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### Mixed Waste Separation Technologies

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## MIXED WASTE SEPARATION TECHNOLOGIES

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### ABSTRACT

The United States Department of Energy policy for management of mixed (radioactive and hazardous) waste is in the development stage. Regardless of the approach to treatment of mixed waste, significant preprocessing will be necessary. The separation requirements for preprocessing of mixed waste will vary greatly depending on the downstream treatment requirements, the contents of the mixed waste stream, and the regulatory requirements at the waste treatment site. During the past year, the Department of Energy's Mixed Waste Integrated Program (MWIP) has begun to identify separation requirements, applicable commercial technologies, and emerging technologies that may meet specific requirements.

The current emphasis on developing emerging separation technologies for the MWIP include: freeze crystallization for gross separation of dissolved solids and organics from an aqueous waste stream; a bio-catalytic process for nitrate destruction; the General Electric KI/I<sub>2</sub> process for separating mercury from noncombustible solids and aqueous sludges; and the 3M/IBC membrane technology for separating mercury, cesium and strontium from aqueous streams.

### INTRODUCTION

The Mixed Waste Integrated Program (MWIP) was created by the DOE Office of Technology Development to perform research, development, demonstration, test, and evaluation (RDD&E) on technologies for the treatment of all DOE

low-level mixed waste. Mixed waste is defined as containing low-level radioactive components and hazardous components regulated under the Resource Conservation and Recovery Act (RCRA) and associated EPA regulations. MWIP goals are to develop improved technologies, in terms of improved treatment and lower cost, and to hand-over proven technologies to the DOE Office of Waste Management (OWM) for implementation at the DOE sites.

The MWIP has established five technical areas based on the primary functional areas of a Mixed Waste Treatment Plant (MWTP) defined by the OWM. These areas are: (1) Front-End Handling (FEH), (2) Chemical/Physical Treatment (CPT), (3) Waste Destruction and Stabilization (WDS), (4) Second-Stage Destruction and Offgas Treatment (OGT), and (5) Final Waste Forms (FWF). The FEH System receives, sorts, and distributes the waste streams to downstream processes. The CPT System pretreats and separates the incoming waste streams for efficient downstream processing in the WDS and FWF Systems. The WDS System destroys the organic components and, in some cases vitrifies the inorganic components, through incineration, plasma arc furnaces, metal melters, and alternate destruction technologies. The OGT System treats the offgas from all other processes within the MWTP. Finally, the FWF System stabilizes the resulting waste into a form that can be delisted and disposed of in a low-level waste landfill.

The Chemical/Physical Treatment System (CPTS) performs the required pretreatment, volume reduction, and/or separations on the waste streams passing through the system for discharge to the environment or efficient downstream processing in the WDS, OGT or FWF Systems. The current philosophy is to minimize the separation requirements in the MWTP in order to minimize the plant and characterization costs. The known separation requirements are the following:

1. Separation of water from solid and liquid organics for efficient thermal treatment of the organic waste stream. This will produce two waste streams, one with less than 10% organics for destruction of dilute organics and one with less than 10% water for efficient organics destruction by thermal (or non-thermal) treatment.
2. Separation of inorganics from the aqueous and wet solids waste streams. The "clean" water would be further treated or discharged, and the low volume of dry inorganic solids would be sent to final forms processing.
3. Separation of metals from organic solids and separation of ferrous and non-ferrous metals for efficient metal processing, and efficient thermal treatment of the organic solids.

4. Separation of mercury from the aqueous, wet solids, and non-combustible solid waste streams. Combustion of mercury contaminated liquid and solid organics (i.e., combustibles) is a BDAT and it is assumed that the mercury will be captured in the offgas system; probably using a sulfur impregnated carbon adsorption bed.

