

Effect of Plasticizer on the Structural and Transport Properties of Mg^{2+} - Ion Conducting Solid Polymer Electrolytes: [PEO: $\text{Mg}(\text{CH}_3\text{COO})_2$]

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Original Research Article

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ABSTRACT

In this study, the use of low molecular weight Ethylene glycol [$\text{CH}_2(\text{OH})\text{CH}_2\text{OH}$] as a new type of plasticizer in polyethylene oxide (PEO)/ $\text{Mg}(\text{CH}_3\text{COO})_2$ solid polymer electrolytes preparation was examined. The complexation of $\text{Mg}(\text{CH}_3\text{COO})_2$ salt with the polymer in the presence and absence of plasticizer was confirmed using Fourier transform infrared spectroscopy (FTIR). The Optical Microscopy (OM) results showed that the microstructural evolution of the pristine polymer is as a result of doping and plasticization. The ionic conductivity was measured for the [PEO: $\text{Mg}(\text{CH}_3\text{COO})_2$] systems in the temperature range 296K – 338K using standard four-probe technique. The addition of Ethylene glycol [$\text{CH}_2(\text{OH})\text{CH}_2\text{OH}$] as plasticizer in the solid polymer electrolytes significantly improved the ionic conductivity. No conduction was observed in the solid polymer electrolyte systems without plasticizer except for the 15-wt-% $\text{Mg}(\text{CH}_3\text{COO})_2$ -doped system. The temperature dependence of the ionic conductivity reveals the conduction mechanism to be an Arrhenius-type thermally activated process.

Keywords: PEO, Ethylene Glycol, $\text{Mg}(\text{CH}_3\text{COO})_2$, FTIR, OM, Ionic conductivity, Polymer electrolyte,

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1. INTRODUCTION

Nowadays, device size and implementation of affordable, compact, lightweight, high-capacity, solid-state rechargeable batteries are the key contenders amongst communication and electronic industries. High ionic conductivity, strong solvating ability with a number of alkali, alkaline and transition metal salts and improved thermal, mechanical and electrochemical properties are desirable characteristics of the extensively studied polyethylene oxide (PEO) based polymer electrolytes [1]. Polyethylene oxide and poly(ethylene glycol) (PEG)-based polymer electrolytes have been regarded as the most suitable electrolytes for rechargeable batteries, amongst the family of polymers (polypropylene oxide (PPO), polyoxymethylene and polyether) with similar structure because of their semicrystalline nature at room temperature, exceptional property to dissolve high concentration of a wide variety of dopants and fast ionic conduction in its molten state when compounded into solid polymer electrolytes [2-5]. In recent years, enormous efforts and several different synthetic techniques have been employed to enhance the ionic conductivity and the cation transport number but creating one universal technique for developing polymer electrolytes is impossible due to the physicochemical differences between each system [6-11]. Most studies have been on the variation of concentration of alkali metal salts (lithium being the most used) and inorganic fillers and on conductivity enhancement of high molecular-weight polymers (e.g., PEO) using mixed phases with great interest on nanosized fillers (like TiO_2 , Al_2O_3 , SiO_2 , ZnO and MgO) [1, 12-18]. As gathered from these literatures reviewed above, the general view is that the underlying principles of the mixed-phase electrolytes is not yet well understood, but the general acceptable view is tied to the fact that fillers improve the mechanical properties and considerably stabilize the electrode-electrolyte interfaces. This implies that more systematic work is needed before these systems can be optimally designed for practical applications [1].

However, much attention has not been paid to the low-molecular-weight ($\text{MW} = 4000$) PEOs and complexing or plasticizing with PEGs. In our laboratory, this strategy has been exploited based on the reasoning that PEG [$\text{CH}_2(\text{OH})\text{CH}_2\text{OH}$] has the same monomeric unit as PEO, but with an end hydroxyl group. The Ethylene Glycol used in our studies for plasticization is a liquid at room temperature, having a melting point close to 60°C . Subsequently, the use of Mg^{2+} ion salt in this work is to proffer solution to the existing problem of chemical instability of Li^+ salt with the electrodes in the Li-ion batteries as claimed by many researchers [5], and to reduce cost and over dependence on Li salts for solid state rechargeable batteries. Thus, solid state rechargeable batteries based on Mg/Mg^{2+} -ion salt solid polymer electrolytes are expected to give battery performance capabilities close to those of Li/Li^+ -ion solid polymer electrolyte batteries due to the fact that Mg^{2+} -ions have ionic size and weight comparable to those of Li^+ -ions. In this work, $\text{Mg}(\text{CH}_3\text{COO})_2$ salt was oven dried for 4hrs and subsequently blended with dissolved Poly-Ethylene Oxide (PEO) in the absence and presence of ethylene glycol employing solution mixing technique. The hybrid solid polymer electrolytes were then characterized by Fourier Transform Infrared (FTIR) spectroscopy, Optical Microscopy (OP) and conductivity measurement for possible utilization in electrochemical devices.

