"Liquid Membrane System for Removal and Concentration of Transuranic Elements"

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Introduction

The treatment and disposal of radioactive waste generated in past plutonium operations represents an immense technical and economic challenge. Current processing objectives are to separate the radioactive from the non-radioactive components of tank sludges in order to reduce the volume of HLW and lower the radiological risk associated with the LLW. It is estimated that 38,000 MT of HLW glass will be generated in processing of Hanford sludges using the baseline process coupled with oxidative leaching of chromium in select cases. The cost of processing and disposing has been estimated to be over \$5 billion.

Two chemical processes are presently being considered at the Hanford and Idaho tank facilities for achieving these separations: the transuranic extraction (TRUEX) process and the strontium extraction (SREX) process. While both TRUEX and SREX processes were originally intended for traditional solvent extraction equipment, this project deploys a unique contacting device for selective removal of transuranic elements and strontium-90 from dissolved tank waste. The contacting device is a unique Moving Liquid Membrane system (MLM) which is adaptable to both TRUEX and SREX processing. The MLM is an indirect contact reactor, which by its structure eliminates cross-contamination between the feed, extractant, and stripping phases. It operates continuously as opposed to the batch processing of conventional solvent extraction equipment.

The major advantage though of the MLM is its potential to operate with drastically lower quantities of extractant. This feature takes on added importance in radionuclide processing, where the cost of extractants can be very high and the size and space requirements for feed and storage tanks can be significantly reduced. Thus, the MLM offers substantial economic processing advantages over direct contact solvent extraction equipment.

Therefore, the thrust of this project is to develop an efficient, reliable and radiation-resistant Moving Liquid Membrane system (MLM) for the selective removal and concentration of TRUs and strontium-90 from dissolved Hanford sludge wastes. The features and advantages of the MLM are summarized as follows:

- Combines non-dispersive extraction and stripping steps within the same processing unit.
- Control and operation are easily managed which results in reduced maintenance and operating costs.
- Configuration is completely stable and not prone to performance-limiting effects such as membrane phase degradation, swelling, and loss of liquid.
- Has high permeability and is flexible and adaptable to different separation requirements.
- High selectivity can be achieved by incorporating the appropriate extractant (or carrier) in the membrane phase.
- Concentrating potential practically unlimited (for metal ion removals).
- Can be designed with a high mass transfer surface area within a small volume which results in small footprint size.

The project team for this work includes LSR Technologies, Inc., the prime contractor to DOE, and Battelle Pacific Northwest National Laboratories (PNNL), which supports the project as a subcontractor to LSR. In Phase I, Battelle provided technical support for testing the MLM with Hanford tank simulants in the LSR laboratory. In Phase II, Battelle will test LSR's membrane technology with actual tank waste in Hanford.

Objective

The goal of this program is to develop an efficient, reliable and radiation-resistant MLM for the selective removal and concentration of transuranic elements (TRUs) and strontium-90 from dissolved Hanford sludge wastes. The efforts are divided into three categories: 1) demonstration and optimization of the MLM for the TRUEX and SREX processes using simulant waste solution; 2) development of a radiation-resistant membrane and membrane module for testing with actual waste solutions; (3) demonstration of the MLM for the TRUEX and SREX processes using actual Hanford waste.

The program shall demonstrate that the MLM is simple, more efficient, and easier to control and operate than conventional solvent extraction processes, such as that using centrifugal contactors. The specific objectives for the proposed research are as follows:

- To demonstrate that the MLM can remove greater than 95% simulant TRUs using CMPO as extractant
- To demonstrate that the MLM can remove greater than 95% simulant strontium using DtBuCH18C6 as extractant
- To develop and evaluate a radiation-resistant support membrane which can withstand up to 0.69 Wh/Kg radiation dose rate and is chemically compatible with feed, strip, and membrane liquid solutions
- To demonstrate that the MLM can effectively remove TRUs (using CMPO) and 90Sr (using DtBuCH18C6) from actual Hanford waste with > 95% removal efficiency at > 10% less extractant consumption.

