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CHEMICAL MODIFICATION OF POLY(ETHYLENE IMINE) FOR
POLYMERIC ELECTROLYTE(U) NATIONAL BUREAU OF STANDARDS
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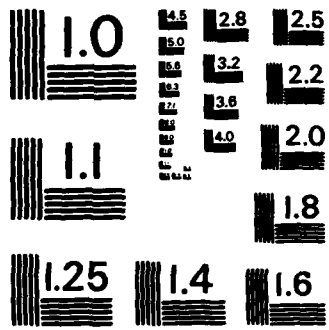
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Linear PEI has been chemically modified in an attempt to prevent formation of a crystalline complex without altering its ability to dissolve salts and conduct ions. Three main systems were investigated: (a) poly(N-acetylene imine), (b) partially quaternized PEI with ethyl or butyl groups, and (c) PEI cross-linked with diepoxyoctane. Dissolution of salt was followed by x-ray diffraction on the mixtures and changes in T_g as determined by DSC. In all cases, the crystallinity was destroyed but conductivity of salt-containing polymer was not improved. However, lightly cross-linked PEI exhibits much improved mechanical

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CHEMICAL MODIFICATION OF POLY(ETHYLENE IMINE) FOR POLYMERIC ELECTROLYTE

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Linear PEI has been chemically modified in an attempt to prevent formation of a crystalline complex without altering its ability to dissolve salts and conduct ions. Three main systems were investigated: (a) poly(N-acetylene imine), (b) partially quaternized PEI with ethyl or butyl groups, and (c) PEI cross-linked with diepoxycyclohexane. Dissolution of salt was followed by x-ray diffraction on the mixtures and changes in T_g as determined by DSC. In all cases, the crystallinity was destroyed but conductivity of salt-containing polymer was not improved. However, lightly cross-linked PEI exhibits much improved mechanical properties and the incorporation of .05 mole NaI/mole of monomer yields a conductivity of 5×10^{-5} S/cm at 100 °C.

1. INTRODUCTION

Polymers which dissolve salts and become ionic conductors show promise as solid electrolytes for high energy density batteries. Oxygen-containing polymers such as polyethylene oxide¹⁻³, polypropylene oxide^{2,4}, polyethylene succinate⁵, and polyphosphazene with ether side chains⁶ have received much attention in recent years as host polymers for dissolved salts. We have shown that linear polyethylene imine $(-CH_2CH_2NH-)_n$ can not only dissolve salts but in the case of sodium iodide, it forms a high melting crystalline complex⁷ analogous to that which has been observed between polyethylene oxide and a variety of alkali metal salts.

The linear polyethylene imine (PEI) which we investigated was of low molecular weight (about 2000). It was brittle when crystalline and paste-like when the crystallinity was destroyed upon incorporation of salts. In this paper, we report three modifications to the PEI and their effects on the physical and chemical properties of the polymer. The modifications were (1) acetyl side group left on the nitrogen which was an intermediate product

in the synthesis of PEI, (2) cross-linking with diepoxycyclohexane, and (3) quaternization with ethyl bromide and butylbromide.

2. PREPARATION OF POLYMERS

1. Linear PAEI and PEI

Linear poly(N-acetyl-ethylene imine), PAEI was synthesized following the procedure of Saegusa et al⁸ in which methyl iodide initiates the ring-opening polymerization of 2-methyl oxazoline in dimethyl formamide. Although this is the intermediate product in the synthesis of linear PEI, we did not examine its properties as a solid electrolyte until after we had investigated the linear PEI. In a second step, the linear PAEI was hydrolyzed in 2.5N NaOH at 100 °C to yield linear polyethylene imine. It was washed repeatedly with water until free of NaOH and vacuum dried at successively higher temperatures with final drying at 100 °C.

2. Cross-linked PEI

The rather low molecular weight linear PEI (about 2000) prepared as described was cross-linked with 1,2,7,8 diepoxycyclohexane in order to improve its mechanical properties.

Initially, the cross-linking was performed in methanol solution at 80 °C. Subsequent evaporation of solvent left a sponge-like material unsuitable as a solid electrolyte. Films for conductivity measurements were prepared by first dissolving salt and linear PEI in acetonitrile followed by evaporation of solvent which results in a non-crystalline paste. Small amounts of diepoxyoctane in methanol solution was stirred into the mixture, pressed into a film on a hydraulic press and cross-linked at 80 °C. The small amount of methanol was then removed by heating to 100 °C under vacuum. These polymers are referred to as PEI-DEO followed by the mole percent of diepoxide that was added.

