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Effect of Ethylene Carbonate (EC) Plasticizer on Poly (Vinyl Chloride)-Liquid 50% Epoxidised Natural Rubber (LENR50) Based Polymer Electrolyte

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ABSTRACT

In this research, new thin film of a free standing electrolyte film containing poly(vinyl) chloride (PVC), 50% liquid epoxidized natural rubber (LENR50), Ethylene carbonate (EC) blends as a host for the electrolyte which was doped with lithium perchlorate ($LiClO_4$) as the dopant salt was successfully prepared with solution casting technique. The polymer electrolyte of PVC-LENR50-EC-$LiClO_4$ was characterized using impedance spectroscopy (EIS), scanning electron microscopy (SEM) and Fourier transform infrared (ATR-FTIR). From the EIS results shows that electrolyte exhibited the highest ionic conductivity of 2.1×10^{-7} *Scm^{-1} at the 30 wt.% of $LiClO_4$. The ionic conductivity result was supported by the morphological studies which revealed the good homogeneity of the PVC-LENR50-EC blends as no phase separation was observed. The smooth surface can ease the mobility of ions in the system complexes. In addition, the formation of micro-pores by introducing lithium salts to the electrolyte also improved the transportation properties of Li^+ ions in the electrolyte system and hence improving its ionic conductivity. The features of complexation of the electrolytes were studied by ATR-FTIR.*

***Keywords*:** *PVC, LENR50, $LiClO_4$, Ionic Conductivity, Polymers Electrolyte, EC*

1. Introduction

Ionic conducting polymer was first suggested by Fenton and Wright in 1973. Since the pioneering work by Wright on ions conductivity in the poly (ethylene oxide)/alkali metal salt complexes, the studies on solid polymer electrolytes (SPEs) have attracted and receiving a great deal of attention due to its proposed large scale use in high energy density secondary lithium ion batteries, sensors, solar cell, fuel cell as well as electrochromic smart windows [1-4]. In the beginning of 21st century, high demands for new renewable energy, storage system and fast emerging of sophisticated microelectronic portable applications, solid state lithium ion batteries have become most preferred power source because of their advantages such as high energy density, long life cycle and absence of a "memory" effect problem. SPEs possessed many advantage over conventional liquid electrolytes in terms of shape, geometry, mechanical strength and good electrode-electrolyte contact [4,5]. However, one of the biggest drawbacks in development of SPE is having low conductivity at ambient temperature [4, 6]. Numerous efforts and approaches have been employed such as addition of nano-size ceramic filler/ additives, plasticizer (EC and PC), radiation, binary blending of polymers and etc [7-12].

Up to date, one of the most famous polymer used in SPE as polymer host is poly (vinyl chloride). PVC is well known for its excellent miscibility and compatibility properties with various low or high molecular weight polymers as well as providing good mechanical strength. This is due to its lone pair of electron from the chlorine atom which can act to stiffen the backbone of the polymer. Besides, the inexpensive PVC can well solvated with the inorganic salts [8,12-16].

Modified natural rubber such as ENR 50 and MG49 has received great attentions from many researches for its distinctive properties of having low glass transition temperature and good elasticity and adhesion properties [5,7,11]. In addition, these modified rubbers can provide coordination sites for Li^+ ions conduction and produce a great number of charge carriers for ionic transport because both have a lone pair of electrons from the oxygen atom. In this work, we have further the modification works for ENR50 by using photochemical method to produce the low molecular weight of liquid epoxidised natural rubber, LENR50. By having lower molecular weight, we expected it would give better property than ENR50 in term of greater penetration onto PVC interstices enabling better solubilisation of the PVC segments in the polymer blend of PVC-LENR50—salt system [9-10,17]. In the present work, we successfully prepared a solid polymer electrolyte consisting of modified low molecular weight rubber LENR50 based PVC-$LiClO_4$ system with enhancement of ethylene carbonate, EC.

2. Experimental

2.1.Materials

PVC (Aldrich) with average molecular weight of 97,000 and ENR50 (Guthrie (M) Bhd.) were used in this work. Lithium perchlorate ($LiClO_4$, purity >95%) and Ethylene carbonate (EC) were obtained from Aldrich. Tetrahydrofuran (THF) was purchased from JT Barker whereas toluene was from R&M Chemical England.

