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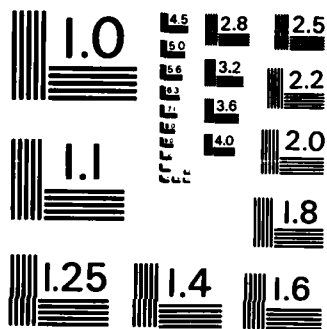
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Conductivity, DSC, FTIR, and NMR Studies of Poly(vinyl acetate)
Complexed with Alkali Metal Salts

by

John J. Fontanella and Mary C. Wintersgill

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In addition to the conductivity, NMR, FTIR, and DSC results are presented. All results are interpreted in terms of the model of Wissbrun and Hannon where water is the solvating species.



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CONDUCTIVITY, DSC, FTIR, AND NMR STUDIES OF POLY(VINYL ACETATE) COMPLEXED WITH ALKALI METAL SALTS*

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Electrical conductivity, differential scanning calorimetry, and nuclear magnetic resonance studies have been carried out on poly(vinyl acetate) after the addition of various lithium salts. In the case of lithium triflate, the resultant polymer is poly(vinyl alcohol). Relatively large conductivities are observed after initial preparation, but these decrease with time when the material is in vacuum. The conductivities exhibit approximately Arrhenius behavior and increases with salt concentration. In addition to the conductivity, NMR, FTIR, and DSC results are presented. All results are interpreted in terms of the model of Wissbrun and Hannon where water is the solvating species.

1. INTRODUCTION

Poly(vinyl acetate) (PVAc) is known to form complexes with inorganic salts.¹ Further, PVAc forms blends with the classic ion conducting polymer, poly(ethylene oxide) (PEO).² Consequently, new studies of the properties of ion containing PVAc have been undertaken. A preliminary note describing some of the properties of PVAc containing LiClO_4 has been published previously.³ In addition, Armstrong and Clarke have recently reported the electrical properties of salts in co-polymers of PVAc and poly(ethylene adipate).⁴ In the present paper, various properties of PVAc after the addition of several lithium salts are reported.

2. EXPERIMENT

The PVAc (MW 1.5×10^6) was obtained from Polysciences, Inc. All of the sample preparation procedures were carried out in a dry box or vacuum oven. The polymer and salts, LiCF_3SO_3 , LiClO_4 , and LiSCN were dissolved in

methanol at about 55°C and cast onto a teflon plate in a glass retaining ring. The samples were initially dried at room temperature in flowing dry nitrogen and then were transferred to a rough pumped vacuum oven for final drying at about 65°C. For all salts except LiCF_3SO_3 , the result was a clear film. For LiCF_3SO_3 a white precipitate was obtained which was vacuum dried and hot-pressed at about 100°C into a disk 25.4 mm in diameter and 1 mm thick. A clear, amber sample was obtained.

All DSC and electrical measurements were carried out using the equipment described elsewhere.⁵ The resultant complex impedance plots were similar to that shown in fig. 1 of ref. 5 and the techniques used to reduce the data are the same as in ref. 5. Some resultant conductivities are plotted in fig. 1 for various samples. Infrared (FTIR) data were obtained using a Perkin Elmer 1750 Infrared Fourier Transform Spectrometer. Thin samples were obtained by casting films on either AgCl ,

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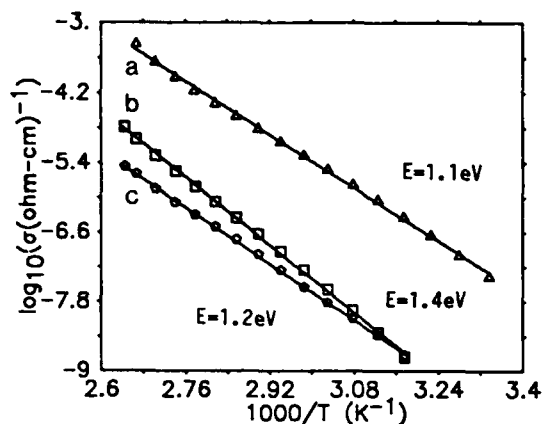


FIGURE 1

Electrical conductivity vs. reciprocal temperature for: (a) PVAc:LiSCN (1:1); (b) PVAc:LiSCN (2:1); (c) PVOH:LiCF₃SO₃.

