offers an attractive alternative to other treatment/encapsulation options is the straightforward nature of its application. Very limited engineering development would be required to build a prototype facility, which could eventually be expanded into a full-production operation capable of handling a variety of waste streams. No major investments would be required to perform engineering design studies; rather, the pilot plant could be designed using system integration of existing off-the-shelf systems.

Stakeholder Concerns

In general' stakeholders desire, low –temperature, nonoffgas producing stabilization technologies that generate no secondary wastes, minimize disposal volumes, and ensure long-term durability. Polyester resin encapsulation meets the first four criteria, but very little data exist to support its long-term effectiveness in maintaining durability over its indefinite disposal life. Critics of the technology question polyester's ability to remain durable over time, especially after encapsulating wastes containing high levels of salts and RCRA hazardous metals. Documentation in the field suggests that polymeric embedded salt solutions may swell the waste form over time as a result of the large osmotic pressures created. This swelling can be of the extent to compromise the barrier. Therefore, testing is necessary to qualify and quantify the long-term durability of polyester microencapsulated salt wastes.

APPENDIX A

BIBLIOGRAPHY

1. Biyani, R. K., J. G. Douglas and D.W. Hendrickson, 1997, *Test Plan for the Immobilization of Salt-containing Surrogate Mixed Wastes Using Polyester Resins*, HNF-SD-RE-TP-026, Rev. 0, SGN Eurisys Corporation, Richland, WA, June 30, 1997.

2. Biyani, R. K. and D.W. Hendrickson, 1997, *Test Procedure for Polyester-Immobilized Salt Containing Surrogate Mixed Wastes*, HNF-SD-RE-TPI-005, Rev. 0, SGN Eurisys Corporation, Richland, WA, July 18, 1997.

3. Biyani, R. K., 1997, *Treatability Test Plan and Procedures for Immobilizing Salt-containing Mixed Wastes Using a Water-extendible Polyester Resin*, SESC-EN-TPI-001, Rev. 0, SGN Eurisys Services Corporation, Richland WA, November 13, 1997.

4. Biyani, R.K., 1998, *Report on Microencapsulation of Surrogate Salt Wastes with Polyester Resins*, SESC-EN-RPT-017, Rev. 1, SGN Eurisys Services Corporation, Richland, WA, January 29, 1998.

5. Biyani, R. K., 1998, *Treatability Test Report on Microencapsulation of Mixed Waste in a Water-extendible Polyester Resin*, SESC-EN-RPT-024, Rev. 0, SGN Eurisys Services Corporation, Richland, WA, March 1998.

6. IAEA, 1988, *Immobilization of Low and Intermediate Level Radioactive Wastes with Polymers*, Technical Report Series No. 289, International Atomic Energy Agency, Vienna 1998.

7. Bleier, A., 1997, *Evaluations of Final Waste Forms and Recommendations for Baseline Alternatives to Grout and Glass*, ORNL/TM Report 13214, Rev. 0, Oak Ridge National Laboratory, Oak Ridge Tennessee.

APPENDIX B
TMS DATA ELEMENTS

Funding Source

This section provides cross-reference information in regards to the EM-50 Mixed Waste Focus Area contract established for development of the polyester salt-containing mixed waste microencapsulation process. The Department of Energy- Headquarters Technology Management System title and tracking number is provided as well as that of the specific Technical Task Plan (TTP).

TMS # 1685 Stabilization of Salt Waste Using Encapsulation with Polyester Resin

TTP # RL47MW42 Micrencapsulation of Salt Waste Using Polyester Resin

APPENDIX C

ACRONYMS

ANL-E	Argonne National Laboratory -East
ANSI	American National Standards Institute
ASTM	American Society for Testing and Materials
cp	centipoise (viscosity units)
DOE	Department of Energy
EM	Environmental Management
EPA	Environmental Protection Agency
FTE	Full-Time Equivalent
ETF	Effluent Treatment Facility at Hanford
INEEL	Idaho National Engineering and Environmental Laboratory
ITSR	Innovative Technology Summary Report
JCG	Japanese Gasoline Corporation
LDR	Land Disposal Restriction
LLW	low-level waste
MEKP	Methyl Ethyl Ketone Peroxide
MLLW	mixed low-level waste
MWFA	Mixed Waste Focus Area
NEPA	National Environmental Policy Act
NESHAPS	National Environmental Standards for Hazardous Air Pollutants
NRC	Nuclear Regulatory Commission
OST	Office of Science and Technology
RCRA	Resource Conservation and Recovery Act
RWMC	Radioactive Waste Management Complex
Sp.Gr.	Specific Gravity
TMS	Technology Management System
TTP	Technical Task Plan
TCLP	toxicity characterization leaching procedure
UTS	Universal Treatment Standard
W.E.P.	Water Extendible Polymer