Experimental testing with simulated waste solutions is to be conducted in the LSR laboratory. The recipes for the simulant feed solution are supplied by PNNL. In parallel, a radiation-resistant support membrane and moving liquid membrane module is to be developed and evaluated at LSR. Following a successful demonstration with the simulant feed solutions and the development of the support membrane and membrane module, a bench-scale MLM will be constructed for testing at PNNL using actual Hanford waste.

Approach

The Department of Energy and its contractors are developing methods to treat and dispose of HLW generated in past plutonium production operations. In the case of the Hanford tank sludge wastes, a baseline approach has been defined in which the sludges will be leached with a high caustic solution (3 M NaOH) to dissolve certain nonradioactive sludge components such as Al, Cr, and P. The leached sludges will be washed with a dilute hydroxide solution to remove most of the added caustic and the dissolved sludge components. Following removal of Cs, the leach and wash solutions will be handled as LLW. The leached sludges will contain the transuranic elements (mainly Pu and Am), 90Sr (strontium-90), and other radionuclides and thus will be handled as HLW, which will be immobilized in borosilicate glass waste form, for geologic disposal.

Using the most favorable assumptions concerning this baseline scheme, it is estimated that at least 22,800 canisters (38,000 MT) of HLW glass will be generated in the processing of the Hanford tank sludges. Using an estimate of \$250,000/canister for production and disposal costs, the cost of processing and disposing of the Hanford tank sludges would be ~ \$5B. This is viewed to be a conservative cost estimate. Furthermore, repository space is limited. There might not be room for 22,800 canisters in the repository. Clearly, there is an incentive to reduce the volume of HLW glass beyond that obtained by implementing the baseline process.

It has been suggested that implementing advanced processes to separate the radioactive components of the Hanford tank sludges from the nonradioactive components could reduce the number of HLW glass canisters to as few as 1,000^[1]. The key radionuclides requiring separation from the Hanford tank sludges are the transuranic elements and 90Sr. Two chemical processes have been investigated recently for achieving these separations: the transuranic extraction (TRUEX) process and the strontium extraction (SREX) process^[2-6]. The TRUEX process involves the extraction of TRUs from nitric acid using octyl(phenyl)-diisobutylcarbamoylmethyl phosphine oxide (CMPO). In the SREX process, Sr is extracted with di-t-butylcyclohexano-18-crown-6 (DtBuCH18C6). Both the TRUEX and SREX processes were originally intended to be implemented using traditional solvent extraction equipment such as centrifugal contactors. However, the chemistry has been applied in the mode of extraction chromatography, mainly for the purpose of analytical sample preparation.

In this project, a unique Moving Liquid Membrane system (MLM) is used to implement the TRUEX and SREX processes. The MLM has been under development at LSR Technologies since 1992. Its feasibility has been demonstrated in nonradioactive environments for gas separation and heavy metal removal. In this project, the proposed work will focus on the development of the MLM for the TRUEX and SREX processes.

Project Description

In Phase I, LSR developed a radiation-resistant membrane and module, developed a predictive model with scale up procedures, optimized the operating parameters, and demonstrated the effectiveness of the MLM apparatus for TRUEX and SREX processing using simulant (non radioactive) waste solutions. The results demonstrated that the process has high separation efficiency, low extractant consumption, and ease of process control and operation.

The Phase I research results can be summarized as follows. Numerous test runs under various experimental conditions were completed, and the results were used to identify optimum conditions. The major accomplishments included:

- Achieved > 97% removal of both TRUs and Sr with no extractant loss.
- Tested 4 membranes types of different thicknesses.
- Found several membrane types that withstand radiation and chemical composition of TRUEX and SREX.
- Developed a predictive model for design of experiments and scale up.
- Experimental data were found to be in excellent agreement with model.

