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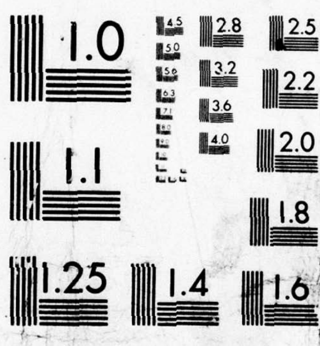

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DIFFUSION STUDIES ON A PPQ-CA MEMBRANE — A COMPARISON WITH STANDARD MEMBRANE MATERIALS

BY WILLIAM P. KILROY LINDA LAUGHLIN

RESEARCH AND TECHNOLOGY DEPARTMENT

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This report discusses zincate and electrolyte diffusion through a new separator material currently being developed for potential use in alkaline AgO-Zn batteries.

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SUMMARY

A polyphenylquinoxaline (PPQ) - cellulose acetate (CA) membrane material is currently being developed by NSWC for potential use as a separator material in alkaline AgO-Zn batteries. One of the criteria used in evaluating the membrane material is to determine the rate at which soluble zinc passes through the membrane.

We have measured the rates of zincate diffusion through a PPQ-CA membrane and compared this flux with that of standard separator materials.

This work was sponsored by the Independent Research Program of the Naval Surface Weapons Center.

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INTRODUCTION

Silver-zinc batteries are widely used by the Navy for many applications. The superiority of the AgO-Zn electrochemical couple as a rechargeable system was largely due to the use of cellophane as a separator. However, it is the oxidative degradation of the cellophane separator by the AgO and KOH electrolyte that limits the cycle life of the battery. This separator failure gives rise to expensive battery replacement costs.

At this center, polymeric membranes are being developed that have excellant stability to AgO-KOH solutions and have shown promise for use as battery separators(1). The primary purpose of this investigation was to select the most promising membrane material, a mixture of 60% polyphenylquinoxaline (PPQ) and 40% cellulose acetate (CA), and measure the diffusive characteristics of the membrane. The objective was to determine if the PPQ-CA membrane had comparable diffusive properties to standard separator materials.

THEORY

An aromatic heterocyclic polymer such as PPQ is nonpolar and hydrophobic to the KOH electrolyte. Since this material does not absorb KOH to swell sufficiently to allow passage of conducting ions, it has a high inherent electrical resistance(2). The electrical resistance can be reduced by incorporating a polar hydrophilic substance such as cellulose acetate to form a codispersed heterogeneous membrane. The polar component is then extracted in a suitable solvent(3) or by hydrolysis in KOH. This creates a semiporous film of a PPQ matrix with an unknown amount of the CA remaining.

---

1. I. Angres, J. V. Duffy, and W. P. Kilroy "Heat and Chemically Resistant Separators", Proceedings --- Twenty Eight Power Sources Symposium, 1978.

2. W. P. Kilroy and J. V. Duffy, "Development of an Improved Separator for Alkaline Silver-Zinc Batteries", NSWC/WOL TR 76-135.

3. I. Angres, "Compatibilization of Polyphemyl-quinoxaline with other polymers using chloroform as the solvent in the preparation of polymeric membranes", NSWC/WOL TR 77-118.

EXPERIMENTAL

Preparation of the PPQ polymer has been previously reported[4]. The procedure for casting the membrane films[2] and preparation of the membranes has been discussed[1,3]. In summary, add CA (40% acetyl content) to a 10% PPQ solution in m-cresol (obtained from the Narmco Division of the Whittaker Corporation). Add 100 ml of chloroform until a 60% PPQ:40% CA homogeneous solution forms. Cast a film and allow the solvent to evaporate slowly for 2 to 3 minutes. Immerse in 1:1 methanol-water mixture for ten minutes, remove, wash with water and air dry.

Diffusion of OH^- ions through the separator was studied by following pH changes across the separator by the method of Harris[5]. The flux of zincate across the separator was determined by a new procedure incorporating differential pulse polarography (DPP)[6].

A membrane material was inserted between two cell compartments of a modified diffusion cell described by Harris[5]. The oriface and consequently the membrane surface area was 0.785 in^2. For the zincate diffusion studies, the zinc-rich compartment contained 250 ml of 1M ZnO in 42% KOH. The zinc poor side contained 250 ml of 42% KOH. At intervals, the zinc-deficient solution was stirred and a 2 ml aliquot was removed and placed in a polarographic cell containing 25 ml of distilled water. The cell employed a working dropping mercury electrode, a platinum wire counter electrode and a Hg/HgO reference electrode. After purging with water saturated argon, the DPP curves were recorded on a PAR model 174A DPP instrument. The peak heights were compared to a previously recorded DPP calibration curve established using 10^{-3}M to 10^{-5}M zinc ion in 0.8M KOH.