AgBr, NaCl, or KCl windows which were dried in a vacuum oven. Nuclear magnetic resonance studies (NMR) were carried out using standard NMR pulse techniques. The samples were sealed in quartz tubes.

3. RESULTS AND DISCUSSION

3.1. LiSCN

As shown in fig. 1, all the samples containing LiSCN are conductive at high temperatures with the conductivity increasing as the amount of salt increases. In the case of LiSCN complexed materials, the maximum polymer:salt ratio was about 1:1. It is apparent that the conductivity for that material is quite high, reaching almost 10^{-3} (ohm-cm)⁻¹ at about 100°C.

The increase in the conductivity with concentration is in marked contrast to the polyethers where the conductivity goes through a maximum at fairly low salt concentrations.⁶ Next, when the materials are placed in a vacuum, the conductivity tends to decrease with time. A plot of the time dependence is shown in fig. 2. This immediately suggests that water is somehow associated with the conductivity. As seen by the FTIR traces in fig. 3, there are traces of water present even though extensive efforts were made to eliminate it.

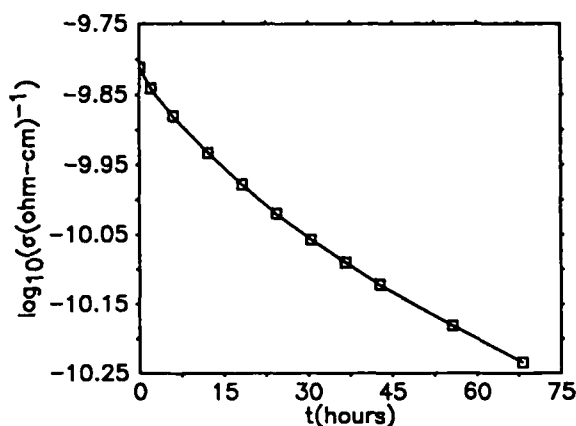


FIGURE 2

Electrical conductivity vs. time at 300K in vacuum for PVAc:LiSCN (2:1).

Further information concerning water in the samples is given by the DSC results shown in fig. 4 for a 2:1 sample. For comparison, DSC traces for both the host polymer and LiSCN are shown in fig. 5. If the polymer is exposed to the atmosphere for a very short amount of time, the glass transition temperature, T_g , drops. For example, fig. 4 shows that a typical dry box sample has a T_g of about 20°C but this drops to about -15°C after being exposed to the atmosphere for about 1/2 hour.

Consequently, one factor contributing to the increase in conductivity is the decrease in T_g . However, classic amorphous behavior is not observed since, as is apparent from fig. 1, the conductivity vs. temperature plots are Arrhenius. Note that the results shown in fig. 1 do not take the time dependence of the conductivity into account, and thus the shape of the curves is uncorrected for this factor. However, those effects are relatively small over the duration of a data run.

Next, the FTIR results in fig. 3 show that the carbonyl frequency decreases in wave number from 1737 cm^{-1} for uncomplexed PVAc to 1708 cm^{-1} for the 2:1 and 1:1 material. Also, the ether frequency increases from 1240 cm^{-1} to 1278 and 1284 cm^{-1} for the 2:1 and 1:1

