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Conductivity, Mechanical and Thermal Studies on Poly(methyl methacrylate)-Based Polymer Electrolytes Complexed with Lithium Tetraborate and Propylene Carbonate

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A series of different composition ratio of polymer electrolytes based on poly(methyl methacrylate) (PMMA) as host polymer, lithium tetraborate ($Li_2B_4O_7$) as salt, and propylene carbonate (PC) as plasticizer is produced by solution casting method. Fourier transform infrared (FTIR) spectroscopy studies are used to confirm the formation of polymer electrolyte complex. PMMA: $Li_2B_4O_7$: PC (52.5:22.5:25.0 wt.%) is obtained as the highest conducting polymer electrolyte with a conductivity of 5.14×10^{-6} S/cm at room temperature (23 °C). The temperature-dependent conductivity of the polymer films shows Arrhenius-like behavior which reveals that the charge carriers move in a liquid-like environment. The addition of PC decreases the Young's modulus and stress at peak values of the complexes. Thermogravimetric analysis (TGA) is employed to study the thermal stability of the electrolytes.

Keywords conductivity, FTIR, mechanical studies, PC, PMMA

1. Introduction

The term "polymer electrolyte" refers to a solvent-free system where the ionically conducting phase is formed by dissolving salts in a high molecular weight polar polymers such as poly(vinyl chloride) (PVC) and poly(ethylene oxide) (PEO) (Ref 1-3).

The intense investigation and development of new polymeric electrolytes is motivated by the possibility of their potential applications in various electrochemical devices, high performance solid-state batteries, energy conversion by fuel cells, chemical sensors, electrochromic windows, analog memory devices and also because of their performance safety, ease in handling, shelf life, cyclability, and processibility (Ref 4-6). In recent years, the polymer-based solid electrolytes are of growing importance for rechargeable batteries with high specific energies (Ref 6-10). Ion conducting polymers have since been extensively investigated because of their application as an electrolyte in solid-state batteries. Compared to molten polymer electrolytes, solid polymer electrolytes have numerous advantages such as no leakage, volumetric stability, solvent-free condition, easy handling, and wide electrochemical stability window (Ref 11).

Early cells consisted of polymers singly or as blend complexes with suitable salt (Ref 12, 13). Although imparted good safety characteristics to lithium cells, such polymers possess very low room temperature conductivities. In order to improve the characteristics of both the electrolytes systems, a hybrid electrolyte concept was proposed (Ref 14-19). In this approach, the polymer matrix is swollen in a plasticizer, the latter being an aprotic solvent with a high dielectric constant. Plasticizers are additives that increase the plasticity or fluidity of the material to which they are added. The introduction of plasticizers with low molecular weight such as propylene carbonate (PC), and ethylene carbonate (EC) play an important role in conducting material with sufficient mobility of ionic conduction. The plasticizer would dissolve enough charge carriers and provide a more mobile medium for the ions so as to enhance the ionic conductivity of the resultant films (Ref 20). Plasticized electrolytes have the advantage over other polymer electrolytes with its relatively higher ionic conductivity. The disadvantages of plasticized electrolytes include poor mechanical properties due to high degree of plasticization, and the reaction of a polar plasticizer with the lithium electrode.

The high dielectric constant of both PC and EC effectively reduces the inter-ion Coulomb interactions. Hence, more cations of the salt contribute to the conductivity of the complexes.

Polymers that have been plasticized to improve its conductivity include poly(acrylo nitrile) (PAN), poly(methyl methacrylate) (PMMA), PVC, and PEO. PMMA is used in out-door electrical applications, high voltage applications, as transparent neutron stopper, standard broad cast television waves, radar bands, electrochemical windows, magnifiers, and automotive tail lights because of its good compatible nature with other polymers, high resistance, non-tracking characteristics, surface resistance, and optical properties (Ref 21).

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PMMA has an amorphous morphology and a polar functional group in its polymer chain that exhibits a high affinity for lithium ions and plasticizing solvents (Ref 22-26). PMMA as a host polymer was first reported by Iijima and Bohnke (Ref 27, 28). Appetecchi and his co-workers studied the kinetics and stability of the lithium electrode in PMMA-based gel electrolytes (Ref 29). Sekhon et al. reported the transport properties of lithium electrolytes gelled with PMMA (Ref 30). Vondrák et al. examined gel polymer electrolytes based on PMMA; the conductivity of gels containing cations of smaller ionic radii (Li and Mg) was lower than that of the others (Ref 31). Deepa et al. studied the ion-ion interactions in liquid and gel polymeric electrolytes. The ion interaction was studied by a detailed FTIR study on aprotic liquid electrolytes ($LiCF_3SO_3$-PC) and the same gelatinized by PMMA incorporation over a wide range of salt (0.025-2 M) and polymer (5-25 wt.%) concentrations (Ref 32).

