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CROSS-LINKED POLYVINYL ALCOHOL FILMS

AS ALKALINE BATTERY SEPARATORS*

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SUMMARY

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Cross-linking methods have been investigated to determine their effect on the performance of polyvinyl alcohol (PVA) films as alkaline battery separators. The following types of cross-linked PVA films are discussed: (1) PVA-dialdehyde blends post-treated with an acid or acid periodate solution (two-step method), (2) PVA-dialdehyde blends cross-linked during film formation (drying) by using a reagent with both aldehyde and acid functionality (one-step method). Laboratory samples of each cross-linked type of film were prepared and evaluated in standard separator screening tests. Then pilot-plant batches of films were prepared and compared to measure differences due to the cross-linking method. The pilot-plant materials were then tested in nickel oxide - zinc cells to compare the two methods with respect to performance characteristics and cycle life. Cell test results are compared with those from tests with Celgard.

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Key-descriptors: Alkaline batteries; Separators; Cross-linked polyvinyl alcohol; Nickel oxide - zinc cells.

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INTRODUCTION

The use of polyvinyl alcohol (PVA) as a separator in various alkaline battery applications is well known. PVA film separators are used because of their insolubility and high ionic conductivity in alkaline electrolytes. Further, PVA film is easily fabricated on a commercial scale from aqueous solution and presents neither a health hazard to personnel nor a pollution threat to the environment.

Cross-linking PVA makes it water insoluble, increases its mechanical strength, and improves its oxidation resistance (ref. 1). Methods of cross-linking PVA have been reported by Philipp and Hsu (ref. 2) with applications for use in alkaline electrolytes. More recently, low-molecular-weight PVA films cross-linked by using dialdehydes, aldehydes with acid functional groups, and polysaccharides (aldehyde precursors) were evaluated as alkaline battery separators by Hsu and Sheibley (ref. 3). This work showed that cross-linked PVA films exhibit acceptable properties in comparative separator screening tests and perform in an acceptable manner as separators in silver oxide - zinc (Ag-Zn) and nickel oxide - zinc (Ni-Zn) cells from the standpoints of cycle life and cell performance.

This paper compares the effect of two cross-linking methods on PVA film properties measured for alkaline battery separators. One cross-linking method involves a two-step process: First, the film is made; then the dry film is treated with acidic solutions to achieve the desired cross-linking. The second method is a one-step method developed at the NASA Lewis Research Center to cross-link PVA films during commercial production operations. The PVA film is cross-linked during conventional oven drying; thus no further treatment is required.

Separator properties were measured for films made by the two-step method where the cross-linking reaction is catalyzed by either acid or acid periodate solutions. Two dialdehyde cross-linking reagents were compared. Then the effect on separator properties of glyoxylic acid (the reagent used to convert the two-step method into a one-step method) was evaluated in both methods.

Finally films made on pilot-plant equipment were evaluated to measure the effect of the two cross-linking methods on separator properties. These films were then performance characterized and cycle tested in small Ni-Zn cells to compare the two cross-linking methods and to compare the cross-linked PVA films to Celgard.

MATERIALS AND PROCEDURE

Materials

A commercially available polyvinyl alcohol (PVA), Elvanol 71-30, a medium-viscosity-grade PVA, was used for this work. This material is greater than 99 percent hydrolyzed. A 4 percent aqueous solution has a viscosity of 28 to 32 cP. The 1,2 diol content was 3.99 groups per 100 monomer units.

The furan 2,5-dialdehyde (DAF) was supplied by Quaker Oats Chemical Co. The terephthalaldehyde (TPA) was obtained from Fisher Scientific. The structural formulas of DAF and TPA are shown in table I. The glyoxylic acid (GOA) also came from Fisher (a 50 percent solution in water). The structural formula of GOA is shown in table II. The sucrose was cane sugar. The filler materials, shown in table III, came from commercial sources. The (143) filler combination was 7.9 vol % SiO_2 (Cab-O-Sil M, Cabot Corp.) and 92.1 vol % 200-mesh white pine wood flour (Wood Flour, Inc.). The (144) filler combination was 7.8 vol % SiO_2 and 92.2 vol % calcium zirconium silicate (TAM Products,

Hightstown, N.J.). The acid cross-linking solution contained 20 milliliters of concentrated H_2SO_4 and 200 g of Na_2SO_4 dissolved in 1 liter of water. The acid periodate solution contained 30 g of KIO_4 , 50 milliliters of concentrated H_2SO_4 , and 200 g of Na_2SO_4 dissolved in 1 liter of water.