In order to prevent an osmotic pressure generated error in the zincate flux, approximately 2 ml of the zincate-rich solution was simultaneously removed.

DISCUSSION OF RESULTS

Diffusion Studies

The rates of diffusion of zincate across several membranes are illustrated in Figure 1. In order to compare the membranes on an equivalent basis, it was necessary to normalize each membrane to a constant thickness of one mil. The zincate flux values reported as moles zincate/min in^2 for a 1.0 mil wet thickness are tabulated in Table I.

---

4. J. K. Stille, U.S. Patent No. 3,661,850, May 1972

5. E. L. Harris, "Electrolyte Diffusion", Characteristics of Separators for Alkaline Silver Oxide Zinc Secondary Batteries - Screening Methods, Edited by J. E. Cooper and A. Fleischer AD-447301, p 93 (1964).

6. W. P. Kilroy and Linda Laughlin, "Measurement of the rates of diffusion of soluble zinc through membrane materials in KOH solution by differential pulse polarography and comparison with potentiometric methods", NSWC/WOL TR 78-172.

Table I

Comparison of Zincate Diffusion in PPQ-CA Membranes with Standard Separator Materials.

Membrane Material	Wet Thickness (mil)	Zincate Flux (moles/min in^2)	Zincate Diffusion Coefficient (cm^2/sec)
pudo cellophane	3.36	6.7×10^{-6}	1.9×10^{-7}
sausage casing	7.24	6.6×10^{-6}	4.0×10^{-7}
silver cellophane	3.06	5.7×10^{-6}	1.5×10^{-7}
60% PPQ - 40% CA	0.88	8.8×10^{-7}	6.5×10^{-9}

Figure 1 illustrates the relative rates of "wetting", that is, the time for diffusion to be appreciable. The cellophane materials and the sausage casing are hydrophilic and have the ability to absorb KOH electrolyte which allows diffusion to occur somewhat faster. The slower wetting time of the PPQ-CA is the result of the nonpolar and hydrophobic nature of the PPQ polymer. It is the CA portion that provides the wettability.

Despite the somewhat slower rates of elecrolyte absorption, the PPQ-CA membrane offers the advantage of slower diffusion of zincate. This may reduce the mechanism of cell failure by electrical short-circuiting, generally initiated by a supply of zincate feeding the growth of conducting metallic zinc dendrites. Table I shows the PPQ-CA has almost an order of magnitude slower zincate flux than the standard separator materials.

In order to determine if there was also an appreciable decrease in the permeation of the KOH electrolyte in the PPQ-CA separator, the diffusion of KOH through a PPQ-CA membrane was compared with that of cellophane. The comparison is shown in Figure 2. In order to compare membranes on an equivalent basis, the flux of hydroxyl ions was normalized to a membrane of one mil thickness. The reported flux for the cellophane membrane[(5)], normalized to 1 mil thickness is approximately 2.9×10^{-3} moles OH^-/min in^2 whereas the PPQ-CA membrane has a value of 3.4×10^{-4} moles OH^-/min in^2. An estimate of the upper current density that the PPQ-CA separator would allow can be calculated from Faradays Law:

$$I = \frac{\text{equivalents} \times F}{t} = \frac{3.4 \times 10^{-4} \times 96485}{60} = 0.55 \frac{\text{amp}}{\text{in}^2}$$

Calculation of the Diffusivity of the Zincate ion in PPQ-CA Membranes

The diffusion coefficient (D) for the zincate ion in a membrane can be calculated from the following equation

$$D = \frac{C_2 - C_1}{t_2 - t_1} \cdot \frac{X}{A} \cdot \frac{1}{d_2 - d_1}$$

where $\Delta c/\Delta t$ is the mass of the substance that diffuses through a membrane of cross-section A and thickness X in time t. If a constant concentration gradient is provided, the flux is constant and if the diffusing material passes into an unchanging volume on the dilute side of the membrane, the concentration varies linearly with time. This is accomplished by using a 1 molar zincate ion solution, d_2, on one side of the membrane, and a dilute 10^{-3} or 10^{-4} molar zincate ion solution, d_1, on the other side.

The calculated diffusivity values for the various membranes have been tabulated in Table I. The diffusion coefficient for the zincate ion at 24°C has been reported to be 9.9×10^{-7} cm^2/sec in 45% KOH.(7) This value is approximately an order of magnitude greater than the diffusivity values of the standard separator materials. The diffusivity of zincate in PPQ-CA membranes is almost two orders of magnitude lower than in the KOH indicating that the PPQ-CA membrane has either a greater tortuosity or a smaller more selective pore size than the standard separator materials.

