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Conductivity and Morphological Studies of Poly(Vinyl Alcohol)-Magnesium Triflate-Ethylene Carbonate Gel Polymer Electrolyte

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ABSTRACT

Gel polymer electrolyte based on poly (vinyl alcohol)(PVA)–magnesium triflate (MgTf) incorporating with different weight percentage (wt.%) of ethylene carbonate (EC) as plasticizer has been prepared by using solution casting method. The ionic conductivity of PVA–MgTf–EC gel polymer electrolyte has been determined by using Electrochemical Impedance Spectroscopy (EIS) and has achieved an optimum value at 1.26 x 10^{-4} *S.*cm^{-1} *when 50 wt.% of EC is added into the system. Fourier Transform Infrared Spectroscopy (FTIR) has been performed to study the molecular interaction between components in PVA-MgTf-EC gel polymer electrolyte. The complexations between the materials used in the electrolyte system are observed based on the shifting of bands and changes in the intensity of peaks in FTIR spectra. The morphological behaviour of electrolytes has been studied by using Field Emission Scanning Electron Spectroscopy (FESEM) to observe the changes of PVA-based gel polymer electrolytes when EC is added to the system. The amorphous peak of PVA-based electrolyte is observed to become broaden with the addition of plasticizer, results in increment in the amorphousness nature of the electrolyte which is determined by X-ray Diffractometer (XRD).*

Keywords: gel polymer electrolyte, PVA, magnesium salt, plasticizer, conductivity, FTIR

INTRODUCTION

Polymer electrolytes have drawn the attention of many scientists since it has been proposed due to its ability to overcome the limitation of classic liquid organic electrolytes such as high vapor pressure low electrochemical stability and high ability to support combustion [1]. The first development of polymer electrolyte was the introduction of solid polymer electrolyte to be implemented in batteries by Wright and Armand in the late 1970s [2]. Solid polymer electrolyte (SPE) possesses non-leakage electrolyte, promising low flammability and high safety, high flexibility, and electrode-electrolyte stability [3]. However, the SPE consists of limitations in producing high ionic conductivity, thus, deteriorate the performance of the electrolyte in the technological application [4]. Therefore, an approach has been made by introducing gel polymer electrolyte (GPE) as an alternative. GPE holds the same advantages as SPE but it can exhibit a high ionic conductivity compared to SPE [5]. GPE is presented in form of thin film by dissolving gelled matrix form solution that consists of polymer host, inorganic salt, plasticizer and solvent [6].

Poly (vinyl alcohol) PVA is a biodegradable and biocompatible polymer that appears semicrystalline in nature, non-toxic, and soluble in water [7]. The usage of PVA in polymer electrolytes has been received great attention among researchers due to its availability, low cost, high film-forming capacity, thermal stability, good chemical and mechanical strength [8]. PVA is found to produce a good polymer electrolyte as it has high hydrogen bonding due to the structure of PVA that consists of a carbon chain backbone structured with a hydroxyl (OH) group linked with the methane carbons [9]. The incorporation of dopant is crucial in producing gel polymer electrolytes as it will promote ionic conduction in the polymer matrix. Most of the alkaline salt that has been used in the production of an electrolyte is Lithium (Li) salt. Due to the drawbacks of Li, which is high reactivity, relatively expensive, and facing problems in safety issues, magnesium (Mg) salt has been chosen as an alternative to be doped in the polymer matrix [10]. Mg is safer to be used as it is environmentally friendly, non-toxic, chemically stable, and can be fabricated without any protection during the preparation process [11–13] .

In order to produce a good performance electrolyte, the ability of a polymer electrolyte system to exhibit a high value of ionic conductivity becomes the priority as it will affect the electrical properties of the electrolyte. However, the incorporation of salt in a polymer matrix is only unable to achieve this target [14]. Therefore, a few approaches have been studied by the researchers to overcome this problem such as by plasticization [15–18], addition of filler in a polymer matrix [19–21] and polymer blending [22, 23]. In this study, a plasticizer, ethylene carbonate (EC) is added to this PVA-based electrolyte system to overcome this limitation and enhance the performance of electrolytes. The addition of plasticizer is found able to promote the amorphous nature of polymer-salt complexes and increase the polymer chain flexibility. The properties of high dielectric constant EC [24] itself contribute to the promotion of ion-pair/aggregation of salt in this GPE system [25]. This is proven by a study done by Hafiza and Isa [18], which recorded an ionic conductivity at 1.17 x 10^{-3} $S.cm^{-1}$ by incorporating 16 wt.% of EC in 2-hydroxyethyl cellulose (2-HEC)–ammonium nitrate (NH_4NO_3) solid polymer electrolyte system.

