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Battery Transport Data*
by
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FINAL REPORT
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to

Lawrence Berkeley Laboratory
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SUMMARY

Measurements of the equilibrium properties have been accomplished for NaOH-H₂O-Nafion® membrane system. The data for 1, 4, 8, and 12 M NaOH at temperatures of 28°C, 50°C, 70°C, and 90°C were obtained by a combination of soaking, blotting, weighing, and analysis techniques. The parameters determined were (i) membrane anion concentration, (ii) membrane water concentration, (iii) membrane fixed ion concentration, and (iv) wet membrane density. The water concentration in the membrane decreased with the external NaOH concentration at a given temperature whereas the other parameters increased. Electrodialysis and dialysis measurements have also been made at room temperature using a cell similar to the one used by Pintauro (1980). There was a problem of gassing of the electrode which made it impossible to accurately measure the volumetric flow through the membrane. Nickel and cadmium porous electrodes were tried unsuccessfully. On the porous Ni electrode, the gassing was not as much as that observed with Cd electrodes. The concentration difference and the volumetric flow rate through the membrane were measured. Using this information, the transference number was calculated using the equation

\[ t_m^+ = \frac{V_0^{F} - V_0^{O}}{F/Q} \]

where \( V_0^{O} \) and \( V_0^{F} \) are the initial and final chamber volumes. \( c_0^{O} \) and \( c_0^{F} \) are the initial and final bulk solution concentration and \( Q \) is the total charge passed. The transference number \( t_m^+ \) decreased with the external NaOH concentration. To overcome the problem of gassing, a weighing technique is now being used. Reverse osmosis experiments were conducted at concentrations of 1, 4, and 8 M NaOH and at temperatures of 23, 50, 70 and 88°C. The flow rate through the membrane decreased with high external concentrations of NaOH at a given temperature. With the increase in temperature, the flow rate increased. At high external concentration the salt rejection was greater than at low concentration.

INTRODUCTION

Until recently membranes have been used only rarely in industrial processing for separation and concentration control functions. Theoretical understanding and quantitative description of membrane transport processes have been developed slowly, and membranes with needed properties have not been commercially available. Although membranes in principle offer the opportunity to develop overall less energy-intensive processing techniques, availability of cheap energy and the uncertainty of developing membrane processes has led to use of more easily understood, high energy-use separations and control techniques. Some examples might be: distillation rather than electrodialysis or reverse osmosis over semipermeable membranes, lead acid batteries with porous, inert separators rather than zinc-nickel oxide batteries which require semipermeable membrane separators, and the use of mercury cells or diaphragm cells for producing chlorine and caustic rather than membrane cells. In each of these examples, the availability of better technical understanding and quantitative description of membrane transport processes along with better, less expensive membranes will lead to more efficient use of energy.

The basic theory and experimental procedures being developed as part of this research effort can be applied to any membrane or diaphragm transport
situation. It is anticipated that in the future the work will be directed towards battery separators, especially separators for zinc-nickel oxide battery systems. However, the success of the perfluorosulfonic acid membranes (Nafion®), has made them an ideal choice for early studies. These membranes have long-term chemical stability, they are readily available, and their structure is being extensively studied (Lopez et al., 1976; a series of papers in the monograph edited by Eisenberg and Yeager, 1982; Hsu and Gierke, 1982; Yeo, 1982; Besso and Eisenberg, 1982; Yarusso, 1982; Covitch, 1982; Yeager, 1982).

The general approach to membrane transport has been described in two publications (Bennion and Pintauro, 1981; Bennion, 1982). Application of this procedure to NaCl in water in a Nafion® membrane at 25°C has been reported by Pintauro (1980). The objective of this research project is to determine the membrane transport and thermodynamic parameters over a range of temperature and concentration. These results are to be used to make a connection between membrane structure, basic transport and thermodynamic parameters which represent that structure, and actual membrane performance. More quantitative and direct connection between the microscopic descriptions of membranes and macroscopic performance is to be accomplished through interpreting observed dependence of the thermodynamic and transport parameters on membrane structure, temperature, and composition of the solutions in which the membrane is immersed.

By way of a short review, there are three independent thermodynamic parameters and six transport parameters which apply to transport of a single electrolyte and water through a membrane. Equilibrium parameters are reported over a range of concentration of NaOH from 1 M to 12 M and temperature of 23°C to 90°C. This data along with some transport data will be reported in this report.

