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SALT SPLITTING USING CERAMIC MEMBRANES

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ABSTRACT

Inorganic ceramic membranes for salt splitting of radioactively contaminated sodium salt solutions are being developed for treating U.S. Department of Energy tank wastes. The process consists of electrochemical separation of sodium ions from the salt solution using sodium(Na) Super Ion Conductors (NaSICON) membranes. In contrast to conventional organic-based bipolar or ion exchange membranes used in salt splitting, NaSICON membranes are resistant to gamma/beta radiation and are highly selective for sodium ions. Potential applications include 1) caustic recycle for sludge leaching, regeneration of ion exchange resins, inhibition of corrosion in carbon steel tanks, or retrieval of tank wastes; 2) pH adjustment and reduction of competing cations to enhance cesium ion exchange processes; 3) sodium reduction in high-level waste sludges; and 4) sodium removal from acidic wastes to facilitate calcining. Initial experiments with dysprosium-based NaSICON membranes have demonstrated the feasibility of the process.

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INTRODUCTION

Nuclear materials production processes within the U.S. Department of Energy (DOE) complex produced acidic waste streams that were made highly alkaline by adding sodium hydroxide to the streams to permit their storage in carbon steel tanks. Many of the wastes have high concentrations of sodium, an element that can negatively affect separations processes such as cesium ion exchange. The sodium also contributes to large disposal volumes and can decrease the durability of glass, the leading candidate for the final waste form. Besides the large amounts of sodium already present in the wastes, many existing disposal strategies (1,2) call for adding more sodium to the wastes as the wastes are prepared for conversion to the final waste forms.

One promising approach for mitigating the impact of sodium is to separate the nonradioactive sodium from the waste using an electrochemical salt-splitting process based on inorganic ceramic membranes, as shown in Figure 1. In this process, the waste is added to the anode compartment of an electrochemical cell and an electrical potential is applied to the cell, subsequently driving sodium ions through the membrane while most other cations (e.g., K^+,Cs^+) are rejected by the membrane. The generation of H^+ from the electrolysis of water maintains the charge balance in the anode compartment. The generation of OH^-, either from electrolysis of water or from oxygen and water using a gas diffusion electrode, maintains the charge balance in the cathode. The normal gas products from the waste electrolysis are oxygen gas at the anode and hydrogen gas at the cathode. The formation of potentially flammable gas mixtures can be prevented by providing adequate volumes of a sweep gas or by using an oxygen and a gas diffusion electrode. As H^+ is generated in the anode compartment, the pH drops. Production of OH^- in the cathode compartment raises the pH as the sodium hydroxide product is recovered.

The membranes under development are from a family of materials known in the electrical battery industry as sodium (Na), super fast ionic conductors

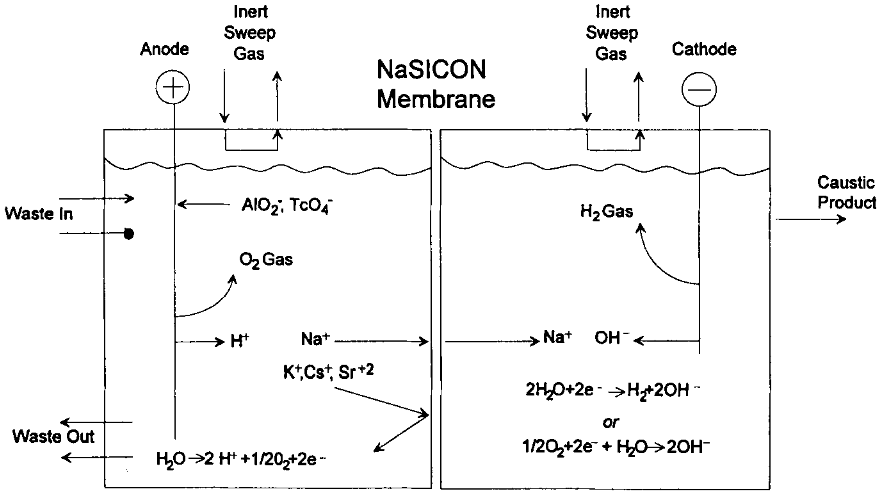

FIGURE 1. Schematic of an electrochemical process using the NaSICON membrane

(NaSICON). The unusual characteristic of NaSICON ceramics is that they possess channels within the crystal structure for fast ion conduction, as shown in Figure 2 (3). "Rings" of silica tetrahedra, which include sodium and heavy metal ions, form the structure around these channels. The channels are filled with highly mobile sodium ions.