Therefore, in this present study, a system of poly (vinyl alcohol) (PVA)–based gel polymer electrolyte doped with magnesium triflate (MgTf) and different weight percent (wt.%) of ethylene carbonate (EC) in distilled water is prepared by using solution casting method. The electrical and morphological behavior of this system is determined by electrochemical impedance spectroscopy (EIS), Fourier transform infrared (FTIR) spectroscopy, field emission scanning electron microscopy (FESEM), and X-ray diffractometer (XRD).

EXPERIMENTAL

Preparation of Gel Polymer Electrolyte

Polyvinyl alcohol (PVA)–Magnesium triflate (MgTf)–ethylene carbonate (EC) gel polymer electrolyte (GPE) films have been prepared by using the solution casting technique. 1.0 g amount of PVA, 40 wt.% of MgTf salt (0.429 g), and different weight percentages of EC (0–50 wt.%) are dissolved in distilled water. The mixture has been magnetically stirred continuously for several hours until a homogenous solution has been obtained. Then, the obtained solution is cast into a petri dish and let dry until a transparent thin film of GPE is formed. The sample of PVA–MgTf electrolyte with addition 0, 10, 20, 30, 40, and 50 wt.% of EC has been denoted as PVA-0, PVA-10, PVA-20, PVA-30, PVA-40, and PVA-50, respectively.

Electrochemical Impedance Spectroscopy

Impedance measurement has been run by using HIOKI 3532-50 LCR Hi-tester at ambient temperature in the frequency range of 100 Hz to 1 MHz. The sample is sandwiched between two stainless steel blocking electrodes with a contact area of 0.2827 cm^2. The conductivity can be calculated by using the formula,

$$\sigma = \frac{t}{R_b A} \tag{1}$$

where σ is the ionic conductivity of sample in S.cm^{-1}, t is the thickness of thin-film in cm, R_b is the bulk resistance that is determined from *Cole-Cole* plots and A is the contact area of electrode-electrolyte in cm^2.

Fourier Transform Infrared Spectroscopy

Fourier transform infrared spectroscopy (FTIR) has been carried out at room temperature by using a Perkin Elmer FTIR model Spectrum 400 at a spectral range between 600 to 4000 cm^{-1} and a spectral resolution of 1 cm^{-1}. This measurement is run by using the Attenuated Total Reflectance (ATR) technique.

Field Emission Scanning Electron Microscopy

Surface morphology of PVA–based gel polymer electrolyte (GPE) with different weight percent of ethylene carbonate (EC) has been measured by using JSM-5600 Field Emission Scanning Electron Microscopy (FESEM) at a size of 10 µm in 1000 magnification. The image of the surface of the electrolyte that records the information such as size, shape, and arrangement of gel polymer electrolyte system can be observed.

X-Ray Diffraction

The X-ray diffraction (XRD) analysis was conducted to determine the structural behaviour of gel polymer electrolytes (GPE) films which are observed by the presence of crystalline or amorphous in nature in the GPE system. The XRD was recorded at room temperature by using PANalytical X'pert PRO diffractometer with CuKα radiation (λ=1.5418 Å) in the range of $10° \leq 2\theta \leq 90°$.

RESULT AND DISCUSSION

Conductivity Studies

AC impedance spectroscopy is a tool for characterising the electrochemical behaviour of an electrolyte, where can determine the ionic conductivity of an electrolyte. The value of R_b can be obtained from the intersection of the Nyquist plot and the real axis. The Nyquist plot, which is also known as impedance and *Cole-cole* plot is illustrated in the graph of the imaginary impedance component (Z") against the real imaginary component (Z'). This plot consisted of two regions, high and low-frequency regions. The high-frequency region that is usually depressed in semicircles shows the bulk effect of polymer electrolyte, which is equivalent to the parallel combination of bulk resistance and capacitance. While the straight sloping line on the low-frequency region is related to the migration of ions, which results in the double-layer capacitance formation at the electrode and electrolyte interface [6].

