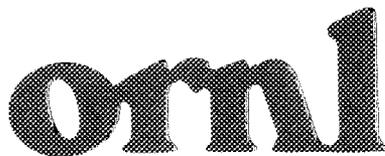




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Results of Pilot-Scale Testing of Electrochemical Processes To Eliminate Production of Liquid Low-Level Waste at ORNL's Process Waste Treatment Plant

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ORNL/TM-13589

Chemical Technology Division

**RESULTS OF PILOT-SCALE TESTING OF ELECTROCHEMICAL PROCESSES TO
ELIMINATE PRODUCTION OF LIQUID LOW-LEVEL WASTE AT ORNL'S
PROCESS WASTE TREATMENT PLANT**

P. A. Taylor, T. E. Kent, D. R. McTaggart, and J. D. Hewitt

Date Published—May 1998

Prepared by the
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831-6285
managed by
LOCKHEED MARTIN ENERGY RESEARCH CORP.
for the
U.S. DEPARTMENT OF ENERGY
under contract DE-AC05-96OR22464



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P. A. Taylor, T. E. Kent, D. R. McTaggart, and J. D. Hewitt

ABSTRACT

The Mixed Waste Focus Area of the U.S. Department of Energy's Office of Science and Technology funded a pilot-scale test of electrochemical processes that could eliminate one of the largest sources of liquid low-level waste (LLLW) at Oak Ridge National Laboratory (ORNL). The Process Waste Treatment Plant (PWTP), which treats 2.7×10^8 L/year of slightly contaminated wastewater, currently produces approximately 40% of the total annual volume of LLLW at ORNL, but contributes less than 1% of the radioactivity. Two electrochemical processes were tested: electrochemical ion exchange and electrodialysis. Both technologies were developed by AEA Technology (AEAT, Harwell, United Kingdom). AEAT conducted laboratory-scale tests and built the pilot-scale unit that was used at ORNL. AEAT will use the data from the pilot-scale tests to evaluate the cost of a full-scale system for the PWTP. A report will be issued later.

The electrochemical ion-exchange process had a low electrical efficiency when regenerating the ion-exchange resin currently used at the PWTP (Dowex HCR-S). The efficiency of regeneration could be improved by using alternative ion-exchange resins, but the loading capacity of these resins was lower, which would increase the number of regenerations required. An electrodialysis process was tested to recycle the nitric acid that is currently used to regenerate the ion-exchange resin at the PWTP. Two cycles of electrodialysis could produce 2.7 M nitric acid from the regenerant solution. The majority of the contaminants in the regenerant solution (Ca, Mg, and Sr) were precipitated inside the cathode compartment of the electrodialysis system, rather than being removed in the catholyte solution. The equipment would need to be modified so that these contaminants would be continuously removed from the system on a continuous basis.

1. INTRODUCTION

Mixed [radioactive and Resource Conservation and Recovery Act (RCRA)-hazardous] liquid wastes are produced at the Process Waste Treatment Plant (PWTP) at Oak Ridge National Laboratory (ORNL) as a result of an ion-exchange process designed to remove radioactive strontium from 2.7×10^8 L/year of ORNL process wastewater. The ion-exchange process creates over 19,000 L/year of a concentrated, radioactive, nitric acid-based ion-exchange regenerant

solution, which contains Sr, Ca, Mg, Cr, and Ni salts, along with radioactive ^{90}Sr and ^{137}Cs . The concentrated regenerant solution currently produced is discharged to the liquid-low-level waste (LLLW) system, where it is mixed with other high-activity and transuranic mixed waste streams for storage and eventual disposal at the Waste Isolation Pilot Plant or the Nevada Test Site. The PWTP currently produces approximately 40% of the total annual volume of LLLW at ORNL, but it contributes less than 1% of the radioactivity. Eliminating this waste stream might make the remaining, currently generated LLLW non-RCRA hazardous by eliminating heavy-metal inputs.

A schematic of the PWTP flowsheet is shown in Fig.1. The plant consists of a softener/clarifier to precipitate water-hardness compounds and ^{90}Sr , followed by ion exchange to polish the wastewater in order to meet the ^{90}Sr discharge limit. ORNL process wastewater is chemically similar to groundwater and contains water hardness compounds such as calcium and magnesium bicarbonates along with small amounts of ^{90}Sr and ^{137}Cs , typically 1200 and 300 Bq/L, respectively. The softening process precipitates 90% of the water-hardness compounds, about 80% of the ^{90}Sr , and about 20% of the ^{137}Cs into a low-level solid waste form that is not characteristically hazardous. The ion-exchange columns contain a strong-acid cation resin (Dowex HCR-S). The columns are operated until initial breakthrough of Ca or Mg, and are then regenerated using two batches of 2.7 M nitric acid. The second batch is fresh nitric acid, and the first batch is recycle acid, which was used as the second batch during the previous regeneration. The acid that has been used twice is sent to an evaporator, where most of the free nitric acid is recovered for reuse at the PWTP. The concentrate stream, which contains the Ca, Mg and Sr removed from the ion-exchange columns, and chromium and nickel corrosion products from the evaporator, enters the ORNL LLLW system.

