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Development and Analysis of Novel Silver Ion-Conducting Glass-Polymer Electrolytes

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Abstract

The development of efficient and stable solid electrolytes is crucial for advancing energy storage technologies such as solid-state batteries and electrochemical devices. In this study, a novel series of silver ion-conducting glass–polymer electrolytes (GPEs) based on the composition (1–x) PEO: x[0.75AgI:0.25(Ag_2O:WO_3)] with x ranging up to 50 wt.% was synthesized and thoroughly analyzed. Unlike conventional techniques such as solution casting or sol–gel methods, these GPEs were fabricated using an innovative hot-pressing approach. The composition 70PEO:30[0.75AgI:0.25(Ag_2O:WO_3)] demonstrated the highest ionic conductivity (~6.5 × 10^{-7} S·cm ⁻¹), and is designated as the optimum conducting composition (OCC). Detailed investigations into polymer–salt interactions and material morphology were carried out using scanning electron microscopy (SEM) and differential scanning calorimetry (DSC). Ionic conductivity (σ) enhancements have been explained by the ionic mobility (μ) and mobile ion concentration (n) measurements with the help of transient ionic current (TIC) technique. Activation energy (E_a) of GPE OCC have been measured with the help of temperature dependent conductivity measurement. The synthesized glass polymer electrolyte exhibits desirable physical, thermal, and electrochemical properties, making it a promising material for future applications in solid-state ionic devices. The combination of high ionic conductivity, structural stability, and good processability presents a compelling case for further investigation into its integration in practical energy storage systems.

Keywords: Glass polymer electrolytes, SEM, DSC, Ionic mobility, Mobile ion concentration.

INTRODUCTION

Solid polymer electrolytes (SPEs) and glass polymer electrolytes (GPEs), appear to be the suited solid electrolytes for applications in solid state devices [1–3]. These ether groups have minimal reactivity and solvate lithium ions in a similar way to crown ethers, boosting safety. Despite their potential, PEO-based polymer electrolytes generally exhibit limited ionic conductivity at room temperature typically in the range of 10^{-7} to 10^{-8} S·cm^{-1} due to reduced charge carrier mobility in their predominantly crystalline state below the melting transition [4, 5]. Flexible solid polymer electrolytes (SPEs) composed of PEO, along with glass–polymer electrolytes (GPEs), are considered promising candidates for applications in advanced energy storage and conversion devices, including polymer-based batteries, smart sensors, electrochromic windows, and various other electrochemical systems. GPEs, in particular, have emerged as a viable class of materials for solid-state devices due to their ability to sustain high ionic conductivity by preserving an amorphous phase. These materials also offer improved interfacial

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compatibility between the electrolyte and electrodes, enhanced ion transport numbers, reduced PEO crystallization rates, and extended operational lifespans [6–8].

Traditionally, SPE and GPE films are fabricated via the solution-casting technique; however, a more recent and efficient hot-pressing method has been introduced for their preparation [9-11]. In this work, we apply the hot-press technique to synthesize a new GPE system with the composition (1–x) PEO: x[0.75AgI:0.25(Ag_2O: WO_3)], where x ranges up to 50 wt.%. The structural and thermal characteristics of the resulting materials were assessed using scanning electron microscopy (SEM) and differential scanning calorimetry (DSC). Additionally, we evaluated the room-temperature ionic conductivity (σ), ionic mobility (μ), charge carrier concentration (n), and ionic transference number (t_{ion}) to better understand the conductivity behaviour of the synthesized electrolytes.

MATERIALS AND METHODS

Analytical reagent (AR) grade materials were utilized in the preparation of the glass–polymer electrolytes (GPEs) with the composition (1–x) PEO: x[0.75AgI:0.25(Ag_2O:WO_3)], where x varies up to 50 wt.%. The starting chemicals included poly(ethylene oxide) (PEO) with a molecular weight of 10^5 (Aldrich, USA), silver iodide (AgI, >98% purity), silver oxide (Ag_2O, >98% purity), and tungsten trioxide (WO_3, 99% purity). Firstly, the host glassy salt: [0.75AgI:0.25(Ag_2O:WO_3)] in mol. wt.% was prepared by melt-quenching technique at ~ 700 °C. The details of the melt-quenching technique were reported in the literature [12,13]. Dry powders of PEO and glassy salt were thoroughly combined and heated to a temperature of 70 °C (far above the melting point of PEO) for about 15 minutes to create a homogeneous slurry for the manufacture of GPEs. The slurry was subsequently compressed (at a rate of around 1.25 tons per square centimeter) between two SS blocks, which produced a mechanically stable polymeric GPE film with a thickness of about 0.024 cm.