2. MATERIAL AND METHODS

Poly-Ethylene Oxide - magnesium acetate tetra hydrate [$\text{PEO}-\text{Mg}(\text{CH}_3\text{COO})_2$] Solid Polymer Electrolytes (SPEs) were produced in two batches involving development with or without Ethylene glycol [$\text{CH}_2(\text{OH})\text{CH}_2\text{OH}$]. In the development of first batch of SPEs, known weight percentages (ranging from 5-25%) of magnesium acetate tetra hydrate were mixed with PEO dissolved in de-ionized water via one-pot reaction. On the other hand, the development of second batch of SPEs was actualized through mixing of same percentages of magnesium

80 acetate tetra hydrate with PEO dissolved in a mixture of de-ionized water (50%) and
81 Ethylene glycol (50%) via one-pot reaction.

82 Following this, five (5) samples for each batch of PEO-Mg(CH₃COO)₂ Solid Polymer
83 Electrolytes (SPEs) containing 5-25% of Mg(CH₃COO)₂ .4H₂O were produced by solution
84 casting into Petri dishes. The samples were oven dried at temperature lower than the
85 melting point of PEO for 12hrs and subsequently, flat and uniform thin samples were
86 obtained. The samples were analyzed by a FTIR spectrophotometer in transmission mode
87 without KBr. The spectra were recorded in the frequency range 400 to 4000 cm⁻¹, after 25
88 scans, with resolution of 2cm⁻¹. The positions and intensities of the IR bands were processed
89 with Spectral Analysis software. Surface morphologies of SPEs were observed using Micro-
90 Capture (Veho-VMS 004) USB microscope employing optical radiation and the images were
91 analyzed using Imaging software (Image-J). Finally, the samples were characterized for their
92 ionic conductivities and transference numbers from room temperature to 65°C using Four-
93 point probe resistivity measurement system. The variation of current with time for the
94 batches of SPEs containing 5-25% of Mg(CH₃COO)₂ are given in Fig. 4(a-c) respectively.
95 From these plots, the initial current (i_i) and final current (i_f) are evaluated, and total ionic
96 transference number (t_{ion}) was calculated using the relation:

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$$t_{ion} = \frac{i_i - i_f}{i_i}$$

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99 3. RESULTS AND DISCUSSION

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101 The micrograph presented in figure 1. represents the pristine polyethylene oxide film wherein
102 the spherulitic growths are discernable with uniform contrast. The average size of the
103 crystalline domain appearing in pristine PEO is 157.20±0.04µm [1]. After blending the
104 pristine polymer with Mg(CH₃COO)₂ .4H₂O the surface topographies of the SPEs [figures 2
105 (a-j)], were modified and no apparent pattern of PEO crystallites is observed under
106 microscope. This can be attributed to the strong nucleating effect of Mg(CH₃COO)₂ .4H₂O
107 salts dispersion, leading to non-observation of the too small spherulitic growths under our
108 experimental condition.

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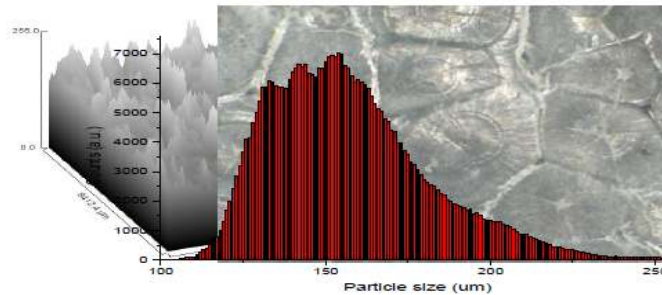
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116 **Figure1: Optical Micrograph, Surface topography and Histogram of particle**
117 **distribution for pure PEO film (Taken from [1]).**