CONCLUSION

This center is currently in the process of developing PPQ-CA membranes for potential use in alkaline AgO-Zn batteries. This report shows that the separator at this stage of development possesses excellant zincate diffusional characteristics.

ACKNOLWDGMENT

We wish to thank Dr. Issac Angres for providing us with a PPQ-CA membrane.

---

7. C. E. May and H. E. Kantz, "Determination of the Zincate Diffusion Coefficient and its Application to Alkaline Battery Problems", Paper presented at the Pittsburg Meeting of the Electrochemical Society, Oct 1978.

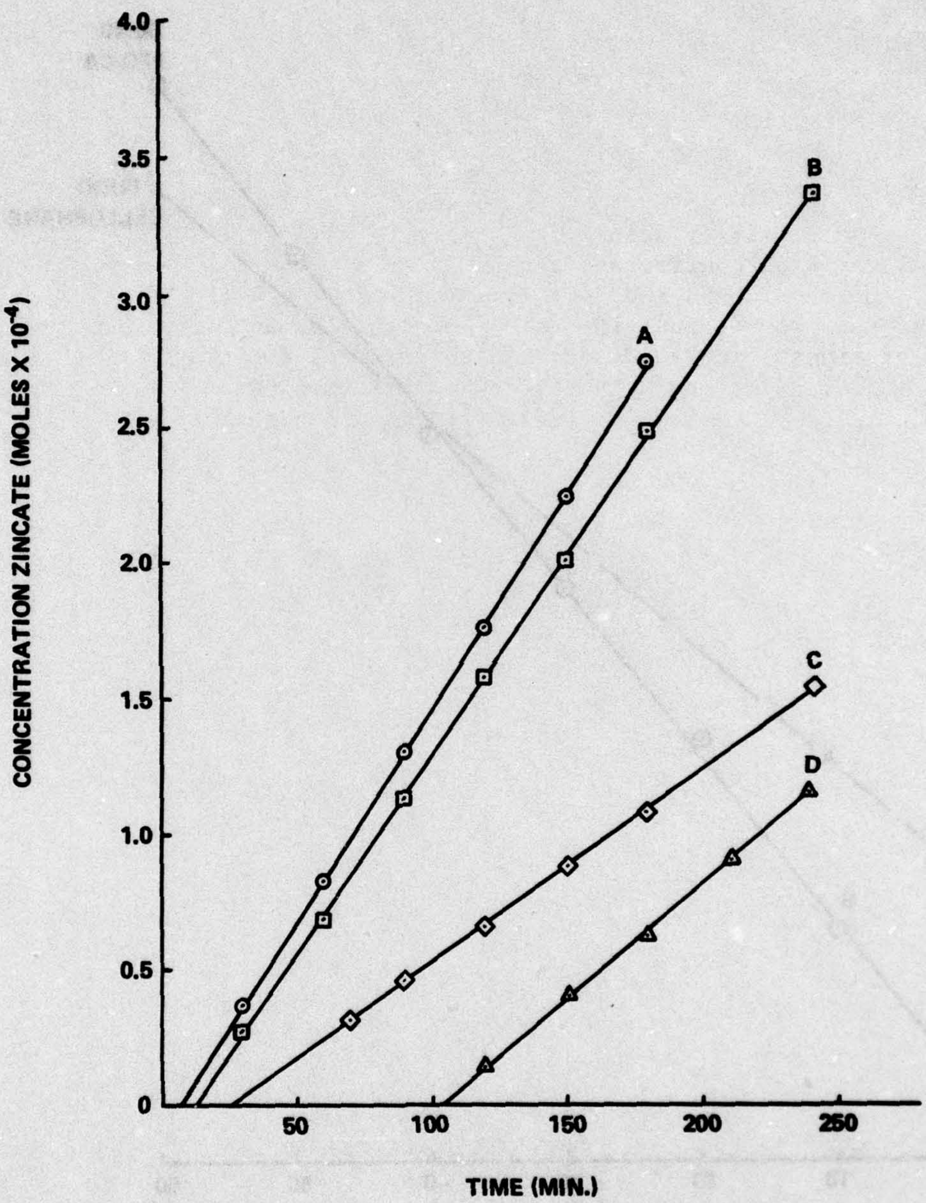

FIGURE 1 COMPARISON OF THE RATES OF ZINCATE DIFFUSION THROUGH VARIOUS STANDARD SEPERATOR MATERIALS WITH DIFFUSION THROUGH A 60% PPQ - 40% CA MEMBRANE. CURVE A - CELLOPHANE, CURVE B - SILVER CELLOPHANE, CURVE C - SAUSAGE CASING, CURVE D - 60% PPQ - 40% CA.