Scanning electron microscopy (SEM) [model: JEOL, JXA-8100, Japan] and differential scanning calorimetric (DSC) analysis [model: Perkin Elmer] have been used to characterize the materials and the polymer-salt complexation. To explain the ion conduction behavior in the present system, ionic conductivity (σ), ionic mobility (μ) and ionic transference number were also determined by using the various experimental techniques in a wide range, as described in our earlier communication [14].

RESULTS AND DISCUSSION

The compositional dependence conductivity (σ) for the silver ion conducting glass electrolytes: [y AgI : (1-y) (Ag_2O:WO_3)], where y in mol. wet.%, synthesized by melt-quenching technique is shown in Figure 1. The conductivity of the glass system initially increased, peaked at x = 0.75, and then started to decline. The composition 0.75AgI:0.25(Ag_2O:WO_3), which exhibits a room temperature ionic conductivity of approximately 3.16×10^{-3} S·cm^{-1}, was selected as the host salt for fabricating the current series of glass–polymer electrolytes (GPEs) with the general formula (1–x) PEO: x[0.75AgI:0.25(Ag_2O:WO_3)], where x ranges up to 50 wt.%.

The variation in room temperature conductivity (σ) for hot-pressed GPEs is shown in Figure 2 for various salt concentrations. Upon the addition of salt to the host polymer, the conductivity abruptly increased (10^2 times). At x = 30 wt.%, a moderately sizable -maxima appeared, however, form, and it subsequently shrank with additional salt addition. Beyond a salt content of 50 wt.%, GPE films became fragile and showed signs of physical instability.

Among the prepared compositions, the GPE film with 70PEO:30[0.75AgI:0.25(Ag_2O:WO_3)] demonstrated the highest ionic conductivity, approximately 6.5×10^{-7} S·cm^{-1}, and is identified as the Optimum Conducting Composition (OCC). This notable enhancement in conductivity by nearly three orders of magnitude is primarily attributed to a higher degree of amorphous character in the material.

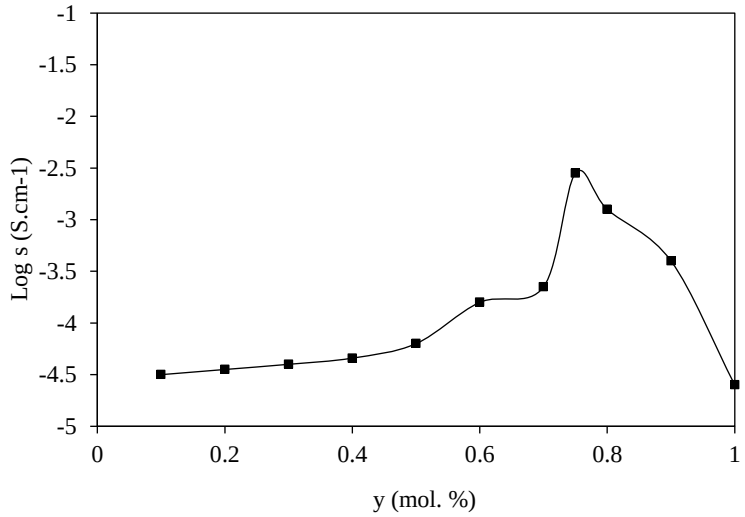

Figure 1. 'Log σ – y' Plot for Glassy Electrolytes: y AgI: (1-y) [Ag_2O:WO_3].