Figure 1 shows the ionic conductivity of the PVA–MgTf system at different concentrations of ethylene carbonate (EC) at room temperature. When MgTf salt was doped to the PVA polymer matrix without the presence of plasticizer EC, the conductivity that has been obtained is 1.82 x 10^{-8} S.cm^{-1}. However, the ionic conductivity is increasing when EC is added to the system. The addition of a plasticizer to the system has enhanced the ionic conductivity to 2.85 x 10^{-6} S.cm^{-1} at 20 wt.% EC and further increased to the optimum value (1.26×10^{-4} S.cm^{-1}) when 50 wt.% EC is added. The enhancement of ionic conductivity happened due to the increasing degree of salt dissociation, which promotes ion mobility, thus improve the conduction process of this system [18]. Furthermore, the increment in ionic conductivity also contributes to the rise of the amorphous nature of electrolytes due to the high segmental motion and flexibility of polymer chains [9, 26].

Fourier Transform Infrared Studies

The possible molecular interaction and complexation of the constituents in the PVA–MgTf–EC gel polymer electrolyte system can be identified by using the Attenuated Total Reflectance-Fourier Transform Infrared (ATR-FTIR) spectroscopy technique.

Figure 2 shows the interaction between pure PVA and PVA–MgTf–EC gel polymer electrolyte. Figure 2 (a) explains the band observed for pure PVA. At the wavenumber of 3000-3500 cm^{-1} region, O–H stretching is observed due to the inter/intra-molecular hydrogen bond presence in PVA polymer. It is supported by Polu & Kumar [27] that had found O–H stretching at 3436 cm^{-1}. While the CH_2 asymmetric stretching band is recorded at 2492 cm^{-1}. At a wavenumber of 1773 cm^{-1}, the absorption band is referring to C=O stretching that is present in the PVA structure. It is almost similar to the studies done by Hema *et al.* [28] and Polu and Kumar [27] that obtained 1736 cm^{-1} for C=O stretching.

The shifting and changes in the intensity of the peak give the information of interaction of constituents in the polymer matrix. Addition of MgTf in the PVA polymer results in the changes of stretching vibration O–H, where the peak and intensity shifted to 2500–3000 cm^{-1} region as illustrated in Figure 2(b). Furthermore, the strong intermolecular interaction between Mg salt and oxygen atoms in polymer–salt complexes contribute to the shifting of carbonyl group C=O. Moreover, a co-ordinate bond will be formed between the oxygen atoms from the PVA polymer, which act as electron donors with magnesium ions from the salt [29]. However, the carbonyl bond peak is disappeared when salt is added to the PVA matrix due to the complexation of polymer.

Figure 2 (c) and (d) indicate the interaction that occurs between PVA–MgTf–EC in the gel polymer electrolyte system. Compared to the pure PVA spectrum, the CH_2 asymmetric stretching has been shifted to 3050 cm^{-1} and C=O stretching shifts to 1805 cm^{-1} due to the increased concentration of EC added to the PVA-based gel polymer electrolyte system. While the O-H band is gradually shifted from 3299 cm^{-1} to 3340 cm^{-1} wavenumbers.

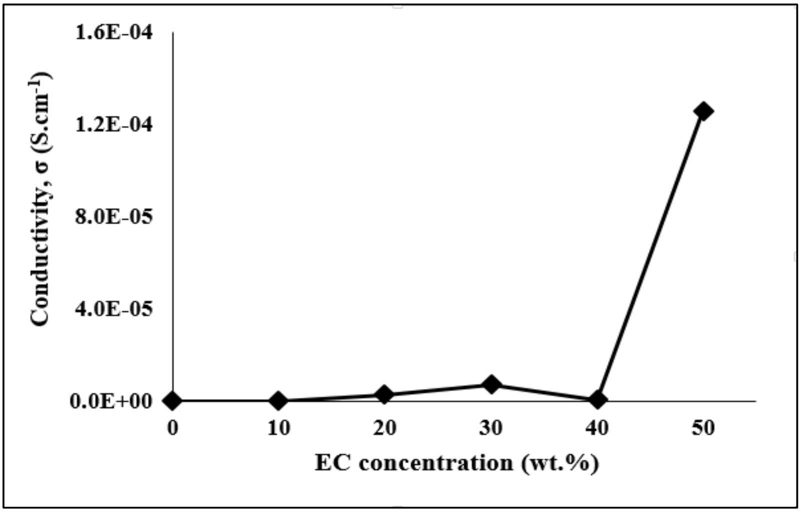

Figure 1: Ionic Conductivity of PVA--MgTf--EC System at Different Concentrations of EC at Room Temperature

