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MEASUREMENT OF THE RATES OF DIFFUSION OF SOLUBLE ZINC THROUGH MEMBRANE MATERIALS IN KOH SOLUTION BY DIFFERENTIAL PULSE POLAROGRAPHY AND COMPARISON WITH POTENTIOMETRIC METHODS,

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RESEARCH AND TECHNOLOGY DEPARTMENT

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The rate of zincate diffusion through battery membrane materials has been measured by potentiometry and compared to a new technique employing differential pulse polarography.

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SUMMARY

A new separator material for alkaline batteries is presently under development at the Naval Surface Weapons Center. One of the criteria used in evaluating the separator is the rate of diffusion of soluble zincate through the separator. In order to assist in the efficient development of a viable separator material, it is essential to have an efficient and reproducible method for evaluating rates of diffusion.

This investigation was undertaken to determine the reliability, convenience, and reproducibility of measuring diffusion of soluble zinc through separators.

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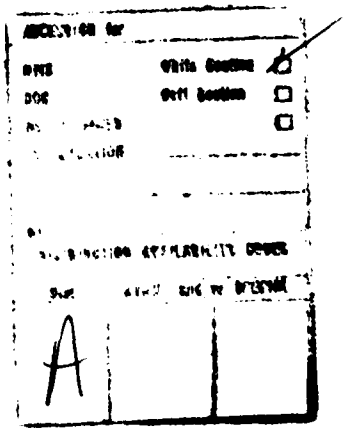

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INTRODUCTION

With the continued development of improved batteries, there has been a simultaneous need for the creation of better synthetic polymeric separators. The complexities associated with improving separators require a thorough knowledge of properties and structure. In order to clearly characterize the physical and chemical properties of separator materials, suitable experimental methods must be established for determining each of the desired parameters.

The accurate measurement of separator resistance has been the subject of the first report.[1] We now wish to report on an improved method for the measurement of zincate ion diffusion.

The standard method for measuring zincate ion diffusion through separators[2] is based on potentiometric theory, i.e., the potential of a zinc-zincate ion couple at constant hydroxyl ion concentration will vary by 0.0295 volts for each 10-fold change in concentration of zincate ion concentration.[3] The potentiometric method was originally selected for its convenience in providing a rapid and strictly instrumental technique. However, we have found that the practical application of this potentiometric technique to be unsatisfactory. The detection of low concentrations of zincate in highly alkaline solutions by potentiometry was found to be subject to numerous experimental difficulties leading to irreproducibility of the potential-concentration calibration curve.

Accurate rates of diffusion can be better achieved by sampling a portion of the solution and analyzing for zinc ion by atomic absorption spectroscopy. Differential pulse polarography was studied as a technique to provide the convenience of the potentiometric method while improving the analysis procedure to rival that of atomic absorption.

---

1. W. P. Kilroy and C. T. Moynihan, "Measurement of Battery Separator Resistances in Low Impedance Conductivity Cells for A-C Bridge Techniques", J. Electrochem. Soc. 125(4), 520 (1978).

2. J. J. Lander, "Zinc Diffusion", Characteristics of Separators for Alkaline Silver Oxide Zinc Secondary Batteries - Screening Methods, Edited by J. E. Cooper and A. Fleischer, 1964.

3. T. P. Dirkse, "The Nature of the Zinc-Containing Ion in Strongly Alkaline Solutions", J. Electrochem. Soc. 101(6), 328 (1954).

THEORY

The polarographic method employs the unique characteristics of the current-voltage curves obtained when solutions of electrooxidizable or electroreducible substances are electrolyzed at a dropping mercury electrode (DME). As a continuously increasing potential is applied to a solution containing inert electrolyte and an electroactive specie, a maximum current is attained that is governed by the rate of supply of the electroactive substance to the electrode surface by a process whose rate is independent of the electrode potential. This maximum current is directly proportional to the concentration of the electroactive substance. Quantitative data is readily obtained via a current-concentration calibration curve.

The sensitivity of the analysis is increased by a technique in which the current flow is sampled before and after application of a potential pulse that is imposed on the linearly increasing dc voltage. This differential pulse polarographic method presents a current signal approximating the derivative of the polarographic wave, and thus yields large easily defined current peaks that are directly proportional to concentrations in the order of ppb or less.