The Mixed Waste Focus Area of DOE's Office of Science and Technology funded a pilot-scale test of two electrochemical processes that could eliminate the production of LLLW at the PWTP. The processes were developed by AEA Technology (AEAT) (Harwell, UK). AEAT conducted lab-scale tests and built a pilot-scale unit that was tested at ORNL.

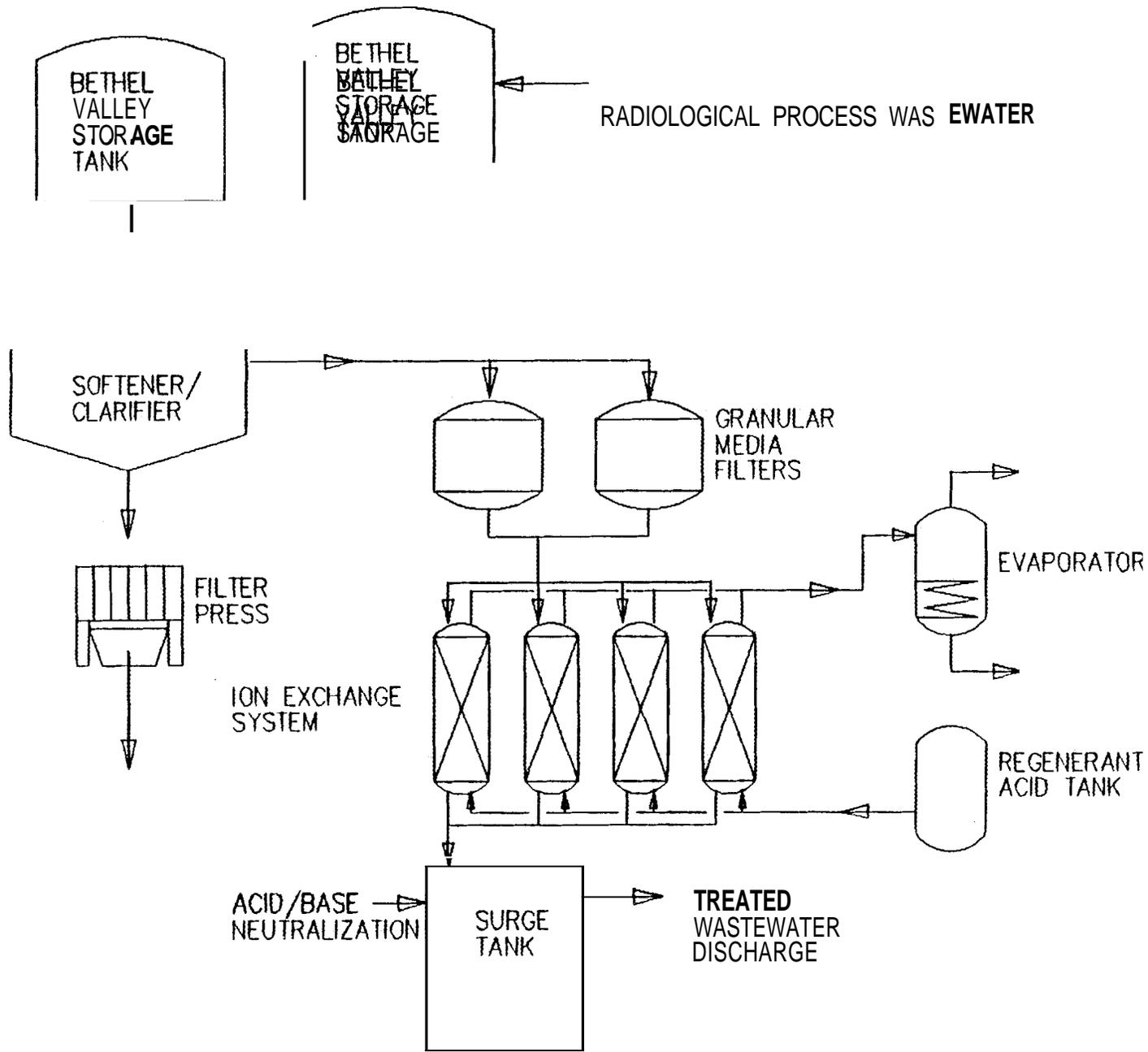


Figure 1. Flow Diagram of the ORNL Process Waste Treatment Plant

2. DESCRIPTION OF EXPERIMENTAL RESULTS

2.1 ELECTROCHEMICAL ION EXCHANGE

Description: The first process recommended by AEAT was electrochemical ion exchange (EIX). In this process the ion-exchange resin is contained within the electrochemical cell. The wastewater is treated the same as at the existing PWTP by softening and ion-exchange. The electrochemical cell is used for regenerating the ion-exchange resin instead of conventional regeneration with nitric acid. A diagram illustrating the EIX regeneration process is shown in Fig. 2. While treating the wastewater, the EIX system operates like a standard ion-exchange column. Wastewater is pumped down through the center compartment of the EIX system, which contains the ion-exchange resin. When the ion-exchange resin becomes loaded with Ca, Mg, and Sr cations from treatment of the wastewater, the loaded resin cell is taken off-line for regeneration.