Analysis of the results leads to the following conclusions:

- There is no cross-contamination between feed, extractant, and stripping solutions.
- Predictions of the model are in excellent agreement with experimental data and experimental data are highly reproducible.
- The MLM separates TRU and Sr with high removal efficiency and selectivity.
- Combination of three streams in one unit enables continuous operation, makes control and scale-up easier.
- The configuration is completely stable.
- MLM is flexible and adaptable to different separation requirements and applications.
- It can be designed with a high mass transfer surface area within a small volume, uses much smaller amounts of extractant than solvent extraction processes.
- The optimized MLM has a small footprint size.
- Although the original proposal focused on the treatment of Hanford sludges, the process and apparatus can be readily applied in other sites, most notably Idaho National Engineering and Environmental Laboratory.

In Phase II, a MLM module is to be shipped to Hanford, Washington where it will be tested with actual radioactive tank waste. Under a subcontract to LSR, Battelle PNNL will oversee a series of tests using the TRUEX/SREX processes with actual waste.

Results

For the MLM to be suitable for the TRUEX and SREX processes, both the support membrane and other components in the module must be radiation-stable and compatible with the streams involved. In the first task, efforts were focused on: (1) identification and evaluation of commercially available membranes for their applicability, (2) identification and evaluation of new (radiation-resistant) membrane material and its fabricability into a desirable support membrane. Each of the efforts is briefly described below.

Most polymeric materials undergo free radical reactions when irradiated, leading to either chain scission or crosslinking. Therefore, polymers with limited free radical reactions will exhibit radiation stability. Amorphous and semicrystalline aromatic polymers with high glass transition temperatures fall into this group and show the greatest resistance to radiation.

Thus, initial efforts focused on the identification of commercially available porous membranes with potential radiation resistance, followed by their evaluation using tensilometer elongation testing of fresh and irradiated polymer samples. Polysulfone (PSU), poly(ethersulfone) (PES) and poly(vinylidene fluoride) (PVDF) show high resistance. These membranes are commercially available and were selected for initial evaluation with PEEK membranes while better alternatives are sought. The object of the elongation at break test is to determine by what percentage a sample of plastic film will stretch before it breaks. This is perhaps the simplest to measure of the numerous mechanical properties which will be adversely affected by the chemical degradation of the polymer chains induced by irradiation. The elongation at break of sample "dogbones" was measured for both untreated samples and samples irradiated to varying total dosage levels, to determine the radiation-resistance threshold of the various candidate plastics. From this information, a suitable membrane material for use in the TRUEX process was chosen.

Before tests, the dogbone samples were checked to be free from obvious defects. The length of the neck region on the dogbone, as given by L_N in Figure 1. The final distance separating the clamps was measured as indicated by L_f in Figure 2. Each test was performed at least three times on each sample material. The elongation at break, *E*, is

$$\mathsf{E} = \frac{(\mathsf{L}_{\mathsf{f}} - \mathsf{L}_{\mathsf{O}})}{\mathsf{L}_{\mathsf{N}}} *100\%$$

In this equation, the L_N refers to the original length of the neck, while $(L_f - L_o)$ is the incremental length by which the neck was stretched. The results of the elongation tests are summarized in Figure 3. They indicate that MLM can withstand radiation which exceeds that expected for 1-3 years of irradiation by Hanford sludges without replacement of polymer membranes.

A second major milestone of this project was to construct an experimental test loop for the expected operational requirements of the MLM and separation demands of high-level waste pretreatment processes. The test loop included two plate-and-frame modules and a flexible control system for testing under both TRUEX and SREX process conditions. A schematic of the test loop is presented in the Figure 4.

A plate-and-frame membrane module was used for testing due to its flexibility and ease of modifying. In order to determine the compatibility of the porous support membrane immobilizing reaction interfaces with the hydrophilic and hydrophobic phases, a series of membrane break through tests were performed for different membrane materials, membrane thickness, pore size, prewet solutions, and challenge solutions. The results of break through testing showed that the PVDF membrane is compatible with the extractant solvents and with the acidic aqueous solutions. Upon the completion of the test loop setup, a series of shakedown tests were performed to evaluate the function of each component and identify necessary modifications.

