Conductivity and transport properties of starch/glycerin-MgSO₄ solid polymer electrolytes

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The thin films of solid polymer electrolytes were based on corn starch doped with magnesium sulphate (MgSO₄) with different ratios of polymer and salt added. They were prepared using a single-solvent technique. The glycerin was added to the mixture of the solution to offer more elasticity to the polymer film and by increasing the flexibility of the thin-film membrane. The conductivity and electric studies were carried out on these thin films to understand the ion transport properties of the polymer electrolytes. The highest conductivity obtained was 8.52 \times 10^{-5} \text{ S cm}^{-1}, which for the 35 wt. % MgSO₄ salt-doped polymer electrolyte system at room temperature. From the evaluation on the transport properties, the conductivity of the system was generally influenced by \( n, \mu \) and \( D \) of charge carriers. MgSO₄ helped to increase the ionic conductivity and further increase the salt content, while the diffusion coefficient and mobility of charge carriers were increased.

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1. Introduction

Solid polymer electrolyte thin films are solid ionic conductors that are made by the dissolving salts in appropriate polymers. This outcome results in solid polymer electrolytes having good flexibility, mechanical stability, light weight, durability, low cost, and corrosion-free properties (Vincent, 1987; Xu, 2014; Kulshrestha et al., 2014; Hassan and Ting, 2018). Solid polymer electrolytes are seen as a vital component; having a wide range of applications in electrochemical devices such as high energy density batteries, super capacitors, fuel cells, sensors, organic transistor and dye-sensitized solar cells (Dai et al., 2018; Verma and Sahu, 2017). Extensive exploration to cultivate a new solid polymer electrolyte with good electrical conductivity and environmental stability has been on the rise, specifically with regard to their ionic, dielectric and transport properties (Prajapati and Gupta, 2009; Zamri et al., 2014; Chai and Isa, 2016; Bandara and Mellander, 2011; Koduru et al., 2017; Gohel and Kanchan, 2018). Electrochemical impedance spectroscopy (EIS) technique is among the most commonly used technique for the evaluation and study of the ionic conductivity, dielectric, and transport properties of solid polymer electrolytes. EIS is a great technique for distinguishing a comprehensive variation of electrochemical systems and for defining the contribution of electrodes or electrolytic processes in these systems (Ribeiro et al., 2015; Hassan and Noruddin, 2018). Many past research works have used EIS to investigate the mentioned parameters.

They found that the irradiated samples had altered significantly on their conductivity and dielectric properties significantly. In the investigation of PVDF-HFP:PMMA-LiClO₄ electrolyte system, Gohel and Kanchan (2018) revealed that at room temperature, the conductivity of 7.5 wt.% LiClO₄ can reach as high as 2.83 \times 10^{-4} \text{ S cm}^{-1} and the ionic conductivity, dielectric and transport properties had changed with the change in temperatures and salt content. The effect of fume silica on PVA:NaI electrolyte system had been studied by Kulshrestha et al. (2014). From the EIS analysis, they found that the maximum conductivity was approximately 3.8 \times 10^{-3} \text{ S cm}^{-1} at room temperature, which was obtained for PVA:NaI (60:40) film containing 0.5% fumed silica nanoparticles and from the dielectric relaxation studies related to temperature. It can be seen that the relaxation time shifted towards higher values for the samples of nanocomposite polymer electrolyte films.
Nowadays, the EIS analysis technique has been improved where Bandara and Mellander (2011) proposed a new approach by employing impedance spectroscopic data that not only can determine the ionic conductivity and dielectric properties, but can also be used to calculate the mobile charge carrier concentration and mobility in different ways. Interestingly, Arof et al. (2014) discovered an alternative method based on impedance spectroscopy to determine transport properties of polymer electrolyte. In the study, they revealed that their novel method was more accurate and could be proven using another technique based on Fourier Transform Infrared (Ftir) analysis. As cited above, there is no doubt that EIS is one of the non-destructive and powerful techniques to investigate ionic conductivity, dielectric, and transport properties which should be applicable for a wide range of electrolytes including ionic solid polymer electrolytes (Hassan and Arof, 2005).