An electrical potential is applied across the resin cell, which includes the anode compartment, cathode compartment, and the center resin bed. The electrical potential ionizes water and produces free hydrogen ions in the anode compartment of the cell. The anolyte stream is a dilute nitric acid solution that is recirculated in a closed loop within the cell. The hydrogen ions generated in the anolyte migrate through a cation membrane into the resin compartment. The hydrogen ions displace the metals (mainly Ca, Mg, and Sr) from the resin, which then pass through a cation membrane to an ion-exchange membrane, which then releases the metals to the deionized water catholyte stream. The ion-exchange membrane was developed by AEAT to reduce the formation of calcium or magnesium hydroxide deposits on the cation membrane by immediately sorbing the cations that pass through the cation membrane, and then slowly releasing them to the catholyte stream. Because the catholyte stream is relatively dilute and

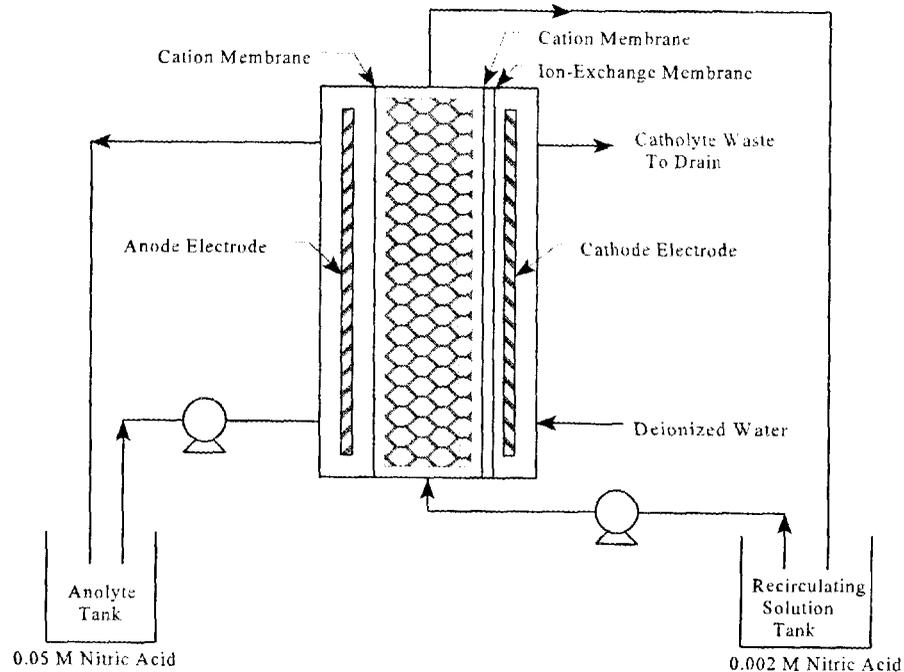


Figure 2. Flow Diagram of Electrochemical Ion-Exchange System During Regeneration of Resin

contains low nitrate, it can be recycled to the PWTP process wastewater feed system for treatment. A dilute nitric acid solution is recirculated up through the resin bed to provide electrical conductivity for the electrochemical process and to keep the calcium or magnesium hydroxides from coating the resin beads.

Results: The EIX System was assembled and installed in the Wastewater Treatment Test Facility at ORNL on June 2, 1997, under the direction of Peter Griffiths of AEAT. The center compartment contained 5 L of Dowex HCR-S cation-exchange resin, the resin currently used at the PWTP. The resin in the system was partially loaded with calcium and magnesium, using tap water as a nonradioactive surrogate for process wastewater. The system treated 1200 L of water over a period of 20.5 h, removing 46.7 g Ca and 11.0 g Mg from the water. The concentration of calcium and magnesium in the treated water was less than 0.5 mg/L.

The first regeneration attempt was performed on June 4, 1997. The resin was rinsed with deionized water to remove any carbonate present in the tap water. A solution of 0.002 *M* nitric acid was recirculated up through the resin bed. A solution of 0.05 *M* nitric acid was recirculated through the anolyte compartment, and deionized water was pumped through the catholyte compartment to the building drain at a flow rate of 100 mL/min. A potential of 60 V was applied between the anode and cathode electrodes, with a maximum current setting of 8 A. Initially, no current was flowing through the system. We noticed that the catholyte membrane was bulging in toward the resin, thus losing electrical contact with the cathode electrodes. The backpressure in the resin compartment was increased by increasing the height of the exit line of the recirculating solution. The current slowly increased, reaching 7.6 A after 9 h of operation, at which time the regeneration was terminated. The concentration of calcium and magnesium in the catholyte was below 0.5 mg/L during the entire regeneration attempt. It was theorized that the ion-exchange membrane installed next to the catholyte membrane retained all of the calcium and magnesium eluted from the resin.