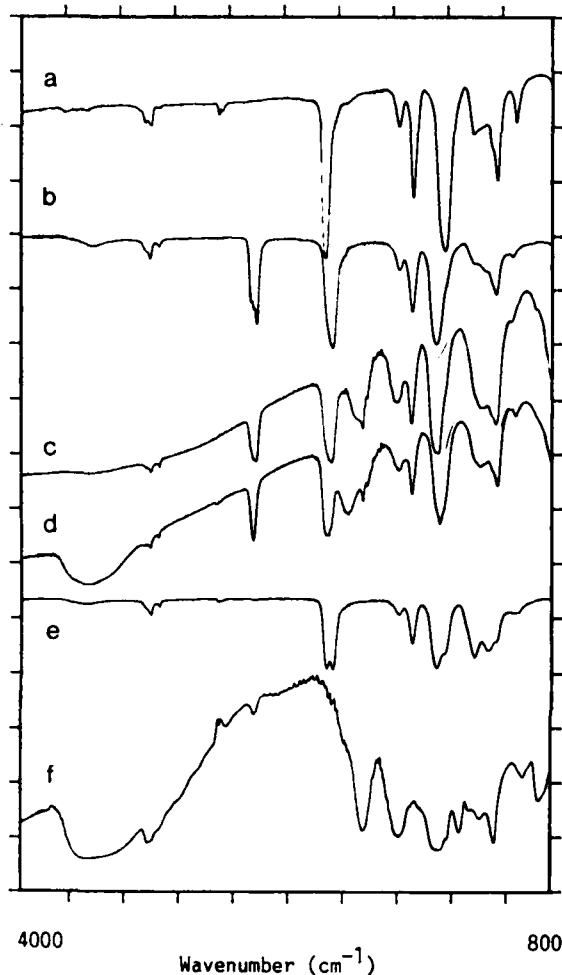
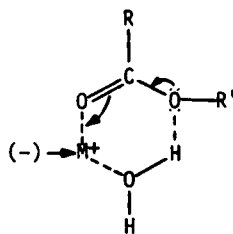


FIGURE 3

FTIR absorption spectra for: (a) uncomplexed PVAc; (b) PVAc:LiSCN (2:1), initially prepared; (c) PVAc:LiSCN (1:1), after drying at 120°C for 24 h; (d) Sample (c) after exposure to the atmosphere for 1 h; (e) PVAc:LiClO₄ (4:1), initially prepared; (f) material which results from adding LiCF₃SO₃ to PVAc.

material, respectively.

This behavior is consistent with the model proposed by Wissbrun and Hannon for PVAc polymer-salt complexes¹ shown to the right.



if the complexed phase is relatively non-conducting. Specifically, it is proposed that while water may be necessary for the complexation, excess water destroys it increasing the conductivity. Some evidence in favor of this model is contained in fig. 3 where the spectrum for a "wet" 1:1 sample is shown. Both the shifts in the carbonyl and ether frequencies are reduced by excess water indicating that the complexation is eliminated by excess water. The result is a highly conducting aqueous solution bound by the polymer.

The conductivity can be reduced even faster by heating to about 110°C in a vacuum. In fact, the conductivity for a 1:1 sample left at 120°C in a vacuum for 48 hours was 10⁴ lower than the initially prepared material. Part of the reason for the drop is shown in fig. 4c which shows a very high T_g of about 90°C. However, the samples also turn dark irreversibly suggesting decomposition. The FTIR results for the low conductivity PVAc containing LiSCN shown in fig. 3 suggest the mechanism for the decomposition. Specifically, IR absorption is induced at about 1600 cm⁻¹ and it appears that the acetate absorption decreases. Consequently, it is suggested that elevated temperatures may affect pyrolysis with the concomitant loss of acetic acid since the induced absorption occurs at about the position expected for an olefin. On the basis of the model described above, the cations would then be released. The conductivity would then be lowered even more rapidly than is expected on the basis of increased loss of water at high temperature if significant ion pairing subsequently takes place.