These are fairly straight-forward separation needs using readily available technologies (except for mercury removal which will require some development work). Costs and difficulties begin to arise if selective separations of specific species from the various waste matrices are required. Although selective species separation requirements have not yet been defined by the processes downstream from the CPTS, removal of certain species from the waste stream may be required in order to produce an acceptable final form, to allow efficient destruction of organics, or to minimize the cost of the waste destruction and/or offgas systems. Potential requirements include the following:

1. Removal of sulfate, phosphate, and chromium salts from aqueous and wet solids waste streams. These species may inhibit or degrade the performance of the waste form or the treatment process. In particular, chromium, cadmium and noble metals degrade performance of glasses and/or glass melters.
2. Removal and/or destruction of nitrates which become reactive or explosive in some waste forms, and which must be removed to regulatory levels in the aqueous stream before discharge.
3. Removal of soluble radionuclides and heavy metals which may prevent formation of a final form that passes the leach criteria. For example cesium, strontium, and technetium have high mobility in many waste forms. Other species, such as nitrates, phosphates, sulfates, and fluorides, may be leachable from the final waste form.
4. Removal of volatile metals from aqueous and wet solids before the dried inorganics are sent to a vitrifier.
5. Removal of chlorides from aqueous and wet solids waste streams. Although chlorides will not prevent formation of an acceptable glass, they will volatilize and will require an extensive offgas system for the melter. The chlorides will then need to be removed from the offgas scrubber solution and subsequently disposed of in a non-vitrified form.

Trade-offs must be made regarding the most cost-effective solution: to characterize the incoming waste stream and remove the metals and chlorides from

the incoming waste before the remaining solids are sent to the vitrifier, or to collect the metals and chlorides in the offgas system and remove them from the scrubber solution. In addition, regardless of the species that are separated from the main waste stream intended for discharge or disposal as a final waste form, development and deployment of a technology to manage and dispose of the recovered and concentrated species will be required.

Since the goal of the MWIP is to develop a final form that contains radionuclides and RCRA hazardous inorganics, and that can pass the EPA leach tests for delisting. Therefore, in general, radionuclides need not be separated from hazardous material (i.e., heavy metals).

As described, the range of separation requirements may be broad including: (1) gross separations in which the organics are separated and concentrated for efficient destruction, and suspended and dissolved solids are separated from the aqueous waste for final form processing; and (2) selective separation in which organics are separated from water and "problematic" species are removed to allow efficient production of a final form that passes the EPA leach tests. However, details of the waste stream compositions, and of the requirements for downstream processing, are not well known. Also, there are many separation processes available, both well developed and emerging, which may be applicable to treating the waste streams in the CPTS depending on the processing requirements. Efforts to identify system requirements, evaluate technologies, and identify gaps in our technology base have begun and are outlined in Reference 1.

Based on preliminary evaluations of technology requirements for the CPTS, the MWIP is currently pursuing efforts to evaluate and demonstrate the following technologies to satisfy the indicated needs:

Technology	Need
Freeze Crystallization	An efficient method for volume reduction and salt removal from the aqueous waste stream, and which is sufficiently versatile to be independent of waste stream variations.
Bio-Catalytic Nitrate Destruction	An efficient and inexpensive method for nitrate destruction.
Mercury Removal	Methods for removal of mercury from aqueous and solid waste streams.

### CURRENT SEPARATION PROJECTS

#### Freeze Crystallization

The freeze crystallization process separates water from solutions by cooling the solution until ice crystals form, usually as a pure material. The ice is separated from the remaining liquid and impurities, washed, and melted to produce a purified aqueous stream containing less than 0.1% of the contaminants (2). The dissolved components are concentrated into a reduced volume containing 99.9% of the contaminants and 10% or less of the water. High decontamination factors on the order of 1,000 to 10,000:1 (ratio of concentration in the feed to concentration in the melted ice) are achievable with large scale systems (2). This includes inorganics, organics (including volatile organics), heavy metals, and radionuclides. A typical process flow is shown in Figure 1.