The differential pulse method offers the advantage in that electrodes other than the DME can be used while maintaining the advantages of DME polarography. In addition, more than one element may be analyzed simultaneously under appropriate conditions in either organic or inorganic solutions.

Comparison of the potentiometric and differential pulse polarographic techniques was accomplished using cellophane membranes in a diffusion cell similar to that used by Harris.[4] The cell allows a membrane to be introduced between cell halves, one half containing a zincate-rich solution and the other half containing a zincate-poor solution. The flux, or moles of zincate diffusing per unit time is directly proportional to this concentration gradient. The zincate ion will diffuse to the zincate-deficient side of the cell at a rate controlled by the membrane thickness, area, total porosity and pore size.

EXPERIMENTAL

A modified version of the cell described by Harris[4] was employed. Details of the potentiometric method have been reported.[2] Standard zincate solutions were prepared by dissolving known amounts of reagent ZnO in Fisher certified 45% KOH.

POTENTIOMETRY. Potentiometric calibration curves were obtained using deaerated standard zincate solutions maintained under a flowing blanket of argon. Changes in potential between a Hg/HgO reference electrode and an amalgamated zinc electrode were recorded to 0.01 mv on a Cimron Model 7200A Voltmeter. The zinc electrode consisted of 1/8 inch 99.99% zinc that was cleaned in HCl, washed in water and heavily amalgamated in a solution of mercuric acetate. In diffusion studies, the Hg/HgO reference and amalgamated zinc electrodes were inserted into the zincate-poor side of the cell under an argon atmosphere and the voltage recorded at various time intervals.

4. 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, 1964.

DIFFERENTIAL PULSE POLAROGRAPHY (DPP). The DPP calibration curve was established using 10^{-3} M to 10^{-5} M zinc ion in 0.8 M KOH and recording the curves with a PAR model 174A DPP instrument. The cell employed a working dropping mercury electrode (DME), a platinum wire counter electrode, and a Hg/HgO reference electrode. All the solutions were deaerated and blanketed with water saturated argon before recording.

In diffusion studies, if the concentration of the KOH employed is less than about 16%, then the DPP measurements can be made insitu. For studies employing a DME electrode in higher KOH concentrations, either another electrode must be used or a portion of the zinc-poor solution must be sampled, diluted and then measured in a separate cell.

PROCEDURE. A membrane material consisting of a 0.785 in^2 surface area was inserted between two cell compartments. The zinc-rich side contained 250 ml of 1M ZnO in 45% KOH whereas the zinc-poor side contained 250 ml of 45% KOH. At intervals, the zinc-poor solution was stirred and a 2 ml aliquot was removed and placed in a polarographic cell containing 25 ml of distilled water. After purging with water soaked argon, the DPP curve was recorded. In order to prevent osmotic pressure generated fluxes of zincate, approximately 2 ml of the zincate-rich solution was simultaneously removed. A correction factor, $0.250 - 0.002\ (n-1)$ where n is the n^{th} aliquot removed, was applied to correct the volume in determining an accurate value of the zincate flux. The flux was reported as moles Zn^{++}/min in^2.

DISCUSSION OF RESULTS

In the process of evaluating new membrane materials for battery separators, we employed the standard potentiometric method[4] for determining rates of diffusion of soluble zinc through these membranes. However, we experienced difficulty in obtaining consistent data. This was attributed to several factors that affect the potentiometric calibration curve. The principal problem encountered was in obtaining a reproducible voltage-concentration calibration curve. Only over the higher zinc ion concentrations from 1.0 M to 10^{-2} M Zn^{++} was the potentiometric method found to be reversible (obeying the Nernst equation) and reproducible. Table 1 illustrates the irreproducible and non-Nernstian behavior observed for more dilute zincate solutions. Table 1 represents the average of six measurements using new zinc amalgam electrodes each time.

Diffusion studies are typically made in the 10^{-3} M to 10^{-4} M zincate concentration region. The irreproducibility of the non-Nernstian 15 mv potential difference over this concentration region is confirmed by the high standard deviation. This poor precision is further accented by the 8 and 10 mv range that was observed between the high and low values of the measured potentials.