A test plan was formulated which addressed various operating conditions, including composition of feed, stripping and membrane solutions, support membrane material in the MLM module, and operating conditions of the MLM (i.e., velocities of fluids in each channel, and thickness of the membrane liquid, operating pressure, etc.)

In the simulant feed solution, non-radioactive europium was used to simulate americium. No attempt was made to simulate plutonium (Pu) since extraction and stripping of Pu is much easier than Eu(III) or Am(III). In other words, Am(III) is the limiting species in the efficiency of the TRUEX process, and development efforts were focused on the removal of Eu(III), a simulant for Am (III). Cold Sr was used to simulate 90Sr.

The testing focused on measurement of TRU and Sr permeabilities, and their removal capacity and concentration factor through the MLM. System stability under various process conditions was also evaluated. Experiments were divided into two groups: (a) TRU extraction testing, and (b) Sr extraction testing. Selected experimental results are summarized in Table 1. The results indicate that MLM efficiently removes more than 97% of both Sr and Eu from the

simulant solutions. Figure 5 presents typical experimental kinetics of MLM-based removal of Eu and compares this data with the LSR performance model.

Application

This project utilizes an improved chemical reactor to effect the separation of HLW. The MLM is an alternative processing method to conventional solvent extraction techniques. The solvent extraction process is traditionally accomplished by dispersing an immiscible phase as droplets in another phase. This creates a large interfacial area and increases the rate considerably. After the extraction is over, the phases are separated and the Insert dispersed phase is coalesced. There are two general categories of equipment for solvent extraction. A mixer-settler arrangement provides a single equilibrium stage; a connected series can provide multistage extraction. Continuous countercurrent contacting equipment either in the form of columns or centrifugal devices can generate the equivalent of many stages in one device.

Regardless of the type of dispersion-based contracting devices used, conventional solvent extraction equipment has many disadvantages including the need for dispersion and coalescence; problems of emulsification and incomplete phase separation; flooding and loading limits in continuous countercurrent devices; the need for density difference between the phases; and the associated chemical loss between phases. A number of newly developed non-dispersive extraction devices appear to eliminate all the problems mentioned above in addition to providing very high mass transfer rates. These devices include: (1) membrane-based solvent extractor ^[7-9], (2) supported liquid membranes ^[10-15], (3) hollow-fiber contained liquid membranes ^[11], and (4) moving liquid membranes ^[16,17].

The supported liquid membrane system offers the following advantages: it is a nondispersive process, yet offers a high mass transfer surface area for extraction. The nondispersive feature minimizes the solvent (membrane phase in the pores) loss to its dissolution into the aqueous phases. In addition, the SLM process combines extraction and stripping into a single unit, thereby eliminating the equilibrium limitations experienced in a conventional two-step extraction and stripping process, and simplifying the extraction/stripping process and its operation ^[15]. However, the SLM configuration is unstable due to the dissolution of the limited membrane phase into both aqueous feed and stripping flows. A number of techniques have been investigated to reduce the dissolution rate, such as using ion-exchange membranes or gel type membrane phases. Despite continuing efforts to remedy this shortcoming, membrane life span remains the major obstacle to the industrial application of this configuration.

An innovative supported liquid membrane configuration, called a Moving Liquid Membrane (MLM) has been tested and extensively investigated. It is a module with two independent streams, one stream carries the feed stream, and the other contains the stripping solution. The membrane liquid is held within the interstice between the two streams. The advantage of this configuration is that the membrane liquid is replenishable and therefore more stable than in a conventional supported liquid membrane. It has the added advantage of drastically reducing the quantity of extractant required, which takes on added importance in radionuclide processing.