This technology could replace evaporation/crystallization used to vaporize water to concentrate contaminants in an aqueous sludge. Evaporation is energy intensive whereas freeze crystallization has significantly lower operating costs. Low operating temperatures keep volatile organics from vaporizing thereby minimizing offgas issues compared with evaporation. A major benefit is the versatility of the technology and its ability to provide high levels of separation and high volume reductions, independent of the waste stream composition or waste stream changes, and in a single process indicating a potentially low cost option.

Freeze crystallization separation is based on the difference in component concentrations between solid and liquid phases in equilibrium. A simple binary solid-liquid equilibrium diagram shows that as a solution is cooled to a temperature at which the solvent begins to freeze, a solid crystalline phase begins to appear in the liquid phase. In the case of an aqueous solution this solid phase will be ice. Only a small amount of crystal forms at the initial freezing temperature. As the ice forms, the concentration of solute in the remaining water increases causing the crystallization (or freezing) temperature of the remaining liquid to drop slightly. Therefore, a lower operating temperature is needed to effect further crystallization.

The eutectic point is eventually reached where the solubility limit of the solute is reached and both the solvent and solute crystallize simultaneously at constant temperature. At this point two separate kinds of crystals are formed, rather than one crystal that incorporates the molecules of both components. In most aqueous

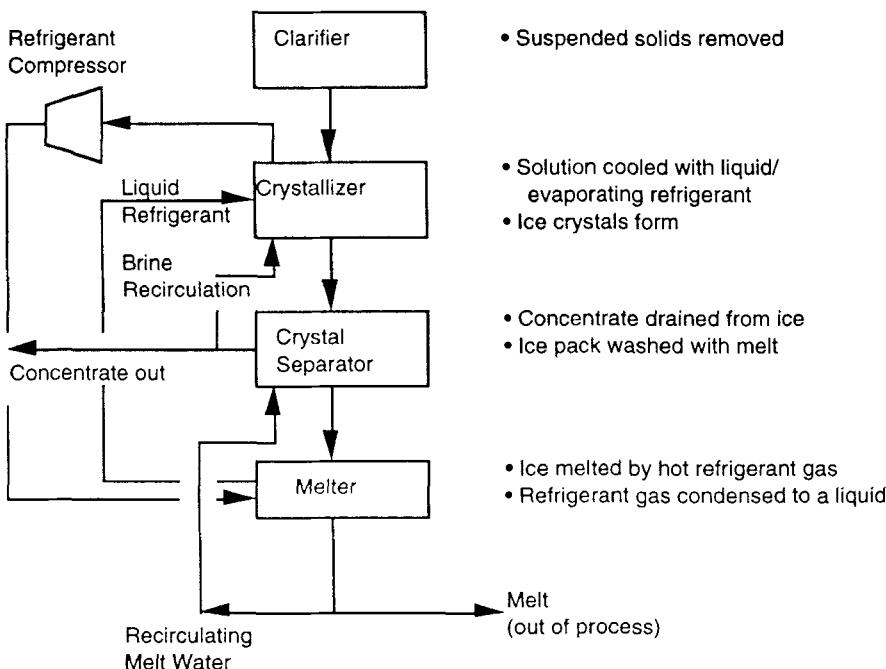


FIGURE 1. Simplified Process Flow

systems, it is possible to fractionate these solid phases by density differences. In multicomponent solutions, the occurrence of a dual-precipitation point may not result in a constant operating temperature. In such systems, the continued concentration of impurities in the liquid phase continues the depression of the freezing point of the eutectic composition by an amount dependent on the concentration of the impurities and the specific contaminants.

Upon leaving the crystallizer, refrigerant vapors are collected and compressed to a point at which the refrigerant can be condensed by either cooling water or by melting the ice crystals. The ice crystals in the system are separated from the rest of the solution and washed with melted ice in the ice washing column. A melted ice stream and a concentrated waste stream are generated in this process. When solubility limits are reached, inorganic salts precipitate and can be separated from

the ice crystals by density differences with a eutectic separator such as a hydrocyclone.