Chemical and mechanical properties of PMMA-PC system allow incorporation of many compounds and can be proton-conducting due to the presence of phosphoric acid or electronically conducting containing composite materials (Ref 33, 34).

Studies show that polymer electrolyte systems based on PMMA have been proposed for lithium battery application particularly because of their beneficial effects on the stabilization of the lithium-electrolyte interface (Ref 35).

A polymer solvent that is capable of strongly coordinating cations is necessary for electrolyte formation. Solvents such as polyethers, polyesters, polyamines, and tetrahydrofuran (THF), which have strong coordinating groups, dissolve salt easily. Solubility can be discussed in terms of the acid-base interactions between solvent and solute molecules, with each solvent being classified as hard or soft. The strength of the interaction between polymer solvent and salt can thus be classified according to the hard/soft acid base (HSAB) principle. The strongest interaction occurs by matching hard with hard or soft with soft (Ref 36). The solvating enthalpy of a salt in a polymer solvent hence depends on the cation polymer interaction.

Many polymers dissolve salts to form mixtures which support ionic conductivity. Low lattice energy is required to facilitate the ionization of the salt (Ref 37, 38). In our study, lithium tetraborate ($Li_2B_4O_7$) was chosen as the salt since it possesses low lattice energy and stability characteristics.

In the present work, PMMA is used as host polymer, $Li_2B_4O_7$ as salt, PC as plasticizer, and THF as solvent. The polymer electrolytes were characterized for electrical, thermal, and mechanical properties.

2. Experimental

2.1 Materials

The host polymer PMMA with average molecular weight of 35,000 g/mol was obtained from Acros Organic. $Li_2B_4O_7$ and PC were purchased from Aldrich and THF was procured from J.T.Baker.

2.2 Preparation

All electrolytes were prepared by solution casting technique. The weight ratio of the PMMA-Li_2B_4O7-PC is shown in Table 1. The prepared materials were dissolved in anhydrous THF and stirred continuously for 24 h to achieve a homogeneous viscous solution. The solution thus obtained was cast on a petri dish and allowed to evaporate slowly inside a dessicator. This procedure yields PMMA-$Li_2B_4O_7$-PC thin films.

Table 1 The weight ratios of the PMMA-$B_4Li_2O_7$-PC prepared

Designation	Composition (PMMA: $Li_2B_4O_7$: PC, wt.%)
PC0%	70.0:30.0:0.0
PC5%	66.5:28.5:5.0
PC10%	63.0:27.0:10.0
PC15%	59.5:25.5:15.0
PC20%	56.0:24.0:20.0
PC25%	52.5:22.5:25.0
PC30%	49.0:21.0:30.0
PC35%	45.5:19.5:35.0
PC40%	42.0:18.0:40.0

2.3 Instrumentation

2.3.1 Fourier Transform Infrared. FTIR analysis was performed by using Perkin-Elmer FTIR spectroscopy RX 1 in the wave region between 4000 and 400 cm^{-1}, with the resolution of 4 cm^{-1}.

2.3.2 Impedance Spectroscopy. The conductivity of the polymer films was measured by using HIOKI Model 3532-50 Bridge interfaced to a computer for data acquisition over the frequency range between 50 Hz and 1 MHz. The thickness of the films was measured with a micrometer screw gage. The films were then sandwiched between two stainless disks. Conductivity-dependent temperature was obtained on a temperature range of 30-100 °C.

2.3.3 Thermogravimetric Analysis. Thermal stability of polymer films was determined by using a Mettler Toledo analyzer that consists of TGA/SDTA851^e main unit and STARe software. The freshly prepared samples were dried and sealed in an inert atmosphere. Inert nitrogen gas was used in the analysis with a rate of 10 mL/min and the temperature was increased from 30 to 400 °C at the rate of 10 °C/min.

2.3.4 Mechanical Studies. An Instron Corporation Series 1X automated Materials Testing System 7.51.00 was used to measure the mechanical properties. The test pieces are designed according to the ASTM designation JIS K 6900. The thickness of the test pieces were between 1 to 3 mm. The crosshead speed for these test pieces was controlled to be 10 mm/min.