In the present study, the effect of MgSO4 salt concentration were studied based on conductivity and transport properties of a new type of solid polymer electrolyte film in which the corn starch is complexed with magnesium sulfate (Starch-MgSO4 system). The picking of the MgSO4 salt is principally because of its ready accessibility, lesser harmfulness, and high potential in electrochemical applications (Byrne et al., 2017; van Essen et al., 2009; Wang et al., 2017; Massé et al., 2015; Lota et al., 2013; Hongois et al., 2011; Zhu et al., 2018; Mahon et al., 2017).

2. Experimental method

The solid polymer thin films were prepared using a single solvent by the solution casting technique (Fig. 1). The mixture was stirred constantly using magnetic stirrers at a temperature of 60°C, to obtain the desired homogenous solution. Subsequently, the solution was transferred into different plastic Petri dishes and was left for the evaporation process to form solid state film. After 24 h, a mechanically strong, transparent, and free-standing film was obtained. The details of the compositions of corn starch doped with MgSO4 (Sigma-Aldrich, 99.99% purity) and their resulting mechanical properties are as per listed in Table 1.

### Table 1: Composition and mechanical strength of starch-MgSO4 films

| MgSO4 content (wt %) | Solvent (ml) | Corn starch (g) | MgSO4 (g) | Film Strength |
|----------------------|-------------|----------------|-----------|--------------|
| 5                    | 30          | 1              | 0.053     | Very good    |
| 10                   | 30          | 1              | 0.111     | Very good    |
| 15                   | 30          | 1              | 0.177     | Very good    |
| 20                   | 30          | 1              | 0.250     | Very good    |
| 25                   | 30          | 1              | 0.333     | Very good    |
| 30                   | 30          | 1              | 0.429     | Very good    |
| 35                   | 30          | 1              | 0.548     | Very good    |
| 40                   | 30          | 1              | 0.667     | Good         |
| 45                   | 30          | 1              | 0.834     | Good         |

The ionic conductivity was measured using HIOKI 3532-02 LCR Hi-Tester which was interfaced to the computer. This tester was used to determine the conductivity of the polymer thin film membrane over a frequency range of 50 Hz to 1MHz.

The prepared samples were cut into 2 cm diameter size and placed between two stainless steel blocking electrodes of the sample holder. The bulk resistance, $R_b$, was obtained from the real impedance value of the Cole–Cole plot. The ionic conductivity was calculated from the measured bulk resistance, area, and thickness of the polymer film using the following formula:

$$\sigma = \frac{1}{R_b A}$$  \hspace{1cm} (1)

where $I$ is the thickness of the polymer electrolyte (cm) measured by the micrometre screw gauge, $A$ is the area of the blocking electrode contact (cm$^2$), and $R_b$ is the bulk resistance ($\Omega$). A digital micrometre-screw gauge was used to measure the thickness of the polymer films.

The dielectric constant, $\varepsilon_r$ for a Starch-MgSO4 system is defined by:

$$\varepsilon_r = \frac{Z_i}{(Z_i^2+Z_o^2)^{1/2}}$$  \hspace{1cm} (2)

where $Z_i$ and $Z_o$ are the real and imaginary part of complex permittivity, $d$ is half the thickness of the polymer electrolyte, $\omega$ is equal to $2\pi f$ or the angular frequency corresponding to the minimum in the imaginary impedance, $f$ being the frequency in Hz, $\varepsilon_o$ is the permittivity of free space ($8.85 \times 10^{-14}$ F cm$^{-1}$), $A$ is the electrolyte-electrode contact area.