3. Quaternized PEI

A portion of the imine groups on the linear polymer were converted to quaternary ammonium groups by reacting with excess butylbromide or ethylbromide in methanol at 80 °C for 24 hours. The solution product was precipitated from ether solution three times and finally dried under vacuum at 100 °C. Based on previous work⁹, one can expect 50% of the nitrogens to be quaternized. The quaternized polymers are designated PEI/BuBr and PEI/EtBr.

4. Polymer-salt mixtures

In all cases except the cross-linked polymer, the incorporation of salt in the polymer was accomplished by dissolving both polymer and salt in methanol for several hours at 80 °C. Films were cast from solution in an argon-filled dry box and dried in vacuum. Precautions were taken at all stages subsequent to the hydrolysis of the PAEI to prevent pick-up of moisture and a final drying step was performed with samples in the conductivity apparatus before measurements were taken.

3. EXPERIMENTAL

1. DSC Analysis

Differential scanning calorimetry was per-

formed on samples hermetically sealed in the dry box using a Perkin-Elmer DSC II¹⁰ equipped with a thermal analysis data station. Cooling capability extended to only -40 °C. Most of the samples examined are amorphous materials and the glass transition temperature is of primary interest from the DSC. Consequently, all samples were heated to 200 °C for 5 minutes and then quenched in the instrument at a setting of 320 °C/minute which seemed to be achievable from 200° to 0 °C but became slower than that at lower temperatures. Scans at increasing temperature were then obtained at a heating rate of 20 °C/min. The glass transition reported was taken as the temperature at the mid point of the increase in heat capacity which occurs at T_g .

2. Conductivity measurements

DC conductivity was deduced from Cole-Cole plots of impedance obtained over the frequency range from 100 Hz to 13 MHz using thin stainless steel discs pressed against the polymer films for electrodes⁷. Samples were exposed to air for a short period of time while loading into the glass apparatus but were dried in situ by evacuating for one day at room temperature followed by one day at 150 °C before commencing the conductivity measurements on the cooling cycle. Changes in temperature were made in increments of five to ten degrees allowing at least 30 minutes for equilibration.

4. RESULTS

DSC traces of linear polyethylene imine and each of the chemically modified polymers are shown in Figure 1. As expected, the heat of fusion of crystalline polymer is absent in each of the four modifications. The only feature present is a change in heat capacity at a temperature which we take to be the glass transition of the resulting amorphous polymers. We were unable to observe the glass

transition temperature of the semicrystalline linear PEI because there is so little of the non-crystalline component and it may be below the temperature range of our instrumentation. However, cross-linking with DEO destroys crystallinity and T_g is observed at a temperature which increases with the concentration of DEO. This variation of T_g with concentration of DEO is shown in Figure 2 which has been arbitrarily extrapolated to zero concentration to yield an estimated T_g of -35°C for the linear PEI.

The addition of .05 mole fraction of NaI to each of these polymers produces a change in T_g but the DSC traces are not shown since they are so similar to those of Figure 1. Thermal data are summarized in Table 1.

Values of DC conductivity measured on some of these polymers before and after the addition of .05 mole fraction NaI are plotted as a function of reciprocal temperature in Figure 3.

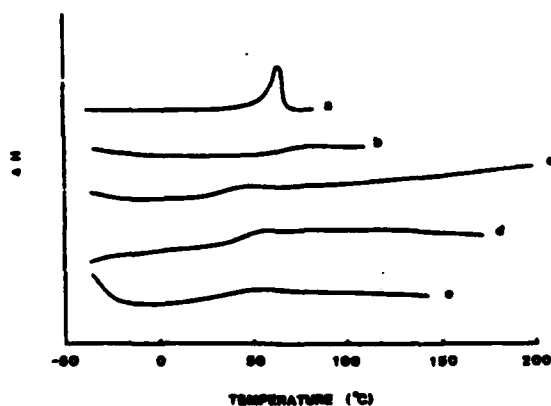


FIGURE 1
DSC traces of linear polyethylene imine (curve a), poly(N acetylene imine) (curve b), PEI quaternized with ethylbromide (curve c), PEI quaternized with butylbromide (curve d), and PEI cross-linked with 25 mole% diepoxycane (curve e).

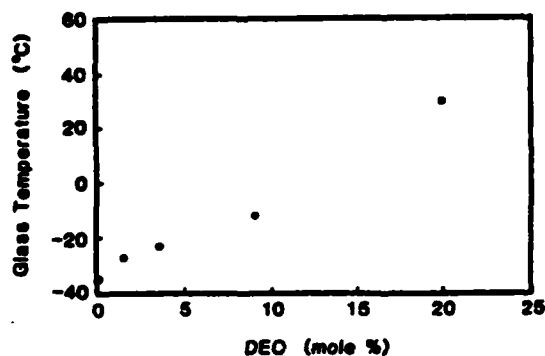


FIGURE 2
Glass transition temperature as determined by DSC versus concentration of diepoxycane used for cross-linking. Point at zero concentration deduced by extrapolation.

