Effect of Glycerin on Electrical and Thermal Properties of PVA/Copper Sulphate Gel Polymer Electrolytes

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Abstract
Copper-ion conducting gel polymer electrolyte (GPE) systems based on polymer poly(vinyl alcohol) (PVA) and copper sulphate salt doped with glycerin as plasticizer have been synthesized by using solution casting technique. Differential scanning calorimetry (DSC) is used to examine the thermal effect of glycerin on a polymer electrolyte. With the addition of various quantities of glycerin, as a plasticizer in pure PVA and PVA + 20 wt% CuSO4 polymer electrolyte shows a decrease in the values of melting temperature, glass transition temperature and percentage of crystallinity. From TGA curves, it is observed that thermal degradation of the glycerin doped polymer electrolyte is shifted towards lower temperature when compared to pristine PVA and the weight loss of the polymer electrolyte increases with increase of glycerin concentration. From DTG analysis, the temperature of maximum decomposition for PVA is 283.4 °C and it is decreased by the addition of 20 wt% CuSO4 and upon increased concentration of the plasticizer from 1 to 3 mL of glycerin. For pure PVA and PVA + 20 wt% CuSO4, ε′ decreases with increasing glycerin concentration and is lowest at 3 mL glycerin concentration. The maximum ionic conductivity obtained was 9.39 × 10−4 S/cm for PVA + 20 wt% CuSO4 + 3 mL glycerin gel polymer electrolyte. The above results suggested that, the optimum conducting sample is suitable as separator in rechargeable batteries.

Keywords Gel electrolyte · PVA · Plasticizer · Glycerin · Flexibility

1 Introduction
In electrochemical devices, rechargeable batteries (RBs) have become an increasingly essential energy storage system [1]. The electrolyte which plays an important role in batteries, which allows ions to travel through to create the battery current [2]. Metal salts and organic solvents are the most common components of liquid electrolytes. However, there are several important considerations for practical applications, such as the safety of liquid electrolyte, particularly when the batteries are subjected to thermal, mechanical, or electrical abuse [3]. Gel polymer electrolytes (GPEs), which contain plasticizers such as ethylene carbonate, propylene carbonate, starch, ionic liquids, glycerin etc. are being explored as a viable alternative to currently available organic liquid electrolytes [4]. GPEs offers more benefits compare with liquid electrolytes and solid polymer electrolytes such as improved ionic conductivity at room temperature, high reliability, high flexibility, no leakage, and good performance. In GPEs, the liquid is immobilized in a polymeric matrix, which may reduce the risk of leakage as compared to commercial separators [5].

GPEs are a type of polymer electrolyte that combines the advantages of liquid and solid electrolytes in one package. GPEs have attracted a lot of attention as a dual nature function of electrolytes [6]. Combining heterogeneous (phase-separated) and homogeneous (uniform) gels results in high ionic conductivity and good interfacial characteristics in the liquid phase, as well as superior mechanical qualities in the solid phase [7]. The majority of GPEs exhibit a remarkable ionic conductivity on the order of 10−3 S/cm at ambient temperature [8]. Li et al. synthesized the composite microporous gel polymer electrolytes (CMGPEs) based on poly(vinylidene fluoride) doped with SiO2(Li+)3 When the content of SiO2(Li+) reached 5 wt% the ionic conductivity of the CMGPEs could reach 10−2 S/cm order of magnitude
at room temperature [9]. Poly(vinyl chloride) (PVC)/poly(ethyl methacrylate) (PEMA) blend-based gel doped and plasticized with zinc triflate [Zn(OTf)₂] salt and 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide (EMIMTFSI) ionic liquid was synthesized by Prasanna et al. For this GPE the highest ionic conductivity of 4.27 × 10⁻⁴ S/cm at ambient temperature [10]. In general, the polymer framework with metal ion transport occurs in the swelling gelled phase or liquid phase for heterogeneous GPEs, which can support the electrochemical performance of the battery cell involving GPEs [11].

PVA, PMMA, PVP, PAN, PEO, and PVDF are some of the polymers that have been used to synthesize polymer electrolytes. Poly(vinyl alcohol) (PVA) is a well-known water-soluble polymer with high transparency and outstanding mechanical properties that has been employed in a wide range of personal, industrial, and electrical applications. PVA is a polyhydroxy polymer that is frequently used in beneficial applications due to its ease of production and biodegradability. PVA can also form films, is hydrophilic, and has a high density of reactive chemical functionalities, making it ideal for chemical, irradiation, or thermal cross-linking. The O–H bonds found in PVA aid in the formation of polymer complexes. PVA also has a lot of good qualities, such as high mechanical strength, strong ionic conductivity, non-toxicity, biocompatibility, biodegradability, and ease of manufacture. [12]