The transport data taking is about half complete. The rest of the data taking will be completed as part of Mr. Bhakta's Ph.D. research program. Reverse osmosis data for NaOH 1 to 8 M and 30 to 90°C is reported. Preliminary dialysis and electrodialysis data at room temperature for 1 to 8 M NaOH is also included. All of this transport data for 1 to 12 M NaOH and 23°C to 90°C will be completed in the next few months. Equipment is now built and tested.

EXPERIMENTAL

The equilibrium parameters are determined using a Nafion® 110 perfluorosulfonic acid cation exchange membrane manufactured by du Pont. The parameters determined are: membrane fixed ion concentration, anion concentration in the membrane, and membrane water concentration. Wet membrane densities were also determined.

Membrane Anion Concentration

A piece of Nafion® membrane of known dry weight and in the H⁺ form (see Figure 1) was immersed in 1.0 M HCl for 24 hours. Afterwards the membrane was leached in distilled water for 24 hours. A known weight of wet membrane was immersed in an aqueous NaOH solution at a given concentration and allowed to soak while stirring for 2.5 hours. The NaOH electrolyte was periodically...
- \[ \left( \text{CF}_2-\text{CF}_2 \right)_m \text{CF}-\text{CF} \] _n -

WHEN:

\( m = 5 \text{ to } 13.5 \)
\( n = \text{ca } 1,000 \)
\( z = 1,2,3--- \)

\[ \begin{array}{c}
\text{CF}_2 \\
\text{O} \\
\text{CF}_2 \\
\text{CF}-\text{CF}_3 \\
\text{SO}_3\text{H}
\end{array} \]

Figure 1. The molecular structure of Nafion®.
replaced to insure that the membrane phase equilibrated with the known concentration of NaOH. After 24 hours the membrane was removed, wiped with a filter paper to remove excess electrolytic solution, and then immersed in distilled water while stirring for two hours. This procedure was repeated to extract all OH\(^{-}\) ions. The concentration of OH\(^{-}\) ions was determined by titration of the mixed wash water with HCl. The mobile anion concentration in moles of OH\(^{-}\) per gram of wet membrane, \(m'_m\), is then known directly from the measurement. The prime is used in the symbol to differentiate the just described definition of molality from the more common definition of moles of solute per kilogram of water, \(m\). The latter definition of molality may have important implications in some theoretical explanation of membrane behavior. The membrane concentration of hydroxyl ions in moles per unit volume is given by

\[ C_- = m'_m \times 1000 \rho_m \]

where \(\rho_m\) is the wet density of the membrane and the 1000 is to convert \(\rho_m\) from g/m\(^3\) to the SI units of kg/m\(^3\). \(C_-\) will then have units of gmole/m\(^3\).

**Membrane Fixed Ion Concentrations**

The membrane fixed ion concentration was calculated using the following relation

\[ C_m = (IEC) \times 1000 \rho_m \]

Here \(\rho_m\) is the wet membrane density and \(\theta\) is the ratio of dry to wet membrane weight. The factor of 1000 converts the units for \(\rho_m\) from gm/cm\(^3\) to kg/m\(^3\). IEC is the ion exchange capacity defined as IEC = 1000/EW. For the 1100 EW membrane used in this work IEC is 0.909 milliequivalents per gram.

**Wet Membrane Density**

The dry weight and size of the membrane were first determined with the membrane in the hydrogen form. The membrane was next immersed in a NaOH-water solution of the desired composition. The NaOH solution was periodically changed to insure the bulk concentration was the desired value. The soaking was continued for 24 hours. The membranes were then removed from the solution and wiped with a filter paper to remove excess surface electrolytic solution. The wiping has to be done with caution so as not to squeeze the electrolyte out of the membrane and also not to leave a film of bulk electrolytic solution over the membrane surface. Moving rapidly to avoid significant evaporation, an outline of the membrane was made on paper. The membrane was then weighed and its thickness determined at five different points using a micrometer. From the outline, the area of the wet membrane was determined. At high NaOH concentrations above 8 M, the membranes tended to curl and were difficult to trace.

**Membrane Water Concentration**

Membrane water content was determined by first letting the membrane equilibrate with NaOH solution of a given concentration as was done earlier. The membrane was then removed from the electrolyte solution, wiped with filter paper to remove excess NaOH solution and weighed in a petri dish. The membrane was dried in a vacuum oven at 37°C for 48 hours (until its weight became constant) then reweighed and the water content calculated.
Transport Experiments