General Procedure for Preparation of Aldehyde Cross-linked
PVA Membrane Separators

Two-step cross-linking method. - A quantity of the dialdehyde or aldehyde precursor is added to an aqueous solution of 10 wt % Elvanol 71-30 in order to achieve the theoretical amount¹ of cross-linking desired (usually 2 to 10 percent cross-linker). The solution is well mixed. Entrapped air bubbles are removed by placing the solution in a bell jar under a vacuum. The solution is cast on a smooth, clean, glass plate by using a Gardner blade. The cast film is air dried at room temperature, usually overnight. For films with the dialdehyde cross-linking reagent, cross-linking is achieved by immersing the dry film in a solution of sulfuric acid (pH, 4 to 5) saturated with sodium sulfate and then gradually increasing the temperature to 90° C over a period of 4 to 16 hr. The films are then rinsed in water to remove the sodium sulfate. The treated film is immersed in boiling water; if it is cross-linked, it will not dissolve. This is a qualitative test for cross-linking.

¹The theoretical amount of cross-linker desired is the percentage of cross-linking reagent required to chemically react with all the hydroxyl groups present in the PVA; e.g., 2 percent cross-linking would be 0.02 times the amount required to react with 100 percent of the PVA hydroxyl groups, assuming one aldehyde group per two hydroxyl groups.

For films containing an aldehyde precursor (e.g., polysaccharides) or where cleavage of the 1,2-diol units of the PVA is desired, cross-linking is achieved by immersing the film in the acid periodate solution (pH, 4 to 5). The same heat-treatment and rinsing step and the boiling water test for cross-linking are performed on the film.

One-step cross-linking method. - A quantity of dialdehyde is added to an aqueous solution of 10 wt % Elvanol 71-30 in order to achieve the theoretical amount of cross-linking desired, and the solution is well mixed. Then, a quantity of acid-aldehyde reagent, glyoxylic acid, is added to attain the theoretical amount of reaction desired, and the solution is well mixed. The amount added is sufficient to produce a pH value below 5. The entrapped air bubbles are removed by vacuum. The film is then cast on a clean, smooth, glass plate by using a Gardner blade. The film is cast before the mixture begins to gel. At a solution temperature of 120° F, gelation starts about 45 min after addition of the GOA. After the film is cast and air dried, the glass plate is placed in an oven for a period of time, the length of which depends on the drying temperature. Time ranges from 4 hr at 90° C to 5 min at 160° C. The film is placed in boiling water to check for cross-linking.

Screening tests of cross-linked PVA separators. - The cross-linked PVA films were evaluated by determining their ionic resistivity (dc method), zinc dendrite penetration rate, and zincate ion diffusivity according to methods described in reference 4.

Cell test evaluation of cross-linked PVA separators. - Cell test evaluation of the cross-linked PVA film separators was performed in nickel oxide - zinc (Ni-Zn) cells of approximately 7.2-A-hr capacity. The electrolyte was 35 wt % KOH. Ampere-hour efficiency reached 95 percent after three formation cycles. Overcharge was limited to 10 percent of the nominal capacity.

The Ni-Zn cell electrode configuration was Ni-Zn-2Ni-Zn-Ni. Electrode sizes were 7.3 cm by 9.4 cm with thickness ranging from 0.075 cm for the nickel electrode to 0.225 cm for the zinc oxide electrode. Performance was characterized at discharge rates of C/8 (the 8-hr rate, the current required to discharge the cell capacity in 8 hr), C/4, C/2, C, and 2C. Midpoint voltage (the voltage after 3.6 A-hr of capacity was removed) was recorded as was the ampere-hour capacity above 1 V.

Cycle testing consisted of 8-hr cycles (6-hr of charge (C/12 rate) and 2 hr of discharge (C/4 rate)). The discharge current density was approximately 7 mA/cm². The testing was continuous: three cycles per day, 7 days per week. Depth of discharge was 50 percent on the basis of the nominal 7.2-A-hr capacity.