118 It can be seen from figures 2(a-j) that the contrast variation is mainly due to the differences
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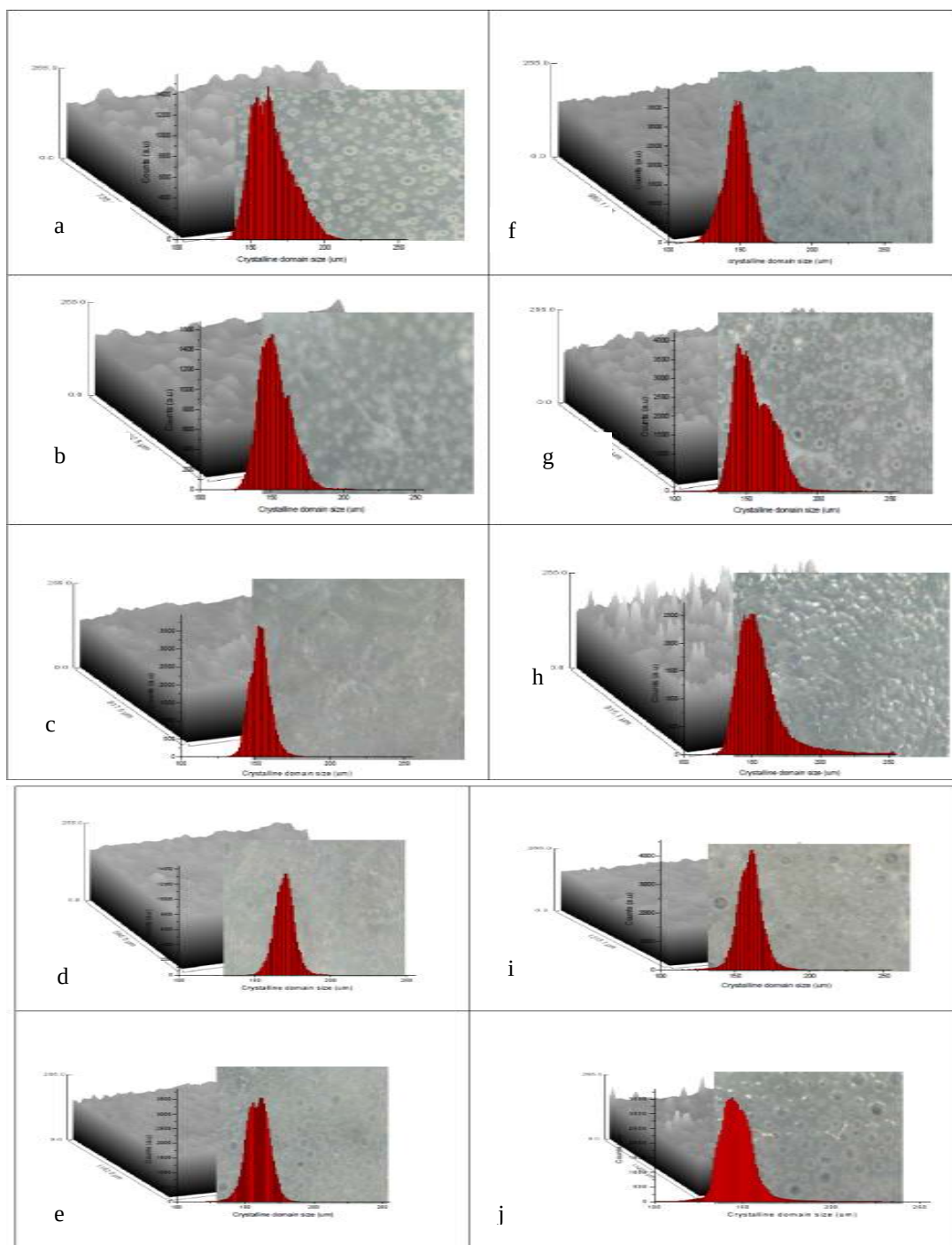
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143 **Figure 2: Optical micrograph, Surface topography and Histogram of particle**
 144 **distribution for (a) PEO/EG/ 5% salt (b) PEO/EG/ 10% salt (c) PEO/EG/ 15% salt (d)**

145 **PEO/EG/ 20% salt (e) PEO/EG/ 25% salt (f) PEO/ 5% salt (g) PEO/ 10% salt (h) PEO/15%**
146 **salt (i) PEO/ 20% salt (j) PEO/ 25% salt**