The resin was loaded with an additional 93.4 g Ca and 22.2 g Mg by treating 2400 L of tap water, at which time initial breakthrough of calcium and magnesium was detected in the effluent. (Note: the ion-exchange columns at the PWTP are regenerated when calcium or magnesium is first detected in the effluent.) The resin was regenerated electrochemically, using the same procedures as before. The current increased steadily to the maximum of 8.0 A after 20 h of operation, and then remained constant. After 29 h of operation, the system was shut down and disassembled to fix a leak in the catholyte membrane. The catholyte membrane was distorted and discolored, and the ion-exchange membrane appeared to have melted in places and was stuck to the electrodes. Both membranes were replaced, and the regeneration was started again. The current immediately increased to 8.0 A and remained constant for an additional 75 h of regeneration. The regeneration took considerably longer than the original goal of 48 h. A total of 695 L of deionized water was pumped through the catholyte chamber. The maximum concentration of calcium and magnesium in the catholyte was 195 and 2.7 mg/L respectively, which occurred after 20 hrs of operation. A total of 32.5 g of Ca and 0.41 g of Mg was removed

from the system in the catholyte stream during regeneration—a recovery of 34.8% and 1.8%, respectively, of what was originally loaded on the resin. The amount retained by the ion-exchange membrane is not accounted for in this calculation; therefore, the actual amount eluted from the resin should be higher.

AEAT determined that the discoloration and wrinkling observed in the catholyte membrane, which eventually led to a break in the membrane, was caused by the precipitation of magnesium hydroxide within the membrane. This determination is consistent with the chemical analysis results during regeneration, which showed a very low concentration of magnesium in the catholyte. A subsequent loading cycle using tap water showed that the resin was about 50% regenerated; the remaining calcium and magnesium, which was removed from the ion-exchange resin during the regeneration, must have been retained in the ion-exchange membrane, which is adjacent to the catholyte membrane. The efficiency of the regeneration, in terms of electrons input vs calcium and magnesium removed from the resin, was only about 10%. Laboratory-scale tests at AEAT showed that the membrane damage could be prevented by adding carbon dioxide gas to the catholyte solution, which lowers the pH and increases the solubility of the magnesium and calcium. The efficiency of the electrochemical regeneration could be improved by using other cation-exchange resins; however, the ^{90}Sr loading capacity of these resins was lower than for the Dowex HCR-S, which would increase the number of regenerations required. Because of the low efficiency of the EIX process, AEAT recommended that we test other electrochemical processes to achieve the goal of eliminating the LLLW generated at the PWTP.

2.2 ELECTRODIALYSIS

Description: Because of the low efficiency of the EIX process, AEAT recommended testing an electrodialysis (ED) process to recycle the nitric acid that is currently used to regenerate the ion-exchange resin at the PWTP. Figure 3 shows a process flow diagram of the ED system. The nitric acid regenerant solution from the PWTP is recirculated through the central compartment of