NaSICON represents a family of materials based upon the crystal structure of compositions such as $Na_{1+x}Zr_2Si_xP_{3-x}O_{12}$ (4,5,6). This structure is known to accommodate a host of other elements in place of the Zr and P while still preserving the unique crystal structure, albeit with very small dimensional changes that can be very important in determining transport properties. The NaSICON compositions investigated were based upon rare earth (RE) ions (i.e., $Na_5RESi_4O_{12}$) where RE is dysprosium (Dy), gadolinium (Gd), and samarium (Sm).

In contrast to conventional organic-based bipolar or ion exchange membranes used in salt splitting, ceramic-based membranes are attractive because

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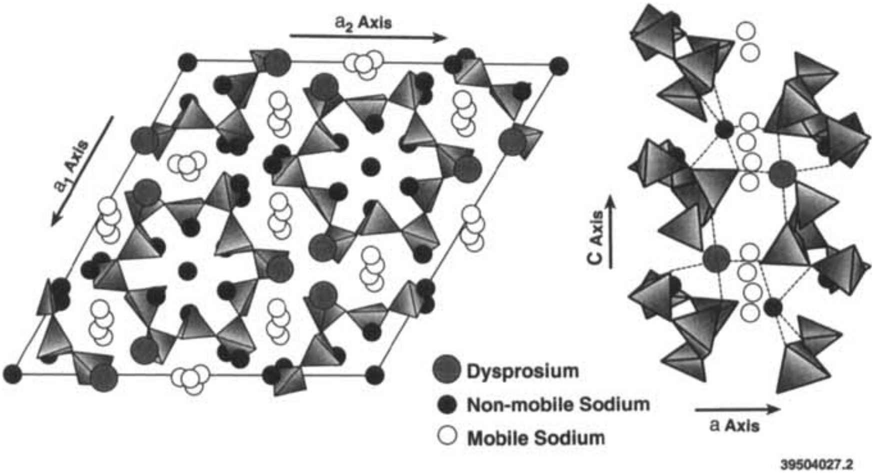

FIGURE 2. NaSICON structure controls sodium transport

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they are much more resistant to gamma/beta radiation and are highly selective for sodium ions. High selectivity for sodium ions is useful for minimizing the transport of fission products (e.g., ^{137}Cs), minimizing the transport of nonradioactive (e.g., K^+) components, minimizing parasitic hydronium conduction, and reducing membrane fouling by multi-valent metal ions (e.g., Ca^{+2}, $Fe^{+2,3}$, Al^{+3}), which precipitate at intermediate pH values that may be encountered during salt splitting. In addition, the radiation effects on the organic membranes could lead to rapid failure of the membranes and high maintenance costs.

The initial goals of this work have been to develop an acceptable material composition and to demonstrate the feasibility of the membrane-based, salt-splitting process. First, the potential applications in the DOE complex were reviewed and several important selection criteria were developed. RE-based NaSICON membrane samples were fabricated using conventional methods and tested in an electrochemical "H" cell to provide information relative to the selection criteria. The data from these experiments were evaluated to select a material composition. The feasibility of the process was demonstrated with a flow-through recirculating plate cell using a Hanford tank waste simulant.

POTENTIAL NEW APPLICATIONS

To select a NaSICON composition, it was necessary to develop functional requirements based upon the potential applications at the Hanford Site, the Idaho National Engineering Laboratory (INEL), and the Savannah River Site (SRS). The potential applications and relevant functional requirements are discussed below.