The freeze crystallization process has been commercialized by several vendors, with varying degrees of success. Two types of crystallizers are used to transform water into ice crystals: the indirect-contact and direct-contact crystallizers. Indirect freezing cycles remove the heat from the liquid solution using a heat transfer surface and a closed-cycle refrigeration system. When the driving force for crystal growth is through a heat-transfer surface, crystallization will normally start on that surface causing a crystal build up and decreased heat transfer rate. HPD Corporation has developed electropolished tubes and a tube coating to eliminate corrosion and minimize nucleation sites to prevent surface crystallization. A system using these tubes is currently being tested (3).

Direct-contact freezing cycles use a secondary refrigerant which is injected, as a liquid, into the process liquid at the bottom of the crystallizing column. Since the pressure of the system is less than the vapor pressure of the refrigerant, the refrigerant boils up through the crystallizing column, removing heat from the process liquid causing ice crystals to form. Although direct contact systems eliminate the corrosion and fouling problems, the refrigerant can become contaminated through entrainment of the waste solution requiring special precautions for maintenance of the refrigeration system.

The direct-contact secondary-refrigerant process is being developed by Freeze Crystallization Technologies Acquisition Corporation (FCTAC) as a separation and volume reduction process on aqueous mixed waste streams. Westinghouse Hanford Corporation (WHC) conducted proof-of-principle tests in early FY92 on waste simulants at FTCAC's pilot plant (4). Results in this non-optimized facility indicated a maximum ice recovery of 71%. Inorganic constituent removal was as high as 96.5% for an average ice melt, and 99.6% for the ice melt grab samples. These removal percentages are equivalent to decontamination factors of 27 for the average ice melt, and 249 for the ice melt grab samples. Problems included (1) loss of refrigerant, due to entrainment in the slurry out line, at a higher rate than was expected or is acceptable; (2) formation of a salt floc in the slurry from the crystallizing column which was not effectively removed using the hydrocyclone; (3) the system did not demonstrate removal of organics: this was attributed to dissolution of organics in tributyl phosphate which remained as an oil slick on top of the waste remaining in the feed tank (i.e., the organics did not enter the

crystallizer); and (4) the system experienced periodic plugging resulting in ice pack surges in the wash column which reduced the ice melt quality.

These systems are still in the pilot stage for application to mixed waste streams and will require additional testing to determine design, scale-up, and optimization parameters. Further development will require low temperature growth data to identify conditions needed to grow crystals and the operating envelope; a wash column to wash crystals with minimal re-dissolution; a control system to insure crystallization without freezing the equipment or system; instrumentation and control to monitor conditions such as foaming and excess ice inventory in the wash area; and development of a robust system that would handle a variety of waste streams without significant modifications.

Freeze crystallization has some pretreatment requirements and limitations. Material that cannot be separated in a gravity column must be identified and removed prior to introducing the waste stream into the freeze crystallizer. This includes colloidal material, and other material that will float to the top of the wash column with the ice crystals. The feed requires a freezing point depression so some minimum concentration, controllable by blowdown and recirculation. Finally, this technology is not applicable to small drum sized quantities; thus, the waste streams need to be segregated and combined into larger quantities for treatment.

#### Bio-Catalytic Nitrate Destruction

It is expected that most of the nitrate-containing mixed waste will ultimately be solidified for final disposal. The nitrates in the waste will generally increase the volume and/or reduce the integrity of all final waste forms that have been proposed. In addition to meeting final waste form requirements, destruction or removal of nitrates may be required to meet environmental discharge limits for aqueous waste water, to reduce NO<sub>x</sub> emissions from the incinerator, or reduce the hazard of vigorous exothermic reactions. Several nitrate destruction technologies are being investigated by the DOE including the nitrate to ammonia and ceramic process, electrochemical ion exchange, and steam reforming. Each of these methods has their advantages and disadvantages including process complexity and cost, and generation of secondary waste streams.