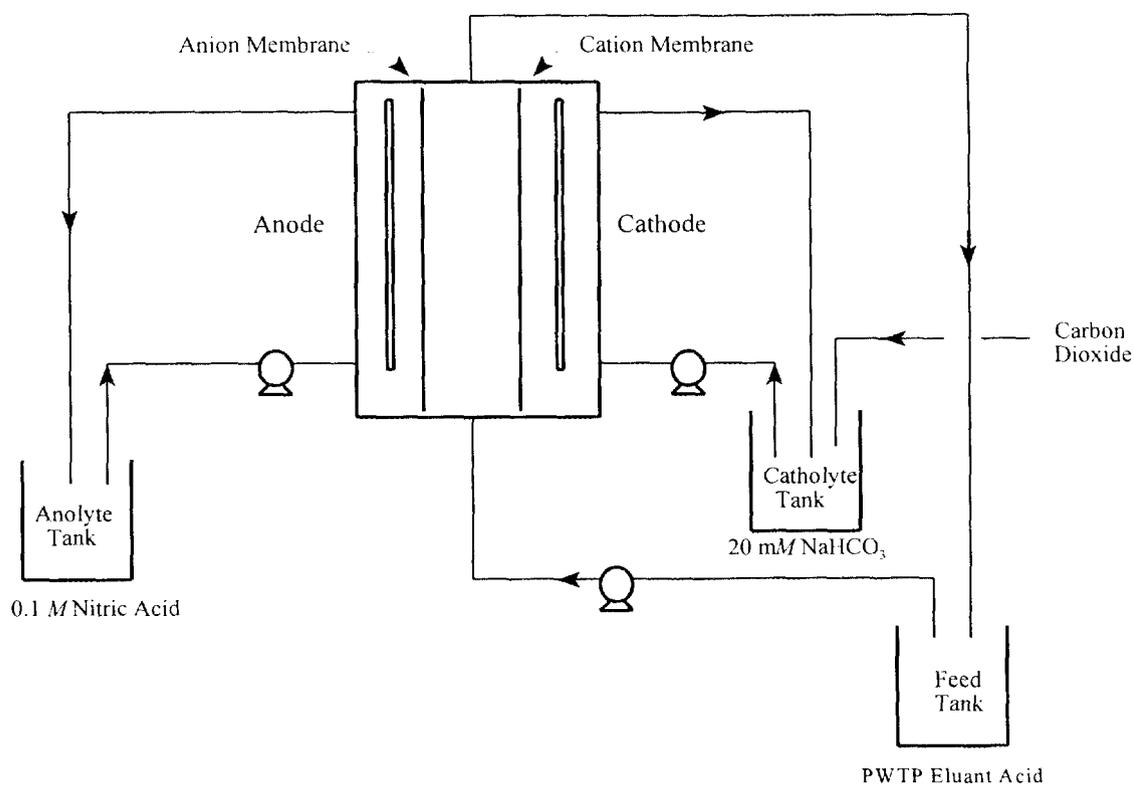


Figure 3. Flow Diagram of the Pilot-Scale Electrodialysis System

the ED system. An electrical potential is applied across the cell, which is divided into three compartments (anode, central, and cathode) with membranes between the compartments. The electrical potential ionizes water to produce free hydrogen ions in the anode compartment of the cell and transports nitrates from the regenerant solution (central compartment), through an anion membrane, and into the anolyte solution. The metal ions (mainly Ca, Mg, and Sr) in the regenerant solution are transported through a cation membrane into a recirculating catholyte stream. Carbon dioxide is added continuously to the catholyte stream to keep the pH of the solution neutral and to increase the solubility of the metals. Initially, the catholyte solution contained about 20 mM NaHCO₃ to provide electrical conductivity. Carbon dioxide gas is bubbled into the catholyte reservoir to solubilize the Ca, Mg, and Sr ions that are transported into the catholyte. The anolyte solution initially contained 0.1M HNO₃, and the acid concentration

increases as nitrate ions are transported into the anolyte and protons are generated electrolytically. The regenerant solution is depleted in Ca, Mg, Sr, and nitrate, and will end up as moderately deionized water, which can be treated through the PWTP, along with the catholyte stream. The existing PWTP chemical precipitation process (softener/clarifier) will precipitate the metals from the catholyte stream and separate the resultant sludge. The anolyte stream increases in nitric acid concentration as nitrate ions are transported into the anode compartment and protons are generated electrolytically.

Results: Laboratory-scale tests using simulant solutions at AEAT showed that 1.5 *M* nitric acid could be produced in one ED stage from a simulant of the PWTP eluant acid. The cathode electrode became coated with calcium carbonate deposits, but this did not stop the current flow in the system when a stainless steel electrode was used. A second ED unit could be used to increase concentration of the 1.5 *M* acid to 2.7 *M*, which is the concentration used for regeneration at the PWTP.

The pilot-scale EIX system was reconfigured to an ED system by (1) removing the ion-exchange resin, (2) using an anion membrane for the anode compartment, and (3) using a solid stainless steel electrode in the cathode compartment. A nonradioactive simulant solution was produced by regenerating 5 L of cation-exchange resin (loaded with calcium and magnesium from tap water) using 2.7 *M* nitric acid. The solution contained 6600 mg/L Ca, 2260 mg/L Mg, 567 mg/L Na, and 58 mg/L Zn. The simulant feed solution was deionized using the ED system, producing 10 L of 1.15 *M* nitric acid anolyte solution, which contained 210 mg/L Ca, 69 mg/L Mg, 33 mg/L Na, and 3 mg/L Zn. The catholyte solution contained 483 mg/L Ca, 1099 mg/L Mg, 1050 mg/L Na and 6 mg/L Zn dissolved in the solution, and a considerable amount of suspended solids. The system operated for 19.5 h at a current of 40 A to deionize 6.8 L of simulant solution. Figure 4 shows a graph of the conductivities (a measure of the dissolved ion concentration) of the feed and anolyte solutions during the run.

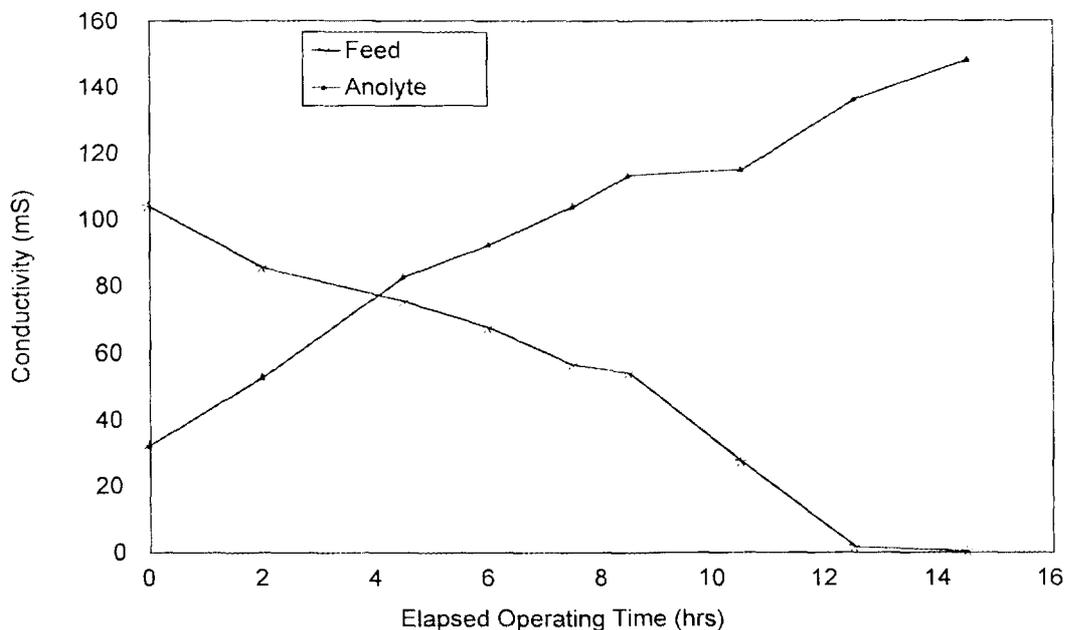


Figure 4. Conductivity of Feed and Anolyte Solutions During Electro dialysis Treatment of Simulant Waste.