Morphological Studies

Morphological studies are conducted by using field emission scanning electron microscope (FESEM) to give an insight into the surface of the gel polymer electrolytes. The FESEM images of pure PVA, PVA–based gel polymer electrolyte without the presence of EC (PVA–0), and PVA–based gel polymer electrolyte with 20 wt.% of EC (PVA–20) membranes are shown in Figure 3. For pure PVA, the network of the polymer does not form clearly due to the higher crystallinity of the polymer. Then, the network started to be observed when MgTf is added to the PVA–based gel polymer electrolyte due to the reduction of the crystalline phase of the system as shown in Figure 3 (b). After 20 wt.% of EC is added to the PVA–MgTf system, the networks that have been reported in Figure 3 (c) are observed on a smooth and clearer surface which results from the complete dissolving of MgTf and EC in the PVA polymer matrix. Moreover, it shows the enhancements of the amorphous region of this system as the fact that 20 wt.% EC exhibits a higher ionic conductivity compared to 0 wt.% of EC added to the PVA–based gel polymer electrolyte. The network formed in the GPE system with the addition of 50 wt.% of EC become more obvious and increase, this indicates that the highest rate of interaction between EC and PVA in the system, which contribute to the highest ionic conductivity.

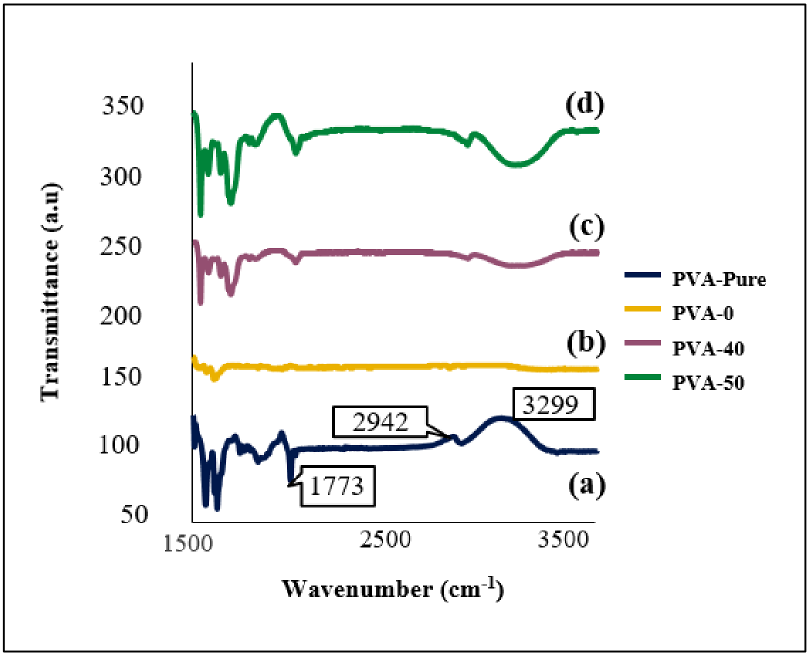

Figure 2: FTIR Spectrum of (a) PVA-pure, (b) PVA-0, (c) PVA-40 and (d) PVA-50

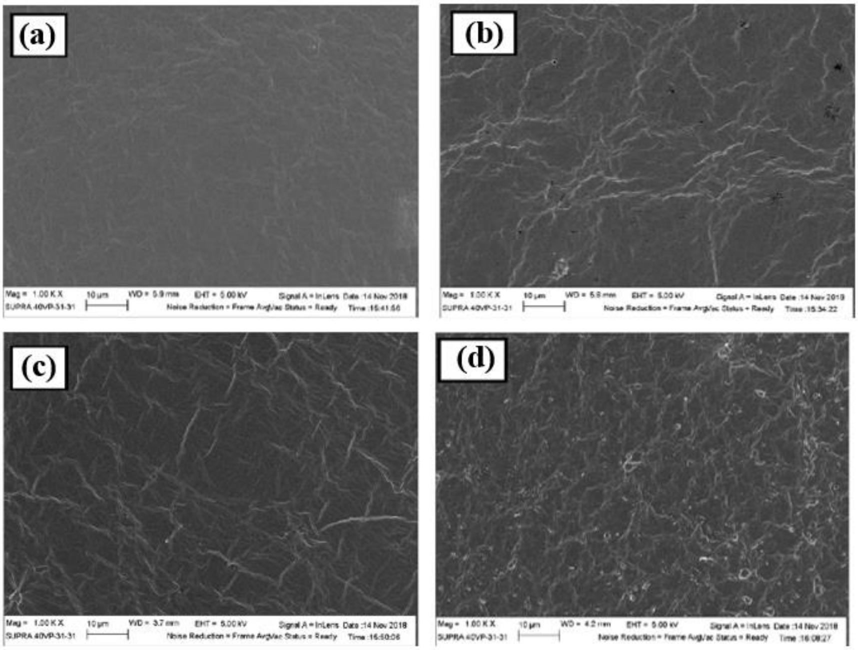

Figure 3: The Surface Image of (a) Pure PVA, (b) PVA–0 (c) PVA–20 and (d) PVA–50

X-Ray Diffraction Studies

X-ray diffraction was run to determine the structural behaviour of plasticized PVA-based GPE by observing the presence of amorphous or/ and crystalline peaks as illustrated in Figure 4. The semicrystalline nature of PVA is proven by the presence of an amorphous peak at $2\theta = 20^{\circ}$ [30–32] in Figure 4 (a). Upon addition of EC in the PVA-MgTf system, the intensity of the peak for the selected samples in Figure 4 (b) and (d) become decreases, thus prove the decrease in crystallinity of GPE. Furthermore, the amorphous peak becomes broader with the addition of plasticizer to PVA–based electrolyte, which again indicates the increase in the amorphous nature of GPE. The broadest peak is observed for the sample of PVA–MgTf–50 wt.