147 yield of the pristine polymer and filler atoms. The pristine polymer matrix appear in dark
148 contrast while the filler matrix in lighter contrast. The nucleating effect of
149 $\text{Mg}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ salts dispersion was observed under microscope and analyzed to bring
150 out representative images of the SPEs with their corresponding 3D surface topography and
151 crystalline domain size distribution. The morphological descriptions for the compositional
152 hierarchies of the SPEs without plasticizer are presented in figure 2(f-j). The images
153 presented in figure 2(a-e) together with the corresponding 3D surface topography and
154 crystalline domain size distribution are the representatives of the microstructural evolution of
155 pristine PEO polymer as a result of incorporation and plasticization with $\text{Mg}(\text{CH}_3\text{COO})_2$ and
156 Ethylene glycol respectively.

157 Microscopic imaging analysis reveals the degree of the $\text{Mg}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ salts
158 dispersion in the PEO- $\text{Mg}(\text{CH}_3\text{COO})_2$ Solid Polymer Electrolytes (SPEs). The variation in
159 contrast and appearance of the images may be attributed the lowering of the crystallinity of
160 PEO as a result of the presence of $\text{Mg}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ salts. The average sizes of the
161 crystalline domain in the SPEs containing 5%-25% $\text{Mg}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ salts with and
162 without plasticizer range from $146.70 \pm 10.99 \mu\text{m}$ to $169.39 \pm 6.32 \mu\text{m}$ while the SPEs containing
163 15% $\text{Mg}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ salts with and without plasticizer possess discernable grain
164 boundaries with average sizes of the crystalline domain being $153.08 \pm 6.79 \mu\text{m}$ and
165 $155.77 \pm 17.75 \mu\text{m}$ respectively.

166 The FTIR spectra for the two batches of PEO- $\text{Mg}(\text{CH}_3\text{COO})_2$ Solid Polymer Electrolytes
167 (SPEs); involving development with or without Ethylene glycol [$\text{CH}_2(\text{OH})\text{CH}_2\text{OH}$] each
168 containing 5-25% of $\text{Mg}(\text{CH}_3\text{COO})_2$ salts given in Figures 3(a-e) and 4(f-j) respectively, were
169 recorded periodically to find out if the $\text{Mg}(\text{CH}_3\text{COO})_2$ salts were embedded in the PEO matrix
170 and chemically bonded to polymer chains. In this work, the significant absorptions observed
171 below 800 cm^{-1} represent metallic bond with oxygen and those above 800 cm^{-1} represent
172 both metallic and metal oxide bonds with hydrogen and hydroxyl groups. The FTIR spectra
173 display broad bands at 3392.69 cm^{-1} - 3905.98 cm^{-1} , which are believed to be associated with
174 the stretching vibrations of hydrogen bonded surface water molecules and hydroxyl groups
175 reflecting the additivity of PEO and Ethylene glycol [$\text{CH}_2(\text{OH})\text{CH}_2\text{OH}$]. It is also noticed that
176 the hydroxyl stretching bands became much broader with increasing $\text{Mg}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$
177 salts content. This strongly supports the idea that a hydrogen bonding can form between
178 either oxygen atoms of PEO and hydroxyl groups of $\text{Mg}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ salts. The
179 asymmetric stretching vibrations occurring in the range 2777.59 cm^{-1} - 2924.18 cm^{-1}
180 represent the characteristic band of Aldehyde groups.

181 The bands occurring at 2166.13 cm^{-1} - 2364.81 cm^{-1} represent the triple carbon bond of the C
182 $\equiv\text{C}$ stretching vibrations. In addition, the bands at 1060.88 cm^{-1} - 1149.61 cm^{-1} are
183 associated with C-OH bending vibrations of the secondary alcohols. The shift to higher
184 frequencies may be attributed to additional bonding of these groups with metallic
185 compounds. The band at 1599.04 cm^{-1} - 1967.46 cm^{-1} is associated with both C=O
186 conjugated and non-conjugated stretching vibration implying the existence of carbonyl
187 absorptions. The changes observed in the vibration frequency of $\nu\text{C}=\text{O}$ in the SPEs indicate
188 that the incorporation of the $\text{Mg}(\text{CH}_3\text{COO})_2$ salts has great influence on the vibration
189 frequency of $\nu\text{C}=\text{O}$. The bands at 1315.50 cm^{-1} - 1458.23 cm^{-1} are associated with the
190 bonding of MgO with ethylenic groups. The characteristic band of PEO was observed at
191 823.63 cm^{-1} - 960.58 cm^{-1} due to the C-O-C bending vibration occurring in the range
192 1263.42 cm^{-1} - 1280.78 cm^{-1} . The changes in the C-O-C band in the spectrum; suggest that
193 hydrogen bonding is the underlying mechanism in the interaction. In addition, hydrogen