2.2. Sample Preparation of LENR50

Before the rubber solution of 5 wt.% was prepared, 250 g of 50% epoxidized natural rubber (ENR50) was cut into smaller size and put into 5 L of straight sided cylindrical flask which contained 4750 g of toluene solvent. The cylindrical flask was fitted together with an immersion well, a stirring assembly and a condenser as shown in **Figure 1**. The ENR50 was stirred until it is completely dissolved. The ENR50 solution was then radiated for 50 hours with a medium pressure mercury lamp of 400 watt which was contained in the double-walled immersion well made from quartz allowing water cooling by a chiller. The temperature was fixed at 20˚C throughout the process. The depolymerised ENR50 was recovered first and concentrating the rubber solution using a rotary evaporator at 60˚C until 60% of dried rubber contained (DRC) in the solution was achieved.

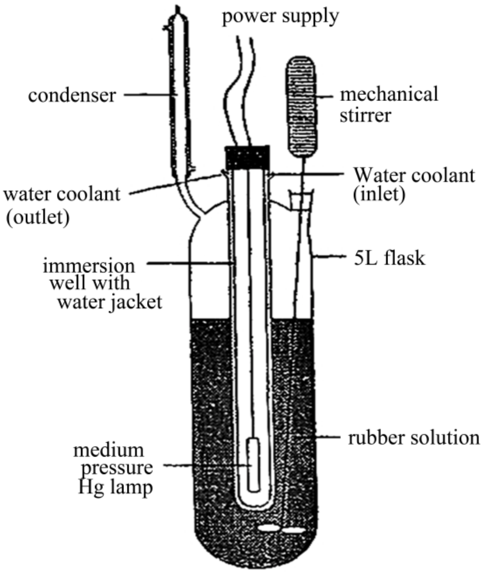

Figure 1. UV irradiation system.

The *M*w of LENR50 before irradiation was 639,661 Da and after irradiation for 50 hours was 76,473 Da [9,10, 17].

Figure 2 show the degradation of ENR50 upon irradiation of UV light while **Figure 3** shows the proposed structure and mechanism routes for irradiation of LENR50. In the process of degradation ENR50, the chain scission take place at C-C bond which bind the two isoprene units together. This was because of the resonance energy which results in it being the weakest bond with energy of only 181 kJ/mol. Besides, it's reported by Dahlan and Abdul Ghani that the liquid form of ENR50 prepared by this technique did not show any significant changes in the absorption peaks of isoprene unit except for the prominent enhanced for—OOH and carbonyl groups in the IR. It's suggested that the formation of carbonyl groups occurred as a result of the ring opening of the epoxy group to produce hydroxylated group [9,10, 17].

2.3. Sample Preparation of SPE

0.9 g of PVC was dissolved into 60 ml of THF and

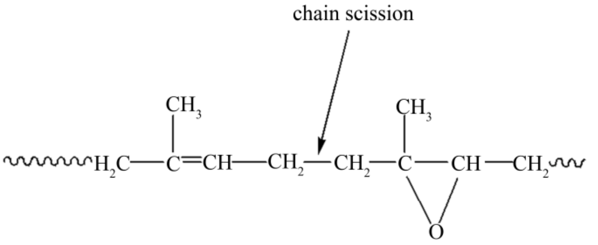

Figure 2. Degradation of ENR50 upon irradiation of UV light.