Future Activities

In Phase II, a MLM module will be shipped to Hanford, Washington where it will be tested with actual radioactive tank waste. Under a subcontract to LSR, Battelle PNNL will oversee a series of tests using the TRUEX/SREX processing. The present status of the joint LSR - PNNL

efforts and readiness for Phase II can be summarized as follows:

- LSR and BATTELLE/PNNL have concluded that from test results the MLM is a promising alternative to solvent extraction technologies, and are confident in the results with actual waste.
- PNNL has obtained a sample of actual waste containing TRU's for Phase II testing.
- PNNL has reserved and allocated a hot cell for testing, obtained instrumentation and equipment, and is ready to perform hot cell tests.
- The MLM module for the actual waste tests will be produced by LSR and have parameters similar to those employed in Phase I.
- A detailed Project Management Plan has been prepared for Phase II testing which includes all necessary permits, licenses, and requirements for environmental handling and disposal.

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Figure 1 Starting position of sample and clamps for elongation test



Figure 2 Position of sample and clamps at the moment of breakage

Yield Strain as a Function of Irradiation (2 Mil Thickness)



Figure 3. Results of tensilometer tests of radiation resistance of commercially available membranes



Figure 4. Schematic of Experimental Test Loop

| Table 1 | MLM Test Summary | |
|---------|------------------|--|
|---------|------------------|--|