This initial research in bio-catalytic nitrate destruction, to be conducted at Argonne National Laboratory, is a proof-of-principle study to show feasibility of

using reductase enzymes, immobilized on a solid support, to reduce nitrates and nitrites in mixed waste to  $N_2$  and water. The reducing equivalents are provided by a low-voltage electrical current which transfers electrons from the cathode to the enzymes via an electron transfer dye. The use of enzymes enables very large specific catalytic activity to be obtained without the need for additional chemical reagents or the production of secondary waste streams. An aqueous biphasic system of waste water and an immiscible liquid phase in contact with the enzymes will be used to protect the enzymes from inactivation in hostile environments. The biphasic system is necessary to protect the enzymes from excessive concentrations of electrolytes, especially  $H^+$  and  $OH^-$ , which would result in enzyme inactivation.

Nitrate reductase enzymes from plant or bacterial sources are capable of reducing nitrate to nitrite and water. Nitrite reductase and nitrous oxide reductase continue the process by acting as electron acceptors and reducing nitrite to nitrous oxide and then to molecular nitrogen. In each reduction step, water is produced as a co-product. Co-immobilized electron-carrying dyes, such as safranin or bromphenol blue, act as electron donors to the enzymes. Reducing equivalents are delivered to the co-immobilized enzymes and electron-carrying dyes by a low voltage electrical current. The most effective reduction of nitrate is achieved when the matrix containing the enzymes is entrapped in a thin layer over a large cathode surface, and the water is first flowed past the anode (5).

Above 1.28 V, the electrochemical potential of water, water molecules are effectively converted to hydroxonium ions at the anode. The ions flow to the cathode, receive an electron, and decompose to water and atomic hydrogen. The co-immobilized redox dye effectively captures such atomic hydrogen before it can combine to produce molecular hydrogen effectively ensuring that molecular hydrogen is not produced. A reactor using reductase enzymes immobilized within a polymer matrix, has been shown to be capable of destroying nitrate and nitrite with specific activities of 500 - 600 kg nitrate per  $m^3$  per day for dilute aqueous streams at room temperature (5).

In the reactor described in Reference 5, enzymes were co-immobilized with electron-carrying dyes in a polymer matrix which was attached in thin layers to the cathode surface. This was a two stage process in which a nitrate reductase enzyme was used in the first stage to reduce nitrate to nitrite, and nitrite and  $NO_2$  reductase enzymes were used in the second stage to produce  $N_2$ . Nitrate laden water was pumped past the anode and through the active matrix on the cathode while a low

voltage was applied resulting in two-stage nitrate reduction. Preliminary tests indicate the reactor was stable over a period of 3 months, retaining more than 50% of its original activity. Extensive tests will be required to demonstrate long term functionality and stability of such a system in continuous operation.

Commercially available nitrate reductase enzymes may be immobilized by entrapment within a polymer matrix, such as polyacrylamide, or covalently bonded onto polymer substrates using mono- and bi-functional cross-linking agents. Co-immobilization of the enzymes with electron-carrier dyes, such as new methylene blue or thionin, which are good enzyme mediators and are easily reduced, increases the activity of the system by 30% over the activity of enzymes in solution (5).

Maintaining enzyme activity at very high ionic strengths and high concentrations of either  $H^+$  or  $OH^-$  is a serious problem and means that most radioactive wastes would require pretreatment before being fed into such a biocatalytic reactor. To protect the enzymes from these harsh environments, a biphasic system has been proposed in which the nitrate/nitrite is partitioned from the waste stream into an immiscible liquid phase, which is then pumped through the enzyme reactor (6). The continual removal of nitrate and nitrite by enzyme action would provide the thermodynamic driving force for mass transfer from the waste stream to the reactor. A proposed reactor concept is shown in Figure 2.