Transport properties ($D, \mu$ and $n$) of the Starch-MgSO4 system are determined by Eqs. 3, 4, and 5:

$$D = \frac{(k_2 + \varepsilon_o A)^2}{\tau_2}$$  \hspace{1cm} (3)

where, $D$ is diffusion coefficient, $k_2$ is capacitance, $\varepsilon_o$ is the vacuum permittivity, $A$ is the electrode/electrolyte contact area and $\tau_2$ is a time constant corresponding to the maximum dissipative loss curve which is equal to $1/\omega_2$.

$$\mu = \frac{\varepsilon (k_{2\text{static}})^2}{k_0 \tau_2}$$  \hspace{1cm} (4)

here, $\mu$ is mobility of the charge carriers, $k_0$ is the Boltzmann constant ($1.38 \times 10^{-23}$ J K$^{-1}$), $T$ is the absolute temperature in Kelvin and $e$ is the electron charge ($1.602 \times 10^{-19}$ C).

The number density of charge carriers ($n$) can be obtained using the Eq. 5:

$$n = \frac{\sigma k T}{k_0 e^2}$$  \hspace{1cm} (5)

$k_2$ in Eqs. 3, 4, and 5 were calculated from

$$k_2 = \frac{\lambda_0}{\varepsilon_r \varepsilon_o A}$$  \hspace{1cm} (6)

where, $\lambda_0$ is the Debye length and its calculation is obtained from Lin et al. (2012). Fig. 2 describes the
The exact location of the Debye length in the solid electrolyte film during EIS measurement. The Stern layer has a very small thickness (< 1nm), so it is not taken into consideration in this study.

Fig. 1: A schematic diagram to prepare starch-MgSO₄ complex films

Fig. 2: A schematic diagram for arrangement of starch-MgSO₄ films during electrochemical impedance measurement

3. Results and discussion

The electrical properties of the starch-MgSO₄ films were studied using the impedance spectroscopy analysis. Eq. 1 was used to calculate the conductivity of each sample and its corresponding electrical conductivity variation was used as a function of the MgSO₄ concentration at room temperature as depicted in Fig. 3. It can be seen from the Fig. 3 that the addition of MgSO₄ had significantly increased the ionic conductivity. By adding 5-15 wt. % MgSO₄ to the starch film, the ionic conductivity gradually increased to 2.59 x 10⁻⁶ S cm⁻¹. The conductivity kept increasing (4.70 x 10⁻⁶) for 20 wt. % MgSO₄. Additional MgSO₄ with 25 wt. % to 35 wt. % evidently increased the ionic conductivity to a high level of (8.52 x 10⁻⁵ S cm⁻¹). The conductivity values started to decrease after further addition of 40 to 45 wt. % MgSO₄.

By using the technique as explained by Arof and coworkers, the dielectric constant, εᵣ, for each sample was obtained by a plot of the real part of the complex permittivity, εᵣ versus frequency, f and were substituted in Eq. 2. In this case, the real part of the complex permittivity, εᵣ, for all samples showed a constant value in between log f = 5.5 and 6.0. Therefore, the values of the dielectric constant, εᵣ, for all samples were taken at 630 kHz and were calculated at a location between 1 and 130, respectively. Another important parameter that had to be determined in the first stage was λₒ and thus it was calculated using the equation mentioned by Lin et al. (2012). These values (εᵣ, λₒ) were used to calculate the parameters of the diffusion coefficient, mobility of ions, the number density of charge carriers and capacitance of samples using Eqs. 3, 4, 5, and 6.

Table 2 shows the values of λₒ, D, μ, n and k₂ of the starch-MgSO₄ electrolytes as a function of MgSO₄ concentration. It was observed that the λₒ had a value in between 1.66 x 10⁻⁸ and 6.38 x 10⁻⁷ cm. The λₒ for the highest conductivity among the samples was 6.38 x 10⁻⁷ cm. The calculated values of k₂ were in between 6.19 x 10³ and 6.49 x 10⁴ F⁻¹ with the highest conductivity found in sample 1.76 x 10⁴ F⁻¹. The value of diffusion coefficient for the highest conductivity sample was 1.03 x 10⁻¹⁸ cm² s⁻¹ with the value located in between 1.02 x 10⁻¹⁸ and 5.18 x 10⁻¹⁷
cm² s⁻¹. It is can be seen that the starch-MgSO₄ films system had mobility values in between 4.08 x 10⁻¹⁰ cm² V⁻¹ s and 2.02 x 10⁻⁵ cm² V⁻¹ s with the highest conductivity found in sample 4.03 x 10⁻⁷ cm² V⁻¹ s. The calculated value of the number density of charge carriers for this system was in between 1.24 x 10²⁰ cm⁻³ and 3.99 x 10²³ cm⁻³ with the highest conductivity sample recording the value of 1.31 x 10²² cm⁻³.