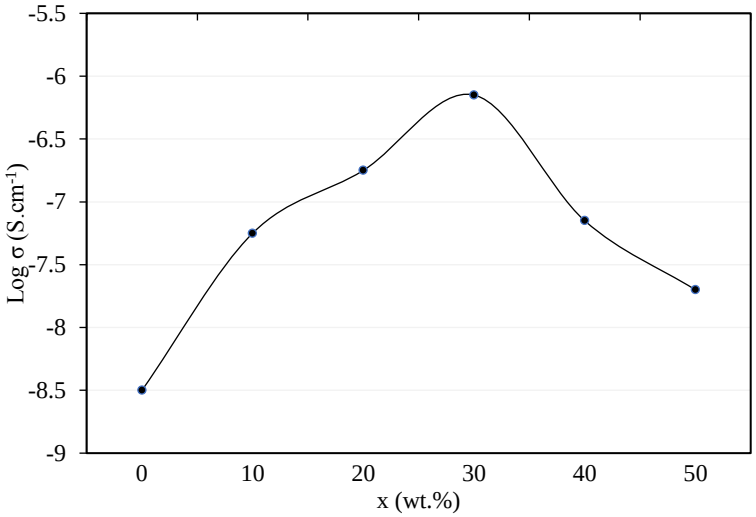

Figure 2. 'Log σ – x' Plot for GPEs: (1-x) PEO: x [0.75AgI:0.25(Ag_2O:WO_3)] where 0≤x≤50 wt.%.

Figure 3 presents the variation of ionic mobility (μ) and mobile ion concentration (n) for the GPE system with the composition: (1–x) PEO: x[0.75AgI:0.25(Ag_2O:WO_3)], where x ranges up to 50 wt.%. The data clearly show that both μ and n reach their maximum values at x = 30 wt.% under ambient conditions. This suggests that the enhanced room-temperature conductivity observed in the optimum composition is the result of simultaneous increases in both ionic mobility and the concentration of charge carriers.

Table 1 lists the room temperature values of some important ionic parameters of pure PEO and GPE OCC for direct comparison. Figure 4 shows the surface morphology (SEM) of GPE OCC: 70PEO: 30[0.75AgI:0.25(Ag_2O: WO_3)]. The fine surface structure of GPE OCC clearly indicates an increase in percentage of amorphicity while pure PEO shows the rough surface morphology [11]. This is because the host glassy salts and polymer have created a cross-link. With the use of the extremely helpful DSC technique, the thermal characteristics of GPE OCC and pure PEO have been identified. Figure 5 displays the DSC thermograms for both pure PEO and the optimum conducting composition (GPE OCC). The large endothermic peak was seen in SPE OCC at 66–67 °C, which is the same as pure PEO's melting point. The complexation of the Ag^+ ion with pure PEO's ether oxygen causes the melting point temperature to slightly shift to the lower side, which also indicates an increase in ionic conductivity.

Table 1. Some Important Ion Transport Parameter Values of σ, μ, n, t_{ion}, and E_a for the GPE OCC, σ – Value of Pure PEO.

Film	σ (S/cm)	μ ($cm^2 V^{-1} s^{-1}$)	n (cm^{-3})	t_{ion}	E_a (eV)
Pure: PEO	3.2×10^{-9}	-	-	-	-
GPE OCC: 70PEO:30[0.75AgI:0.25(Ag_2O:WO_3)]	6.5×10^{-7}	1.54×10^{-3}	5.88×10^{14}	0.98	0.37

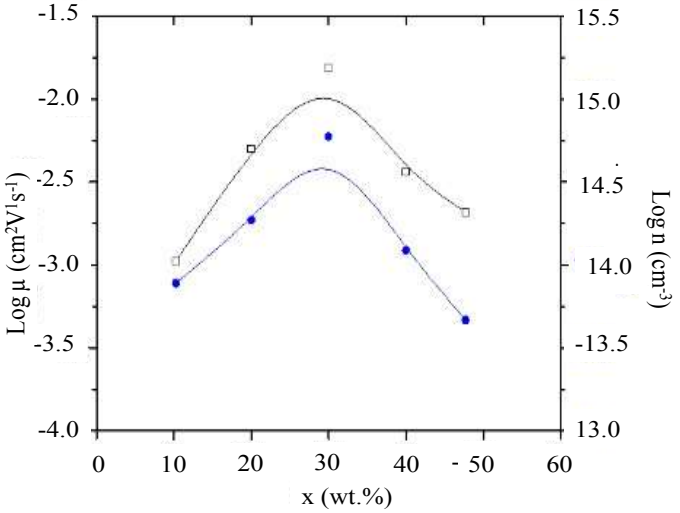

Figure 3. 'Log μ-x' and 'log n-x' Plots for GPEs: (1-x) PEO: x [0.75AgI:0.25(Ag_2O:WO_3)].