Table 1
Glass Transition Temperatures of
Some Modified Poly(ethylene imine)

Sample	PEI	PEI	PEI-EB	PEI-DB	PEI-DEO(1,2)
Side Group	H	CH ₃	(C ₂ H ₅) ₂	(C ₄ H ₉) ₂	(CH ₂), (CH ₂ CH ₂) ₂
T_g ($^{\circ}\text{C}$) (0.05 NaI)	-35	25	32	41	-19
T_g ($^{\circ}\text{C}$) (0.05 NaI)	-21	31	17	42	-19

5. DISCUSSION

Mixtures of linear poly(ethylene imine) and alkali metal salts exhibit many properties analogous to those of poly(ethylene oxide)-salt mixtures. For example, sodium iodide dissolves in the polymer, initially reduces crystallinity and increases ionic conductivity but at higher concentrations forms a high-melting crystalline phase in which conductivity is decreased⁷. Lithium salts also dissolve in the polymer, decrease crystallinity but do not form a crystalline complex¹¹. The linear PEI prepared as described above is a low molecular weight so that when crystallinity is destroyed upon dis-

solution of salt, the mixture is paste-like rather than a solid polymer electrolyte. When the crystal complex forms between PEI and NaI, the polymer becomes brittle and exhibits conductivity even lower than was measured before salt was purposely added. The purpose of this work was to modify the polymer so as to improve mechanical properties and prevent the loss of charge carriers to an immobile complex crystal phase. All of the modifications described resulted in disrupting the crystallinity and the formation of a crystalline complex but they also resulted in a large increase in glass transition temperature (see Table 1) and reduced mobility of charge carriers. The unhydrolyzed precursor of linear PEI

has a glass transition well above room temperature and the addition of .05 mole fraction NaI increases T_g to 81 °C which is too high to be practical in most applications envisioned for polymeric electrolytes. Quaternization with ethylbromide and butylbromide also increased T_g to the vicinity of room temperature. The small change upon addition of salt may result from low dissolution of salt. The conductivity of the PEI/BuBr and PEI/EtBr to which NaI has been added is not much larger than before the addition of salts. Data for the butyl quaternized polymer before and after addition of salt are presented in Figure 3. Note that the ethyl-quaternized polymer with NaI has nearly identical conductivity to that of the butyl-quaternized polymer with salt.

The most encouraging results were obtained with the lightly cross-linked sample. Addition of 3.5 mole% diepoxyoctane cross-linked the PEI sufficiently to increase the T_g to about -20 C which results in a flexible film at room temperature. Incorporation of .05 mole fraction NaI yields the highest conductivities shown in Figure 3. At the highest temperature shown, the conductivity is comparable to that of polyethylene oxide containing a mole ratio of NaI of 0.2¹². However, it should be noted that the conductivity of the cross-linked PEI decreases with temperature more than the PEI system and that incorporation of more salt increases T_g with a further reduction in conductivity.

6. ACKNOWLEDGEMENTS

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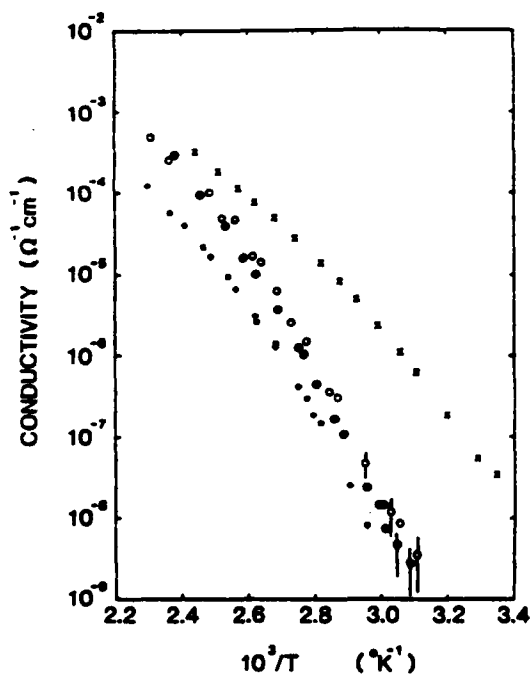


FIGURE 3
DC conductivity vs reciprocal of absolute temperature for modified polyethylene imines
● PEI/BuBr (no salt); ○ PEI/BuBr (.05 NaI);
○ PEI/EtBr (.05 NaI); x PEI/DEO-3.5% (.05 NaI).

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