194 bonding has a strong influence on the dispersoid (in our case the --OH of $\text{Mg}(\text{CH}_3\text{COO})_2$
195 $\cdot 4\text{H}_2\text{O}$ salts) and the absorption maxima of stretching vibration shifts toward higher
196 frequencies. Finally, the out of plane bending vibrations appearing at 823.63cm^{-1} – 960.58
197 cm^{-1} are due to slight transformation in the ethylenic groups as a result of oxidation and
198 hydrolysis.

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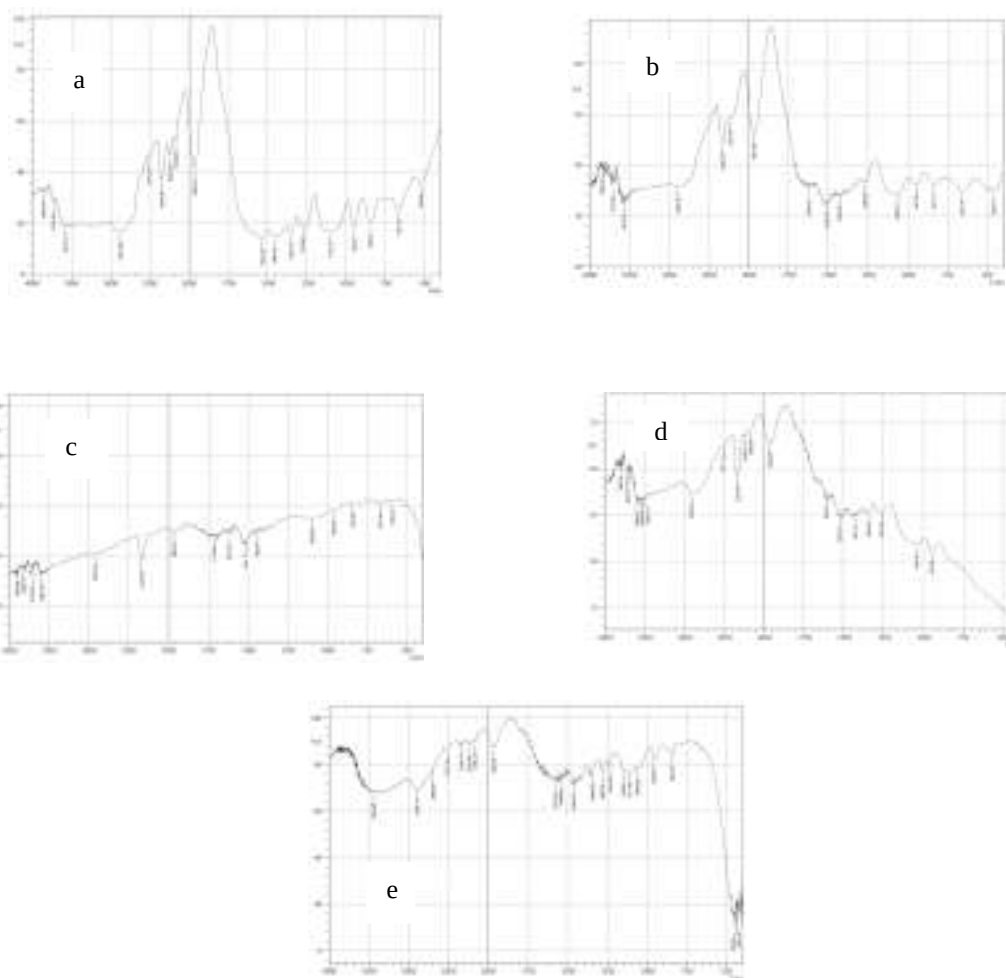
217 **Figure 3: FTIR Spectra for the SPEs containing (a) PEO/EG/5% salt (b) PEO/EG/10%**
218 **salt (c) PEO/EG/15% salt (d) PEO/EG/20% salt (e) PEO/25% salt**