However, the nature of the charge carriers in PVAc has not yet been unambiguously identified. In fact, there has been a report of electronic conductivity in iodine containing PVAc⁷ In that case the temperature dependence did not resemble that observed in the present work. The pressure dependence of the conductivity gives some information concerning the nature of the charge carriers. Such experiments have been carried out and a value

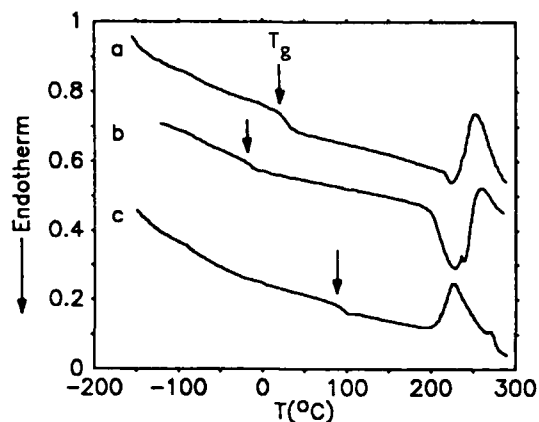


FIGURE 4

DSC thermograms for PVAc:LiSCN (2:1): (a) initially prepared; (b) after exposure to the atmosphere for 0.5 h; (c) after drying at 120°C for 24 h.

of $\ln G/dP = -9.8/GPa$ is obtained which is on the order of that observed in the polyethers^{5,8} and thus ionic conduction is inferred.

3.2. LiClO₄

The electrical conductivity in PVAc containing LiClO₄ also shows Arrhenius behavior as is apparent in fig. 1 of ref. 3. It was found that 4:1 material had a larger conductivity, however, higher concentrations were not tested. As is apparent from the FTIR traces in fig. 3, traces of water were present and thus the comments concerning the conductivity mechanism are the same as discussed above for PVAc:LiSCN.

In order to gain further information concerning the nature of these materials, NMR studies have been performed. Previous ⁷Li NMR measurements on 8:1 material demonstrated classic motional narrowing above room temperature, reflecting a corresponding increase in chain and ion mobility.³ Spin-lattice relaxation (T_1) data, obtained using a steady state $(\pi/2-\pi)_n$ pulse sequence, at a ⁷Li frequency of 155 MHz for both 8:1 and 4:1 are displayed in fig. 6a. The relaxation behaviors of both materials exhibit strong similarities, with one important exception. At lower temperatures, the 4:1 recovery profile indicates the presence

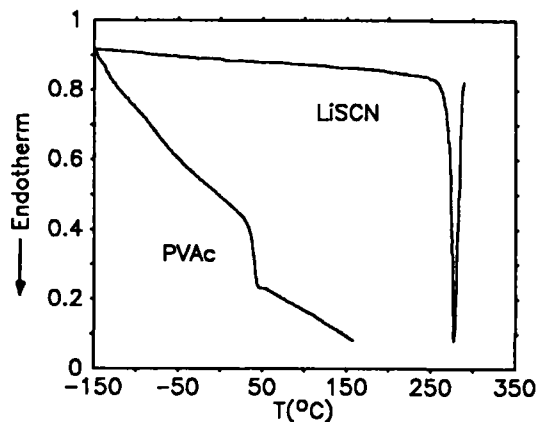


FIGURE 5

DSC thermograms for two of the starting materials used in the present work.

of at least two separate processes which differ in T_1 by an order of magnitude. For example, the 4:1 data at 234K, 263K, and 273K are best fit by two T_1 values: 59s, 8.2s (234K); 51s, 5.9s (263K); and 51s, 4.7s (273K). At higher temperatures ($\geq 330K$) the difference between the two T_1 's becomes less pronounced; for example the data at 405K are consistent with separate T_1 values of 4+2s and 1s. The 4:1 T_1 data in fig. 6a represent only the shorter values extracted from the recovery curves. Although slightly nonexponential relaxation was noted in the earlier 8:1 work,³ single-valued T_1 's were still obtainable. A comparison of the recovery profiles of 8:1 and 4:1 at similar temperatures, shown in fig. 6b, demonstrates the important qualitative difference between the two materials. Different T_1 's in the same sample (4:1) suggest the possibility of different Li-sites in the polymer complex. The presence of more than one Li-site in 4:1 is consistent with the model proposed above assuming the presence of excess water in the samples, the two sites being complexed and uncomplexed Li. The FTIR results shown in fig. 3 for the 4:1 material indicate that complexation takes place in that the carbonyl region is composed both of an unshifted (1734 cm^{-1})