| MgSO₄ content (wt%) | λₑ(cm) | kₑ(F⁻¹) | τₑ(s⁻¹) | D(cm² s⁻¹) | µ(cm² V⁻¹s) | n(cm⁻³) |
|---------------------|---------|----------|---------|------------|-------------|---------|
| 5                   | 1.67 x 10⁻⁸ | 6.32 x 10⁻⁵ | 2.65 x 10⁻⁸ | 1.05 x 10⁻¹¹ | 4.08 x 10⁻¹⁰ | 3.18 x 10²³ |
| 10                  | 7.45 x 10⁻⁸ | 6.19 x 10⁻⁵ | 1.22 x 10⁻⁸ | 4.54 x 10⁻¹⁰ | 1.77 x 10⁻⁷ | 7.74 x 10²⁰ |
| 15                  | 1.08 x 10⁻⁸ | 6.49 x 10⁻⁵ | 3.18 x 10⁻⁸ | 1.03 x 10⁻¹¹ | 4.00 x 10⁻¹⁰ | 3.99 x 10²³ |
| 20                  | 6.99 x 10⁻⁹ | 3.35 x 10⁻⁵ | 2.65 x 10⁻⁸ | 1.85 x 10⁻¹⁰ | 7.19 x 10⁻⁸ | 4.03 x 10²³ |
| 25                  | 7.76 x 10⁻⁹ | 5.35 x 10⁻⁵ | 5.30 x 10⁻⁸ | 1.14 x 10⁻⁹ | 4.42 x 10⁻⁸ | 3.97 x 10²² |
| 30                  | 4.53 x 10⁻⁸ | 1.52 x 10⁻⁵ | 3.18 x 10⁻⁸ | 6.45 x 10⁻¹⁰ | 2.51 x 10⁻⁷ | 1.36 x 10²¹ |
| 35                  | 4.53 x 10⁻⁷ | 1.76 x 10⁻⁵ | 3.94 x 10⁻⁸ | 1.03 x 10⁻⁸ | 4.03 x 10⁻⁷ | 1.30 x 10²² |
| 40                  | 4.58 x 10⁻⁷ | 5.08 x 10⁻⁵ | 8.84 x 10⁻⁸ | 1.22 x 10⁻⁷ | 4.74 x 10⁻⁷ | 7.15 x 10²⁰ |
| 45                  | 4.91 x 10⁻⁷ | 5.31 x 10⁻⁵ | 4.65 x 10⁻⁷ | 5.18 x 10⁻⁷ | 2.02 x 10⁻⁵ | 1.24 x 10²⁰ |

The graphs of D, µ, and n were plotted versus the salt content; using the Origin software and can be seen in Fig. 4. From this software, the non-linear curve fitting technique based on Boltzmann and Dose-response functions were used to obtain the fitting curves. It can be observed that the n of the system is opposite trend to the conductivity, whereas the D and µ had followed the trend of conductivity value, respectively. It seems that the increasing trend of conductivity is due to an increase in the D and µ of charge carriers as the MgSO₄ concentration increased in the samples. As discussed by Hassan and coworkers, the increasing trend of ionic conductivity had been contributed by two main factors, the amorphous structure of complex samples and the sufficient ionic conductors in the close system (Hassan et al., 2018). For the amorphous structure, it could act as a supportive medium for fast ionic movement. A few researchers claimed that the ionic conductivity is higher in amorphous complex structures as compared to crystalline polymer salt complex; this is due to the interface resistance is low in between the solid electrolyte and the electrode surface (Bruce and Vincent, 1993; Maurya et al., 1992; Reddy et al., 2007; Srivastava et al., 1995). Another aspect is associated to the D and µ of ionic conductors in the electrolyte system. The increase in conductivity with the salt content is due to the increase in diffusion coefficient and mobility of charge carrier as shown in Fig. 4. As for the decreasing trend in conductivity, this could be because of the reduction on the number of charge carriers and increase the empty space in the electrolyte system (Rajendran et al., 2000). The wrecked film and the increase in crystallinity of film might be the extra factors of the conductivity drop in this electrolyte system.