% EC system due to the highest amorphous region that contributes to high ionic diffusity and optimum ionic conductivity, thus support the discussion of ionic conductivity result [30].

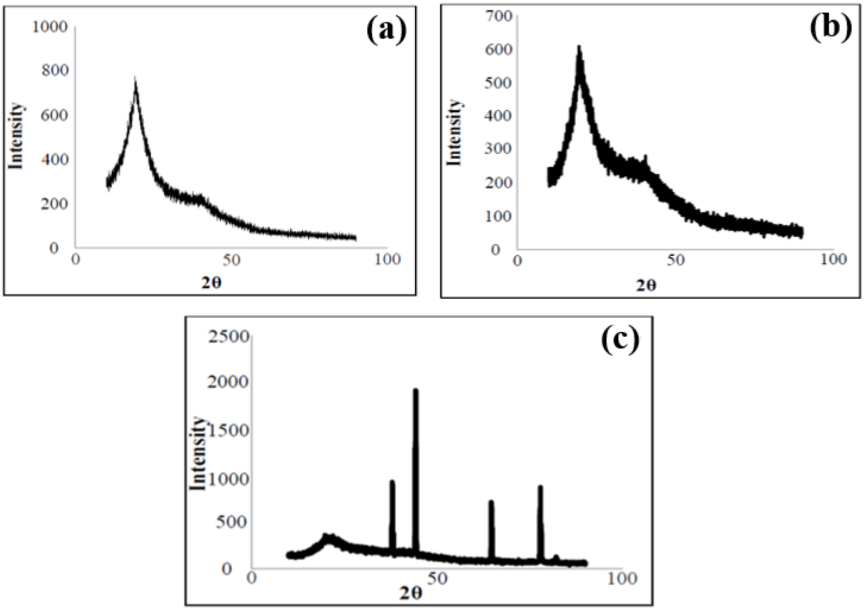

Figure 4: XRD Pattern of (a) Pure PVA, (b) PVA–40 and (c) PVA–50

CONCLUSION

Gel polymer electrolyte system that consists of poly (vinyl alcohol) (PVA), magnesium triflate (MgTf), and ethylene carbonate (EC) has been successfully prepared by using the solution casting method. PVA and MgTf are dissolved in distilled water and EC is added in different weight percent. The thin film of PVA–MgTf–EC gel polymer electrolyte has been characterised using EIS to determine the electrical behaviour of the electrolyte. The highest amount of ionic conductivity has been obtained after 50 wt.% of EC is added to PVA–MgTf system at a value of 1.26 x 10^{-4} $S.cm^{-1}$. It shows that the addition of EC to the PVA–MgTf system can increase the ionic conductivity of gel polymer electrolytes due to the increased number of a charge carrier or free mobile ions. While for FTIR spectroscopy, the molecular interactions between PVA–MgTf–EC have been studied and the presence of O–H, CH_2, and C=O stretching peak is observed. For morphological studies, FESEM images clearly show the compatibility of networks between the constituents of PVA–based gel polymer electrolyte when 50 wt.% of EC is added to the system. The highest amorphousness nature of GPE is recorded at 50 wt.% addition of EC by observing the broadest and the lowest intensity of the amorphous peak XRD pattern. The results obtained in FTIR, FESEM, and XRD are in accordance with the composition of EC in PVA–gel polymer electrolyte that exhibits the highest value in ionic conductivity.

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