Aqueous solutions of either polyethylene glycol (PEG) or polypropylene glycol (PPG) are immiscible with high-ionic-strength aqueous electrolyte solutions. The polymer solutions contain 70-95% water permitting partitioning of fully hydrated species (7). The partition coefficients of PEG-4000 in equilibrium with sodium sulfate or sodium carbonate solutions are approximately 50 and 1000, respectively (8). Although PEG does not form a biphasic with nitrate or nitrite solutions, PPG does have this ability. While PEG is completely miscible with nitrate and nitrite solutions, high concentrations of  $OH^-$  and  $CO_3^{2-}$ , or moderate concentrations of  $PO_4^{3-}$ , in combination with nitrate or nitrite, will promote aqueous biphasic formation (8). These polymers are inexpensive, nontoxic, and nonflammable, and are known to prevent protein denaturation in harsh environments. Immobilizing the enzymes onto solid substrates would also contribute to enhanced resistance to inactivation.

This process would eliminate the need for chemical reagents, and minimize or eliminate secondary wastes such as  $NO_x$ , and secondary products such as  $NH_3$ ,

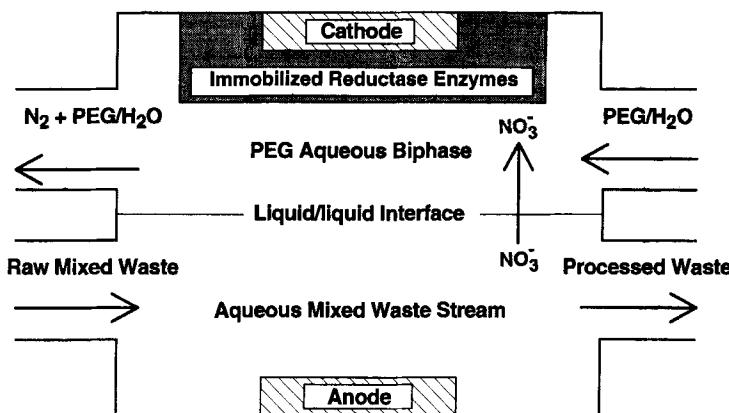


FIGURE 2. Conceptual Bio-Catalytic Reactor

H<sub>2</sub>, O<sub>2</sub>, and NaOH prevalent with other nitrate destruction processes. It is applicable to a wide range of aqueous waste streams with highly variable composition, and because living matter is not involved, it is not subject to shock of bacteria in typical biodenitrification systems due to sudden changes in the contents of the input stream. If this process is determined to be feasible it could provide a compact, low cost reactor to treat aqueous mixed waste streams.

#### Mercury Removal Technologies

The goal of this program is to develop low cost technologies to replace thermal bakeout processes and the attendant costs of permitting and offgas systems. This program will investigate and apply advanced treatment methods for mercury removal to specific waste streams selected from several DOE sites. The methods include acid leaching and the General Electric KI/I<sub>2</sub> leaching process for noncombustible solids and aqueous sludges, and activated carbon beds impregnated with sulfur for aqueous streams. Two other methods that will be investigated include ion exchangers and 3M membrane technology. Results of this effort will be sufficient data for pilot scale design.

**General Electric KI/I<sub>2</sub> Technology.** This process involves leaching of solid matrices with KI/I<sub>2</sub> solution to solubilize the mercury. This process was

invented and patented by General Electric Company at their Environmental Technology Laboratory in Schenectady, NY (9). Tests on a synthetic soil matrix dosed with metallic mercury and several mercury compounds including oxides, chlorides, sulfide, phosphate, nitrate, and methyl mercury chloride gave excellent separations. Tests were also made with numerous solid matrices including soils, plastic, concrete and brick.