Electrodialysis

Measurements were made at room temperature using the cell similar to that used by Pintauro (1980). In this experiment both the cell compartments are filled with the same NaOH solution. The compartments were separated by the Nafion® membrane (presoaked in the electrolyte for 1 day). A precalculated current is applied for a predetermined time. The average applied experimental current is determined by dividing the charge passed, as recorded by a coulometer, by the total experimental time. This method proved to be more accurate than reading the current directly from the ammeter because the current fluctuated slightly during an experiment. After passing the current, the cell is taken apart and the solutions analyzed. The main problem encountered was "gassing." Gassing is not desired because there is no outlet from the cell for the gas to escape and it builds up pressure, forcing liquid out of the cell, affecting the reading in the horizontal capillary tubes required to know the volumetric flow. Porous cadmium electrodes (supplied by Eagle Picher Co., Joplin, MO) were tried as working electrodes, but gassing was observed, even at as low a current as 0.3 A for 19.6 cm² electrodes. Porous nickel electrodes supplied by Eagle Picher Co. were next tried. It also led to gassing, but it was not as much as that observed with Cd electrodes.

Dialysis Experiment

The same cell as used in the electrodialysis experiment was used here, but no current was passed. Measurements were made at room temperature. In the experiment an initial concentration difference was allowed to decay with time. The concentration difference was the same in all the experiments, ± 0.4 M. The lower compartment was filled with the less-concentrated solution. A Nafion® membrane was soaked in the lower-compartment NaOH solution for 24 hours prior to a dialysis experiment. The upper chamber of the cell was filled with concentrated salt solution after the lower compartment was full and the membrane put in place. After about 7 hours the cell was taken apart and the solutions analyzed. Some problems are being encountered in determining the flow rate. The final salt concentration in the upper chamber was the key information obtained.

Reverse Osmosis

The apparatus used in the reverse osmosis experiments is shown in Figure 2. The main component of the RO cell was a 10 liter Teflon®-coated stainless steel vessel. A Nafion® membrane (40.7 cm² in area) supported by a porous stainless steel disk was placed in the bottom of the vessel. Mixing was provided by a mechanical, rotating stirrer which was driven by a magnetic drive. A rotational stirrer speed (928 rpm) was used to insure that boundary layer effects were negligible (Choi and Bennion, 1974). The temperature in the cell was held constant by pumping water from a Fischer Isotemp Bath through cooling coils in the cell. Pressure was applied to the fluid in the cell through two diaphragms in series. The diaphragms separated oil from a water solution and the water solution from the test electrolytic solution. A constant pressure was applied by a weight from an Amthor dead weight testing device using oil as the pressure transmitting fluid. A glass capillary tube
Figure 2. Reverse osmosis cell.

Dimensions are in inches
was connected to the exit port of the cell to measure fluid flow through the membrane.

Pretreated Nafion® membranes were presoaked in the NaOH electrolytic solution of interest prior to the RO experiment. Because of the very small flow rates through the membrane in these experiments and the large void space in the porous stainless steel disk which supported the membrane, the porous disk was vacuum filled with distilled water before being inserted into the RO cell. The upstream pressure was maintained constant at 73 psi. This low-pressure driving force was used to insure that there was no compaction of the membrane during an experiment. The volumetric flow rate of fluid through the membrane was determined by monitoring the change in the liquid position in the capillary tube. Steady-state conditions were obtained when the flow rate stabilized to a change of less than 10% in two hours. This occurred after approximately 15 hours. For the next 30 hours flow rate data were periodically taken. After the steady state was reached, downstream salt concentrations were measured by titrating the exit solution with standardized HCl.

The data obtained from the reverse osmosis experiments were the steady-state volumetric flow rate through the membrane and the steady-state downstream salt concentration.

RESULTS AND DISCUSSION

At a particular external temperature the fixed ion concentration, the anion concentration, and the cation concentration increase with the external NaOH concentration as shown in Figures 3-17. The fixed ion concentration increase is explained qualitatively by the membrane swelling less in concentrated electrolyte solution leading to a smaller wet membrane volume. The water concentration, which is one of the most important parameters in determining the membrane performance, decreases with increasing external concentration of NaOH at a particular temperature (Figure 18). It increases with temperature, however, at a fixed external concentration (Figure 19).