Copper ion conducting polymer electrolytes with no discernible electronic conductivities are discovered, they could be used in solid polymer batteries. As an anode material in solid state batteries, copper has various benefits over metallic lithium. Copper is more environmentally friendly and less expensive than metallic lithium [13]. The literature survey reveals that the PVA complexes with copper salts are rare. The problem with polymer-salt complexes was low ionic conductivity. That we can enhance by plasticizers, composite addition, polymer blending, and in-situ polymerization techniques. This will lower the crystallinity of the resultant matrix as a result of higher chain flexibility of the polymeric backbone have all been utilized to improve the conductivity of PVA-based polymer electrolytes [14].

Different plasticizers have been used in various polymer electrolyte systems by different research groups. Out of all plasticizers, glycerin is one the good one to increase the performance of polymer electrolytes. In present paper, we report the gel polymer electrolytes prepared by the addition of glycerin to PVA-CuSO₄ (80–20). The purpose of this work is to emphasize the extraordinary effect occurring in the PVA-CuSO₄-glycerin gel polymer electrolytes. Our results demonstrate that the dispersion of glycerin in the PVA-CuSO₄ matrix leads to an increase in the ionic conductivity of the gel polymer electrolytes. The resultant electrolyte films have been characterized by DSC, TGA-DTG analyses.

The conductivity of the polymer electrolytes is measured using ac impedance technique.

2 Experimental Details

2.1 Materials

Poly(vinyl alcohol) (PVA) as a white powder with molecular weight 14,000 produced by Sigma-Aldrich, USA, ionic conductor Copper sulphate (CuSO₄) salt from Sigma-Aldrich, USA and glycerin (HOCH₂CH(OH)CH₂HO as a liquid plasticizer with density (1.26 g/cm³) received from India.

2.2 Preparation PVA Based GPE Films

The solution cast process was used to make gel polymer electrolyte samples. PVA (molecular weight of 14,000) was used as the polymer. CuSO₄ was added accordingly. Distilled water was employed as the solvent in this experiment. To get a homogenous solution, the mixture was agitated at room temperature for up to 10 h. After adding the needed amount of glycerin as a plasticizer, the solution was suspended and agitated for roughly 4 h. These solution mixtures were then poured into glass petri dishes and dried for 72 h, resulting in a thick film with thickness about ~ 65 μm that was peeled off and stored in desiccators to dry further. The composition used for making gel polymer electrolytes is shown in Table 1.

3 Characterization

The melting and glass transition temperature was measured using a Differential Scanning Calorimeter (DSC) powered by Mettler- Toledo, USA over a temperature range of 40 to 90 °C, and thermal stability was measured using a thermogravimetric analyzer (TGA-DTG, Mettler- Toledo, USA).

Table 1 Compositions of the pure PVA and PVA/CuSO₄/Glycerin electrolyte films

| Sample                        | PVA Weight (in gm) | CuSO₄ Weight (in gm) | Amount of glycerin |
|-------------------------------|--------------------|---------------------|-------------------|
| Pure PVA                      | 0.8                | 0                   | 0                 |
| PVA/20 wt%CuSO₄               | 0.64               | 0.16                | 0 mL              |
| PVA/20 wt% CuSO₄/1 mL glycerin| 0.64               | 0.16                | 1 mL              |
| PVA/20 wt% CuSO₄/2 mL glycerin| 0.64               | 0.16                | 2 mL              |
| PVA/20 wt% CuSO₄/3 mL glycerin| 0.64               | 0.16                | 3 mL              |
under N$_2$ gas and heating at a rate of 10 °C/min over a temperature range of 25 to 300 °C. The resistivities were measured at room temperature using GWINSTEK LCR-6100 with a frequency range of 1–100 kHz.

4 Results and Discussion

4.1 Thermal Analysis

The thermal effect of glycerin on a plasticized polymer electrolyte is investigated using differential scanning calorimetry (DSC). In Fig. 1, the DSC curves of pure PVA, PVA + 20 wt% CuSO$_4$, and PVA + 20 wt% CuSO$_4$ plasticized by 1, 2 and 3 mL of glycerin are presented. An endothermic peak corresponding to the melting temperature (T$_m$) and the glass transition temperature (T$_g$) of pure PVA is discovered at 73.12 °C and 66.02 °C, respectively. When 20 wt% of CuSO$_4$ salt was added to the polymer matrix, the melting temperature (T$_m$), glass transition temperature (T$_g$), and degree of crystallinity ($\chi_c$) were all increased.