RESULTS AND DISCUSSION

The two-step method of cross-linking PVA uses either an acid solution or an acid periodate solution. The acid solution is used when a dialdehyde is incorporated in the film. The acid catalyzes the acetalization reaction and this leads to cross-linking. The acid periodate method is used to cross-link PVA under several conditions. It can be used to cross-link PVA alone through oxidative cleavage of its 1,2-diol units in order to form aldehyde groups that react with the secondary alcohol groups of PVA (ref. 1). The addition of aldehyde precursors (polysaccharides such as sucrose or alginic acid) to PVA requires the acid periodate treatment to cleave the saccharide ring structure, resulting in aldehyde formation and cross-linking of PVA (ref. 5). Dialdehydes can also be used in the acid periodate method.

The effects of the acid and acid periodate catalysis methods were compared in reference 3 for cross-linked PVA films made from low-viscosity PVA.

Results indicated that periodate-treated PVA films exhibited higher ionic resistivity but longer cycle life in Ag-Zn and Ni-Zn cells than did acid-treated films.

Two-Step Cross-Linking Methods

Table I compares the two-step cross-linking method using either acid or acid periodate and two cross-linking reagents: furan 2,5-dialdehyde (DAF) and terephthaldehyde (TPA) in medium-viscosity PVA. Properties measured were volume resistivity, zinc dendrite penetration rate, and zincate diffusivity. The data show that resistivity is increased by use of acid periodate treatment. However, periodate treatment results in a longer time for zinc dendrite penetration and a lower zincate diffusion rate. Un-cross-linked PVA has generally higher values of resistivity and zinc dendrite penetration rate, but the unaltered PVA structure shows the lowest zincate diffusion rate. From these data it appears that the cross-linked films generally represent an improvement in PVA used as an alkaline separator. The structures of the DAF and TPA are shown beneath table I.

The data presented in table II show the effect of incorporating GOA into the PVA. The reagent contains both an aldehyde and a carboxylic acid group. The carboxylic acid group affects the resistivity and zincate diffusion. The reaction of the aldehyde with PVA can follow two routes: either form the cyclic acetal with PVA (the intramolecular reaction) and/or cross-link it (the intermolecular reaction). The films were cast with the solutions buffered to a pH of 7 with ammonia solution. The dry films were then treated with the acid periodate solution according to the method described in the procedure section. The extent of cross-linking as compared with cyclic acetal formation with the GOA is not known. The percentage of GOA shown in table II represents

the percentage of the theoretical amount needed to cross-link all the OH groups on the PVA.

As the amount of GOA in the film increases, the volume resistivity decreases with a corresponding increase in zincate diffusion rate. Between 10 and 50 percent GOA, the resistivity is halved (24 ohm-cm to 13 ohm-cm) while the zincate diffusion rate increases approximately 10 percent. The use of smaller amounts of GOA appears to represent a method of resistivity control with minimal change in zincate diffusion.

The effect of GOA on lowering resistivity is very pronounced in the data shown in table IV. The films contained 2 wt % sucrose in PVA with the amounts of GOA required to achieve the percentages of the theoretical amount to react with all the PVA. The film-casting solution was buffered with ammonia solution to a pH of 7. The dry film was then treated with acid periodate solution to cross-link the PVA through the tetra aldehyde fragments resulting from oxidative cleavage of the sucrose ring structure (ref. 5). The resistivity is decreased significantly (approx 90 percent) over the range 10 to 35 percent GOA. However, zincate diffusivity increases approximately 20 times over the same range. But the zincate diffusivity does not approach the 2×10^{-6} mole/cm² min value of commercially available film separators (ref. 6) until about 70 percent GOA in the film is reached.

One-Step Cross-Linking Method

The two-step methods of cross-linking were judged not commercially desirable because the dry films when immersed in the acid treatment solutions lost much of their strength. They tore easily when handled. The use of machine processing appeared very unlikely. An evaluation of a batch-processing method to achieve cross-linking showed that a wound roll of un-cross-linked PVA film

required a layer of material to separate the layers of PVA film to permit penetration of the acid treating solution and to prevent "adhesion" of adjacent film layers of PVA because of the cross-linking reaction. Further, batch processing would lead to nonuniform cross-linking as a result of varying solution concentration and temperature.

An apparent manufacturing method was found when the one-step cross-linking method described in the procedure section was worked out. The GOA, selected as the model reagent, was used to convert the cross-linking method to a one-step film-manufacturing process. The GOA contains the necessary acid to catalyze the cross-linking reactions; and since GOA reacts chemically with PVA, it leaves no residual products in the film that need to be rinsed out. The only constraints on the film-casting process are time and temperature. The film has to be cast before gelation starts in order to prevent casting a film containing domains of cross-linked product. As temperature is lowered, the time to gelation increases.