Several experimental difficulties were observed to contribute to the irreproducible nature of the potential-concentration curve. If one desires to reuse the zinc amalgam, selection of an appropriate solvent (neutral, acidic or basic) and the extent of exposure to oxygen affects the electrode behavior. At the low concentrations (10^{-4} M), slow dissolution of zinc from the amalgam, the proximity of the reference and the speed of stirring the solution affect the observed potential.

Differential pulse polarography was investigated as an alternate method. It is inherently more sensitive, routinely detecting 10^{-5} M concentrations. However, the question remained as to the reproducibility (precision) and convenience of the method.

The method is less convenient for measuring zincate in solutions containing more than approximately 16% KOH. The polarographic half wave potential and the DPP peak potential of the zinc ion becomes more negative with increasing KOH concentration until it eventually merges with the solvent reduction potential and becomes immeasurable at a DME electrode. Therefore, for concentrated KOH solutions, an aliquot of the solution must be removed and placed into a separate cell for measurement. The measuring cell contains the electrodes and 25 ml of deaerated water.

In order to demonstrate the reproducibility of the DPP method, two typical concentrations, 7.5×10^{-4} M and 7.5×10^{-5} M Zn^{++}, were selected. Three distinct standard solutions of each were prepared and five to six values of the peak current for each solution was measured. The results are shown in Table 2.

The precision of the two methods can be observed by comparing Tables 1 and 2. For a 10-fold concentration change and a corresponding potential change of 15 mv, the potentiometric method exhibits an 8 to 10 mv range. This contrasts with the very low 0.1 to 0.8 ua range observed over a 36 ua difference for the DPP method over a similar 10-fold concentration change.

We wished to compare the precision and accuracy of the zincate flux across a membrane using the potentiometric and DPP methods. Cellophane and silvered cellophane were selected as membrane materials because of their homogeneity. An example of a zincate flux measurement for each method is illustrated in Figure 1. The scatter and/or curvature was found to be characteristic of the potentiometric method. This arises from the irreproducibility of the potentiometric calibration curve. Considering the poor precision of the method (Table 1), the accuracy of flux values obtained by this procedure is questionable.

The excellent precision of zincate flux measured across cellophane and silvered cellophane by the DPP method is illustrated in Table 3.

CONCLUSION

The potentiometric method, although convenient, has been found to be subject to numerous experimental difficulties that can lead to variable flux values, easily diverging by a factor of two. This method may be viable as a screening method when relative flux values of two or more membranes are to be compared. However, in reporting accurate flux values, a method such as the DPP method is recommended.

Table 1. Reproducibility of the Potential of the Zinc-Zincate Couple in 45% KOH

Zn^{++} (moles/liter)	Potential*(volts)	S	Range(mv)
10^{-2}	1.433	.0036	8
10^{-3}	1.459	.0040	10
10^{-4}	1.474	.0031	8

* Average of six measurements
S - Standard deviation

Table 2. Reproducibility of the DPP Method for Measuring Zn^{++} in KOH

(7.50×10^{-4} M Zn^{++})

Solution	Peak Current (ua)*	S	Range (ua)
1	41.09	0.28	0.62
2	40.03	0.38	0.75
3	40.40	0.31	0.75
AV.	40.51		

(7.50×10^{-5} M Zn^{++})

Solution	Peak Current (ua)*	S	Range (ua)
1	4.171	0.026	0.040
2	4.122	0.061	0.124
3	4.223	0.100	0.150
AV.	4.138		

* Average of 5 to 6 measurements
S - Standard Deviation

Table 3. Reproducibility of the DPP Method in Measuring Zincate Flux through Membranes

Membrane Sample	Zincate Flux ($^{moles}/_{min\text{-}in^2}$)* Cellophane	Ag-Cellophane
1	7.15×10^{-6}	5.83×10^{-6}
2	7.06×10^{-6}	6.28×10^{-6}
3	6.46×10^{-6}	5.28×10^{-6}
4	6.55×10^{-6}	-
5	6.46×10^{-6}	-
AV.	6.72×10^{-6}	5.78×10^{-6}

* The flux was measured through a 0.785 in^2 oriface. The average wet thickness for cellophane was 3.36 mil and for Ag-cellophane was 3.06 mil. The data has been reported as moles/min.-in^2 for a 1.0 mil membrane thickness.