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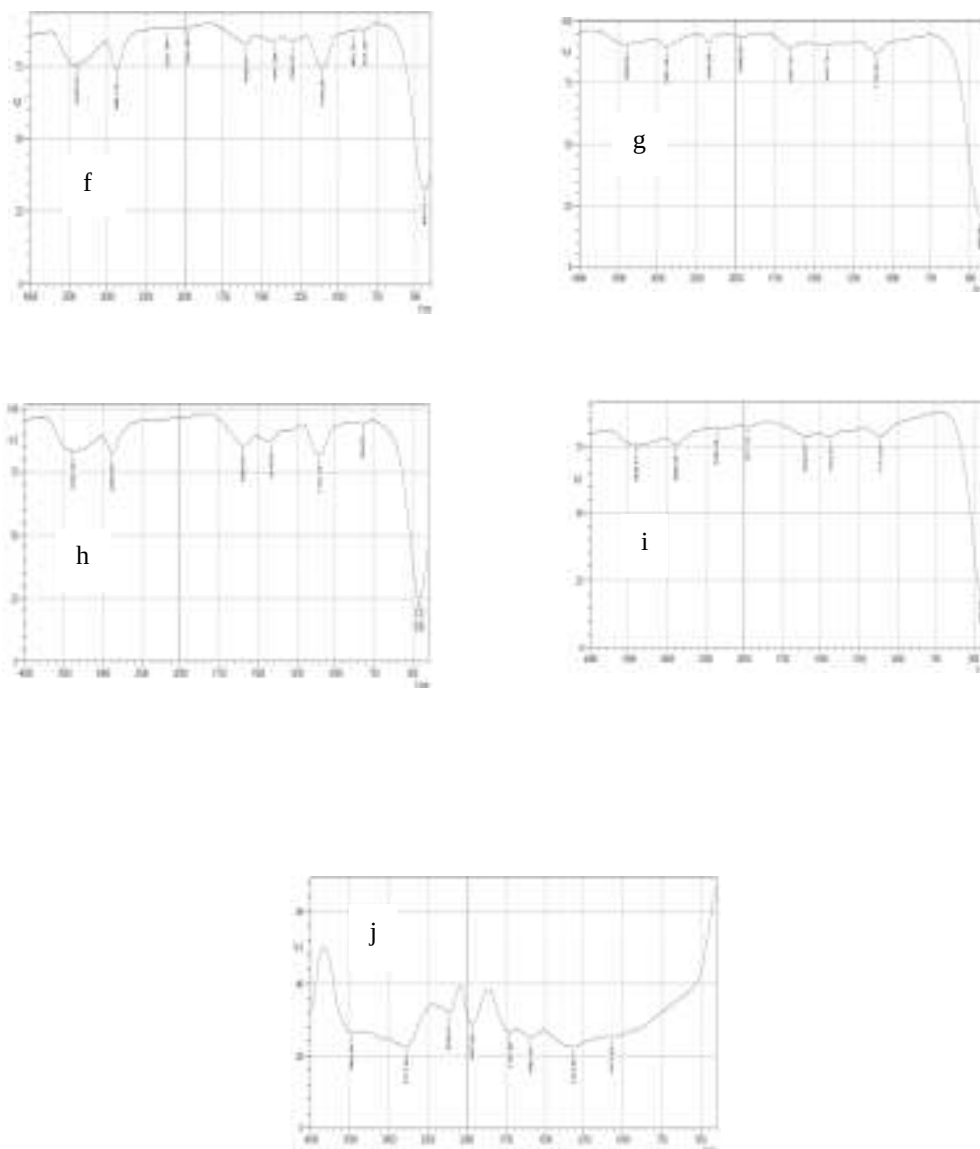
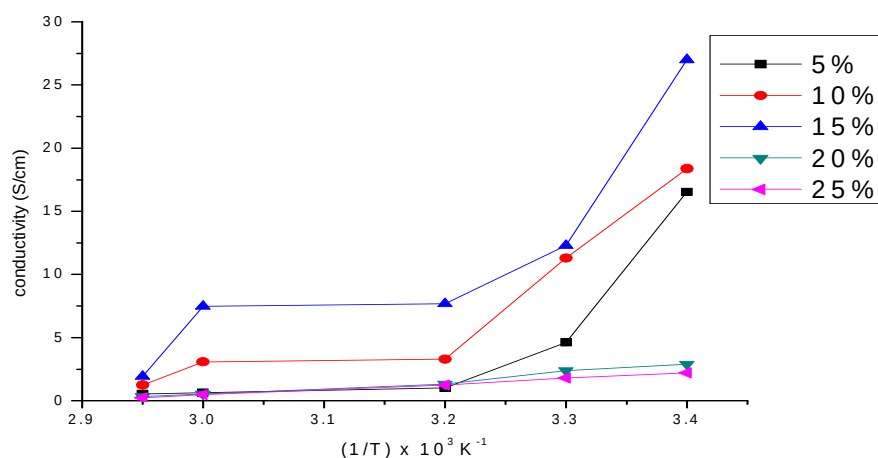