Chilled water was recirculated through cooling coils in the holding tanks to keep the solution temperatures below the recommended maximum of 50°C. Nitrate ions were detected in the catholyte solution after running for 4 h. The ED system was disassembled to check for leaks. No leaks were visible, the system was reassembled using a new cation membrane, and the catholyte solution was replaced. Nitrate was detected again after restarting the system. The nitrate concentration leveled off at about 1000 mg/L after running for several hours. Nitrite ions were also indicated in the catholyte solution, but the test strips that we used can not quantify nitrite. Because of the reducing conditions in the cathode compartment, nitrate ions entering the catholyte could be reduced to nitrite, and possibly, ammonia. These results showed that some nitrate ions can diffuse through the cation membrane. In a full-scale system, the catholyte solution would be treated in the PWTP softener, so any soluble ions in the catholyte would end up in the effluent from the PWTP. Low concentrations of ammonia, nitrite and nitrate could be tolerated in the catholyte, but high concentrations could affect discharge compliance.

Following the successful treatment of the simulant solution, a large sample of ion-exchange column regenerant solution was obtained from the PWTP. The solution contained 30 Bq/mL ^{90}Sr , 3.5 Bq/mL ^{137}Cs , 3800 mg/L Ca, 97 mg/L Mg, 1330 mg/L Na and 133,000 mg/L nitrate. Three runs were completed, each using a 6-L batch of this regenerant solution as the feed to the ED system. The catholyte solution was 10 L of 0.02 M NaHCO_3 , and the anolyte was 10 L of 0.1 M HNO_3 acid for each run. Carbon dioxide was bubbled into the catholyte tank at a rate of 11 L/h to keep the pH below 7.0. The suspended solids concentration in the catholyte was very low for all of these runs, unlike for the simulant run, which had more calcium and magnesium in the feed solution. The amount of Ca, Mg, and Sr in the catholyte solution at the end of each run was much lower than the amount in the starting feed solution, which indicates that a significant amount of these compounds was deposited inside the cathode chamber.

Run 1 lasted for 18 h at a current of 40 A. The potential across the ED cell started at 37 V, decreased to 14 V during the middle of the run, and then, as the feed became deionized, increased to 40 V at the end of the run. The deionized feed had a conductivity of 0.4 mS and a hardness of <5 mg/L. The acid concentration of the anolyte solution increased from 0.1 M to 1.4 M.

The feed acid for run 2 was pumped through the cathode compartment to dissolve any deposits from run 1. After dissolving the deposits, the concentration of contaminants in the acid increased to 36 Bq/mL ^{90}Sr , 4.0 Bq/mL ^{137}Cs , 5750 mg/L Ca, and 125 mg/L Mg. Run 2 also lasted for 18 h, and the current and voltage profiles were similar to those for run 1. The deionized feed had a conductivity of 0.59 mS, a hardness of <5 mg/L, and contaminant concentrations of <0.2 Bq/mL for ^{90}Sr and ^{137}Cs , <0.2 mg/L for Ca and Mg, and 1.5 mg/L for Na. The catholyte contained <0.2 Bq/mL ^{90}Sr , 2.1 Bq/mL ^{137}Cs , 99 mg/L Ca, 49 mg/L Mg, 1080 mg/L Na, 1140 mg/L nitrate, 55 mg/L nitrite, and 300 mg/L ammonia. The anolyte acid concentration increased from 0.1 M to 1.4 M.

The feed acid for run 3 had contaminant concentrations of 64 Bq/mL ^{90}Sr , 3.5 Bq/mL ^{137}Cs , 7160 mg/L Ca and 135 mg/L Mg after dissolving the cathode deposits from the previous run. Run 3 lasted for 15.5 h, and the contaminant concentrations in the deionized feed were similar to runs 1 and 2. The electrical efficiency of these runs averaged 60%. The results of the feed hardness titrations and anolyte acid concentration measurements are shown in Fig. 5. We obtained a new supply of nitrate test strips and an ammonia test kit prior to this run, so that the catholyte concentrations could be monitored. These results are shown in Fig. 6. These concentrations could cause an increase of 33 mg/L nitrate and 1.1 mg/L ammonia in the effluent from the PWTP if the catholyte solution from a full-scale ED system was returned to the PWTP feed tanks at one time.