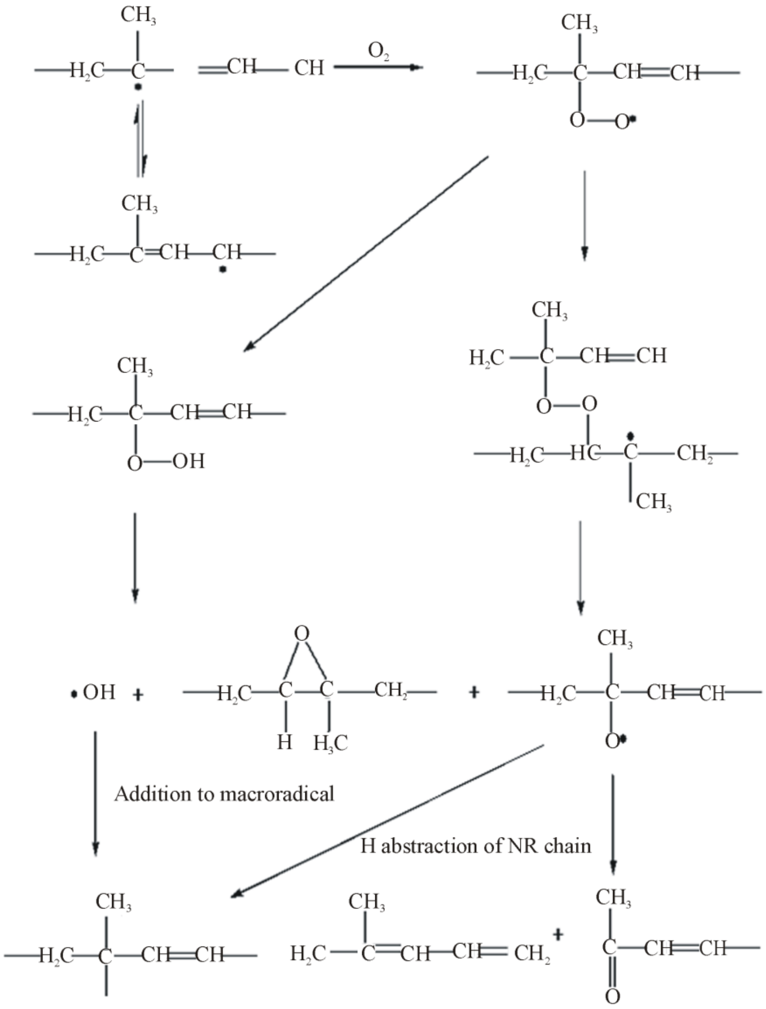

Figure 3. Structure and mechanism routes for irradiation of LENR50.

stirred using a magnetic stirrer until all PVC was dissolved completely. 3.5 g of LENR50 which contains 2.1 g of DRC was poured into the solution and stirred for 24 hours to form a homogenous solution. The 5 wt.% $LiClO_4$ were then dissolved in EC aand THF before added into the mixtures solution and continued to stirred for another 24 hours before being cast onto a finely cleaned petri dish before allowing it to evaporate slowly in a fume hood at room temperature for a day. After appropriate amounts of THF solvent dried off, the sample was further dried in a vacuum oven at 50˚C and 0.2 atm for 24 hours. The dried film was obtained after THF solvent has completely evaporated. The film was then peeled off form the dish. These steps were repeated for preparing PVC-LENR50 (30/70)-EC(70)-$LiClO_4$ with 10, 15, 20, 25, 30, 35 and 40 wt.% of $LiClO_4$. This process will produce a mechanically stable and a free standing electrolytes film.

2.4. Sample Characterization

The ionic conductivity measurement was performed by alternate current (AC) impedance spectroscopy using high frequency response analyzer (HFRA Solartron 1256, Schlumberger) in the frequency range of 0.1 Hz to 1 MHz. The electrolyte films were sandwiched between two stainless steel electrodes with a surface contact area of 2.0 cm^2 and mounted onto the holder. From the Cole-cole plots obtained, the bulks resistance, R_b (Ω) of the samples was determined with *Z*-View software. The conductivity was calculated based on the equation $\sigma = l / R_b A$, where l is the film thickness (cm) and A (cm^2) is the effective contact area of electrolyte and the electrode. The morphological studies on the fractured surfaces of polymer electrolyte samples were done using SEM with 2000× magnification at 25 kV electron beam. ATR-FTIR analysis was performed on polymer-based and lithium salt using the Perking Elmer Spectrum 2000 in the range of 4000 cm^{-1} to 500 cm^{-1} with its scanning solution of 4 cm^{-1}.

3. Result and Discussion

3.1. Ionic Conductivity

Figure 4 shows the variation of conductivity as a function of weight percentage of EC in PVC-LENR50 polymer blends. There is a significant conductivity enhancement after the addition of plasticizer to the electrolytes system. The lowest ionic conductivity obtained was 2.0×10^{-11} Scm^{-1} at 0 wt.% of EC content while the highest ionic conductivity was observed at 70 wt.% of EC loading with conductivity value of 6.4×10^{-9} Scm^{-1}. The conductivity of the blends has been improved by 326 times with introduction of 70 wt.% of EC. This step was carried out to optimize the coordination sites of the polymer host by increased the number of oxygen atom for lithium salt to interact. The increased in conductivity of the polymer blends system is due to the decreased of bulk resistance in the system.