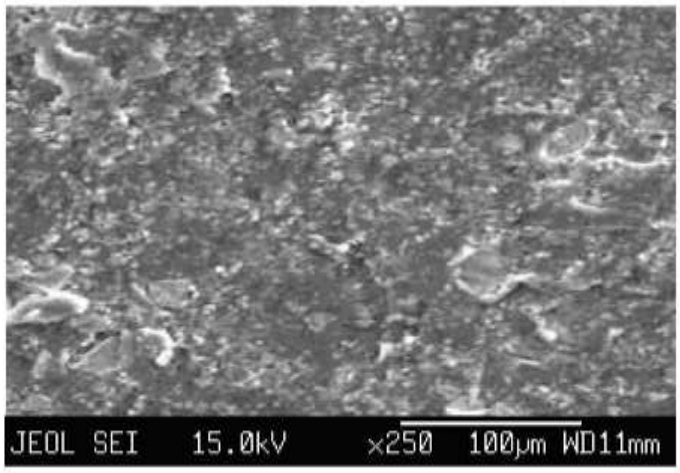

Figure 4. SEM Picture of GPE OCC: 70 PEO: 30 [0.75AgI:0.25(Ag_2O:WO_3)].

Figure 6 illustrates the current versus time response for the optimum conducting composition (GPE OCC), specifically 70PEO:30[0.75AgI:0.25(Ag_2O:WO_3)], measured at room temperature. t_{ion} was calculated with the help of the following equation:

$$t_{ion} = 1 - (I_e/I_T) \quad (1)$$

where I_e is the electronic current and I_T is the total current. t_{ion} ~ 0.98 was found by utilising the aforementioned equation, and it showed that the majority of charge carriers (98%) are cations Ag^+ and that other charge carriers account for just a very tiny proportion (2%). t_{ion} is nearly one, making the current GPE systems ideal for creating of electrochemical devices.

Figure 7 shows the temperature-dependent conductivity variation of GPE OCC: 70PEO: 30[0.75AgI:0.25(Ag_2O:WO_3)]. As the temperature, the conductivity increases practically linearly up to ~ 65 °C at which an upward jump in conductivity was observed.

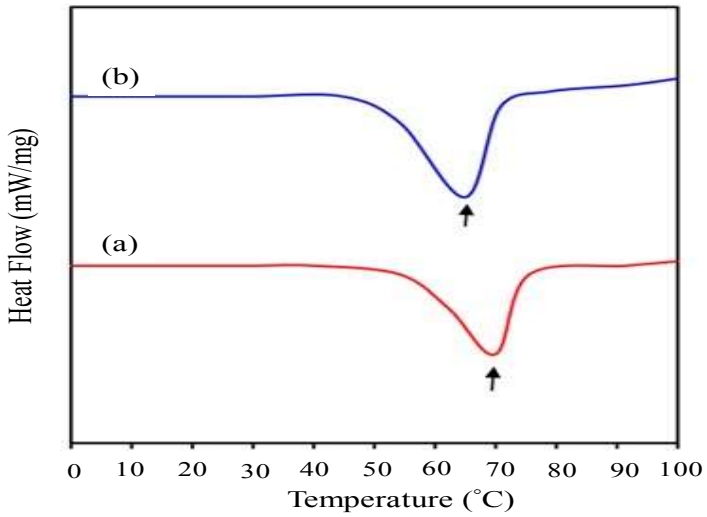

Figure 5. DSC Curves: (a) Pure PEO and (b) GPE OCC: 70 PEO: 30 [0.75AgI:0.25(Ag_2O:WO_3)].