3. Results and Discussion

3.1 FTIR Studies

FTIR spectroscopy is a powerful tool to monitor the vibrational energy levels in different molecules. FTIR spectra were recorded in the transmittance mode. FTIR spectra of PMMA, PC0%, and PC25% are shown in Fig. 1. In PMMA, peak at 2974 cm^{-1} is assigned to C-H stretching, peak at 2039 cm^{-1} to CH_3 asymmetric stretching mode, peak at 1734 cm^{-1} to C=O symmetrical stretching, peak at 1487 cm^{-1} to O-CH_3 asymmetric bending, peak at 1388 cm^{-1} is assigned to CH_2 twisting mode, peak at 1192 cm^{-1} is assigned to O-CH_3 stretching mode, and peak at 986 cm^{-1} is assigned to C-O vibrational mode of C-O-C bond. In PC0% spectrum, the peaks

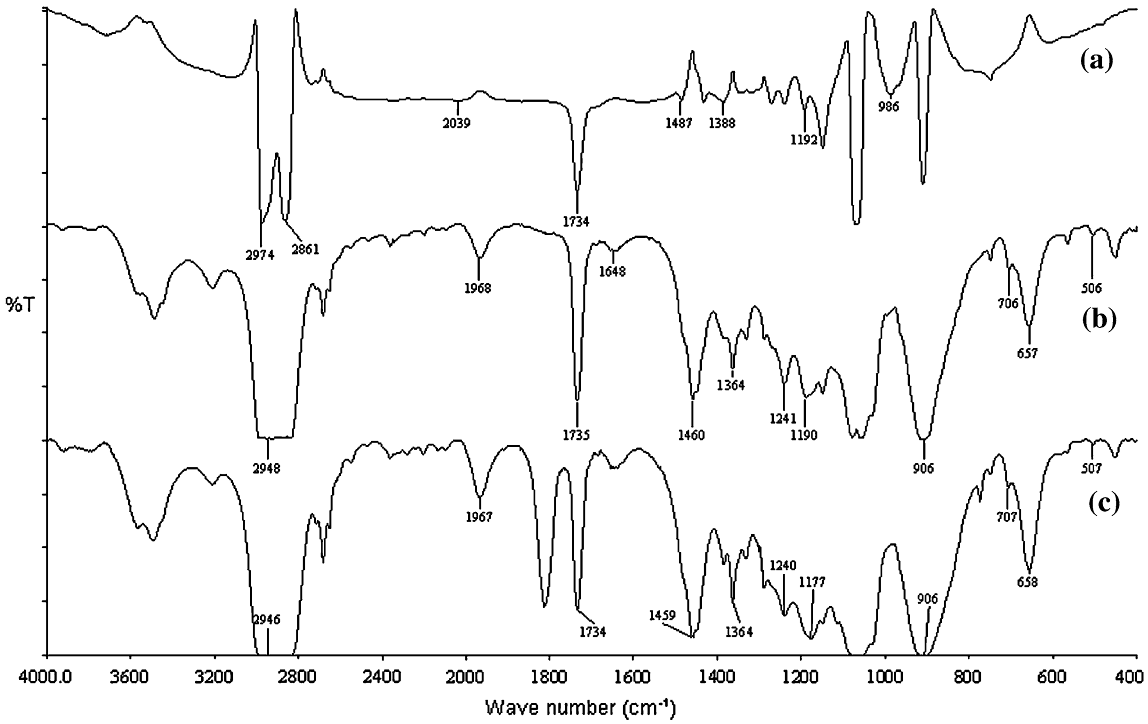

Fig. 1 FTIR spectra for (a) PMMA, (b) PC0%, and (c) PC25%

at 1487, 1388, 2039, and 2974 cm^{-1} have shifted to 1460, 1364, 1968, and 2948 cm^{-1}, respectively. In addition, the peak at 986 cm^{-1} is absent. This is due to the complexation between PMMA and $Li_2B_4O_7$. The intensity of peak at 1734 cm^{-1} is different when comparing pure PMMA and PC25%. The intensity of peak at 1734 cm^{-1} for pure PMMA is 75% while for PC25% is 45%. By comparing PMMA and PC0%, there are new peaks occurring at 506, 657, 706, 906, and 1241 cm^{-1} in PC0% FTIR spectrum. Peaks at 506 and 657 cm^{-1} are assigned to the O-B-O deforming vibration of BO_4 tetrahedral, peaks at 706 and 906 cm^{-1} are assigned to the B-O stretching vibration of BO_4 tetrahedral, and peak at 1241 cm^{-1} is assigned to the B-O stretching mode of BO_3 triangle. Another new peak occurs at 1812 cm^{-1} on addition of PC at PC25%. This peak is assigned to C=O stretching mode of PC. Besides that, by comparing PMMA and PC0%, two sharp peaks for PMMA at 2974 and 2861 cm^{-1} have become a broad peak at 2948 cm^{-1}.