Figure 5 shows the relationship between the ionic conductivity at the different concentrations of $LiClO_4$ salt at room temperature. It was observed that the ionic conductivity of PVC-LENR50 (30/70)-EC(70)-$LiClO_4$ salt increases from 0 wt.% $LiClO_4$ to 30 wt.% before started to drop until 40 wt.%. From **Figure 5**, the lowest ionic conductivity was 6.4×10^{-9} Scm^{-1} obtained at 0 wt.% $LiClO_4$ and the highest value of 2.1×10^{-7} Scm^{-1} was obtained at 30 wt.% $LiClO_4$. In **Figure 6** shows the bulk resistance which is described by an arc was interpreted from the simulation (semicircle) line performed on the impedance spectra which the slanted spike is represents the high frequency semi circular region, attributed to the electrolytes resistance [20]. The semicircle and a spike observed in the impedance plots indicated the occurrence of ion diffusion. The increase in the ionic conductivity after introduction of $LiClO_4$ salt into the electrolytes system was due to the fact of increasing charge carriers in the system increase. This can be shown by the equation: $\sigma = ce(u_+ + u_-) = c\Lambda$ where σ is conductivity of electrolytes, Λ is molar conductivity, c is salt concentration, e is charge on an electron, and u_+ and u_- represent the ion mobilities. As number of charges increases, the ionic conductivity also increases [18,19]. From previous researches, it's reported that plasticizer such as EC exhibit high dielectric constant which can increases the number of mobile ions by weakening the columbic force between the anions and cations of the salt [3]. Besides, plasticizer created and promotes more free volume in the electrolyte system and decreased the viscosity of the electrolyte making the mobility of ions became easier. Another factor contribute to the rise in ionic conductivity of the SPE is the large volume of ClO_4^- which may elongate the pitch of PVC spiral structure and thus provide a bigger transfer space for Li^+ ions [4]. However, after the optimum value of concentration $LiClO_4$ salt introduced, the ionic conductivity decreased. This implied that ions association was likely occurred in the electrolytes system. Ions association

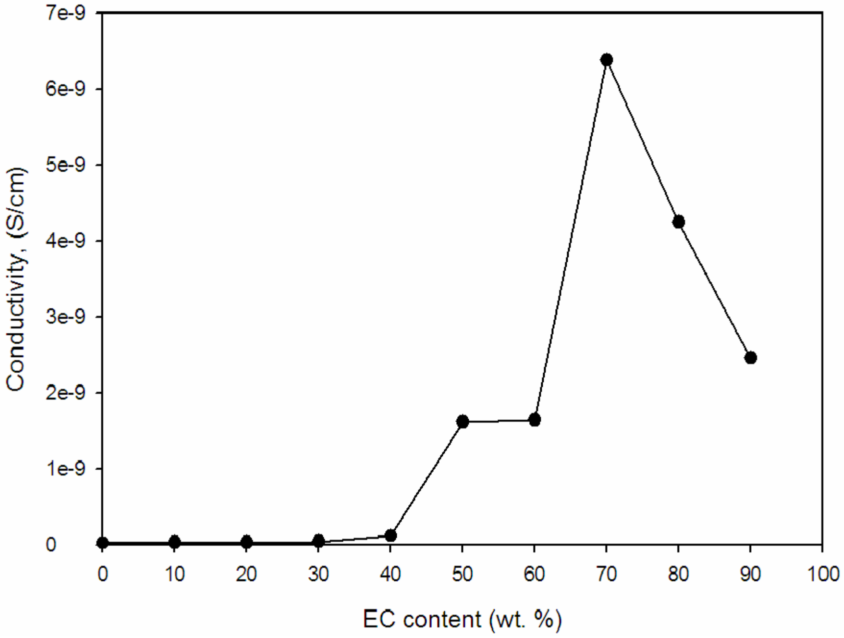

Figure 4. Variation of conductivity as a function of weight percentage of EC in PVC-LENR50 polymer blends.