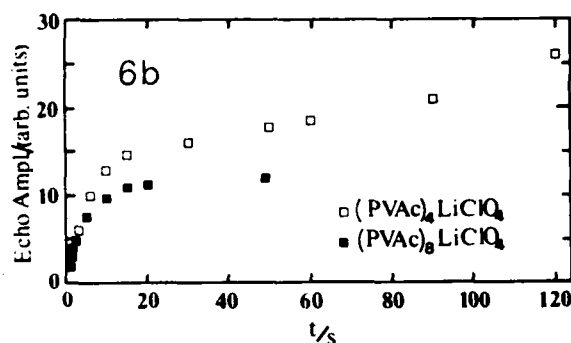
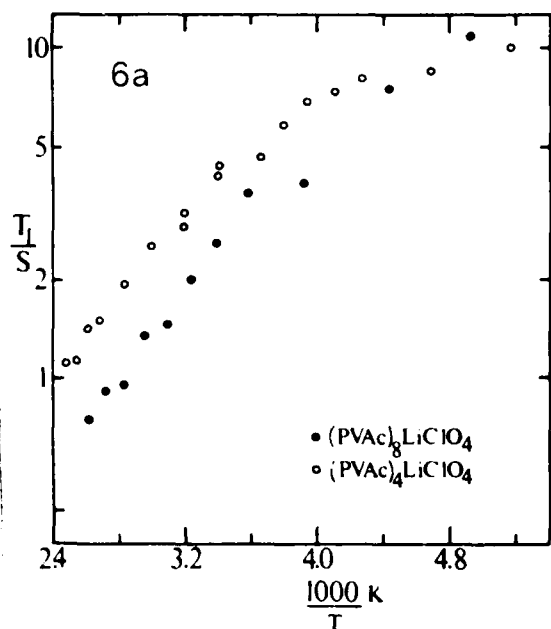


FIGURE 6

(a) ${}^7\text{Li}$ T_1 values for PVAc:LiClO₄ 4:1 (o) (only the shorter T_1 is included) and 8:1 (o). Least squares fits to the data yield activation energies of 0.11 eV for 4:1 and 0.14 eV for 8:1. (b) T_1 recovery profiles for 4:1 (o) at 254K and 8:1 (o) at 256K.

and a shifted (1708 cm^{-1}) peak. In addition, the ether absorption is complicated. It will be of interest to perform high resolution NMR measurements (i.e. magic angle spinning) in order to resolve the two sites structurally.

The temperature dependence of the ${}^7\text{Li}$ linewidths of 4:1 and 8:1 are shown in fig. 7. Both materials exhibit a comparable degree of

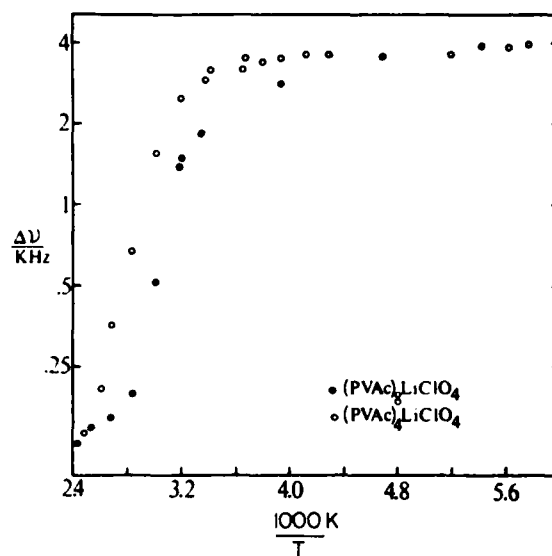


FIGURE 7

${}^7\text{Li}$ linewidth temperature dependence for PVAc:LiClO₄ 4:1 (o) and 8:1 (o).