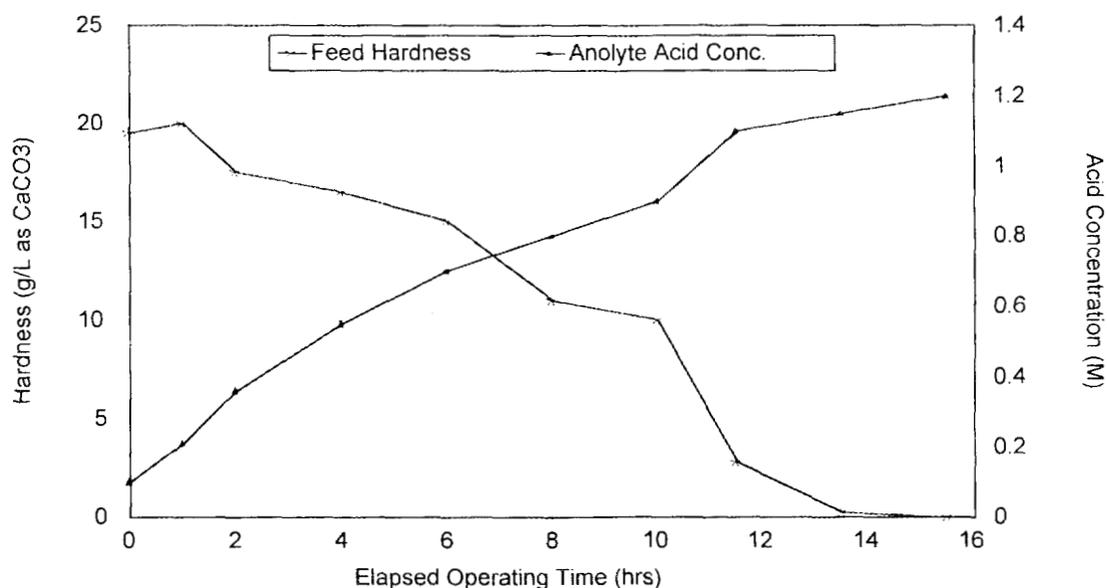


Figure 5. Feed Hardness and Anolyte Acid Concentration During Electrodialysis Treatment of PWTP Eluant Acid.

For each of the runs, most of the hardness compounds and ^{90}Sr were retained in the system as deposits in the cathode compartment. Table 1 shows a material balance for ^{90}Sr , ^{137}Cs , and hardness compounds for each run. The input column is the amount of each contaminant in the feed solution, including the amounts dissolved from the cathode. The output column is the sum

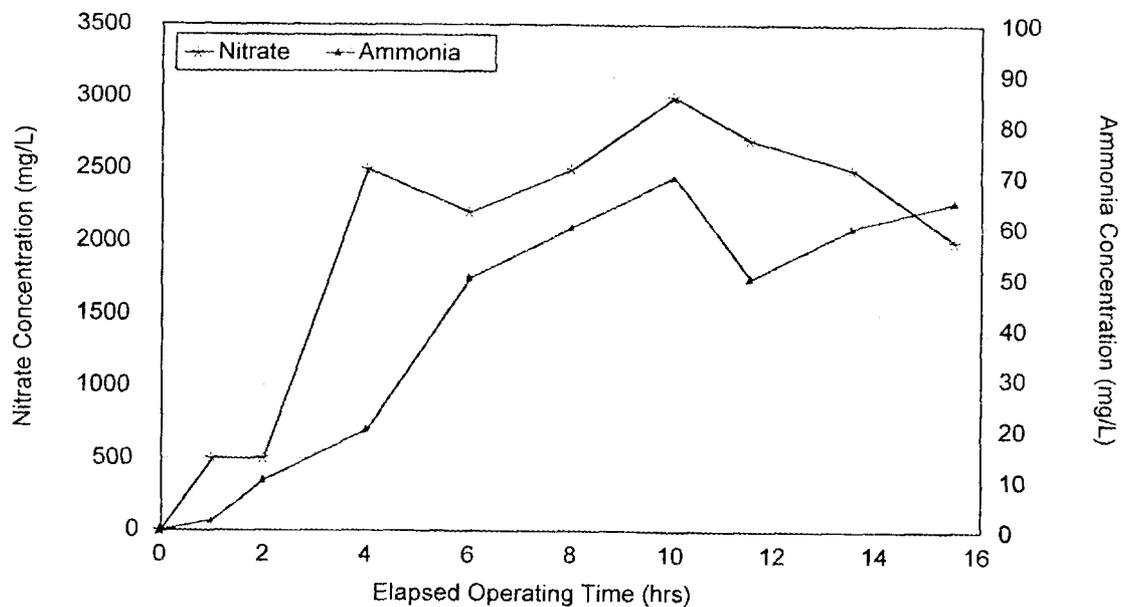


Figure 6. Nitrate and Ammonia Concentrations in Catholyte Solution During Electro dialysis Treatment of PWTP Eluant Acid

of the contaminants in the feed, catholyte, and anolyte at the end of the run. After completing three runs, 94.7% of the ^{90}Sr and 96.3% of the hardness compounds were retained inside of the ED system. Inspection of the inside of the system showed a thick layer of sludge in the bottom of the cathode compartment. The cathode compartment was rinsed with acid to dissolve the contaminants.