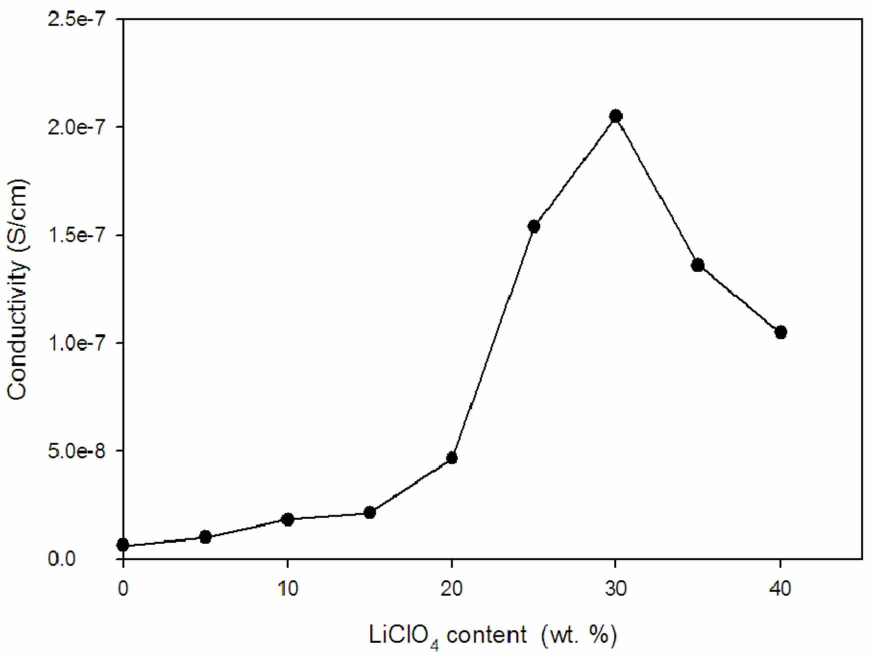

Figure 5. Ionic conductivity of PVC-LENR50 (70/30)-EC(70) doped with $LiClO_4$ salt.

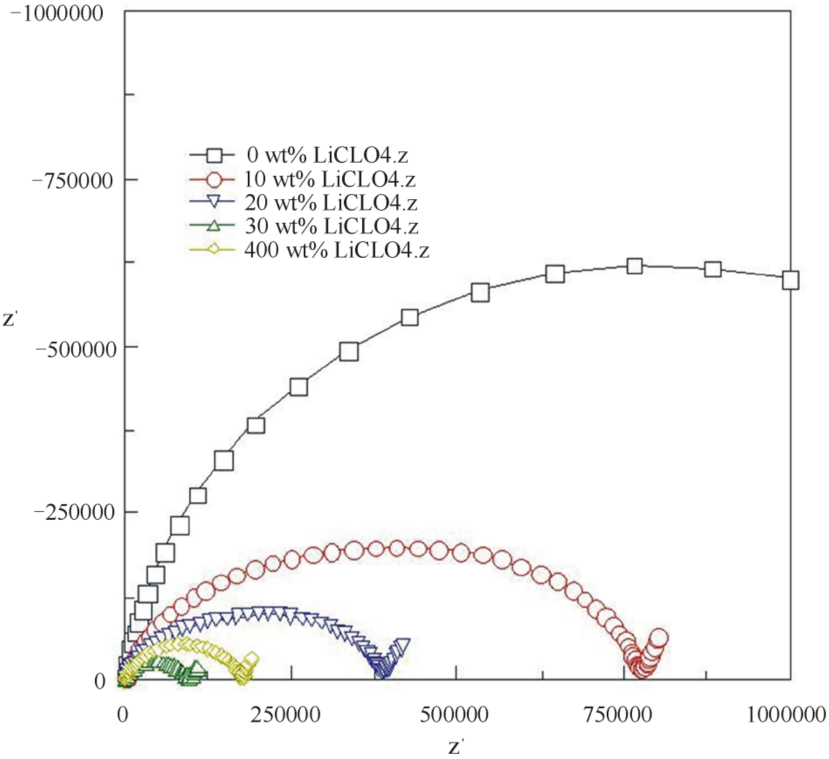

Figure 6. Typical Cole-cole plot for PVC-LENR50 (30/70)-EC(70)-$LiClO_4$.

causes the number of free ions to decrease which leads to the lower ionic conductivity values. This can be explained which at extreme low salts concentrations the salts exist in the form of isolated Li^+ and ClO_4^- ions. As the concentration increases, mutual interactions between ions are sufficiently strong to promote the formation of ion pairs, which are in equilibrium with the free ions: $Li^+ + ClO_4^- \rightleftharpoons [LiClO_4]^0$. Since the ions pairs carry no charge, the conductivity per unit salt concentration will drop as observed in **Figure 5**. The formation of the PVC -LENR50-EC-LiClO$_4$ complex is described in for which: [4,18-21]

$$LiClO_4 + LENR50 + EC \rightarrow Li^+(LENR50)_x (EC)_y]ClO_4^- \quad (1)$$

$$Li^+[(LENR50)_x (EC)_y]ClO_4^- + (CH_2CHCl)_n^- \rightarrow (CH_2CHCl)_n^- - Li^+[(LENR50)_x (EC)_y]ClO_4^- \quad (2)$$

3.2. Morphological Studies

The SEM micrographs shown in **Figure 7** were the surface morphology of the electrolytes systems which were characterized at the cross-sectional area. As we can see, the surface of pure PVC was rough and cracked

