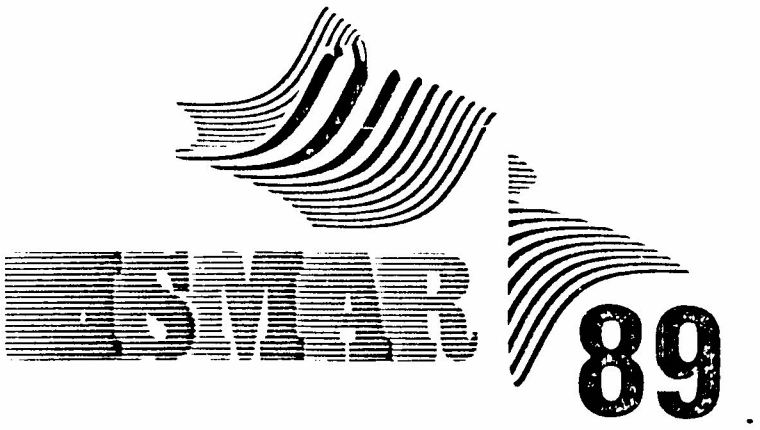

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ABSTRACTS

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NMR AND IR STUDY OF WATER EFFECTS OF LITHIUM SALT-PEO COMPLEXES

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The complexes between Poly(ethylene oxide), (PEO), and various lithium salts have raised a widespread interest because their high ionic conductivity at moderate temperatures coupled with the flexible nature of the films, made them potentially useful as electrolytes in high energy density battery applications[1]. Recently, this concept has been extended to humidity sensors[2]: thin films of lithium salt-PEO complex show a appreciable impedance variation with humidity, but the detailed mechanism of the water-polymer interaction are not fully understood. Here we present preliminary results of a Infrared (IR) and Nuclear Magnetic Resonance (NMR) study of the water effects in the amorphous system $P(EO)_6\ LiC_8F_{17}SO_3$ (T_g ~ 247 K).

Required amount of polymer and salt were weighted and dissolved in acetonitrile, under a dry atmosphere in a glove box, to the desired Lithium oxygen in PEO ratio. Three samples were prepared: (1) under anhydrous condition using double distilled acetonitrile as solvent; (2) under anhydrous condition using commercial grade acetonitrile; (3) same as sample (1) but exposing the film to a humid atmosphere (66% relative for 20 days).

The IR analysis was made at room temperature between 4200 - 600 cm^{-1}. The resulting spectra were similar to those observed in PEO:NaX and PEO:LiX complexed[3], but several differences between samples (1) and (3) were revealed. The hydrated sample (3) show hydrated lithium salt bands at 1620 and 3500 cm^{-1}, also observed by Weston and Steele[4], and their "high purity polymer". Slow heating of the film support cause a reduction in the intensity of these water bands, and after 30 minutes give a IR spectrum identical to that of sample (1). A strong peak at 1050 cm^{-1} in sample (1) was absent in the spectra of sample (3), and peak at 830 and 930 cm^{-1} were markedly reduced, suggesting that the form of the lithium salt-PEO complex may be influenced by the water addition.

The proton spin-lattice relaxation measurements were performed at 24.4 MHz in the temperature range 299 - 397 K. A $(T_1)^{-1}$ maximum, associated to the segmental motions of the chains, was observed around 317 K in samples (1) and (2) and around 358 K in sample (3). Activation energies may be estimated from the slope of the $\ln(T_1)^{-1}$ vs T^{-1} curve. From the high T side of the $(T_1)^{-1}$ maximum we have $E^{(1)} = 0.27$ eV and $E^{(2)} = 0.33$ eV. For sample (3), $E^{(3)} = 0.23$ eV was extracted from the low T side of the $(T_1)^{-1}$ maximum. This behaviour may be interpreted in terms of the expansion of the free volume by the plasticizing effect of sorbed water[2].

The IR spectra and the behaviour of the NMR relaxation rate are not fully understood yet, and further investigation, including DSC, X-ray and ^{19}F NMR are under course. (Partially supported by CNPq and FINEP).

References

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