The process uses a hydrometallurgical approach in which a solution of  $KI/I_2$  (a complexant and an oxidant) is used to solubilize various mercury compounds from solid substrates. The solution is collected and the mercury precipitated in the form of metallic mercury, and the  $KI$  and  $I_2$  recovered and recycled. The reagent is selective, with a high affinity for mercury over many other metals (e.g., Fe, Ca, Mg, Mn, etc.), and is capable of solubilizing many forms of mercury. Typical reactions of mercury derivatives with  $KI/I_2$  are shown in Figure 3. The residual mercury levels decrease with time, increased temperature, increased number of extractions, and higher  $KI$  and  $I_2$  concentrations. Results of preliminary data regarding residual mercury levels as a function of processing time and temperature are shown in Figures 4 and 5, respectively (10). This extraction process typically results in < 50 ppm residual mercury levels on the treated solids, which have passed the EPA TCLP tests independent of the media, Hg form, or Hg concentration (10).

The recycling loop removes mercury from the  $KI/I_2$  solution to reconstitute the extractant and maintain solution chemistry. This recycling process recovers >97% of the iodine making this process very cost effective (10).

**3M/IBC Membrane Technology.** The Efficient Separations/Processing Integrated Program is sponsoring a collaboration between the 3M Company and IBC Adavanced Technologies, Inc., to work with Pacific Northwest Laboratories to develop membrane systems that will selectively remove cesium and strontium from DOE wastes. IBC has developed a method of making highly selective, non-ion exchange, organic ligands chemically bonded to solid supports such as silica particles. 3M has developed methods for incorporating these particles into matrices resulting in membranes that are highly porous to afford very high flow rates. This technology is promising in applications where highly selective removal of various species, including mercury, is required from aqueous streams.

The IBC technology produces particles that can be incorporated into a variety of matrices. To date, the primary approach has been to attach synthetic ligands to

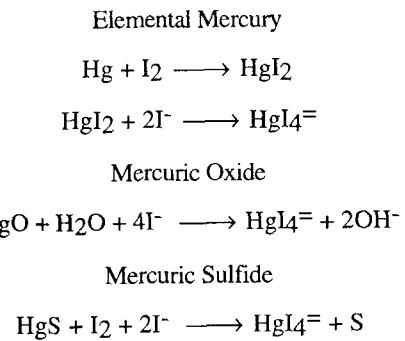
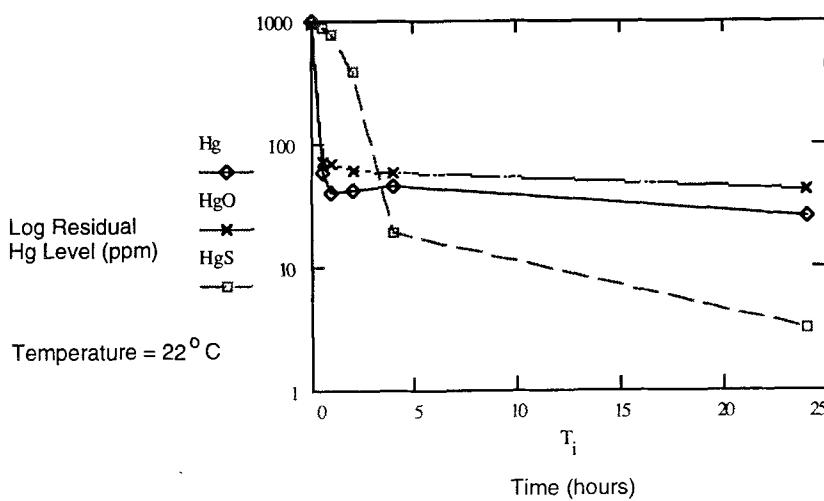
FIGURE 3. Typical Reactions of Mercury Derivatives with KI/I<sub>2</sub>