Figure 4: FTIR Spectra for the SPEs containing (f) PEO/5% salt (g) PEO/10% salt (h) PEO/15% salt (i) PEO/20% salt (j) PEO/25% salt

In the present study, the likely mobile species are protonic and the total transference number was evaluated using equation (1) above. The calculated values of t_{ion} for the two batches of SPEs ranges from 0.74 to 0.98 where the SPE containing 15% $Mg(CH_3COO)_2 \cdot 4H_2O$ salts has the highest value. From these values, it is obvious that PEO- $Mg(CH_3COO)_2$ Solid Polymer Electrolytes (SPEs) are essentially ionic materials. The quality of the values is limited due to uncertainty in the measurement of initial current due to quick onset of polarisation [1]. The temperature dependence of the conductivity (i.e. $\ln \sigma$ versus $1/T$ and σ versus $1/T$ plots) for the SPEs containing Ethylene glycol $[CH_2(OH).CH_2OH]$ are presented in Figures 5 and 6 while that for the SPE containing 15% salt without Ethylene glycol $[CH_2$

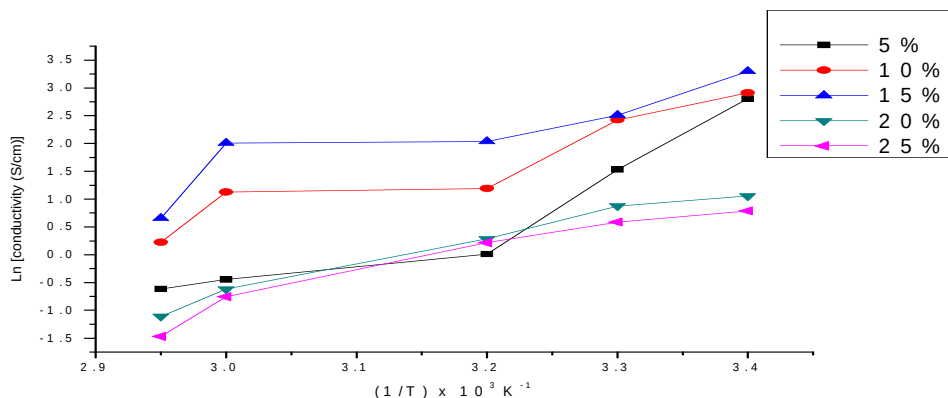
(OH).CH₂OH] is presented in figure 7. The addition of Ethylene glycol [CH₂(OH).CH₂OH] as a plasticizer enhances the conductivity after T_m, in each of the five compositions shown in Figures 5 and 6; this may be attributed to phase transition from semicrystalline to amorphous phase as a result of plasticization whereas the batch without plasticizer did not show any conductivity except for the sample containing 15% Mg(CH₃COO)₂ .4H₂O salts. Thus, for both batches, there exists continuous decrease in conductivity with temperature due to the size of mobile species and the values for the sample are presented in table 1 near the phase transition. The calculated values for activation energy obtained from the Arrhenius plots and the ionic conductivity at 338K for the SPEs are given in Table 1. The activation energies for the SPEs range from 1.23×10⁻³eV to 1.97 ×10⁻³eV where the sample containing 15% salt has the lowest activation energy. The reduction in activation energy leading to higher conductivity may be due to smoother morphologies of the films upon addition of Ethylene glycol [CH₂(OH).CH₂OH].