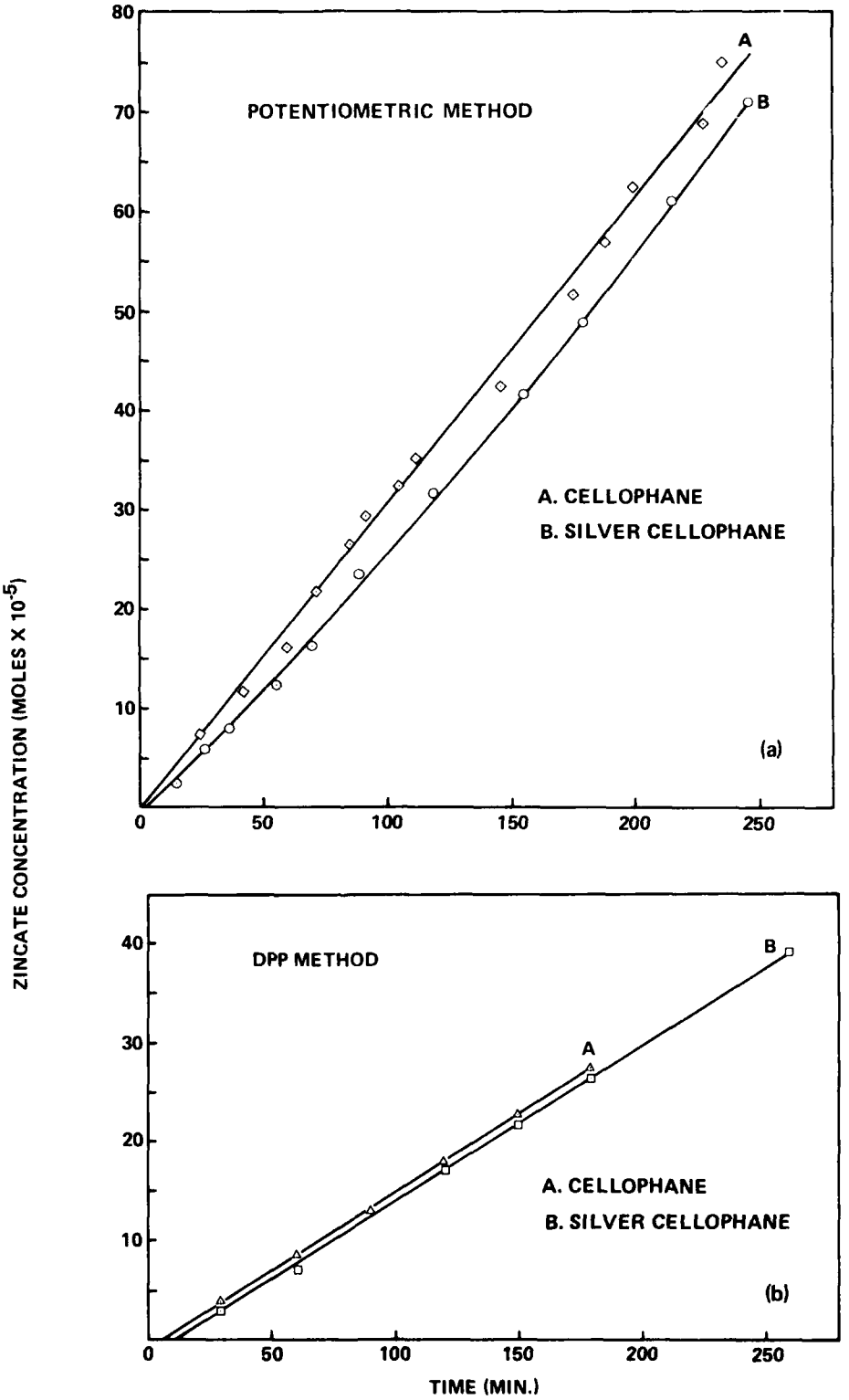

FIGURE 1 COMPARISON OF ZINCATE FLUX THROUGH CELLOPHANE MEMBRANES

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