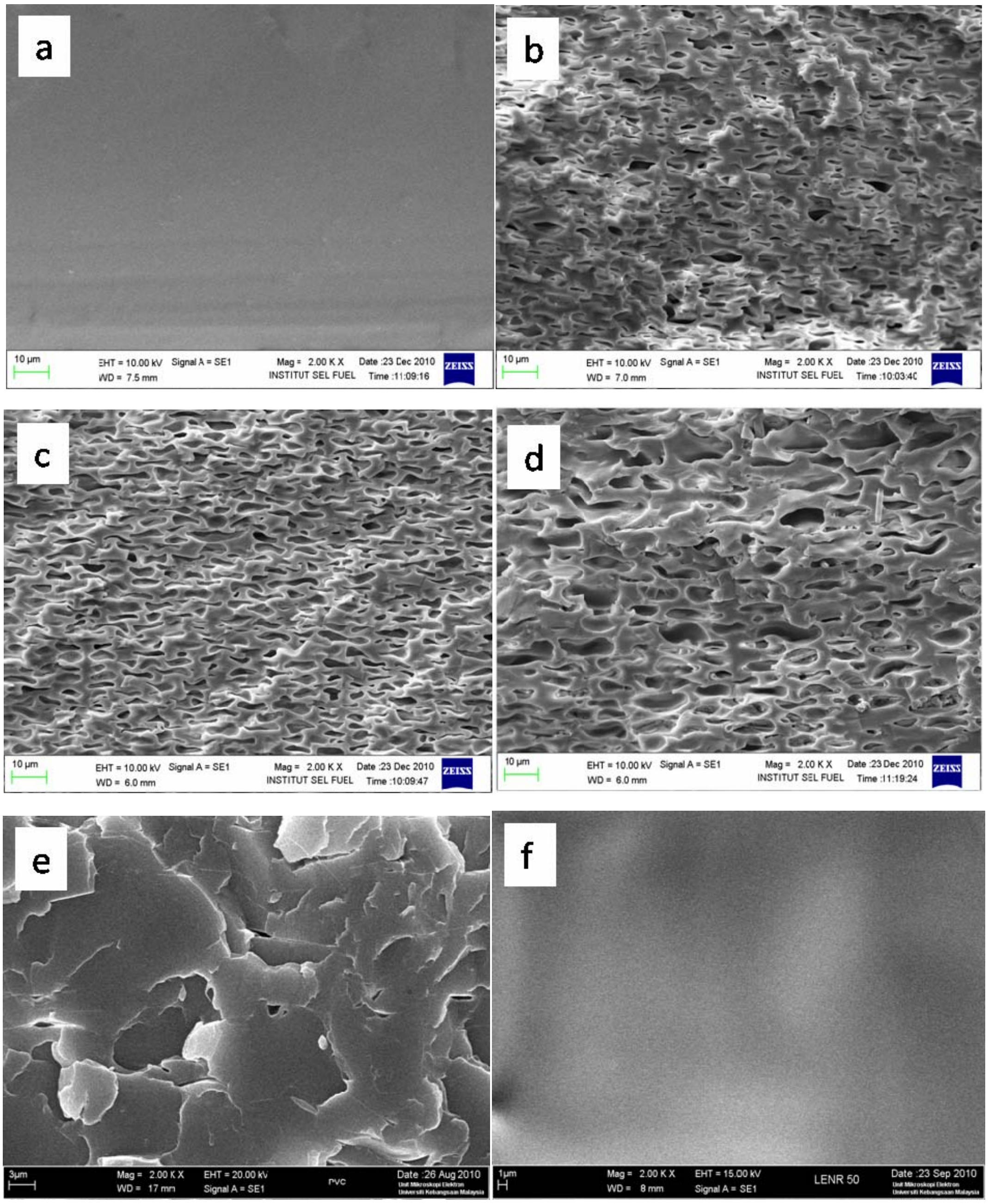

Figure 7. SEM micrograph of (a) SPE 0 wt.% salt, (b) SPE 10 wt.% salt, (c) SPE 30 wt.% salt, (d) SPE 40 wt.% salt, (e) pure PVC (f) pure LENR50.

whereas the surface morphology for LENR50 is smooth and clean. However, when PVC was blended with EC and LENR50, the brittleness and cracked surface was improved as shown in **Figure 7(a)**. It's observed that from **Figures 7(a)-(d)** indicated that LENR50 forms miscible blends with PVC and enhances the tear strength of the PVC, resulting better physical properties. In facts, LENR50 has better miscibility than ENR50 because LENR50 has larger inter-phasing area and higher interaction with PVC owning to its smaller molecular size [9,16]. According to the previous reports [3,22], ions can move more freely in the electrolytes with smoother surface.