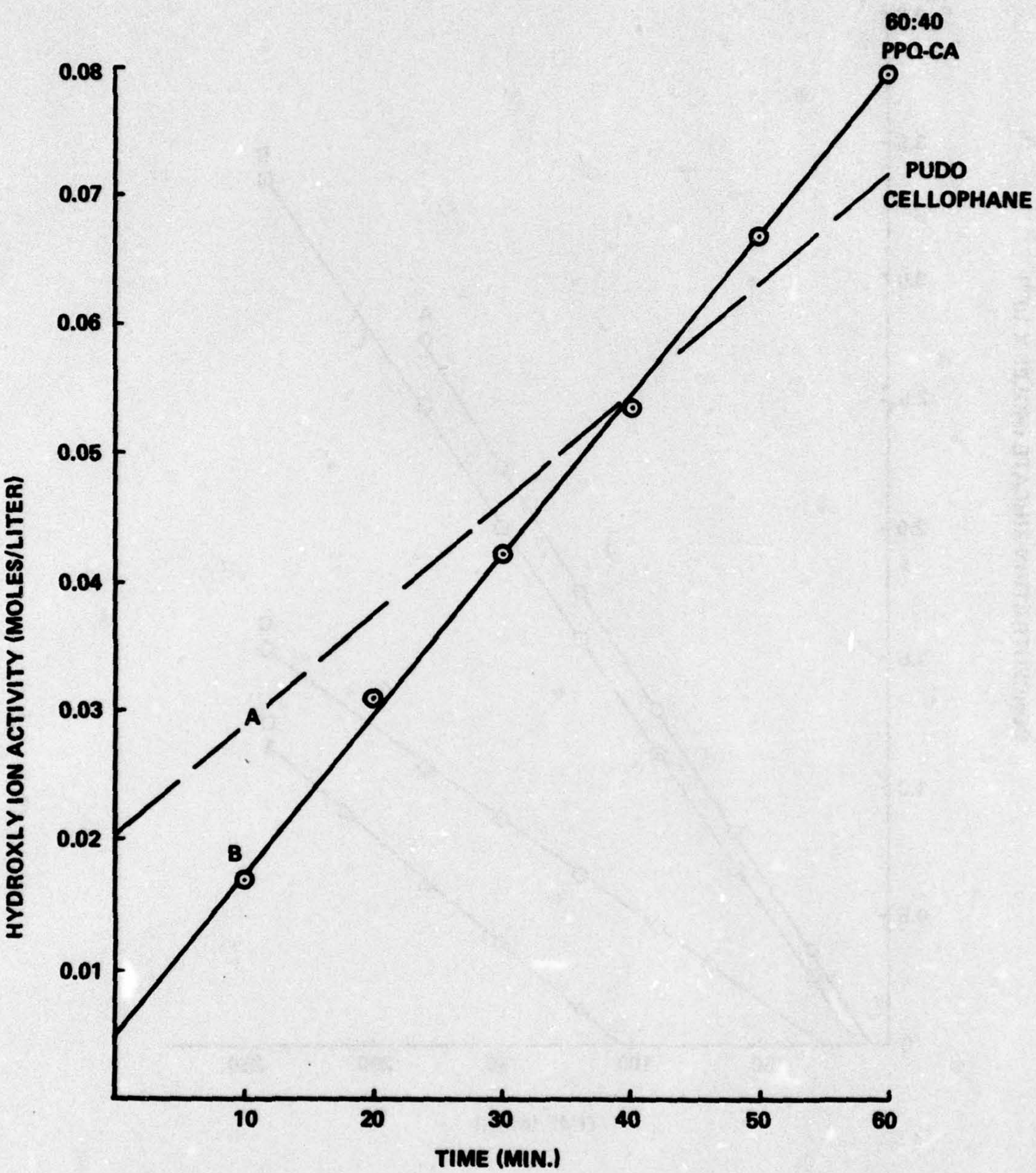

FIGURE 2 COMPARISON OF KOH DIFFUSION THROUGH 60% PPQ-40%CA AND PUDO CELLOPHANE MEMBRANES. CURVE A - KOH DIFFUSION THROUGH 3 MIL (WET) CELLOPHANE AS REPORTED BY HARRIS [5]. CURVE B - KOH DIFFUSION THROUGH A 0.88 MIL (WET) 60% PPQ-40% CA MEMBRANE.

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PENALTY FOR PRIVATE USE, $300

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DOD 316

COMMANDER
NAVAL SURFACE WEAPONS CENTER
WHITE OAK, SILVER SPRING, MARYLAND 20910

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