Caustic Recycle

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The Hanford Site and the SRS have large inventories of radioactively contaminated sludge, salt cake, and aqueous liquids. The salt cake and aqueous liquids consist primarily of sodium salts (nitrate, nitrite, hydroxide, aluminate). The primary goal of waste treatment prior to disposal (1,2) is to separate the waste into a low-volume fraction containing most of the radioactivity and a relatively large volume fraction containing most of the nonradioactive chemicals, referred to as high-level waste (HLW) and low-level waste (LLW), respectively. At both sites, the HLW will be converted into a borosilicate glass. At Hanford, the LLW will be converted into a high-sodium LLW glass and at SRS, into a saltstone. Because of its predominance, sodium is the component that controls the volume of the LLW. In sufficient amounts, the residual sodium in the HLW could also impact the volume or the quality of the HLW glass. Significant amounts of caustic are required for proposed waste treatment operations and other ongoing operations. Separation and recycle of the caustic from the tank wastes have the potential to reduce the waste disposal volume and avoid the need to purchase fresh caustic. Potential uses for the caustic include sludge leaching, regeneration of ion exchange resins, inhibition of corrosion in carbon steel storage tanks, and retrieval of tank wastes.

Three operational configurations are available for recovering caustic from the aqueous tank wastes: (1) anolyte pH>12, which would prevent the precipitation of $Al(OH)_3$; (2) anolyte pH in the range of 3 to 12, in which $Al(OH)_3$ would precipitate; and (3) operation with an acidic anolyte in which aluminum would be soluble as Al^{+3}. In all three modes, the catholyte would remain highly alkaline. Configuration 3 would allow the recovery of the greatest amount of caustic, while configuration 1 would recover caustic present only in excess quantities. Preliminary material balances indicate that sufficient caustic is available without using configuration 3, so membrane compatibility with acidic solutions may not be required for this application.

pH Adjustment for Cesium Ion Exchange

At Hanford, it is currently planned to remove ^{137}Cs using an ion exchange process. The efficiency and chemical stability of some ion exchange materials are decreased by the high pH of the waste. For example, the distribution coefficient (Kd) for a powder form of crystalline silico-titanate (TAM5-11) was reported (7) to increase from about 150 mL/g in 1.3 M NaOH to about 1600 mL/g at pH 12. This difference would result in a tenfold reduction in the required amount of exchanger. The reduction in pH would also allow the use of granular potassium cobalt hexacyanoferrate(II). This material has extremely high cesium Kds (8), but it has not been considered for application at Hanford because it is unstable in solutions having a pH >12. Since this application would be used before the cesium is removed, radiation resistance of the membrane is of paramount importance.

Sodium Reduction in HLW Sludge

Many of the stored sludges at Hanford and SRS are anticipated to possess interstitial liquid containing sodium concentrations as high as 5 M. Sodium carry-over with the sludge is undesirable because of the limited levels of sodium the HLW glass composition can accommodate. Current plans are to remove the sodium by diluting the interstitial liquid with 0.01 M sodium hydroxide and sodium nitrite with subsequent solid/liquid separation via settle/decant. Electrochemical

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sodium separation offers the potential for directly removing sodium from the sludge, thereby reducing water usage and solids carry-over during solid/liquid separation.

Treatment of Sodium-Bearing Waste

Sodium removal may also prove beneficial for treating 5.7 million liters of sodium-bearing waste (SBW) at the Idaho Chemical Processing Plant (ICPP) (9). Calcination is an established process at the ICPP and uses high temperatures to convert nonsodium-bearing liquid waste into a granular solid. The presence of sodium tends to cause bed agglomeration, anion volatilization, and alpha aluminum formation. Current mitigation strategies include adding aluminum nitrate, calcium nitrate, and boric acid or modifying the calciner equipment. The addition of chemicals slows processing rates and increases the waste volume, while equipment modifications are expected to be expensive. Removing the sodium before calcining the waste would allow direct calcination of the waste. In contrast to the wastes described under the other applications, the SBW is acidic, so the membrane must be compatible with acidic solutions.