motional narrowing above 320K. The rigid ($T_c \leq 220\text{K}$) linewidths in the two samples are also comparable. The ${}^7\text{Li}$ lineshape in the polymer complex is attributable primarily to the nuclear dipole-dipole interaction between ${}^7\text{Li}$ and ${}^1\text{H}$ (heteronuclear) and between ${}^7\text{Li}$ and ${}^7\text{Li}$ (homonuclear); where other possible interactions such as ${}^7\text{Li}$ - ${}^6\text{Li}$ or ${}^7\text{Li}$ - ${}^{13}\text{C}$ have been neglected. Careful measurements of $\pi/2$ pulse widths (relative to a liquid standard) and the absence of structures in the lineshapes suggest a negligibly small nuclear quadrupole interaction which, in turn, implies a highly symmetric environment for the Li^+ ions.⁹

It is possible to distinguish homonuclear from heteronuclear contributions to the linewidths simply by measuring the spin-spin relaxation time (T_2), where $\Delta\nu(\text{hom}) = 1/\pi T_2$. Rigid ($T = 178\text{K}$) T_2 values have been obtained for 4:1 (320 s) and 8:1 (410 s), using a Carr-Purcell-Meiboom-Gill pulse sequence. These results can be used in conjunction with a standard second moment formalism¹⁰ to deduce an average Li-Li separation, d_{Li} , in the complexes. The second moment contains the factor $\sum_k 1/r_{jk}^6$ where r_{jk}

are the internuclear distances. For simple cubic symmetry the summation reduces to $8.5d^{-6}$ where d is the lattice constant. Although there is no compelling physical justification for using this simple expression in our calculations, we have chosen to do so on the basis that departures from the numerical factor 8.5 will be reduced by the sixth root in the final result for d_{Li} . We obtain $d_{Li}=4.7\text{\AA}$ for 8:1 and $d_{Li}=4.3\text{\AA}$ for 4:1. Note that the T_2 measurements in 4:1 were performed so as not to saturate the long T_1 component, which would have yielded an erroneously longer T_2 . The observation that T_2 does not quite scale with Li-concentration in the two complexes implies that the Li is not uniformly or randomly distributed in the polymer matrix.

3.2. LiCF_3SO_3

Finally, the results for the conductivity of the as-prepared material obtained after adding LiCF_3SO_3 to PVAc are also shown in fig. 1. Once again the conductivities are reasonably high. However, that material also exhibits time dependence when placed in a vacuum.

In order to determine the nature of the material FTIR studies were also carried out and the results are shown in fig. 4. Clearly, the material is not PVAc. In fact, the FTIR studies indicate that the new material is probably poly(vinyl alcohol) (PVOH). Presumably, the triflate ion is sufficiently nucleophilic to remove the acetate moiety leaving lithium alkoxide at those locations where acetate cleavage occurs. That is not surprising as Wissbrun and Hannon studied complexing of salts with PVOH and found that the results were quite similar to that observed for PVAc.¹

4. CONCLUSIONS

In summary, then, the electrical properties of PVAc complexed with lithium salts have been studied. In the case of LiCF_3SO_3 , the resultant polymer is PVOH. Relatively large conductivities are observed after initial

preparation, but decrease with time when the material is in vacuum. Approximately Arrhenius behavior is observed and the conductivity increases with concentration of salt. In addition, NMR, FTIR, and DSC results have been presented. All results are interpreted in terms of the model of Wissbrun and Hannon where water is the solvating species.

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