The acid periodate cross-linking treatment does not appear easily adaptable to the one-step method. The oxidation-reduction reaction involving the hydroxyl and the periodate leaves an iodine reaction product in the membrane. This can be removed by rinsing with water but involves an additional treatment step. This approach was not pursued.

In table III the effect of the cross-linking methods (two step vs. one step) on resistivity and zincate diffusivity is measured for two cross-linking reagents (DAF and TPA) and on films with 2 wt % DAF and 5 wt % fillers. The one-step method generally results in a cross-linked film having a higher (but acceptable) resistivity and a lower zincate diffusion rate.

Evaluation of Pilot-Plant Materials

Two pilot-plant batches of film materials were processed. The purpose was to establish the feasibility of the one-step method on a PVA-film-casting machine. The compositions of the two materials are shown in table V. The major difference in composition was the 13 wt % GOA (manufacturer error, should have been 10%) added to the one-step material to cross-link it during the drying process. The one-step material came off the line as a cross-linked product. The two-step material was processed and dried and subsequently cross-linked by using the acid treatment procedure.

The two cross-linked films were then compared. The data on the two-step film in table VI are quite similar to the 2-wt %-DAF two-step data in table III. The area resistivity of 0.037 ohm-cm^2 shown in table VI is quite close to the 0.04 ohm-cm^2 value for the laboratory-prepared sample. The 6.8×10^{-7} -mole/ $\text{cm}^2 \text{ min}$ zincate diffusion values are identical. Comparing the measured separator properties of the PVA films in table VI shows no significant difference in properties when films are made on pilot-plant equipment. Table VI also contains measured properties of Celgard 3401 and Cellophane, which are commercially available alkaline battery separators. The PVA films exhibit lower resistivity values, lower zincate diffusion rates, and equivalent hydroxyl diffusion rates. The measured rate of water diffusion was approximately 50 percent higher in the one-step cross-linked PVA than in Celgard.

The two PVA films were compared with Celgard in cycle tests in 7.2-A-hr Ni-Zn cells. The construction of these cells differed from that of the cells described in reference 7 in that the nickel electrodes were contained in an absorber bag of beater-treated, fuel-cell-grade asbestos (ref. 6), while the zinc electrodes each received two wraps of the film separator (the PVA or the Celgard).

A plot of discharge voltage as a function of ampere-hour capacity is shown in figure 1 for the two types of cross-linked PVA films and Celgard. The ampere-hour capacity was measured to a 1-V cutoff. The C/2 discharge rate was 3.6 A (approx 14-mA/cm^2 current density) for a constant current discharge. An examination of figure 1 shows that although the ampere-hour capacities did not vary much for the cells, the PVA films showed a midpoint voltage that was approximately 50 mV higher than that of the Celgard film. The plots in figure 1 were taken from cycle 6, the C/2 rate performance characterization cycle.

Results of cycle testing in Ni-Zn cells are shown in figure 2. The PVA film from the one-step method was cycle tested in cells built by two suppliers (A and B). The average (and standard deviation) of two cells built by supplier A was 117 (± 4) cycles; the average of three cells from supplier B was 116 (± 10) cycles. A combined (A + B) average of 116 (± 8) is shown in figure 2. The average cycle life for the two-step method was 108 (± 4) cycles based on three cells from supplier A. The 80-cycle result for Celgard is based on one cell from supplier A.

From the results of performance characterization and cycle life tests in small Ni-Zn cells, the one-step method of cross-linking provides essentially the same results as the two-step method. Hence it appears that cross-linked PVA film made on commercial production equipment has acceptable separator properties and performs at least as well as the commercially available separators to which it was compared.

CONCLUDING REMARKS

The development work on cross-linked polyvinyl alcohol (PVA) films for use as alkaline battery separators has produced the following results:

1. Chemically cross-linked PVA films generally exhibited better separator properties and better cell performance than un-cross-linked PVA.

2. The introduction of carboxylate functional groups to the cross-linked film reduced resistivity and, when used in smaller amounts (<35 percent theoretical), did not appear to alter other film properties significantly.

3. A one-step cross-linking method appears adaptable to the commercial PVA film production process.

4. Cross-linked films produced on pilot-plant equipment by the one-step method did not exhibit significant differences from films similarly made by a two-step process.