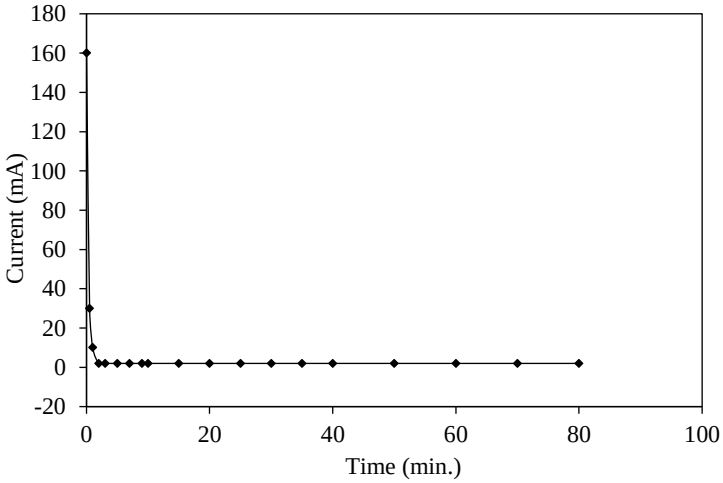

Figure 6. 'Current vs time' Plot for GPE OCC: 70 PEO: 30 [0.75AgI:0.25(Ag_2O:WO_3).

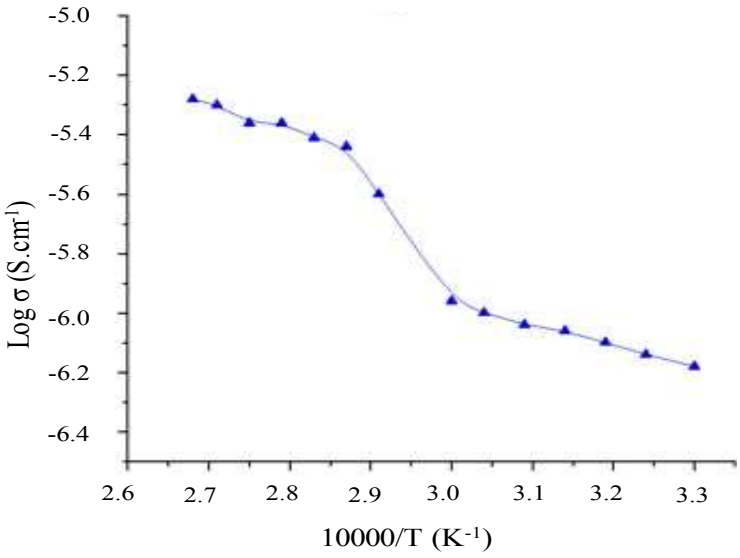

Figure 7. 'Log σ –1/T' Plot for GPE OCC: 70 PEO: 30 [0.75AgI:0.25(Ag_2O:WO_3)].

The abrupt change in conductivity at around this temperature is due to the phase transition from semi-crystalline to amorphous. Below the transition temperature, the plot governing the Arrhenius type behavior and can be expressed as:

$$\sigma (T) = 4.7\times10^{-2} \exp [-0.37/ kT] [Scm^{-1}] \quad (2)$$

where $E_a \simeq 0.37$ is the activation energy in eV for GPE OCC, which is low and it is indicative of easy ion transport in the system and hence this can be potentially used for device fabrication such as batteries, sensors etc.

CONCLUSIONS

A new silver ion conducting GPE: 70PEO:30[0.75AgI:0.25(Ag_2O:WO_3)] with ionic conductivity (σ) 6.5×10^{-7} S.cm^{-1} has been synthesized using a hot-press technique. The conductivity has increased by two orders of magnitude compared to that of pure PEO polymer. Glass-polymer complexations of the present GPEs were studied by SEM and DSC techniques. The characterization of ion transport behavior was done in terms of the basic ionic parameters: σ, μ, n, t_{ion} etc. The t_{ion} measurements indicates that 98% of Ag^+ ions are mobile in the system and this can be used for the fabrication of different electrochemical devices.

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