The electrodialysis data is shown in Figure 20. The transference number was calculated using a simple minded equation

\[ t^m = \frac{|V^0 C_e - V^F C_e| F}{Q} \]

where \( V^0 \) and \( V^F \) are the initial and final bulk solution concentration and \( Q \) is the total charge passed during an experiment. As porous electrodes are charged/discharged, a concentration gradient builds up within the thickness of the electrode. If the cell compartment is small, this difference (unaccounted for in the solution analysis) could be significant, so corrections are required in the above equation. But in the preliminary calculation no corrections are made. The data in Figure 20 follows the trend observed by Yeager (1982). The electro osmotic coefficient of water or the water transference number is not reported because the capillary flow rates could not be determined due to gassing. As a suitable working electrode, which would prevent the problem of gassing, could not be found, we feel that a weighing technique should be used to determine transference numbers. A new cell as shown in Figure 24 has been built. This cell will be used to make
Figure 3. 1100 EW Nafion\textsuperscript{R} fixed ion concentration vs. the external NaOH concentration ○ 25\textdegree{}C, ● Pintauro's data (1980).
Figure 4. Membrane anion concentration vs. the external NaOH concentration $25^\circ C$, Pintauro's data (1980).
Figure 5. Membrane cation concentration at 25°C vs. the external NaOH concentration.
Figure 6. Wet membrane density at 25°C vs. the external NaOH concentration.
Figure 7. 1100 EW Nafion® fixed ion concentration at 50°C vs. the external NaOH concentration.
Figure 8. Membrane anion concentration at 50°C vs. the external NaOH concentration.
Figure 9. Membrane cation concentration at 50°C vs. the external NaOH concentration.
Figure 10. Wet membrane density at 50°C vs. the external NaOH concentration.
Figure 11. Membrane fixed ion concentration at 70°C versus the external NaOH concentration.
Figure 12. Membrane anion concentration at 70°C versus the external NaOH concentration.
Figure 13. Membrane cation concentration at 70°C vs. the external NaOH concentration.
Figure 14. Membrane fixed ion concentration at 900°C vs. the external NaOH concentration.
Figure 15. Membrane anion concentration at 90°C vs. the external NaOH concentration.
Figure 16. Membrane cation concentration at 90°C vs. the external NaOH concentration.
Figure 17. Nafion® wet membrane density at 90°C vs. the external NaOH concentration. ◎ Run #1, △ Run #2, □ Run #3, ▼ Run #4.
Figure 18. Membrane water content versus the external NaOH concentration (○ 90°C, △ 70°C, ● 50°C, ■ 23°C.

Figure 19. Membrane water content vs. temperature (■ 1M NaOH, 4M NaOH, △ 8M NaOH, ○ 12M NaOH.)
Figure 20. Nafion® cation transference number at 25°C vs. the external NaOH concentration.
measurements at various concentrations and temperatures. Figure 21 shows the percent change in the upper-chamber salt concentration as a function of the initial chamber concentration.

Figures 22 and 23 show the reverse osmosis data. The volumetric flow rate through the membrane decreases with increasing external concentrations of NaOH at a given temperature. Similar results were obtained by Pintauro (1980) for NaCl. The observed Nafion® flow rates are low compared to skinned cellulose acetate membranes (Choi and Bennion, 1975). The increase in volumetric flow with temperature can be explained both by increased molecular activity and by an increase in free water concentration as shown in Figure 19. The relative flow rates of NaOH and water are illustrated in Table 1. At high external electrolyte concentration the electrolyte flow rate is relatively less than water while at low concentration the relative flow rate of electrolyte is greater. The salt rejection is not comparable to that observed with typical RO membranes. But low-pressure reverse osmosis experiments with ionic membranes typically reject only 0-5% of the upstream salt (Michaels et al., 1973).

INTERPRETATION OF RESULTS

There are various models which are used to explain the water concentration data. Rodmacq and coworkers (1980) propose a three-phase model in which fluorocarbon microcrystallites, ion water clusters, and a second ionic region of lower water content coexist. Falk (1980) sees evidence for two environments of sorbed water in Nafion® from infrared spectroscopic studies. The first environment appears to be aqueous in nature, with the strength of intermolecular hydrogen bonding reduced from that in pure water. In the second environment, the water molecules are not hydrogen bonded and appear to be exposed mainly to fluorocarbon. Thus, in essence, the water in the Nafion® membrane (C₀) can be visualized as consisting of free water (C₀ᶠ) and bound water (C₀ˢ). The free water exists in the aqueous phase and the bound water exists in the fluorocarbon phase

\[ C₀ = C₀ᶠ + C₀ˢ \]

Assuming Langmuir type adsorption for the bound water phase, we have

\[ rₐ = kₖ C₀ᶠ \frac{(C₀ˢ₀ - C₀ˢ)}{C₀} \]

\[ rₐ = \text{rate of adsorption} \]

\[ C₀ˢ₀ = \text{total number of sites} \]

\[ r_d = k_b C₀ˢ \]

\[ r_d = \text{rate of desorption} \]

\[ kₖ = \text{rate constant for adsorption which is temperature dependent but independent of composition} \]

\[ k_b = \text{rate constant for desorption which is temperature dependent but independent of composition} \]
Figure 21. Dialysis concentration data. The percent change in the upper chamber concentration vs. the initial upper chamber concentration.
Figure 22. Steady state reverse osmosis volumetric flow rate vs. temperature. Upstream pressure: 84 psi. ○ upstream concentration 1M NaOH.
Figure 23. Steady state reverse osmosis volumetric flow rate vs. temperature. Upstream pressure: 73 psi. 
△ upstream concentration 4M NaOH, □ upstream concentration 8M NaOH.
Figure 24. Electrodialysis/dialysis cell.
Table 1