These films are now under further development by W. R. Grace & Co., Columbia, Md., under a cost-sharing contract with NASA (Contract NAS 3-22223). Producing a cross-linked film in a one-step process during conventional film drying opens up other areas for separator development. An obvious approach would be to apply the separator directly to the electrode as a result of a multiple dipping operation, using drying steps between dipping steps to achieve a satisfactory, defect-free separator.

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TABLE I. - RESULTS OF TREATING PVA^a-DIALDEHYDE BLENDS WITH
ACID AND ACID PERIODATE SOLUTIONS

Cross-linker, percent ^b	Method	Volume ^c resistivity, ohm-cm	Zinc dendrite ^c penetration rate, cm/min	Zincate ^c diffusivity, moles/cm ² min
2 DAF ^d	Acid	10	1.0x10 ⁻⁴	2.6x10 ⁻⁷
2 DAF	Periodate	102	<9.0x10 ⁻⁵	2.2x10 ⁻⁷
2 TPA ^e	Acid	20	1.1x10 ⁻⁴	1.2x10 ⁻⁶
2 TPA	Periodate	54	5.8x10 ⁻⁵	7.2x10 ⁻⁸
None	Un-cross- linked	69	1.4x10 ⁻⁴	5.1x10 ⁻⁸

^aMedium-viscosity grade Elvanol 71-30.

^bPercentage of theoretical amount required to react with all hydroxyl groups in PVA.

^cWet film thickness ranged from 0.013 to 0.017 cm.

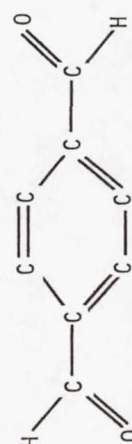
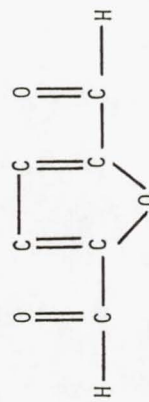
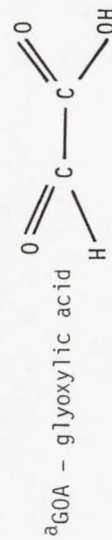


TABLE II. - EFFECT OF GLYOXYLIC ACID IN POLYVINYL
ALCOHOL ON RESISTIVITY AND ZINCATE DIFFUSIVITY
[Acid periodate cross-linked.]

GOA ^a content in film, percent ^b	Volume resistivity, ohm-cm	Zincate diffusivity, ^c moles/cm ² min
10	24	5.5x10 ⁻⁷
25	21	3.9x10 ⁻⁷
50	13	6.2x10 ⁻⁷
100	4	2.0x10 ⁻⁶



^bPercentage of theoretical amount required to react with all hydroxyl groups in PVA.

^cWet film thickness ranged from 0.013 to 0.017 cm.

TABLE III. - COMPARISON OF FILMS MADE WITH TWO- AND ONE-STEP CROSS-LINKING METHODS

Cross-linker, ^a percent	Volume resistivity, ohm-cm		Zincate diffusivity, moles/cm ² min	
	Two-step method	One-step method	Two-step method	One-step method
2 DAF	21	40	6.8×10^{-7}	3.1×10^{-7}
2 TPA	21	20	1.2×10^{-6}	4.7×10^{-7}
2 DAF + 5 wt % fillers (143)	5	17	1.6×10^{-6}	3.0×10^{-7}
2 DAF + 5 wt % fillers (144)	7	23	3.8×10^{-6}	6.9×10^{-7}

^aPercentage of theoretical amount required to react with all hydroxyl groups present in PVA.

TABLE IV. - COMPARISON OF POLYVINYL ALCOHOL FILMS WITH VARYING GLYOXYLIC ACID CONTENT

[Sucrose content, 2 wt %; wet film thickness, 0.014 to 0.018 cm; acid periodate cross-link.]

GOA content in film, percent ^a	Volume resistivity, ohm-cm	Zincate diffusivity, moles/cm ² min	Zinc dendrite penetration rate, cm/min
0	122	2.3×10^{-8}	$< 4 \times 10^{-5}$
10	28	3.1×10^{-7}	6×10^{-5}
35	14	5.5×10^{-7}	9×10^{-5}
70	12	3.9×10^{-6}	2×10^{-4}

^aPercentage of theoretical amount required to react with all hydroxyl groups present in PVA.