The disappearance and shifting of some bands, formation of new peaks and changes in intensity of the peaks in the FTIR spectra of SPEs suggest that some extent of coordination or complexation has occurred between the constituents in these SPEs. In summary, these observations establish the complexation of PMMA with $Li_2B_4O_7$ and PC. These results correlate with the findings from previous studies (Ref 39, 40).

3.2 Conductivity Studies

Impedance spectroscopy was employed and the conductivities of the polymer complexes were calculated from the bulk resistance obtained by the intercepts of the typical impedance curves. Ionic conductivity was calculated using the relation $\sigma = l/R_bA$, where l is the thickness of the film, R_b is the bulk resistance, and A is the known area. PC25% has the highest ionic conductivity value of 5.14×10^{-6} S/cm at room temperature (23 °C). These results are in the same range with the results obtained by Ali et al. (Ref 41) and Uma et al. (Ref 42) who performed studies on PMMA-$LiCF_3SO_3$-PC and PMMA-Li_2SO_4-DBP, respectively.

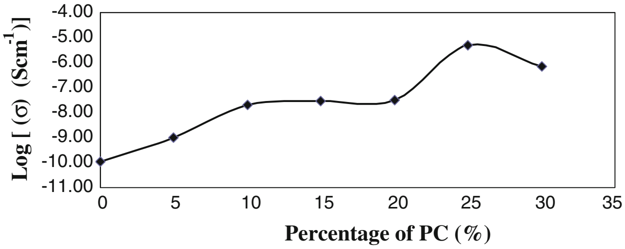

Fig. 2 The logarithmic conductivity against various percentage of PC at room temperature (23 °C)

The graph of the logarithmic conductivity against various percentage of PC content at room temperature (23 °C) is shown in Fig. 2. From Fig. 2, it is observed that as the percentage of PC increases, the conductivity increases. This is because the plasticizer acts as a conductivity enhancer and has a higher dielectric constant. The plasticizer would dissolve enough charge carriers and provide a more mobile medium for the ions so as to enhance the ionic conductivity of the films (Ref 20). Addition of a plasticizer modifies the electrolyte by lowering T_g through an isothermal increase in the system's configurational entropy and this consequently increases the mobility of all particles (Ref 36), which enables the easy flow of ions through the material when there is an applied electric field. Thus, conductivity increases.

However, as the concentration of PC increase to greater than 25%, the ionic conductivity decreases. This is attributed to the formation of linkages between the plasticizer itself, thus causing it to crystallize, resulting in the decrease in ionic conductivity (Ref 43).

3.3 Temperature-Dependent Conductivity Studies

Conductivity varies with temperature, according to the Arrhenius (Eq 1) relationship:

$$\log \sigma = \log A - \frac{E_a}{RT}, \qquad \text{(Eq 1)}$$

where A is the pre-exponential factor, E_a is the activation energy, and R is the gas constant, which is 8.314 J/mol/K.

Figure 3 shows the Arrhenius plot for PC25% from 30 to 100 °C. The regression (R^2) value for the graph is 0.9899. The linear relationship of the temperature-dependent conductivity reveals that the conductivity of PC25% followed an Arrhenius relationship. This reveals that the charge carriers move in a liquid-like environment (Ref 44). The conductivity was found to increase with increase in temperature. This is because as the temperature increases, the ion mobility and segmental mobility increases as well, assisting ion transport and causing higher conductivity.

3.4 Thermal Studies

TGA studies were carried out under nitrogen atmosphere to examine the thermal stability of the polymer electrolytes. The first and second decomposition temperatures and percentages of total weight loss are shown in Table 2. Figure 4 shows the TGA curves for PC20%, PC25%, and PC40%.