The glass transition temperature (T$_g$) moved towards the lower temperature side when different amounts of glycerin were added to the prepared electrolyte membrane PVA + 20 wt% CuSO$_4$ polymer electrolyte. The decrease in T$_g$ of the polymer electrolyte with increased glycerin content suggests a weaker intermolecular connection between the glycerin, CuSO$_4$, and PVA, allowing the polymer network to move more segmentally by making the polymer matrix more flexible [15, 16]. Glycerin’s plasticizing action produces a reduction in the T$_m$ and T$_g$ of polymer gel electrolyte membranes when it is added. The degree of crystallinity ($\chi_c$) and melting temperature increased when 20 wt% of CuSO$_4$ salt was added to the polymer matrix (T$_m$) were initially elevated, but this increased crystallinity and melting temperature was successfully controlled by the addition of glycerin and achieved the lowest value (approximately 11.65%) for the membrane contains a higher amount of glycerin (3 mL).

The DSC parameters are presented in Table 2. The relative percentage of crystallinity ($\chi_c$) has been calculated with the Eq. (1).

$$\chi = \frac{\Delta H_f}{\Delta H_{fo}} \times 100$$

where $\Delta H_{fo}$ = 2.65 (J/g). The calculated heat of fusion ($H_f$), melting temperature (T$_m$), and percentage crystallinity ($\chi_c$) values are shown in Table 2.

The heat of fusion ($H_f$), melting temperature (T$_m$), and percentage of crystallinity all decreased when plasticized with 1, 2 and 3 mL of glycerin. This is owing to a decrease in PVA polymer electrolyte crystallinity, which is a well-known favorable condition for boosting ionic conductivity.

TGA thermographs of pure PVA, PVA + 20 wt% CuSO$_4$, and PVA + 20 wt% CuSO$_4$ plasticized by 1, 2, and 3 mL of glycerin gel polymer electrolytes are shown in Fig. 2. The plot clearly shows two stages of weight loss, the first of which is a 5% weight loss at 54.3 °C and the second of which is a maximum weight loss at 285.8 °C for pure PVA and PVA + 20 wt% CuSO$_4$, both of which can be attributed to evaporation of water and degradation of PVA by the polymer chain’s dehydration reaction [16]. Water absorption, heat degradation of functional groups, and thermal oxidation of the polymer backbone are the processes in which the PVA + 20 wt% CuSO$_4$/Glycerin gel polymer electrolyte loses weight [17].

| Samples | Melting temperature (°C) | Glass transition temperature (°C) | $\Delta H_f$ (J/g) | Percentage of crystallinity ($\chi_c$) |
|---------|--------------------------|-------------------------------|-----------------|-------------------------------------|
| Pure PVA | 73.12 | 66.02 | 85.23 | 32.12 |
| PVA + 20 wt% CuSO$_4$ | 74.24 | 67.34 | 87.34 | 32.95 |
| PVA + 20 wt% CuSO$_4$/1 mL glycerin | 64.78 | 53.12 | 50.12 | 18.91 |
| PVA + 20 wt% CuSO$_4$/2 mL glycerin | 59.89 | 42.62 | 34.22 | 12.91 |
| PVA + 20 wt% CuSO$_4$/3 mL glycerin | 54.34 | 37.22 | 30.89 | 11.65 |

**Fig. 1** DSC curves of pure PVA, PVA + 20 wt% CuSO$_4$, and PVA + 20 wt% CuSO$_4$ plasticized with 1, 2 and 3 mL of glycerin

**Table 2** DSC parameters of pure PVA, PVA + 20 wt% of CuSO$_4$ and PVA + 20 wt% of CuSO$_4$ plasticized by 1, 2 and 3 mL of glycerin
All membranes lose weight before reaching 50 °C in the first phase, which can be attributed to the evaporation of bound water in the samples. The release of the quaternary ammonium group’s degraded product causes weight loss in the second phase in the range 124 to 180 °C [15]. Weight loss in the third phase at 200–230 °C is assumed to be caused by the release of residual quaternary ammonium group. At 270–300 °C, the fourth and final weight loss was discovered, which was caused by polymer chain breakdown [15, 17].

The weight loss of the polymer electrolyte increases as the glycerin concentration rises, which is due to scission monomers and bonds in the polymeric backbone cracking and loss of dopant due to heat energy [18]. The decomposition of organic polymer chains, both the hard segment of PVA linkage and the soft segment from glycerin, was attributed to the decomposition of plasticizer with polymer [17, 19].