TABLE V. - COMPOSITION OF PILOT-PLANT
FILMS^a

[Film drying temperature, 350° F;
line speed, 25 to 30 ft/min; dry
film thickness, ~1.5 mils.]

Component	Two-step method	One-step method
	Composition, percent	
DAF	2.3	2.0
GOA	0	13.0
Triton X-100	.7	.6
Zelec UN	.56	.5
Film color	Yellow	Clear

^aElvanol 71-30.

TABLE VI. - PILOT-PLANT MATERIALS: MEASURED PROPERTIES OF CROSS-LINKED
PVA COMPARED WITH THOSE OF CELGARD AND CELLOPHANE

	Dry film thick- ness, cm	Area resis- tivity ohm-cm ²	Volume resis- tivity, ohm-cm	Zinc dendrite pene- tration rate, cm/min	Zincate diffu- sivity, moles/cm ² min	OH diffusivity, moles/cm ² min	H ₂ O diffusivity, moles/cm ² min
PVA (one-step method)	0.0044	0.038	8.6	2.0x10 ⁻⁴	7.1x10 ⁻⁷	2.6x10 ⁻⁴	4.3x10 ⁻⁴
PVA (two-step method)	.0036	.037	10.3	1.1x10 ⁻⁴	6.8x10 ⁻⁷	3.0x10 ⁻⁴	3.1x10 ⁻⁴
Celgard 3401	.0025	.078	31.0	8.5x10 ⁻⁴	29.4x10 ⁻⁷	2.4x10 ⁻⁴	2.5x10 ⁻⁴
Cellophane	.0025	.051	20.0	.4x10 ⁻⁴	22.9x10 ⁻⁷	2.3x10 ⁻⁴	-----

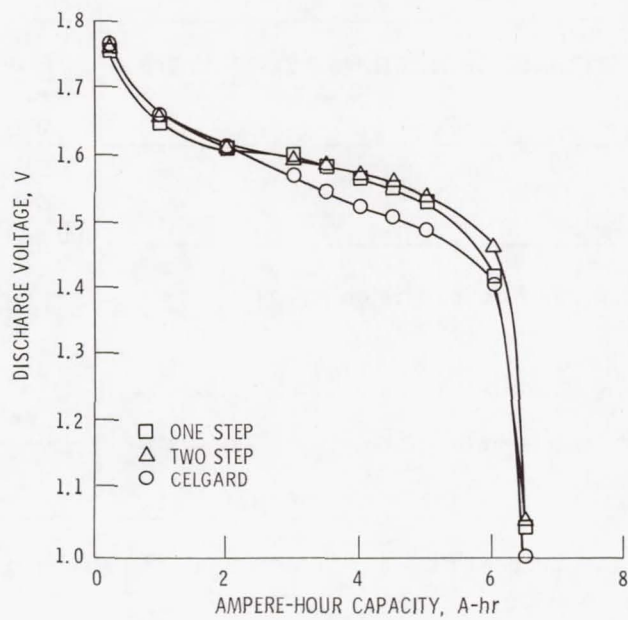


Figure 1. - Comparison of discharge voltage at C/2 rate - pilot-plant-made PVA cross-linked film and Celgard.

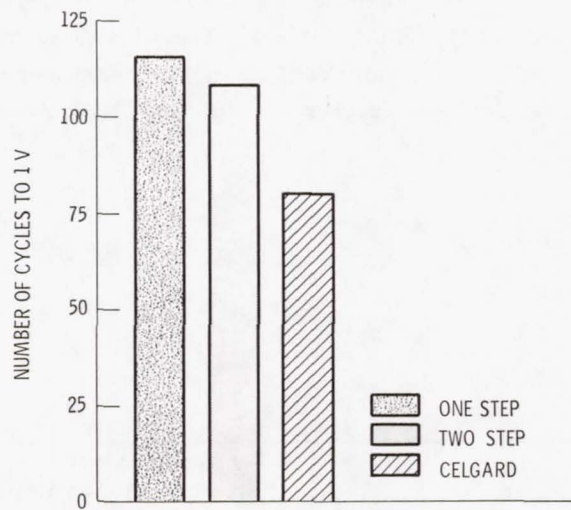


Figure 2. - Results of cycle life tests of pilot-plant-made PVA cross-linked film and Celgard - 7.2-A-hr Ni/Zn cells.

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