The fractured structural view of the samples in **Figures 7(b)** to **(d)** shows the formation of micro-pores that occurs from the complex process of interaction between solvent, plasticizer, lithium salt and the polymers during evaporation. According to Watchanida Chinpa, the high porosity might result from the repulsive forces between the carboxylic acid groups with the polymers host which the carboxylic acid form from the OOH group [25]. Furthermore, it is also kinetically controlled by the relative rate of evaporation of the compounds. Nevertheless, the formation of the fine-pores in the polymer-salt matrix will improve the mobility of ions by providing and creating more path ways for ions transportation [23,24]. However, the analysis of impedance shows that after the addition of 30 wt.% of salt, the value of ionic conductivity dropped because the number of charge carriers decreased. The presences of higher lithium salt concentration can cause phase separation and agglomeration that hinder migration of Li^+ ions in the polymer, resulting in lower ionic conductivity. This was proved in **Figure 7 (d)** as the surface of the cross-sectional show brighter spots than **Figures 7(b)-(c)**. The bright spots were caused by the addition of lithium salt [14]. Besides, the agglomeration was clearly observed in **Figure 7(d)** too [3].

3.3. ATR-FTIR Studies

ATR-FTIR is a very useful tool to study the local structural changes as well as occurrence of the complexation and interaction between various constituents. The FTIR spectra of polymer electrolytes complexes are shown in **Figure 8**. The peaks observed at 3450 cm^{-1} to 3600 cm^{-1} shows OH and –OOH groups. This was caused by hygroscopic nature of lithium salt and THF solvent that absorbed moisture from the air. In addition, the –OOH group was resulted from the irradiation process of LENR50 which oxygen interact with unsaturated double bonds of the isoprene units to form hydroperoxide. While the 771 cm^{-1} peak referred to EC ring-stretching mode[27].

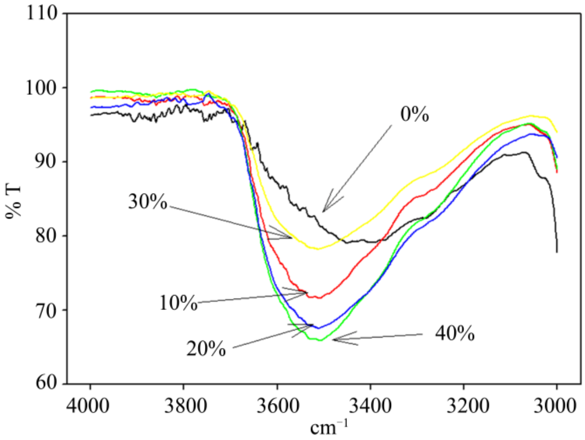

Figure 8. FTIR spectra for OH and OOH groups of PVC-LENR50-EC-LiClO$_4$ (0 wt.%, 10 wt.%, 20 wt.%, 30 wt.% and 40 wt.%.

The peaks 2963 cm^{-1}, 2923 cm^{-1}, and 2860 cm^{-1} shown in **Figure 9** were belong to saturated aliphatic stretching C-H of isoprene. There were significant changes as the intensity of the peaks became less sharp when the concentration of lithium salt increased. From **Figure 10**, the absorption peaks at 1449 cm cm^{-1} and 1329 cm^{-1} are corresponding to CH_2 scissoring of isoprene and CH_2 wagging of PVC respectively [2]. It was clearly seen that the peak at 1329 cm^{-1} and disappeared upon adding of lithium salt. The peaks at 1449 cm^{-1} was shifted to 1481 cm^{-1} and the intensity decreased, while peak at 1380 cm^{-1} shifted to 1403 cm^{-1}.

The peak absorption of C-O-C group was found at 1251 cm^{-1} and 873 cm^{-1} were from the epoxy group of the isoprene units. Peak at 1251 cm^{-1} gradually

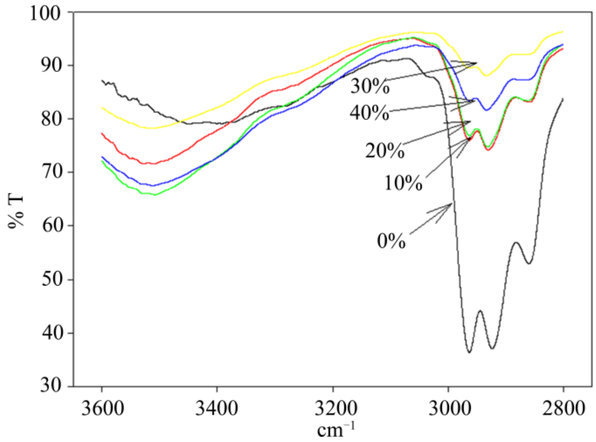

Figure 9. FTIR spectra for aliphatic stretching CH of PVC-LENR50-EC-LiClO$_4$ (0 wt.%, 10 wt.%, 20 wt.%, 30 wt.% and 40 wt.%).