The DTG thermograms of pure PVA, PVA + 20 wt% CuSO₄, and PVA + 20 wt% CuSO₄ plasticized by 1, 2, and 3 mL glycerin are shown in Fig. 3. The maximal decomposition temperature of PVA is 283.4 °C, and this temperature drops when 20 wt% of CuSO₄ is added and the plasticizer concentration is increased from 1 to 3 mL. For PVA + 20 wt% CuSO₄ had a T_{max} of 272 °C, which dropped to 261 °C for the electrolyte PVA + 20 wt% CuSO₄/3mL glycerin. This phenomenon has been linked to the low T_g value of the plasticized gel polymer electrolyte. The dipole-dipole interaction of polymer chains was reduced when glycerin was added, because it softens the polymer chain’s backbone and lowers the polymer’s T_g. The same pattern was found by Abdulkadir et al. [20].

Figure 4 shows the results of separating CuSO₄ salt to cations and anions in PVA based GPE. The GPEs were created by injecting conducting salt (CuSO₄) into the polymer host (PVA) and plasticizing it with glycerin, as described in the experimental section. PVA and glycerin with hydroxyl or polar groups (−OH) produced a covalent dative bond with the CuSO₄ cations in the electrolytes [20, 21]. This is because polymer materials (PVA) and plasticizers (glycerin) have −OH groups in their macromolecular chains and three-dimensional networks that can react with various inorganic salts [22, 23]. The presence of polar −OH groups enable chemical (complexing processes) and physical connections (via H bonding, Van der Waals dipole–ion interactions, or dipole–dipole interactions) [24]. CuSO₄ dissociates into cations (Cu^{2+}) and anions (SO₄^{2−}) when dissolved in solvent is shown in Eq. 2.

\[
\text{CuSO}_4 \rightarrow \text{Cu}^{2+} + \text{SO}_4^{2-}
\] (2)

The cations formed when coordination with the polar groups (−OH) of the plasticized polymer matrix, resulting in a complex molecule, as shown in Fig. 4. Because of the interaction between the plasticized polymer polar
The dielectric constant decreases with increasing frequency and reaches a stable value at high frequencies, resulting in the frequency-independent \( \varepsilon' \) decrease with increasing glycerin content for pure PVA and PVA + 20 wt% \( \text{CuSO}_4 \) in the above frequency range 5 and 6. It achieves a high value at 3 mL glycerin concentration. This type of behaviour has also been observed in electrical conductivity investigations (see Table 3). The increase in \( \varepsilon' \) is due to a reduction in mobile charge carrier density. The addition of plasticizer to the polymer salt system introduces additional ions, lowering the density of charge carriers and thereby lowering the gel polymer electrolyte system’s dielectric constant [24].

We can see from Table 3 that when pure PVA and PVA + 20 wt% \( \text{CuSO}_4 \) are exposed to an electric field, the cations from the salts can migrate from one coordinated site to another. This is due to the weak coordinates of the cations with sites along the polymer chain [19]. According to prior research [24], ions, primarily cations, connected to functional groups of the host polymer chains can move along the polymer backbone by recoordination. After that, the polymer chains are folded to form tunnels in which the functional groups locate and coordinate the cations. Cations can readily flow via these tunnels, which form channels [17, 26].

Conducting salts have also been found to reduce the number of active centers in polymer chains, diminishing intermolecular and intramolecular interactions [17, 30]. As a result, the stiffness of the host polymer will be lowered, and the mechanical and thermomechanical properties of the polymer will be altered [21, 26]. Similarly, introducing high-conducting salts reduces the glass transition temperature (\( T_g \)) of the polymer system (as would be stated in DSC data) [17, 31]. As a result, crystallinity will decrease and salt dissociation capacity will increase, resulting in enhanced charge carrier transport and greater ionic conductivity [25, 30]. An increase in glycerin concentration, which results in the formation of a complex between the polymer matrix and the conducting salt (PVA + \( \text{CuSO}_4 \)), would raise entropy, which will improve the composite’s segmental motion. Reduced crystallinity (greater
flexibility) and enhanced electrolyte ionic conductivity will result from increased segmental motion [26, 32].

5 Conclusions

In this work, PVA + 20 wt% CuSO4 + Glycerin gel polymer electrolytes have been produced with great flexibility, low glass transition temperature, low dielectric constant and strong ionic conductivity. The glass transition temperature (Tg) of the GPE films shifted to the lower temperature side with the addition of glycerin as a plasticizer in pure PVA and PVA + 20 wt% CuSO4 gel polymer electrolyte. When compared to pristine PVA, the glycerin doped gel polymer electrolytes thermal decomposition is moved towards lower temperatures, and the weight loss of the gel polymer electrolyte increases with the glycerin concentration. By the incorporation of glycerin to a PVA + 20 wt% CuSO4 film, the conductivity was increased to the order of 10−4 S/cm at room temperature. These results suggested that, this would be suitable separator for rechargeable batteries.

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Declarations

Conflict of interest  The authors have not disclosed any competing interests.

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