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MATERIAL SELECTION CRITERIA AND EVALUATION

Several criteria were considered for membrane material selection. The criteria are considered below, along with experimental results.

Fabricability, Strength, and Hermetic Qualities

For ceramic membranes, fabricability, strength, and hermetic qualities are somewhat related. Fabricability means that the membranes must be manufactured using a conventional fabrication process that has a wide range of acceptable process conditions and is readily scalable to commercialization. Strength and hermetic qualities refer to the ability of the membranes to provide acceptable separations during operation. Fabrication under the proper conditions is crucial to the performance of the membranes. Second-phase formation at the grain boundaries and other crystallographic imperfections can degrade the ionic conductivity of the membrane. Adequate densities must be achieved since greater than 6% to 8% porosity may compromise the hermetic quality of the membrane, thereby reducing separation performance. The theoretical density of the RE-based NaSICON is about 3.26 g/cm^3.

RE-based NaSICON disk samples, 2-4 cm in diameter and 1-3 mm thick, were prepared using the following steps: powder mixing, pressing, and sintering (10). The samples were characterized by density measurements and x-ray diffraction (XRD) using CuKα radiation prior to electrochemical testing. The RE-based NaSICON samples were found to exhibit adequate densification with densities generally greater than 3.01 g/cm^3. These densities are greater than 92% of theoretical and indicate a porosity <8%. The XRD traces indicated that no phases other than the desired crystal structure were present in the Dy-based NaSICON samples. The Gd- and Sm-based samples were found to contain multiple unknown phases. Although the fabrication conditions for these samples were not optimum, the process seemed sensitive to the fabrication conditions. It was concluded that the Dy-based NaSICON appeared to have a greater tolerance to fabrication parameters than the other compositions.

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Chemical Stability

All applications require that the NaSICON membranes be compatible with alkaline solutions, and some process configurations require compatibility with acidic solutions. Chemical stability was demonstrated by exposing the RE-based NaSICON membranes to alkaline solutions at pH >13 for as long as 1000 hours with no observable corrosion, as determined by mass measurements and scanning electron microscopy (SEM). Subsequent operation in an electrochemical cell also demonstrated no mass loss when operating with alkaline solutions up to 2.7 M NaOH. Exposure of the membrane samples to a solution with a pH of 2.19 resulted in the formation of visible cracks and corrosion as revealed by SEM. Other compositions that are stable in acidic solutions are under development.

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Ionic Conductivity

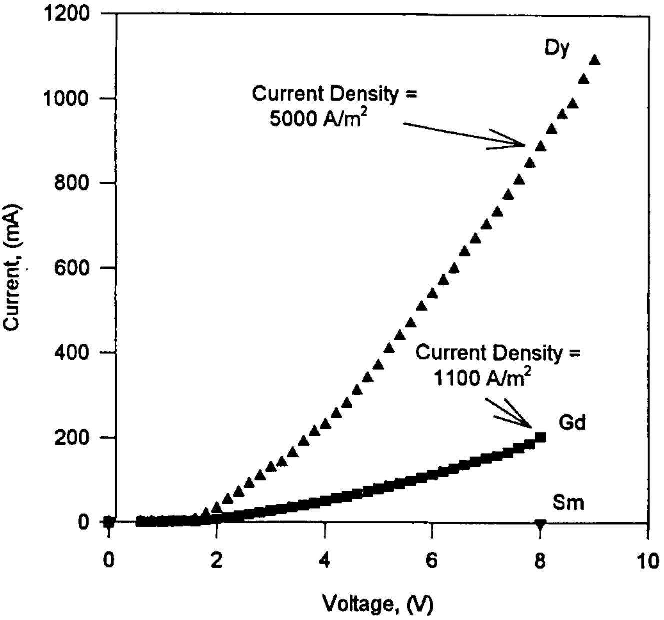
Sufficient ionic conductivity to achieve a current density of >2500 A/m^2 with 6-8 volts is necessary. Current density is normally limited by either excess ohmic heating or parasitic water-splitting reactions. A low ionic conductivity would require larger cells and greater power consumption.