Downstream Concentration
Kgmoles/M³

<table>
<thead>
<tr>
<th>Upstream Concentration Kgmoles/M³</th>
<th>T = 32°C</th>
<th>T = 50°C</th>
<th>T = 70°C</th>
<th>T = 88°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.160</td>
<td>1.062</td>
<td>1.1335</td>
<td>1.071</td>
</tr>
<tr>
<td>4</td>
<td>3.781</td>
<td>3.829</td>
<td>3.8048</td>
<td>3.899</td>
</tr>
<tr>
<td>8</td>
<td>6.372</td>
<td>6.714</td>
<td>6.558</td>
<td>5.9089</td>
</tr>
</tbody>
</table>
At equilibrium

\[ r_a = r_d \]
\[ k_f C_0 \left( C_{so} - C_s \right) / C_o = k_b C_s \]

Eliminating \( C_f \) and solving for \( C_s \) yields

\[ C_s = \frac{C_o (1+K) + C_{so} - \sqrt{(C_o (1+K) + C_{so})^2 - 4C_s C_o}}{2} \]  

(1)

\[ K = \frac{k_f}{k_b} \]

There are various models to correlate the diffusion coefficient (D) with the water content. Yasuda and coworkers (1968) have used the function \( V_p/(1-V_p) \) to correlate with D. \( V_p \) is the volume fraction of polymer in a water-swollen material. The denominator is, therefore, the volume fraction of water in the membrane calculated from sorption results. This function was developed by Yasuda and coworkers to treat diffusion in various hydrophilic polymers. Their equation

\[ \overline{D} = D^0 \exp \left[ -bV_p/(1-V_p) \right] \]

describes the relationship between the aqueous and the polymer diffusion coefficients of a species, \( D^0 \) and \( \overline{D} \) respectively. The constant \( b \) in the above equation is related to the free volume of water. This is one of the models to correlate the diffusion coefficient with the water concentration. Other simpler models should be considered like

\[ D_1 = D_s \frac{C_s}{C_o} + D_1 \frac{(1-C_s)}{C_o} \]

The above model is an extension of concepts taught to Bennion by Henry Eyring in a course in 1966. The general idea of Eyring's significant structure theory can be found in Eyring et al. (1964) and Eyring and Jhar (1970). The idea can be further traced to Einstein's theory of viscosity of solutions (1905). The usefulness of the models could be evaluated by determining the parameters and fitting the calculated value of the diffusion coefficient to that experimentally determined by transport experiment. This model is now in infancy, and it will be tested later. The water concentration data can also be explained using the ion clustering model of Gierke and Hsu (1982). At low external NaOH concentrations the ionic cluster diameter is larger, leading to high water content in the clusters. The clusters are formed at a minimum free energy, so at high temperature the free energy is larger as a result of which the cluster diameter increases leading to the increased water content in the membrane.

FUTURE WORK

Future work will be concentrated in obtaining the rest of the transport data by electrodialysis and dialysis over the range 1 M to 12 M NaOH and temperatures from 25°C to 90°C. The mathematical model presented by Pintauro (1980) will be used to obtain the "best" transport parameter values to fit the data and to predict the concentration, pressure, and potential profiles in the
membranes for the various operating conditions. The transport and thermodynamic parameters will be tabulated as functions of temperature and external and internal electrolyte concentration. Attempts will be made to explain the observed dependence of the parameters on temperature and composition in terms of some structural model of the membrane. Further refinement to include structural information now becoming available will be considered. The results here on NaOH will be combined with results from ETC (1983) and Pintauro on NaCl (1980) to achieve a more complete description of ion and water transport in multicomponent situations. Attempts will be made to predict cross terms associated with mixtures of NaCl and NaOH. If time permits, a few key measurements on mixtures of NaCl and NaOH will be attempted. Consideration will be given to correlating transport performance to membrane structure and to optimizing membrane structure to achieve desired performance. We also intend to apply this procedure to membranes in zinc-nickel oxide batteries.

REFERENCES


E. Besso and A. Eisenberg, "Properties and Structure of Ionomers and Ionomeric Membranes," ibid.


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