Table 1. Input and Output Amounts of Contaminants During Electro dialysis Runs

Contaminant	Run No. 1		Run No. 2		Run No. 3	
	Input	Output	Input	Output	Input	Output
^{90}Sr (kBq)	180	7	216	28	384	6
^{137}Cs (kBq)	21	20	20	21	21	21
Hardness (g of CaCO_3)	60	2	96	3	117	5

Anolyte acid from the three first-cycle runs (30 L) was saved and then used as feed for two second-cycle runs to increase the acid concentration to 2.7 M, which is used at the PWTP for regenerating the ion-exchange columns. For the first acid concentration run, 10 L of anolyte acid was placed in the feed tank and 9.5 L was placed in the anolyte tank. The catholyte tank was filled with 10 L of 0.1 M NaOH. The ED process transports the nitrate ions from the feed solution into the anolyte and generates protons electrolytically at the anode. The protons from the feed acid are transported into the catholyte, where they combine with hydroxide ions generated at the cathode to form water. The run lasted for 31 h at a current of 40 A. The voltage started at 8 V and slowly increased to 13.5 V. The acid concentrations of the feed and the anolyte during the run are shown in Fig. 7. The nitrate concentration in the catholyte solution increased to 600 mg/L after 12 h of operation, and then decreased to 5 mg/L at the end of the run. The ammonia concentration slowly increased to 100 mg/L after 22, and then remained constant. Water was transported from the anolyte to the feed solution by the ED process. The final volume of anolyte was 7 L, and 3.5 L of feed solution was removed during the middle part of the run to keep from overflowing the feed tank. The acid concentration in the feed solution that was removed averaged 0.38 M. The electrical efficiency of this run was only about 22.7%.

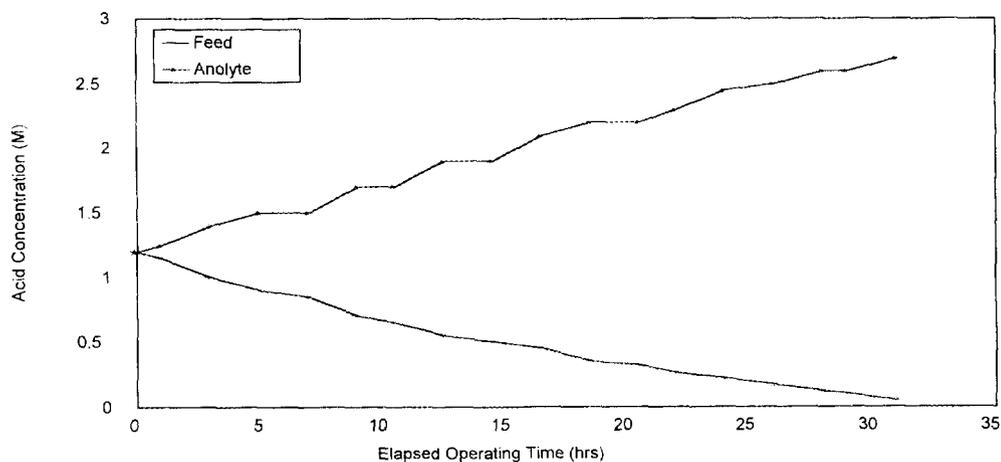


Figure 7. Feed and Anolyte Acid Concentration During Second Cycle Electrodialysis Treatment of PWTP Eluant Acid

For the second acid concentration run, 4.2 L of the first-cycle anolyte acid was used in the feed tank and 5.0 L was used in the anolyte tank. The catholyte tank contained 10 L of 0.1 M NaOH. The feed acid was reduced to <0.01 M after 9.5 h, and then 3.5 L of 0.38 M nitric acid (removed from the feed tank during the first run) was added to the feed; the run continued for an additional 6 h. The nitrate concentration in the catholyte increased to 250 mg/L and then decreased to 5 mg/L; the ammonia concentration increased to 60 mg/L. The electrical efficiency of this run was 21%. The efficiency of the process would be improved by maintaining a higher nitrate concentration in the feed solution, which would be possible if a separate electro dialysis unit was used to concentrate the first-cycle anolyte acid on a continuous basis.

3. CONCLUSIONS

Pilot-scale tests were conducted using two electrochemical processes that could eliminate production of LLLW at the PWTP. The EIX process had a low electrical efficiency when regenerating the ion-exchange resin currently used at the PWTP (Dowex HCR-S). The efficiency of regeneration could be improved by using alternative ion-exchange resins. The loading capacity of these resins was lower, however, which would increase the number of regenerations required. An ED process was tested to recycle the nitric acid that is currently used to regenerate the ion-exchange resins at the PWTP. Two cycles of electro dialysis could produce 2.7 M nitric acid from the regenerant solution. The majority of the contaminants in the regenerant solution (Ca, Mg, and Sr) were precipitated inside the cathode compartment of the electro dialysis system, rather than being removed in the catholyte solution. The equipment would need to be modified so that these contaminants would be removed from the system on a continuous basis.

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