Experiments were conducted in an H cell to measure the current passed as a function of the applied potential (Figure 3). The area of the membrane disks was 1.76 cm^2 and the thickness 1.8 mm. The operating temperature was 80°C using a simplified Hanford waste simulant (1.5 M $NaNO_3$, 1.5 M $NaNO_2$, 0.3 M $Al(NO_3)_3$, 2.0 M NaOH) as the anolyte. The catholyte was 0.1 M NaOH. At 8 volts, the current density of the Dy-based NaSICON was about 5000 A/m^2, the current density of the Gd-based NaSICON was about 1100 A/m^2, and the Sm-based NaSICON appeared to pass no measurable current.

High Selectivity for Sodium

Selectivity for sodium is important for three reasons: 1) separation of the radionuclides from the recycled caustic to minimize shielding requirements, 2) elimination of fouling by multi-valent cations, and 3) reduction of any parasitic ion conduction that increases power requirements. Experiments were conducted in an "H" cell to assess the selectivity of Dy-based NaSICON membranes for sodium relative to Al^{+3}, Sr^{+2}, Cs^+ in neutral to slightly acidic solutions. The initial catholyte composition was 0.1 M KNO_3 and none of the anolyte components were present.

Aluminum ion conduction. To assess the transport of Al^{+3}, an "H" cell with an initial anolyte composition of 1 M $NaNO_3$ + 0.1 M $Al(NO_3)_3$ was operated at 60°C and 4.5 volts. During the experiment, the anolyte pH decreased from 2.93 to 2.25, so the aluminum ions were present as Al^{+3} rather than the AlO_2^- ion found in alkaline solutions. Transport of aluminum in an alkaline anolyte is expected to be minimal since the AlO_2 ion would migrate to the anode and away from the cathode. After the experiment, the composition of the catholyte was determined to contain 650 mg/L of Na and 2 mg/L of aluminum. The aluminum level was at the



NaSICON Disks: Area = 1.76 cm², Thickness = 1.8 mm
Temperature: 80°C

FIGURE 3. Current as a function of applied potential

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detection limit of the analytical method. Thus, it can be concluded that the Dy-based NaSICON has good selectivity for sodium relative to Al^{+3}.

Cesium ion conduction. To assess the transport of Cs^+, an "H" cell with an initial anolyte composition of 0.1M $CsNO_3$ was operated at 65°C and 4.5 volts. At the completion of the experiment, the catholyte contained 22 mg/L of sodium and less than the detection limit for cesium(<1 mg/L). The cesium transport was anticipated to be very low because of its large ionic radius (1.67 Å) relative to the ionic radius of sodium (0.97 Å). The presence of sodium in the catholyte probably resulted from electrochemical flushing of the mobile sodium initially present in the membrane. It is proposed that hydronium ions replaced the sodium ions in the conduction channels of the Dy-based NaSICON. EDAX measurements on the surface of the Dy-based NaSICON indicated a depletion of sodium and no cesium in the crystal structure. If all of the sodium in the center of the NaSICON disk had been displaced into the anolyte, the concentration of sodium would have been 26 mg/L. This amount is in agreement with the measured concentration. Thus, it can be concluded that the Dy-based NaSICON has excellent selectivity for sodium relative to Cs^+.

Strontium ion conduction. To assess the transport of Sr^{+2}, an "H" cell with an initial anolyte composition of 0.1 M $Sr(NO_3)_2$ was operated at 60°C and 4.5 volts with the pH of the anolyte changing from 7 to 2.81. The current decreased from 40-45 mA during the experiment to 30 mA. After the experiment, the catholyte contained 6.3 mg/L of strontium and 220 mg/L of sodium. As with the cesium nitrate anolyte, the sodium in the catholyte is assumed to be that which was displaced from the sodium originally in the NaSICON disk. The divalent strontium ion (1.12Å) is somewhat larger than the monovalent sodium ions (0.97Å). The difference in charge between the strontium and sodium may inhibit the movement of strontium down the sodium ion channels in the NaSICON crystal structure. In any case, the Dy-based NaSICON appears to have good selectivity for sodium relative to Sr^{+2}.