FIGURE 4. Residual Mercury Levels Versus Process Time

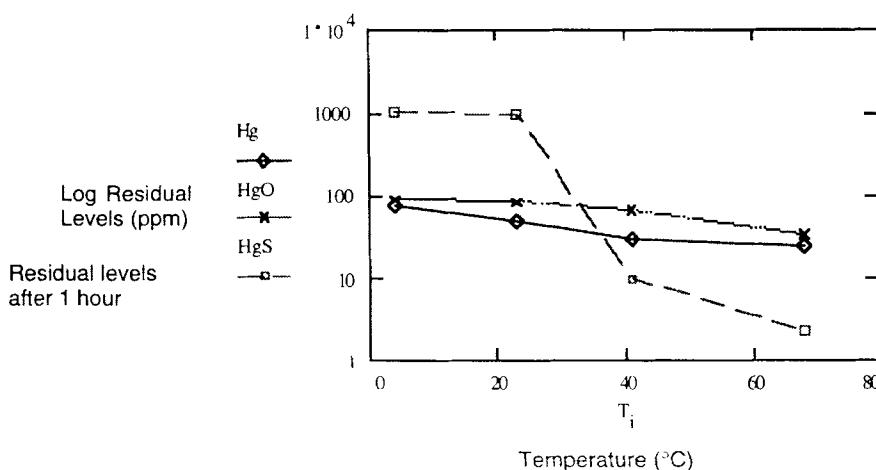


FIGURE 5. Residual Mercury Levels Versus Temperature

silica gel and utilize the modified solid particles in fixed bed columns. These products have been commercialized under the trade name SuperLig<sup>TM</sup>. SuperLig<sup>TM</sup> based systems can selectively bind a single or group of guest molecules, and can treat large volumes of contaminated mixtures with flow rates over ten times those of ion exchange systems. SuperLig<sup>TM</sup> systems exhibit several orders of magnitude selection preference for specific ionic species as compared to traditional separation techniques such as precipitation, ion exchange, and solvent extraction. Decontamination factors greater than  $10^6$  have been achieved and high selectivity in binding  $\text{Cs}^+$  and  $\text{Sr}^{2+}$  has been demonstrated, and binding constants, depending on the molecule used for  $\text{Hg}^{2+}$ , have been shown to exceed  $10^{14}$  (11). The Superlig<sup>TM</sup> materials have also shown good radiolytic stability at  $10^7$  to  $10^8$  rads of gamma radiation in  $\text{HN}_3$  (12).

The 3M Company's Empore<sup>TM</sup> membrane technology provides a method for enmeshing surface active particles in a net-like matrix of PTFE (polytetrafluoroethylene) fibers to form a coherent porous membrane with good integrity and handling strength, an extremely high particle surface availability, and extremely uniform particle distribution thereby eliminating channeling characteristic of particle based separation systems such as adsorption columns. These membranes provide very close, uniform particle spacing in thin, uniform cross-sections where

90% of the membrane sheet consists of particles. The membrane structures have tightly controlled physical properties such as pore size, pore volume, permeability, flexibility, and strength while maintaining good radiation stability, low pressure drop, and rapid flow using the fast kinetics of the coated particles. This combination of 3M membrane and IBC molecular recognition technology has demonstrated high levels of ion removal at very low flow rates (e.g., 200 bed volumes/minute) (12). The Empore<sup>TM</sup> technology can produce multi-layer membranes with different properties depending on the chemical characteristics of the particles used. Because of this, a broad range of chemical separation systems can be produced.

Tests in nonradioactive environments show that these membranes can isolate cesium and strontium, even when they are present at concentrations as low as a few parts per billion. Future efforts will involve evaluating these membranes in high radiation fields, testing the membranes on actual waste from the Hanford site, and engineering the membranes so they can be manufactured as appropriately shaped cartridges. The MWIP role will be to evaluate this technology for application to mercury removal and other selective separation requirements.

### SUMMARY

The treatment of mixed waste may require a wide range of separation technologies depending on the waste stream characteristics, downstream processing requirements, and the federal, state and local regulatory requirements at the site performing treatment. This paper described the known and potential separation requirements for a mixed waste treatment plant, and described the technologies under investigation for the MWIP.

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