As for comparison purposes, the conductivity and transport property values of our prepared starch-MgSO₄ films in this study and those other systems reported in the literature are updated in Table 3.

The results studied here showed that the current Starch-MgSO₄ films had high conductivity (8.52 x 10⁻⁵ S cm⁻¹) and comparable transport properties (D = 1.04 x 10⁻⁸ cm² s⁻¹, µ = 4.03 x 10⁻⁷ cm² V⁻¹ s, n = 1.31 x 10⁻²² cm⁻³) to those studies in similar starch based compounds. These results are important as a reference for current and future work, and most significant since such information is seldom documented in other reports.

![Fig. 4: Plots of diffusion coefficient, D, charge carrier mobility, µ and charge carrier number density, n, against the MgSO₄ concentration](image)

4. Conclusion

As seen in this study, polymer electrolytes based on corn starch and magnesium sulphate were prepared via a solution casting method. The transport properties of the corn starch and magnesium sulphate were determined. Magnesium sulphate contents with Mg²⁺ mobile ions directly affected transport parameters and ionic conductivity. The addition of MgSO₄ resulted in an increase of diffussion coefficient and mobility of mobile ions which are main reasons to increase and enhancement in ionic conductivity. The highest conductivity was achieved at 8.52 x 10⁻⁵ S cm⁻¹.
Table 3: Comparison of the conductivity and transport values of hybrid starch-MgSO₄ film prepared in this work with other systems which reported in literature

| System                  | Conductivity (S cm⁻¹) | μ (cm² V⁻¹ s⁻¹) | n (cm⁻¹) | Reference |
|-------------------------|------------------------|----------------|----------|-----------|
| Corn starch-MgSO₄      | 8.52 × 10⁻⁵            | 1.04 × 10⁻⁶    | 4.03 × 10⁻⁵ | This work |
| Rice Starch-Li        | 4.68 × 10⁻⁵            | 5.53 × 10⁻⁹    | 2.15 × 10⁻⁵ | (Khammirzaei and Ramesh, 2013) |
| CMC-NH₄Br-PEG         | 2.48 × 10⁻⁴            | 1.40 × 10⁻⁸    | 1.80 × 10⁻⁷ | (Zainuddin et al., 2018) |
| CMC-NH₄F             | 1.52 × 10⁻⁶            | 9.66 × 10⁻⁷    | 1.12 × 10⁻⁵ | (Ramli and Isa, 2016) |
| Starch-chitosan-NH₄   | 3.04 × 10⁻⁴            | ~3.45 × 10⁻⁹   | 5.50 × 10⁻⁸ | (Yusof et al., 2014) |
| Potato Starch/Chitosan-LiCF₄SO₄ | 7.11 × 10⁻⁷ | 1.12 × 10⁻⁹ | 4.38 × 10⁻⁵ | (Navaratnam et al., 2015) |
| Cellulose Acetate-NH₄NO₃ | 1.02 × 10⁻³ | 3.28 × 10⁻⁶ | 9.84 × 10⁻⁵ | (Monisha et al., 2017) |
| Chitosan-NH₄NO₃-Al₂SiO₅ | 2.10 × 10⁻⁵ | ~1.51 × 10⁻⁸ | ~3.52 × 10⁻⁷ | (Majid et al., 2005) |
| Corn Starch-Na-GO      | 2.61 × 10⁻³            | ~              | ~        | (Shahrudin and Ahmad, 2016) |

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Compliance with ethical standards

Conflict of interest

The authors declare that they have no conflict of interest.

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