Hydronium ion conduction. In a similar "H" cell experiment using a Hanford simulant (1.5M $NaNO_3$, 1.5M $NaNO_2$, 0.2M $Al(NO_3)_3$, 1M NaOH) as the anolyte, 9800 coulombs were passed at a cell temperature of 60°C with an applied potential of 4.5 volts. During the experiment, the pH of the anolyte fell from 12.1 to 10. Within the experimental error, all of the current was carried by the transport of sodium. Parasitic conduction by hydronium ion was negligible, which is not surprising since H^+ is present in such low concentrations in alkaline solutions.

Radiation Stability

Membrane tolerance to damage by radiation is required since the applications are expected to be in a radioactive environment. Six Dy-based

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NaSICON disks were irradiated to a dose of 1E+09 rads with a ^{60}Co gamma source. This dose is approximately 100 times that expected in actual operation over 15 years, even if cesium is not removed before caustic recycle. Following the irradiation, the disks were placed in an "H" cell and the current was measured as a function of time for applied potentials of 6 and 8 volts. The initial anolyte solution was 1.5 M $NaNO_3$, 1.5 M $NaNO_2$, 0.3 M $Al(NO_3)_3$, and 2 M NaOH. The initial catholyte solution was 0.1 M NaOH. The cell temperature was maintained at 80°C.

No physical changes could be observed after irradiation, except for a slight change in color. Figure 4 shows a plot of current versus time for the sample after irradiation to 10^9 R. Note that the initial current is approximately 50% of that expected for nonirradiated Dy-NaSICON at 6 volts (approximately 500 mA). As current flowed through the NaSICON, the current recovered almost completely. As the voltage was increased to 8 volts, the current recovered to the level anticipated for the nonirradiated material. It is anticipated that if the current was being passed at the same time as the irradiation, no gamma irradiation effect would be present.

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Process Test

Recirculating cell experiments were also conducted with an advanced RE-NaSICON membrane to demonstrate the potential of sodium removal from Hanford tank waste using a simplified Hanford waste simulant as the anolyte (1.5 M $NaNO_3$, 1.5 M $NaNO_2$, 0.2M $Al(NO_3)_3$, 1 M NaOH). The actual Hanford tank waste contains a number of other ions and has a pH which is generally greater than 14. The initial catholyte solution was 1 M NaOH and the cell temperature was maintained at 70°C. Figure 5 plots the total amount of sodium transported and the pH of the anolyte as a function of time for one test in the recirculating cell. The pH of the anolyte decreased from approximately 12.6 to almost 7 as 60 g of sodium were removed through the advanced RE-NaSICON membrane. At pH levels of less than 9, precipitation of aluminum-hydroxide (or salts) occurred in the

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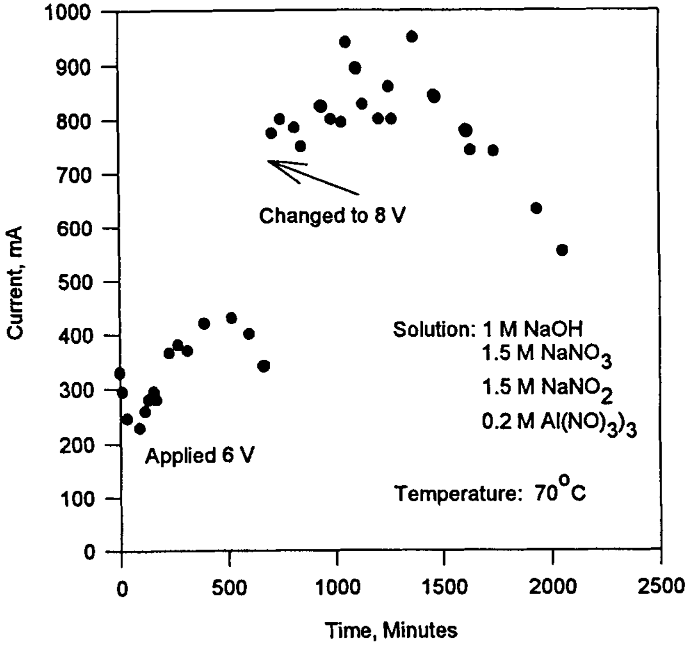