Studies by Uma et al. exhibited the first weight loss at 46 °C for PMMA polymer electrolytes with Li_2SO_4 as salt (Ref 42). In another study by Rajendran et al. for PMMA blends with $LiClO_4$, the researchers found that their initial weight loss began at 50 °C (Ref 45). In this study, with $Li_2B_4O_7$ as salt, it was found that the initial weight loss for all the samples is quite constant up to 100 °C. This weight loss is due to the evaporation of residual solvent and moisture from the samples. These comparative observations show that PMMA-PC-$Li_2B_4O_7$ films can be operated up to 100 °C and are preferred in the lithium polymer batteries as its operating temperature is normally in the range of 40-70 °C.

The samples exhibit first decomposition temperature above 100 °C. This decomposition is due to the scission of the main chains at weak head-to-head linkages. The second decomposition takes place at temperatures about 370 °C. This decomposition is caused by the crystallization of the polymer complex and the separation of methyl methacrylate monomers from vinylidene chain ends (Ref 46). With the addition of the plasticizer, the total weight loss increases as shown in Fig. 4. This is because plasticizer reduces the T_g of the polymer complex and decreases the stability of the polymer films. Therefore, as the percentage of PC increases, the stability decreases and an increase in weight loss is observed.

3.5 Mechanical Studies

The results of stress-strain measurement for PC0%, PC25%, and PC40% are summarized in Table 3. The addition of PC to PMMA: $Li_2B_4O_7$ decreases the Young's modulus and stress at peak values of the complexes. When the plasticizer content is increased from 25 to 40 wt.%, there is an obvious decrease in Young's modulus and stress at peak values of the polymer electrolytes. The influence of PC on the mechanical properties of PMMA: $Li_2B_4O_7$ film resembles the plasticization effect. The interaction between PMMA and $Li_2B_4O_7$ are weakened by the presence of PC, increasing its flexibility and reducing the viscosity, the Young's modulus and stress at peak values.

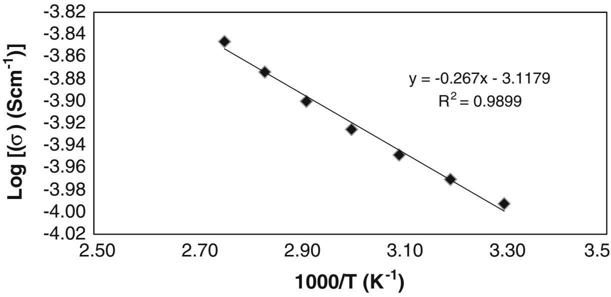

Fig. 3 Arrhenius plot of ionic conductivity for PC25% from 30 to 100 °C

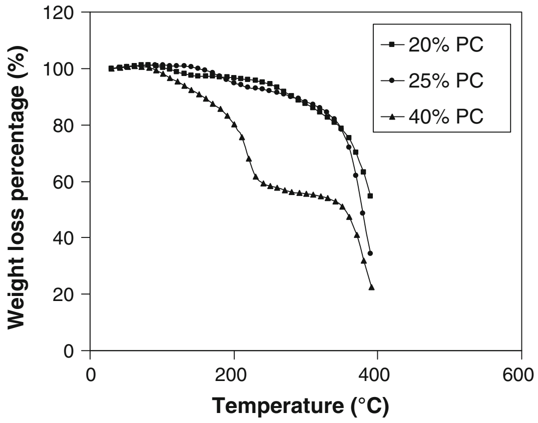

Fig. 4 TGA curves for PC20%, PC25%, and PC40%

Table 2 First and second decomposition temperatures and percentages of total weight loss for various composition polymer films

Sample	First decomposition temperature,°C	Second decomposition temperature, °C	Total weight loss, wt.%
PC20%	119.0	376.3	45.33
PC25%	180.2	373.4	64.88
PC40%	119.8	376.2	77.54

Table 3 Stress-strain results of plasticized PMMA-$Li_2B_4O_7$-PC complexes

Sample	Young's modulus, MPa	Stress at peak, MPa
PC0%	$\sim 10^{-2}$	2.00
PC25%	$\sim 10^{-3}$	0.12
PC40%	$\sim 10^{-4}$	0.08

4. Conclusion

Complex formation in PMMA-$Li_2B_4O_7$-PC has been confirmed from FTIR studies. The highest conducting sample is PC25% with conductivity of 5.14×10^{-6} S/cm. The temperature-dependent conductivity agrees with Arrhenius behavior and reveals that the charge carriers move in a liquid-like environment. From the TGA analysis, it is observed that the stability of the polymer films decline as the content of plasticizer, PC increases. It can be concluded that the addition of PC decreases the mechanical properties and improves the flexibility of the complexes.

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