| | Experimental Condition | | | | | | Results | | | | Experimental Result | | | | | |
|-----|------------------------|--------|----------|-----------|-------|------|-------------|-------------------|------------|------|---------------------|------|------|------------|--------------|--------------|
| Run | Α | В | С | D | Ε | F | G | Soln' Composition | | [Fi] | [Ff] | [Si] | [Sf] | Efficiency | Extraction R | ate mg/cm2/s |
| # | ml/min. | ml/min | .ml/min. | | | | EXTR/TBP | Feed/HNO3 | Strip/HNO3 | ppm | ppm | ppm | ppm | [F] | [F] | [S] |
| | | | | | | | | | | | | | | | | |
| 8 | 199.92 | 40.35 | 182.08 | PVDF/Dod. | u-d-u | Dod. | 0.2M/1.4M | Eu/1.0M | 0.2M | 54.0 | 23.0 | 1.8 | 0 | 57% | 1.11E-05 | 3.67E-07 |
| 9 | 199.56 | 41.06 | 206.93 | PVDF/Dod. | u-d-u | Dod. | 0.2M/1.4M | Eu/1.0M | 0.2M | 41.8 | 8.96 | 1.2 | 6.2 | 79% | 5.39E-06 | 8.20E-07 |
| 10 | 291.38 | 47.17 | 286.25 | PVDF/Dod. | u-d-u | Dod. | 0.2M/1.4M | Eu/1.0M | 0.2M | 39.4 | 10.2 | 7.2 | 19.2 | 74% | 4.96E-06 | 2.09E-06 |
| 11 | 291.38 | 40.07 | 294.96 | PVDF/Dod. | u-d-u | Dod. | 0.05M/0.35M | Eu/1.0M | 0.2M | 223 | 132 | 13 | 16.4 | 41% | 1.55E-05 | 5.92E-07 |
| 12 | 315.98 | 38.63 | 307.78 | PVDF/Dod. | u-d-u | Dod. | 0.2M/1.4M | Eu/1.0M | 0.2M | 211 | 55.8 | 16 | 21.2 | 74% | 2.64E-05 | 8.70E-07 |
| 13 | 317.00 | 39.24 | 163.18 | PVDF/Dod. | u-d-u | Dod. | 0.2M/1.4M | Eu/1.0M | 0.2M | 138 | 36.5 | 21 | 23.6 | 73% | 1.94E-05 | 5.74E-07 |
| 14 | 317.00 | 44.86 | 179.17 | PVDF/Dod. | u-d-u | Dod. | 0.2M/1.4M | Eu/1.0M | 0.2M | 83.4 | 14.4 | 0 | 28.6 | 83% | 9.78E-06 | 4.10E-06 |
| 15 | 176.00 | 38.89 | 177.90 | PVDF/Dod. | u-u-u | Dod. | 0.2M/1.4M | Eu/1.0M | 0.04M | 47.8 | 17.6 | 0 | 27.0 | 63% | 3.85E-06 | 3.52E-06 |
| 16 | 176.00 | 38.89 | 177.90 | PVDF/Dod. | u-u-u | Dod. | 0.2M/1.4M | Eu/1.0M | 0.02M | 18.8 | 5.7 | 1.8 | 46.6 | 70% | 9.28E-07 | 3.25E-06 |
| 17 | 184.41 | 40.81 | 180.61 | PVDF/Dod. | u-u-u | Dod. | 0.2M/1.4M | Eu/1.0M | 0.02M | 302 | 83.8 | 2.8 | 72.8 | 72% | 1.50E-05 | 4.93E-06 |
| 18 | 191.58 | 40.55 | 158.69 | PVDF/Dod. | u-u-u | Dod. | 0.2M/1.4M | Eu/1.0M | 0.002M | 174 | 28.7 | 1.8 | 119 | 83% | 5.71E-06 | 4.71E-06 |
| 19 | 195.00 | 40.64 | 186.98 | PVDF/Dod. | u-u-u | Dod. | 0.5M/1.4M | Eu/1.0M | 0.05M | 162 | 5.2 | 3.2 | 19 | 97% | 5.44E-06 | 5.46E-07 |
| 20‡ | 195.00 | 42.09 | 195.00 | PVDF/Dod. | u-d-u | Dod. | 0.5M/1.4M | Eu/1.0M | 0.02M | 236 | 117 | 0.5 | 63 | 79% | 5.39E-06 | 8.20E-07 |
| 22‡ | 173.72 | 40.81 | 186.26 | PVDF/Dod. | u-d-u | Dod. | 0.5M/1.4M | Eu/1.0M | 0.002M | 236 | 6.4 | 0.5 | 56 | 97% | 3.38E-06 | 8.35E-07 |
| 23 | 170.93 | 43.70 | 169.67 | PVDF/Oct. | u-d-u | Oct. | 0.2M | Sr/3.0M | 0.004M | 168 | 36.1 | 0 | 0.16 | 79% | 5.25E-05 | 8.12E-08 |
| 24 | 282.66 | 48.22 | 285.74 | PVDF/Oct. | u-u-u | Oct. | 0.2M | Sr/3.0M | 0.5M | 17 | 9.3 | 0 | 1.52 | 45% | 2.04E-06 | 5.80E-07 |
| 28 | 195.00 | 47.60 | 176.54 | PVDF/Oct. | u-u-u | Oct. | 0.2M | Sr/2.0M | 1 M | 276 | 7.4 | 1.6 | 13 | 97% | 3.56E-05 | 2.13E-06 |
| 29 | 179.26 | 47.78 | 172.20 | PVDF/ISO. | u-u-u | ISO. | 0.2M | Sr/3.0M | 0.0M | 181 | 31.2 | 0 | 8.58 | 83% | 6.60E-06 | 5.50E-07 |
| 30‡ | 176.35 | 43.74 | 214.75 | PVDF/ISO. | u-u-u | ISO. | 0.2M | Sr/3.0M | 0.01M* | 47 | 1.3 | 0.1 | 9.9 | 97% | 6.77E-07 | 1.50E-07 |
| 31‡ | 314.27 | 48.11 | 316.54 | PVDF/ISO. | u-u-u | ISO. | 0.2M | Sr/3.0M | 0.1M* | 211 | 55.8 | 16 | 21.2 | 74% | 2.64E-05 | 8.70E-07 |

Note A-Feed flowrate; B-MLM flowrate; C-Strip flowrate, D-Support/prewet; E-Flow arrangement: u-u-u: All streams - same direction;

u-d-u: Feed & Strip - same direction, MLM - opposite; F-MLM dilute, G-Extractant concentration

* 0.1M HNO3 and 0.25M HEDPA; ‡ experiment with real simulate



Figure 5. Typical time dependencies of feed and strip concentrations