FIGURE 4. Performance of Dy-based NaSICON disk after irradiation to 10^9 rads with ^{60}Co gamma rays

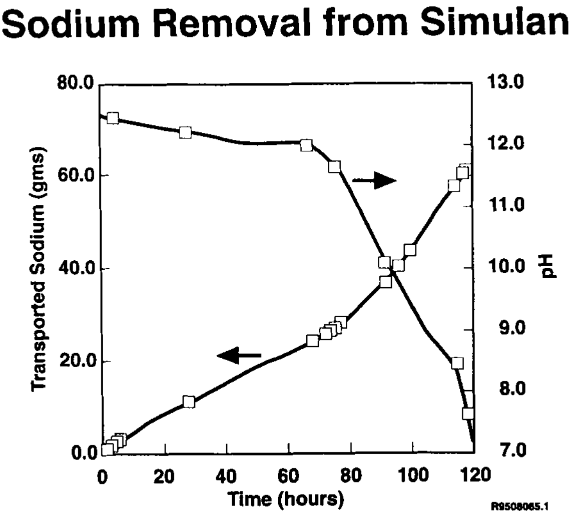

FIGURE 5. Bench-scale process demonstration with Hanford waste simulant

TABLE 1. PROPERTIES OF NaSICON MATERIALS TESTED

Parameter	Dy-based NaSICON	Gd-based NaSICON	Sm-based NaSICON
Fabricability	Good	Poor	Poor
Compatibility			
-Basic	Excellent	Excellent	Excellent
-Acid	Fair	Poor	Poor
Current Density/ Conductivity	Excellent	Moderate	Poor
Selectivity			
-Cesium	Excellent	ND	ND
-Aluminum	Excellent	ND	ND
-Strontium	Excellent	ND	ND
Irradiation Stability	Excellent	ND	ND

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anolyte. However, the throughput of the cell appeared to fall only slightly. The voltage was increased to 10 volts in this particular cell to achieve a current density of 2600 A/m^2.

Additional testing was conducted with a sodium-bearing acidic anolyte with an initial pH of 1.5. The intent was to investigate the operation of the cell under acidic conditions. After 400 additional hours of operation at 3000 A/m^2, a total of 200 g of sodium had been transported through a NaSICON disk weighing 1.2 g. The final pH of the solution was 0.6. As might be expected from the "H" cell corrosion testing described above, the disk thinned in the acidic environment.

SUMMARY AND CONCLUSIONS

Table 1 summarizes the experimental evaluation. Because of difficulties in fabricating Gd- and Sm-based NaSICON and their associated poor ionic conductivity, extensive testing of these compositions was not pursued. Instead, the testing program evaluated other properties of Dy-based NaSICON that could be readily fabricated into membranes with a single crystalline phase type. Experiments conducted in an H cell achieved a current density of 5000 A/m^2 with an applied potential of 8 volts, indicating the Dy-based NaSICON has a good ionic conductivity. The lower ionic conductivity of the Sm- and Gd-based NaSICON probably is due to the presence of multiple phases. The selectivity of the Dy-based NaSICON for sodium relative to Al^{+3}, Sr^{+2}, and Cs^{+} is very high. Parasitic loss in current efficiency resulting from transport of hydronium ion in neutral to alkaline solutions is low. The Dy-based NaSICON demonstrated good chemical stability in solutions ranging from neutral to alkaline, and demonstrated good radiation stability even at doses much greater than those anticipated for actual operation. Based on the experimental results, an advanced RE-based NaSICON material such as Dy-based NaSICON is the preferred material for use in electrochemical processing of tank wastes in which neutral to alkaline solutions will be encountered.

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