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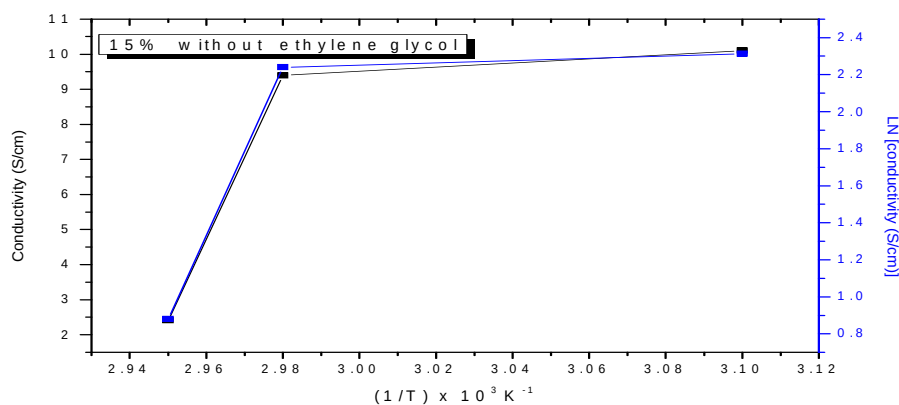
268 **Figure 5: Variation of conductivity with Temperature for the SPEs containing 5% -25%**
 269 **Mg(CH₃COO)₂ .4H₂O with Ethylene Glycol**

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272 **Figure 6: Variation of Ln conductivity with Temperature for the SPEs containing 5% -**
 273 **25% $\text{Mg}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ with Ethylene Glycol**



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275 **Figure 7: Variation of conductivity and Ln conductivity with Temperature for the SPEs**
 276 **containing 15% $\text{Mg}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ without ethylene glycol**

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278 **Table 1: Transport properties for SPEs containing 5%- 25% $\text{Mg}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ salts**
 279 **with Ethylene Glycol**

Salt Con.(%)	Cond. (S/cm) @338K ($\times 10^{-2}$)	Transference Number	Activation Energy (eV) ($\times 10^{-3}$)
5	5.4	0.88	1.97
10	12.5	0.91	1.45
15	19.3	0.98	1.23
20	3.3	0.77	1.33
25	2.3	0.74	1.34

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283 4. CONCLUSION

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285 The use of low molecular weight Ethylene glycol [$\text{CH}_2(\text{OH})\text{CH}_2\text{OH}$] as a new type of
 286 plasticizer in addition to the versatility of PEO have been exploited for the development of
 287 (PEO)/ $\text{Mg}(\text{CH}_3\text{COO})_2$ solid polymer electrolytes. The morphological characteristics and

transport properties of the developed SPEs were examined employing Optical Microscopy, FTIR and dc conductivity. The FTIR spectroscopy revealed the existence of strong interfacial interaction between PEO and $\text{Mg}(\text{CH}_3\text{COO})_2$ as the pristine polymer matrix are uniformly interspersed with $\text{Mg}(\text{CH}_3\text{COO})_2$ salts showing strong absorption bands for the polymer and the $\text{Mg}(\text{CH}_3\text{COO})_2$ salts. Thus, the significant absorptions observed below 800cm^{-1} represent metallic bond with oxygen and those above 800 cm^{-1} represent both metallic and metal oxide bonds with hydrogen and hydroxyl groups. Observation with optical microscope also revealed that the crystallinity of the pristine PEO decreases with increase in percentage weight of $\text{Mg}(\text{CH}_3\text{COO})_2$ salts. Finally, polarisation and conductivity studies confirmed that the transference number and ionic conductivity were found to increase with increase in $\text{Mg}(\text{CH}_3\text{COO})_2$ salts content up to 15% and above this, a decrease in the conductivity was observed. However, a maximum conductivity of $19.3 \times 10^{-2}\text{ S/cm}$ and transference number of 0.98 was observed for sample containing 15 wt% $\text{Mg}(\text{CH}_3\text{COO})_2$ salts at 338 K. The temperature dependence of ionic conductivity of the SPEs seems to follow an Arrhenius-type, thermally activated process with activation energies reducing with addition of Ethylene glycol $[\text{CH}_2(\text{OH}).\text{CH}_2\text{OH}]$.

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306 ACKNOWLEDGEMENTS

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308 We thank Materials Science Unit of the Department of Physics, Ahmadu Bello University,
309 Zaria, Nigeria and National Research Institute for Chemical Technology, Zaria, Nigeria for
310 technical assistance during the experimental characterization.

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315 COMPETING INTERESTS

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317 Authors have declared that no competing interests exist.

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320 AUTHORS' CONTRIBUTIONS

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322 This work was carried out in collaboration between all authors. Authors TOA and AI
323 designed the study. Author TOA wrote the protocol and wrote the first draft while, author
324 TOA and author EON carried out the experimental work and analyses. Author TOA and
325 author EON managed the literature searches. All authors read and approved the final
326 manuscript.

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