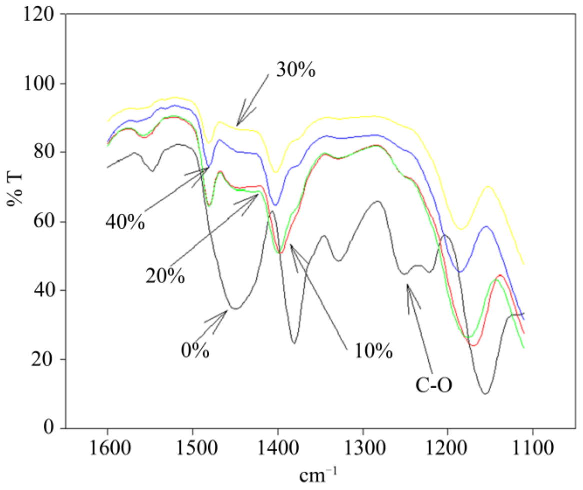

Figure 10. FTIR spectra for CH_2 of PVC-LENR50-EC-$LiClO_4$ (0 wt.%, 10 wt.%, 20 wt.%, 30 wt.% and 40 wt.%).

disappeared and peak at 873 cm^{-1} shifted to 901 cm^{-1} when the lithium salt added [5]. This shows that the complexation between Li$^+$ ions and ether group occurred. Moreover, the vibrational peak observed at 711 cm^{-1} and shifted to 721 cm^{-1} was attributed to Li$^+$ ions.

The peaks at 1804 cm^{-1} and 1774 cm^{-1} peaks observed for the complexes of lithium with C = O of LiCO which was shown in **Figure 11** [26]. The intensity of C = O peak decreased and shifting was observed too as salt was introduced. The peak shifted slightly from 1804 cm^{-1} to 1802 cm^{-1} while peak at 1774 cm^{-1} shifted to 1768 cm^{-1}. The shifting and changes in intensity peaks as well as shape of the peaks shows that complexation between the lithium salts and the oxygen atoms in the polymer host occurred.

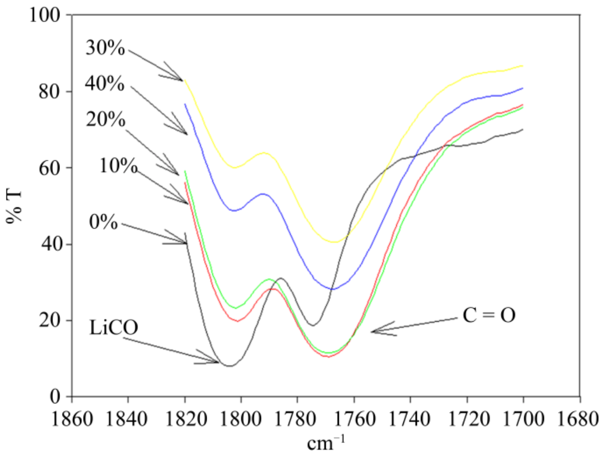

Figure 11. FTIR spectra for C = O of PVC-LENR50-EC-$LiClO_4$ (0 wt.%, 10 wt.%, 20 wt.%, 30 wt.% and 40 wt.%).

4. Conclusions

Solid polymeric electrolyte of PVC-LENR50 (30/70)-EC (70) with function of $LiClO_4$ salt concentration was successfully prepared by solution casting technique. The highest ionic conductivity obtained is 2.1×10^{-7} S cm^{-1} at 30 wt.% of $LiClO_4$ salt. The above results also show that the conductivity of the polymer blends of PVC-LENR50 with addition of EC can lower the resistance bulk of the system as the flexibility of the polymer host increased. The SEM studies showed that PVC, LENR50 and EC were well miscible as suggested by other researchers. It also reveals that the SPEs with lithium salts produce almost consistent sizes and well-distributed micro-pores. The micro-pores aid in mobility of the ions in the system complexes. Results of the ATR-FTIR proved that complexation occurred between Li$^+$ ions and C-O-C as well as C = O groups.

5. Acknowledgements

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